THE BRIX-FREE WATER CAPACITY AND SORPTION BEHAVIOUR OF FIBRE COMPONENTS OF SUGAR CANE (*Saccharum officinarum*)

by

YIN LUN WONG SAK HOI

B.Sc. (University of Hull, UK), M.Sc. (University of London), C Chem, FRSC

A thesis submitted in fulfilment of the academic requirements for the degree of Doctor of Philosophy in the School of Chemistry,
University of KwaZulu-Natal, Durban.

2008

ABSTRACT

Milling data from sugar factories in Mauritius were examined from 1960 to 2004 to assess the trend in the quality of cane received at mills and the change in factory performance. A deterioration in overall quality was apparent due to the increased level of extraneous matter delivered in the cane supply. Comparison was made with available data from other countries in the world, notably those of South Africa and Australia.

Controlled addition of extraneous matter to clean cane was effected under laboratory conditions to determine the relative impact of dry leaves, green leaves and cane tops on the quality of cane and the resulting juice, and to predict through derived equations, their impact on cane processing. The addition of dry leaves was found to have the most adverse effect followed by green leaves and cane tops. In the case of dry leaf addition to cane the detrimental effects were found to be masked by an increase in the concentration of solutes in the juice extracted. This phenomenon was thought to be due to the selective sorption of water (so-called Brix-free water) by dry leaves. To test this assertion, the sugar cane stalks of four different cane varieties aged 52, 44 and 36 weeks were separated into their component parts by means of a method devised in this work. There were nine component parts: stalk fibre, stalk pith, rind fibre, rind fines, top fibre, dry leaf fibre, dry leaf fines, green leaf fibre and green leaf fines which, on characterisation by Fourier transform infrared spectroscopy and scanning electron microscopy, were very similar except that stalk pith was more flaky and had a higher surface area than the others.

Various analytical techniques were tested for the determination of Brix-free water. The most convenient method proved to be a refractometric method which was improved so as to be applicable to the wide range of cane components fibres studied. Statistical analysis of the Brix-free water content of the separated samples showed that when the combined effect of fibre and pith in the cane stalk of three ages was considered, the four cane varieties were not different. This was not the case for dry leaf, green leaf, top and rind. Of the nine cane components, stalk pith exhibited the highest Brix-free water value of about 20 g/100 g fibre, whereas all the other components exhibited values of about 15 g/100 g fibre, which are much lower than the traditionally accepted value of 25% for cane. The latter was found to be the fibre saturation point of bound water determined at 20 ^{o}C, which is the sum of

dissolved and hydrated waters, and which is normally greater than the Brix-free water value as determined in this work.

The water sorption characteristics of the various cane component parts were further investigated by making measurements to determine the equilibrium moisture contents at various water activity values. These data were used to construct adsorption isotherms. These were fitted to 17 existing isotherm models, of which two, namely, the Hailwood-Horrobin and Guggenheim-Anderson-de Boer models, gave the best fit.

The sorbed water was subsequently characterised in terms of various parameters, namely, the monolayer moisture content, the number of adsorbed monolayers, the percentage of bound water, the total surface area for hydrophilic binding, the heats of sorption of the monolayer and multilayers, the net and total isosteric heats of sorption and the entropy of sorption.

From the monolayer moisture content and the amount of "hydrated water" as calculated from the Hailwood-Horrobin model, it is clear that at EMC values between 0 and 5% (a_w = 0 – 0.3), the non-freezable water is tightly bound to the surface of the fibre. The second region starts at EMC values from 5% to 10 – 15% (a_w = 0.3 to 0.6 – 0.8) depending on the cane components, and the bound water in this region is termed the freezable water. The third type of water is essentially free water, it exists after the second region and ends at EMC values of about 25%. From this study, it is apparent that the Brix-free water as measured in this work measures the amount of water bound in the first two regions.

PREFACE

The experimental work described in this thesis was carried out in the laboratories of the Mauritius Sugar Industry Research Institute (MSIRI) from January 2000 to March 2005.

These studies represent the original work of the author and have not been submitted in whole or in part to any other university/tertiary institution as candidature to the award of a degree/diploma. Where use was made of the work of others, it has been duly acknowledged in the text.

ACKNOWLEDGEMENTS

I am particularly grateful to Professor Bice Martincigh of the School of Chemistry, University of KwaZulu-Natal, and Dr Raoul Lionnet for their supervision of this work, and Dr Louis Jean Claude Autrey, who acted as my local supervisor. They all provided invaluable guidance and encouragement.

I wish to record my sincere thanks to Drs Louis Jean Claude Autrey and René Ng Kee Kwong, respectively the former and the present Director of the Mauritius Sugar Industry Research Institute (MSIRI) for extending resources and facilities to me in the pursuing of this work.

Thanks are also due to Mrs Cheenta Ramnawaz for assistance in the statistical analysis of data and modelling by means of the non-linear regression procedure of Sigma Plot (SPSS Inc.), Mr Rasack Nayamuth, Head of Plant Physiology Department of the MSIRI, for providing the cane materials for fibre extraction, and the members of the Sugar Technology Department of the MSIRI, who helped in the fibre extraction.

I am indebted to the Late Dr Fiona Graham for carrying out the scanning electron microscopic examination of the extracted cane fibres.

I am also grateful to Mrs Michaela Chung Po Chuen for typing the thesis and artwork, and Mr Ronald Bégué for photographs.

TABLE OF CONTENTS

LIST OF FIGURES

Page

Page

Page

Page

LIST OF TABLES

Page

Page

Page

GLOSSARY OF TERMS

Absorption is the incorporation of a substance in one state into another of a different state (e.g. liquids being absorbed by a solid or gases being absorbed by water).

Adsorption is the physical adherence or bonding of ions and molecules onto the surface of another molecule. Unless it is clear absorption or adsorption process is operative, sorption is the preferred term.

Adsorption and desorption are used to indicate the direction from which the equilibrium states have been approached.

Adsorption hyteresis arises when the amount adsorbed is not brought to the same level by the adsorption and desorption approach to a given "equilibrium" pressure or bulk concentration.

Adorption isotherm is the relationship, at constant temperature, between the amount adsorbed by a substrate (adsorbent) and the equilibrium pressure, or concentrations of a fluid (adsorbate).

Ash % cane is the ash content in cane. Ash is the residue remaining after burning off all organic matter. Ash may be determined as carbonated ash, conductivity ash or sulfated ash.

Bagacillo are very small particles of bagasse separated either from pre-clarification juices or from the final bagasse for filtration or other purposes.

Bagasse is the residue obtained from crushing cane in a mill. Depending on the number of mills, it is referred to as first mill bagasse, second mill bagasse, etc.

Boiling house recovery is the percentage of the pol in mixed juice that passes into the sugar produced, whereas the percentage of the pol in cane that passes into mixed juice is termed extraction. The product of these two is known as overall recovery.

Brix of a pure solution is the concentration of a pure sucrose solution in water (expressed by mass as parts of sucrose per 100 parts of solution), having, at a given temperature, the same density as the solution under examination.

Brix (refractometric) is the percentage by mass of soluble solid matter (sucrose and soluble non-sucrose) in solution as indicated by a sugar refractometer or as derived from the refractive index of a solution and reference to tables of equivalent percent sucrose and refractive indices.

Brix-free water or "hydrated water" as referred to in South Africa, or "adsorption water" or "hygroscopic water" as in Australia, is defined as the water strongly adsorbed onto the cane fibre and, unavailable for dissolving the soluble components in sugar cane. It cannot be separated from the natural cane fibre by mechanical means, only at elevated temperatures, and it is assumed to be 25% on dry fibre.

Cane crushing rate is the rate at which cane is crushed by the mills.

Chemisorption is the chemical adsorption process in which the adsorbed molecules are attached by strong chemical bonding.

Desorption – see adsorption.

Dextran is a high molecular mass polysaccharide formed by the action of certain species of bacteria, mainly *leuconostoc mesenteroides*, on sucrose.

Dry matter (in cane) is taken as the Brix and fibre in the cane.

Equilibrium moisture content (EMC) is the moisture content attained when a hygroscopic material is kept in contact with air at constant temperature and humidity until equilibrium is reached.

Extraction (pol) is the percentage of pol in cane which passes into mixed juice. Analogous definitions apply to sucrose extraction, Brix extraction and juice extraction.

Extraneous matter in cane is all foreign matter (e.g. cane tops, dry and green leaves, soil, rocks, cane roots, etc) delivered with the cane. Some authors used the word "trash" as a collective term for all extraneous matter, or more frequently, as the dry leaves associated with cane stalks. In this study, the term "trash", if used, will refer to the dry leaves associated with cane stalks.

Fibre in cane is the dry water-insoluble component of cane. Natural fibre is that fibre with chemically bound (Brix-free) water present in its structure.

Filter cake is the residue removed by filtration in the process of juice clarification.

Filterability of a raw sugar is measured by comparing the filtration rate of the sugar with that of a standard sucrose solution under specified conditions. It is expressed as a percentage of the filtration rate of the standard sugar.

Imbibition is the process in which water or juice is applied to bagasse to enhance juice extraction at the mill tandem. Imbibition water is the water used in the imbibition process.

Lime saccharate is obtained from a mixture of milk of lime and a sucrose solution in a certain proportion, and is used in cane juice clarification.

Massecuite is the mixture of sugar crystals and mother liquor discharged from a vacuum pan. Massecuite are classified according to descending purity as A, B, etc or first, second, etc.

Mill extraction is the percent mass of sucrose originally in the cane that has been extracted into the mixed juice.

Mixed juice is the mixture of juices leaving the milling train or a cane diffuser for further processing.

Molasses is the mother liquor of massecuite separated from the crystals by mechanical means. It is designated as for the massecuite from which it is extracted, e.g. A-molasses. Final molasses is molasses obtained from the final massecuite and from which no further sugar will be removed.

Net isosteric heat of sorption (q_{st}), or the enthalpy of sorption is defined as the isosteric heat of sorption (Q_{st}) minus the heat of vaporisation of water at the system temperature.

Non-pol is dry substance minus pol, and dry substance is the material remaining after drying a product to constant mass under such conditions that it does not suffer chemical change.

Non-sucrose is analogous to non-pol, it is dry substance minus sucrose.

Overall recovery is the percentage of the pol in cane that passes into sugar (see boiling house recovery).

Physisorption is the physical adsorption process in which weak Van der Waals interactions are involved.

Pol is the apparent sucrose content of a sugar product determined by direct or single polarization. The term is used in calculations as if it were a real substance, e.g. kg pol.

Purity (apparent) is the ratio of pol in the Brix or gravity solids, expressed as a percentage.

Purity (Clerget) is the ratio of sucrose as determined by the Clerget method in the Brix or gravity solids, expressed as a percentage.

Purity (gravity) is the ratio of sucrose in the Brix or gravity solids, expressed as a percentage.

Purity (target) – It is widely recognised that below a certain sucrose or purity level, no more sucrose can be recovered from molasses. This target purity depends on a number of factors, most importantly, the viscosity and the crystal contents of the massecuites, the saturation temperature at crystallization and the nature of the non-sucrose fraction in the final molasses, namely fructose, glucose and the inorganic ash such as potassium, sodium, calcium and magnesium.

Purity (true) is the ratio of sucrose in the dry solids, expressed as a percentage.

Ratoon is the re-growth after crop of a perennial plant.

Recovery is the ratio of sucrose actually recovered to that entering, expressed as a percentage.

Reducing sugars are the reducing substances in the cane and its products, calculated as invert sugar. Two familiar examples are fructose (laevulose) and glucose (dextrose).

Seed – Small sugar crystals serving as nuclei for crystallization in the sugar boiling process.

SJM formula states that given a juice (or initial material e.g. syrup or massecuite) of J purity and producing a sugar of S purity with a molasses of M purity, the percentage of the total sucrose (or pol) in the original material to go into the sugar will be 100S(J-M)/[J(S-M)]. The formula predicts the portion of the sucrose (or pol) in the original material that goes to the sugar produced, and the remainder to the molasses.

Sorption is the generic term used when adsorption and absorption processes cannot be distinguished experimentally.

Sucrose % cane is the sucrose of formula $C_{12}H_{22}O_{11}$ in the absolute juice of the cane expressed as a percentage.

Suspended solids are the dry solids in juice or other products removable by physical means such as decantation, filtration or centrifugation.

Syrup is the concentrated sugar solution leaving the evaporators.

Trash (see extraneous matter).

Water activity a_w of a substrate is defined as the ratio of the equilibrium vapour pressure of water (p') in the substrate to the vapour pressure of pure water (p^o) at the same temperature, i.e. $a_w = \frac{p'}{p^o} = \frac{R_H}{100} = H_R$, where R_H and H_R are the equilibrium relative humidity expressed respectively as a percentage and fraction.

Undetermined loss is the unaccounted sucrose loss when sucrose is extracted from cane after taking into account the sucrose losses in bagasse, in filter cake and in molasses.

ABBREVIATIONS

Ash	Sulfated ash
BHR	Boiling house recovery
cm	centimetre
CCS	Commercial cane sugar
d.f.	Number of degrees of freedom
D	Dry leaf
EM	Extraneous matter
E_s	Standard error of the mean
f	fines
F	fibre
F pr.	F-test probability
FTIR	Fourier transform infrared
g	Gram
G	Green leaf
G/F	Glucose/fructose ratio
HPIC	High performance ion chromatography
Hz	Hertz
ICUMSA	International Commission for Uniform Methods of Sugar Analysis
IUPAC	International Union of Pure and Applied Chemistry
L	Litre
m	Metre
mL	Millilitre
mm	Millimetre
MPa	Mega Pascal
m.s.	Mean squares
MSIRI	Mauritius Sugar Industry Research Institute
nm	Nanometre
NS	Not significant
P	Pith
rpm	Revolutions per minute
R	Rind
R^2	Coefficient of determination
s	Second
s.s.	Sum of squares
S	Stalk
S.D.	Standard deviation
T	Tops
TCH	Tons cane per hour
TP	Target purity
TPD	Target purity difference
v.r.	Variance ratio
µm	Micrometre

LIST OF MAIN SYMBOLS

Symbol	Description and unit (none unless specified in brackets)
a	Adsorbed water (% fibre) in Freundlich isotherm equation
a_w	Water activity = p'/p^o
A	Surface area for adsorption ($m^2\ g^{-1}$)
A_m	Area of a water molecule ($1.06 \times 10^{-19}\ m^2$ molecule $^{-1}$)
b, c, d, f	Constants in sorption models
b_o, c_o	Constants
BFW	Brix-free water (% db)
D'_{df}	Dry mass fraction of dry leaf fines
D'_{dF}	Dry mass fraction of dry leaf fibre
D'_{gf}	Dry mass fraction of green leaf fines
D'_{gF}	Dry mass fraction of green leaf fibre
D'_{rf}	Dry mass fraction of rind fines
D'_{rF}	Dry mass fraction of rind fibre
D'_{sF}	Dry mass fraction of stalk fibre
D'_{sp}	Dry mass fraction of stalk pith
D'_{ss}	Dry mass fraction of stalk skin
EMC	Equilibrium moisture content (% db for all isotherm models and decimal in the Guggenheim-Anderson-de Boer isotherm model)
GCV	Gross calorific value ($kJ\ kg^{-1}$)
H_1	Heat of sorption of the monolayer ($kJ\ mol^{-1}$)
H_L	Latent heat of vaporisation of pure water (43.53 $kJ\ mol^{-1}$ at 35 ^oC)
H_m	Heat of sorption of the multilayer ($kJ\ mol^{-1}$)
H_R	Equilibrium relative humidity (decimal)
m	Measured EMC (% db)
$\hat{m}$	Predicted EMC (% db)
m_{df}	Measured Brix-free water value of dry leaf fines (% db)
m_{dF}	Measured Brix-free water value of dry leaf fibre (% db)
m_{dl}	Estimated Brix-free water value of reconstituted dry leaf (% db)
m_{gf}	Measured Brix-free water value of green leaf fines (% db)

m_{gF}	Measured Brix-free water value of green leaf fibre (% db)
m_{gl}	Estimated Brix-free water value of reconstituted green leaf (% db)
m_h	Mass of hydrated water (g)
m_o	Moisture content equivalent to the monolayer (% db for all isotherm models and decimal in Guggenheim-Anderson-de Boer isotherm model)
m_{rf}	Measured Brix-free water value of rind fines (% db)
m_{rF}	Measured Brix-free water value of rind fibre (% db)
m_s	Mass of dissolved water (g)
m_{sF}	Measured Brix-free water value of stalk fibre (% db)
m_{sp}	Measured Brix-free water value of stalk pith (% db)
m_{st}	Estimated Brix-free water value of reconstituted cane stalk (% db)
m_v	Material constant which approximates the fibre saturation point for desorption (%)
m_w	Molar mass of water (18.02 g mol^{-1})
M_{rf}	Measured EMC of rind fines (% db)
M_{rF}	Measured EMC of rind fibre (% db)
M_{sF}	Measured EMC of stalk fibre (% db)
M_{sp}	Measured EMC of stalk pith (% db)
M_{ss}	Measured EMC of stalk skin (% db)
M_{st}	Estimated EMC of reconstituted stalk (% db)
n_i	Total number of isotherms
N	Number of data points
N_A	Avogadro's number (6.022 x 10^{23} molecules mol^{-1})
N_o	Number of adsorbed monolayers
P	Water vapour pressure in mm Hg in the Freundlich's isotherm equation
p'/p^o	relative pressure
p_1, p_2	Brix of the sample blank before and after equilibrium (%)
p_3, p_4	Brix of the test solution before and after equilibrium (%)
p	Net Brix increase (%) = p_4 – corrected blank
P	Mean relative deviation modulus
q_{st}	Net isosteric heat of sorption (kJ mol^{-1})
Q_{st}	Total isosteric heat of sorption (kJ mol^{-1})
R	Universal gas constant (8.314 J mol^{-1} K^{-1})
R_H	Equilibrium relative humidity (%)

s_c	Slope of Caurie I isotherm plot
s	Slope of Freundlich isotherm plot
S_d	Entropy of sorption (kJ mol^{-1})
T	Temperature (^oC unless specified otherwise)
T_{hm}	Harmonic mean temperature (K)
T_β	Isokinetic temperature (K)
w_1, w_3	Mass of sample in the blank and test solution respectively (g)
w_2	Mass of distilled water in the blank (g)
w_4	Mass of the contacting sucrose solution in the test solution (g)
W	Molecular mass of the adsorbate substance necessary to bond one molecular mass of water (mol/mol)

CHAPTER 1. EXTRANEOUS MATTER IN CANE

This thesis describes the work undertaken to examine the effect of extraneous matter on cane, bagasse and juice quality and its impact on milling performance, and subsequently to study certain chemical properties of sugar cane component parts in an attempt to explain the effects observed.

1.1 BACKGROUND TO THE MAURITIAN SUGAR INDUSTRY

Mauritius is an island in the Indian Ocean; it covers an area of 1840 km^2 and is situated 880 km off the east coast of Madagascar, near the intersection of latitude 20° South and longitude 57° East. The climate is sub-tropical with only two seasons: winter prevailing from May to September and summer from October to April. Mauritius is prone to cyclones between the months of December and April.

The Dutch took possession of the island, to which they gave the name Mauritius, in 1598. Sugar cane was introduced in 1639 in Mauritius from Batavia, the Dutch name for Jakarta of Indonesia. The intent was apparently to produce sugar but, at first, only a spirit known as “arrack” was produced. Later, the Dutch colonists managed to produce a dark brown sugar cake, but crystalline sugar was only successfully made in 1694.

The Dutch abandoned Mauritius in 1710. Later, the French took possession of it, and the production of sugar on a significant scale was encouraged and developed. By 1755, sufficient sugar was produced to meet the needs of the local inhabitants and its neighbouring islands. In 1801 cane plantations covered 4220 hectares and 60 mills were producing 3000 tonnes of sugar (Anon., 1997).

The British captured the island in 1810. It remained under British rule up to 1968 when it acceded to independence and became a sovereign state within the Commonwealth until 1992, when it became the Republic of Mauritius. Under British rule the cultivation of sugar cane was encouraged since it is more resistant than other crops to both drought and cyclones. In 1820, 106 mills were in operation and by 1825 the area under cane cultivation had reached 10 975 hectares, and the sugar production, 10 870 tonnes (Rouillard, 2001; Anon., 1997). Since then, the sugar industry has become the major agro-industry, and has remained so to this day. In 1858, the number of sugar mills in operation reached the record

number of 255, and cane land covered 46 025 hectares, producing 125 002 tonnes of sugar (Anon., 1858).

The industry continues to evolve. In 1953, the annual sugar production amounted to 512 000 tonnes produced by 27 factories (Anon., 1997). A record production of 718 362 tonnes sugar was achieved in 1973 by 21 factories (d'Espaignet, 1974; Wong Sak Hoi, 2003). Today, the production is 504 653 tonnes in 2006 (Anon., 2007), and centralization of sugar factories has reduced their number to ten (see Fig 1.1). Mauritius has no option but to pursue the centralization of sugar factories in order to improve the efficiency of the industry, and to modernize the remaining factories so as to reduce the costs of sugar production.

Since 1920, Mauritian sugar factories have produced electricity from bagasse, a by-product of the sugar industry. At first this was for their own demand, but since 1957, they have also been supplying to the national grid (Kong Win Chang *et al*., 2001). By 2004, they contributed to about 22% of the primary energy supply, and about 55% of the electricity sold by the grid (Central Electricity Board) was generated by sugar factories from bagasse (302 000 MWh) and from coal (835 000 MWh).

Since the 1970s, the Mauritian economy has diversified from a sugar cane mono-crop economy based on sugar, to manufacturing (mainly textiles and garments) as well as tourism in the 1980s, while still relying heavily on sugar. More recently, it has started developing financial services as the fourth pillar of the economy.

Because of the threats facing the Mauritian sugar industry, namely, the erosion of preferential access on its traditional export markets for sugar, and the challenges imposed by trade liberalisation, the Government decided that a sugar sector strategic plan for the period 2001-2005 be elaborated.

The plan (Anon., 2001a) implied more centralisation, cost reduction, enhanced productivity, manpower rightsizing, an optimal use of cane sugar resources, well-planned diversification activities, improvement of value added products and the creation of new opportunities. Among the targets cited are: on the field side, the preparation of a substantial proportion of land that can be totally mechanised and the supply of irrigation water to land requiring irrigation, among others. On the factory side, the reduction of sugar factories from 14 to 7 or 8, the reduction of the cost of sugar production from 18 US cents/lb to 14 US cents/lb

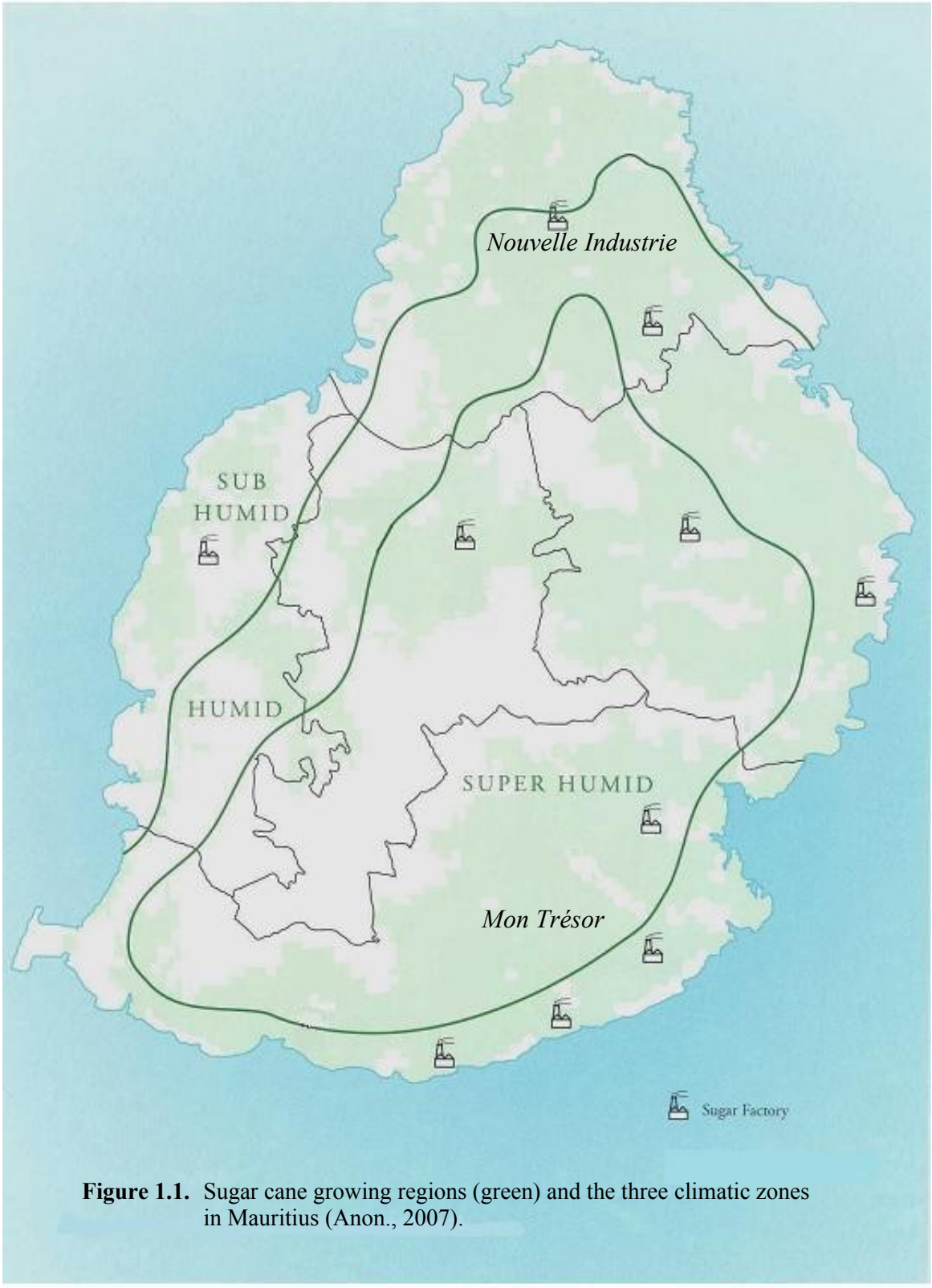


Figure 1.1. Sugar cane growing regions (green) and the three climatic zones in Mauritius (Anon., 2007).

and eventually to 10 US cents/lb, the generation of as much electricity as possible from bagasse, the running of sugar factories for 7 days a week for up to 150 days, increased effort to market specialty sugars, the review of new technologies such as molasses desugarisation, dry cane cleaning to recover trash prior to cane crushing, improvement of sugar recovery from cane, energy and water economy, and the production of co-products such as ethanol and *rhum agricole*, a high value-added product produced from cane juice.

This thesis examines the effect of extraneous matter on sugar recovery and investigates the avenues which could lead to the improvement of sugar recovery from cane. In order to place this work in context, a brief description of the Mauritian raw sugar manufacturing practices is presented in the next section. The meaning of terms associated with sugar processing is given in the section entitled "Glossary of Terms".

1.2 RAW SUGAR MANUFACTURING PROCESS IN MAURITIUS

The raw sugar manufacturing process in Mauritius is outlined in the flow-diagram shown in Fig 1.2. The description of general practices in sugar processing is extracted from Chen and Chou (1993). No attempt will be made to indicate the number, type and capacity of each equipment used. For these details, see Kong Win Chang and Wong Sak Hoi (1999) who have compiled the plant data of Mauritian sugar factories.

1.2.1 Cane harvest

In the early 1970s, almost all the cane stalks in Mauritius were stripped of all the extraneous materials before being manually harvested and loaded. In 1975, due to the shortage of manpower in the agricultural sector, grab loading of manually harvested cane was resorted to, and represented 60% of the cane production in 1996. Since 1988, mechanical harvesters were introduced and cane harvested by chopper harvester increased steadily. It represented 16.7, 21.6, 24.3, 27.3, 28.6 and 32.3% of the total cane harvested from 2001 – 2006 (Anon., 2001b; Anon., 2002a; Anon., 2003; Anon., 2004; Anon., 2005a and Anon., 2006) respectively. Whole-stalk harvested cane represented only about 1% of the machine-harvested cane in the early 2000s. The trend was towards green cane harvesting, it increased from 67% of the cane mass harvested in 2002 to 72, 76 and 85% in 2003, 2004 and 2005 respectively.

1.2.2 Cane reception

The cane supply arriving at a factory or a cane reception centre is first sampled at the weighbridge by means of a core-sampler (Fig 1.3) and then weighed before being unloaded on the platform for crushing. The sample is sent to the laboratory of the Cane Planters and Millers Arbitration and Control Board for analysis of Brix and pol of press juice, mass of press cake and fibre % cane, all of which are required for cane payment purposes.

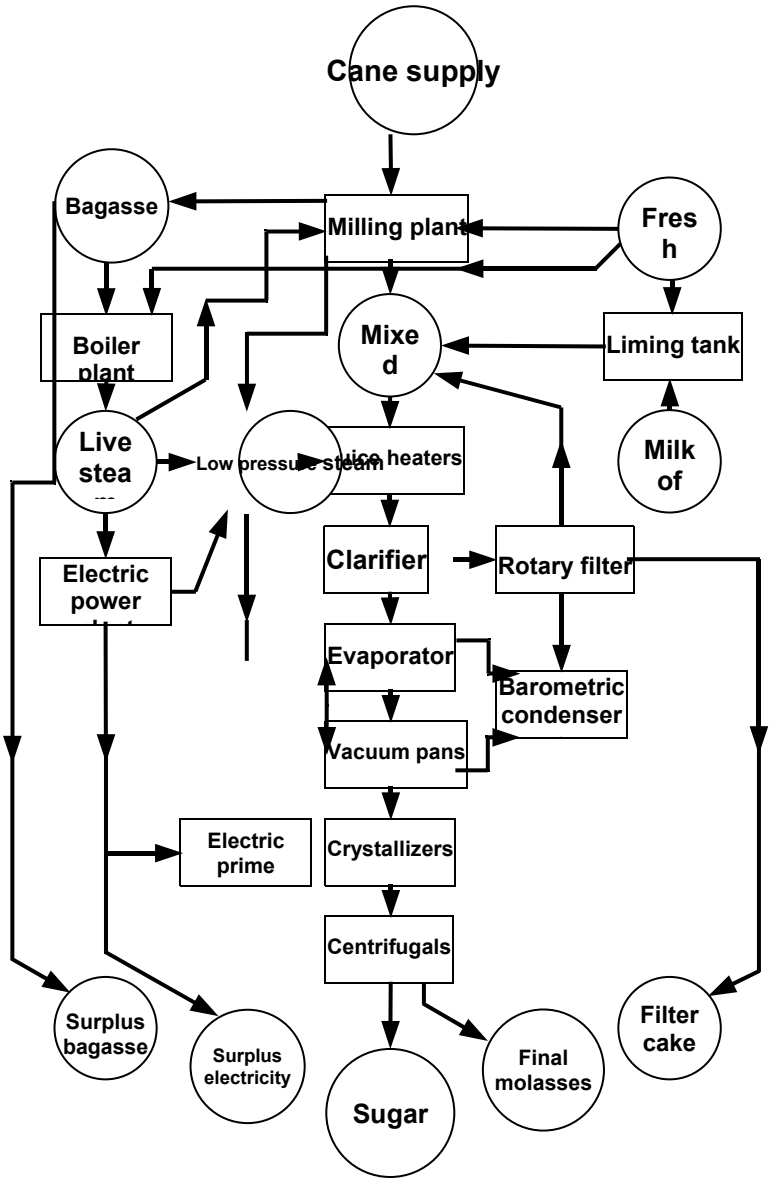


Figure 1.2. Flow-diagram of raw sugar manufacturing process in Mauritius (Anon., 1997).

Figure 1.3. Cane sampling by core sampler for analysis.

1.2.3 Juice extraction

The cane is first prepared by revolving knives that cut the stalks into chips which are then cut by heavy duty shredder into long fibres before entering the milling plant. The latter consists of multiple units (4 – 7) of three-roller combinations through which the crushed cane or the cellulosic fibre (bagasse) successively passes. Fresh water is sprayed on the mat of bagasse as it enters the last mill to help leach out the maximum sugar. In this context, the thin juice extracted by each mill is sprayed on the bagasse entering the previous mill. This process is termed imbibition, and the water used, imbibition or maceration water. The combined juice collected after the first mill is called mixed juice. The fibre from the last mill, the final bagasse, contains unextracted sugar and 45 – 55% moisture. Bagasse normally goes to the boiler plant as fuel to produce steam and electricity for the factory's own demand. The surplus bagasse can be used to produce electricity for the national grid.

1.2.4 Clarification and filtration

The mixed juice extracted from the cane is acidic in nature, of pH about 5.2; it consists of a solution of sucrose mixed with soluble and insoluble impurities. The clarification (or defecation) process removes most of the turbid impurities by the action of heat and lime. The mixed juice is heated, in most cases, to about 75 °C, and the juice pH is changed from

acidic to neutral or slightly alkaline by the use of milk of lime, which causes coagulation of some colloids, and forms a heavy precipitate of complex composition containing insoluble lime salts, coagulated albumin and some of the fats, waxes and gums.

The phosphate content of the juice is the most important factor in efficient clarification. If the cane is grossly deficient in natural phosphate, or is otherwise very difficult to clarify, phosphate may have to be added before liming. Nowadays, liming techniques can overcome most cane deficiencies, and satisfactorily clarify juice with natural phosphate levels down to half or less of the normally accepted requirement of 300 ppm P_2O_5 (Whayman, 1992).

When sugar solution containing soluble phosphate comes into contact with excess calcium ions, an amorphous calcium phosphate is formed, which crystallizes as octa-calcium phosphate and hydroxyapatite (Bennett, 1975). The two-stage precipitation first causes small particles to form, which grow and rearrange into a very intricate floc that entraps and adsorbs other non-sugars that are precipitated by the reaction change, by the heat, by the calcium and by the increase in pH. The precipitate entraps most of the fine suspended solids originally present in the juice. Bennett (1957, 1975) showed that the impurities are bound by the mechanism of bridging by calcium phosphate precipitation (Fig 1.4).

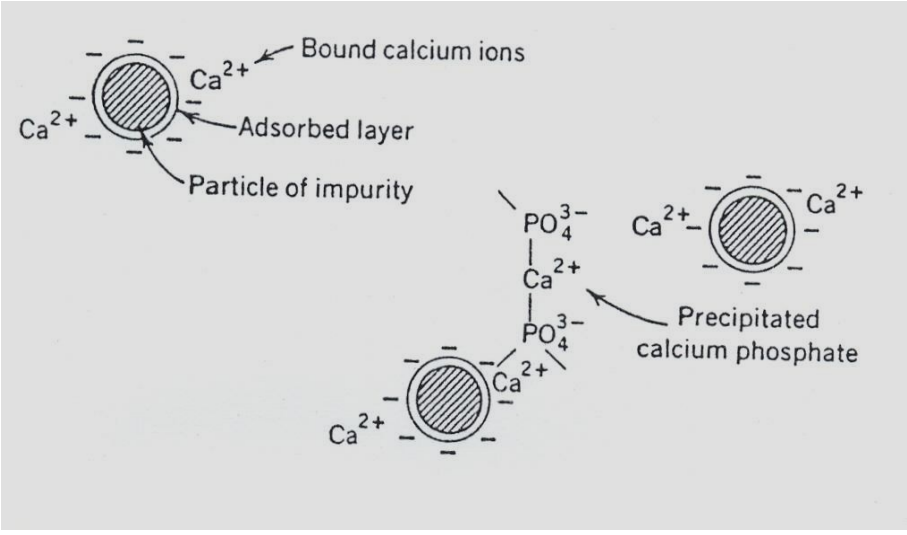


Figure 1.4. Bridging mechanism formed by calcium and phosphate ions during cane juice clarification (Bennett, 1975).

In Mauritius, most of the sugar factories use lime saccharate, a mixture of milk of lime and sucrose syrup, as this liming agent has been found more efficient than milk of lime alone (Wong Sak Hoi and Chung, 1996). After liming, the juice is superheated to 103 – 105 °C and allowed to flash to the constant boiling point at atmospheric pressure. This forces out the air present in the juice and also causes bagacillo particles to burst and sink with the solids. The flashed limed juice is then sent to decant in a large vessel called a clarifier where the clear juice separates from the precipitated solids. Flocculation and settling are aided by the addition of synthetic water soluble flocculants, which are partially hydrolysed polyacrylamides. The clear juice is removed from the top and sent to evaporators while the muds are pumped from the bottom of the clarifier to a rotary vacuum filter. The filtered juice is sent back to process while the filter cake is returned to the fields as soil conditioner or fertilizer.

The techniques used for clarification and filtration were comprehensively reviewed by Whayman (1992).

1.2.5 Evaporation

The clear juice from the clarifier contains about 85% water. Two-thirds of this water is evaporated in a vacuum multiple-effects evaporator consisting of a succession of vacuum-boiling vessels (usually 4 – 5) arranged in series or in parallel so that each succeeding body has a higher vacuum, and therefore boils at a lower temperature. The vapours from one body can thus boil the juice in the next one. With this arrangement, the exhaust steam introduced into the first body of a quadruple-effect evaporator evaporates four times its mass of water. The vapour from the final body of the evaporator goes to a barometric condenser. The syrup leaving the evaporator contains about 65% refractometric solids (Brix) and 35% water.

1.2.6 Crystallization

Crystallization takes place in single-effect vacuum pans, where the syrup is further evaporated until saturated with sugar. "Seed grain" is then added to serve as nuclei for the formation of sugar crystals, and more syrup is added as the water evaporates. Crystal growth continues until the pan is full. For a skilled sugar boiler, the original crystals can be grown without the formation of additional crystals (false grain), so that when the pan is

full, the crystals are all of the same desired size. The crystals and syrup form a dense mass called massecuite, and the strike (the contents of the pan) is then discharged into a mixer or a crystallizer, where the crystals continue to grow.

In the three-boiling system, the first boiling of raw syrup yields raw sugar and A molasses, which is returned to the vacuum pan to be reboiled on a "footing" of first-grade massecuite to a second massecuite (B) that in turn yields a second crop of crystals (B sugar). This is usually mixed with the A sugar forming the commercial output of the factory. The second or B molasses is in turn reboiled on a footing of syrup crystals to form C massecuite. The C sugar is mingled with syrup and used for A and B massecuite "seed". The final cane molasses contains approximately one-third sucrose, one-fifth reducing sugars, and the remainder, ash, organic non-sugars and water. It is used as cattle feed, and in the manufacture of alcohol and yeast.

1.2.7 Centrifugation

The crystals in the massecuite are separated from the surrounding mother liquor (molasses) by centrifugal force in a machine called a centrifuge. It consists of a perforated drum or basket so arranged that it may revolve on a vertical shaft or axis called the spindle. The perforated basket is lined with a metal screen containing 400 to 600 perforations per square inch. The basket revolves at 1000 to 1800 rpm. The perforated screen retains the sugar crystals which may be washed with water or steam while the molasses passes through the lining. The raw sugar obtained is then sent to a dryer and cooled.

1.3 MEASURE OF CANE QUALITY

The amount of sugar recovered from cane is highly dependent on the raw material processed. The next section examines the quality of cane received at the mills.

The quality of cane received at Mauritian sugar factories has deteriorated in recent years to such an extent that the raw material appears more like a few cane stalks buried in extraneous matter, i.e. non-cane, which contains little sucrose and inflates the mass of cane processed.

Extraneous matter in cane is defined as any material that is delivered to a sugar factory as part of the cane but which does not contribute to increasing the amount of sugar produced, or has the tendency to lower the purity of the mixed juice. Included in this classification

are: cane tops, dry and green leaves, soil, rocks, cane roots, etc. Cane top is defined as the top part of the cane stalk above the actively growing apical internode with attached green leaves. Some authors use the word "trash" as a collective term for all extraneous matter, or more frequently, as the dry leaves associated with cane stalks. To avoid confusion, the term extraneous matter will be used in this thesis as the collective name and its individual components, by their specific names such as tops, green leaves, dry leaves, soil, etc. The term trash, if used, will refer to the dry leaves associated with cane stalks.

1.3.1 Direct measure of extraneous matter in cane

A method exists to determine directly the amount of extraneous matter in a cane supply. This is carried out on a consignment of 5-6 tonnes of cane after the separation of all non-cane materials, i.e. trash, green leaves, tops and young shoots to determine their respective masses. The number of samples handled in a day is limited and the results obtained are far from being representative of the total mass of cane crushed during the day. As this method is too time-consuming and costly to be practicable, factories do not systematically determine extraneous matter in cane, and its exact level is therefore unknown. A more common practice nowadays is to use a smaller sample size of about 400 kg taken by a loader from a cane consignment. Fig 1.5 shows such a test being carried out with the removed non-cane placed on both sides and cleaned cane in the middle. Results obtained on a single cane consignment over a fortnight showed mean extraneous matter of 8.3% ranging from 4.7% to 11.5% at one factory, and 24.0% with a range from 17.2% to 34.0% at another factory (Wong Sak Hoi *et al.*, 1999).

In South Africa, Cargill (1976) described sampling and analytical procedures involving a grab sampler positioned next to a main cane carrier for estimating the amount of tops and trash in a cane consignment. A sample size of 100 kg is recommended (Anon., 1985b).

Wong Sak Hoi *et al.* (1999) devised a rapid and reliable method of estimating extraneous matter in a cane consignment, by using grab samples of cane bundles containing about 40 cane stalks. The method was found to yield reasonably accurate estimate of extraneous matter in cane. Results obtained at one factory on sample involving 40 cane stalks taken from a consignment of 400 kg, was compared with the true value of the whole consignment. Statistical analysis of ten such paired-comparisons showed a mean coefficient of variation of 11.6.

Figure 1.5. Determination of extraneous matter in cane from a 400 kg load, with removed non-cane on both sides and cleaned cane in the middle.

1.3.2 Other cane quality yardsticks

As indicators of cane quality, suspended solids in mixed juice, ash % cane and also sucrose % dry matter in cane (i.e. sucrose expressed as a percentage of both Brix and fibre in cane) have been used in South Africa (Lionnet, 1996). Ethanol was first employed by Lionnet (1986) to measure cane deterioration after harvesting, and the concept of estimated recoverable crystal in cane (ERC) was introduced by Van Hengel (1974) to estimate the contribution of each cane consignment towards the total industrial production of a standard sugar. However, past performance figures are needed to establish factors relating recovery to each of the three parameters: sucrose, non-sucrose and fibre. The ageing of cane after harvesting and infection by micro-organisms with the formation of dextran are also criteria commonly considered.

Clarke and Legendre (1996) listed, as factors for cane quality, the ratio of green tops and leafy trash, mud or field soil, staleness, stress factors affecting cane (drought, irrigation, disease, pest, frost) and physical damage of cane stalks by mechanical handling, as well as the harvesting method, cane transport and delivery procedures.

All the parameters cited above are not systematically measured in the routine control of factory performance. Fortunately, changes in some measured parameters such as fibre % cane, sucrose % cane, sucrose/fibre ratio in cane and mixed juice purity, can give an indirect measure of cane quality received at factories.

1.4 TRENDS IN CANE QUALITY RECEIVED AT SUGAR MILLS

Wong Sak Hoi and Autrey (1997) examined average data for Mauritian sugar factory performance from 1960 to 1996 and found that, with the advent of mechanical cane loading in 1975, the increase in extraneous matter in cane has increased fibre % cane by 2 units, decreased sucrose % cane by 0.3 unit and boiling house recovery, which is defined as the percentage of sucrose originally present in mixed juice that has passed into sugar, by 0.7 unit.

Cane quality has deteriorated to such an extent that, in a factory situated in the north of the island, fibre % cane which averaged 14% in the pre-mechanization era of 1975 had increased by 5 units in 1997, representing a 36% increase attributable to the increased extraneous matter received (Wong Sak Hoi and Autrey, 1998). Surveys carried out over a three-week period in 1998 showed that leafy trash received at the same factory averaged 15% of the cane loads and ranged from 7% to 30%.

This trend is a serious concern since factory performance will be badly affected.

While it is probably true that twenty years ago extraneous matter in cane consisted mainly of dry trash, nowadays it is common practice to send green leaves and immature cane tops with the cane.

In the following section, the previous examination of trends in cane quality from 1960 to 1996 carried out by Wong Sak Hoi and Autrey (1997) has been updated to 2004, with particular attention paid to the period 1996 to 2004. Parameters such as fibre % cane and sucrose % cane were examined to see whether the deteriorating trend in cane quality received at the factories was maintained.

1.4.1 Trends in Mauritian cane quality

Four parameters were monitored in order to assess the trend in cane quality received at Mauritian sugar factories and each of these will be discussed in turn.

Changes from 1960 to 2004 in island average data on fibre % cane, sucrose % cane and sucrose/fibre ratio in cane are shown in Fig 1.6.

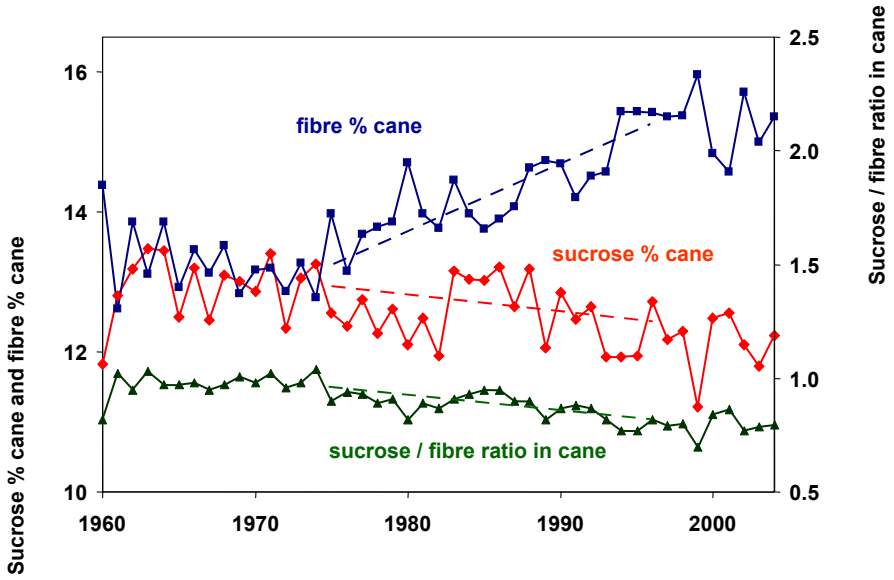


Figure 1.6. Changes in fibre % cane, sucrose % cane and sucrose/fibre ratio in cane from 1960-2004.

1.4.1.1 *Fibre % cane*

The true cane fibre content varies with cane varieties and climatic conditions, such as cyclones and drought, and although there were variations in fibre % cane in the pre-mechanization era of 1960-1975, the average of 13.2% showed that the true cane fibre had remained constant in spite of changes in varietal composition of the cane crushed.

Since increased mechanization started in 1975 with grab loading of manually harvested cane to lorry, fibre % cane no longer represents the true cane fibre. It also includes the fibrous material from trash and the soil from cane, reflecting to some extent the extraneous matter in cane received at the factory. Changes in fibre % cane from 1975 to 1996 (see Fig 1.6) represent an increase of 2.0 units, and correspond to the upward trend in the extent of mechanical loading. In 1990, when mechanical loading of cane was at 50% of the total production and when the amount of mechanical harvesting was still insignificant, fibre % cane was at 14.7, i.e. 1.5 units higher than the previous 13.2 fibre % cane.

In general, a higher fibre content of the raw material due to more cane trash reduces mill capacity, increases power consumption and causes more wear and tear of the equipment.

From Fig 1.6, it is evident that the upward trend in fibre % cane seems to have levelled off since 1996. The increase in 1999 was due to the high proportion of immature cane sent to factories together with cane tops, and dry and green leaves with adhering soil, because of the most severe drought ever faced by the country during that year. A clean cane

campaign was launched to encourage growers to send cane with less than 10% extraneous matter, and the effects were seen in the drop in fibre % cane in 2000 and 2001.

1.4.1.2 *Sucrose % cane*

Sucrose % cane is also subject to changes in cane varieties and climatic conditions. In the pre-mechanization era, sucrose % cane averaged 12.9, whereas from 1975 to 1996 there was a gradual decline of about 0.3 unit.

Fig 1.6 shows that when fibre % cane is high, sucrose % cane is low, mainly because trash is weighed as cane and does not contain any sucrose.

1.4.1.3 *Sucrose/fibre ratio in cane*

Lionnet (1992a) introduced the concept of sucrose/fibre ratio in cane to indicate the amount of extraneous matter in cane. From Fig 1.6, this ratio was steady at almost 1.0 from 1960 to 1975, with a gradual downward trend from 1975 to 1996 probably due to increased trash in the cane as a result of increased mechanization in cane loading and harvesting. From 1996 to 2004, sucrose/fibre ratio in cane appears to be stable.

1.4.1.4 *Mixed juice purity*

Mixed juice purity is the ratio of sucrose to total solids in solution. It is a good indicator of the freshness of the cane. It is adversely affected by cane tops but not by trash and soil. Changes in mixed juice purity and boiling house recovery from 1960 to 2004 are shown in Fig 1.7. Since mixed juice purity is closely related to boiling house recovery, the direct relationship between the two parameters, as indicated by the same pattern of the curves, is as expected.

Without taking into account the cyclonic years in the early 1960s, mixed juice purity can be seen to decrease from 1965 to 1975. There was then an upward surge to 2004 except during the drought year of 1999, which may be attributed to more fresh cane being sent to the mills as a result of mechanization.

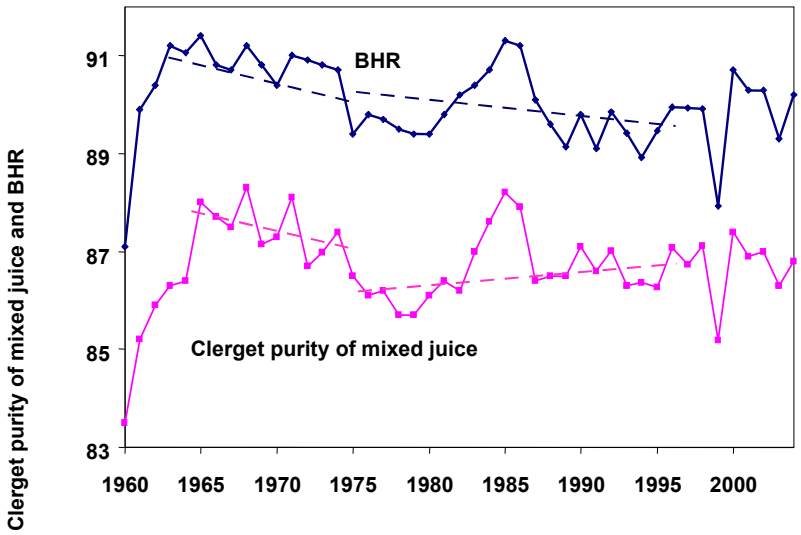


Figure 1.7. Changes in Clerget purity of mixed juice and boiling house recovery (BHR) from 1960-2004.

During the pre-mechanization era of 1960 to 1975, the decrease in boiling house recovery is in line with the decrease in mixed juice purity as indicated by the same slope of the two downward curves. From 1975 to 1996, however, the boiling house recovery curve did not parallel the increase in mixed juice purity and in fact, registered a decrease of 0.7 unit. Wong Sak Hoi and Autrey (1997) attributed this to the result of extraction of melassigenic impurities in trash and tops, the concentration of which is not high enough in juice to affect mixed juice purity, but is sufficiently high in molasses for their effect to be felt. From 1996 to 2004, the boiling house recovery curve seems to have levelled off except during the drought year of 1999.

1.4.2 Cane quality trends in other sugar-producing countries

Ideally, cane delivered to sugar factories should be cleaned of immature tops, leaves whether green or dry, and soil. This practice was possible when man-power was abundant; however, with economic development in other sectors, the shortage of agricultural labour led to mechanization of cane loading in Louisiana in mid-1940. In Australia, mechanical loading started in mid-1950 and mechanical harvesting in early 1960 (King, 1969). In South Africa, cane loading started in the early 1970s, while in Mauritius, in the mid-1970s; with the consequence that a progressive increase is observed in the quantity of extraneous matter being introduced at the mills with the cane.

The level of extraneous matter in cane depends on the weather at harvest, and the harvesting and loading methods chosen. The loading method known as push-piling entrains a lot of soil and trash in the cane, and is banned in certain parts of the world.

In his review of cane quality in South Africa, Lionnet (1996) examined the industrial average of pol % cane (the data became sucrose % cane as from 1981 onwards), fibre % cane and pol purity of mixed juice from 1960 to 1995, and produced graphs to show the trend in cane quality received at South African sugar mills over the years. These graphs have been updated to 2004 (Figs 1.8 and 1.9) by making use of South African published data from 1960-2004 (Anon., 1985a; Anon., 2002b and Anon., 2005b).

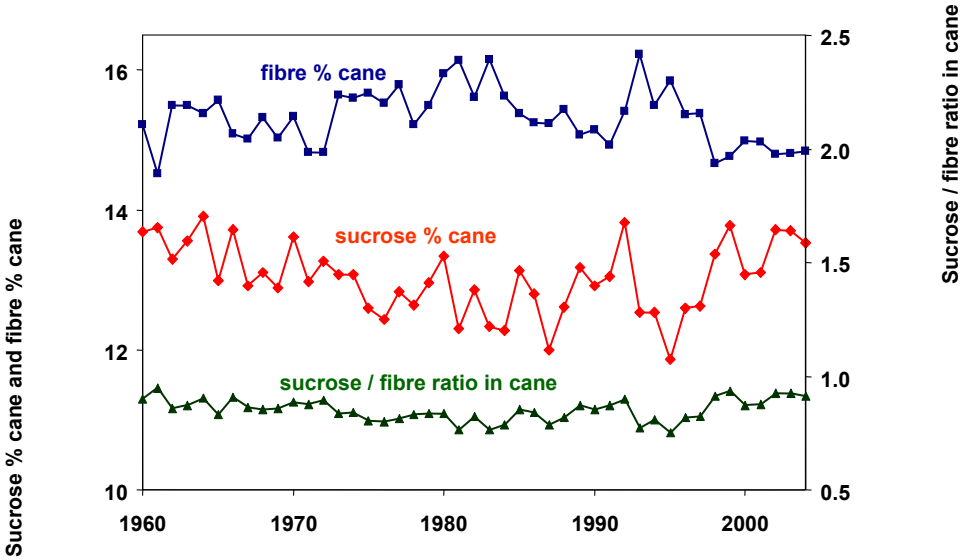


Figure 1.8. Changes in fibre % cane, sucrose % cane and sucrose/fibre ratio in South African cane from 1960-2004.

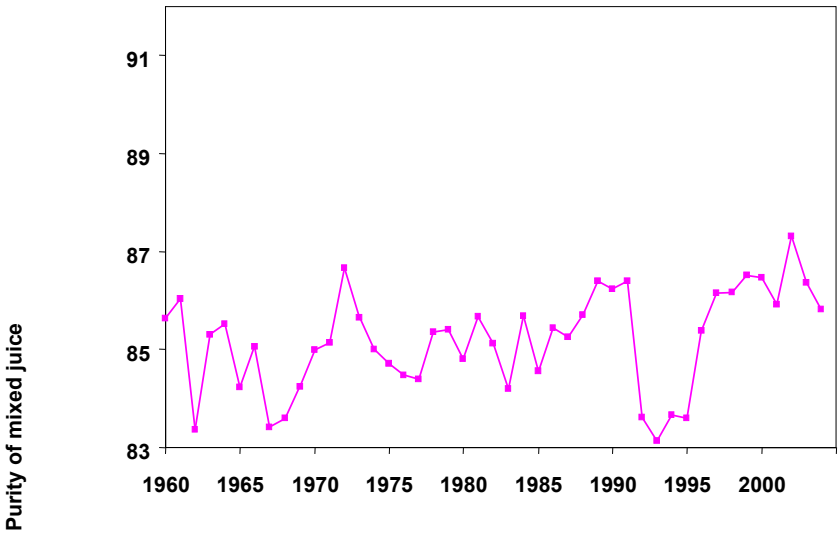


Figure 1.9. Changes in purity of South African mixed juice from 1960-2004.

Lionnet (1996) explained that for pol % cane, there was a slow decrease over the years until about 1987 when a strong recovery took place. This was unfortunately stopped by the droughts which lasted from 1992 to 1995. Fibre % cane shows a clear decrease from 1983 onwards due to harvesting of younger cane with the incidence of the Eldana borer, which tends to affect older or carry-over cane. Lionnet (1996) also noted that an improvement in mixed juice purity (see Fig 1.9) started in 1985, except for the drought years of 1992-1995.

Compared to the trends of fibre % cane and sucrose % cane in Mauritius (see Fig 1.6), those in South Africa (refer to Fig 1.8) do not show great changes over the years, although a downward trend in fibre % cane and an upward trend in sucrose % cane are evident as from 1985 onwards (except for the drought years). It is worth pointing out that while the values of sucrose % cane and sucrose/fibre ratio in cane are comparable in the two countries, this is not the case with the fibre % cane data. In Mauritius, fibre % cane was stable at 13.2 up to 1975, and gradually increased to about 15.0, whereas in South Africa, it averages 15.3% over the years. Purity of mixed juice is higher in Mauritius; it averages 86.7%, whereas in South Africa it is 85.6%.

Numerous literature exists on trends in cane quality. Clayton and Roberts (1971) stated that in Florida, the trash content of cane just before harvest is about 30%. Kampen (1974) had shown that extraneous matter in Louisiana cane was very much dependent on cane variety and the harvest method used, and it had gradually increased from less than 1% in 1945 to more than 13% in the early 1970s, with total mechanization of field operations since 1950. Legendre and Irvine (1974) reported that when hand cutting, stripping and loading were practised in Louisiana prior to 1942, the amounts of extraneous matter rarely exceeded 4%; by 1947, when mechanical harvesting had become predominant, the total extraneous matter level had nearly doubled. In Puerto Rico, Cabrer *et al.* (1965) estimated that as high as 25% of cane crushed consists of tops, trash, sand and other extraneous matter while in South Africa, according to Smits and Blunt (1976), the amount may be about 12% or more during rainy periods. Lamusse and Munsamy (1979) reported that after measuring tops and trash at five factories in South Africa during the whole season, the cane was found to contain on average 2.1% tops and 5.3% trash; de Beer *et al.* (1989) carried out an experiment with 800 tonnes of cane, and found that unburnt, untopped cane contained 22.2% extraneous matter whereas burnt, topped cane, contained only 3.2%.

In Australia (Cargill, 1976), cane quality was defined according to the content of extraneous matter in the cane: from 0 – 3% acceptable, 4 – 5% poor, and > 6% very poor. Cargill (1976) also reported that trials carried out in Cuba for five consecutive seasons showed that extraneous matter in green cane harvested and loaded manually amounted to 3%, and in green cane harvested manually and loaded mechanically, 4%. He also reported that in Louisiana, whole stalk green cane harvested mechanically contained 8% extraneous matter.

In Mauritius, due to shortage of labour, when grab loading of manually harvested cane was resorted to in 1975, as much as 15% extraneous matter, including tops, trash, soil and rocks were reported (d'Espaignet, 1977). St Antoine (1977) first raised the alarm on the deteriorating quality of cane received at Mauritian factories.

From these trends it can be seen that steps must be taken to improve the cane quality received at Mauritian mills. Cane quality has deteriorated worldwide, however, Mauritius is at the higher end and this poses a number of deleterious effects which are enumerated in Section 1.5.

1.5 THE DELETERIOUS EFFECTS OF EXTRANEOUS MATTER ON CANE PROCESSING

The adverse effects of sand and trash on milling have been explicitly described by Graham and Gunn (1971), and by Kent *et al.* (1999), and on milling and boiling house performance by Smits and Blunt (1976). St Antoine (1977) discussed the effect of cane quality on the efficiency of sugar factories. Lamusse and Munsamy (1979) examined the effect of trash on mills and diffuser performance, and Clarke and Legendre (1996) described the impact of cane quality on the yield and quality of the sugar produced. Lionnet (1992a, 1996) reviewed some aspects of cane quality in South Africa and its effects on factory performance.

1.5.1 Effects of soil on cane processing

Dirt or field soil is not desirable in cane, but some is inevitable, especially with mechanical harvesting and loading systems operating in wet conditions. A fair amount of soil finds its way into processing causing clarification and filtration problems. A number of process difficulties have been attributed to the detrimental effect of soil: higher turbidity of

clarified juice, increased sucrose losses in filter cake and viscosity of massecuites which are difficult to exhaust (Anon., 1977).

Field soil entering the factory reduces the overall plant capacity and efficiency, increases the cost per ton cane crushed and results in extensive wear to knives, mill rolls and conveyors; poor calorific value of bagasse, poor settling in clarifiers with a high mud volume and large volumes of filter cake and high sucrose losses in filter cake and molasses (Smits and Blunt, 1976; Muller *et al.*, 1982; Kent *et al.*, 1999).

The trend in the decline of cane quality delivered to the mills has resulted in an intolerable amount of extraneous material especially soil which, if left unremoved, causes increased maintenance and replacement costs due to heavy wear and tear of factory equipment and more or less serious difficulties of processing. Cane washing experiments effected by Vignes (1980) showed that process difficulties could be minimized, however, a certain amount of sugar was lost in the process.

In South Africa, a measure of soil in cane was introduced in mid-1970 (Lionnet and Wagener, 1976; Brokensha and Mellet, 1977). It involved the ashing of a prepared cane sample in a furnace. As it had been found that ash content in clean cane averaged 0.6%, this value would be deducted from the ash content found in the prepared cane to give the soil content in cane. Industrial values reported were 1.51% in 1991/92, 1.74% in 1993/94 (Lionnet, 1992a, 1994), and 1.67% in 2004 ranging from 1.17 to 3.08 for the eleven factories which supplied the data (Anon., 2005b).

In Australia, Atherton *et al.* (1992) reported that the natural gamma-ray technique can be used to monitor the soil content of sugar cane. Subsequently, a soil monitor based on natural radioactivity has been developed for the on-stream monitoring of the soil content of sugar cane on a moving conveyor belt (Mathew *et al.*, 1994). It consists of a gamma-ray detector and associated electronics, a belt-weigher and a personal computer. The soil content of cane is computed from the average gamma-ray activity of a rake, the specific gamma-ray activity of the soil and the conveyor load by using a calibration equation. Subsequently, a commercial soil monitor was developed to measure soil in cane to within 1% (Olson *et al.*, 1999). The predictions of soil in cane in one factory from 1994 to 1998 were: 1.7, 1.6, 1.8, 2.1 and 2.0%, respectively.

The effect of soil on factory performance has been the subject of investigation by Muller *et al.* (1982) who found that, through a material balance of ash in the factory, 40% of the soil present in cane goes to the juice and 60% to the bagasse.

1.5.2 Effects of tops and trash on cane processing

St Antoine (1977) stated that cane tops contain relatively little sucrose and a high proportion of non-sugars and a juice of relatively low purity, hence entails a high production of molasses. He also reported that cane leaves absorb a certain quantity of juice during crushing and increase sucrose loss in bagasse. They tend to make the mill roll slip, and reduce mill extraction. Cane leaves, especially green ones, contain soluble non-sugars which increase sucrose loss in molasses.

Lamusse and Munsamy (1979) and Cargill (1976) showed that high extraneous matter in cane increases transportation costs, reduces mill throughput and increases sucrose losses. Clarke (2003) stated that these losses occur in bagasse, and in the boiling house due to the extraction from the trash of non-sucrose materials that interfere with clarification and sucrose crystallisation.

The traditional method of reducing extraneous matter of cane, namely burning, has become unacceptable because of the environmental consequences (Bernhardt, 1994). Since trash has the benefit of providing additional biomass fuel for steam generation and power production, it could be collected separately from the field or separated from the cane upon arrival at the mill.

Dry cane cleaning is now a means of removing a significant proportion of this material before the cane is shredded, thus avoiding the detrimental effects it has on cane processing. Dry cleaning also provides the potential of supplying large quantities of energy-rich fibre for steam generation and power production for off-crop refining, by-product manufacture or supply to the national grid. Bernhardt (1994) has reviewed in detail various methods of dry-cleaning sugar cane.

For each 1% increase in trash, Keller and Schaffer (1951) showed that fibre % cane is increased by 2.75%, as confirmed by Cargill (1976) in Natal; Blanchi & Keller (1952) showed that mill extraction is reduced by 0.40%; Cargill (1976) in South Africa found that the crushing rate is reduced by 3%; and Tsai Ming Chuin (1973) indicated that the overall recovery is reduced by 0.34%.

Rein (1975) investigated the effects of fibre and pol % cane on the extraction achieved by milling tandems, by using a statistical approach. He showed that extraction is adversely affected by high fibre levels but improves as pol % cane rises.

A cost analysis done by Cargill (1976) showed the following:

- Reducing tops and trash in cane by 3% would increase throughput (tons cane per hour TCH) by 9% with the same installed capacity, and reduce transport costs by 3%.
- Decreasing trash in cane by 1% would increase the overall recovery of pol by 0.3%.

Scott (1977) carried out full-scale tests at two milling tandems to measure the effect of fibre % cane, trash and tops in cane on throughput. Except for tops in cane, he obtained statistically significant regressions:

$$TCH = 203.3 - 5.3 \text{ fibre \% cane}$$

$$TCH = 134.3 - 3.0 \text{ trash \% cane}$$

The overall results showed that the crushing rate is reduced by about 4% for a unit rise in fibre % cane and by 2.2 to 3.0% for a unit rise in trash % cane.

Reid and Lionnet (1989) carried out full-scale processing of different types of harvested cane: clean stalk, stalk without removal of tops, trash, and tops and trash. Compared to clean stalk, the results showed that:

- The mill throughput with unburnt, untopped cane was 30% less than that with burnt, topped cane, while that with unburnt, topped cane was 22% less.
- Compared to burnt, topped cane, boiling house recovery was predicted to drop by 6% with unburnt, untopped cane.

Similar results were obtained when Lionnet (1992b) carried out tests with a diffuser instead of the milling tandems.

The effect of trash on milling has been studied by numerous workers. Most of the studies either dealt with dry trash only or a fixed pre-defined proportion of dry trash, cane tops and soil, while many others were conducted as factory trials. Using an experimental three-roller mill equipped with rollers of 10 inches diameter and 14 inches long with a hydraulic pressure of 30 tons, Arceneaux and Davidson (1944) crushed clean cane of several varieties to which were added separately green and dry trash in amounts of 2.5, 5.0 and 7.5%, with maceration water applied four times at 20% on the cane. They found that green

leaves increased the level of impurities in the juice but had little influence on the sucrose retention in the bagasse, while the opposite was true for dry trash, as they showed that green leaves contain twice as much soluble solids as did the same quantity of dry matter from dry leaves. This laboratory scale experiment had the merit of adding accurately measured amount of extraneous matter to clean cane, and determining the quality of the resulting cane, bagasse and juice. There was no evidence to suggest that the prevailing experimental conditions would be different from those which may be expected under industrial conditions.

Tsai *et al.* (1961) also used an experimental mill to crush mixtures of various types of extraneous matter with clean cane, and found the purity of the crusher juice extracted dropped by 0.5 – 0.6 unit for each unit of extraneous matter added irrespective of whether it was dry trash, green leaves, tops, roots, dead stem or soil. With a similar experimental mill, Legendre and Irvine (1974) studied the effects of a mixture of 40% dry trash and 60% cane tops in increments of 0, 5, 10, 20 and 30% by mass of cane on milling quality such as fibre % cane, juice extraction and purity of juice extracted. Hemaida *et al.* (1977) also investigated the effects of dry and green leaves at 0, 2.5, 5.0 and 7.5% cane on mixed juice extraction, bagasse % cane, sucrose lost in bagasse % cane and mixed juice purity. All the above workers also estimated the theoretical sugar recovery and sucrose loss in molasses.

1.5.3 Effects of cane quality on cane processing in Mauritius

The analysis of factory performance data conducted by Wong Sak Hoi and Autrey (1997) over the period 1960 to 1996 showed that the increase in extraneous matter in cane has increased sucrose losses in filter cake, bagasse and molasses. This is in contrast to the fact that over the same period sucrose extraction at the milling plant (mill extraction) has actually been improved as a result of heavy investment in cane preparation equipment and in milling tandems, and by the application of more imbibition water % fibre. One favourable aspect of the extra trash in cane is the increase in the mass of bagasse % cane by 4 units, which is good for energy production. However, this advantage is outweighed by the capital investment in the installation or modification of various factory equipment such as the feed table, heavy duty shredders, juice screening system and modern boilers, and by higher maintenance costs to off-set the effect of extraneous matter on factory performance.

In this section, the previous examination of the effects of cane quality on cane processing from 1960 to 1996 carried out by Wong Sak Hoi and Autrey (1997), has been updated to 2004. Parameters such as mill extraction, sucrose lost in filter cake % sucrose in cane, Clerget purity of molasses and mass of molasses at 85° Brix % cane and sugar quality were examined.

1.5.3.1 *Mill extraction*

With increased trash in cane, extraction is adversely affected. There is an increase in the energy requirement at knives and shredders, a decrease in crushing rate, imbibition efficiency and mill extraction, with increased occurrences of roll slip. The "sponge" effect of trash will also increase the sucrose lost in bagasse.

As shown in Fig 1.10, the changes in mill extraction and sucrose lost in bagasse % cane are quite complex, they have been broadly divided into three periods by Wong Sak Hoi and Autrey (1997):

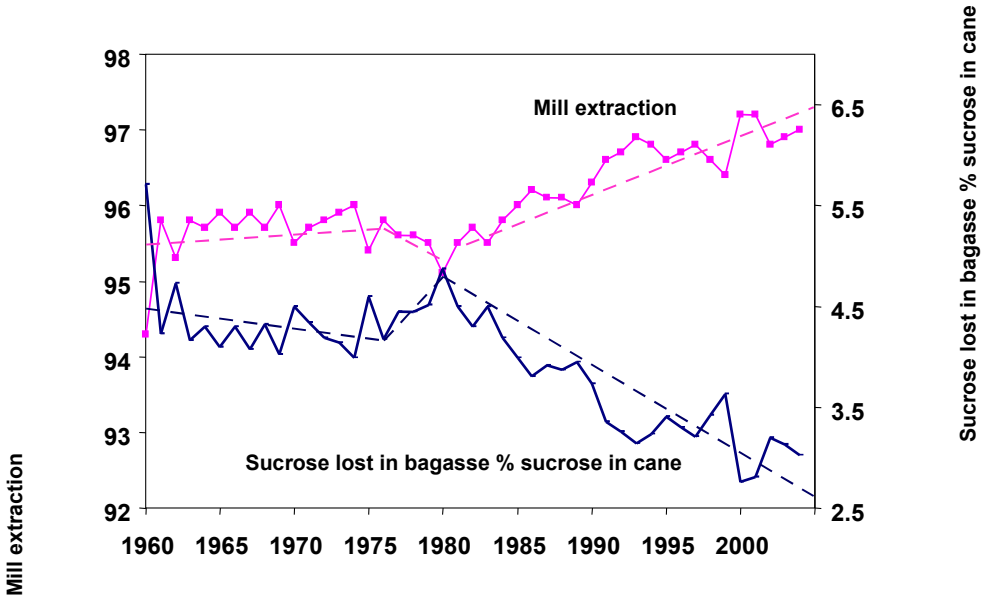


Figure 1.10. Changes in mill extraction and sucrose lost in bagasse % sucrose in cane from 1960-2004.

- From 1960 to 1976, when mill extraction was improving slightly. This was the period when mills were modernized, cane preparation was improved and arcing of mill rollers was started.

- From 1976 to 1980, when the proportion of mechanical loading increased from about 10% to 40%, the full impact of extraneous matter on mill extraction was felt, milling work was greatly hampered, and mechanical time efficiency at this period dropped steadily from 96% to 93%.
- From 1980 to 2004, there was an upward trend in mill extraction mainly due to the investment by various factories on heavy duty shredders, new milling units and mill-feeding control. Carding drums have also recently started to replace knives in cane preparation installations, and hot imbibition is being practised by certain factories since the early 1990s.

It is evident from Fig 1.10 that sucrose lost in bagasse % sucrose in cane is the mirror image of mill extraction. It is worth pointing out that since co-generation of electricity for export to the national grid has become a priority for the Mauritian sugar industry, efforts to improve the calorific value of bagasse by reducing its moisture content has resulted in record low values of pol % bagasse (Fig 1.11) and sucrose lost in bagasse % sucrose in cane, of 1.22 and 2.81 respectively in 2000 (Wong Sak Hoi, 2001), while imbibition % fibre remained low at 225 compared to South Africa, where the industrial average of imbibition % fibre was reported to be 348 in 2000 and 369 in 2004 (Anon., 2005b).

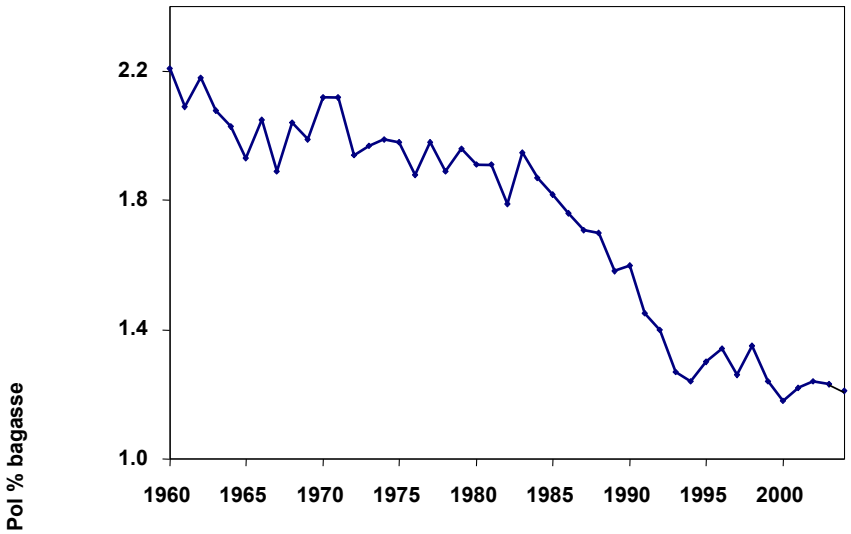


Figure 1.11. Changes in pol % bagasse from 1960-2004.

1.5.3.2 *Sucrose lost in filter cake % sucrose in cane*

Data on sucrose lost in filter cake % sucrose in cane are shown in Fig 1.12, and are of interest because from 1960 to 1975, this parameter had been decreasing, indicating an improvement in the filter station. Indeed, the industry had been gradually replacing filter presses by more efficient rotary vacuum filters. However, when mechanized cane loading was started in 1975, increased trash worsened the work at the filters and more sucrose was lost in the filter cake from 1975 to 2004. In particular, there is a marked increase from 1990, mainly because the mass of filter cake % cane had increased considerably from 3.6 in 1975 to 5.4 in 2004, because mud from effluent treatment plants is mixed with filter cake in some factories.

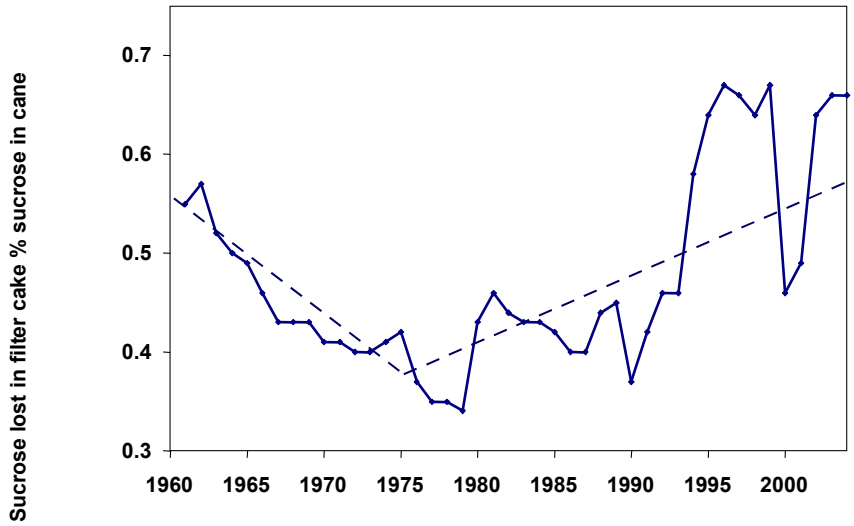


Figure 1.12. Changes in sucrose lost in filter cake % sucrose in cane from 1960-2004.

1.5.3.3 *Clerget purity of molasses and mass of molasses at 85° Brix % cane*

Changes in Clerget purity of molasses and mass of molasses at 85° Brix % cane are shown in Fig 1.13. Without taking into consideration the cyclonic years in the early 1960s, the Clerget purity of molasses before 1975 was decreasing steadily implying good molasses exhaustion. However, during the same period, the mass of molasses at 85° Brix % cane had increased with the net result that the sucrose lost in molasses % cane remained stable (Fig 1.14).

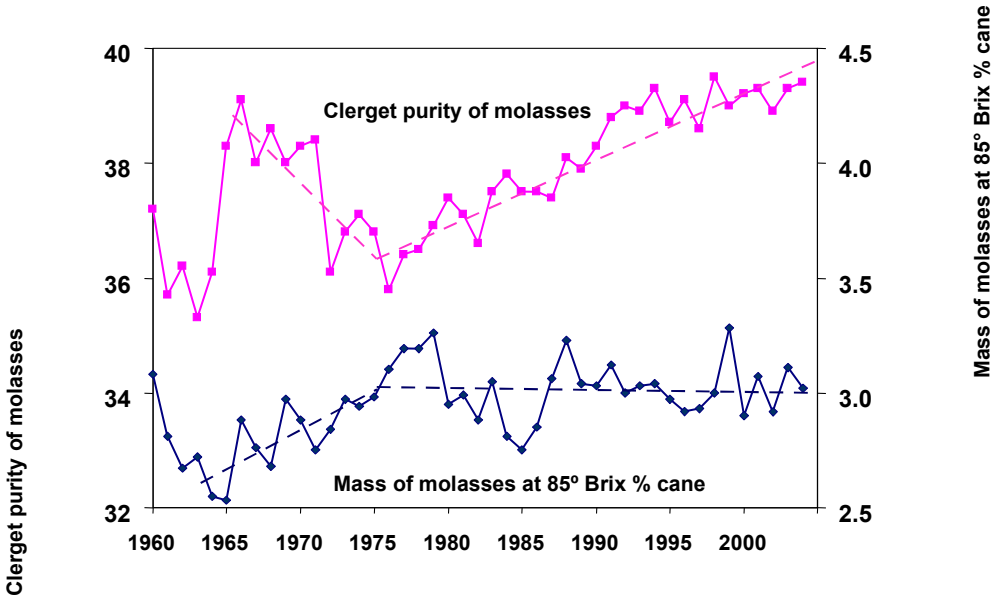


Figure 1.13. Changes in Clerget purity of molasses and mass of molasses at 85° Brix % cane from 1960-2004.

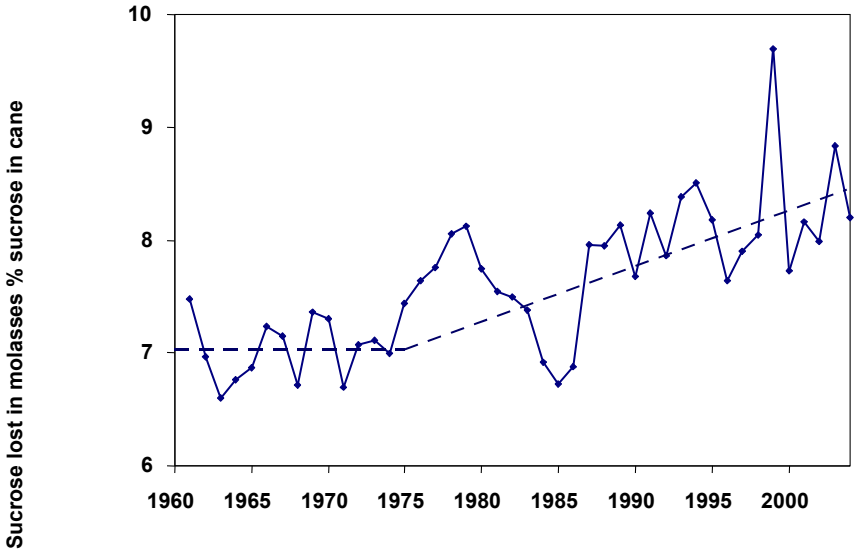


Figure 1.14. Changes in sucrose lost in molasses % sucrose in cane from 1960-2004.

After 1975, while molasses purity had increased by 3 units, the quantity of molasses produced % cane had only slightly decreased probably due to better clarification. The increase in sucrose lost in molasses % sucrose in cane was 0.8 unit from 7.4 in 1975 to 8.2 in 2004 as shown in Fig 1.14. With harvesting difficulties, considerable amounts of extraneous matter found its way into the mills together with a fair amount of immature

shoots and tops, which contain a lot of melassigenic non-sugars, retaining more sucrose in the molasses.

1.5.3.4 *Sugar quality*

Dry leaves will produce more colour initially in juice and eventually in sugar. Deterioration in sugar quality has been noted in recent years with respect to the decreasing raw sugar filterability, which is attributable to suspended solids from soil brought to the factories (Lee and Donovan, 1996).

1.6 OBJECTIVES OF THIS STUDY

The objectives of this study were to devise experiments to quantify the effect of extraneous matter on cane, bagasse and juice quality and its impact on milling performance; to develop methods to separate the sugar cane plant into fibres of its various components; and to study certain chemical properties such as the water adsorbing power and adsorption isotherms of these separated fibres in order to explain some of the effects observed when extraneous matter especially trash is added to cane.

CHAPTER 2. IMPACT OF EXTRANEOUS MATTER ON CANE JUICE QUALITY AND MILLING PERFORMANCE, AND THE PHENOMENON OF BRIX-FREE WATER IN DRY LEAF

With the increasing trend in mechanised loading of cane which started in Mauritius in the mid-1970s, more extraneous matter (EM) is now being sent with the cane to the mills. Extraneous matter in cane consists of dry and green leaves, immature cane tops, roots, dead stems, soil and any other non-cane material, all of which increase the costs of harvest and transport, as well as the cost of mill maintenance. Extraneous matter in cane may also necessitate investment in new equipment to cope with the increased crushing and milling capacity of the factory (Wong Sak Hoi and Autrey, 1997). It also lengthens the crushing season. It is therefore essential to investigate and quantify the effects of certain kinds of extraneous matter, in particular, dry leaves, green leaves and tops on cane and juice quality, on milling operations and on sugar recovery.

As already mentioned in Section 1.3, the use of the term trash or dry trash will be used to refer to the dry leaves associated with cane stalks.

In this chapter, experiments performed to study the effect of the controlled addition of extraneous matter to clean cane will be described and discussed.

2.1 EFFECT OF EXTRANEOUS MATTER ON CANE JUICE QUALITY AND MILLING PERFORMANCE

In the present study, the effects of dry trash, green leaves and immature cane tops were investigated by adding each type of EM to sub-samples of clean cane so that it constitutes 5, 10 and 20% of the total mass, and applying maceration water at 33% on cane to obtain "mixed juice", since the average maceration water used on cane in all Mauritian sugar factories in 2000 was 33.3% (Anon., 2001b). The extracted "mixed juice" samples were analysed, in particular, for fructose (F), glucose (G) and sulfated ash (Ash), to predict the sucrose loss in molasses by means of the target purity (TP) of molasses by using the South African equation (Rein and Smith, 1981):

$$TP = 33.9 - 13.4 \log_{10}\left(\frac{F + G}{Ash}\right)$$

Target purity of molasses is that level of purity below which no more sucrose can be recovered from molasses. This target purity depends on a number of factors, most importantly, the viscosity and the crystal content of the massecuite, the saturation temperature at crystallization and the nature of the non-sucrose fraction in the final molasses, namely, the reducing sugars, notably fructose and glucose, as well as the inorganic ash such as potassium, sodium, calcium and magnesium.

Normally the TP of molasses is predicted from molasses data; however, in this experiment, mixed juice data were used to calculate mixed juice-based TP by using the above equation, from which molasses-based TP can be deduced since Lionnet and Koster (1986) have shown that molasses-based TP is two units higher than the mixed juice-based TP.

In Mauritius, average monthly molasses from 17 sugar factories analysed by the Mauritius Sugar Industry Research Institute (MSIRI) over the years 1996-1999 (unpublished data) show that the average target purity difference (TPD), the difference between the molasses true purity (the percentage of sucrose in the dry solids) and its target purity, is on the rise (Table 2.1). As 1999 was an abnormal year when severe drought conditions prevailed, the 1998 average of 2.4 units was added to the estimated molasses-based TP to predict the expected true purity of Mauritian molasses. According to the 1998 data, Clerget purity is 0.1 unit higher than the true purity (not shown), therefore a further 0.1 unit was added. Hence, a constant totalling 4.5 units (2 + 2.4 + 0.1) was added to the mixed juice-based TP to predict Clerget purity of molasses.

Table 2.1. Mauritian average of molasses target purity difference (TPD) compiled from MSIRI unpublished data.

	July	August	September	October	Mean
1999	3.6	3.4	2.5	1.7	**2.8**
1998	3.4	1.9	2.2	2.2	**2.4**
1997	2.6	2.3	1.3	1.9	**2.0**
1996	-	1.7	1.7	1.8	**1.7**

Since the extrapolated purity of molasses (M) and the purities of mixed juice (J) and sugar (S) were known, the SJM formula of Deerr (1921) could be used to predict the portion of the sucrose in the original material that goes to the sugar produced, after deduction of sucrose losses in filter cake and undetermined losses. The latter is the unaccounted sucrose loss when sucrose is extracted from cane after taking into account the sucrose losses in bagasse, in filter cake and in molasses.

The SJM formula states that given a juice (or initial material, e.g. syrup or massecuite) of J purity and producing a sugar of S purity with a molasses of M purity, the percentage of the total sucrose (or pol) in the original material to go into the sugar will be 100S(J-M)/[J(S-M)]. The formula predicts the portion of the sucrose (or pol) in the original material that goes to the sugar produced, and the remainder goes to the molasses. No losses of any kind are taken into account in this formula.

In this experiment, sucrose losses in filter cake and undetermined losses were unknown, and they were assumed to be 0.08 and 0.13% cane, respectively. These were the 1998 island average (Anon., 1999). When these values are compared to the 2004 values (Anon., 2005a) of 0.08 and 0.08% cane, respectively, it can be seen that they have remained more or less constant.

The experiment also enabled the estimation of mill extraction (or more appropriately, press extraction, in the context in which the experiment was carried out); sucrose losses in bagasse and in molasses, boiling house recovery and overall recovery. Mill extraction is defined as the percent mass of pol originally present in the cane that has been extracted into the mixed juice. Boiling house recovery is the percentage of the pol in mixed juice that passes into the sugar produced. The product of mill extraction and boiling house recovery is known as overall recovery.

2.1.1 Materials

For each trial of EM addition to clean cane, about 2 kg each of dry leaves, green leaves and cane tops, and about 20 kg of cane stalks were obtained from a neighbouring sugar factory. The cane stalks were cleaned of dry leaves, green leaves and cane tops.

1.3.1 *Mercuric iodide juice preservative*

This is prepared by dissolving 500 g of red mercuric iodide in a saturated aqueous solution of potassium iodide and made up to a total volume of 1 L. The potassium iodide solution

is prepared by dissolving 800 g potassium iodide in 500 mL of warm distilled water. The juice preservative is applied at a rate of 0.5 mL per litre of sample.

2.1.2 Equipment

A heavy duty universal model 264 Jeffco cutter-grinder (Fig 2.1) with a 7500 W, three phase motor, was suitable for shredding the sugar cane stalk.

A cane chipper (Fig 2.2) with four steel blades bolted onto a horizontal, cylindrical shaft, which rotates at a high speed, was used to shred cane into pieces about 2 – 3 cm long. Cane was fed manually to the electrically-driven rotating knives.

Juice was expressed from the cane by means of a Pinette Emidecau hydraulic press (Fig 2.3) capable of exerting a pressure of up to 25 MPa to sample contained in a stainless steel cup.

A Schmidt and Haensch DUR W2 sugar refractometer (Fig 2.4) was used to measure the refractive index of sugar solutions and displayed the results as dissolved solids (g) in 100 g solution (Brix).

A Schmidt and Haensch Saccharomat sugar polarimter (Fig 2.5) was used to measure the pol and Clerget sucrose of mixed juice, and pol in bagasse, cane, dry leaf, green leaf and in tops.

A Dionex high performance ion chromatograph (Fig 2.6) equipped with an eluant degas module model EDM-2 and a pulsed amperometric detector was used to determine glucose, fructose and sucrose. Separation was achieved at ambient temperature on a Dionex CarboPac PA1 column of 250 mm length and 4 mm internal diameter. The column was preceded by a guard column packed with the same material. Sodium hydroxide solution (150 mmol L^{-1}) was used as eluant at a flow rate of 1 mL per minute.

Figure 2.1. Jeffco cutter grinder.

Figure 2.2. Cane chipper.

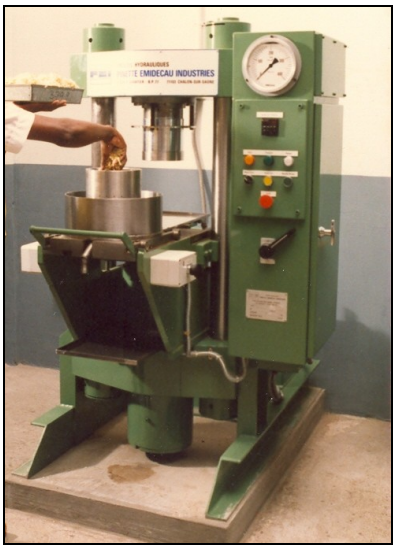


Figure 2.3. Pinette Emidecau Press.

Figure 2.4. Sugar refractometer.

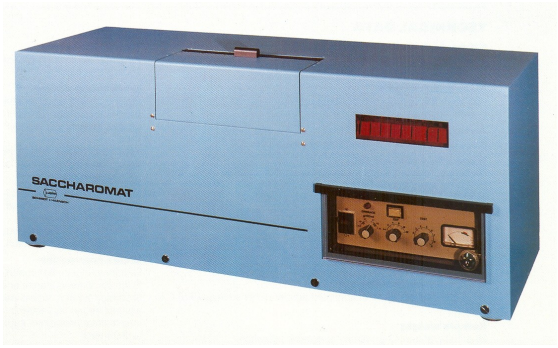


Figure 2.5. Sugar polarimeter

Figure 2.6. High Performance Ion Chromatograph

2.1.3 Methodology

A mass of 2 kg each of dry trash, green leaves and immature cane tops without attached foliage above the natural breaking point of cane stalk, were shredded separately in a Jeffco cutter-grinder (Fig 2.1). Subsequently 20 kg of clean cane stalks, which had previously been chipped coarsely in a cane chipper (Fig 2.2), were shredded. The finely divided clean cane was well mixed; a sub-sample of 1329 g was taken, 329 g of which was analysed for pol % cane and fibre % cane by the *Société de Technologie Agricole et Sucrière de Maurice* (STASM) method (Anon., 1991). Simultaneously, one kg of the clean cane was subjected to a pressure of 20 MPa (200 bar) for two minutes in a Pinette Emidecau hydraulic press (Fig 2.3) to obtain first expressed juice. Distilled water (330 mL) was sprayed onto the pressed cake and a second pressing carried out to obtain "mixed juice". The mass of the cake was weighed as bagasse, of which 50 g were analysed for moisture content and 150 g for Brix and pol (for method, see Appendix 1). Fibre % bagasse was taken as 100 – moisture % bagasse – Brix % bagasse. Mercuric iodide juice preservative was added to the mixed juice at the rate of 0.5 mL L^{-1} before analysis for Brix by a refractometer (Fig 2.4), pol and Clerget sucrose by a polarimeter (Fig 2.5) by the STASM method (Anon., 1991), sulfated ash content (for method, see Appendix 2), and glucose, fructose and sucrose by high performance ion chromatography making use of the official International Commission for Uniform Methods of Sugar Analysis (ICUMSA) method for molasses modified for juices (Schäffler, 1994) using lactose as internal standard. Clerget purity was obtained by dividing Clerget sucrose by the Brix, expressed as a percentage.

The experiment was repeated with shredded dry trash, green leaves and cane tops added separately to sub-samples of the shredded clean cane so that the added EM constituted 5%, 10% and 20% of the total mass which was 1329 g. The experiment was concluded with a second sub-sample of clean cane alone, to ascertain that no deterioration of the cane had occurred. This was assessed by ensuring that the pol of the second mixed juice was not less than 0.05% lower than the first one, and the average of the two sets of clean cane data was taken. As an indication, the eleven "mixed juice" samples were processed not later than two hours after the start of the experiment. The composition in terms of moisture, Brix and pol of shredded dry trash, green leaves and cane tops were determined as for bagasse (Appendix 1).

Six trials were carried out on fresh supply of cane and EM.

2.1.4 Results

The analytical results of the six trials with addition of dry trash, green leaves and cane tops to clean cane are presented on CD (file: Raw data for Tables 2.2-2.4.xls). Averages of the six trials with addition of dry trash, green leaf and tops are shown in Tables 2.2 – 2.4. The first series of data in each category of clean cane and various additions of EM refer to the first trial, and so on. It is of note that Clerget sucrose results are in agreement with HPIC sucrose. The composition of the extraneous matter is compared to that of clean cane in Table 2.5 (samples from Trial 1 were not analysed). It is worth pointing out that the moisture of dry trash collected could be twice as much on a wet day than on a dry day (34.4% compared to 17.9%). In Table 2.5, apparent purity is obtained by dividing pol % by Brix %, expressed as percentage, and non-pol is taken as: Brix - pol.

The influence of extraneous matter in cane is indicated by the mean analytical data compiled on the quality of mixed juice, bagasse and cane.

Glucose, fructose and ash contents of mixed juice were used to calculate mixed juice-based target purity, to which were added 4.5 units to predict Clerget purity of molasses (Table 2.6), as explained earlier.

Calculation of SJM sucrose recovery % sucrose in mixed juice leads to (i) g sucrose in sugar/kg cane, which after subtracting the assumed sucrose losses in filter cake and undetermined losses totalling 0.21% cane, yields sucrose recovery % cane, and (ii) g sucrose in molasses/kg cane and mass of molasses % cane.

The data summarised in Table 2.6 enabled the calculation of mill extraction, boiling house recovery and overall recovery (Table 2.7) since, by definition, mill or press extraction is the percentage mass of sucrose originally in the cane that has been extracted into the mixed juice. Boiling house recovery is by definition, the percentage of sucrose in the mixed juice that passes into the sugar produced, and overall recovery is the percentage of sucrose in the cane that passes into the sugar produced. Brix and pol data of mixed juice in Tables 2.2 – 2.4 also allowed the determination of commercial cane sugar (CCS), which is computed from the following Australian equation (Anon., 1984):

$$CCS = \frac{3}{2}\,pol\left[\frac{100 - (fibre\,\%\,cane + 5)}{100}\right] - \frac{1}{2}\,Brix\left[\frac{100 - (fibre\,\%\,cane + 3)}{100}\right]$$

in which pol and Brix refer to the first expressed juice.

Table 2.2. Analytical results of resulting mixed juice, bagasse and cane with increased addition of dry trash (trials 1-6).

		Mixed juice										Bagasse					Cane	
Additive to clean cane		Mass/g from one kg gross cane*	Clerget sucrose (%)	Brix (%)	Pol (%)	Clerget purity	HPIC glucose (%)	HPIC fructose (%)	HPIC sucrose (%)	Ash (%)	Mixed juice based TP	Mass/g from one kg gross cane*	Pol (%)	Brix (%)	Moisture (%)	Fibre (%)	Pol (%)	Fibre (%)
Nil	1	1077.8	14.78	16.29	15.09	90.7	0.074	0.068	14.69	0.284	37.9	252.2	3.18	4.74	38.87	56.4	17.28	14.96
	2	1105.0	13.16	14.43	13.37	91.2	0.042	0.041	13.23	0.386	42.8	225.0	1.67	5.95	43.51	50.5	15.80	10.81
	3	1066.6	14.69	16.48	15.02	89.1	0.136	0.136	14.72	0.423	36.5	263.4	2.23	5.78	40.23	54.0	17.07	14.61
	4	1064.9	14.24	15.76	14.47	90.4	0.151	0.144	14.17	0.341	34.7	265.1	2.51	6.90	44.14	49.0	16.60	12.77
	5	1063.7	13.95	15.85	14.09	88.0	0.284	0.261	13.94	0.305	30.5	266.3	2.53	7.04	42.68	50.3	16.28	13.33
	6	1081.3	15.29	16.88	15.49	90.6	0.159	0.160	15.30	0.376	34.9	248.7	1.23	5.10	40.71	54.2	17.06	13.42
Mean		1076.6	14.35	15.95	14.59	90.0	0.141	0.135	14.34	0.353	36.2	253.5	2.23	5.92	41.69	52.4	16.68	13.32
Dry trash 5%	1	1024.6	14.53	16.25	14.96	89.4	0.080	0.072	14.62	0.321	38.3	305.4	3.06	4.85	39.30	55.9	16.66	17.63
	2	1046.6	12.91	**14.49**	13.26	89.1	0.083	0.067	12.95	0.460	40.4	283.4	2.01	6.39	46.80	46.8	15.23	13.25
	3	1003.9	**14.82**	**16.63**	**15.06**	89.1	0.148	0.145	**14.92**	0.467	36.6	326.1	2.17	4.99	38.96	56.1	16.62	17.02
	4	1010.3	14.21	**16.11**	**14.56**	88.2	0.175	0.167	14.15	0.435	35.3	319.7	2.25	5.51	40.02	54.5	15.95	15.99
	5	1014.8	13.85	15.79	14.06	87.7	0.277	0.250	13.89	0.391	32.2	315.2	2.25	6.03	40.18	53.8	15.58	16.53
	6	1017.0	15.14	**16.98**	15.43	89.2	0.171	0.169	15.14	0.432	35.3	313.0	2.18	5.32	42.90	51.8	16.33	16.50
Mean		1019.5	14.24	16.04	14.56	88.8	0.156	0.145	14.28	0.418	36.3	310.5	2.32	5.52	41.36	53.1	16.06	16.15
Dry trash 10%	1	970.8	14.28	**16.48**	14.98	86.7	0.116	0.100	**15.39**	0.390	37.3	359.2	3.68	5.31	37.11	57.6	15.87	20.91
	2	1000.7	12.58	14.31	12.81	87.9	0.135	0.109	12.57	0.528	38.4	329.3	1.37	6.84	42.66	50.5	14.58	14.89
	3	950.1	14.67	**16.69**	14.90	87.9	0.159	0.165	**14.81**	0.500	36.4	379.9	2.12	6.96	39.34	53.7	15.80	20.33
	4	959.9	13.71	15.69	14.09	87.4	0.184	0.180	13.82	0.436	35.0	370.1	2.39	6.99	42.62	50.4	15.45	18.88
	5	944.7	13.54	15.55	13.73	87.1	0.285	0.259	13.53	0.405	32.2	385.3	2.05	6.55	40.06	53.4	14.78	18.97
	6	958.0	14.77	16.72	15.02	88.3	0.183	0.179	14.85	0.486	35.6	372.0	1.77	6.14	41.06	52.8	15.96	19.12
Mean		964.0	13.93	15.91	14.26	87.5	0.177	0.165	14.16	0.458	35.8	366.0	2.23	6.47	40.48	53.1	15.41	18.85
Dry trash 20%	1	823.9	13.78	16.07	14.30	85.7	0.149	0.138	14.83	0.504	37.2	506.1	3.60	5.70	38.12	56.2	14.96	28.42
	2	882.9	11.83	14.11	12.20	83.8	0.217	0.166	11.81	0.664	37.1	447.1	2.13	7.95	40.96	51.1	13.21	19.85
	3	840.2	13.96	16.24	14.26	86.0	0.175	0.195	13.93	0.660	37.3	489.8	2.01	6.44	38.00	55.6	14.92	25.78
	4	841.0	13.27	15.58	13.65	85.2	0.215	0.213	13.25	0.543	35.3	489.0	2.25	7.61	42.90	49.5	13.90	24.50
	5	807.7	13.00	15.26	13.21	85.2	0.294	0.263	12.96	0.513	33.4	522.3	2.46	7.18	40.74	52.1	13.79	24.68
	6	837.0	13.28	16.26	14.33	81.7	0.200	0.200	13.44	0.567	35.9	493.0	2.12	6.75	38.56	54.7	14.32	25.60
Mean		838.8	13.19	15.59	13.66	84.6	0.208	0.196	13.37	0.575	36.0	491.2	2.43	6.94	39.88	53.2	14.18	24.81

* Gross cane refers to clean cane + EM

Table 2.3. Analytical results of resulting mixed juice, bagasse and cane with increased addition of green leaf (trials 1-6).

		Mixed juice										Bagasse					Cane	
Additive to clean cane		Mass/g from one kg gross cane*	Clerget sucrose (%)	Brix (%)	Pol (%)	Clerget purity	HPIC glucose (%)	HPIC fructose (%)	HPIC sucrose (%)	Ash (%)	Mixed juice based TP	Mass/g from one kg gross cane*	Pol (%)	Brix (%)	Moisture (%)	Fibre (%)	Pol (%)	Fibre (%)
Nil	1	1077.8	14.78	16.29	15.09	90.7	0.074	0.068	14.69	0.284	37.9	252.2	3.18	4.74	38.87	56.4	17.28	14.96
	2	1105.0	13.16	14.43	13.37	91.2	0.042	0.041	13.23	0.386	42.8	225.0	1.67	5.95	43.51	50.5	15.80	10.81
	3	1066.6	14.69	16.48	15.02	89.1	0.136	0.136	14.72	0.423	36.5	263.4	2.23	5.78	40.23	54.0	17.07	14.61
	4	1064.9	14.24	15.76	14.47	90.4	0.151	0.144	14.17	0.341	34.7	265.1	2.51	6.90	44.14	49.0	16.60	12.77
	5	1063.7	13.95	15.85	14.09	88.0	0.284	0.261	13.94	0.305	30.5	266.3	2.53	7.04	42.68	50.3	16.28	13.33
	6	1081.3	15.29	16.88	15.49	90.6	0.159	0.160	15.30	0.376	34.9	248.7	1.23	5.10	40.71	54.2	17.06	13.42
Mean		1076.6	14.35	15.95	14.59	90.0	0.141	0.135	14.34	0.353	36.2	253.5	2.23	5.92	41.69	52.4	16.68	13.32
Green leaf	1	1060.7	14.34	16.10	14.68	89.1	0.098	0.085	14.31	0.334	37.4	269.3	2.90	4.54	38.26	57.2	16.63	15.68
	2	1086.2	12.49	14.29	13.02	87.4	0.074	0.068	12.56	0.454	40.7	243.8	2.40	5.33	45.34	49.3	14.93	11.55
	3	1044.9	14.15	16.15	14.47	87.6	0.183	0.211	14.23	0.470	34.9	285.1	0.68	5.31	40.60	54.1	16.09	15.53
	4	1048.4	13.91	15.53	14.10	89.6	0.167	0.150	13.76	0.408	35.4	281.6	2.25	6.06	45.64	48.3	16.00	13.62
	5	1040.3	13.53	15.53	13.75	87.1	0.289	0.258	13.63	0.359	31.4	289.7	2.53	7.10	43.82	49.1	15.49	14.62
	6	1060.0	14.57	16.43	14.84	88.7	0.176	0.171	14.58	0.428	35.1	270.0	1.91	5.43	44.32	50.3	16.27	14.35
Mean		1056.8	13.83	15.67	14.14	88.2	0.165	0.157	13.85	0.409	35.8	273.3	2.11	5.63	43.00	51.4	15.90	14.23
Green leaf 10%	1	1051.8	14.31	15.73	14.41	91.0	0.117	0.130	14.25	0.404	36.8	278.2	1.64	4.31	38.15	57.5	15.83	16.35
	2	1077.2	12.08	13.70	12.47	88.2	0.098	0.088	12.05	0.458	39.1	252.8	2.39	5.12	45.82	49.1	14.69	11.95
	3	1036.6	13.83	15.93	13.99	86.8	0.223	0.209	13.87	0.505	34.8	293.4	1.91	5.20	39.56	55.2	15.62	15.71
	4	1027.4	13.37	15.48	13.78	86.4	0.189	0.173	13.45	0.491	35.7	302.6	2.12	6.48	44.70	48.8	15.60	14.41
	5	1037.3	13.12	15.33	13.38	85.6	0.319	0.275	12.15	0.421	31.9	292.7	2.46	6.46	41.62	51.9	15.27	14.98
	6	1050.1	13.93	15.90	14.21	87.6	0.183	0.175	13.97	0.464	35.4	279.9	1.37	5.32	43.02	51.7	15.96	15.32
Mean		1046.7	13.44	15.35	13.71	87.6	0.188	0.175	13.29	0.457	35.6	283.3	1.98	5.48	42.15	52.4	15.50	14.79
Green leaf 20%	1	1006.6	13.49	15.21	13.56	88.7	0.149	0.169	13.60	0.516	36.7	323.4	2.94	4.54	41.46	54.0	14.79	17.20
	2	1066.7	11.02	12.81	11.45	86.0	0.145	0.133	11.18	0.531	37.7	263.3	1.77	4.91	45.08	50.0	13.30	12.52
	3	1014.4	12.64	14.94	12.68	84.6	0.303	0.285	12.58	0.546	33.5	315.6	1.57	5.32	42.34	52.3	14.03	16.47
	4	1001.0	12.37	14.78	12.79	83.7	0.201	0.187	12.49	0.625	36.7	329.0	1.98	5.64	43.58	50.8	14.15	15.93
	5	1000.1	12.17	14.66	12.43	83.0	0.305	0.269	13.10	0.531	33.4	329.9	2.39	6.36	43.30	50.3	14.00	16.44
	6	1017.0	12.99	15.25	13.39	85.2	0.199	0.186	13.08	0.562	36.1	313.0	2.05	5.32	42.88	51.8	14.80	16.22
Mean		1017.6	12.45	14.61	12.72	85.2	0.217	0.205	12.67	0.552	35.7	312.4	2.12	5.35	43.11	51.5	14.18	15.80

* Gross cane refers to clean cane + EM

Table 2.4. Analytical results of resulting mixed juice, bagasse and cane with increased addition of cane tops (trials 1-6).

		Mixed juice										Bagasse					Cane	
Additive to clean cane		Mass/g from one kg gross cane*	Clerget sucrose (%)	Brix (%)	Pol (%)	Clerget purity	HPIC glucose (%)	HPIC fructose (%)	HPIC sucrose (%)	Ash (%)	Mixed juice based TP	Mass/g from one kg gross cane*	Pol (%)	Brix (%)	Moisture (%)	Fibre (%)	Pol (%)	Fibre (%)
Nil	1	1077.8	14.78	16.29	15.09	90.7	0.074	0.068	14.69	0.284	37.9	252.2	3.18	4.74	38.87	56.4	17.28	14.96
	2	1105.0	13.16	14.43	13.37	91.2	0.042	0.041	13.23	0.386	42.8	225.0	1.67	5.95	43.51	50.5	15.80	10.81
	3	1066.6	14.69	16.48	15.02	89.1	0.136	0.136	14.72	0.423	36.5	263.4	2.23	5.78	40.23	54.0	17.07	14.61
	4	1064.9	14.24	15.76	14.47	90.4	0.151	0.144	14.17	0.341	34.7	265.1	2.51	6.90	44.14	49.0	16.60	12.77
	5	1063.7	13.95	15.85	14.09	88.0	0.284	0.261	13.94	0.305	30.5	266.3	2.53	7.04	42.68	50.3	16.28	13.33
	6	1081.3	15.29	16.88	15.49	90.6	0.159	0.160	15.30	0.376	34.9	248.7	1.23	5.10	40.71	54.2	17.06	13.42
Mean		1076.6	14.35	15.95	14.59	90.0	0.141	0.135	14.34	0.353	36.2	253.5	2.23	5.92	41.69	52.4	16.68	13.32
Tops 5%	1	1079.3	14.19	15.84	14.71	89.6	0.126	0.114	14.09	0.354	36.2	250.7	3.28	4.70	37.23	58.1	16.59	14.80
	2	1113.7	12.37	13.93	12.77	88.8	0.118	0.115	12.32	0.415	37.3	216.3	1.37	5.12	44.34	50.5	14.90	10.52
	3	1068.8	14.13	15.95	14.29	88.6	0.171	0.167	14.16	0.482	36.0	261.2	2.39	5.00	41.30	53.7	16.19	14.16
	4	1063.5	13.71	15.40	13.95	89.0	0.179	0.184	13.68	0.373	34.1	266.5	2.05	6.16	44.36	49.5	16.15	13.28
	5	1048.5	13.35	15.35	13.54	87.0	0.294	0.265	13.33	0.343	31.1	281.5	2.66	6.57	42.92	50.5	15.59	13.98
	6	1073.6	14.77	16.59	14.89	89.0	0.211	0.216	14.73	0.425	33.9	256.2	0.96	5.64	43.96	50.4	16.67	13.92
Mean		1074.6	13.75	15.51	14.03	88.7	0.183	0.177	13.72	0.399	34.7	255.4	2.12	5.53	42.35	52.1	16.02	13.44
Tops 10%	1	1068.5	13.69	15.43	14.04	88.7	0.158	0.204	13.61	0.421	34.8	261.5	2.22	4.47	40.81	54.7	15.97	14.69
	2	1122.4	11.75	13.43	12.08	87.5	0.160	0.158	11.80	0.419	35.5	207.6	0.75	5.12	45.22	49.7	14.31	10.33
	3	1074.2	13.04	15.22	13.43	85.7	0.194	0.189	12.97	0.511	35.6	255.8	2.18	5.30	40.16	54.5	15.52	13.89
	4	1065.6	13.22	15.04	13.47	87.9	0.201	0.204	13.17	0.406	33.9	264.4	0.68	6.16	43.66	50.2	15.70	13.28
	5	1056.0	12.69	14.99	12.98	84.7	0.351	0.331	12.74	0.379	30.5	274.0	2.59	5.73	41.44	52.8	14.83	13.95
	6	1080.7	14.09	16.50	14.62	85.4	0.264	0.263	14.01	0.460	33.1	249.3	1.77	5.11	42.96	51.9	16.44	14.16
Mean		1077.9	13.08	15.10	13.44	86.6	0.221	0.225	13.05	0.433	33.9	252.1	1.70	5.32	42.38	52.3	15.46	13.38
Tops 20%	1	1086.1	12.38	14.49	12.65	85.4	0.236	0.231	12.46	0.557	34.9	243.9	2.70	4.54	41.92	53.5	14.88	14.18
	2	1125.3	10.53	12.41	10.78	84.9	0.234	0.233	10.66	0.480	34.1	204.7	0.67	4.39	44.90	50.7	12.79	10.30
	3	1078.5	11.79	14.21	12.17	83.0	0.234	0.235	11.82	0.569	35.0	251.5	1.09	4.58	40.40	55.0	14.14	13.86
	4	1068.9	12.23	14.32	12.52	86.1	0.240	0.260	12.30	0.424	32.9	261.1	2.46	6.16	43.32	50.5	14.45	13.22
	5	1060.2	11.44	13.90	11.59	82.3	0.417	0.402	11.50	0.452	30.4	269.8	2.39	6.26	43.62	50.1	13.78	13.47
	6	1086.1	13.63	16.13	13.90	84.5	0.340	0.340	13.62	0.543	32.6	243.9	2.39	5.10	41.12	53.8	15.69	14.25
Mean		1084.2	12.00	14.24	12.27	84.4	0.284	0.284	12.06	0.504	33.3	245.8	1.95	5.17	42.55	52.3	14.29	13.21

* Gross cane refers to clean cane + EM

Table 2.5. Composition of dry trash, green leaf, cane top and clean cane.

		Brix/%	Pol/%	Apparent purity	Moisture/ %	Fibre/%	Non-pol/%
Dry trash	2	9.31	0.19	2.04	34.38	56.31	9.12
	3	8.67	0.19	2.19	20.36	70.97	8.48
	4	9.98	0.34	3.41	17.88	72.14	9.64
	5	8.31	0.41	4.93	20.08	71.61	7.90
	6	8.57	0.48	5.60	20.22	71.21	8.09
Mean		8.97	0.32	3.63	22.58	68.45	8.65
Green leaf	2	6.11	0.90	14.73	71.38	22.51	5.21
	3	6.73	0.82	12.18	68.94	24.33	5.91
	4	9.77	1.77	18.12	61.64	28.59	8.00
	5	10.08	1.71	16.96	59.40	30.52	8.37
	6	8.33	1.09	13.09	68.34	23.33	7.24
Mean		8.20	1.26	15.02	65.94	25.86	6.95
Tops	2	4.45	0.61	13.71	86.30	9.25	3.84
	3	4.65	0.48	10.32	82.30	13.05	4.17
	4	8.29	0.48	5.79	76.06	15.65	7.81
	5	6.95	0.27	3.88	77.14	15.91	6.68
	6	13.35	3.07	23.00	68.14	18.51	10.28
Mean		7.54	0.98	11.34	77.99	14.47	6.56
Clean cane	2	19.57	17.28	88.30	65.47	14.96	2.29
	3	18.64	15.80	84.76	70.55	10.81	2.84
	4	19.57	17.07	87.23	65.82	14.61	2.50
	5	19.31	16.60	85.97	67.92	12.77	2.71
	6	19.03	16.28	85.55	67.64	13.33	2.75
Mean		19.22	16.61	86.36	67.48	13.30	2.62

Table 2.6. Predicted molasses Clerget purity and sugar recovery with increasing amounts of extraneous matter (EM) in cane.

EM added		Sucrose (g) from kg gross cane in			Predicted Clerget purity of molasses	SJM recovery in sugar (%)	Sucrose in sugar (g)/ kg cane	Sucrose in molasses (g)/ kg cane	Mass of molasses at 85° Brix % cane	Sucrose recovery % cane	98.5° sugar recovery % cane
		bagasse	mixed juice	cane							
	0%	5.7	154.5	160.1	40.7	92.4	142.7	11.7	2.5	14.06	14.28
Dry trash	5%	7.2	145.1	152.3	40.8	91.3	132.5	12.6	2.6	13.04	13.24
	10%	8.2	134.2	142.3	40.3	90.4	121.2	12.9	2.7	11.91	12.09
	20%	12.0	110.5	122.5	40.5	87.6	96.8	13.8	2.9	9.47	9.61
Green leaf	5%	5.7	146.1	151.8	40.3	90.9	132.9	13.2	2.8	13.08	13.28
	10%	5.6	140.6	146.2	40.1	90.5	127.4	13.3	2.8	12.53	12.72
	20%	6.7	126.5	133.2	40.2	88.3	111.8	14.7	3.1	10.97	11.14
Cane tops	5%	5.5	147.7	153.2	39.2	91.8	135.6	12.2	2.6	13.35	13.55
	10%	4.4	140.9	145.3	38.4	90.4	127.4	13.5	3.0	12.53	12.72
	20%	4.9	130.0	134.9	37.8	88.7	115.4	14.7	3.3	11.33	11.50

Table 2.7. Predicted factory performance with increased extraneous matter (EM) in cane.

EM added		Press extraction/%	Boiling house recovery/%	Overall recovery/%	CCS calculated from mixed juice
	0%	96.5	91.0	87.8	14.89
Dry trash	5%	95.3	89.8	85.6	14.26
	10%	94.3	88.8	83.7	13.37
	20%	90.3	85.7	77.3	11.62
Green leaves	5%	96.2	89.5	86.1	14.16
	10%	96.1	89.0	85.6	13.53
	20%	95.0	86.7	82.4	12.20
Cane tops	5%	96.4	90.3	87.1	14.19
	10%	97.0	88.9	86.3	13.47
	20%	96.4	87.1	84.0	12.07

As the mixed juice was diluted 1.33 times by the imbibition water sprayed on the cake, CCS values were calculated from the mixed juice data and then multiplied by a factor of 1.33 (Table 2.7).

The manner in which the parameters listed in Tables 2.2 – 2.7 were calculated are detailed in Appendix 3.

2.1.5 Validity of the assumption made in the experimentation

This experiment provides an insight into the relative effect of measured quantities of dry leaves, green leaves and tops added to clean cane of the same quality on cane processing which is impossible to obtain under factory conditions. The mixed juiced-based TP, the Mauritian TP and the 'mixed juice' quality leads to the quality and quantity of molasses, which enables the prediction of values to the whole host of factory performance parameters, which has never been done before.

The only one laboratory experiment carried out with known quantities of 2.5, 5.0 and 7.5% each of dry leaves and green leaves added to clean cane was done by Hemaida *et al.* (1977), who reported on the quality and quantity of the mixed juice and bagasse obtained, and calculated the sugar yield and sugar losses in molasses by a formula simplified by Herbert (1973).

Until there is a simple method of quantifying extraneous matter in cane and representative sub-samples of all the cane supplied to factories are analysed, no industrial average of EM in cane will be available. Tulip and Moore (2007) recently showed that using a technique

known as 'linear spectral unmixing', the differences among cane, leaves and top could be detected by visible and very near infrared (VIS-VNIR) spectra, showing promise for online monitoring of trash levels in cane fed to milling train.

In Mauritius at the end of the millennium, the guess was that dry trash was as high as 7%, and that green leaves and tops each amounted to less than 5%. Examination of the experimental and predicted factory performance data obtained in the trial tests due to 5% each of dry trash, green leaves and cane tops shows that they agree well with the Mauritian industrial average, for example in 1998 (Anon., 1999) and more recently in 2006 (Anon., 2007) (Table 2.8). Experimental data indicate that the clean cane used is very rich in sucrose, hence the relatively high figures of experimental mixed juice purity and the predicted sucrose recovery % cane. Although press extraction is probably not as efficient as mill extraction, the predicted boiling house recovery and overall recovery are comparable with the industrial data.

This shows the validity of the assumption made in the experiment that molasses-based TP is two units higher than mixed juice-based TP. The experiment enables the precise measure of each type of EM in cane, as well as the relative effect of dry trash, green leaves and cane tops on factory performance, contrary to trials carried out by various research workers in factories, where the level and type of EM are difficult to maintain constant.

Whether the effect of each type of EM on milling performance is additive, however, could be the subject of further study, and mill modelling with the juice quality obtained could also be undertaken.

The results obtained will be further discussed in the following section in terms of the impact of extraneous matter on factory performance.

Table 2.8. Comparison of experimental and predicted data with Mauritian industrial average.

	Experimental and predicted data				Mauritian industrial average	
	clean cane	EM in cane			1998	2006
		5% dry trash	5% green leaves	5% cane tops		
Sucrose % cane	16.0	15.2	15.2	15.3	11.2	12.0
Fibre % cane	13.3	16.2	14.2	13.4	16.0	15.9
Mixed juice purity	90.0	88.8	88.2	88.7	85.2	86.4
Pol % bagasse	2.2	2.3	2.1	2.1	1.2	1.2
Clerget purity of molasses	40.7	40.8	40.3	39.2	39.0	39.5
Mass of molasses at 85°Brix % cane	2.5	2.6	2.8	2.6	3.3	3.0
Sucrose recovery % cane	14.1	13.0	13.1	13.4	9.5	10.5
Press extraction	96.5	95.3	96.2	96.4	-	-
Mill extraction	-	-	-	-	96.4	96.9

Boiling house recovery	91.0	89.8	89.5	90.3	87.9	90.0
Overall recovery	87.8	85.6	86.1	87.1	84.7	87.2

2.1.6 Impact of extraneous matter on milling performance

The influence of increasing levels of dry trash, green leaves and cane tops on the quality of cane, bagasse, mixed juice and molasses, juice extraction, sugar recovery, CCS, boiling house recovery and overall recovery are clearly illustrated in Figs 2.7 – 2.22. In most cases, linear relationships were found. Each of these will now be discussed in turn.

2.1.6.1 *Cane quality*

The change of pol % cane due to increasing levels of extraneous matter (Fig 2.7) clearly shows that each unit increase in dry trash (D) causes a 0.13 unit decrease in pol % cane. Similarly, each unit increase in green leaves (G) and tops (T) entails corresponding decreases of 0.13 and 0.12 in pol % cane, all changes being related to a pol % clean cane value of 16.7 (Table 2.2). Sucrose % cane which was not determined directly but calculated from the sucrose contents in mixed juice and bagasse, was affected to different degrees by the nature of the extraneous matter; the decrease in sucrose % cane is more severe in the case of dry trash than for green leaves and cane tops (Fig 2.8).

Fibre % cane is markedly increased by dry trash, less so by green leaves and remains unaffected by cane tops (Fig 2.9), probably because the cane tops used were without attached foliage. It can be deduced from Fig 2.9 that increases of 2 and 5 units in fibre % cane mentioned in Chapter 1 are in fact due to the presence of about 3.5% and 8.5% dry trash respectively.

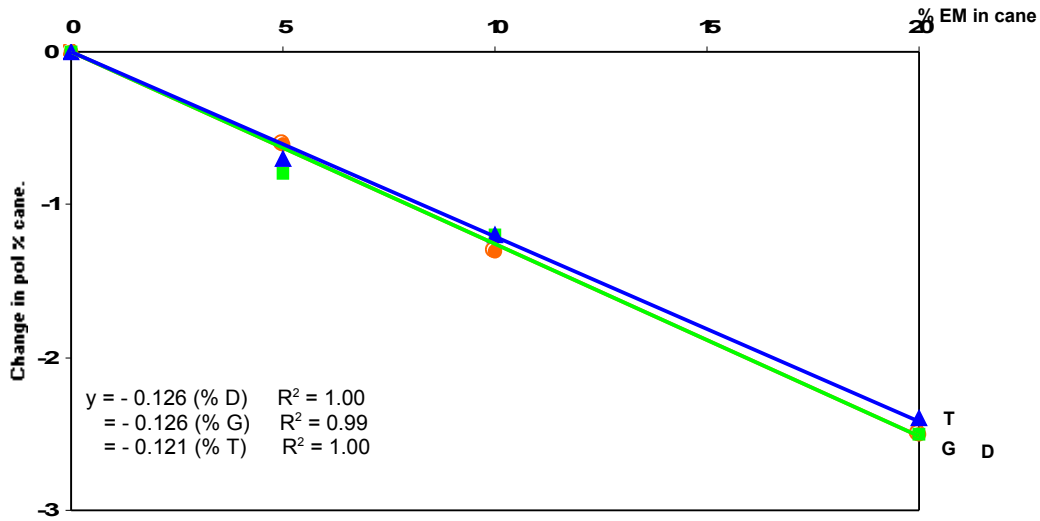


Figure 2.7. Changes in pol % cane due to dry trash (D), green leaves (G) and cane tops (T).

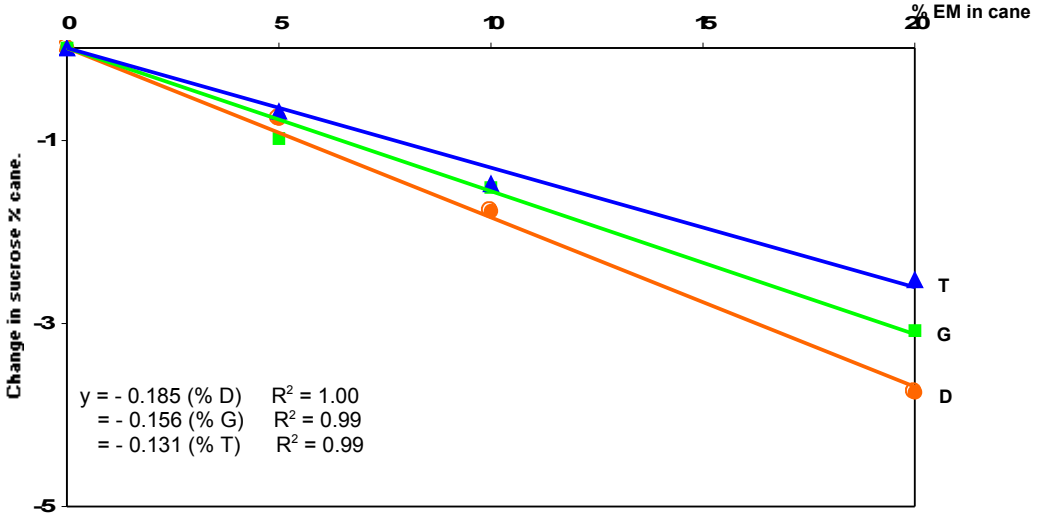


Figure 2.8. Changes in sucrose % cane due to dry trash (D), green leaves (G) and cane tops (T).

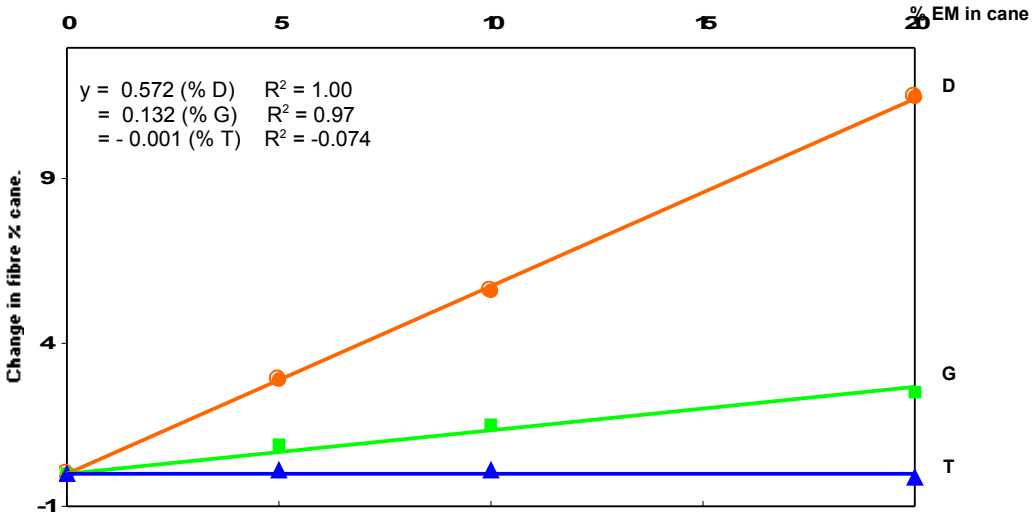


Figure 2.9. Changes in fibre % cane due to dry trash (D), green leaves (G) and cane tops (T).

2.1.6.2 *Bagasse quality*

As expected dry trash significantly increases the mass of bagasse % cane and sucrose lost in bagasse % cane (Figs 2.10 and 2.11), whereas cane tops have no effect, again because they did not have attached foliage. Although cane tops actually decrease the loss of sucrose in bagasse % cane, this should not, however, be taken as an encouragement to send cane tops to the mill as it will be shown that the loss of sucrose in molasses will be significantly increased by cane tops.

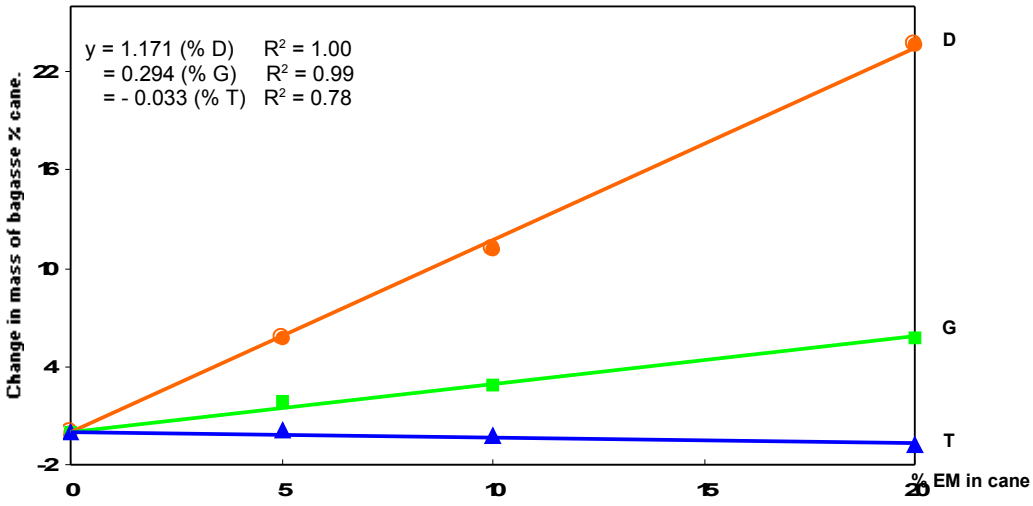


Figure 2.10. Changes in mass of bagasse % cane due to dry trash (D), green leaves (G) and cane tops (T).

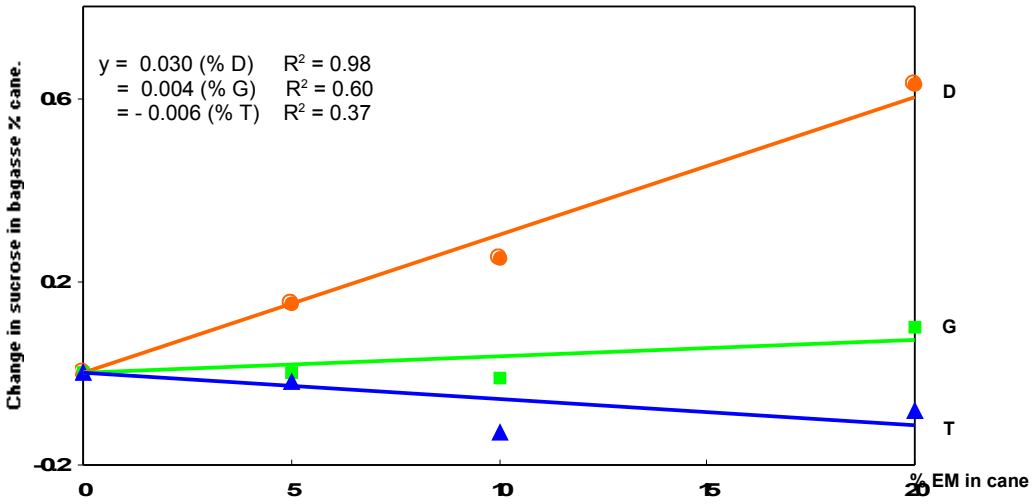


Figure 2.11. Changes in sucrose in bagasse % cane due to dry trash (D), green leaves (G) and cane tops (T).

2.1.6.3 *Mixed juice quality*

The decrease in mixed juice Clerget purity by the different kinds of extraneous matter studied appears to be more or less the same (Fig 2.12). The same applies to the ash content in mixed juice (Fig 2.13). However, the increase in reducing sugars, namely fructose and glucose, in mixed juice is most pronounced in the case of cane tops (Fig 2.14).

As shown in Fig 2.12, each 1% increase in dry trash causes a decrease in mixed juice Clerget purity of 0.26 unit; for green leaves and cane tops, it is 0.25 and 0.29 respectively. This concurs well with the findings of factory trials performed by other workers. For 1% increase in trash, the decrease in mixed juice purity found in South Africa by Reid and Lionnet (1989) was 0.43, and in Australia, 0.30 and 0.27 by Kent *et al.* (1999, 2003).

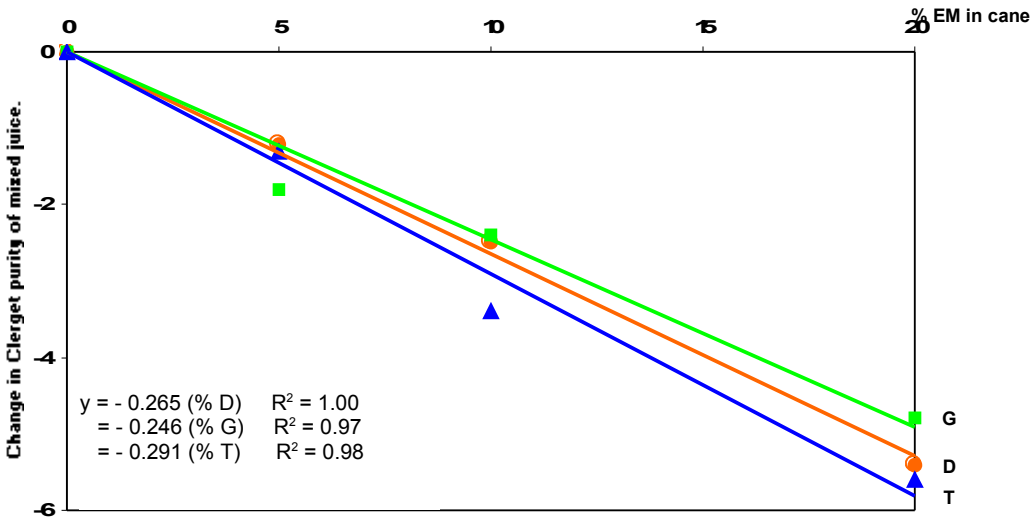


Figure 2.12. Changes in Clerget purity of mixed juice due to dry trash (D), green leaves (G) and cane tops (T).

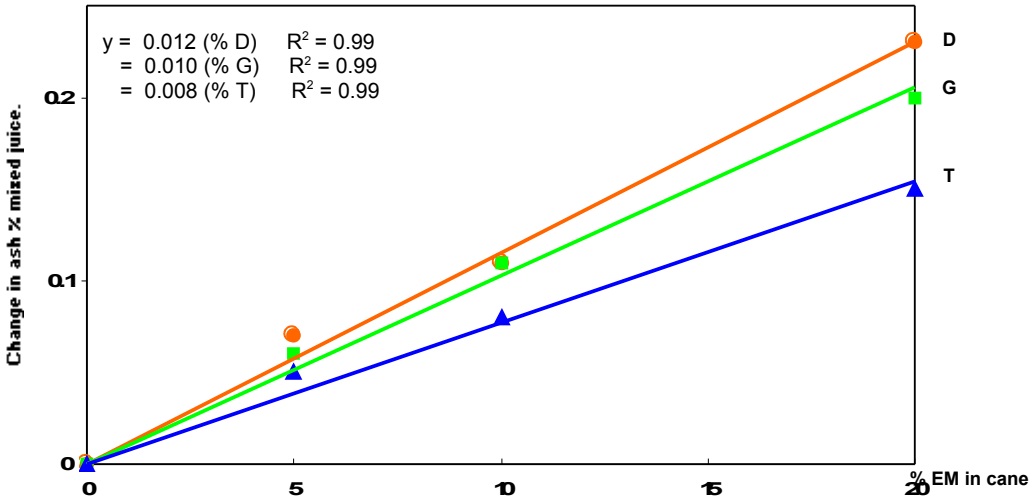


Figure 2.13. Changes in ash % mixed juice due to dry trash (D), green leaves (G) and cane tops (T).

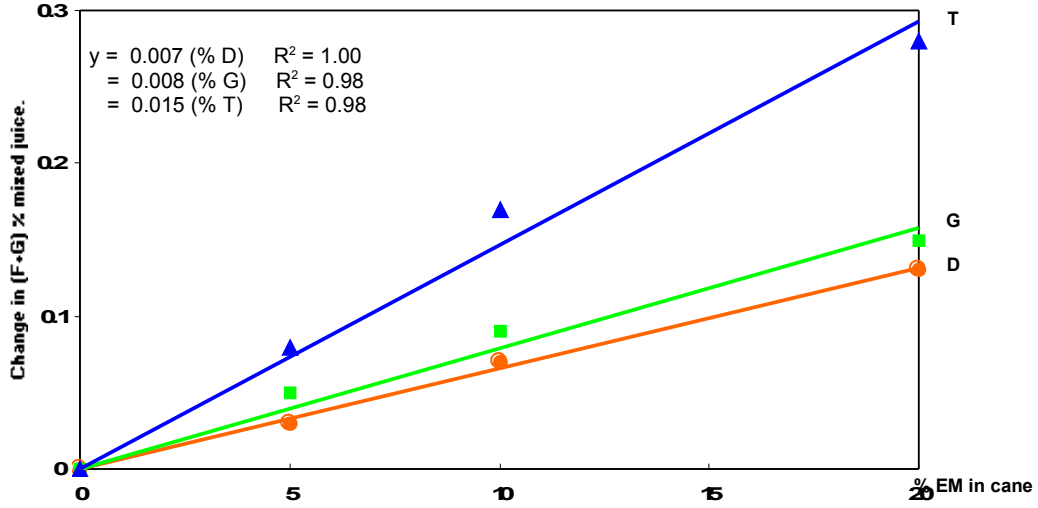


Figure 2.14. Changes in (F+G) % mixed juice due to dry trash (D), green leaves (G) and cane tops (T).

2.1.6.4 *Molasses quality*

Changes in the quality of molasses are most interesting: extraneous matter decreases the Clerget purity of molasses. The effect is most marked with cane tops (Fig 2.15), most probably because of their higher content of reducing sugars. Both cane tops and green leaves exhibit quadratic curve rather than linear relationship as do dry leaves. However, cane tops increase the mass of molasses produced % cane (Fig 2.16) with the result that the loss of sucrose in molasses is high in the case of cane tops (Fig 2.17).

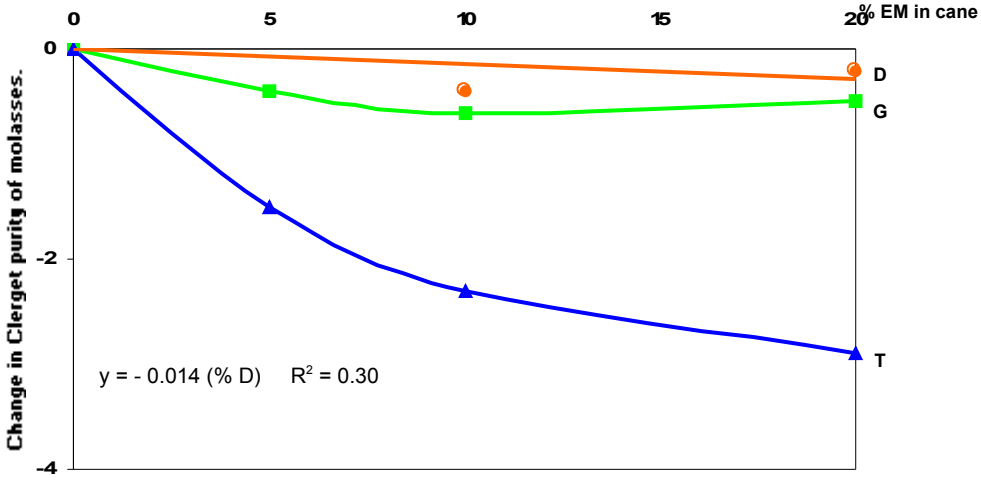


Figure 2.15. Changes in Clerget purity of molasses due to dry trash (D), green leaves (G) and cane tops (T).

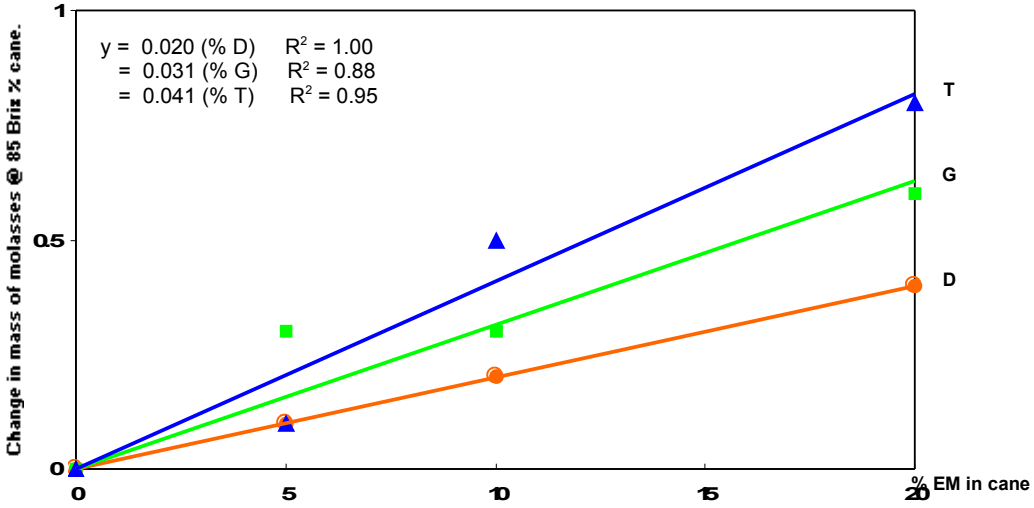


Figure 2.16. Changes in mass of molasses at 85° Brix % cane due to dry trash (D), green leaves (G) and cane tops (T).

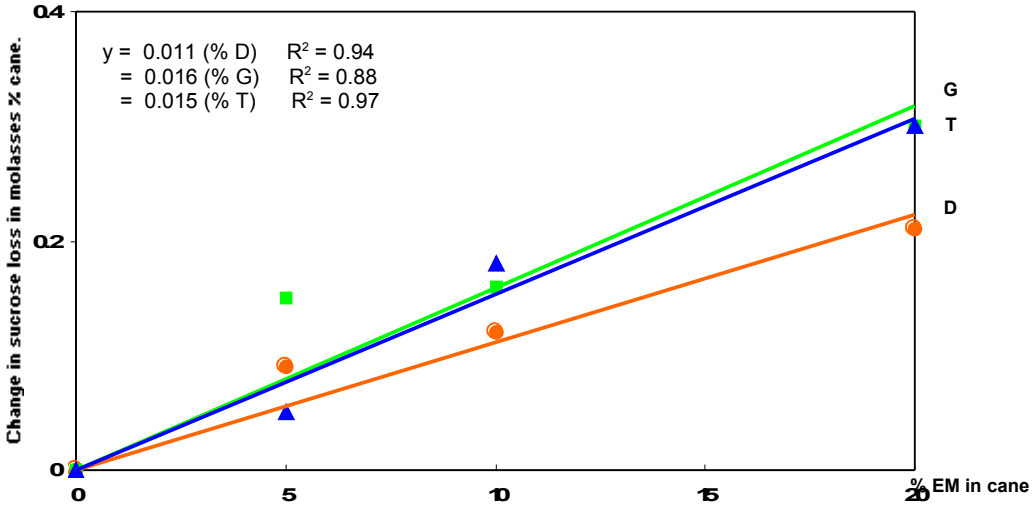


Figure 2.17. Changes in sucrose lost in molasses % cane due to dry trash (D), green leaves (G) and cane tops (T).

2.1.6.5 *Sugar recovery*

Dry trash decreases sucrose extracted in juice % cane, while the effect of green leaves and cane tops are less pronounced (Fig 2.18).

Decrease in the predicted sugar recovery % cane and CCS caused by green leaves and tops appear to be in good agreement, but that caused by dry trash is much more substantial in the case of sugar recovery % cane (Figs 2.19 and 2.20).

The extent of the drop in CCS agrees well with that estimated by Brotherton (1980) who showed that the rate of change of CCS per unit of extraneous matter was -0.16, as compared to -0.16, -0.14 and -0.14 for 1 unit of dry trash, green leaves and cane top respectively shown in Fig 2.20.

The influence of extraneous matter on boiling house recovery and overall recovery are shown in Figs 2.21 and 2.22. The effects of dry trash appear to be most detrimental on these two aspects of milling quality.

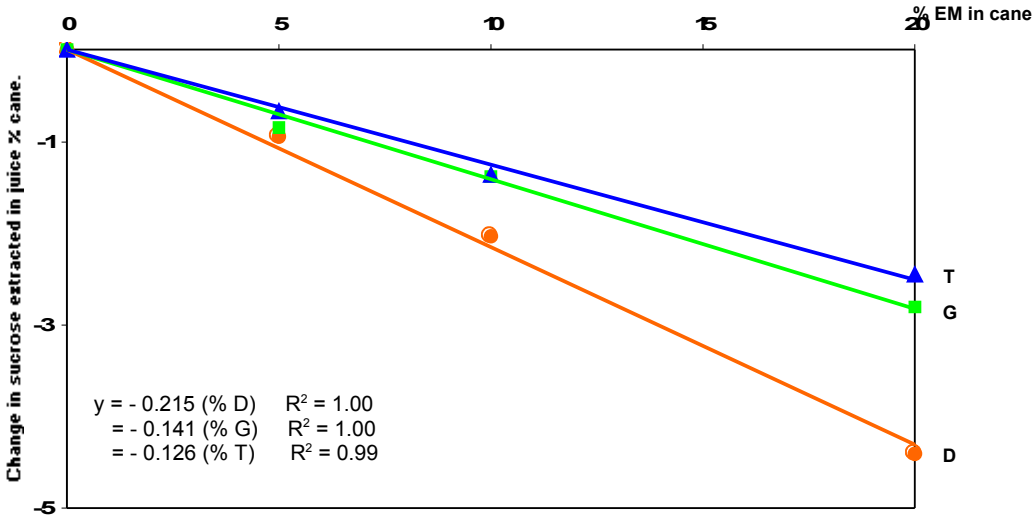


Figure 2.18. Changes in sucrose extracted in juice % cane due to dry trash (D), green leaves (G) and cane tops (T).

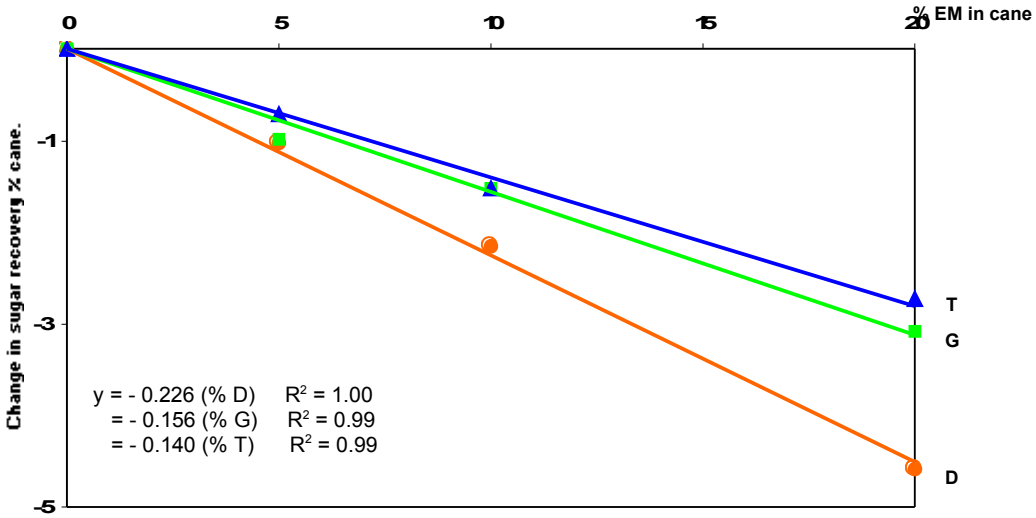


Figure 2.19. Changes in sugar recovery % cane due to dry trash (D), green leaves (G) and cane tops (T).

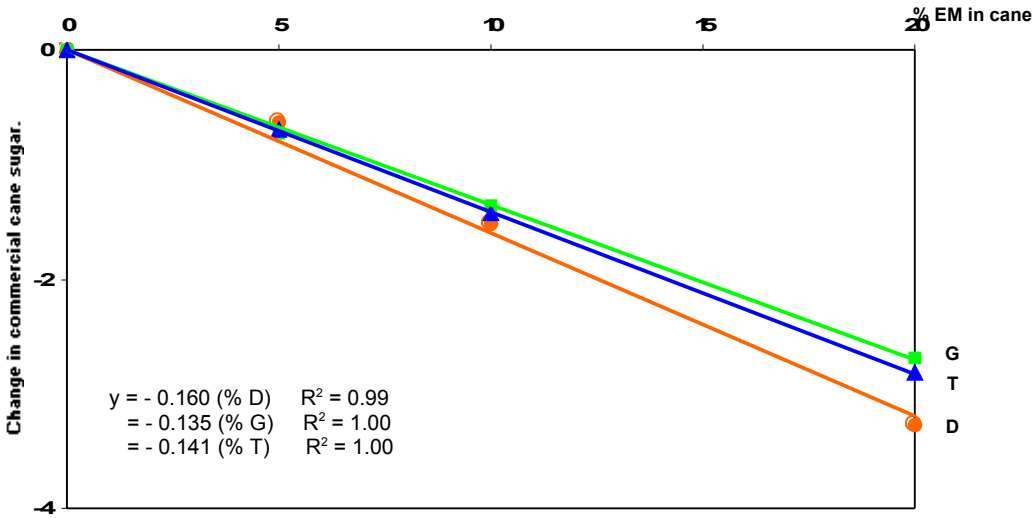


Figure 2.20. Changes in commercial cane sugar due to dry trash (D), green leaves (G) and cane tops (T).

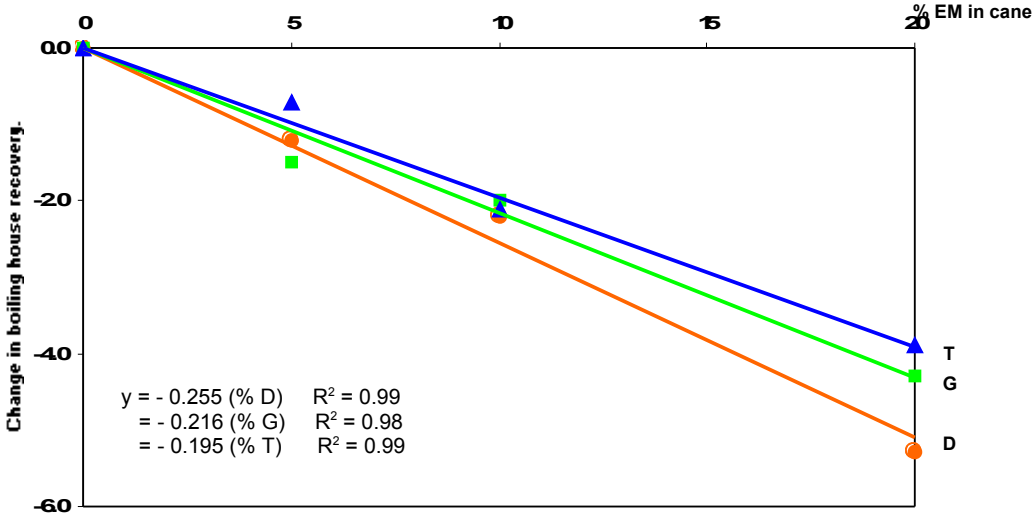


Figure 2.21. Changes in boiling house recovery due to dry trash (D), green leaves (G) and cane tops (T).

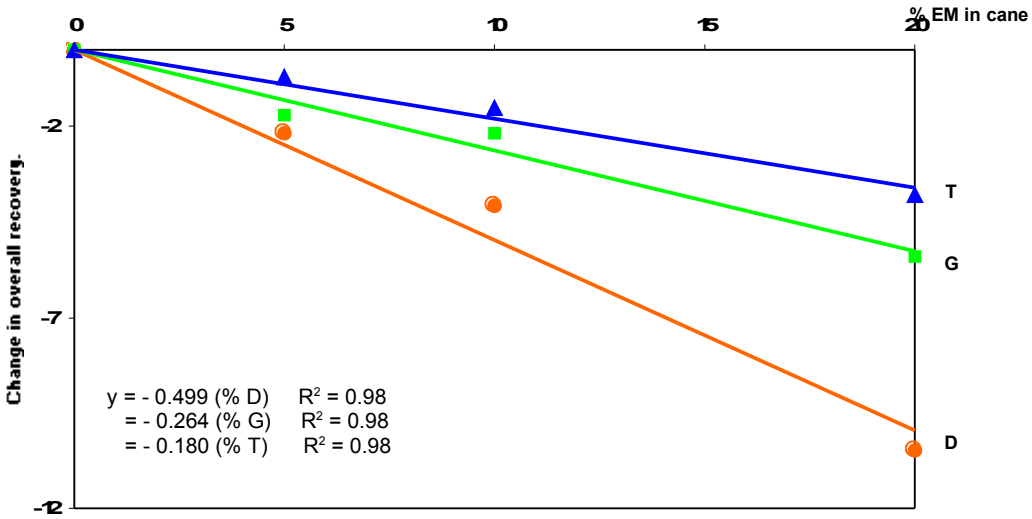


Figure 2.22. Changes in overall recovery due to dry trash (D), green leaves (G) and cane tops (T).

2.1.7 Conclusions

The different effects of various kinds of extraneous matter, notably dry trash, green leaves and cane tops, have been clearly demonstrated in the experiment.

Among the most important findings are:

- Extraneous matter, whatever its nature, serves as a diluent in direct analyses such as pol % cane.
- Dry trash has by far a more adverse effect than green leaves or tops on juice extraction, sugar recovery, boiling house recovery and overall recovery.

  One unit of dry trash increases fibre % cane, mass of bagasse % cane and sucrose loss in bagasse % cane and in molasses % cane by 0.57, 1.17, 0.030 and 0.011 units respectively, and decreases juice extraction and sugar recovery by 0.22 and 0.23 units, respectively.
- The detrimental effect of green leaves is intermediate between those of dry trash and cane tops.
- Cane tops do not seem to affect fibre % cane, the mass of bagasse % cane and juice extraction.

  One unit of cane tops increases the non-sucrose level in mixed juice, which increases the mass of molasses at 85° Brix produced % cane by 0.041 unit,

resulting in 0.015 unit sucrose loss in molasses % cane, a much higher loss than that produced by the same amount of dry trash.

The economic implication of the presence of extraneous matter in cane should be of great concern to both cane growers and millers, as besides a reduction in processing efficiency and in income, the following additional expenses should be considered: cost of harvest and transport of the extraneous matter, the increased cost of maintenance of the factory equipment, investment in new equipment to cope with the trash, soil and rocks, and the cost of lengthening of the crushing season.

2.2 THE PHENOMENON OF BRIX-FREE WATER IN DRY LEAF AND ITS IMPACT ON CANE JUICE QUALITY

On examination of Tables 2.2 – 2.4, it is evident that while green leaves and cane tops invariably lowered the Brix, pol and sucrose content in mixed juice, the effect of adding dry leaves would sometimes raise the analytes (marked in bold in Table 2.2), in particular when their moisture content was below a certain critical value, of about 20% (Table 2.5). This increase in analytes by dry leaf addition can be explained by the phenomenon of Brix-free water in dry leaf.

Similar results have been reported by Hemaida *et al.* (1977) while investigating the effects of EM addition on cane. They found that with up to 7.5% green leaves, no effect on the extraction of both mixed juice and sugar was detected, whereas dry trash (7.5%) markedly increased Brix, sucrose % and glucose % mixed juice by 0.94, 0.37 and 0.80 units respectively. They attributed these increases to the higher rate of absorption of water than of sugar by the dry leaves.

The existence of Brix-free water in leafy trash is rarely reported, while that in cane fibre/bagasse is well known. It is referred to as "Brix-free water" or "hydrated water" in South Africa and "adsorption water" or "hygroscopic water" in Australia. It is defined as the water strongly adsorbed onto the cane fibre and, therefore unavailable for dissolving the soluble components in sugar cane. It cannot be separated from the natural cane fibre by mechanical means, but only by elevated temperatures; and it is assumed to be 25% on dry fibre (Anon., 1984).

Prinsen Geerligs (1904) first determined Brix-free water in fibre by contacting dried washed fibre with a salt solution, allowing it to equilibrate, and then determining the increase in the concentration of the salt solution. Steuerwald (1912) applied sucrose solutions instead of salt solutions, and used two methods to measure Brix-free water: a contact method and a press method.

Experiments were thus conducted to better discern the Brix-free water in dry leaves and its impact on cane juice quality.

This section examines the effects of dry and green leafy trash on juice extraction, predicts sugar recovery from the quality of the cane and first expressed juice and determines the value of Brix-free water in dry cane leaves.

2.2.1 Experimental procedure

Cane materials and extraneous matter such as green leaves, dry leaves and bagasse were obtained from neighbouring factories, and the experiments were carried out in the MSIRI laboratory as described below.

2.2.1.1 *Addition of green leaves, dry leaves and bagasse to cane sample*

In the first trial, which was carried out late in the 2000 season, four whole canes which were cane stalks with attached cane tops were sampled simultaneously with 40 kg cane stalks. The tops of the whole cane (125 cm measured from the apex) were removed, chipped and kept aside, while the four stalks were weighed and chipped together with the 40 kg sample of cane stalks. The four whole canes were then reconstituted by mixing the chipped tops with the recorded mass of the homogenized chipped cane stalks. One representative sample (1329 g) of the chipped whole cane was taken, 329 g of which was analysed for pol % cane and fibre % cane by the STASM method (Anon., 1991). The remaining one kg was subjected to a pressure of 20 MPa (200 bar) for two minutes in a Pinette Emidecau hydraulic press to obtain juice for analysis of Brix, pol and Clerget sucrose by the STASM method (Anon., 1991) and glucose, fructose and sucrose by high performance ion chromatography (HPIC) using the official ICUMSA method (Schaffler, 1994).

After the reconstitution of the whole cane, the remaining chipped cane stalk served as a control sample and material to which EM would be added. The control sample was analysed as above.

Green leaves (2 kg) were shredded in a Jeffco cutter grinder, and added to sub-samples of chipped cane stalks so that green leaves constituted 5, 10 and 20% of the total mass which was 1329 g. The resulting samples were analysed as above.

The experiment was then repeated with shredded dry leaves (2 kg) added to the same chipped cane sample.

The whole experiment was repeated four times.

In Trial II, the effect of the whole cane was not investigated. However, the effect of the moisture content in the dry leaves was investigated. In order to vary the moisture content in the dry leaves, four kg dry leaves were collected, two kg of which were subjected to a

wetting process, in which they were soaked in a bucket of tap water for about twenty minutes, the surface water was then allowed to run off before placing the leaves between sheets of absorbent paper for drying. Shredded original dry leaves, wetted dry leaves and green leaves were then added to chipped cane stalk as in trial I, and analyses carried out. The moisture content of all types of trash samples was also determined by oven-drying 100 g of the sample at 105 °C to constant mass (about 3 hours) and weighing immediately after removal from the oven as per the standard practice, since the leaves absorb atmospheric moisture quickly on cooling.

The whole experiment was repeated four times, as in Trial I.

In Trial III, the moisture content in dry leaves was varied even further. Six kg dry leaves were collected, 2 kg were used as is after being shredded, 2 kg were wetted and 2 kg were oven dried at 105 °C for 3 hours to drive off most of the moisture, before being shredded and used.

In Trial IV, an air-dried bagasse sample was substituted for the dry leaf sample, part of which was wetted and oven dried as the dry leaves in Trial III. Only one set of tests was carried out to check the Brix-free water effect of bagasse.

2.2.1.2 *Determination of Brix-free water in dry leaf*

An analytical method similar to that used by Mangion and Player (1991) to determine the Brix-free water in various parts of cane was adopted. Ten dry leaves each, of the four main cane varieties cultivated in Mauritius (M 695/69, M 1658/78, M 3035/66 and R 570) were collected, cut into strips 1 cm wide, washed repeatedly in running cold water until the solution gave no Brix reading. The four samples were then separately disintegrated in 1 L of water in a wet disintegrator at 8000 rpm for 10 minutes, sieved and air-dried.

About 8 g each of the four samples were placed in pre-weighed 250 mL glass bottles and dried in a vacuum oven at 80 °C overnight. After drying, the bottles were stoppered and cooled in a desiccator before weighing to determine the mass of the samples. A 10° Brix sucrose solution was prepared and mercuric iodide juice preservative solution added at the rate of 0.5 mL/L. To each trash sample were then added 150 mL of the 10° Brix sucrose solution, the mass of which was accurately determined. The contact time between the trash sample and the sucrose solution was one-and-a-half hours, during which period the bottles were shaken from time to time. The solutions were then filtered through a Whatman 91

filter paper, rejecting the first few mL of the filtrate. The Brix of the filtrate together with the original 10° Brix sucrose solution were determined in triplicate.

The Brix-free water in the dry leaf was then calculated from the following equation (Mangion and Player, 1991):

$$\text{Brix - free water in dry leaf} = 100\left[\frac{\text{Mass of sucrose solution}}{\text{Mass of dry leaf sample}}\right]\left[1 - \frac{\text{Brix before contacting}}{\text{Brix after contacting}}\right]$$

2.2.2 Results and discussion

The average results for Trials I - III, with green and dry trash are shown in Tables 2.9 – 2.11, and the results of Trial IV with bagasse are presented in Table 2.12. Values of CCS were computed by using the Australian method of calculation (Anon., 1984) described in Section 2.1.4.

The effect on press juice and cane qualities due to the additions of green leaves, dry leaves and bagasse was assessed from the data presented in Tables 2.9 – 2.12, and the average values are compiled in Table 2.13.

The increase of non-sucrose in press juice, taken as Brix minus Clerget sucrose, due to the addition of EM was also calculated, and the percentage increase or decrease presented in Table 2.13.

2.2.2.1 *Glucose/fructose ratio in green and dry leaves*

The green leaves used in Trial I, in contrast to those that were used in Trials II – IV, contained very little glucose but had a relatively high fructose concentration as shown by the constant amount of glucose with increased addition of green leaves while the fructose content increased significantly. The low glucose in these green leaves indicates leuconostoc growth and formation of dextran, which is a polymer of glucose. This explains the decrease in G/F ratio, from 0.96 to 0.53, with the addition of the first 5% green leaves, and to 0.34% with a further 5% addition, representing decreases of 0.43 and 0.19 units respectively. This concurs with Legendre's finding (1991) that green (but not dry) leaves reduced the G/F ratio. He also reported that a G/F ratio of < 1.6, might give rise to false low pol. The aforesaid, however, does not apply to the green leaves used in Trials II – IV in which both glucose and fructose contents rose with increasing additions of green leaves, with the result that the G/F ratio remained more or less constant at 0.87 and

0.95 respectively (Tables 2.10 and 2.12). It even increased (Table 2.11) with increasing green leaves addition. The only difference among the green leaves was that those used for Trial I had been treated with cane ripener, with their addition to cane stalk giving rise to a false pol situation.

2.2.2.2 *Changes in sucrose, Brix and pol in press juice*

Table 2.13 shows that while the addition of dry trash and air-dry bagasse increased the sucrose (Clerget as well as HPIC), Brix and pol in press juice, green leaves, wetted dry trash and wetted air-dry bagasse (except HPIC sucrose) did the opposite. This increase was even higher when some of the moisture present in the dry trash and in air-dried bagasse was removed. Increases in non-sucrose produced by the addition of EM amounted to 7.4, 14 and 36% for 5, 10 and 20% green leaves, and are similar to the corresponding increases due to dry leaves which were 12, 18 and 38%. This is in disagreement with observations of Arceneaux and Davidson (1944) who found that green leaves, contrary to dry trash, had a marked influence on the impurities of the juice. The corresponding changes in non-sucrose due to the addition of air-dried bagasse were –0.2, 0.7 and 1.0%, much less than the effect produced by the addition of green and dry leaves.

2.2.2.3 *Changes in pol and fibre % cane*

Addition of all types of EM (green leaves, dry leaves, wetted and extra dry, and bagasse, wetted and extra dry) produced decreases in pol % cane by about 5, 9 and 18% at the level of 5, 10 and 20% EM (Table 2.13). The effect of EM on fibre % cane however, was dependent mainly on the moisture content of the EM added.

2.2.2.4 *Losses in sugar recovery*

The loss in CCS due to the presence of 5, 10 and 20% green leaves was 4.4, 9.6 and 18% respectively (Table 2.13), these being the same as for wetted dry leaves and air-dried bagasse. The losses in sugar recovered due to dry leaves were slightly lower at high concentrations, i.e. 4.3, 6.5 and 14%, while those due to air-dried bagasse were 4.5, 8.4 and 17% at 5, 10 and 20% trash levels, respectively.

Table 2.9. Effect of increased addition of EM on press juice and cane quality (Trial I).

			PRESS JUICE								CANE		
Level of EM added to stalk (%)	Type of EM	Moisture % EM	Volume extracted (mL)	HPIC glucose (%)	HPIC fructose (%)	G/F	HPIC sucrose (%)	Clerget sucrose (%)	Brix (%)	Pol (%)	Pol (%)	Fibre (%)	CCS
Whole cane	-		631	0.225	0.228	0.99	17.79	17.69	19.60	17.62	13.08	16.22	12.91
Control (0)	-		686	0.070	0.073	0.96	19.21	19.16	20.62	19.32	15.79	14.02	14.91
5	Green	N.A.	677	0.080	0.149	0.53	18.78	18.73	20.30	18.68	14.96	15.02	14.09
10	Green		663	0.080	0.244	0.34	18.21	18.31	20.01	17.78	13.91	15.81	13.00
20	Green		629	0.087	0.407	0.26	17.18	17.06	19.34	16.46	12.41	17.00	11.52
5	Dry	N.A.	645	0.100	0.117	0.85	**19.62**	**19.50**	**21.09**	**19.63**	14.74	18.18	14.31
10	Dry		578	0.127	0.135	0.94	**20.02**	**19.95**	**21.59**	**20.00**	14.63	19.94	14.20
20	Dry		489	0.181	0.191	0.95	**20.36**	**20.38**	**22.28**	**20.49**	13.65	26.28	13.24

Note: All data are averages of four results.

N.A.: not analysed.

Table 2.10. Effect of increased addition of EM on press juice and cane quality (Trial II).

			PRESS JUICE								CANE		
Level of EM added to stalk (%)	Type of EM	Moisture % EM	Volume extracted (mL)	HPIC glucose (%)	HPIC fructose (%)	G/F	HPIC sucrose (%)	Clerget sucrose (%)	Brix (%)	Pol (%)	Pol (%)	Fibre (%)	CCS
Control (0)	-		707	0.035	0.042	0.83	14.44	14.46	15.92	14.38	12.06	10.99	11.27
5	Green	70.6	707	0.055	0.063	0.87	14.10	14.12	15.75	14.08	11.52	11.41	10.91
10	Green		697	0.069	0.081	0.85	13.78	13.75	15.40	13.59	11.01	12.33	10.33
20	Green		687	0.108	0.124	0.87	12.86	12.82	14.72	12.67	9.67	13.30	9.37
5	Dry	29.8	687	0.065	0.072	0.90	14.25	14.29	**15.93**	14.22	11.45	13.88	10.68
10	Dry		665	0.091	0.101	0.91	14.36	14.32	**16.01**	14.24	11.25	15.63	10.44
20	Dry		559	0.125	0.138	0.91	14.39	14.35	**16.32**	14.29	10.11	21.50	9.59
5	Dry (wetted)	59.4	701	0.066	0.072	0.92	14.09	14.15	15.74	14.07	11.44	12.24	10.80
10	Dry (wetted)		685	0.085	0.092	0.92	13.69	13.76	15.45	13.73	10.86	14.01	10.27
20	Dry (wetted)		652	0.120	0.131	0.92	13.33	13.20	14.89	13.07	9.80	16.83	9.36

Note: All data are averages of four results.

Table 2.11. Effect of increased addition of EM on press juice and cane quality (Trial III).

			PRESS JUICE								CANE		
Level of EM added to stalk (%)	Type of EM	Moisture % EM	Volume extracted (mL)	HPIC glucose (%)	HPIC fructose (%)	G/F	HPIC sucrose (%)	Clerget sucrose (%)	Brix (%)	Pol (%)	Pol (%)	Fibre (%)	CCS
Control (0)			741	0.083	0.089	0.93	20.25	20.07	21.61	20.18	16.94	11.31	16.07
5	Green	72.2	723	0.106	0.111	0.95	19.65	19.54	21.15	19.61	16.25	11.93	15.44
10	Green		711	0.130	0.128	1.02	18.95	18.81	20.58	18.89	15.53	12.37	14.70
20	Green		685	0.172	0.166	1.04	17.56	17.39	19.39	17.44	13.68	12.92	13.32
5	Dry	15.1	693	0.135	0.137	0.99	20.23	**20.08**	**21.84**	20.18	15.90	13.96	15.46
10	Dry		645	0.155	0.157	0.99	**20.43**	**20.25**	**22.17**	**20.37**	14.90	17.34	14.90
20	Dry		563	0.258	0.260	0.99	**20.65**	**20.49**	**22.77**	**20.57**	13.60	23.64	13.67
5	Dry (wetted)	65.4	727	0.111	0.116	0.96	19.44	19.33	20.97	19.44	16.01	12.60	15.18
10	Dry (wetted)		716	0.136	0.134	1.01	18.89	18.73	20.41	18.79	15.16	13.25	14.49
20	Dry (wetted)		693	0.174	0.175	0.99	17.43	17.19	19.02	17.27	13.63	14.90	12.94
5	Dry (oven-dried)	7.1	689	0.130	0.132	0.98	**20.46**	**20.29**	**22.09**	**20.40**	16.16	14.48	15.52
10	Dry (oven-dried)		636	0.157	0.152	1.04	**20.74**	**20.54**	**22.46**	**20.64**	15.40	17.86	15.00
20	Dry (oven-dried)		547	0.194	0.189	1.03	**21.11**	**20.98**	**23.25**	**21.05**	14.03	25.17	13.70

Note: All data are averages of four results.

Table 2.12. Effect of increased addition of EM and bagasse on press juice and cane quality (Trial IV).

			PRESS JUICE								CANE		
Level added to stalk (%)	Type of additive	Moisture % additive	Volume extracted (mL)	HPIC glucose (%)	HPIC fructose (%)	G/F	HPIC sucrose (%)	Clerget sucrose (%)	Brix (%)	Pol (%)	Pol (%)	Fibre (%)	CCS
Control (0)			745	0.043	0.049	0.88	20.24	19.90	21.62	20.30	17.30	11.03	16.28
5	Green leaf	70.2	743	0.084	0.088	0.95	19.42	19.25	21.07	19.57	16.50	11.61	15.48
10	Green leaf		724	0.106	0.112	0.95	19.02	18.79	20.72	19.01	15.70	12.16	14.83
20	Green leaf		709	0.144	0.150	0.96	17.88	17.55	19.73	17.76	14.50	12.83	13.59
5	Dry bagasse	22.2	743	0.070	0.074	0.95	**20.30**	19.88	**21.70**	20.25	16.00	14.26	15.55
10	Dry bagasse		734	0.082	0.088	0.93	**20.48**	**20.13**	**21.88**	**20.45**	15.75	18.27	14.92
20	Dry bagasse		732	0.104	0.114	0.91	**20.56**	**20.20**	**22.02**	**20.50**	14.00	25.59	13.48
5	Dry bagasse (wetted)	80.0	700	0.056	0.060	0.93	19.24	18.94	20.60	19.30	15.90	12.34	15.21
10	Dry bagasse (wetted)		644	0.050	0.056	0.89	18.14	17.84	19.40	18.17	15.80	13.10	14.18
20	Dry bagasse (wetted)		528	0.054	0.060	0.90	16.66	16.90	17.61	16.50	14.10	13.83	12.77
5	Dry bagasse (oven-dried)	10.7	684	0.082	0.086	0.95	**20.74**	**20.35**	**22.18**	**20.69**	16.20	14.89	15.76
10	Dry bagasse (oven-dried)		654	0.080	0.082	0.98	**20.84**	**20.40**	**22.26**	**20.78**	16.15	17.66	15.28
20	Dry bagasse (oven-dried)		527	0.076	0.078	0.97	**21.16**	**20.72**	**22.70**	**21.17**	13.75	27.23	13.60

Table 2.13. Effect of increased addition of various types of EM on press juice and cane quality.

Level of EM added to stalk (%)		Average % increase/decrease in parameters relative to control										
		PRESS JUICE								CANE		CCS
		Volume extracted	HPIC glucose	HPIC fructose	HPIC sucrose	Clerget sucrose	Brix	pol	Non sucrose	Pol (%)	Fibre (%)	
Top + stalk to reconstitute whole cane		-8.0	221	212	-7.4	-7.7	-4.9	-8.8	31	-17	16	-13
5%	green leaf	-1.0	49	65	-2.9	-2.6	-1.8	-3.0	7.4	-4.6	5.4	-4.4
10%		-2.9	79	125	-5.6	-5.6	-3.8	-6.6	14	-9.5	11	-9.6
20%		-5.9	144	236	-12	-12	-8.2	-13	36	-19	18	-18
5%	dry trash	-5.1	64	62	0.3	0.2	1.2	0.2	12	-5.9	25	-4.3
10%		-11	109	101	1.5	1.3	3.0	0.6	18	-8.7	46	-6.5
20%		-25	209	194	2.6	2.6	5.3	2.5	38	-17	97	-14
5%	dry trash (wetted)	-1.3	61	51	-3.2	-2.9	-2.1	-3.0	7.7	-5.3	11	-4.9
10%		-3.3	103	85	-6.0	-5.8	-4.3	-5.6	13	-10	22	-9.4
20%		-7.2	176	154	-11	-12	-9.3	-12	17	-19	42	-18
5%	dry trash (oven-dried)	-6.9	57	48	1.0	1.1	2.2	1.1	17	-4.6	28	-3.4
10%		-14	89	71	2.4	2.3	3.9	2.3	25	-9.1	58	-6.7
20%		-26	134	112	4.2	4.5	7.6	4.3	47	-17	123	-15
5%	air-dry bagasse	-0.3	63	51	8.0	0.3	-0.1	0.4	-0.2	-7.5	29	-4.5
10%		-1.5	91	80	5.7	1.2	1.2	1.2	0.7	-9.0	66	-8.4
20%		-1.7	142	133	3.4	1.6	1.5	1.9	1.0	-19	132	-17
5%	air-dry bagasse (wetted)	-6.0	30	22	5.7	-4.9	-4.8	-4.7	-4.9	-8.1	12	-6.6
10%		-14	16	14	1.1	-10	-10	-10	-11	-8.7	19	-13
20%		-29	26	22	2.3	-18	-15	-19	-19	-19	25	-22
5%	air-dry bagasse (oven-dried)	-8.2	91	76	8.0	2.5	2.3	2.6	1.9	-6.4	35	-3.2
10%		-12	86	67	11.0	3.0	2.5	3.0	2.4	-6.6	60	-6.1
20%		-29	77	59	10.0	4.5	4.1	5.0	4.3	-21	147	-17

2.2.2.5 *Estimation of EM in cane using sucrose ratio in dirty cane relative to clean cane*

Larrahondo *et al.* (1998) derived an equation to predict the trash level in cane by assuming that sucrose in EM was negligible.

$$\text{Thus, \% EM in cane} = \left(1 - \frac{\text{sucrose in dirty cane}}{\text{sucrose in clean cane}}\right)100$$

If pol instead of sucrose % cane data from Tables 2.9 – 2.12 are substituted in the above equation, calculated EM values can be obtained as shown in Table 2.14. When they were compared with known added EM contents, the equation appeared to apply well to green and wetted dry leaves, but at high levels of dry and extra dry leaves, the theoretical values tended to be underestimated.

2.2.2.6 *Brix-free water determination in dry leaves*

The results of the Brix-free water determination by Mangion and Player's (1991) method in dry leaves of the four main cane varieties cultivated in Mauritius are summarised in Table 2.15. Only M 695/69 had a low Brix-free water of 25.6% while the other three varieties averaged 28.3% Brix-free water. This is in agreement with the data obtained in Trial II (Table 2.10), carried out during the rainy season, when the trash was not quite dry, and averaged a moisture content of 29.8%. In this trial, the phenomenon of Brix-free water was not observed to be operating, as evidenced by the lower Clerget sucrose % press juice (14.29%) due to the addition of 5% dry trash than that in the control sample (14.46%). The same applies to the 10% and 20% addition of dry trash. This is also true for the values of pol % press juice. The indication would be that the Brix-free water of the dry leaves in this case was below 29.8%.

This method of determining Brix-free water, gave reproducible results for dry leaves, but when tried on bagasse samples, the results were not reproducible. According to Qin and White's (1991) finding, there are significant differences in the Brix-free water values of rind, stalk fibre and stalk pith fractions in bagasse sample, which explains why the Brix-free water value of bagasse is variable. This is probably because in each sample of bagasse tested there were varying proportions of fibre, pith, rind and trash, each with their own Brix-free water contents. It would therefore be of interest to separate sugar cane into its

various components such as stalk fibre, pith, rind fibre, top fibre, dry leaf fibre and green leaf fibre, and determine their Brix-free water content.

Table 2.14. Comparison of actual and calculated EM % cane as obtained from the formula of Larrahondo *et al*. (1998).

Extraneous matter actually added	Theoretical EM % cane (t) as calculated from $t = \left(1 - \frac{\text{pol \% dirty cane}}{\text{pol \% clean cane}}\right) \times 100$ using experimental data from Tables 2.9 - 2.12				
	Trial I	Trial II	Trial III	Trial IV	Mean
5% green leaf	5.3	4.5	4.1	4.6	4.6
10%	11.9	8.7	8.3	9.2	9.5
20%	21.4	19.8	19.2	16.2	19.2
5% dry leaf	6.6	5.1	6.1	-	5.9
10%	7.3	6.7	12.0	-	8.7
20%	13.6	16.2	19.7	-	16.5
5% dry leaf (wetted)	-	5.1	5.5	-	5.3
10%	-	10.0	10.5	-	10.3
20%	-	18.7	19.5	-	19.1
5% dry leaf (oven-dried)	-	-	4.6	-	4.6
10%	-	-	9.1	-	9.1
20%	-	-	17.2	-	17.2

Table 2.15. Brix-free water content of dry leaf from the four main cane varieties cultivated in Mauritius.

Cane variety	Brix-free water of dry leaf/% dry fibre		Average
M 695/69	25.0	26.3	25.6
M 3035/66	28.9	28.0	28.5
R 570	28.2	29.2	28.7
M 1658/78	27.9	27.8	27.8

2.2.3 Survey of moisture content in dry trash

The cane payment system in Mauritius has been described in details by Anon. (1991). Essentially, cane is purchased on its quality, assessed by analysing its press juice. Therefore, if the cane is supplied with dry trash which has low moisture content below its Brix-free water content, the press juice analysed will have inflated results, which will certainly have an impact on the payment of cane.

A survey was carried out in 2003 in four sugar factories to determine the moisture content in dry trash received with the cane supply. About 50 g of the dry trash were put in a

weighed bag made of mosquito net, and measuring about 30 cm x 15 cm. The bag could be tied closed with a draw string. After weighing to determine the initial sample mass, drying was effected in a thermostatic oven at 105 °C to constant mass, after which, the dried sample was weighed while still hot to calculate the moisture content of the trash. The results obtained are shown in Tables 2.16 – 2.19. Most of the dry trash samples had a low moisture content below the typical Brix-free water value of 28.3% found in the main Mauritian cane varieties. Tables 2.16 – 2.19 show that 93, 94, 85 and 100% of the 192, 98, 60 and 22 samples respectively contain less than 28% moisture.

How the Brix-free water in dry trash affects the cane payment system could be the subject of a future study.

2.2.4 Conclusions

The effect of processing clean cane stalks together with their attached tops in the reconstituted whole cane experiment reduced the volume of juice extracted, pol % cane and sugar recovered, and increased non-sucrose in press juice and fibre % cane. This is in agreement with the results obtained in Section 2.1.6 except in the case of fibre % cane (see Fig 2.9), where there was practically no change when measured quantities of cane tops were added to clean cane, whereas with whole cane, there was an increase of 16% in fibre % cane. The difference is probably due to the different amount of foliage attached to the cane tops. The effect produced by whole cane is roughly the same extent as produced by the presence of 15% green leaves on cane.

Dry leaves absorb more juice than green leaves during cane crushing. The effect of dry leaves on non-sucrose contents in the juice extracted was of the same order of magnitude as that produced by green leaves. The overall effect of wet dry leaves on the quality of both press juice and cane approximated that of green leaves.

Dry leaves have a much higher juice absorbing power than green leaves. When the moisture content of dry leaves is below a certain critical Brix-free water value, determined by Mangion and Player's (1991) method to be 28.3% for typical Mauritian cane varieties, the press juice obtained in contact with dry leaves had increased the concentrations of sucrose, Brix and pol (marked bold in Tables 2.2, 2.9 – 2.12), as the trash absorbs water in preference to juice during pressing to satisfy its Brix-free water capacity. This implies that

in the presence of dry leaves, if cane quality is assessed by the above parameters, the results will be over-estimated.

During the period under investigation, a survey carried out during three weeks at one of the factories where the cane samples were collected showed 15.1% trash in cane and 18.4 % fibre in cane. In the pre-mechanisation period of the 1970s when the cane was relatively clean, fibre % cane at this factory was found to be 14.0% (Wong Sak Hoi and Autrey, 1998). The increase in fibre % cane is therefore 31.4%, corresponding to 15% increase in wet trash. According to Table 2.13, the loss in sugar recovery alone at this trash level amounts to 13.5% if the mill extraction parameters are kept unchanged.

Since the existence of Brix-free water in dry leaf has been proven, and its impact on cane juice quality shown; it would be of interest to develop methods to separate the sugar cane plant into fibres of its various components, and to determine their Brix-free water content.

The work undertaken to achieve this will be described in Chapters 3 and 4.

Table 2.16. Moisture content in sugar cane dry leaves at Belle Vue sugar factory.

Date	% moisture	Date	% moisture	Date	% moisture	Date	% moisture
9.09.03	41.7	1.10.03	15.2	17.10.03	11.5	6.11.03	8.3
9.09.03	44.1	1.10.03	10.2	17.10.03	7.2	6.11.03	8.6
9.09.03	26.0	2.10.03	22.0	18.10.03	22.4	12.11.03	13.7
9.09.03	22.4	2.10.03	22.1	18.10.03	15.6	12.11.03	10.5
10.09.03	29.1	2.10.03	11.3	20.10.03	17.5	12.11.03	8.7
10.09.03	20.0	2.10.03	18.2	20.10.03	16.0	12.11.03	10.3
10.09.03	12.3	3.10.03	24.5	20.10.03	12.7	13.11.03	9.0
10.09.03	9.5	3.10.03	19.9	20.10.03	6.6	13.11.03	18.5
11.09.03	22.8	3.10.03	12.3	21.10.03	25.8	13.11.03	14.7
11.09.03	26.8	3.10.03	19.6	21.10.03	28.9	13.11.03	11.2
11.09.03	11.5	6.10.03	21.3	21.10.03	12.5	14.11.03	12.3
11.09.03	10.3	6.10.03	19.7	21.10.03	7.4	14.11.03	11.6
12.09.03	19.7	6.10.03	18.2	22.10.03	11.9	14.11.03	9.1
12.09.03	14.1	6.10.03	10.7	22.10.03	15.9	14.11.03	9.0
12.09.03	12.2	7.10.03	14.8	22.10.03	6.7	17.11.03	19.3
12.09.03	11.7	7.10.03	15.1	22.10.03	7.3	17.11.03	16.5
13.09.03	26.6	7.10.03	19.3	23.10.03	22.8	17.11.03	23.0
13.09.03	22.3	7.10.03	20.3	23.10.03	20.1	17.11.03	24.5
15.09.03	24.0	8.10.03	28.0	23.10.03	9.4	18.11.03	11.4
15.09.03	26.7	8.10.03	30.2	23.10.03	14.1	18.11.03	12.1
15.09.03	11.5	8.10.03	14.6	27.10.03	9.4	18.11.03	10.1
15.09.03	12.5	8.10.03	9.5	27.10.03	15.5	18.11.03	11.0
16.09.03	16.9	9.10.03	15.8	27.10.03	24.6	19.11.03	16.8
16.09.03	17.7	9.10.03	15.5	27.10.03	13.5	19.11.03	15.7
22.09.03	28.5	9.10.03	15.0	28.10.03	17.2	19.11.03	12.7
22.09.03	29.8	9.10.03	23.9	28.10.03	18.1	19.11.03	6.5
22.09.03	32.7	10.10.03	16.7	28.10.03	9.8	20.11.03	13.6
22.09.03	37.8	10.10.03	13.1	28.10.03	7.7	20.11.03	23.4
23.09.03	30.1	10.10.03	24.5	29.10.03	23.1	21.11.03	11.8
23.09.03	28.7	10.10.03	9.0	29.10.03	23.9	21.11.03	15.6
23.09.03	15.4	13.10.03	17.4	29.10.03	13.0	21.11.03	13.5
23.09.03	15.2	13.10.03	23.7	29.10.03	19.3	21.11.03	12.5
23.09.03	15.9	13.10.03	12.0	30.10.03	11.7	24.11.03	33.1
23.09.03	19.8	13.10.03	20.5	30.10.03	12.7	24.11.03	34.2
26.09.03	18.1	14.10.03	15.1	30.10.03	9.6	24.11.03	29.9
26.09.03	23.9	14.10.03	19.6	30.10.03	8.1	24.11.03	16.3
26.09.03	9.4	14.10.03	11.5	31.10.03	12.6	25.11.03	16.6
26.09.03	10.8	14.10.03	10.9	31.10.03	16.9	25.11.03	15.2
29.09.03	17.3	15.10.03	14.6	3.11.03	21.8	25.11.03	11.8
29.09.03	14.8	15.10.03	14.6	3.11.03	14.5	25.11.03	14.9
29.09.03	14.4	15.10.03	9.3	3.11.03	7.9	27.11.03	5.2
29.09.03	10.8	15.10.03	11.9	3.11.03	8.2	27.11.03	6.1
30.09.03	12.0	16.10.03	22.0	5.11.03	11.6	27.11.03	12.8
30.09.03	11.5	16.10.03	18.0	5.11.03	22.4	27.11.03	6.5
30.09.03	10.3	16.10.03	6.3	5.11.03	9.1	28.11.03	12.9
30.09.03	9.5	16.10.03	10.2	5.11.03	14.8	28.11.03	13.4
1.10.03	15.1	17.10.03	19.3	6.11.03	8.5	28.11.03	7.4
1.10.03	18.9	1.10.03	16.3	6.11.03	8.1	28.11.03	8.9

Mean of 192 determinations = 16.16
Standard deviation of the mean = 7.06

Table 2.17. Moisture content in sugar cane dry leaves at Beau Champ sugar factory.

Date	% moisture	Date	% moisture	Date	% moisture	Date	% moisture
9.08.03	31.0	4.09.03	14.2	3.10.03	13.6	28.10.03	14.8
11.08.03	13.2	5.09.03	24.2	3.10.03	12.0	29.10.03	16.0
11.08.03	25.0	8.09.03	27.2	6.10.03	12.8	29.10.03	14.6
12.08.03	12.8	8.09.03	48.2	6.10.03	9.2	30.10.03	13.6
12.08.03	14.4	9.09.03	28.8	7.10.03	12.0	30.10.03	20.0
13.08.03	15.8	10.09.03	14.6	7.10.03	13.2	31.10.03	14.8
13.08.03	15.8	13.09.03	15.2	8.10.03	11.2	31.10.03	12.6
14.08.03	17.6	15.09.03	13.8	8.10.03	10.0	3.11.03	11.6
14.08.03	15.0	15.09.03	12.2	9.10.03	10.6	3.11.03	11.2
16.08.03	20.8	17.09.03	19.4	10.10.03	14.4	4.11.03	11.6
20.08.03	16.6	18.09.03	21.8	13.10.03	14.0	4.11.03	13.0
21.08.03	13.8	18.09.03	10.8	14.10.03	11.2	5.11.03	13.6
22.08.03	15.0	19.09.03	20.2	15.10.03	12.2	5.11.03	13.2
22.08.03	12.0	22.09.03	12.8	15.10.03	14.4	6.11.03	15.4
25.08.03	15.8	22.09.03	14.6	16.10.03	18.2	7.11.03	13.4
27.08.03	14.4	24.09.03	49.8	16.10.03	16.0	7.11.03	14.0
27.08.03	28.6	25.09.03	20.4	20.10.03	13.2	10.11.03	12.6
28.08.03	13.8	26.09.03	16.0	22.10.03	10.6	10.11.03	11.4
28.08.03	16.0	26.09.03	17.8	22.10.03	9.4	11.11.03	13.4
29.08.03	12.8	27.09.03	46.0	23.10.03	11.2	11.11.03	12.4
29.08.03	13.6	29.09.03	14.0	23.10.03	18.0	12.11.03	18.2
2.09.03	16.6	29.09.03	10.4	24.10.03	13.0	12.11.03	20.6
2.09.03	13.4	30.09.03	10.8	27.10.03	27.0	13.11.03	12.2
3.09.03	19.2	1.10.03	16.4	27.10.03	12.2		
3.09.03	12.2	1.10.03	10.8	28.10.03	12.6		

Mean of 98 determinations = 16.14
Standard deviation of the mean = 7.18

Table 2.18. Moisture content in sugar cane dry leaves at Mon Desert Alma sugar factory.

Date	% moisture	Date	% moisture	Date	% moisture	Date	% moisture
26.09.03	14.4	11.10.03	30.4	28.10.03	28.7	6.11.03	17.3
26.09.03	12.6	13.10.03	24.0	28.10.03	30.8	7.11.03	7.5
30.09.03	23.5	13.10.03	27.8	29.10.03	28.3	7.11.03	10.9
30.09.03	27.9	14.10.03	22.5	29.10.03	28.0	10.11.03	5.4
7.10.03	20.2	14.10.03	34.3	30.10.03	31.9	10.11.03	8.7
7.10.03	22.3	15.10.03	19.5	30.10.03	12.3	11.11.03	10.1
8.10.03	12.2	15.10.03	18.1	3.11.03	19.6	11.11.03	17.9
8.10.03	15.6	17.10.03	9.9	3.11.03	12.6	12.11.03	4.8
9.10.03	30.7	17.10.03	21.1	4.11.03	21.7	12.11.03	18.2
9.10.03	19.4	18.10.03	36.4	4.11.03	26.7	13.11.03	5.8
10.10.03	30.3	18.10.03	22.2	5.11.03	8.8	13.11.03	21.3
10.10.03	18.0	20.10.03	43.1	5.11.03	16.5	14.11.03	7.9
11.10.03	19.2	20.10.03	67.7	6.11.03	24.9	14.11.03	12.5

Mean of 52 determinations = 20.81
Standard deviation of the mean = 10.98

Table 2.19. Moisture content in sugar cane dry leaves at FUEL sugar factory.

Date	% moisture	Date	% moisture	Date	% moisture	Date	% moisture
23.10.03	24.2	31.10.03	9.2	6.11.03	17.1	10.11.03	8.2
24.10.03	18.8	3.11.03	16.7	6.11.03	13.3	10.11.03	8.9
29.10.03	18.9	4.11.03	10.1	7.11.03	10.7	11.11.03	11.4
30.10.03	15.8	4.11.03	8.6	7.11.03	10.1	11.11.03	8.7
30.10.03	7.3	5.11.03	11.2	8.11.03	22.1		
31.10.03	11.5	5.11.03	12.4	8.11.03	19.9		

Mean of 22 determinations = 13.41
Standard deviation of the mean = 4.95

CHAPTER 3. SEPARATION OF THE SUGAR CANE PLANT INTO FIBRES OF VARIOUS COMPONENT PARTS

This chapter describes the separation of the sugar cane plant, of four cane varieties and of three different ages, into fibres of various component parts by means of a method devised in this work. This was carried out in order to study their Brix-free water capacity and sorption behaviour.

3.1 THE SUGAR CANE PLANT

Sugar cane is a perennial tropical grass of the genus *saccharum*, thought to have evolved in the Burma-China-India area of southern Asia, and later spread to other areas. It produces sturdy stalks 2 to 5 m in height and 3 to 5 cm in diameter. Cane leaves take up water, nutrients and carbon dioxide in the air to form photosynthate (glucose and fructose), the condensation reaction of which produces sucrose, a disaccharide, α-D-glucopyranosyl-β-D-fructofuranoside, which is then stored in the internodes of the cane stalk.

$$6CO_2 + 6\,H_2O \rightleftharpoons C_6H_{12}O_6 + 6O_2$$

$$C_6H_{12}O_6 + C_6H_{12}O_6 \rightleftharpoons C_{12}H_{22}O_{11} + H_2O$$

glucose fructose sucrose

The structures of glucose, fructose and their condensation product sucrose are shown in Fig 3.1.

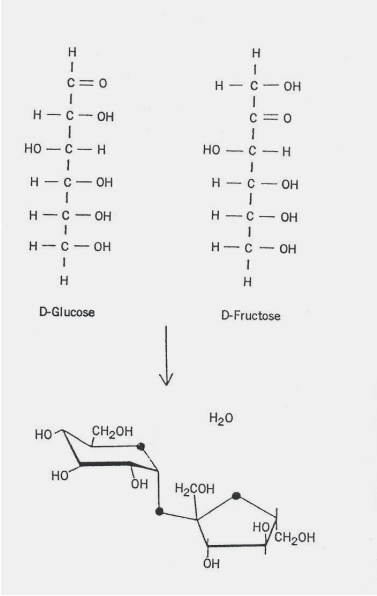


Figure 3.1. Glucose and fructose and their condensation product sucrose (Chen and Chou, 1993).

When the plant needs the stored energy during photosynthesis, the above equations are reversed (respiration) into active energy for synthesis of fats, proteins, etc., for growth and production. There is competition within the plant for the products of photosynthesis. On one hand, glucose and fructose are necessary for rapid growth, and on the other hand, sucrose is needed for storage. Anything that stimulates growth, such as high temperatures, adequate water and nutrients, results in low sucrose % cane and a low juice purity. Conversely, drought and winter conditions result in a high sucrose % cane and high juice purity. The growing season should therefore be warm with high mean day temperatures of about 25 to 30 °C, with adequate moisture and high incident solar radiation; whereas the ripening and harvesting season should be cool, with mean day temperatures of between 10 and 20 °C, frost-free, dry and with high incident radiation.

The composition of the sugar cane depends very much on the cane variety, the region and the climatic conditions under which it is grown, the degree of maturity of the cane and so on. Sugar cane is composed mainly of sucrose, fibre and water, usually in the proportion of about 12%, 15% and 70%, respectively (Anon., 2007), in Mauritius. The remaining constituents are other sugars (glucose and fructose), inorganic materials, nitrogeneous substances, gums, waxes and organic acids (Chen and Chou, 1993).

The sugar cane plant can be divided into three parts: roots, stalk and leaves, the composition of which has been reported by Brown and Blouin (1907) and is shown in

Table 3.1. It can be seen that the main part of the sugar cane plant from which sugar can be extracted is the stalk (Fig 3.2).

Table 3.1. Composition of different parts of cane (Brown and Blouin, 1907).

Composition/%	Stalks	Roots	Leaves
Water	74.96	68.79	74.38
Ash	0.64	1.87	2.23
Fats and wax	0.38	0.54	0.69
Nitrogeneous matter	0.58	1.59	1.70
Fibre { crude cellulose	4.86	9.58	9.18
Fibre { pentosans	3.04	7.04	5.49
Fibre { ligneous bodies	2.14	4.25	4.13
Sugars, etc.	13.40	6.34	2.21

According to Van Dillewijn (1952), the structure of cane stalk can be roughly divided into two parts: an outer peripheral region or rind, and an inner soft pith section, which differ mainly in the relative concentrations of juice-containing cells (parenchyma) and fibrovascular bundles. The rind has a strong outer cuticle often covered with a layer of wax, which prevents evaporation of water from the inner cells as well as giving them protection against mechanical injuries and attack by micro-organisms. The term “rind” normally refers to the relatively thin external layer of the stalk, but it is also taken as the adjacent layer of dense tissue in which there is a high concentration of vascular bundles and relatively few juice cells (Fig 3.3). By virtue of its high fibre content this is the strongest portion of the plant and it gives the stalk much of its characteristic strength and rigidity.

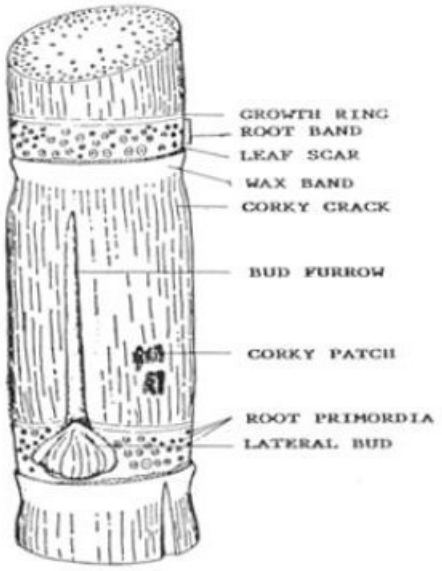


Figure 3.2. Cane stalk.
(Van Dillewijn, 1952)

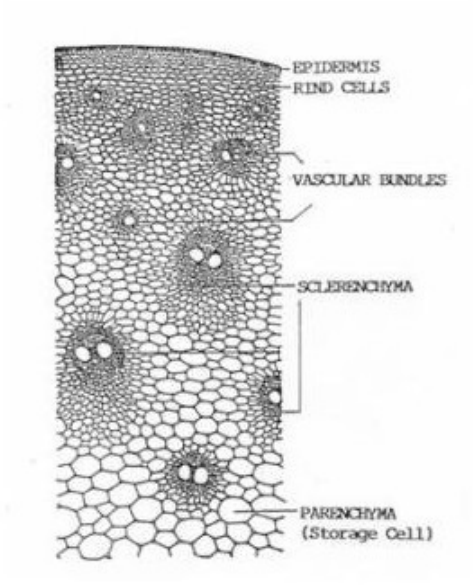


Figure 3.3. Cross-section of stem.
(Van Dillewijn, 1952)

The juice-containing parenchyma cells are thin-walled and contribute only to a small part of the total fibre in the cane. The vascular bundles on the other hand contain many small thick-walled cells which surround the large vessels and sieve tubes. The vessels carry water from the roots and the sieve tubes conduct sugary juices from the leaves. High tensile strength in the vascular bundles appears to impart good milling quality to the cane variety.

The fibrous residue of the sugar cane stalk after crushing and juice extraction is known as bagasse, it consists mainly of cellulose, pentosans and lignin. Paturau (1989) enumerated two important types of fibrous residue occurring in bagasse: a) the tough, hard-walled, cylindrical cells of the rind and vascular tissues (true fibre); and b) the soft, thin-walled, irregularly shaped parenchymatous cells of the inner stalk tissue (pith). The vessel segments, also associated with the vascular bundles, because of their non-fibrous character and because any depithing steps (the separation of pith from fibres) will remove a large portion of the cells, are often, but not always, considered as a pith fraction.

The fibrous physical composition of bagasse can be summarized as shown in Table 3.2.

Table 3.2. Fibrous physical composition of bagasse (Villavicencio, 1974).

		Mass/%
Fibres suitable for paper-making	True fibres	55
	Vessel segments	20
Pith	Parenchyma cells	20
	Other non-fibrous components, e.g. nodes	5

According to Paturau (1989), the true fibre and the pith have almost the same chemical composition, but their structure differs widely. The true fibres have a fairly high ratio of length-to-diameter (approximately 70), and a relatively high coefficient of expansion and contraction upon wetting and subsequent drying. This results in close bonding of one fibre with another and accounts for the strength, cohesiveness and the ability to become matted when subjected to pulping processes.

The pith cells are of irregular size and shape, with a length-to-diameter ratio of about 5. They are characterised by their absorbent properties. They do not bond together and so tend to weaken any pulp in which they are incorporated and, further, prevent its rapid drying. However, they can absorb many times their mass of liquid.

3.2 THE CHOICE OF SUGAR CANE SAMPLES FOR FIBRE EXTRACTION

In this work, in order to obtain cane samples of different varieties and of differing ages in the same environment, the materials were removed "randomly" from the experimental rows of maturity testing trials of recently released cane varieties. These trials were established by the Plant Physiology Department of the MSIRI in various agro-climatic environments of the island, having as treatments three harvest dates and ten cane varieties. As cane harvesting in Mauritius usually starts at the beginning of June and ends in November, three harvest dates were included in these trials: early, middle and late, i.e. June, September and November respectively, apart from the cane varieties that are being compared.

The trial was planted in 2000 and grown under the same conditions as in the North of Mauritius (in the area called Nouvelle Industrie – see Fig 1.1). The experimental design was a split-split plot with three replicates. The main plots consisted of three harvest dates (July, September and November – all plots were of 52 weeks of age at harvest of ratoon crop), the sub-plots represented the ten cane varieties and the sub-sub-plots, represented the three sampling dates for the Plant Physiology Department. The plot consisted of six rows, each of which was 10 m long. The samples were removed during the second half of July 2003 for four cane varieties (early maturing variety M 1557/70, middle-to-late maturing varieties R 579 and M 1400/86, and late maturing variety R 570). The ages of the crops at the time of sampling were: 52, 44 and 36 weeks (after the last harvest in 2002). The samples removed for fibre extraction consisted of four stalks each from each of the three replicates with all their attached dry and green leaves. Additional cane tops and dry leaves for each sample were collected since a trial series of cane samples harvested and processed previously, in 2001, had shown that the fibres extracted from cane tops and dry leaves were insufficient for the subsequent Brix-free water determination. The cane samples harvested are detailed in Table 3.3 and the cane varieties chosen are illustrated in Fig 3.4.

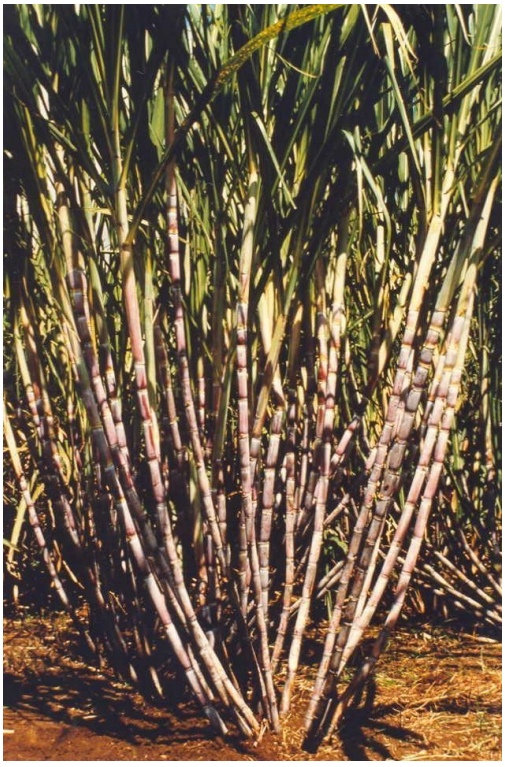
The cane variety R 570 which formed part of the trial series grown at Mon Trésor (Fig 1.1) and harvested in 2001 was included in this work for comparison with the R 570 grown at Nouvelle Industrie and harvested in 2003, to see the effect of different growing regions on the subsequent Brix-free water values.

Table 3.3. Cane samples harvested for fibre extraction.

Variety	Maturing characteristic	Age/weeks	Year of harvest	Growing site
R 570	late	37, 44 and 52	2001	Mon Trésor
R 579 R 570 M 1557/70 M 1400/86	middle-to-late late early middle-to-late	36, 44 and 52	2003	Nouvelle Industrie

R 579

R 570



M 1557/70

M 1400/86

Figure 3.4. The four cane varieties chosen for fibre extraction.

3.3 EXTRACTION OF PLANT FIBRES

Some fibres are embedded within the tissues of the plant, which have to be removed to obtain the fibres, while fibres of cotton and kapok need only be removed from the seed case. Fibres can be extracted from tissue either by beating and/or scraping the fresh leaves or by softening the tissues by retting. For some fibres, retting alone is not sufficient and a further chemical treatment needs to be carried out, for example, the use of alkaline solutions in degumming of ramie (Jarman, 1998), which is an Asiatic shrub, *Baehmeria nivea*, in the nettle family, yielding a fibre used in making textiles, upholstery, thread and paper.

3.3.1 Industrial extraction of fibres from flax, hemp, jute and sisal

Jarman (1998) described the dew retting and water retting methods of fibre extraction from flax and hemp stem. In the former, freshly pulled straw is spread in the field in relatively thin layers; rain, snow or dew causes fungi and bacteria to enter the stomata of the stems and attack the pectin binding the fibres together. In the water retting method, retting can be carried out either in cold water in rivers, pools or ditches: the small, loosely bound bundles are submerged in water and weighted down with large stones to keep them submerged; or in a tank using warm water at about 28 °C. In cold water retting, at temperatures below 15 °C, there is little bacterial activity whereas in warm water retting, the principal retting organisms are anaerobic bacteria. Once the retting is completed after days, the straw bundles are rinsed and taken back to the field where they are piled in open "wigwam" fashion and allowed to dry completely before fibre extraction. The process consists of beating and shaking the broken straw to remove most of the wood and finally "hackling" to align the fibres and remove the woody stem portions (Jarman, 1998).

In jute stems, Jarman (1998) also described the retting method whereby the bundles of jute stems are immersed in pools, canals, slow-moving streams or ponds at a depth of 1-2 m. The bacteria on the stem will attack the plant tissues surrounding the fibres, softening them sufficiently so that they can be washed away leaving the fibres unaffected. When retting is complete, the bundles must be removed from the water and the fibres stripped from the stem before it over-rets. The retted stems are first gently beaten at the base with a mallet to

loosen the fibre. The woody core is then broken near the base and the broken pieces discarded. The fibre is stripped from the core; afterwards, adhering pieces of bark and broken stick are removed by lashing the stripped fibre on the surface of the water. The fibre is then washed, wrung dry and sun-dried on horizontal wires or poles. Retting is the most important step in the production of good quality jute; under-retting or over-retting can lead to downgrading of the jute fibres produced.

Leaf fibres are usually extracted without recourse to retting. In its simplest form the extraction is carried out by first beating the leaf to soften the non-fibrous soft tissue, and then scraping the fibres clean. Sisal leaf is such an example requiring a mechanical device to clean the fibres (Jarman, 1998). However, some leaves containing high levels of pectins will require retting as well as chemical treatment.

3.3.2 Methods reported for the sugar cane plant

In an attempt to extract fibres from sugar cane for use as textile fibres, Jhingooree *et al.* (2000) applied chemical degumming treatments to the rind section of the sugar cane stalk, which they boiled in sodium hydroxide solutions of various concentrations (5, 10 and 15%) for a limited period (2, 4 and 5 hours). The fibrous tissues obtained after the degumming treatment still contained debris from degraded vegetable matter, and required combing with a hair comb to get rid of the undesirable intercellular gummy matter binding the fibres together. The combing process could not be carried out too many times as it led to the breakage of the fibres. They found that the tenacity of the subsequently separated fibres decreased with increase of the alkali concentration as well as the boiling time. Instead of cane rind, when whole cane was crushed by a hammer before the treatment of alkali, a lower concentration of alkali (< 5%) was required to achieve the same results as with 15% alkali on the rind section. When crushed cane was treated with high concentrations of alkali, the fibres deteriorated. Softeners and crease-resisting agents were also applied to the fibres before combing to enable the fibres to swell and become looser. In so doing, the combing process became easier.

To extract fibres from cane samples, Mangion and Player (1991) processed cane samples *via* the use of a wet disintegrator, a Jeffco cutter-grinder and a gyratory-type machine grinder. In physical appearance, the wet disintegrator and Jeffco cutter grinder materials were similar, with pith and long fibre in the sample being easily distinguishable and separable. The mechanical grinder which consisted of agitated concentric steel rings

which ground the material, produced a fine powdery product, with pith and fibre being macerated into one homogeneous sample. Subsequent Brix-free water determination showed similar results for the wet disintegrator and Jeffco cutter-grinder materials while the mechanical grinder material had higher results by five units, which they attributed to the increased sample surface area per unit mass by the use of the mechanical grinder.

Moodley (1991) reported that cane stalks (2 kg) were cut into 10-15 cm sections, which were shredded in a Wadell shredder for a period of 15 seconds and then mixed thoroughly. Water (2600 mL) was added to the crushed cane (400 g), and the sample was further prepared in a cold digester for two minutes. The material was then transferred to a fibre-pith separator (Fig 3.5) consisting of a perforated stainless steel plate of 2 mm diameter holes made into a basket (35 x 15 x 15 cm) submerged in a trough (45 x 39 x 20 cm) filled with water (25 L) and rotated for 20 minutes at 23 rpm by means of an electric motor. The water containing fibre and pith was filtered through a 1.651 mm screen to retain the fibre and a 100 micron screen to collect the pith.

Figure 3.5. Fibre-pith separator (Moodley, 1991).

A recent version of a fibre-pith separator was reported by Chinsamy *et al.* (2004). It consisted of a sample pot of which the bottom and the lid were made of a perforated plate of 1.6 mm diameter holes. Prepared shredded cane was placed in the pot, water flowed in from the bottom of the pot and compressed air was introduced at the top of the pot to

agitate the mixture in the pot, and assist the separation of pith from the fibre. Coarse fibres were retained in the pot, finer ones and pith fell through a funnel (cone-shaped vessel) into a secondary screening pot with 850 micron mesh lid, which retained fine fibres while the pith fell into a cone-shaped vessel made of fine mesh screen, on which the pith collected, and through which water flowed out to drain.

Snow (1974) described a fibre-pith separator consisting of a drum made by rolling a piece of brass screen 30.5 cm wide and 30.5 cm long into a cylinder. The perforations consisted of 3.18 mm round holes on 6.36 mm centres. Cane, which had previously been shredded in a 4 L commercial Waring Blendor with 1 L of water at a high speed for 1 minute, was put into the drum which in turn was put in the apparatus, the tank of which was filled with water. The drum was rotated for 5 minutes at 18 rpm by means of a 190 W electric motor. The water containing the pith was drawn off and the pith collected on a Tyler 100 mesh sieve. The hard fibre was recovered from the rotary drum. Commercial processes also exist to separate bagasse into fibre and pith for the manufacture of paper and fibre board (Keller 1966, Atchison 1971).

Another effective method for fibre extraction is the sample preparation method currently used in Mauritius for direct cane analysis for pol % cane and fibre % cane (Anon., 1991). This involves shredding the cane with a cane chipper (Fig 2.2). A mass of 329 g of this shredded cane is weighed out and transferred to a wet disintegrator (Fig 3.6). After adding 1 L of water, the wet disintegrator is operated for 8 minutes at a speed of 8000 rpm. After the treatment, the fibres are normally well separated.

Figure 3.6. Jeffco wet disintegrator.

This last method using a wet disintegrator looked very promising as confirmed by Mangion and Player (1991); however, it would be best if the use of the cane chipper as well as any chemicals could be avoided. Since it was desired to extract fibres from various components of cane, it was necessary to experiment on cane samples so as to find the optimum conditions for fibre extraction from each of the cane components without inflicting too much injury to their fibres.

With the objective of separating fibres from pith, a basket identical to that described by Moodley (1991) was built into a commercially available water bath with an outlet orifice at the inclined base to facilitate drainage of the water from the bath (Fig 3.7). Experimentation showed that the separation of fibre from pith by using this method was poor and time-consuming, as recycling of the pith into the fibre occurred when the sample basket rotated in a trough of water, where the pith was in suspension. Considering the number and the size of samples requiring the treatment, a different method had to be found.

Figure 3.7. Custom-built fibre-pith separator.

3.4 EXPERIMENTAL

The actual method used for fibre extraction from sugar cane samples is detailed in the following section.

3.4.1 Materials

The samples used for fibre extraction are shown in Table 3.3. The cane harvested in 2003 consisted of four varieties: R 579, R 570, M 1557/70 and M 1400/86, each aged 36, 44 and 52 weeks. The cane variety R 570 harvested in 2001 aged 37, 44 and 52 weeks was also included in this report for comparison purposes. For each sample, three replicates consisting of four cane stalks were processed.

3.4.2 Equipment

The Jeffco cutter-grinder described in Section 2.1.2 was used to pre-treat peeled cane stalk, rind, dry leaves and green leaves prior to fibre extraction in a Jeffco wet disintegrator.

The Jeffco wet disintegrator used (Fig 3.6) was a Model 292 with a water-jacketed 7 L-bowl and a standard 50 Hz AC motor of 2250 W, 3 phase, 2880 rpm and 415 volt. The bowl unit could be easily lowered or raised for sample disintegration and tilted for sample unloading. Up to four blades could be fitted at right angles to each other on the central vertical spindle which can be rotated at high speed to disintegrate the cane sample.

A Pinette Emidecau press (see Section 2.1.2) was used to press out liquid from fibre cake prepared from sugar cane components.

A sugar refractometer (see Section 2.1.2) was needed to measure the Brix of the washings from the fibre cake to ensure no residual Brix remained in the extracted fibre.

A selection of three sieves with openings of 2 mm, 1.18 mm and 38 µm were assembled on top of each other. The top two sieves served to retain the fibre and the bottom sieve of 38 µm, the pith.

Because of the large number of samples required to be dried, a large capacity industrial oven equipped with a thermostat was required (Fig 3.8).

Figure 3.8. Industrial drying oven.

3.4.3 Method of fibre extraction from the sugar cane plant

When the three replicates (each consisting of four stalks) of the four varieties aged 52 weeks were delivered at the laboratory, for each sample (a total of 12), the number of dry leaves and green leaves were noted, separated from the stalks and weighed. The cane tops, after being detached from the stalks at the point where they naturally broke off from the stalks, were weighed. All the nodes were removed, weighed and discarded (this was done because the nodes could not be separated into homogeneous fibres and proved difficult to disintegrate). The rind was peeled off longitudinally from the stalk. The masses of the tops, rind and peeled stalk were determined.

The extraction of fibres starting first with the tops, followed by green leaves, peeled stalk, rind and dry leaves, could be completed within one week. All the samples (12 x 5) were separated into fibre and pith/fines, dried and sealed in plastic bags pending dry sieving at a later stage.

The optimum conditions for fibre extraction from the various components of the sugar cane plant depend on the type of sample processed: the sample mass used and the disintegrating time vary, as detailed in the following section.

3.4.3.1 *Cane stalk*

The peeled cane stalks were cut longitudinally into thin sticks (about 6-8 pieces depending on the diameter of the stalk), and put through a Jeffco cutter-grinder. About 500 g of the sample was then placed in a Jeffco wet disintegrator followed by 4 L of water. The disintegrator was operated at 8000 rpm for 30 seconds. The fibres were then inspected to see whether they were well separated and soft; if not, the disintegration was continued until good separation was obtained. After all the cutter-ground stalks had been processed in this manner, the resulting fibres and pith were washed free from gummy matter and rinsed under tap water, using a 38 μm sieve to catch the sample. Retting of the sample was then effected overnight in a bucket of water.

The determination of Brix-free water in fibres involves contacting the fibres with a 10° Brix sucrose solution and measuring the increase in the Brix of the resulting solution. In order to ensure that any increase in Brix is due to the preferential absorption of the water by the fibres and not the elution of the residual Brix in the fibres into the solution, the fibres must be washed free of sucrose. Hence after retting, the sample which consisted of a mixture of fibres and pith, was well washed by filling the cup of the Pinette Emidecau press with some of the sample, which was then washed with 3 L of tap water. The washing was repeated two more times. The water in the sample was pressed out by using a pressure of 20 MPa and the cake was weighed. To test for residual Brix in subsequent washing, if the mass of the cake was for example 180 g, then (150 x 180)/16=1687.5 mL water were used to wash the sample. The wash liquid was tested for Brix by using a refractometer, and the Brix value was required to be less than 0.01. If not, the washing and testing was repeated until the Brix of the washings was less than 0.01. The volume of water calculated above to wash the sample, and to test for residual Brix in the washing, is

based on the manner in which the Brix-free water determination is performed on the sample (Mangion and Player, 1991). This is done by adding 150 mL of a 10° Brix sucrose solution to 8 g of dry sample (here the moist cake is assumed to contain 50% moisture and hence double this mass is used in the calculation), and measuring the increase of Brix in the resulting solution. The washing was repeated on the remainder of the sample until all was free from residual Brix.

To separate pith from the fibre, the cakes from the Pinette Emidecau press were soaked in a bucket of water. A sieve assembly was mounted with a 2 mm, 1.18 mm and 38 μm sieve in order on top of each other. A handful of fibre and pith mixture was put onto the top sieve, and running tap water was used to wash the pith into the bottom sieve. Separation of pith from the fibre in the sample was thus effected. The separated fibre was hand pressed and placed in a metal tray for drying in an oven at 65 °C to constant mass. The separated pith was also dried in the same way.

The dried fibre samples sometimes still contained some pith, and dry sieving by using a 1.18 mm sieve mounted on top of a receiver was used to ensure complete separation.

3.4.3.2 *Rind, dry leaves and green leaves*

These samples were treated as for the cane stalk. Pre-treatment in a Jeffco cutter-grinder was necessary, the sample mass and the time of disintegration are indicated in Table 3.4.

Table 3.4. Preparatory treatment process for fibre extraction from sugar cane plant components.

Sample	Cutter-grinder	Mass used in the Jeffco wet disintegrator/g	Disintegration time/s
Top	-	400	60
Stalk	✓	500	30
Rind	✓	300	75
Dry leaves	✓	100	60
Green leaves	✓	300	30

3.4.3.3 *Cane tops*

After the cane tops were detached from the stalks at the point where they naturally broke off the stalks, they were cut into 10 cm lengths and into thin strips lengthwise; in so doing,

pre-treatment in a Jeffco cutter-grinder was not necessary. The top sample was then treated as for the cane stalk; the sample mass and the time of disintegration required are indicated in Table 3.4.

The three replicates (four stalks each) of the four varieties aged 44 weeks were treated in the same manner the following week, and those aged 36 weeks the week thereafter.

Some relevant fibre extraction processes from the sugar cane plant are illustrated in Figs 3.9 – Fig 3.21.

Figure 3.9. Removal of cane nodes.

Figure 3.10. Removal of green leaves.

Figure 3.11. Green leaf sample before and after cutter-grinding.

Figure 3.12. Washing of green leaf sample after cutter-grinding and retting.

Figure 3.13. Green leaf cake washed free from Brix (top) and three fractions after wet-sieving.

Figure 3.14. Dry leaf sample before and after cutter-grinding.

Figure 3.15. Dry leaf cake washed free from Brix (top) and three fractions after wet-sieving.

Figure 3.16. Cane top sample cut into 10-cm lengths and then in thin strips before being treated in a wet disintegrator.

Figure 3.17. Cane stalk with rind (top), with rind removed and cut into thin sticks (left) and rind (right).

Figure 3.18. Stalk sample before and after cutter-grinding.

Figure 3.19. Stalk cake washed free from Brix (top) and three fractions after wet-sieving.

Figure 3.20. Rind sample before and after cutter-grinding.

Figure 3.21. Rind cake washed free from Brix (top) and three fractions after wet-sieving.

3.4.4 Characterisation of the sugar cane component parts

3.4.4.1 *Gross calorific value*

The gross calorific value (GCV) of the extracted fibres was measured with a Parr model 1241 adiabatic oxygen bomb calorimeter (Fig 3.22).

Samples of the four cane varieties R 579, R 570, M 1557/70 and M 1400/86 of the same age (about 52 weeks old) were obtained. After the nodes were rejected, rind was peeled off the stalk, and fibres were extracted from both rind and stalk. The combined rind fibre and fines, as well as stalk fibre, stalk pith and dry leaf were examined. Long fibres in rind, stalk and dry leaf were cut into fine particles with a pair of scissors, before drying at 105 °C for three hours in a drying oven. Each sample, when required, was removed from the oven, pressed into a pellet and burnt in the adiabatic oxygen bomb calorimeter to determine its GCV.

The calorific value of fibre (or bagasse) is determined by burning a weighed sample in an adiabatic bomb calorimeter under controlled conditions and is computed from the temperature observations made before and after the combustion, with allowances being made for the thermometer and thermochemical reactions. Benzoic acid pellets weighing between 0.9 and 1.25 g were used to calibrate the calorimeter.

Figure 3.22. Parr model 1241 adiabatic oxygen bomb calorimeter with pellet press on the left.

3.4.4.2 *Infrared spectroscopy and scanning electron microscopy*

Fibres were also investigated by infrared spectroscopy and scanning electron microscopy in order to observe any structural and morphological differences.

For the infrared technique, the instrument used was a Nicolet IR spectrophotometer and potassium bromide disc of the sample was analysed. Samples of stalk fibre and pith of four different cane varieties aged 37, 44 and 52 weeks, and hard fibres extracted from the top, rind, dry leaf and green leaf of the same four cane varieties aged 52 weeks were examined.

For the scanning electron microscopy, samples were mounted on an aluminium stub by using double-sided carbon tapes, the specimens were then sputter-coated with gold in a Polaron coating unit, and viewed in a Leo 1450 scanning electron microscope at 10 kV. Fibres of stalk, rind, top, dry leaf and green leaf together with stalk pith of cane variety R 575 aged 52 weeks were examined (this variety was used in the initial stages of the work to test the various procedures).

3.5 RESULTS AND DISCUSSION

Results of the initial masses of the sugar cane parts prior to fibre extraction of the four varieties aged 52 weeks are shown on the left hand side of Table 3.5. Since most stalks did

not have many dry leaves attached to them, additional material pertaining to the relevant variety and replicates were collected from the field, the number of leaves was counted, weighed and are shown in Table 3.5. Green leaves attached to the samples were also counted and shown. As tops from the four-stalk sample were insufficient, additional material was collected from the field. The masses of stalk (internode) with rind, nodes, rind only, and stalk only, when measured, are also shown in Table 3.5. The mass of cane is taken as the sum of the mass of stalk with rind and nodes. Results of the dry mass of these sugar cane components are shown on the right hand side of Table 3.5; they are the sum of the three sieved fractions after oven-drying. The dry mass result for tops is the combined mass of the tops from the cane source and the additional material collected.

Similarly, results obtained for the cane varieties aged 44 weeks and 36 weeks are indicated in Tables 3.6 and 3.7 respectively.

Details of the three sieved fractions after oven-drying (> 2 mm, > 1.18 mm and > 38 μm) of the sugar cane components of the four varieties aged 52 weeks are shown in Table 3.8, which also shows details of the two fractions after dry-sieving (> 1.18 mm and < 1.18 mm). The latter enables the calculation of fibre/fines ratio within each component part, which, in the case of stalk, implies fibre/pith ratio. Similar results for samples aged 44 weeks and 36 weeks are shown in Tables 3.9 and 3.10 respectively.

During fibre extraction from the cane variety R 570 harvested in 2001, the use of an assembly of three sieves for wet sieving had not yet been thought of, all the fibre and fines/pith were retained on the 38 μm sieve. Dry sieving was subsequently effected by using a 1.18 mm sieve on top of a receiver to ensure separation of fibre and pith/fines. Details of dry masses of the sugar cane components of R 570 of three ages harvested in 2001 after fibre extraction and dry sieving are shown in Table 3.11. From Tables 3.8 – 3.11, it can be seen that the total mass after dry sieving is greater than the mass after fibre extraction; this is because the extracted fibres were weighed after oven drying; the dry sieving was effected much later and the sieved fractions were not re-dried in the oven. The fibre/fines or fibre/pith ratios are calculated.

3.5.1 Material loss during fibre extraction

From Tables 3.5, 3.6 and 3.7, the material loss in each component after the fibre extraction process can be estimated. This is shown for cane samples of the four varieties and three

ages in Table 3.12. The material loss consists principally of the loss of water and to a much lesser extent, sucrose and other constituents of the sugar cane component parts.

The greatest loss appears to be in the stalk, followed by top, rind, green leaf, and then dry leaf. For the dry leaf, the loss for the 36-week batch was 37.4%, for the 52-week batch, 40.1% and for the 44-week batch, 62.6%. This is probably because the 44-week batch samples were harvested on a rainy day; the leaves had picked up rain water and had a higher moisture content than normal.

3.5.2 Dry mass % cane extracted from sugar cane component parts

From Tables 3.5 – 3.7, it is also possible to calculate the dry mass % cane extracted as rind, stalk fibre and stalk pith. The results are presented in Table 3.13. Since the nodes had been excluded from fibre extraction, the mass of cane was taken to be the mass of stalk plus rind only. The lowest dry mass extracted was from the hard fibre of the stalk, followed by stalk pith and rind.

Table 3.5. Masses of cane samples aged 52 weeks prior to and after fibre extraction.

Sample		Mass of cane components prior to extraction/g										Dry mass of cane components after extraction/g					
		Cane source								Additional material							
		Dry leaf	Green leaf	Top	Stalk with rind	Nodes	Cane	Rind only	Stalk only	Dry leaf	Top	Dry leaf	Green leaf	Top	Rind	Stalk fibre	Stalk pith
R 579	1	neg.	(34) 760	280	5700	1580	7280	NA	NA	(20) 140	240	92.9	179.3	50.2	204.0	131.0	105.4
	2	neg.	(36) 820	320	5760	1940	7700	NA	NA	(35) 300	380	183.7	195.1	45.4	204.4	100.1	118.2
	3	neg.	(31) 580	220	2760	920	3680	NA	NA	(19) 180	370	112.9	120.7	39.3	101.3	28.6	51.4
R 570	1	neg.	(32) 960	180	4420	1100	5520	NA	NA	(27) 300	260	180.0	221.0	47.2	135.1	57.8	64.0
	2	neg.	(38) 800	380	3220	1040	4260	NA	NA	(30) 280	440	149.7	184.7	68.4	151.0	48.5	57.2
	3	neg.	(31) 760	360	4420	1140	5560	NA	NA	(15) 180	420	110.9	172.3	41.4	225.6	55.5	87.4
M 1557/70	1	neg.	(52) 1000	420	7080	2080	9160	NA	NA	(26) 250	370	149.8	257.4	96.7	363.6	101.6	150.3
	2	neg.	(42) 740	290	2530	700	3230	NA	NA	(14) 120	240	63.6	176.0	71.9	124.1	45.0	48.7
	3	neg.	(52) 1200	380	5650	1540	7190	NA	NA	(42) 320	280	192.5	285.8	35.8	269.2	93.1	124.3
M 1400/86	1	neg.	(29) 500	160	4200	1120	5320	NA	NA	(49) 380	250	224.6	110.4	63.2	178.9	58.1	100.2
	2	neg.	(61) 1030	320	5830	2110	7940	NA	NA	(38) 340	140	225.5	277.2	68.4	288.2	95.0	137.7
	3	neg.	(30) 460	200	3930	1220	5150	NA	NA	(14) 140	310	77.5	126.0	60.8	203.8	58.9	77.2

Note: Numbers in parentheses indicate the number of leaves.

Table 3.6. Masses of cane samples aged 44 weeks prior to and after fibre extraction.

Sample		Mass of cane components prior to extraction/g										Dry mass of cane components after extraction/g					
		Cane source								Additional material							
		Dry leaf	Green leaf	Top	Stalk with rind	Nodes	Cane	Rind only	Stalk only	Dry leaf	Top	Dry leaf	Green leaf	Top	Rind	Stalk fibre	Stalk pith
R 579	1	neg.	(39) 1060	300	4040	1520	5560	920	3120	(33) 440	210	136.2	201.5	56.5	161.3	59.2	93.4
	2	neg.	(36) 760	220	3480	1280	4760	1020	2460	(67) 660	200	285.4	158.8	46.6	145.9	37.6	58.1
	3	neg.	(39) 820	250	6100	1830	7930	1260	4840	(61) 650	170	274.4	166.4	51.0	196.1	79.9	129.8
R 570	1	neg.	(36) 860	300	5490	1350	6840	1400	4090	(37) 540	270	216.3	190.5	77.8	311.2	86.7	106.0
	2	neg.	(35) 790	280	3410	920	4330	930	2480	(29) 360	280	150.4	160.7	71.2	198.2	54.6	63.9
	3	neg.	(43) 1040	350	4560	1340	5900	1200	3360	(52) 700	230	243.9	224.8	70.1	282.7	74.2	88.7
M 1557/70	1	neg.	(36) 780	260	4350	1220	5570	1370	2980	(33) 630	160	183.9	160.7	56.1	267.4	51.2	86.2
	2	neg.	(44) 900	250	3490	840	4330	920	2570	(40) 550	240	161.2	210.7	57.2	213.2	56.5	90.8
	3	neg.	(38) 880	200	3850	890	4740	1020	2830	(37) 480	190	187.8	167.0	47.2	188.5	46.7	69.8
M 1400/86	1	neg.	(37) 890	350	4330	1430	5760	1260	3070	(43) 580	210	201.1	217.6	77.2	212.7	54.8	92.4
	2	neg.	(36) 720	320	4700	1430	6130	1190	3510	(52) 640	220	276.2	167.6	74.1	234.8	62.7	122.6
	3	neg.	(38) 790	300	4160	1080	5240	1030	3130	(29) 420	180	167.9	188.8	70.2	208.9	58.6	100.3

Note: Numbers in parentheses indicate the number of leaves.

Table 3.7. Masses of cane samples aged 36 weeks prior to and after fibre extraction.

Sample		Mass of cane components prior to extraction/g										Dry mass of cane components after extraction/g					
		Cane source								Additional material							
		Dry leaf	Green leaf	Top	Stalk with rind	Nodes	Cane	Rind only	Stalk only	Dry leaf	Top	Dry leaf	Green leaf	Top	Rind	Stalk fibre	Stalk pith
R 579	1	neg.	(33) 480	140	2920	790	3710	1365	1555	(57) 380	200	218.5	111.7	32.9	111.7	41.4	52.9
	2	neg.	(33) 520	120	2920	860	3780	1250	1670	(27) 220	180	135.2	118.1	35.4	95.2	31.5	47.6
	3	neg.	(34) 750	150	2640	880	3520	700	1940	(23) 160	110	89.5	157.1	33.7	139.7	31.1	46.9
R 570	1	neg.	(40) 960	210	2660	960	3620	850	1810	(28) 270	170	181.5	244.9	53.1	162.1	36.8	63.4
	2	neg.	(46) 1200	250	4800	1580	6380	525	4275	(44) 410	180	278.9	305.1	53.1	343.3	56.4	99.9
	3	neg.	(36) 870	250	4600	1300	5900	590	4010	(27) 290	180	185.5	206.8	55.8	308.8	44.2	85.8
M 1557/70	1	neg.	(40) 770	280	2690	190	2880	804	1886	(26) 240	140	139.9	164.0	47.5	144.7	26.6	47.9
	2	neg.	(44) 880	200	3070	1080	4150	930	2140	(23) 200	170	115.7	187.4	40.5	207.7	28.5	63.8
	3	neg.	(39) 620	160	2480	840	3320	723	1757	(18) 140	160	87.8	148.0	40.5	143.2	20.6	43.8
M 1400/86	1	neg.	(38) 790	200	3350	1340	4690	970	2380	(30) 300	180	209.2	219.1	57.0	158.5	30.0	62.4
	2	neg.	(41) 770	230	3860	1520	5380	1260	2600	(26) 220	190	135.6	172.7	58.8	231.5	36.1	88.3
	3	neg.	(40) 770	210	2810	1140	3950	635	2175	(46) 350	150	232.8	193.7	47.9	122.1	35.6	55.9

Note: Numbers in parentheses indicate the number of leaves.

Table 3.8. Masses of cane components aged 52 weeks after wet and dry sieving.

Sample		Dry mass of three fractions* after wet sieving/g					Dry mass of two fractions+ after dry sieving/g					Fibre/fines ratio				
		Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk
R 579	1	68.1	149.6	37.9	178.2	131.0	54.5	136.9	37.9	159.0	96.6	1.2	2.21	6.1	2.80	0.88
		9.0	23.2	6.1	0.0	4.2	44.3	62.0	6.2	56.8	109.4					
		15.8	6.5	6.2	25.8	101.2										
	2	137.2	160.1	33.6	180.5	100.1	110.0	138.1	33.6	138.5	72.7	1.3	2.17	4.8	1.79	0.59
		13.4	29.6	4.9	0.0	8.2	80.4	63.6	6.9	77.3	123.6					
		33.1	5.4	6.9	23.9	110.0										
	3	84.0	97.3	28.6	92.8	28.6	69.6	78.8	28.6	67.0	22.6	1.5	1.85	5.3	1.95	0.43
		10.3	18.0	5.3	0.0	5.8	45.4	42.6	5.4	34.3	52.2					
		18.6	5.4	5.4	8.5	45.6										
											Mean	1.3	2.08	5.4	2.18	0.63
R 570	1	129.7	182.4	35.3	128.0	57.8	109.0	140.2	35.3	89.1	48.3	1.4	1.60	6.0	2.03	0.75
		16.5	33.1	6.1	0.0	14.3	76.1	87.5	5.8	43.9	64.4					
		33.8	5.5	5.8	7.1	49.7										
	2	106.7	153.5	53.6	141.1	48.5	96.1	130.4	53.6	73.7	41.1	1.6	2.33	6.3	0.93	0.72
		14.6	25.1	6.4	0.0	8.7	58.4	55.9	8.4	79.1	57.4					
		28.4	6.1	8.4	9.9	48.5										
	3	71.1	145.0	29.5	209.5	55.5	60.4	119.3	29.5	139.4	53.0	1.1	2.30	6.5	1.69	0.61
		12.6	20.1	7.4	0.0	9.1	54.1	51.9	4.5	82.4	87.6					
		27.2	7.2	4.5	16.1	78.3										
											Mean	1.4	2.08	6.3	1.55	0.69
M 1557/70	1	96.7	224.5	77.7	328.3	101.6	80.9	174.5	77.7	218.2	82.7	1.2	2.45	6.0	1.49	0.55
		17.9	23.3	6.0	0.0	14.4	66.1	71.2	12.9	146.8	151.3					
		35.2	9.6	12.9	35.3	135.9										
	2	42.7	149.0	56.6	110.1	45.0	36.0	126.4	56.6	79.3	36.7	1.2	2.33	6.2	2.19	0.76
		6.4	19.2	6.2	0.0	5.6	27.8	54.3	9.1	36.2	48.4					
		14.5	7.8	9.1	14.0	43.1										
	3	121.0	244.5	25.6	238.3	93.1	115.5	203.9	25.6	180.0	76.6	1.4	2.35	5.4	2.05	0.62
		24.5	33.6	5.5	0.0	13.5	82.7	86.8	4.7	87.9	123.5					
		47.0	7.7	4.7	30.9	110.8										
											Mean	1.3	2.38	5.9	1.91	0.64
M 1400/86	1	151.8	91.0	49.2	158.9	58.1	134.6	82.3	49.2	93.3	54.3	1.4	3.06	6.9	1.32	0.55
		20.0	11.4	6.9	0.0	13.7	95.2	26.9	7.1	70.6	98.8					
		52.8	8.0	7.1	20.0	86.5										
	2	150.5	239.1	53.5	252.1	95.0	131.0	191.4	53.5	189.2	74.1	1.3	2.21	6.0	1.66	0.54
		24.0	30.2	6.1	0.0	22.0	96.1	86.5	8.8	113.7	136.0					
		51.0	7.9	8.8	36.1	115.7										
	3	49.4	104.7	47.0	176.4	58.9	42.5	89.2	47.0	142.4	42.0	1.2	2.66	7.7	1.96	0.56
		9.1	13.7	7.7	0.0	13.8	34.1	33.5	6.1	72.7	75.2					
		19.0	7.6	6.1	27.4	63.4										
											Mean	1.3	2.64	6.9	1.65	0.55

* The three fractions are > 2 mm, > 1.18 mm and > 38 µm.

+ The two fractions are > 1.18 mm and < 1.18 mm.

Table 3.9. Masses of cane components aged 44 weeks after wet and dry sieving.

Sample		Dry mass of three fractions* after wet sieving/g					Dry mass of two fractions+ after dry sieving/g					Fibre/fines ratio				
		Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk
R 579	1	92.7	157.7	43.2	127.3	59.2	72.1	147.6	41.7	111.0	66.8	1.0	2.19	3.1	2.07	0.68
		11.5	12.4	7.8	6.0	10.3	69.8	67.5	13.4	53.5	98.0					
		32.0	31.4	5.5	28.0	83.1										
	2	214.9	128.8	34.8	109.0	37.6	155.6	119.9	34.3	103.7	40.2	1.1	2.55	3.5	1.74	0.64
		22.0	8.4	5.5	6.1	6.3	133.2	47.0	9.7	59.6	62.9					
		48.5	21.6	6.3	30.8	51.8										
	3	212.8	135.3	38.5	145.6	79.9	153.1	127.9	38.7	137.5	81.7	1.1	2.65	4.0	1.93	0.57
		17.6	8.9	5.4	11.1	12.0	129.9	48.2	9.5	71.3	143.5					
		44.0	22.2	7.1	39.4	117.8										
											Mean	1.1	2.46	3.5	1.91	0.63
R 570	1	171.3	156.8	60.8	238.8	86.7	126.9	143.8	59.3	228.5	80.2	1.2	2.67	3.1	2.21	0.63
		13.3	10.9	11.7	13.6	12.0	101.2	53.9	19.1	103.2	126.5					
		31.7	22.8	5.3	58.8	94.0										
	2	117.1	132.7	55.7	164.1	54.6	83.1	122.9	53.4	148.6	51.1	1.1	2.55	3.2	2.34	0.67
		10.0	8.6	9.8	6.4	8.0	73.6	48.2	16.7	63.5	75.8					
		23.3	19.4	5.7	27.7	55.7										
	3	185.8	180.6	54.9	224.1	74.2	137.0	152.2	54.9	182.8	75.0	1.0	2.03	3.7	1.57	0.67
		22.5	15.8	9.2	11.1	10.6	126.6	74.9	14.6	116.1	112.0					
		35.6	28.4	6.0	47.5	78.1										
											Mean	1.1	2.42	3.3	2.04	0.66
M 1557/70	1	133.5	132.6	42.7	212.0	51.2	94.0	117.8	39.6	164.9	52.6	1.0	2.29	2.3	1.47	0.54
		15.3	8.1	8.3	8.8	9.6	93.1	51.5	16.9	112.1	97.8					
		35.1	20.0	5.1	46.6	76.6										
	2	107.0	173.9	43.8	180.1	56.5	74.1	155.0	41.0	142.9	55.8	0.7	2.38	2.8	1.86	0.55
		16.7	13.7	7.8	4.5	12.1	95.7	65.0	14.5	76.9	101.6					
		37.5	23.1	5.6	28.6	78.7										
	3	123.3	138.2	35.0	155.7	46.7	92.3	121.5	32.3	131.9	44.8	0.8	2.22	2.5	1.88	0.56
		15.6	8.8	7.7	5.3	8.4	105.0	54.8	12.9	70.1	80.7					
		48.9	20.0	4.5	27.5	61.4										
											Mean	0.8	2.30	2.5	1.74	0.55
M 1400/86	1	149.9	175.0	60.2	166.0	54.8	101.0	153.6	56.5	135.6	54.8	0.9	2.30	2.8	1.44	0.51
		13.6	16.0	11.9	10.8	12.6	109.5	66.9	19.8	94.0	106.8					
		37.6	26.6	5.1	35.9	79.8										
	2	211.0	136.2	58.4	189.8	62.7	170.7	119.9	54.6	142.5	62.3	1.2	2.24	3.0	1.36	0.53
		21.5	11.5	9.5	9.6	16.8	139.3	53.6	17.7	105.1	117.8					
		43.7	19.9	6.2	35.4	105.8										
	3	117.3	157.4	55.1	165.3	58.6	88.5	137.1	51.9	125.8	58.1	0.9	2.38	3.1	1.39	0.50
		14.6	11.1	9.0	9.1	22.0	89.8	57.5	16.3	90.8	115.2					
		36.0	20.3	6.1	34.5	78.3										
											Mean	1.0	2.31	3.0	1.39	0.52

* The three fractions are > 2 mm, > 1.18 mm and > 38 µm.

+ The two fractions are > 1.18 mm and < 1.18 mm.

Table 3.10. Masses of cane components aged 36 weeks after wet and dry sieving.

Sample		Dry mass of three fractions* after wet sieving/g					Dry mass of two fractions+ after dry sieving/g					Fibre/fines ratio				
		Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk
R 579	1	150.2	91.2	22.1	79.6	41.4	140.0	86.9	30.3	85.3	38.0	1.5	2.92	2.4	2.44	0.60
		27.3	6.1	8.1	7.2	5.7	92.8	29.8	12.6	34.9	63.0					
		41.0	14.4	2.7	24.9	47.2										
	2	91.2	95.2	24.5	67.8	31.5	84.5	84.2	22.6	70.8	30.0	1.4	2.17	1.9	2.38	0.52
		15.2	7.3	7.7	6.1	6.4	58.2	38.8	11.7	29.8	57.7					
		28.8	15.6	3.2	21.3	41.2										

	3	57.6	126.3	23.7	97.2	31.1	53.8	115.8	22.7	97.3	28.3	1.3	2.33	2.4	1.87	0.52
		10.6	5.9	6.2	8.2	3.8	41.4	49.8	9.4	52.0	54.7					
		21.4	24.9	3.8	34.3	43.1										
											Mean	1.4	2.47	2.2	2.23	0.55
R 570	1	129.2	198.8	39.3	113.2	36.8	117.5	183.8	37.7	106.5	41.6	1.8	2.56	2.5	1.71	0.61
		18.4	11.3	9.8	8.8	7.3	62.9	71.8	14.6	62.4	67.7					
		33.9	34.8	4.0	40.1	55.6										
	2	183.5	240.8	38.7	223.0	56.4	172.8	215.2	37.2	220.2	60.4	1.5	2.06	2.3	1.55	0.55
		35.8	16.0	10.8	28.0	15.5	112.6	104.5	16.0	141.8	109.7					
		59.6	48.3	3.6	92.3	84.4										
	3	125.0	163.0	40.2	205.1	44.2	122.5	154.6	38.8	189.1	45.6	1.7	2.46	2.2	1.32	0.48
		24.4	15.3	12.3	20.8	9.4	70.3	62.9	17.5	143.7	94.4					
		36.1	28.5	3.3	82.9	76.4										
											Mean	1.7	2.36	2.3	1.53	0.55
M 1557/70	1	104.4	135.2	33.5	105.6	26.6	99.6	129.2	33.3	97.4	25.6	2.3	2.87	2.2	1.73	0.47
		13.7	6.4	10.9	6.8	6.3	42.6	45.0	14.8	56.2	54.2					
		21.8	22.4	3.1	32.3	41.6										
	2	85.0	144.6	28.8	147.4	28.5	79.7	137.0	28.2	138.8	30.1	2.3	2.22	2.3	1.65	0.43
		11.8	9.5	8.2	12.0	7.3	34.3	61.8	11.8	84.1	69.2					
		18.9	33.3	3.5	48.3	56.5										
	3	58.8	114.6	28.7	109.1	20.6	58.6	107.0	27.7	92.4	21.7	1.7	2.07	2.2	1.72	0.44
		10.3	6.9	8.3	5.6	5.4	33.3	51.8	12.1	53.6	48.9					
		18.7	26.5	3.5	28.5	38.4										
											Mean	2.1	2.38	2.3	1.70	0.45
M 1400/86	1	152.8	171.2	42.6	110.6	30.0	148.0	158.0	39.9	106.2	30.5	2.3	2.24	2.3	1.65	0.43
		22.4	13.0	10.2	8.6	8.9	63.5	70.6	16.8	64.2	70.6					
		34.0	34.9	4.2	39.3	53.5										
	2	88.7	138.2	44.5	160.6	36.1	88.4	130.9	42.8	146.2	38.4	1.7	2.56	2.7	1.46	0.39
		18.2	8.2	9.8	13.6	11.7	51.5	51.2	15.8	100.2	97.9					
		28.7	26.3	4.5	57.3	76.6										
	3	170.4	153.9	35.6	89.2	35.6	145.0	145.5	34.6	79.8	34.6	1.7	2.54	2.8	1.61	0.51
		22.1	10.8	7.7	6.6	7.4	85.5	57.3	12.0	49.6	67.3					
		40.3	29.0	4.6	26.3	48.5										
											Mean	1.9	2.44	2.6	1.57	0.45

* The three fractions are > 2 mm, > 1.18 mm and > 38 µm.

+ The two fractions are > 1.18 mm and < 1.18 mm.

Table 3.11. Masses of cane components of R 570 of three ages harvested in 2001 after fibre extraction and dry sieving.

Sample		Total dry mass after fibre extraction/g					Dry mass of two fractions* after dry sieving/g					Fibre/fines ratio				
		Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk
52 weeks	1	137.8	111.0	91.1	183.8	115.5	62.9	80.7	71.1	113.7	40.8	0.76	2.24	2.45	1.43	0.50
							82.4	36.1	29.0	79.3	82.2					
	2	191.0	102.0	80.2	132.3	92.5	80.3	73.3	63.8	86.0	31.8	0.65	1.98	2.64	1.63	0.48
							122.8	37.0	24.2	52.7	66.7					
	3	143.3	119.2	88.7	103.4	81.7	53.3	88.1	72.8	73.0	26.7	0.53	2.34	2.95	1.96	0.42
							101.1	37.6	24.7	37.3	64.2					
											Mean	**0.65**	**2.19**	**2.68**	**1.67**	**0.46**
44 weeks	1	28.4	174.7	6.4	95.2	109.9	21.2	91.9	4.9	71.0	30.3	2.10	1.59	3.06	3.74	0.63
							10.1	57.8	1.6	19.0	47.9					
	2	27.6	177.0	11.1	122.7	147.0	21.3	112.5	8.9	82.1	35.4	2.45	1.99	3.30	2.70	0.60
							8.7	56.4	2.7	30.4	59.3					
	3	4.4	182.2	6.0	94.5	85.7	2.6	123.5	6.0	59.6	22.4	1.24	2.09	10.00	2.27	0.50
							2.1	59.1	0.6	26.2	45.1					
											Mean	**1.93**	**1.89**	**5.45**	**2.90**	**0.58**
37 weeks	1	112.4	136.2	20.5	120.8	94.2	69.6	93.2	18.2	83.9	40.5	1.39	1.82	4.33	2.01	0.68
							49.9	51.2	4.2	41.7	59.8					
	2	97.8	95.9	14.0	145.5	121.0	57.4	66.2	12.5	97.0	54.8	1.21	1.82	4.31	2.10	0.73
							47.3	36.3	2.9	46.1	74.7					
	3	92.0	173.1	16.2	159.2	121.2	48.8	114.3	14.0	114.5	62.0	0.98	1.69	3.50	2.36	0.83
							49.6	67.5	4.0	48.6	74.6					
											Mean	**1.20**	**1.78**	**4.05**	**2.16**	**0.75**

* The two fractions are > 1.18 mm and < 1.18 mm.

Table 3.12. Material loss (%) from sugarcane component parts after fibre extraction.

Sample		Sample aged 52 weeks					Sample aged 44 weeks					Sample aged 36 weeks				
		Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk	Dry leaf	Green leaf	Top	Rind	Stalk
R 579	1	33.6	76.4	90.3	NA	NA	69.0	81.0	88.9	82.5	95.1	42.5	76.7	90.3	91.8	93.9
	2	38.8	76.2	93.5	NA	NA	56.8	79.1	88.9	85.7	96.1	38.5	77.3	88.2	92.4	95.3
	3	37.3	79.2	93.3	NA	NA	57.8	79.7	87.9	84.4	95.7	44.1	79.1	87.0	80.0	96.0
R 570	1	40.0	77.0	89.3	NA	NA	59.9	77.8	86.4	77.8	95.3	32.8	74.5	86.0	80.9	94.5
	2	46.5	76.9	91.7	NA	NA	58.2	79.7	87.3	78.7	95.2	32.0	74.6	87.7	34.6	96.3
	3	38.4	77.3	94.7	NA	NA	65.2	78.4	87.9	76.4	95.2	36.0	76.2	87.0	47.7	96.8
M 1557/70	1	40.1	74.3	87.8	NA	NA	70.8	79.4	86.6	80.5	95.4	41.7	78.7	88.7	82.0	96.0
	2	47.0	76.2	86.4	NA	NA	70.7	76.6	88.3	76.8	94.3	42.2	78.7	89.1	77.7	95.7
	3	39.8	76.2	94.6	NA	NA	60.9	81.0	87.9	81.5	95.9	37.3	76.1	87.3	80.2	96.3
M 1400/86	1	40.9	77.9	84.6	NA	NA	65.3	75.6	86.2	83.1	95.2	30.3	72.3	85.0	83.7	96.1
	2	33.7	73.1	85.1	NA	NA	56.8	76.7	86.3	80.3	94.7	38.4	77.6	86.0	81.6	95.2
	3	44.6	72.6	88.1	NA	NA	60.0	76.1	85.4	79.7	94.9	33.5	74.8	86.7	80.8	95.8

Table 3.13. Dry mass % cane extracted from sugarcane components.

Sample		Sample aged 52 weeks			Sample aged 44 weeks			Sample aged 36 weeks		
		Rind	Stalk fibre	Stalk pith	Rind	Stalk fibre	Stalk pith	Rind	Stalk fibre	Stalk pith
R 579	1	3.58	2.30	1.85	3.99	1.47	2.31	3.83	1.42	1.81
	2	3.55	1.74	2.05	4.19	1.08	1.67	3.26	1.08	1.63
	3	3.67	1.04	1.86	3.21	1.31	2.13	5.29	1.18	1.78
	Mean	**3.60**	**1.69**	**1.92**	**3.80**	**1.29**	**2.04**	**4.13**	**1.22**	**1.74**
R 570	1	3.06	1.31	1.45	5.67	1.58	1.93	6.09	1.38	2.38
	2	4.69	1.51	1.78	5.81	1.60	1.87	7.15	1.18	2.08
	3	5.10	1.26	1.98	6.20	1.63	1.95	6.71	0.96	1.87
	Mean	**4.28**	**1.36**	**1.73**	**5.89**	**1.60**	**1.92**	**6.65**	**1.17**	**2.11**
M 1557/70	1	5.14	1.44	2.12	6.15	1.18	1.98	5.38	0.99	1.78
	2	4.91	1.78	1.92	6.11	1.62	2.60	6.77	0.93	2.08
	3	4.76	1.65	2.20	4.90	1.21	1.81	5.77	0.83	1.77
	Mean	**4.94**	**1.62**	**2.08**	**5.72**	**1.34**	**2.13**	**5.97**	**0.92**	**1.87**
M 1400/86	1	4.26	1.38	2.39	4.91	1.27	2.13	4.73	0.90	1.86
	2	4.94	1.63	2.36	5.00	1.33	2.61	6.00	0.94	2.29
	3	5.19	1.50	1.96	5.02	1.41	2.41	4.35	1.27	1.99
	Mean	**4.80**	**1.50**	**2.24**	**4.98**	**1.34**	**2.38**	**5.02**	**1.03**	**2.05**

3.5.3 Fibre/pith ratios in cane component parts and in cane

Bernhardt (1998) in characterising the particle properties of bagasse suggested that not only the mean fibre length obtained by sieving with an assembly of sieves of apertures 6.7, 4.0, 2.8, 2.0, 1.4, 0.85, 0.60, 0.425 and 0.30 mm was important, but it was also important to have the coarse to fine ratio of bagasse, which he defined as the mass of particles retained by the sieves with apertures of 2 mm and higher, divided by the mass of particles that pass through the 0.85 mm sieve.

In this work it was found much simpler to separate the fibres into two fractions, namely, one that is retained by a 1.18 mm sieve and one that passes through it. The former is referred to as fibre and the latter as fines in the case of dry leaf, green leaf, top and rind, and as pith in the case of stalk, if the hard fibre is not rendered brittle and breaks into short pieces.

Results of the fibre/fines ratios of dry leaf, green leaf, top, rind and stalk in cane samples are given in Tables 3.8 – 3.11. It can be seen that irrespective of the cane variety and age, stalk has the lowest fibre/pith value, followed by dry leaf, rind, green leaf and top.

By combining the mass of fibre or pith in both rind and stalk, and expressing the hard fibre, pith and total fibre in terms of % cane, some meaningful results, including the fibre/pith ratio in cane were obtained and are presented in Tables 3.14 – 3.16 for the four cane varieties of three ages. The change in these parameters due to the presence of extraneous matter, namely dry leaf, green leaf and tops, was also calculated and presented in Tables 3.14 – 3.16. Again, since the nodes had been excluded from the fibre extraction, the mass of cane was taken to be the mass of stalk plus rind only.

3.5.3.1 *Effect of cane age on fibre and pith contents of cane*

As the cane matures, the hard fibre % cane is expected to increase, so does the total fibre % cane.

Moodley (1991) tested two cane varieties aged 6 months and 19 months; with one variety, the fibre/pith ratio did not vary to a large extent, it was 2.22 and 2.27 for the 6-month and 19-month samples respectively, whereas with the other, it was 2.94 and 3.45 respectively.

Table 3.14. Effect of extraneous matter on fibre/pith ratio in four cane varieties aged 52 weeks.

		R 579				R 570				M 1557/70				M 1400/86			
Replicates and mean		1	2	3	Mean	1	2	3	Mean	1	2	3	Mean	1	2	3	Mean
Initial	Mass of cane/g	5700	5760	2760		4420	3220	4420		7080	2530	5650		4200	5830	3930	
	Hard fibre % cane	4.48	3.67	3.25	**3.80**	3.11	3.57	4.35	**3.68**	4.25	4.58	4.54	**4.46**	3.51	4.52	4.69	**4.24**
	Pith % cane	2.92	3.49	3.13	**3.18**	2.45	4.24	3.85	**3.51**	4.21	3.34	3.74	**3.77**	4.03	4.28	3.76	**4.03**
	Total fibre % cane	7.40	7.15	6.38	**6.98**	5.56	7.80	8.20	**7.19**	8.46	7.93	8.28	**8.22**	7.55	8.80	8.46	**8.27**
	Fibre/pith ratio	1.54	1.05	1.04	**1.21**	1.27	0.84	1.13	**1.08**	1.01	1.37	1.21	**1.20**	0.87	1.05	1.25	**1.06**
Dry leaf	Mass added/g	140	300	180		300	280	180		250	120	320		380	340	140	
	% gross cane	2.40	4.95	6.12	**4.49**	6.36	8.00	3.91	**6.09**	3.41	4.53	5.36	**4.43**	8.30	5.51	3.44	**5.75**
	Hard fibre % cane	5.31	5.30	5.41	**5.34**	5.22	6.03	5.50	**5.58**	5.21	5.74	6.23	**5.73**	6.16	6.39	5.57	**6.04**
	Pith % cane	3.60	4.64	4.49	**4.24**	3.91	5.57	4.87	**4.78**	4.97	4.24	4.93	**4.71**	5.78	5.60	4.47	**5.28**
	Total fibre % cane	8.91	9.94	9.90	**9.59**	9.13	11.59	10.37	**10.36**	10.18	9.98	11.16	**10.44**	11.94	12.00	10.05	**11.33**
	Fibre/pith ratio	1.47	1.14	1.21	**1.27**	1.34	1.08	1.13	**1.18**	1.05	1.35	1.27	**1.22**	1.07	1.14	1.25	**1.15**
Green leaf	Mass added/g	760	820	580		960	800	760		1000	740	1200		500	1030	460	
	% gross cane	11.76	12.46	17.37	**13.86**	17.84	19.90	14.67	**17.47**	12.38	22.63	17.52	**17.51**	10.64	15.01	10.48	**12.04**
	Hard fibre % cane	6.08	5.31	5.04	**5.48**	5.16	6.10	6.02	**5.76**	5.88	7.41	6.72	**6.67**	4.89	6.63	6.23	**5.92**
	Pith % cane	3.53	4.02	3.87	**3.81**	3.64	4.79	4.28	**4.24**	4.57	4.25	4.35	**4.39**	4.18	4.90	4.13	**4.40**
	Total fibre % cane	9.61	9.33	8.91	**9.28**	8.80	10.89	10.30	**10.00**	10.45	11.66	11.08	**11.06**	9.07	11.53	10.36	**10.32**
	Fibre/pith ratio	1.72	1.32	1.30	**1.45**	1.42	1.27	1.40	**1.37**	1.29	1.75	1.54	**1.53**	1.17	1.35	1.51	**1.34**
Tops	Mass added/g	520	700	590		440	820	780		790	530	660		410	460	510	
	% gross cane	8.36	10.84	17.61	**12.27**	9.05	20.30	15.00	**14.78**	10.04	17.32	10.46	**12.61**	8.89	7.31	11.49	**9.23**
	Hard fibre % cane	4.72	3.79	3.53	**4.01**	3.55	4.17	4.27	**4.00**	4.81	5.64	4.47	**4.97**	4.27	5.04	5.21	**4.84**
	Pith % cane	2.77	3.22	2.74	**2.91**	2.35	3.59	3.36	**3.10**	3.95	3.06	3.42	**3.48**	3.83	4.11	3.47	**3.80**
	Total fibre % cane	7.49	7.01	6.27	**6.92**	5.90	7.75	7.62	**7.09**	8.76	8.70	7.90	**8.45**	8.10	9.15	8.68	**8.64**
	Fibre/pith ratio	1.70	1.18	1.29	**1.39**	1.51	1.16	1.27	**1.32**	1.22	1.84	1.31	**1.46**	1.12	1.23	1.50	**1.28**

Table 3.15. Effect of extraneous matter on fibre/pith ratio in four cane varieties aged 44 weeks.

		R 579				R 570				M 1557/70				M 1400/86			
Replicates and mean		1	2	3	Mean	1	2	3	Mean	1	2	3	Mean	1	2	3	Mean
Initial	Mass of cane/g	4040	3480	6100		5490	3410	4560		4350	3490	3850		4330	4700	4160	
	Hard fibre % cane	4.40	4.14	3.59	**4.04**	5.62	5.86	5.65	**5.71**	5.00	5.69	4.59	**5.09**	4.40	4.36	4.42	**4.39**
	Pith % cane	3.75	3.52	3.52	**3.60**	4.18	4.09	5.00	**4.42**	4.83	5.11	3.92	**4.62**	4.64	4.74	4.95	**4.78**
	Total fibre % cane	8.15	7.66	7.11	**7.64**	9.81	9.94	10.66	**10.13**	9.83	10.81	8.51	**9.71**	9.03	9.10	9.37	**9.17**
	Fibre/pith ratio	1.17	1.17	1.02	**1.12**	1.34	1.43	1.13	**1.30**	1.04	1.11	1.17	**1.11**	0.95	0.92	0.89	**0.92**
Dry leaf	Mass added/g	440	660	650		540	360	700		630	550	480		580	640	420	
	% gross cane	9.82	15.94	9.63	**11.80**	8.96	9.55	13.31	**10.60**	12.65	13.61	11.09	**12.45**	11.81	11.99	9.17	**10.99**
	Hard fibre % cane	5.58	7.23	5.52	**6.11**	7.22	7.50	7.51	**7.41**	6.26	6.75	6.21	**6.41**	5.93	7.03	5.95	**6.30**
	Pith % cane	4.94	6.18	5.11	**5.41**	5.49	5.65	6.74	**5.96**	6.08	6.79	5.91	**6.26**	6.32	6.78	6.46	**6.52**
	Total fibre % cane	10.52	13.41	10.62	**11.52**	12.71	13.15	14.25	**13.37**	12.34	13.54	12.12	**12.67**	12.25	13.81	12.41	**12.83**
	Fibre/pith ratio	1.13	1.17	1.08	**1.13**	1.32	1.33	1.11	**1.25**	1.03	0.99	1.05	**1.02**	0.94	1.04	0.92	**0.97**
Green leaf	Mass added/g	1060	760	820		860	790	1040		780	900	880		890	720	790	
	% gross cane	20.78	17.92	11.85	**16.85**	13.54	18.81	18.57	**16.97**	15.20	20.50	18.60	**18.10**	17.05	13.28	15.96	**15.43**
	Hard fibre % cane	6.38	6.22	5.02	**5.87**	7.13	7.68	7.32	**7.38**	6.54	8.06	6.30	**6.97**	6.59	5.99	6.48	**6.36**
	Pith % cane	4.29	4.00	3.80	**4.03**	4.47	4.46	5.41	**4.78**	5.10	5.55	4.35	**5.00**	5.13	5.10	5.32	**5.18**
	Total fibre % cane	10.67	10.22	8.82	**9.90**	11.59	12.15	12.73	**12.16**	11.63	13.60	10.65	**11.96**	11.72	11.09	11.81	**11.54**
	Fibre/pith ratio	1.49	1.56	1.32	**1.45**	1.60	1.72	1.35	**1.56**	1.28	1.45	1.45	**1.40**	1.29	1.17	1.22	**1.23**
Tops	Mass added/g	510	420	420		570	560	580		420	490	390		560	540	480	
	% gross cane	11.21	10.77	6.44	**9.47**	9.41	14.11	11.28	**11.60**	8.81	12.31	9.20	**10.10**	11.45	10.31	10.34	**10.70**
	Hard fibre % cane	4.82	4.57	3.96	**4.45**	6.07	6.38	6.08	**6.18**	5.39	6.02	4.93	**5.45**	5.05	4.95	5.08	**5.03**
	Pith % cane	3.62	3.39	3.44	**3.48**	4.11	3.93	4.72	**4.25**	4.75	4.85	3.86	**4.49**	4.51	4.59	4.79	**4.63**
	Total fibre % cane	8.45	7.96	7.40	**7.93**	10.18	10.30	10.81	**10.43**	10.14	10.87	8.79	**9.94**	9.56	9.54	9.87	**9.66**
	Fibre/pith ratio	1.33	1.35	1.15	**1.28**	1.48	1.62	1.29	**1.46**	1.13	1.24	1.28	**1.22**	1.12	1.08	1.06	**1.09**

Table 3.16. Effect of extraneous matter on fibre/pith ratio in four cane varieties aged 36 weeks.

		R 579				R 570				M 1557/70				M 1400/86			
Replicates and mean		1	2	3	Mean	1	2	3	Mean	1	2	3	Mean	1	2	3	Mean
Initial	Mass of cane/g	2920	2920	2640		2660	4800	4600		2690	3070	2480		3350	3860	2810	
	Hard fibre % cane	4.22	3.45	4.76	**4.14**	5.57	5.85	5.10	**5.51**	4.57	5.50	4.60	**4.89**	4.08	4.78	4.07	**4.31**
	Pith % cane	3.35	3.00	4.04	**3.46**	4.89	5.24	5.18	**5.10**	4.10	4.99	4.13	**4.41**	4.02	5.13	4.16	**4.44**
	Total fibre % cane	7.58	6.45	8.80	**7.61**	10.46	11.09	10.28	**10.61**	8.68	10.50	8.73	**9.30**	8.10	9.91	8.23	**8.75**
	Fibre/pith ratio	1.26	1.15	1.18	**1.20**	1.14	1.12	0.99	**1.08**	1.11	1.10	1.11	**1.11**	1.01	0.93	0.98	**0.97**
Dry leaf	Mass added/g	380	220	160		270	410	290		240	200	140		300	220	350	
	% gross cane	11.52	7.01	5.71	**8.08**	9.22	7.87	5.93	**7.67**	8.19	6.12	5.34	**6.55**	8.22	5.39	11.08	**8.23**
	Hard fibre % cane	7.98	5.90	6.41	**6.76**	9.06	8.70	7.30	**8.36**	7.60	7.60	6.59	**7.26**	7.80	6.69	8.21	**7.57**
	Pith % cane	5.78	4.64	5.29	**5.24**	6.59	6.99	6.31	**6.63**	5.22	5.74	5.18	**5.38**	5.43	6.12	6.41	**5.99**
	Total fibre % cane	13.76	10.54	11.70	**12.00**	15.65	15.69	13.61	**14.98**	12.82	13.34	11.77	**12.64**	13.23	12.81	14.61	**13.55**
	Fibre/pith ratio	1.38	1.27	1.21	**1.29**	1.38	1.25	1.16	**1.26**	1.45	1.33	1.27	**1.35**	1.44	1.09	1.28	**1.27**
Green leaf	Mass added/g	480	520	750		960	1200	870		770	880	620		790	770	770	
	% gross cane	14.12	15.12	22.12	**17.12**	26.52	20.00	15.90	**20.81**	22.25	22.28	20.00	**21.51**	19.08	16.63	21.51	**19.07**
	Hard fibre % cane	6.18	5.38	7.12	**6.23**	9.17	8.26	7.12	**8.18**	7.29	7.74	7.13	**7.39**	7.12	6.81	7.26	**7.06**
	Pith % cane	3.76	3.67	4.62	**4.01**	5.58	5.93	5.50	**5.67**	4.49	5.45	4.98	**4.97**	4.96	5.38	4.87	**5.07**
	Total fibre % cane	9.94	9.05	11.74	**10.24**	14.75	14.20	12.62	**13.85**	11.78	13.19	12.11	**12.36**	12.08	12.20	12.13	**12.13**
	Fibre/pith ratio	1.65	1.46	1.54	**1.55**	1.64	1.39	1.29	**1.44**	1.62	1.42	1.43	**1.49**	1.43	1.27	1.49	**1.40**
Tops	Mass added/g	340	300	260		380	430	430		420	370	320		380	420	360	
	% gross cane	10.43	9.32	8.97	**9.57**	12.50	8.22	8.55	**9.76**	13.50	10.76	11.43	**11.90**	10.19	9.81	11.36	**10.45**
	Hard fibre % cane	4.92	3.98	5.39	**4.77**	6.57	6.38	5.72	**6.22**	5.52	6.08	5.37	**5.66**	5.00	5.61	5.03	**5.22**
	Pith % cane	3.54	3.20	4.22	**3.65**	5.11	5.37	5.35	**5.28**	4.42	5.10	4.34	**4.62**	4.29	5.28	4.35	**4.64**
	Total fibre % cane	8.46	7.18	9.61	**8.42**	11.68	11.75	11.07	**11.50**	9.95	11.18	9.71	**10.28**	9.30	10.90	9.39	**9.86**
	Fibre/pith ratio	1.39	1.24	1.28	**1.30**	1.28	1.19	1.07	**1.18**	1.25	1.19	1.24	**1.23**	1.16	1.06	1.16	**1.13**

Snow (1974) investigated the seasonal variations in the ratio of hard fibre to pith within a cane variety at regular monthly intervals for 7 through 12-month old cane. Young or immature cane had more pith than hard fibre with a fibre/pith ratio of about 0.58 at 7 months. As the cane reached maturity (9 months) more hard fibre was present in the stalks giving approximately equal amounts of hard fibre and pith. This fibre/pith ratio of 1 remained constant for the duration of the season.

From Tables 3.14 – 3.16 for the four cane varieties, the pith % cane increases in all cases (except R 570) as the cane matures from 36 to 44 weeks, but decreases (except M 1400/86) from 36 to 52 weeks. However, there is no indication of an increase of hard fibre % cane with age in any of the four cane varieties. In fact, the value found for the total fibre % cane was on the low side. This can be attributed to the fact that the samples examined had the nodes eliminated, some probably had more stalk and rind removed in the process as in the case of cane aged 52 weeks. However, R 570, M 1557/70 and M 1400/86 did show an increase in hard fibre % cane from 36 weeks to 44 weeks. In spite of this shortcoming, the fibre/pith value in cane calculated can still be considered as reliable since both Moodley (1991) and Snow (1974) had found that fibre/pith ratio remained more or less constant.

3.5.3.2 *Effect of cane variety on fibre/pith ratio in cane*

From Table 3.14, for the mature cane aged 52 weeks, the fibre/pith ratios of R 570 and M 1400/86 were close to the expected value of one with values of 1.08 and 1.06 respectively, whereas those of R 579 and M 1557/70 were high, at about 1.20. According to Snow (1974), cane with high ratio of hard fibre to pith e.g. 1.52, causes problems in milling due to the toughness of the stalks entailing excessive maintenance of the equipment due to stress and, mill chokes induced by bagasse jamming in the radial and juice grooves, whereas cane varieties with more pith than hard fibre, with a fibre/pith ratio of about 0.6, cause an overall slowdown in mill operations manifested through occasional reduced grinding rates due to the soft “mushy” consistency of the macerated material leading to mill roll slippage or chokes, and high residual moisture content of the bagasse. Varieties with approximately equal amounts of hard fibre and pith proved to be the best milling varieties. Hence the varieties investigated here should be considered good milling varieties if judged on their fibre/pith ratios.

Snow (1974) tested eight cane varieties of 11-month maturity, where the fibre/pith ratio varied from 0.66 to 1.52 and fibre % cane (presumably total fibre % cane) ranged from 9.0 to 12.2.

Moodley (1991) tested four different varieties of cane aged 19 months and found that although the fibre/pith ratio did vary from 2.00 to 2.86, there was not much difference in pith % cane, but the hard fibre % cane did vary over a wide range from 8.85 to 12.86.

In this study, with the four cane varieties aged 52 weeks, fibre/pith ratio in cane ranged from 1.06 to 1.21, in agreement with values found by Snow (1974). Hard fibre % cane varied from 3.68 to 4.46, pith % cane from 3.18 to 4.03, and fibre % cane from 6.98 to 8.27. As mentioned earlier, the fibre % cane values appear to be low, and so do those of hard fibre % cane.

3.5.3.3 *Effect of extraneous matter on fibre/pith ratio in cane*

Moodley (1991) found that, by adding 12% by mass of cane tops to clean cane, hard fibre % cane is greatly increased from 8.78 to 10.99, and the pith % cane changes from 4.03 to 3.16 giving a fibre/pith ratio of 3.45, whereas with the addition of 12% by mass of dry leaves, hard fibre % cane was greatly increased to 14.06 and the pith % cane to 4.11 giving a fibre/pith ratio of 3.45 also. He also found that drought conditions affect fibre/pith ratio, normally it is 60:40, whereas during drought it changes to 40:60.

In this study, by using data from Tables 3.8 – 3.10, the mass of stalk plus rind can be considered as clean cane, the effect of the presence of dry leaf in gross cane (clean cane + dry leaf) on hard fibre % cane, pith % cane, total fibre % cane and fibre/pith ratio in cane was evaluated and shown in Tables 3.14 – 3.16 for the four cane varieties at three ages. The first fraction > 1.18 mm collected after dry sieving was considered to be the hard fibre and the second fraction < 1.18 mm, the fines or pith. Similarly, the effect of green leaf and tops on the above parameters was also assessed. It can be seen that all the extraneous matter studied increases the fibre/pith ratio in cane (except dry leaf when added to 44 weeks old cane), hard fibre % cane, pith % cane and total fibre in cane.

3.5.3.4 *Effect of extraneous matter on fibre % cane*

From Tables 3.14 – 3.16, it is evident that the addition of dry leaf and green leaf, but not cane tops, to clean cane increases fibre % cane. These increases in fibre % cane due to the presence of dry leaf and green leaf are calculated and shown in Table 3.17 for the four cane varieties of three ages. These calculated increases compare favourably with the prediction

from Figure 2.9 that the change in fibre % cane is 0.572, 0.132 and –0.001 due to one unit of dry leaf, green leaf and cane tops respectively; except in the case of dry leaf addition to 44 weeks old cane.

3.5.3.5 *Fibre % cane results by direct cane analysis*

It has been pointed out that the fibre % cane calculated from the mass of the extracted fibres appear to be on the low side. This is confirmed by the fibre % cane results obtained by direct cane analysis (Anon., 1991) performed on parallel samples and shown in Table 3.18. For the samples aged 52 weeks, the calculated fibre % cane values are 2 – 4 units lower than the analytical results, whereas for the samples aged 44 and 36 weeks, only about one unit (except the M 1557/60 aged 44 weeks) difference was found.

3.5.4 Characterisation of sugar cane component parts

The components separated from the sugar cane plant were characterised by measuring their gross calorific values and by investigating their structure and morphology by means of infra-red spectroscopy and scanning electron microscopy, respectively.

3.5.4.1 *Gross calorific value*

Snow (1974) also mentioned that with a high pith content in cane, extensive problems would be encountered by the high residual moisture content of bagasse due to the fact that pith can easily pick up atmospheric moisture and absorb more water during cane processing than fibre, entailing additional expenses through increased consumption of auxiliary fuel oil to burn the bagasse.

The results of the determination of the calorific value of the various cane components are shown in Table 3.19; it is evident that while extracted rind has the highest GCV of 19 443 kJ kg^{-1}, followed by stalk fibre (19 041 kJ kg^{-1}) and dry leaf (18 268 kJ kg^{-1}); stalk pith has the lowest GCV (17 512 kJ kg^{-1}) of about 8% less than stalk fibre. The stalk fibres of R 579 and R 570 appear to have higher GCV than those of the other two cane varieties.

Table 3.17. Effect of extraneous matter on fibre % cane in four cane varieties of three ages (using data in Tables 3.8.-3.10).

			R 579			R 570			M 1557/70			M 1400/86		
Replicates and mean			1	2	3	1	2	3	1	2	3	1	2	3
52 weeks	Initial	Fibre % cane	7.40	7.15	6.38	5.56	7.80	8.20	8.46	7.93	8.28	7.55	8.80	8.46
	Dry leaf	% gross cane	2.40	4.95	6.12	6.36	8.00	3.91	3.41	4.53	5.36	8.30	5.51	3.44
		Total fibre % cane	8.91	9.94	9.90	9.13	11.59	10.37	10.18	9.98	11.16	11.94	12.00	10.05
		Increase	1.51	2.79	3.52	3.57	3.79	2.17	1.72	2.05	2.88	4.39	3.20	1.59
		Prediction*	1.37	2.83	3.50	3.64	4.58	2.24	1.95	2.59	3.07	4.75	3.15	1.97
	Green leaf	% gross cane	11.76	12.46	17.37	17.84	19.90	14.67	12.38	22.63	17.52	10.64	15.01	10.48
		Total fibre % cane	9.61	9.33	8.91	8.80	10.89	10.30	10.45	11.66	11.08	9.07	11.53	10.36
		Increase	2.21	2.17	2.53	3.24	3.08	2.10	1.99	3.73	2.79	1.52	2.73	1.91
		Prediction*	1.55	1.64	2.29	2.36	2.63	1.94	1.63	2.99	2.31	1.40	1.98	1.38
44 weeks	Initial	Fibre % cane	8.15	7.66	7.11	9.81	9.94	10.66	9.83	10.81	8.51	9.03	9.10	9.37
	Dry leaf	% gross cane	9.82	15.94	9.63	8.96	9.55	13.31	12.65	13.61	11.09	11.81	11.99	9.17
		Total fibre % cane	10.52	13.41	10.62	12.71	13.15	14.25	12.34	13.54	12.12	12.25	13.81	12.41
		Increase	2.37	5.76	3.51	2.90	3.21	3.59	2.51	2.73	3.61	3.22	4.71	3.03
		Prediction*	5.62	9.12	5.51	5.12	5.46	7.61	7.24	7.79	6.34	6.76	6.86	5.25
	Green leaf	% gross cane	20.78	17.92	11.85	13.54	18.81	18.57	15.20	20.50	18.60	17.05	13.28	15.96
		Total fibre % cane	10.67	10.22	8.82	11.59	12.15	12.73	11.63	13.60	10.65	11.72	11.09	11.81
		Increase	2.52	2.56	1.70	1.79	2.20	2.08	1.81	2.80	2.14	2.68	1.99	2.44
		Prediction*	2.74	2.37	1.56	1.79	2.48	2.45	2.01	2.71	2.46	2.25	1.75	2.11
36 weeks	Initial	Fibre % cane	7.58	6.45	8.80	10.46	11.09	10.28	8.68	10.50	8.73	8.10	9.91	8.23
	Dry leaf	% gross cane	11.52	7.01	5.71	9.22	7.87	5.93	8.19	6.12	5.34	8.22	5.39	11.08
		Total fibre % cane	13.76	10.54	11.70	15.65	15.69	13.61	12.82	13.34	11.77	13.23	12.81	14.61
		Increase	6.18	4.09	2.90	5.19	4.61	3.33	4.14	2.84	3.04	5.13	2.89	6.38
		Prediction*	6.59	4.01	3.27	5.27	4.50	3.39	4.69	3.50	3.06	4.70	3.08	6.34
	Green leaf	% gross cane	14.12	15.12	22.12	26.52	20.00	15.90	22.25	22.28	20.00	19.08	16.63	21.51
		Total fibre % cane	9.94	9.05	11.74	14.75	14.20	12.62	11.78	13.19	12.11	12.08	12.20	12.13
		Increase	2.36	2.60	2.94	4.29	3.11	2.34	3.10	2.69	3.38	3.98	2.28	3.89
		Prediction*	1.86	2.00	2.92	3.50	2.64	2.10	2.94	2.94	2.64	2.52	2.20	2.84

* Values predicted from the linear equation given in Fig 2.9.

Table 3.18. Fibre % cane results by direct cane analysis.

	R 579				R 570				M 1557/70				M 1400/86			
Replicates and mean	**1**	**2**	**3**	**Mean**	**1**	**2**	**3**	**Mean**	**1**	**2**	**3**	**Mean**	**1**	**2**	**3**	**Mean**
Sample age/weeks 52	10.12	10.15	10.15	**10.14**	10.12	13.53	10.67	**11.44**	11.28	11.73	10.09	**11.03**	10.70	10.43	11.25	**10.79**
44	8.33	10.40	6.81	**8.51**	10.82	10.85	9.21	**10.29**	11.98	12.43	11.82	**12.08**	8.97	9.70	8.81	**9.16**
36	8.45	10.00	9.67	**9.37**	12.58	11.34	11.64	**11.85**	10.27	10.82	6.72	**9.27**	10.33	11.40	9.24	**10.32**

Table 3.19. Gross calorific value of sugarcane components.

		GCV of dried sample /kJ kg^{-1}			
Sample		Rind	Stalk fibre	Stalk pith	Dry leaf
R 579	1	19243	19233	17975	18112
	2	19311	19478	17988	18260
	3	19168	19521	17819	18262
	Mean	19241	19411	17927	18211
R 570	1	19726	19552	17380	19216
	2	20026	19258	17356	19126
	3	19903	19095	17218	18912
	Mean	19885	19302	17318	19085
M 1557/70	1	19204	19098	17000	17703
	2	19325	18811	17130	17853
	3	19080	19025	17129	17646
	Mean	19203	18978	17086	17734
M 1400/86	1	19382	18323	17714	17912
	2	19339	18641	17802	18080
	3	19610	18452	17633	18134
	Mean	19444	18472	17716	18042
	Mean	19443	19041	17512	18268

3.5.4.2 *Infrared spectroscopy and scanning electron microscopy*

No differences were observed in the infrared spectra of the various cane component parts. This indicates that all the components are composed of essentially the same constituents. Typical Fourier transform infrared (FTIR) spectra of stalk fibre, dried in an oven at 70 °C for 0.5, 1.0, 1.5, 2.0, 2.5, 3.0 and 48 hours, before preparation into a KBr disc (2 mg sample + 200 mg KBr) are shown in Fig 3.23 (bottom to top spectrum).

Sun *et al.* (2004) attributed the absorption bands in the FTIR spectrum of sugar cane bagasse as follows: the adsorption band at 3420 cm^{-1} is due to the stretching of C-H groups, that of 2900 cm^{-1} to the C-H stretching, and that at 1745 cm^{-1} is assigned as the C=O ester band. The band at 1639 cm^{-1} is attributed to the bending mode of the adsorbed water, that at 1428 cm^{-1} as CH_2 bending and that at 1328 cm^{-1} as from C-C and C-O skeletal vibrations. The C-O-C pyranose ring skeletal vibrations occurs in the region 1076-1023 cm^{-1}. The peak at 903 cm^{-1} originates from β-glycosidic linkages between glucose units in cellulose. In their study of accelerated ageing of paper under controlled conditions, Qojewska *et al.* (2005) also pointed out that the bending vibration band at 1640 cm^{-1} in the FTIR spectrum is a good indicator of bound water. From Fig 3.23, the presence of this peak is well pronounced, and could perhaps provide another way of quantitatively determining the bound water in fibres.

All the electron scanning micrographs showed normal wood tissues such as parenchyma and wood fibres, and no difference was detected in the fibres of dry leaf, green leaf, rind, stalk and top (Fig 3.24a-f). Stalk pith (Fig 3.24f) appears to be different, it is more flaky and has higher surface area than other components.

3.6 CONCLUSIONS

The sugar cane plant of four cane varieties and of three ages has been successfully separated into fibres of its component parts by means of a simple method specially developed for the purpose. The use of a 1.18 mm sieve ensures complete separation of fibres from pith, of which the ratio gives an indication of the milling quality of the cane variety.

The four cane varieties under study show fibre/pith ratio approaching to one, indicating good millability. The addition of extraneous matter to clean cane increases the fibre % cane to the same extent as predicted by the linear equation presented in Fig 2.9 of Chapter 2.

The Brix-free water of the cane component parts will be discussed in Chapter 4 and their sorption properties in Chapters 5 and 6.

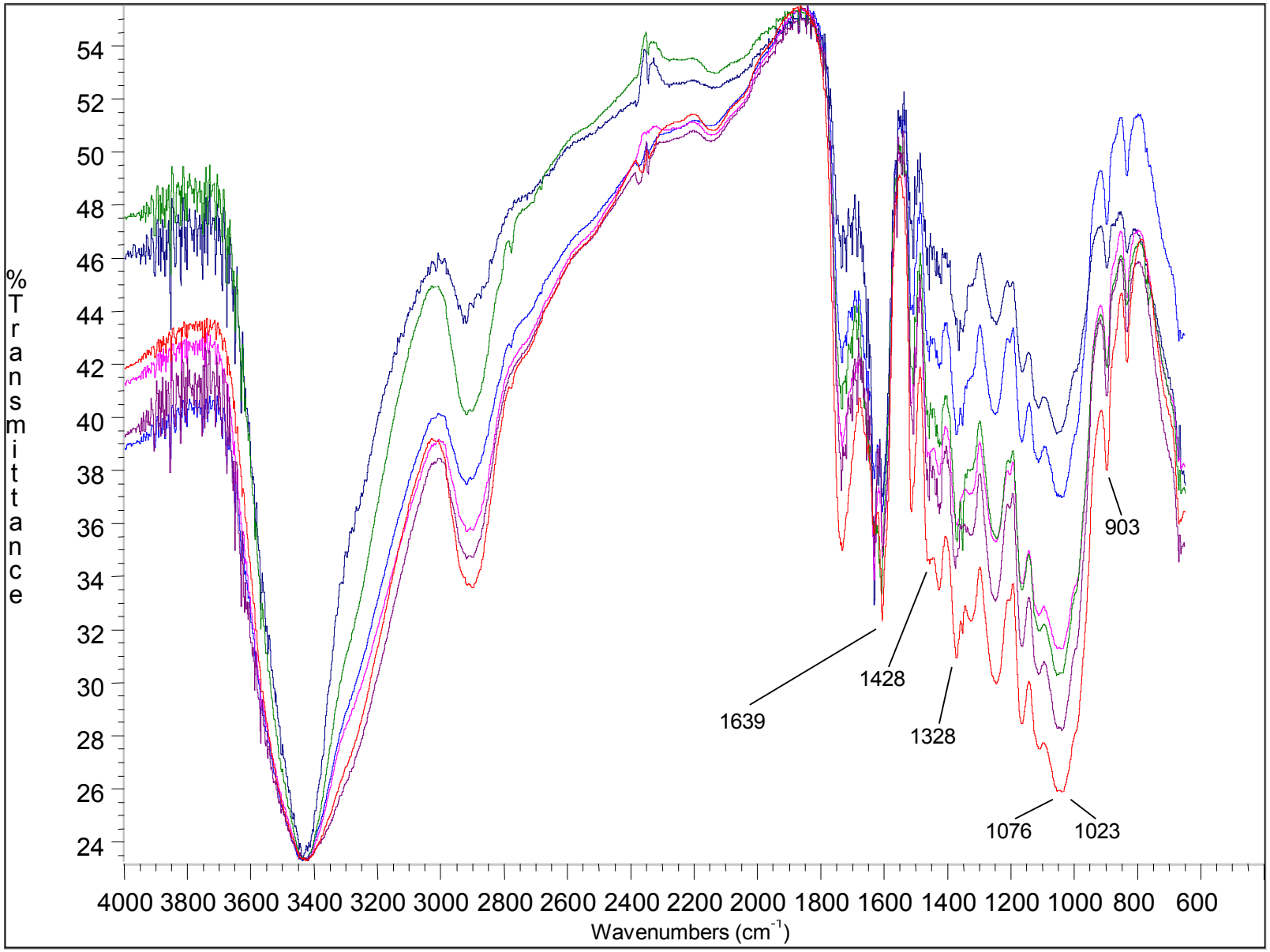
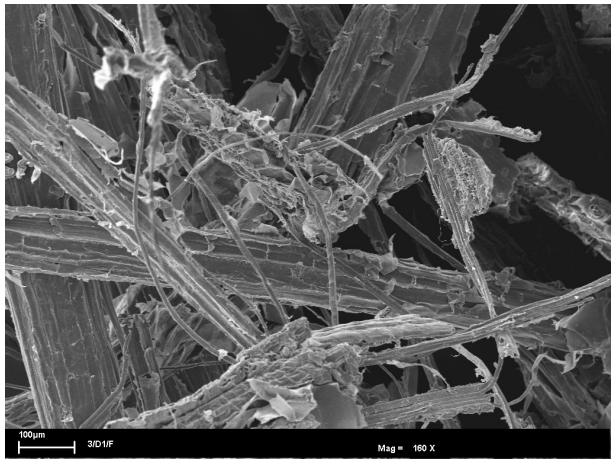
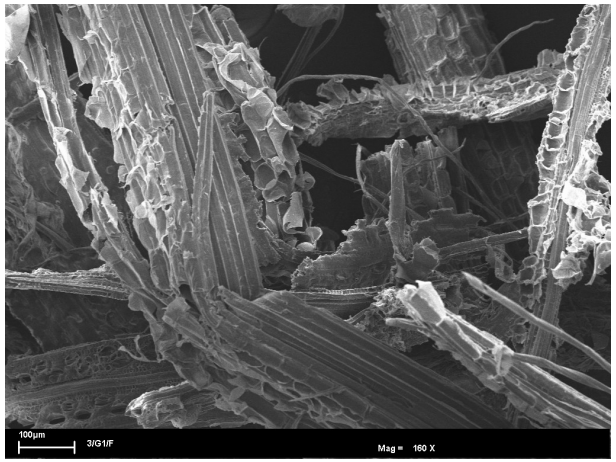


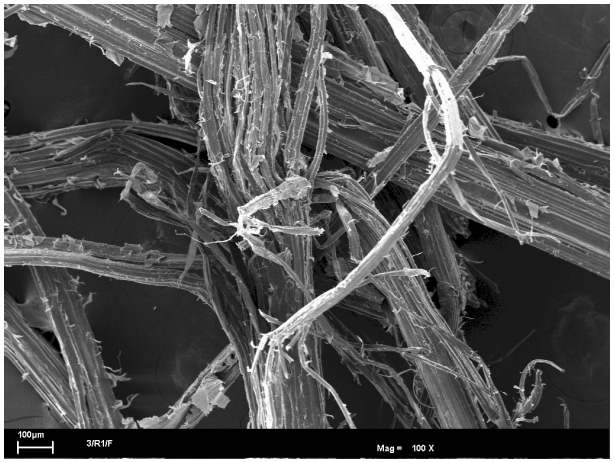
Figure 3.23. FTIR spectra of stalk fibre dried in oven at 70 °C for various lengths of time.



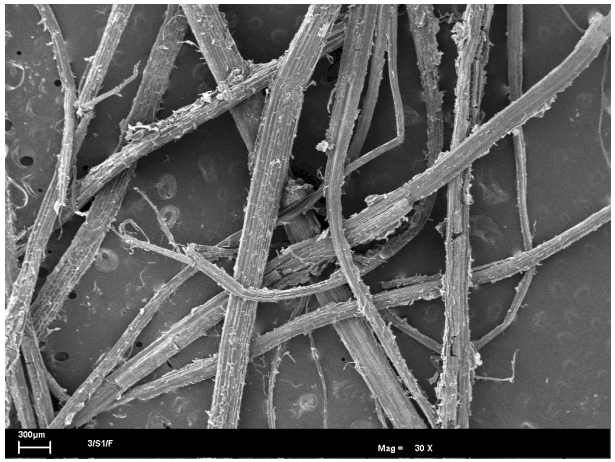
(a). Dry leaf



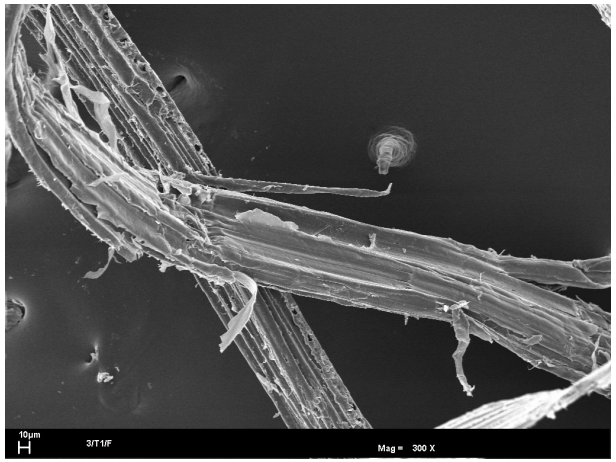
(b). Green leaf



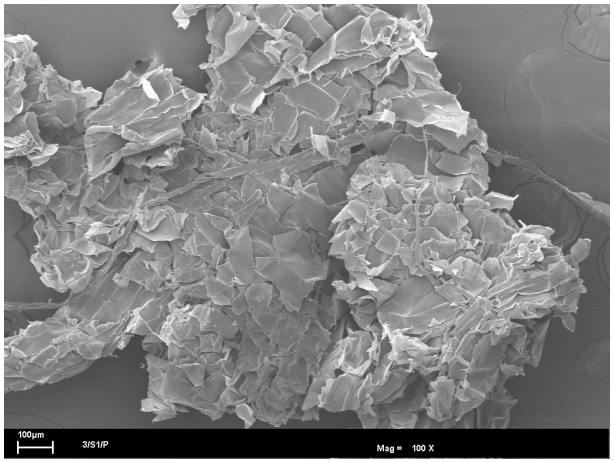
(c). Rind



(d). Stalk



(e). Top



(f). Stalk pith

Figure 3.24. Fibres of sugar cane components observed under a scanning electron microscope.

CHAPTER 4. BRIX-FREE WATER IN CANE FIBRES

As mentioned in Chapter 2 (Section 2.2), natural fibres have associated water, which is separate from the water in the juice and which cannot be removed by mechanical means. This water is generally termed Brix-free water or hydrated water in South Africa, and adsorption water or hygroscopic water in Australia. Brix-free water is present in varying amounts for a particular substance depending on the vapour pressure of water in the atmosphere and the temperature. Prinsen Geerligs (1897) and Steuerwald (1912) studied the change in concentration of solutes dissolved in water brought about by the addition of fibre to the solution. Previous determinations of Brix-free water in cane reported by Steuerwald (1912), Foster (1962, 1963) and Richardson (1970) gave values ranging from 10% to 50% on fibre.

4.1 THE SIGNIFICANCE OF BRIX-FREE WATER IN CANE

The existence of Brix-free water in cane and its unavailability for the dissolution of sucrose affect milling control and direct cane (or bagasse) analyses. Brix-free water in extraneous matter, and particularly in dry trash, when present with cane, can inflate the press juice analytes.

4.1.1 Effect of Brix-free water in cane on milling control

Brix-free water is characterised as water strongly adsorbed onto the cane fibre and unavailable for the solution of the soluble components present in sugar cane. As a result, it is incorrect to assume that the mass of juice that can be extracted from cane can be obtained by deducting the mass of fibre as found by drying, from the mass of the cane sample from which the juice is extracted. The mass of fibre plus Brix-free water should be subtracted:

Mass of juice = mass of cane - mass of fibre - mass of Brix-free water

This juice is termed undiluted (or normal) juice and is the juice expressed by the mills or retained in the bagasse corrected for Brix-free water. For purposes of calculation, it has the Brix of the primary juice. The concept of Brix-free water expressed as a percentage of

dry fibre, or a factor, is used in assessing the accuracy of the various variables used in milling control, to correct the Brix of primary juice to that of juice in cane and so bridge the gap in the mass balance.

To calculate the Brix of the undiluted juice from the Brix of the first expressed juice, a 'dry milling factor' can be used. This is obtained by periodically operating the mills briefly without imbibition water, determining the Brix of the first expressed juice and of the mixed juice so obtained. For example, if they are 20.0 and 19.4 respectively, the dry milling factor is 19.4 divided by 20.0 = 0.97. It is then assumed that this factor is the relationship between the first expressed juice and the undiluted juice when imbibition water is used, and under these conditions, mixed juice is of much lower density. For example, if the Brix of the first expressed juice is 19.21 in regular milling, the undiluted juice Brix will be 19.21 times 0.97 = 18.63 (Chen and Chou, 1993).

4.1.2 Effect of Brix-free water in cane on direct cane (or bagasse) analyses

Because of the unavailability of the Brix-free water for the solution of the soluble components present in sugar cane, methods utilising the wet disintegrator technique, e.g. pol or Brix in cane (or bagasse), produce higher pol or Brix results than would be observed if all the water present in cane fibre were available for solution of soluble substances. Hence, in the direct cane analysis of cane (or bagasse) by liquid extraction, practised in Mauritius, South Africa and all over the world, the concept of Brix-free water should be taken into consideration in calculations.

4.1.3 Effect of Brix-free water in dry trash on cane quality

It emerges from Chapter 2 (Section 2.2.3) that when the moisture content of dry trash in particular is below a certain critical value (Brix-free water content), the press juice obtained in contact with dry trash has a higher concentration in sucrose, Brix and pol. It was surmised that this occurs because the trash absorbs water in preference to juice during pressing to satisfy its Brix-free water capacity. This implies that if cane quality is assessed by the above parameters, the results will be over-estimated.

4.2 METHODS OF DETERMINATION OF BRIX-FREE WATER IN CANE FIBRES

Published literature shows that Brix-free water of sugar cane fibre may be determined by two main methods:

1) a contacting method where dry sugar-free cane fibre is placed in a sucrose (or any suitable compound) solution of known concentration, is allowed to equilibrate and the hygroscopic water calculated from the change in sucrose concentration. The concentration change needs to be determined with great accuracy and it is uncertain whether the fibre is absolutely dry, due to the extremely hygroscopic nature of cellulose.

2) a vapour sorption method, where water is sorbed by dry sugar-free cane fibre from an atmosphere of controlled relative humidity. This method requires that the experiment be carried out at constant temperature. Since in the contacting method, a small amount of sucrose may be sorbed by the fibre in addition to the water, this method measures the differential sorption of water over sucrose (Kelly and Rutherford, 1957). According to Downing and McBain (2000), the difference between the two methods is somewhat indistinct. The two methods may differ due to the contribution of capillary condensation; however, condensation of water in preformed spaces within dry fibres is not expected to be more than 2 – 3% by mass on cellulose (Valko, 1943).

As early as the end of the 19th century, Prinsen Geerligs (1897) described experiments designed to determine directly Brix-free water, which he called colloidal water in fibre. He added known masses of completely washed bagasse, of known moisture content to solutions of sodium chloride of a known concentration. After a mixing time of 24 hours, the salt concentration was re-determined. From the increase in salt concentration, he was able to calculate the Brix-free water associated with the fibre by subtracting the amount of water in which the salt was dissolved from the total amount of water in the system. He obtained a value of 35% Brix-free water in bagasse. It was evident that absorption of sodium chloride on the fibre also took place, especially at high salt concentrations, leading to negative results at times. With sucrose solutions, he found the value of 20% Brix-free water on fibre.

Steuerwald (1912) applied sucrose solutions instead of salt solutions, and used two methods to measure Brix-free water: a contact method and a press method. In the former, a known mass of sucrose solution was allowed to remain in contact with a known mass of fibre, the increase in concentration of the sucrose solution was a measure of the amount of water adsorbed into the fibre. He obtained an average value of 22% for Brix-free water in fibre, with a range from 15 to 28%. The latter method consisted of adding pure sugar solution to cane fibre; after pressing, a residue was obtained; by analysing its sugar content and the total water present, the adsorbed water could be measured. From the mass of sugar in the residue and the composition of the expressed juice, the respective masses of sugar solution and adsorbed water could be calculated. With the press method, the value decreased with increasing pressure until he obtained 16.5% at 300 atmospheres.

Spoelstra (1935) repeated these experiments slightly differently, in that he added completely dry fibre to salt solutions and determined the increase in concentration of sodium chloride after equilibrium was obtained. He found Brix-free water values between 26 and 30%.

Van der Pol *et al.* (1957) pointed out that in the original Prinsen Geerligs method, the use of solutions of an electrolyte presented conditions far remote from conditions existing in a cane stalk, and also that the use of high temperatures in either extracting the sucrose from the fibre or drying the fibre, introduced an uncertainty due to possible irreversible chemical reactions which could affect the affinity of the fibre for water. They dried fibre in vacuum over phosphorus pentoxide, P_2O_5, at a temperature lower than 60 °C, and then used sucrose solutions to contact the fibre. After equilibration, the sucrose concentration was determined with a saccharimeter. They found that at high Brix (60°), the amount of water absorbed by the dry fibre was less than at lower Brix. The same phenomenon had been observed by Prinsen Geerligs who attributed it to the absorption of sucrose by the fibre. Since fibre which had been dried in vacuum over P_2O_5 at 60 °C still contained 5% moisture whereas if it had been dried in oven at 105 °C-110 °C, the residual moisture would be about 0.5%. Van der Pol *et al.* (1957) corrected their experiments for a residual moisture content of 5%. They found that by using a 20% sucrose solution, the average Brix-free water content was 29%.

Kelly and Rutherford (1957) also repeated the experiments of Prinsen Geerligs. After drying fibre at 120 °C for 5 hours, they put the fibre into various solutions, containing known concentrations of sucrose, glycerol, sodium chloride and potassium chloride. They

found that the amount of absorbed water on the fibre was dependent on the cane variety and the concentration of the solution used. They also did an experiment with pure cellulose in a 20% sucrose solution and found a value of 8%.

Foster (1963) used a different method to determine Brix-free water in cane fibre. Cane (2 kg) was weighed into a wet disintegrator, and reduced to a slurry with water (6 kg). A sample of the extract was taken for Brix reading. The extract was then removed until the slurry mass was 4000 g. To this was added an exact mass of 1500 g of 67.00° Brix sucrose solution. After five minutes of further mixing in the disintegrator, the resultant Brix was measured, and the actual mass of solvent plus Brix could be calculated. Subtracting this mass from 4000 g, gave the mass of fibre plus Brix-free water. He obtained values of 25 to 40% Brix-free water on fibre.

Mangion and Player (1991) ensured that all the adsorbed water was removed from a pre-washed, essentially sucrose-free fibre sample, and then contacted this fibre with a sucrose solution of known concentration. They employed a method that used eight grams of fibre sample weighed into a pre-weighed jar. The fibre was vacuum dried at 80 °C, and 825 mbar for 3 hours to avoid any heat damage of the fibre prior to analysis. After which, the sample mass was determined, and 150 g of a 10° Brix sucrose solution was added and well mixed to ensure all fibre was wetted by the sucrose solution. Mixing was effected from time to time for one and a half hours. The sample was then filtered through a covered Whatman 91 filter paper prior to the Brix measurement. The Brix of the original sucrose solution was also measured. The Brix-free water of the fibre was calculated by means of the following equation:

$$\% \text{ Brix-free water} = [100\, w_4(1 - p_3\, p_4^{-1})]/w_3$$

where w_3 and w_4 are the mass of fibre sample and the mass of sucrose contact solution respectively, and p_3 and p_4 are the Brix of the sucrose solution before and after mixing with the fibre sample.

Mangion and Player (1991) found from over 250 data points, an average of 20.6% Brix-free water in present day cane varieties with a standard deviation of 2.2 units.

Qin and White (1991) adopted the same method and reasoned that with unit mass of fibre added to 20 times its mass of sucrose solution, if the Brix-free water was 20% and the initial sucrose solution was 10° Brix, one could only expect an increase of 0.1 unit Brix, which was a small change and very difficult to measure with any accuracy. If the contact

solution/fibre ratio was lowered from 20 to 3, this would give a much larger change in Brix from 10.0 to about 10.7 enhancing the accuracy of the measurement. However, with such a low contact solution/fibre ratio, the fibre would not be fully wetted. Hence, a hydraulic press (see Fig 4.1), was devised to repeatedly compress the fibre so as to ensure good mixing of the contacting solution.

The press is a modification of a hydraulic press unit used to obtain pressure-filtered molasses samples from massecuites. The bottom of the cylinder was sealed, and the close-fitting internal piston was replaced by a loose open-holed piston, which compressed the fibre but allowed the press juice to flow out. The piston was moved by using a hydraulic car jack. The apparatus was used in a controlled humidity cabinet.

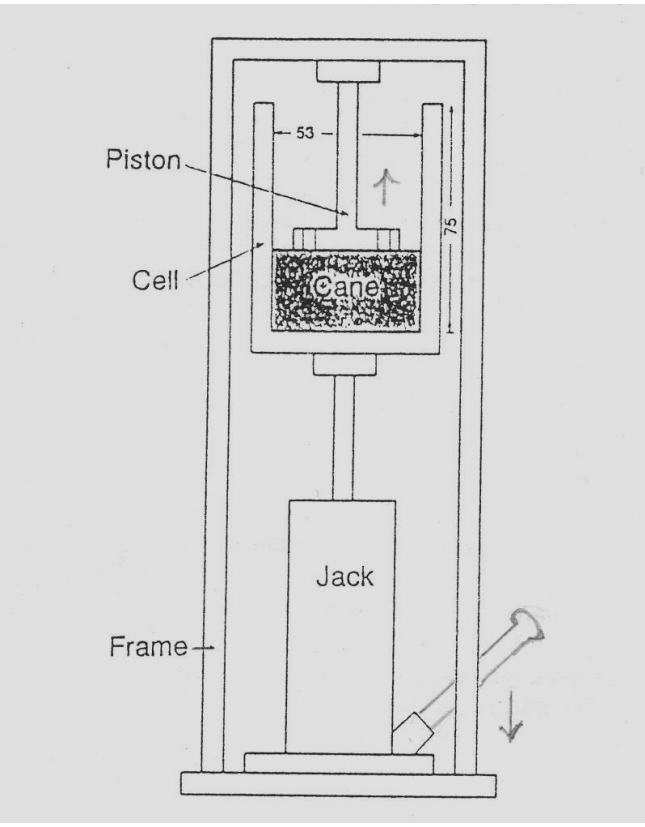


Figure 4.1. Diagram of press cell used by Qin and White (1991) for Brix-free water measurements.

Qin and White (1991) dried well-washed and sieved bagasse at 60 °C in an atmospheric oven for 8 hours and stored it in a desiccator. A weighed amount (8 g) of dry washed fibre was transferred to the cylinder of the hydraulic press (see Fig 4.1) and a weighed mass (24 g to 48 g) of sucrose solution of known Brix was added. The fibre was then compressed by the press causing liquid to appear through the open cylinder. When the hydraulic pressure was released, the fibre stayed as a compressed plug and needed to be released by digging with a small fork. The pressing and forking procedure was then repeated for the required number of pressings. A sample of the final juice was taken with a syringe, filtered and analysed in a refractometer. The same equation as that of Mangion and Player above applies.

Qin and White (1991) found significant differences in Brix-free water values for rind fibre, stalk fibre and stalk pith fractions in the test bagasse; they were 11.9, 18.3 and 22.1% respectively when a 5° Brix sucrose solution was used as the contact solution.

4.3 VARIABLES THAT AFFECT BRIX-FREE WATER DETERMINATIONS

Factors which affect the Brix-free water values are numerous and have been investigated by various research workers. They are enumerated in the following section.

4.3.1 Concentration of contacting solution

Qin and White (1991) found that the Brix-free water of cane fibre decreased as the concentration of the contacting sucrose solution increased. Also the analytical errors decreased because the concentration change became larger. This trend agreed with the observations of Van der Pol *et al.* (1957) and Kelly and Rutherford (1957), although the absolute values found by Qin and White for sieved bagasse were lower than their values for cane fibres (with higher temperature fibre drying). The values obtained by Kelly and Rutherford varied with the cane variety, with Brix-free water values up to 130% for one cane variety.

4.3.2 Drying temperature of fibre

The morphological history of the fibre is reported to have considerable effect on the Brix-free water value. Van der Pol *et al.* (1957) found a difference of five units in the Brix-free water values for cane dried at 60 °C (over phosphorus pentoxide and under vacuum) compared to the higher value obtained for the same fibre at 125 °C. Drying at 60 °C may

leave some residual moisture on the fibres; on the other hand, high temperature drying could have altered the characteristics of the fibre, e.g. activating it. Qin and White (1991) dried the fibres at 60 °C in an atmospheric oven for eight hours. Mangion and Player (1991) vacuum dried the fibres at 80 °C before performing the Brix-free water determination.

4.3.3 Temperature at which Brix-free water is measured

Qin and White (1991) changed the temperature of the controlled atmosphere cabinet, and tests carried out at three temperatures with a 5° Brix sucrose solution and at a 4:1 contact solution/fibre ratio showed no trend.

4.3.4 Method of sample preparation

Mangion and Player (1991) used three different types of equipment to extract fibre: a wet disintegrator, a Jeffco cutter-grinder and a gyratory-type machine grinder. The fibre obtained from the first two devices looked similar and gave comparable Brix-free water values, while that from the last device looked finer and yielded a higher Brix-free water value by 5 units. They attributed the higher value to an increased sample surface area per unit mass.

4.3.5 Methods of measurement

Mangion and Player (1991) also showed that Brix-free water results depend on the analytical techniques used to detect the change in concentration of the contacting solution after mixing with the fibre. They found parallel results on 13 analyses by a refractometric technique gave values two units higher than a polarimetric measurement (22.1% compared to 19.8%).

4.3.6 Cane variety

Mangion and Player (1991) carried out 20 analyses each of six cane varieties in the same region, and obtained values ranging from 17.0 to 20.3% Brix-free water. They attributed the variation in results to possible differences in fibre/pith ratios in the samples analysed and errors associated with the Brix measurements.

4.3.7 Pith and rind

Qin and White (1991) separated fibre, pith and rind from bagasse samples by sieving and manual selection. Brix-free water results, obtained with a 5° Brix sucrose contacting

solution at room temperature, showed significant differences between fibre (18.3%), pith (22.1%) and rind (11.9%). However, Kelly and Rutherford (1957) did not find any difference between these materials for their cane samples.

4.3.8 Sugar cane sections

Mangion and Player (1991) analysed various sections of the sugar cane plant and found the highest Brix-free water value in cane tops and the lowest in cane roots. For one cane variety, they obtained the following results: cane top (23.5%), 1/3 top (19.2%), 1/3 middle (18.9%), 1/3 bottom (20.3%), roots (6.7%) and trash (19.7%).

4.3.9 Crop location

Mangion and Player (1991) analysed 20 samples each of two cane varieties grown in three locations, and found that crop location did not consistently affect Brix-free water results for the varieties tested. While there was a statistical difference in the values obtained between two locations for a particular variety, it was not true for the other cane variety grown in the same cane areas.

4.4 DEVELOPMENT OF A BRIX-FREE WATER DETERMINATION METHOD FOR CANE FIBRES

From what has been described in the previous section it is clear that there is no one accepted method for the determination of Brix-free water in fibres. Also no one has previously determined the Brix-free water content for the range of sugar cane component parts studied in this work. There was therefore a need to devise a suitable method applicable to the samples investigated in this work.

The method of Mangion and Player (1991) for Brix-free water determination described in Section 4.2 was tried, and various aspects were investigated in order to adapt the method to the sugar cane plant fibre samples obtained by the separation method described in Chapter 3.

4.4.1 Residual sucrose in cane fibres

Some stalk fibre, stalk pith, green leaf fibre and rind fibre samples from a trial series were analysed for Brix-free water content according to Mangion and Player's method, with the difference that instead of the 8 g sample, a smaller sample size of 6 g was used; also,

instead of 150 g of 10° Brix sucrose contacting solution, a mass of 110 g was used to keep the ratio of the contact solution to sample size at about 20. Drying was effected in a vacuum oven under a vacuum of 875 mbar at 65 °C overnight, instead of 80 °C for three hours. The results obtained for the various stalk fibre and stalk pith samples (Table 4.1) were erratic and much higher than reported by other workers on these types of fibre. In one case, Brix-free water as high as 107.3% on stalk fibre was obtained. For fibres from different samples of green leaf and rind, results also appeared to be high (see Table 4.2).

Table 4.1. Determination of Brix-free water in stalk fibre and pith by Mangion and Player's (1991) method.

Sample	Mass of empty bottle/g	Mass of bottle + sample/g	Mass of bottle + sample + 150 g solution/g	Mass of sample/g W_3	Mass of soln./g W_4	Brix before P_3	Mean p_3	Brix after P_4	Mean p_4	Brix-free water $[100w_4(1-p_3\ p_4^{-1})]/w_3$
Stalk fibre 1	216.47	224.16	379.50	7.69	155.34	11.14,11.14,11.14	11.14	11.33, 11.34, 11.34	11.333	34.40
Stalk fibre 2	217.78	225.19	382.78	7.41	157.59	11.14, 11.14, 11.14	11.14	11.32, 11.32, 11.33	11.323	34.37
Stalk fibre 3	217.02	224.72	380.01	7.70	155.29		11.14	11.45, 11.44, 11.46	11.450	54.60
Stalk fibre 4	212.96	220.62	374.43	7.66	153.81		11.14	11.35, 11.34, 11.35	11.347	36.63
Stalk fibre 5	225.62	233.16	388.77	7.54	155.61		11.14	11.26, 11.26, 11.27	11.263	22.54
Stalk fibre 6	215.07	222.55	378.13	7.48	155.58		11.14	11.29, 11.30, 11.30	11.297	28.91
Stalk fibre 7	228.47	236.09	390.41	7.62	154.32		11.14	11.28, 11.28, 11.29	11.283	25.67
Stalk fibre 8	213.01	216.62	291.81	3.61	75.19		11.14	11.47, 11.48	11.475	60.81
Stalk fibre 9	213.82	221.43	375.38	7.61	153.95		11.14	11.37, 11.37, 11.37	11.370	40.92
Stalk fibre 10	212.73	216.50	294.06	3.77	77.56		11.14	11.56, 11.56	11.560	74.75
Stalk fibre 11	213.71	217.55	294.52	3.84	76.97		11.14	11.77, 11.77	11.770	107.29
Stalk fibre 12	214.84	218.42	293.10	3.58	74.68		11.14	11.60, 11.60	11.600	82.72
Stalk pith 1	227.31	234.81	392.23	7.50	157.42		11.14	11.38, 11.38, 11.38	11.380	44.27
Stalk pith 2	226.10	233.63	390.21	7.53	156.58		11.14	11.34, 11.34, 11.33	11.337	36.13
Stalk pith 3	216.43	223.88	374.37	7.45	150.49		11.14	11.43, 11.43, 11.42	11.427	50.73
Stalk pith 4	214.18	221.90	375.46	7.72	153.56		11.14	11.36, 11.36, 11.36	11.360	38.52
Stalk pith 5	214.34	221.62	374.95	7.28	153.33		11.14	11..30, 11.30, 11.30	11.300	29.82
Stalk pith 6	212.95	220.40	373.71	7.45	153.31		11.14	11.33, 11.32, 11.32	11.323	33.26
Stalk pith 7	215.42	222.46	377.86	7.04	155.40		11.14	11.32, 11.32, 11.32	11.320	35.10
Stalk pith 8	215.05	220.70	336.51	5.65	115.81		11.14	11.48, 11.48, 11.49	11.483	61.23

Table 4.2. Determination of Brix-free water in green leaf and rind fibres by Mangion and Player's (1991) method.

Sample	Mass of empty bottle/g	Mass of bottle + sample/g	Mass of bottle + sample + 150 g solution/g	Mass of sample/g W_3	Mass of soln./g W_4	Brix before P_3	Mean p_3	Brix after P_4	Mean p_4	Brix free-water $[100w_4(1-p_3\ p_4^{-1})]/w_3$
Green leaf fibre 1	216.48	222.54	340.85	6.06	118.31	10.39, 10.40, 10.40	10.40	10.58. 10.58, 10.58	10.580	33.77
Green leaf fibre 2	217.78	223.70	340.20	5.92	116.50	10.40, 10.40, 10.40	10.40	10.54, 10.55, 10.55	10.546	27.80
Green leaf fibre 3	217.02	222.52	338.11	5.50	115.59		10.40	10.54, 10.54, 10.54	10.540	28.51
Green leaf fibre 4	212.97	219.26	334.58	6.29	115.32		10.40	10.56, 10.56, 10.55	10.557	27.79
Green leaf fibre 5	225.64	231.80	334.59	6.16	102.79		10.40	10.56, 10.56, 10.55	10.557	25.29
Green leaf fibre 6	215.07	221.16	332.41	6.09	111.25		10.40	10.57, 10.59, 10.57	10.577	31.09
Green leaf fibre 7	228.47	234.41	350.97	5.94	116.56		10.40	10.56, 10.56, 10.56	10.560	30.29
Green leaf fibre 8	213.01	218.91	331.00	5.90	112.09		10.40	10.53, 10.53, 10.53	10.530	24.00
Green leaf fibre 9	213.81	220.35	335.99	6.54	115.64		10.40	10.54, 10.54, 10.54	10.540	23.99
Green leaf fibre 10	212.73	218.83	336.20	6.10	117.37		10.40	10.55, 10.54, 10.54	10.543	26.65
Green leaf fibre 11	213.72	219.56	331.68	5.84	112.12		10.40	10.56, 10.56, 10.57	10.563	29.63
Green leaf fibre 12	214.74	220.82	336.22	6.08	115.40		10.40	10.55, 10.55, 10.54	10.547	26.45
Rind fibre 1	227.31	232.93	345.83	5.62	112.90		10.40	10.56, 10.57, 10.56	10.563	31.00
Rind fibre 2	226.10	231.77	343.24	5.67	111.47		10.40	10.63, 10.63, 10.61	10.623	41.27
Rind fibre 3	216.41	222.28	337.76	5.87	115.48		10.40	10.54, 10.54, 10.54	10.540	26.13
Rind fibre 4	214.18	220.08	337.56	5.90	117.48		10.40	10.54, 10.54, 10.54	10.540	26.45
Rind fibre 5	214.34	220.46	337.16	6.12	116.70		10.40	10.54, 10.54, 10.54	10.540	25.33
Rind fibre 6	212.95	218.56	334.23	5.61	115.67		10.40	10.51, 10.51, 10.51	10.510	21.58
Rind fibre 7	215.43	221.06	333.91	5.63	112.85		10.40	10.51, 10.51, 10.51	10.510	20.98
Rind fibre 8	215.06	221.03	338.56	5.97	117.53		10.40	10.52, 10.52, 10.52	10.520	22.46

This is attributable to possible residual sucrose in the fibre sample, despite repeated washing and pressing during the fibre extraction process, resulting in an over-estimation of the Brix increase in the sucrose contacting solution, and hence an over-estimation of the Brix-free water content.

Reports exist on the use of detergent to clean plant tissue prior to certain analyses, e.g. Van Soest (1963) experimented with detergents to dissolve forage nitrogen in the preparation of fibrous portions of plant tissue of low nitrogen content for analysis such as lignin, and found cetyl trimethyl ammonium bromide in strongly acid solution or sodium lauryl sulfate in slightly alkaline solutions most effective. However, one must ensure that any residual reagent does not affect the subsequent Brix-free water determination. It would be much simpler to effect a blank determination in distilled water, and subtract the Brix value of the sample blank from that of the test solution. Since it would be difficult to adjust to have the same mass of distilled water in the blank as that of the contact solution in the test solution, let alone to have the same mass of fibre sample in the blank and test solution, bearing in mind the hygroscopic nature of cellulose fibre; a corrected blank is needed to compensate for the differences in masses. The advantage of the corrected blank is that if one sample is used several times in the same experiment for different test purposes, only one blank will be required.

Thus, if w_1 and w_3 are the mass of sample in the blank and test solution respectively, and w_2 is the mass of distilled water in the blank and w_4, the mass of the contacting solution in the test solution; p_1 and p_2 are the Brix of the sample blank before and after equilibrium, and p_3 and p_4 are the Brix of the test solution before and after equilibrium, the corrected blank, b, is given by $b = p_2 w_2 w_3/(w_1 w_4)$ and the net Brix increase by $p = p_4 - b$.

Hence the Brix-free water in the sample is given by $BFW = [100\ w_4\ (1 - p_3\ p^{-1})]/w_3$.

Results obtained from almost sucrose-free fibre samples (Table 4.3) confirmed that the samples still had high residual sucrose content as evidenced by the high Brix p_2 after equilibration with distilled water, and the Brix-free water results are now 30-40% lower than those obtained had the sample blank not been incorporated in the method. Most other workers performed their studies on bagasse which probably has a low residual sucrose.

Table 4.3. Comparison of Brix-free water determined in fibre samples with and without the incorporation of a blank.

Determination of blank value

	Sample	Mass of empty bottle/g	Mass of bottle + sample/g	Mass of bottle + sample + 110 g distilled water/g	Mass of sample/g w_1	Mass of dist. H_2O/g w_2	Brix before p_1	Mean p_1	Brix after p_2	Mean p_2	Adjusted blank (b) $p_2w_2w_3/(w_1w_4)$
1b	Stalk fibre	216.48	222.69	336.55	6.21	113.86	0.00, 0.00, 0.00	0.000	0.04, 0.04, 0.03	0.037	0.035
2b	Stalk pith	217.78	223.62	337.51	5.84	113.89		0.000	0.04, 0.04	0.040	0.039
3b	Rind fibre	217.03	223.28	334.74	6.25	111.46		0.000	0.04, 0.04, 0.04	0.040	0.039
4b	Rind fines	212.97	219.10	331.97	6.13	112.87		0.000	0.03, 0.03, 0.03	0.030	0.028
5b	Dry leaf fibre	225.64	232.26	345.44	6.62	113.18		0.000	0.05, 0.06, 0.06	0.057	0.056
6b	Dry leaf fines	215.08	221.66	333.87	6.58	112.21		0.000	0.07, 0.07, 0.05	0.063	0.057

Determination of Brix-free water in sample

	Sample	Mass of bottle empty/g	Mass of bottle + sample/g	Mass of bottle + sample + 110 g 10º Brix solution/g	Mass of sample/g w_3	Mass of soln./g w_4	Brix before p_3	Mean p_3	Brix after p_4	Mean p_4	net p p_4 - b	Brix-free water $[100w_4(1-p_3\,p^{-1})]/w_3$	Brix-free water **without sample blank**
1	Stalk fibre	216.48	222.48	339.67	6.00	117.19	10.04, 10.04,10.05	10.048	10.14, 10.15, 10.14	10.143	10.109	11.71	18.36
2	Stalk pith	217.78	223.69	341.58	5.91	117.89	10.05, 10.05, 10.06	10.048	10.16, 10.16	10.160	10.121	14.37	21.99
3	Rind fibre	217.03	223.34	337.78	6.31	114.44		10.048	10.15, 10.14, 10.15	10.147	10.107	10.65	17.64
4	Rind fines	212.96	218.90	336.18	5.94	117.28		10.048	10.13, 10.13, 10.13	10.130	10.102	10.56	15.98
5	Dry leaf fibre	225.63	232.12	344.99	6.49	112.87		10.048	10.18, 10.18, 10.18	10.180	10.124	13.05	22.55
6	Dry leaf fines	215.07	221.11	334.15	6.04	113.04		10.048	10.19, 10.19, 10.19	10.190	10.133	15.62	25.48

4.4.2 Brix measurement

As seen in Table 4.3, when the volume of the sample solution permits, three Brix values have been recorded for each sample. This was done by pouring part of the solution into the refractometer, three Brix readings were taken and an average was recorded. The process was repeated two more times by pouring more sample solution into the refractometer. For the original contact solution, six such values were recorded, three at the beginning and three more at the end of the Brix measurement. It is understood that all Brix values were corrected for instrument zero by using distilled water, as shown by the zero Brix before contact p_1, in the sample blank determination. As pointed out by Qin and White (1991) only a small difference of about 0.1 unit Brix is expected (see Section 4.2); such a precaution is therefore essential to ensure accuracy in the results obtained.

4.4.3 Re-generation of fibre samples

The incorporation of a blank determination does, however, imply the necessity of having twice as much sample for the Brix-free water determination.

Since samples to be tested can sometimes be limited in quantity, the possibility of re-generation of the sample after analysis was contemplated. Fibre samples as listed in Table 4.4 were therefore analysed and then washed free of Brix (< 0.01 Brix), dried overnight in an air-oven at 70 °C and re-analysed incorporating the blank determination. The results obtained were much greater and highly different ($p < 0.001$) from the original results. This was confirmed by analysis of further rind fibre and stalk pith samples. This concurs well with the findings of Oguri (1932) and Stamm and Hansen (1938) who found that re-generated cellulose adsorbed more water than the original material.

Consequently, this test showed that re-regeneration of samples for further tests is not viable and that sufficient fibre samples must be prepared beforehand.

Table 4.4. Results of the determination of Brix-free water on re-generated samples.

Sample	Brix-free water % on sample	
	Original	Re-generated
Stalk fibre	10.3	12.8
Stalk pith	9.0	15.8
Rind fibre	12.8	15.4
Rind fines	7.4	18.2
Dry leaf fibre	15.8	18.1
Dry leaf fines	15.3	19.4

4.4.4 Homogeneity of fibre samples

When the samples analysed previously (Table 4.3) were examined closely, it was evident that the fibre/fines separation was not perfect; discrepancy could arise due to the heterogeneous nature of the samples since the results obtained so far indicated that the finer fractions of a cane component have higher Brix-free water values than the coarser fibre fractions. It was therefore decided to sieve all samples through a 1.18 mm sieve as described in Section 3.4.3.1. The fraction retained on the sieve will be termed 'fibre' and that which passed through the sieve, as 'fines', or 'pith' in the case of stalk fines. Samples of rind, stalk, top and green leaf fibres were separately sieved through a 1.18 mm sieve, and 4 x 6 g of each were weighed out in four bottles for triplicate determinations of Brix-free water content. The fourth replicate was used for the blank determination. Drying was effected in a vacuum oven at 65 °C under 875 mbar vacuum overnight for 16 hours. The triplicate results obtained for each cane component appeared consistent except for that of stalk fibre (Table 4.5).

Table 4.5. Reproducibility of Brix-free water determinations on fibre samples.

	Sample	Mass of bottle/g	Mass of bottle sample/g	Mass of bottle + sample 110 g distilled water/g	Mass of w_1	Mass of dis. H_2O/g w_2	Brix before p_1	Mean p_1	Brix after p_2	Mean p_2	Adjusted blank (b) $p_2w_2w_3/(w_1w_4)$
1b	Rind fibre	225.48	231.24	342.47	5.76	111.23	0.00 0.00 0.00	0.000	0.01 0.01 0.01	0.010	0.009
2b	Rind fibre				5.76	111.23					0.009
3b	Rind fibre				5.76	111.23					0.009
4b	Stalk fibre	226.13	231.45	344.88	5.32	113.43	0.00 0.00 0.00	0.000	0.01 0.01 0.02	0.012	0.012
5b	Stalk fibre				5.32	113.43					0.012
6b	Stalk fibre				5.32	113.43					0.012
7b	Top fibre	202.07	208.02	319.60	5.95	111.58	0.00 0.00 0.00	0.000	0.07 0.06 0.07	0.067	0.061
8b	Top fibre				5.95	111.58					0.064
9b	Top fibre				5.95	111.58					0.064
10	Green leaf	220.77	226.34	340.25	5.57	113.91	0.00 0.00 0.00	0.000	0.03 0.03 0.04	0.033	0.039
11	Green leaf				5.57	113.91					0.035
12	Green leaf				5.57	113.91					0.036

	Sample	Mass of bottle empty/g	Mass of bottle sample/g	Mass of bottle + sample 110 g 10º Brix solution/g	Mass of w_3	Mass of soln./g w_4	Brix before p_3	Mean p_3	Brix after p_4	Mean p_4	net p p_4 - b	Brix-free water $[100w_4(1-p_3p^{-1})]/w_3$	Standard deviation
1	Rind fibre	226.29	231.40	344.72	5.11	113.32	9.97 9.98 9.98	9.978	10.07 10.06 10.06	10.067	10.05	17.56	
2	Rind fibre	212.64	217.74	326.96	5.10	109.22		9.978	10.07 10.07 10.08	10.073	10.06	18.30	
3	Rind fibre	216.80	222.05	337.42	5.25	115.37		9.978	10.07 10.07 10.07	10.070	10.06	18.10	0.383
4	Stalk fibre	216.30	221.49	333.15	5.19	111.66		9.978	10.07 10.07 10.07	10.072	10.06	17.49	
5	Stalk fibre	225.36	230.53	340.71	5.17	110.18		9.978	10.08 10.09 10.09	10.087	10.07	20.45	
6	Stalk fibre	214.09	219.49	333.30	5.40	113.81		9.978	10.08 10.09 10.08	10.085	10.07	19.85	1.565
7	Top fibre	180.48	186.20	303.13	5.72	116.93	9.74 9.74 9.75	9.745	9.890 9.900 9.910	9.900	9.839	19.50	
8	Top fibre	175.38	181.10	292.88	5.72	111.78		9.745	9.900 9.910 9.915	9.908	9.844	19.72	
9	Top fibre	178.31	184.33	301.07	6.02	116.74		9.745	9.900 9.910 9.915	9.908	9.844	19.48	0.133
10	Green leaf	183.50	189.69	298.29	6.19	108.60		9.745	9.87 9.875 9.885	9.877	9.838	16.55	
11	Green leaf	165.85	171.86	288.55	6.01	116.69		9.745	9.86 9.875 9.865	9.867	9.832	17.09	
12	Green leaf	180.63	186.87	305.25	6.24	118.38		9.745	9.87 9.875 9.88	9.875	9.839	18.14	0.809

In order to test for homogeneity of samples, fibres of various components of cane were extracted from 15 kg of cane. After removing and discarding the nodes, the rind was separated from the stalk. Fibres were extracted from the rind and the stalk, washed free of sucrose and dried as per the method described in Chapter 3 (Sections 3.4.3.1 and 3.4.3.2). Fibres were separated from the fines/pith as much as possible to obtain four well-separated fractions: stalk fibre (620 g), pith (1175 g), rind fibre (1240 g) and rind fines (830 g). There were also two mixtures: stalk with pith weighing 250 g and rind fibre with fines weighing 915 g. No effort was made to further separate these two mixtures as they would serve to see to what extent reproducible results could be obtained from them.

Each of these six fractions was tested in five replicates and one blank determination was effected. Drying was effected in a vacuum oven at 80 °C for 3 hours. To 6 g of sample when cool were added 110 g of 10° Brix sucrose solution. A contact time of one and a half hours was allowed during which period, the sample was shaken every ten minutes.

The Brix-free water results are shown in Table 4.6, which indicate that the fractions of stalk fibre, rind fibre, rind fibre + fines, and rind fines were homogeneous, despite such a small sample (6 g) taken from a large sample, e.g. 1240 g, in the case of rind fibres. For the stalk fibre + pith mixture and stalk pith sample, results were not reproducible, probably because of the difficulty associated with drying the pith. Hence the drying condition prior to analysis needed investigation.

4.4.5 Drying conditions prior to analysis

When more of the samples which had been examined previously (results shown in Table 4.3) were analysed again two days later for their Brix-free water content under the same analytical conditions and incorporating the blank value, significantly higher results ($P < 0.05$) were obtained (see Table 4.7). This would indicate that the samples were drier on the second occasion than when first examined, and took up more water to satisfy their Brix-free water capacity. Thus, the overnight vacuum drying at 65 °C so far adopted needed to be re-examined.

One green leaf fibre sample was well mixed, and sub-samples of 6 g were weighed into separate bottles. Samples were dried at 65 °C under vacuum of 875 mbar for 2, 4, 6 and 16 hours. The Brix-free water results (Table 4.8) show increasing values from 2, 4, 6 hours reaching the highest value at 16 hours, implying that drying under these conditions for less than 16 hours is insufficient to drive off the moisture originally present in the sample.

Table 4.6. Homogeneity test for fibre samples.

Sample	Brix-free water/%			Sample	Brix-free water/%			Sample	Brix-free water/%		
	Value	Mean	S.D.		Value	Mean	S.D.		Value	Mean	S.D.
Stalk fibre	16.52			Rind fibre + fines	11.70			Stalk fibre + pith	19.80		
Stalk fibre	15.25			Rind fibre + fines	12.27			Stalk fibre + pith	20.02		
Stalk fibre	17.07			Rind fibre + fines	12.88			Stalk fibre + pith	20.72		
Stalk fibre	16.64			Rind fibre + fines	12.27			Stalk fibre + pith	22.65		
Stalk fibre	15.23	16.14	0.849	Rind fibre + fines	12.15	12.25	0.424	Stalk fibre + pith	20.23	20.68	1.151
Rind fibre	13.15			Rind fines	15.04			Stalk pith	29.99		
Rind fibre	12.64			Rind fines	15.30			Stalk pith	27.31		
Rind fibre	12.31			Rind fines	15.11			Stalk pith	25.53		
Rind fibre	12.53			Rind fines	14.79			Stalk pith	24.73		
Rind fibre	12.71	12.67	0.307	Rind fines	14.10	14.87	0.466	Stalk pith	27.39	26.99	2.029

S.D. is the standard deviation.

Table 4.7. Results of the repeat determination of Brix-free water in the same fibre samples as in Table 4.3 incorporating the blank determination.

	Sample	Mass of empty bottle/g	Mass of bottle + sample/g	Mass of bottle + sample + 110 g distilled water/g	Mass of sample/g w_1	Mass of dist. H_2O/g w_2	Brix before p_1	Mean p_1	Brix after p_2	Mean p_2	Adjusted blank (b) $p_2w_2w_3/(w_1w_4)$
1b	Stalk fibre	216.48	222.63	331.99	6.15	109.36	0.00, 0.00, 0.00	0.000	0.03, 0.03, 0.02	0.027	0.025
2b	Stalk pith	217.78	224.64	334.41	6.86	109.77	0.00, 0.00, 0.00	0.000	0.02, 0.01	0.015	0.014
3b	Rind fibre	217.03	223.53	336.64	6.50	113.11		0.000	0.03, 0.03, 0.03	0.027	0.025
4b	Rind fines	212.97	219.22	330.29	6.25	111.07		0.000	0.02, 0.02	0.020	0.019
5b	Dry leaf fibre	225.62	231.72	346.69	6.10	114.97		0.000	0.03, 0.03, 0.03	0.030	0.029
6b	Dry leaf fines	215.08	221.77	330.89	6.69	109.12		0.000	0.04, 0.04, 0.04	0.040	0.034

	Sample	Mass of bottle empty/g	Mass of bottle + sample/g	Mass of bottle + sample + 110 g 10º Brix solution/g	Mass of sample/g w_3	Mass of soln./g w_4	Brix before p_3	Mean p_3	Brix after p_4	Mean p_4	net p p_4 - b	Brix free water $[100w_4(1-p_3\,p^{-1})]/w_3$
1	Stalk fibre	228.46	235.04	360.83	6.58	125.79	10.00, 9.99,9.99	9.992	10.10, 10.09, 10.10	10.097	10.070	14.81
2	Stalk pith	213.01	219.47	332.46	6.46	112.99	9.99, 9.99, 9.99	9.992	10.13, 10.13	10.130	10.115	21.27
3	Rind fibre	213.82	220.14	340.27	6.32	120.13		9.992	10.09, 10.09, 10.08	10.087	10.060	12.85
4	Rind fines	212.73	219.02	338.38	6.29	119.36		9.992	10.09, 10.09, 10.09	10.090	10.070	14.70
5	Dry leaf fibre	213.71	220.00	340.69	6.29	120.69		9.992	10.11, 10.11, 10.10	10.107	10.077	16.18
6	Dry leaf fines	214.74	221.03	340.84	6.29	119.81		9.992	10.13, 10.13, 10.13	10.130	10.090	18.50

Table 4.8. Brix-free water results for fibre samples which had either been vacuum or air dried for varying lengths of time prior to analysis.

Sample	**Vacuum** oven drying		Brix-free water/%	Sample	**Air** oven drying		Brix-free water/%
	Temperature/^{o}C	Duration/h			Temperature/^{o}C	Duration/h	
Green leaf fibre	65	2	12.95	Rind fibre	65	1	9.90
Green leaf fibre	65	4	14.20	Rind fibre	65	4	11.86
Green leaf fibre	65	6	13.51	Rind fibre	65	6	11.93
Green leaf fibre	65	16	16.85	Rind fibre	65	15	13.56
				Rind fibre	65	21	13.85
Green leaf fibre	80	3	17.42	Rind fibre	105	1	15.75
Green leaf fibre	80	4	17.33	Rind fibre	105	3	15.56
Green leaf fibre	80	5	16.83	Rind fibre	105	6	17.01
Green leaf fibre	80	7	17.87	Rind fibre	105	14	16.82
Green leaf fibre	80	16	15.73	Rind fibre	105	20	17.37

The experiment was repeated with the remaining sub-samples after drying at 80 °C in a vacuum oven under 875 mbar vacuum for 3, 4, 5, 7 and 16 hours.

Results obtained (Table 4.8) after vacuum drying at 80 °C for 3, 4, 5 and 7 hours are all comparable to those obtained after drying overnight for 16 hours at 65 °C (average of 17.4% c.f 16.9%). However, after overnight drying at 80 °C, the Brix-free water value obtained was much lower at 15.7%.

Air-oven drying was tried on subsamples of a rind fibre at 65 °C and 105 °C for various lengths of time up to 21 hours. Results (Table 4.8) show that 65 °C was insufficient to dry the samples even after 21 hours whereas 105 °C yielded the highest Brix-free value of the sample after 6-20 hours. This means that at 105 °C after 6-20 hours, the Brix-free water originally in the sample has been completely driven out of the sample, enabling 'new' water to be adsorbed during analysis and determined as Brix-free water. However, it would be unwise to adopt this drying method prior to analysis, since many workers have warned against the use of high temperatures, notably Kollmann and Schneider (1963) who indicated that elevated temperatures reduce timber water adsorption capacity.

From the data in Table 4.8, it was evident that after one hour of drying at 65 °C, the Brix-free water result of 9.9% represented more than half of the maximum value of 17.37% achieved after 20 hours of drying at 105 °C. Hence it was thought reasonable to use one hour air-oven drying at 65 °C prior to vacuum drying to boost the drying process. Results for one hour air-oven drying at 65 °C followed by 65 °C or 80 °C vacuum-oven drying for various lengths of time are shown in Table 4.9.

It appears that more consistent results are obtained after air-oven drying at 65 °C for one hour followed by overnight vacuum-oven drying for 16 hours at 65 °C under 875 mbar vacuum. (See also Section 4.4.6.).

4.4.6 Residual moisture in fibre samples prior to Brix-free water determination

The Brix-free water values obtained for certain sugar cane component parts were in general lower than those obtained by Mangion and Player (1991), for example, 13 – 15% both for cane top and dry leaf fibres compared to those of Mangion and Player of 23 – 24% for cane top and 20 – 21% for dry leaf. It was therefore thought opportune to check the remaining moisture content of fibres after the method of vacuum oven drying at 65 °C for 16 hours under 875 mbar vacuum prior to the Brix-free water determination. Should there

Table 4.9. Analysis of Brix-free water in samples dried in an air - oven at 65 ^{o}C for one hour before vacuum drying.

Sample	Vacuum oven drying		Brix free water/%	Sample	Vacuum oven drying		Brix free water/%
	Temperature/^{o}C	Duration/h			Temperature/^{o}C	Duration/h	
Rind fibre	65	2	14.82	Stalk fibre	80	2	15.62
Rind fibre	65	4	14.98	Stalk fibre	80	4	17.33
Rind fibre	65	6	15.42	Stalk fibre	80	6	15.96
Rind fibre	65	16	14.32	Stalk fibre	80	16	14.34
Rind fibre	65	21	13.12	Stalk fibre	80	21	17.04
Rind fines	65	2	10.74	Stalk pith	80	2	26.38
Rind fines	65	4	16.63	Stalk pith	80	4	25.10
Rind fines	65	6	16.51	Stalk pith	80	6	24.70
Rind fines	65	16	16.11	Stalk pith	80	16	20.20
Rind fines	65	21	12.81	Stalk pith	80	21	21.43

be any residual moisture in the sample, it would imply under-estimation in the subsequent Brix-free water determination.

One gram each of the nine cane component fibres was placed in separate pre-weighed glass bottles of 60 mm diameter and 70 mm height with snap-on metallic caps. The samples were dried in an air oven at 65 °C for one hour with occasional stirring, followed by vacuum oven drying at 65 °C for 16 hours under 875 mbar vacuum, and weighed when cool. They were subjected to further drying in an air oven at 105 °C for three hours to drive off any residual moisture present, and weighed when cool. The difference in the two masses affords an estimation of the residual moisture in the samples after the vacuum oven drying method. The experiment was performed eleven times, and the results (Table 4.10) show that the average residual moisture in the samples varied from 0.63% for cane top fibre to 1.42% for dry leaf fines, and the average for all fibre samples was 1.12%.

The drying method used by Mangion and Player (1991), i.e. vacuum oven drying at 80 °C and 825 mbar vacuum for three hours, was also checked to see if it was sufficient to dry the samples completely. The same samples were tested again eleven times and the results thereof are presented in Table 4.10. They varied from 1.38% for rind fibre to 2.22% for dry leaf fibre, and the average for all fibre samples was 1.72%.

Since the accepted standard method of moisture determination by drying at 105 °C to constant mass takes a long time, i.e. at least three hours, it was thought useful to have a rapid method for residual moisture determination in fibres.

4.4.6.1 *Halogen thermogravimetry as a technique to determine residual moisture in fibres*

Wong Sak Hoi (1997) tested the suitability of a halogen moisture analyser for rapid moisture determination in sugars. The method devised was found to give results comparable to those of the reference oven drying method at 105 °C for three hours, with a shorter analysis time ranging from two to twelve minutes depending on the moisture content of the sugar. In addition, good precision and accuracy was obtained for moisture levels in sugars as low as < 0.1% and as high as 4%.

Table 4.10. Residual moisture in fibres of cane component parts after vacuum drying and prior to Brix-free water analysis.

Sample	Residual moisture after one hour drying at 65 °C in an air oven followed by vacuum drying at 65 °C for 16 hours/%											
	1	2	3	4	5	6	7	8	9	10	11	Mean
Stalk fibre	2.34	0.96	0.43	1.13	0.46	1.88	3.30	2.13	0.90	0.95	0.64	1.38
Stalk pith	1.17	1.21	0.85	1.04	3.53	0.56	2.33	0.71	0.89	0.01	1.21	1.23
Rind fibre	1.57	-0.27	1.74	0.15	2.25	0.20	3.40	0.25	1.62	-0.17	0.93	1.06
Rind fines	0.63	0.99	0.80	1.74	1.31	0.22	0.59	0.66	0.75	2.11	1.16	1.00
Top	1.07	-0.32	1.66	0.25	0.38	0.15	0.44	-0.93	0.84	1.07	2.36	0.63
Dry leaf fibre	1.13	1.11	0.52	0.39	1.18	0.32	2.18	0.98	1.02	1.33	2.82	1.18
Dry leaf fines	1.87	1.30	0.88	1.13	0.82	0.16	0.68	3.19	2.85	1.32	1.37	1.42
Green leaf fibre	1.12	1.37	0.84	1.94	2.20	0.32	0.54	0.78	0.69	1.31	1.25	1.13
Green leaf fines	-0.14	1.48	0.94	1.83	0.82	0.27	0.76	0.85	1.55	1.41	1.44	1.02
												1.12

Sample	Residual moisture after vacuum drying at 80 °C for 3 hours after Mangion and Player (1991)/%											
	1	2	3	4	5	6	7	8	9	10	11	Mean
Stalk fibre	1.57	1.35	2.14	1.04	2.91	0.85	1.32	-0.22	2.77	1.55	1.96	1.57
Stalk pith	1.87	1.52	1.30	1.33	1.46	2.92	-0.18	1.47	1.74	1.43	1.44	1.48
Rind fibre	1.59	1.36	1.03	1.04	1.71	1.08	1.31	2.12	1.52	1.27	1.09	1.38
Rind fines	3.74	1.40	1.70	1.13	1.48	1.83	1.38	1.34	1.74	1.23	2.54	1.77
Top	1.83	1.99	0.92	0.13	-0.19	1.84	4.64	-0.72	1.78	2.42	1.30	1.45
Dry leaf fibre	2.16	0.29	4.01	1.15	4.60	1.84	1.55	1.81	2.54	2.39	2.04	2.22
Dry leaf fines	2.04	1.46	2.09	1.35	1.57	1.28	1.60	1.27	2.05	1.68	1.51	1.63
Green leaf fibre	2.19	1.73	4.24	1.32	1.72	3.22	1.83	1.51	2.18	1.38	1.52	2.08
Green leaf fines	2.42	2.52	3.16	1.62	0.12	2.61	2.00	0.99	2.29	1.73	1.50	1.90
												1.72

By means of the same technique, Wong Sak Hoi (1998) subsequently developed a rapid moisture determination method programmed for products of high moisture content such as filter cake, bagasse and cane. The application of the method to these products was found successful with regard to both precision and accuracy of the method compared with the reference methods, and with great savings in analysis time, which is essential in process control.

It was thought useful to develop a method for rapid moisture determination in fibre by using halogen thermogravimetry; this will be particularly useful to check any residual moisture in the fibre after vacuum oven drying.

The Mettler Toledo (Model HR73) moisture analyser (Fig 4.2) is a development of the infrared dryer. It consists of a glass tube filled with an inert halogen gas and contains a tungsten wire heating element. The geometry of the halogen radiation provides uniform distribution of the infrared radiation over the whole surface of the samples through a large gold-plated reflector. It is claimed by the manufacturer that the analyser cools faster and provides more precise temperature control than a conventional infrared dryer.

Figure 4.2. The Mettler Toledo HR73 halogen thermogravimetric moisture analyser.

A precision balance integrated in the instrument continuously monitors the mass change of the sample under test, which is either dried for a pre-set period or to constant mass. The apparatus offers four drying programmes, namely standard, rapid, gentle and stepwise drying for different kinds of products, and five automatic switch-off criteria (1-5) for

decreasing specific mass loss, the drying is automatically ended as soon as the mean mass loss of 1 mg drops below 10, 20, 50, 90 and 140 seconds respectively. A built-in printer records the sample mass, the analysis method, the time and the results in the form of percentage moisture content or percentage dry matter. The apparatus also provides an integrated programme for statistical analysis of the last five test results.

4.4.6.2 *A rapid method developed to determine residual moisture in fibres by halogen thermogravimetry*

After much experimentation, the following method was found to be precise and suitable for the rapid determination of moisture in fibres. It consists of the use of a glass fibre filter previously dried under the same conditions as the sample for use as a cover for the drying of the sample.

4.4.6.2.1 Equipment

A Whatman GF/C glass fibre filter of 9 cm diameter and a Mettler Toledo (Model HR73) moisture analyser were used.

4.4.6.2.2 Method of determination of residual moisture in fibres

The integrated balance of the moisture analyser was tared with the aluminium pan on the balance. A Whatman GF/C glass fibre filter was placed on the pan to dry by using the standard drying programme at 105 °C and automatic switch-off mode 3. At the end of the drying programme, the filter was quickly removed with a pair of tweezers, placed in a petri-dish with cover, and allowed to cool in a desiccator.

After the halogen moisture analyser was cooled to about 65 °C, the balance was quickly tared with the dried filter, after which, the filter was quickly put back into the petri-dish and covered. About 0.6 g of the fibre sample was quickly shaken onto the aluminium pan, spread out evenly with a spatula, and covered with the dried filter. Drying was then started by using the same standard drying programme as above at 105 °C with automatic switch-off mode 3. At the end of the heating programme, the moisture content in the sample was displayed. This method of moisture determination in fibres had the great advantage of requiring only a few minutes instead of the three hours essential in the standard drying method.

After the determination, the aluminium pan was wiped with a clean and dry cloth, and any fibre adhering to the glass filter was shaken off. The above cycle could be repeated for the next sample by first drying the filter again. This could be done while the moisture analyser

was still hot. Since the filter was used only as a cover for the fibre sample, it could be used over and over again.

4.4.6.2.3 Comparison of the halogen drying method with the standard oven drying method

About 3.5 g each of the fibres of nine sugar cane component parts were separately weighed into pre-weighed 250 mL glass bottles with stoppers, and dried in a vacuum oven at 65 °C and 875 mbar vacuum for 16 hours after a pre-heating period of one hour at 65 °C in an air oven.

After drying, the bottles were cooled in desiccators. About 0.8 g of each sample was quickly transferred into two small pre-weighed bottles, which were then weighed on the analytical balance to four decimal places and dried by using the standard method of 105 °C in an air oven to constant mass (about three hours). Two portions (about 0.6 g) of the same sample were also examined by the halogen moisture analyser to yield duplicate moisture content values of the sample.

Five replicates of nine fibre samples were examined. The results are shown in Table 4.11.

Statistical analysis by paired comparison of the averages of the duplicate results of residual moisture content determined by the standard drying method and the halogen thermogravimetric method showed that the difference was not significant; and for 45 data, the standard error of the mean was 0.104% for a mean of 2.48% by the halogen drying method compared with 0.096% for a mean of 2.36% by the standard oven drying method.

It was concluded that the method of moisture determination in fibre by halogen thermogravimetry gives results comparable to the reference oven drying method at 105 °C to constant mass, with a much shorter analysis time. The method was subsequently used whenever it was required to check the moisture content in fibres.

Also, fibre which had been dried in a vacuum oven at 65 °C for 16 hours prior to Brix-free water determination still contained about 1.1% moisture. As mentioned in Section 4.3.2, Van der Pol *et al.* (1957) found a difference of five units in the Brix-free water values for cane, dried at 60 °C over phosphorus pentoxide and under vacuum, compared to the higher value for the same fibre dried at 125 °C. Since high temperature drying could have altered the characteristics of the fibre (i.e. activating it), the method of drying in a vacuum oven at 65 °C for 16 hours was adopted for fibres in this study, and if required, the Brix-free water

Table 4.11. Comparison of moisture determination in fibre samples by halogen thermogravimetry and the standard drying method.

Sample	Moisture/%											
	Halogen	Oven	Halogen	Oven	Halogen	Oven	Halogen	Oven	Halogen	Oven	**Mean halogen**	**Mean oven**
Stalk fibre	3.68	1.79	4.31	2.51	3.35	2.71	3.07	3.27	4.19	2.45	**3.72**	**2.55**
Stalk pith	2.76	3.58	2.52	1.92	2.26	3.42	2.15	2.10	2.45	2.13	**2.43**	**2.63**
Rind fibre	2.00	3.05	2.39	2.90	2.65	2.40	2.05	2.28	2.22	2.18	**2.26**	**2.56**
Rind fines	2.46	2.55	2.12	1.83	2.12	2.58	1.79	1.11	1.88	1.02	**2.07**	**1.82**
Top	3.79	3.18	2.79	3.03	3.70	3.03	3.34	3.57	3.10	1.59	**3.34**	**2.88**
Dry leaf fibre	2.55	2.86	2.62	3.21	2.18	2.72	2.43	2.90	2.34	1.47	**2.42**	**2.63**
Dry leaf fines	2.29	2.04	2.08	2.12	1.57	2.92	1.56	1.29	2.06	2.05	**1.91**	**2.08**
Green leaf fibre	3.10	2.13	2.00	1.93	2.44	1.99	2.34	2.47	2.18	2.45	**2.41**	**2.19**
Green leaf fines	2.09	1.88	1.70	1.92	1.57	1.66	1.58	1.58	1.68	2.62	**1.72**	**1.93**
											2.48	**2.36**

value determined could be corrected for the residual moisture after the vacuum drying procedure.

4.4.7 Concentration of the contact solution

By varying the concentration of the contact solution from 5 to 10 and 15° Brix sucrose solution, it was found that the Brix-free water in a rind fibre and stalk pith varied with a change in sucrose concentration (Table 4.12), and to increase with increased concentration of sucrose solution (Table 4.13) in a later experiment with a stalk fibre and a rind fibre. This is in contradiction to the findings of Qin and White (1991), Van der Pol *et al.* (1957) and Kelly and Rutherford (1957), who found that the Brix-free water values decreased as the concentration of the contact solution increased.

With a 5° Brix sucrose solution, the increase in Brix after contacting would be smaller than 0.1 unit and hence difficult to detect, whereas with a 15° Brix solution, the stability of the sucrose would be doubtful after the long procedure of determination. It was therefore decided to adopt a 10° Brix sucrose solution as the contact solution, since it was also adopted by Mangion and Player (1991).

4.4.8 Ratio of the contact solution to sample size

In this experiment, a 10° Brix sucrose solution was used and four samples were examined: two rind fibres and two stalk piths. The ratio of contact solution to sample mass was varied from 25 to 10. At a high ratio, e.g. 25, the errors became large as the increase in Brix after equilibration was less than 0.1 unit, and at a low ratio, e.g. 15, the volume of the solution obtained from pith samples after equilibration was not sufficient for the Brix determination. At a ratio of 5, pith samples were not completely wetted. It was therefore found that a ratio of 20 was most appropriate. The higher surface area of stalk pith as shown by scanning electron microscopy (Fig 3.24f) may explain its higher liquid absorption than sample of other cane components.

Two samples showed a decrease in Brix-free water with decrease in the ratio of contact solution/sample, while one stalk pith showed the opposite (Table 4.14). Qin and White (1991) with their press cell method, however, found that this ratio does not affect the Brix-free water results, although the errors become larger at the higher juice/fibre ratios.

Table 4.12. Impact of concentration of contact solution on Brix-free water.

Sample	Concentration of sucrose contact solution/°Brix	Brix-free water/%
Rind fibre	5	10.15
Rind fibre	10	9.76
Rind fibre	15	14.03
Stalk pith	5	15.80
Stalk pith	10	19.41
Stalk pith	15	19.19

Table 4.13. Impact of concentration of contact solution on Brix-free water.

Sample	Concentration of sucrose contact solution/°Brix	Brix-free water/%	Mean/%
Stalk fibre	5	9.43	8.22
Stalk fibre	5	7.01	
Stalk fibre	10	13.02	13.89
Stalk fibre	10	14.75	
Stalk fibre	15	14.22	14.46
Stalk fibre	15	14.70	
Rind fibre	5	6.25	7.74
Rind fibre	5	9.22	
Rind fibre	10	10.70	10.84
Rind fibre	10	10.97	
Rind fibre	15	11.17	11.11
Rind fibre	15	11.05	

Table 4.14. Impact of ratio of contact solution to sample size on Brix-free water.

Sample	Ratio of contact solution to sample	Brix-free water/%
Rind fibre 1	19	21.69
Rind fibre 1	14	17.72
Rind fibre 1	9	16.87
Stalk pith 1	20	28.14
Stalk pith 1	14	24.72
Stalk pith 1	9	20.75
Rind fibre 2	25	14.36
Rind fibre 2	20	10.04
Rind fibre 2	15	11.99
Stalk pith 2	24	15.16
Stalk pith 2	20	15.80
Stalk pith 2	13	20.40

Qin and White's finding was confirmed in a later experiment with a stalk fibre sample, when 10° Brix and 15° Brix sucrose solutions were used; the ratio of the contact solution was varied from 14.0 to 25.6 (Table 4.15).

Table 4.15. Impact of ratio of contact solution to sample size on Brix-free water.

Sample	Concentration of contact sucrose solution/°Brix	Ratio of contact solution to sample	Brix-free water/%	Mean/%
Stalk fibre	10	25.0	15.36	15.71
Stalk fibre	10	25.6	16.06	
Stalk fibre	10	18.8	15.47	15.75
Stalk fibre	10	19.3	16.02	
Stalk fibre	10	14.3	16.65	16.55
Stalk fibre	10	14.0	16.45	
Stalk fibre	15	25.5	16.99	16.96
Stalk fibre	15	25.2	16.92	
Stalk fibre	15	18.9	15.94	16.26
Stalk fibre	15	19.0	16.57	
Stalk fibre	15	14.4	16.33	16.21
Stalk fibre	15	14.9	16.09	

4.4.9 Contact time

The contact time was also checked, it appears that one and a half hours is sufficient to reach equilibrium.

4.4.10 Sample fineness

According to Mangion and Player (1991), a sample in a finer state has a higher Brix-free water value due to the increased surface area per unit mass. It was therefore decided to reduce some fibre samples to a finer state (about 3 mm long) by using a pair of scissors, and to carry out Brix-free water determinations before and after the operation. Results obtained for duplicate analyses of 21 fibre samples are tabulated in Table 4.16. Statistical analysis by the Student's t-test showed that there was no significant difference ($P = 0.89$) between the samples whether in their original or finer states. The standard error of the mean for 21 data points is 0.547 for a mean of 12.82 on the original samples, and 0.500 for a mean of 12.72 on finely cut samples. This shows that increased surface area does not increase the Brix-free water value, contrary to the finding of Mangion and Player (1991).

Table 4.16. Comparison of the Brix-free water of a sample in its original state with that in a finely divided state.

Sample	Brix-free water in the original state/%	Mean/%	Brix-free water of finely cut sample/%	Mean/%
Stalk fibre 1	10.89		10.66	
	10.54	10.72	10.59	10.62
Stalk fibre 2	11.63		10.26	
	11.82	11.73	11.34	10.80
Stalk fibre 3	10.57		12.19	
	10.15	10.36	11.33	11.76
Stalk fibre 4	12.41		12.47	
	12.82	12.61	13.57	13.02
Stalk fibre 5	14.08		12.76	
	14.23	14.16	14.21	13.49
Stalk fibre 6	16.97		16.86	
	16.96	16.96	17.28	17.07
Rind fibre 1	8.57		8.86	
	8.12	8.34	8.67	8.77
Rind fibre 2	8.38		8.89	
	8.52	8.45	9.21	9.05
Rind fibre 3	8.51		8.47	
	8.56	8.54	8.47	8.47
Rind fibre 4	11.53		12.82	
	12.44	11.98	12.22	12.52
Rind fibre 5	13.89		13.66	
	14.60	14.24	13.78	13.72
Rind fibre 6	12.66		11.59	
	12.90	12.78	12.08	11.83
Dry leaf fibre 1	14.96		14.27	
	14.89	14.93	14.38	14.32
Dry leaf fibre 2	12.77		13.08	
	13.14	12.96	13.21	13.15
Dry leaf fibre 3	12.92		12.31	
	12.85	12.89	12.84	12.57
Green leaf fibre 1	13.55		13.63	
	13.10	13.32	13.03	13.33
Green leaf fibre 2	13.32		12.31	
	12.87	13.09	12.56	12.44
Green leaf fibre 3	13.58		14.07	
	13.42	13.50	13.37	13.72
Top fibre 1	14.80		15.39	
	15.91	15.35	15.72	15.55
Top fibre 2	17.03		15.55	
	16.13	16.58	15.36	15.46
Top fibre 3	16.07		15.98	
	15.57	15.82	15.02	15.50

4.4.11 Sample size and precision of the method

In general, the use of 6 g samples and the corresponding 110 g contact solution, with the ratio of contact solution/sample set at 20, had been adopted for all previous Brix-free water determinations. However, with pith sample, a test sample of 6 g is very bulky, making it difficult to dry and handle when filtering after contact with the sucrose solution. The sample size was therefore reduced to 3.5 g in the above experiment. The filtrate obtained was found sufficient to satisfy the test volume requirement of Brix measurement as described in Section 4.4.2.

A sample size of 3.5 g with 75 g contact solution was henceforth adopted for pith samples.

4.5 OTHER METHODS OF DETERMINING BRIX-FREE WATER

So far in the literature and here, the method used to determine Brix-free water in cane fibre involves mixing dried fibre with a solution of 10° Brix sucrose solution. From the increase in Brix as detected by a refractometer, the amount of water absorbed by the fibre is calculated leading to the Brix-free water value of the fibre. In theory, the fibre can be made to contact a solution other than that of sucrose, the increase in the analyte as detected by an appropriate analytical technique, should also lead to the Brix-free water value of the fibre, provided that the technique is sensitive enough to detect the increase in the analyte. In this context, various analytes and techniques were explored, for example, lithium by flame photometry, conductivity by using a conductivity meter, chloride by amperometric potentiometric titrimetry, lactose by high performance ion chromatography and pol by saccharimeter. The details of these experiments are given in Appendix 4.

All these alternative methods (except the polarimetric one) still require further development to give acceptable Brix-free water results. Although the polarimetric method showed promise, it is much more time-consuming and requires special equipment, this is the reason why the refractometric method was preferred in this study.

4.6 EXPERIMENTAL

The following procedure was followed to determine the Brix-free water content in fibres of sugar cane component parts.

4.6.1 Materials

4.6.1.1 *Samples examined*

Brix-free water was determined in duplicate for the three replicates of four cane varieties (R 579, R 570, M 1557/70 and M 1400/86) aged 52, 44 and 36 weeks. For each age, nine cane component parts, namely dry leaf fibre and fines, green leaf fibre and fines, top fibre, rind fibre and fines and stalk fibre and pith obtained as described in Section 3.4.3 were studied.

For comparison, those nine component parts of R 570 aged 52, 44 and 37 weeks harvested in 2001 were also studied.

4.6.1.2 *10° Brix sucrose solution*

Refined white sugar (100 g) was weighed into a 1 L volumetric flask, followed by distilled water to make up to mark. The solution was preserved with mercuric iodide solution at the rate of 0.5 mL L^{-1}. The mercury iodide solution was prepared as described in Chapter 2, Section 2.1.1.1.

4.6.2 Equipment

Glass bottles of a nominal size of 250 mL with a plastic stopper were found suitable as sample bottles for sample drying and contacting with the sucrose solution.

A glass rod with a rounded button end (Fig 4.3) was used to squeeze out solution from fibre for filtration. This rod is readily obtainable from suppliers of laboratory equipment.

Figure 4.3. Glass rod with button-end.

A two-decimal place Mettler model PJ 3600 analytical balance was used for all mass determinations.

A Gallenkamp vacuum oven with a power rating of 1000 W and a capacity of 31 L connected to a Fisons single stage vacuum pump of 50 Hz and 185 W was used to dry the samples. Dishes containing silica gel were placed in the oven to absorb moisture (see Fig 4.4).

Figure 4.4. Fibre samples drying in the Gallenkamp vacuum oven.
Take note of the dish containing silica gel placed below the sample bottles.

A sugar refractometer was used to measure the refractive index of the sucrose solutions and to convert the readings to dissolved solids (g) in 100 g solution (Brix).

4.6.3 Method for Brix-free water determination in cane fibres

About 3.5 g of the fibre sample was weighed out in three pre-weighed glass bottles of 250 mL nominal size with the stopper on. One served as the sample blank and the other two as duplicate tests. The bottles without the stoppers were then put in an air-oven at 65 °C for one hour. During the drying period, the samples were stirred from time to time (particularly with fine samples such as pith) after which they were dried in a vacuum oven at 65 °C for 16 hours under 875 mbar vacuum. The bottles were then removed from the vacuum oven, well stoppered, cooled and weighed to determine the mass of the samples. To one bottle, about 75 g distilled water was quickly added, followed by the addition of 75 g, 10° Brix sucrose solution to the other two bottles. The mass of the distilled water and sucrose solution added was accurately determined by re-weighing the bottles.

The bottles were shaken every 10 minutes for one and a half hours to reach equilibrium. The solutions were then filtered through Whatman 91 filter paper in a funnel which was covered to prevent evaporation. The first few mL of the filtrate was rejected. The use of a glass rod with button end helped to squeeze out the solution for filtration (see Fig 4.5).

The Brix of the filtrates together with the original 10° Brix sucrose solution were determined. For each portion of the filtrate poured into the refractometer, three Brix readings were taken and an average recorded. Three such averages were recorded after

examination of three portions of each filtrate. For the original contact solution, six average values were recorded, three at the beginning and three at the end of the Brix determination. All Brix values were corrected for instrument zero by using distilled water.

Figure 4.5. Use of glass rod with button-end to squeeze out solution.

If w_1 and w_3 are the mass of sample in the blank and test solution respectively, and w_2 is the mass of distilled water in the blank, and w_4 the mass of the contact solution in the test solution; p_1 and p_2 are the Brix of the sample blank before and after equilibration, and p_3 and p_4 are the Brix of the test solution before and after equilibration, the corrected blank $b = p_2w_2w_3/(w_1w_4)$, the net Brix increase $p = p_4 - b$, and the Brix-free water in the sample = $[100w_4 (1 - p_3 p^{-1})]/w_3$.

(Note: A series of twelve samples could be determined in one batch.)

A number of precautions were taken:

- If a sample size smaller than 3 g was used, the mass of the contact solution and distilled water was adjusted proportionately so as to keep the ratio of the contact solution/sample at about 20.
- During the cooling process, the stopper was kept well pushed in to ensure that no air entered the bottle before the introduction of the contact solution, since cellulose fibres are avid absorbers of moisture; otherwise, the results will be under-estimated.
- Since stalk pith is fluffy and light, 3.5 g occupied a large volume in the bottle. To ensure that it was well dried, it proved better to have the bottle lying down in the oven rather than standing up, as there was more drying surface for the sample.

- One large fibre sample (rind) of known Brix-free water value (by accurate determination) was used as a control sample. If for some reason, the value re-determined differed by more than 2 units from the value first determined, the whole batch of samples was re-analysed.
- The silica gel used in the vacuum oven was re-generated each time the vacuum oven was used.
- Since the weighing of the bottles and fibre samples extended over two days, the analytical balance used was calibrated against a standard mass every day before use.

4.7 RESULTS AND DISCUSSION

The duplicate Brix-free water results of the nine cane components (dry leaf fibre and fines, green leaf fibre and fines, top fibre, rind fibre and fines, and stalk fibre and pith) of triplicate cane samples of four cane varieties and three ages are presented in Tables 4.17 to 4.21. The tables also show the average of both the duplicate analytical results and the replications.

The duplicate Brix-free water results of the nine cane components of triplicate cane samples of the variety R 570 of three ages obtained in 2001 are also presented in Tables 4.17 to 4.21.

4.7.1 Raw data

The raw data leading to the Brix-free water values are presented on CD (file: BFW test series.xls) and for the R 570 variety of 2001 (file: BFW R 570 of 2001.xls). Results of those component parts marked in bold were analysed again at the end of the component series, and shown in Tables 4.17 – 4.21..

4.7.2 Sample code

The samples aged 52, 44 and 36 weeks are designated as 1, 2 and 3 respectively, followed by the types of samples, dry leaf (D), green leaf (G), tops (T), rind (R) and stalk (S). The three replicates of the first of the four cane varieties harvested in 2003, R 579 are represented by 1, 2 and 3, those of the second variety R 570 by 4, 5 and 6, the third variety M 1557/70 by 7, 8 and 9 and the last variety M 1400/86 by 10, 11 and 12 and those of the R 570 harvested in 2001 by 13, 14 and 15. Fibre is designated by F, pith by P and fines by f: thus 2/R5/f is rind fines aged 44 weeks of the second replicate of the second variety R 570 harvested in 2003.

Table 4.17. Brix-free water values/% for **dry leaf** fibres (fibre F and fines f) of triplicate cane samples of four cane varieties and three ages.

Sample*		52 weeks				44 weeks				36 weeks			
		F	mean	f	mean	F	mean	f	mean	F	mean	f	mean
R 579	1	14.14	**14.92**	16.07	**16.86**	15.69	**15.81**	15.06	**15.63**	14.98	**15.57**	16.42	**16.37**
		15.69		17.64		15.93		16.19		16.16		16.32	
	2	15.24	**15.11**	16.76	**17.16**	15.78	**15.50**	15.56	**15.43**	14.41	**14.77**	15.35	**15.45**
		14.98		17.56		15.22		15.29		15.12		15.54	
	3	15.67	**15.77**	17.11	**17.03**	14.63	**14.94**	17.26	**17.79**	14.54	**14.36**	15.19	**15.94**
		15.86		16.95		15.25		18.31		14.18		16.68	
			15.26		**17.02**		**15.42**		**16.28**		**14.90**		**15.92**
R 570	1	14.15	**14.57**	17.59	**17.31**	15.00	**14.52**	16.49	**16.27**	14.59	**14.44**	17.85	**17.78**
		14.98		17.03		14.04		16.04		14.28		17.70	
	2	15.68	**15.73**	15.68	**16.55**	16.47	**17.10**	14.45	**14.42**	14.47	**14.85**	16.69	**16.75**
		15.77		17.41		17.72		14.39		15.22		16.81	
	3	15.25	**14.79**	17.26	**17.36**	14.97	**15.28**	18.15	**17.40**	14.59	**14.51**	15.61	**15.11**
		14.32		17.46		15.58		16.65		14.43		14.60	
			15.03		**17.07**		**15.63**		**16.03**		**14.60**		**16.54**
M 1557/70	1	15.74	**15.52**	13.77	**13.94**	14.61	**15.41**	13.56	**14.26**	15.19	**15.30**	19.98	**19.76**
		15.29		14.11		16.21		14.95		15.40		19.54	
	2	15.55	**15.50**	14.78	**14.64**	14.09	**14.28**	13.19	**13.45**	15.43	**15.27**	16.75	**16.84**
		15.44		14.49		14.47		13.71		15.11		16.92	
	3	14.01	**14.43**	15.43	**15.07**	13.77	**14.05**	17.18	**17.02**	13.67	**14.69**	15.29	**15.40**
		14.84		14.70		14.32		16.86		15.71		15.51	
			15.15		**14.55**		**14.58**		**14.91**		**15.09**		**17.33**
M 1400/86	1	15.02	**14.39**	12.02	**12.85**	14.45	**14.51**	15.03	**15.08**	16.29	**16.07**	18.00	**17.58**
		13.75		13.67		14.57		15.12		15.85		17.15	
	2	13.17	**13.43**	13.34	**13.28**	15.08	**15.17**	13.41	**14.07**	14.25	**14.87**	14.56	**15.00**
		13.68		13.22		15.26		14.73		15.48		15.43	
	3	13.51	**13.65**	16.36	**16.45**	14.00	**13.97**	16.88	**16.49**	17.17	**17.06**	14.25	**14.45**
		13.78		16.53		13.94		16.10		16.95		14.65	
			13.82		**14.19**		**14.55**		**15.21**		**16.00**		**15.67**
R 570 (2001)	1	14.78	**14.28**	14.86	**15.13**	16.20	**16.46**	18.47	**17.66**	12.91	**13.64**	15.55	**14.79**
		13.78		15.39		16.71		16.84		14.37		14.03	
	2	14.31	**14.55**	14.48	**14.58**	16.85	**16.93**	14.21	**14.76**	15.31	**15.12**	12.32	**12.80**
		14.78		14.68		17.01		15.31		14.92		13.27	
	3	14.26	**14.42**	14.99	**14.00**	15.95	**15.95**	13.67	**13.67**	14.06	**14.75**	13.46	**14.07**
		14.57		13.00		-		-		15.44		14.67	
			14.41		**14.57**		**16.45**		**15.36**		**14.50**		**13.88**

* Samples were harvested in 2003 except where indicated

Table 4.18. Brix-free water values/% for **green leaf** fibres (fibre F and fines f) of triplicate cane samples of four cane varieties and three ages.

Sample*		52 weeks				44 weeks				36 weeks			
		F	mean	f	mean	F	mean	f	mean	F	mean	f	mean
R 579	1	13.27	**14.00**	14.19	**14.41**	12.72	**12.95**	11.69	**12.07**	13.29	**13.77**	13.32	**13.17**
		14.73		14.62		13.18		12.45		14.25		13.02	
	2	14.14	**14.16**	12.52	**12.43**	14.69	**14.60**	13.21	**13.97**	14.57	**14.18**	15.58	**16.04**
		14.18		12.33		14.50		14.72		13.78		16.50	
	3	13.25	**13.83**	14.04	**13.55**	12.17	**12.70**	14.25	**14.02**	13.85	**13.39**	17.08	**17.44**
		14.41		13.05		13.23		13.78		12.92		17.80	
			14.00		**13.46**		**13.42**		**13.35**		**13.78**		**15.55**
R 570	1	13.01	**12.53**	15.31	**14.60**	14.96	**14.39**	15.21	**15.00**	12.95	**12.44**	15.63	**14.88**
		12.05		13.89		13.81		14.79		11.93		14.13	
	2	13.53	**13.53**	13.18	**13.24**	11.28	**12.12**	11.31	**11.11**	11.97	**12.29**	14.19	**14.55**
		13.52		13.29		12.95		10.90		12.60		14.91	
	3	11.76	**11.96**	13.62	**13.23**	12.33	**12.35**	10.33	**11.07**	12.79	**12.85**	15.93	**15.87**
		12.16		12.84		12.36		11.81		12.91		15.81	
			12.67		**13.69**		**12.95**		**12.39**		**12.53**		**15.10**
M 1557/70	1	14.37	**14.85**	15.64	**16.10**	12.98	**13.43**	15.91	**15.55**	13.75	**13.45**	13.55	**13.41**
		15.33		16.56		13.88		15.19		13.15		13.26	
	2	15.23	**15.28**	16.89	**17.87**	11.61	**12.34**	17.54	**17.64**	13.32	**13.55**	14.65	**14.67**
		15.32		18.84		13.06		17.73		13.77		14.69	
	3	15.10	**15.03**	17.08	**18.09**	13.20	**13.64**	18.31	**17.90**	12.28	**13.02**	16.02	**15.68**
		14.95		19.10		14.07		17.49		13.75		15.34	
			15.05		**17.35**		**13.13**		**17.03**		**13.34**		**14.59**
M 1400/86	1	14.54	**14.48**	14.30	**14.90**	13.45	**13.18**	15.27	**15.42**	13.80	**13.45**	13.37	**12.86**
		14.41		15.49		12.91		15.56		13.10		12.34	
	2	14.61	**14.79**	14.84	**14.94**	12.60	**12.64**	14.27	**14.70**	12.03	**12.35**	12.96	**13.13**
		14.97		15.03		12.68		15.12		12.67		13.29	
	3	13.59	**14.01**	13.99	**14.75**	13.87	**13.91**	14.93	**15.15**	12.49	**12.59**	14.22	**13.94**
		14.43		15.50		13.94		15.37		12.69		13.65	
			14.43		**14.86**		**13.24**		**15.09**		**12.80**		**13.31**
R 570 (2001)	1	14.22	**14.16**	14.11	**13.74**	14.24	**14.18**	15.77	**15.03**	14.58	**14.97**	14.80	**14.35**
		14.09		13.36		14.11		14.28		15.36		13.89	
	2	14.72	**14.48**	11.87	**12.19**	13.93	**13.95**	14.39	**14.60**	15.10	**15.16**	14.47	**15.15**
		14.23		12.50		13.97		14.81		15.22		15.82	
	3	14.23	**14.06**	11.80	**11.82**	15.00	**15.08**	16.01	**15.92**	14.62	**14.89**	15.74	**15.75**
		13.89		11.84		15.16		15.83		15.15		15.75	
			14.23		**12.58**		**14.40**		**15.18**		**15.01**		**15.08**

* Samples were harvested in 2003 except where indicated

Table 4.19. Brix-free water values/% for top fibres (fibre F) of triplicate cane samples of four cane varieties and three ages.

Sample*		52 weeks		44 weeks		36 weeks	
		F	mean	F	mean	F	mean
R 579	1	14.48	**14.51**	14.43	**15.24**	13.72	**14.07**
		14.54		16.05		14.41	
	2	15.73	**15.73**	16.41	**16.36**	14.15	**14.83**
		15.72		16.31		15.51	
	3	13.77	**14.59**	16.28	**16.85**	14.82	**14.30**
		15.40		17.42		13.77	
			14.94		**16.15**		**14.40**
R 570	1	15.75	**16.13**	16.55	**16.52**	15.86	**15.59**
		16.50		16.48		15.31	
	2	14.58	**14.82**	16.08	**16.30**	14.22	**13.69**
		15.06		16.51		13.16	
	3	15.30	**16.03**	16.53	**16.68**	14.89	**14.87**
		16.75		16.82		14.85	
			15.66		**16.50**		**14.72**
M 1557/70	1	13.30	**13.64**	14.62	**15.39**	15.42	**15.75**
		13.98		16.16		16.07	
	2	15.00	**15.68**	17.28	**16.36**	16.21	**16.52**
		16.36		15.44		16.83	
	3	15.19	**15.87**	17.49	**17.47**	15.28	**15.16**
		16.54		17.45		15.03	
			15.06		**16.41**		**15.81**
M 1400/86	1	13.97	**13.99**	13.90	**14.05**	15.46	**15.84**
		14.01		14.19		16.22	
	2	12.56	**12.27**	14.28	**14.27**	11.76	**12.60**
		11.97		14.26		13.43	
	3	13.55	**13.63**	16.22	**15.89**	13.41	**13.53**
		13.71		15.56		13.64	
			13.30		**14.74**		**13.99**
R 570 (2001)	1	14.10	**14.36**	11.56	**11.56**	13.66	**14.13**
		14.61		-		14.59	
	2	17.03	**16.79**	13.14	**13.09**	14.14	**14.34**
		16.54		13.04		14.54	
	3	15.38	**16.41**	13.74	**13.74**	13.13	**13.13**
		17.43		-		13.13	
			15.85		**12.80**		**13.87**

* Samples were harvested in 2003 except where indicated

Table 4.20. Brix-free water values/% for **rind** fibres (fibre F and fines f) of triplicate cane samples of four cane varieties and three ages.

Sample*		52 weeks				44 weeks				36 weeks			
		F	mean	f	mean	F	mean	f	mean	F	mean	f	mean
R 579	1	10.72	**11.58**	16.41	**15.76**	11.39	**11.87**	13.54	**13.02**	11.83	**12.24**	15.50	**15.53**
		12.43		15.11		12.34		12.49		12.65		15.56	
	2	12.09	**12.10**	16.71	**16.66**	13.72	**13.24**	11.40	**11.54**	12.25	**12.08**	16.55	**17.05**
		12.10		16.61		12.75		11.67		11.90		17.54	
	3	11.37	**11.95**	15.34	**14.89**	11.78	**12.04**	15.58	**15.47**	11.04	**11.29**	18.43	**19.08**
		12.53		14.44		12.29		15.35		11.53		19.73	
			11.87		**15.77**		**12.38**		**13.34**		**11.87**		**17.22**
R 570	1	10.85	**10.80**	9.69	**10.00**	11.42	**11.66**	11.15	**11.01**	12.12	**12.32**	16.02	**16.16**
		10.74		10.30		11.90		10.87		12.52		16.30	
	2	10.52	**10.82**	10.79	**10.84**	10.24	**11.14**	11.24	**11.18**	12.35	**12.63**	13.64	**13.66**
		11.12		10.88		12.04		11.12		12.90		13.68	
	3	11.07	**11.15**	9.56	**10.27**	10.65	**10.92**	11.64	**11.37**	12.65	**12.71**	13.73	**13.62**
		11.23		10.98		11.19		11.09		12.76		13.51	
			10.92		**10.37**		**11.24**		**11.19**		**12.55**		**14.48**
M 1557/70	1	10.26	**11.09**	12.33	**12.48**	14.56	**14.55**	13.87	**13.61**	12.17	**12.25**	17.04	**16.90**
		11.91		12.62		14.53		13.35		12.32		16.76	
	2	15.05	**15.37**	15.67	**16.42**	14.82	**15.12**	15.58	**15.43**	11.93	**12.03**	14.75	**13.93**
		15.68		17.17		15.42		15.27		12.13		13.10	
	3	11.70	**12.38**	14.14	**13.92**	13.85	**13.79**	13.54	**13.80**	13.13	**13.61**	14.27	**13.62**
		13.05		13.69		13.72		14.05		14.08		12.96	
			12.94		**14.27**		**14.48**		**14.28**		**12.63**		**14.81**
M 1400/86	1	13.05	**13.31**	16.07	**16.35**	11.01	**11.13**	13.67	**13.17**	11.89	**12.15**	15.18	**14.81**
		13.56		16.62		11.24		12.67		12.40		14.44	
	2	11.87	**12.01**	13.20	**13.28**	10.04	**10.85**	10.89	**10.90**	12.20	**12.24**	13.13	**13.92**
		12.15		13.36		11.66		10.91		12.27		14.71	
	3	11.98	**12.41**	14.33	**13.89**	10.19	**10.60**	11.32	**11.50**	12.91	**12.98**	15.32	**15.00**
		12.83		13.44		11.00		11.67		13.04		14.68	
			12.57		**14.50**		**10.86**		**11.86**		**12.45**		**14.58**
R 570 (2001)	1	12.35	**12.58**	14.14	**13.49**	14.53	**15.05**	17.68	**16.68**	11.51	**11.77**	16.33	**15.84**
		12.81		12.84		15.56		15.68		12.02		15.34	
	2	13.33	**13.38**	13.38	**13.30**	14.71	**15.07**	12.87	**12.93**	13.55	**13.68**	16.14	**16.04**
		13.43		13.21		15.42		12.99		13.80		15.94	
	3	14.36	**14.56**	12.22	**12.32**	13.86	**14.47**	14.28	**15.14**	12.52	**12.75**	13.15	**13.01**
		14.76		12.41		15.08		16.00		12.97		12.86	
			13.51		**13.03**		**14.86**		**14.92**		**12.73**		**14.96**

* Samples were harvested in 2003 except where indicated

Table 4.21. Brix-free water values/% for **stalk** fibres (fibre F and pith P) of triplicate cane samples of four varieties and three ages.

Sample*		52 weeks				44 weeks				36 weeks			
		F	mean	P	mean	F	mean	P	mean	F	mean	P	mean
R 579	1	9.84	**10.55**	23.89	**23.94**	11.10	**10.67**	17.81	**18.09**	14.27	**14.42**	19.47	**20.41**
		11.26		23.99		10.23		18.36		14.57		21.35	
	2	10.20	**10.25**	22.98	**22.74**	12.16	**11.29**	16.71	**16.98**	14.23	**14.36**	22.31	**21.65**
		10.30		22.50		10.41		17.25		14.49		20.99	
	3	10.32	**10.88**	26.58	**26.18**	10.64	**10.84**	19.36	**19.43**	13.19	**14.05**	18.97	**18.80**
		11.43		25.78		11.04		19.49		14.90		18.63	
			10.56		**24.29**		**10.93**		**18.16**		**14.28**		**20.29**
R 570	1	10.83	**10.84**	26.87	**26.60**	13.26	**12.81**	18.34	**18.73**	13.73	**13.50**	22.91	**22.74**
		10.84		26.32		12.36		19.12		13.26		22.56	
	2	11.75	**11.09**	23.41	**23.13**	13.77	**13.72**	17.37	**18.01**	13.48	**13.23**	20.31	**20.82**
		10.42		22.84		13.66		18.64		12.98		21.32	
	3	11.69	**11.89**	21.33	**22.13**	13.21	**12.26**	18.31	**18.36**	12.95	**13.07**	20.37	**20.60**
		12.09		22.93		11.31		18.41		13.19		20.82	
			11.27		**23.95**		**12.93**		**18.37**		**13.27**		**21.38**
M 1557/70	1	13.81	**14.19**	18.89	**18.59**	11.37	**11.52**	16.12	**17.07**	15.83	**16.36**	16.61	**17.49**
		14.57		18.29		11.67		18.01		16.89		18.36	
	2	13.56	**13.38**	23.27	**23.33**	12.77	**13.12**	19.56	**19.69**	15.66	**15.90**	18.01	**18.25**
		13.20		23.38		13.46		19.82		16.13		18.49	
	3	11.74	**11.66**	19.97	**20.46**	12.59	**12.79**	16.36	**16.81**	12.64	**13.04**	15.67	**16.45**
		11.57		20.95		12.98		17.25		13.44		17.23	
			13.08		**20.79**		**12.47**		**17.85**		**15.10**		**17.40**
M 1400/86	1	14.21	**14.24**	23.70	**23.30**	14.10	**14.31**	19.44	**19.62**	13.96	**14.53**	21.10	**20.26**
		14.27		22.89		14.51		19.79		15.09		19.42	
	2	12.41	**12.73**	19.73	**20.42**	13.63	**13.25**	18.24	**18.97**	14.84	**14.99**	18.85	**19.13**
		13.05		21.11		12.86		19.69		15.13		19.41	
	3	9.98	**10.55**	21.62	**21.90**	13.60	**12.59**	17.92	**16.96**	14.20	**14.02**	16.94	**16.72**
		11.11		22.17		11.57		16.00		13.83		16.50	
			12.51		**21.87**		**13.38**		**18.51**		**14.51**		**18.70**
R 570 (2001)	1	11.62	**10.79**	19.19	**17.86**	12.21	**13.01**	17.85	**17.82**	9.57	**9.37**	14.09	**14.09**
		9.96		16.53		13.81		17.78		9.17		14.09	
	2	11.01	**10.82**	15.01	**16.22**	14.18	**14.07**	18.55	**18.22**	14.30	**14.30**	11.36	**11.76**
		10.63		17.43		13.95		17.89		14.29		12.15	
	3	11.37	**12.27**	15.96	**16.49**	15.20	**14.90**	20.20	**19.74**	11.39	**10.38**	12.30	**12.27**
		13.16		17.02		14.60		19.27		9.36		12.23	
			11.29		**16.86**		**13.99**		**18.59**		**11.35**		**12.70**

* Samples were harvested in 2003 except where indicated

4.7.3 Statistical analysis

It is essential to establish, first of all, whether the difference in the duplicate results is significant.

4.7.3.1 *Validity of the duplicate analyses*

Calculations were carried out by using the one sample Student's t-test with the null hypothesis that the mean of the duplicate analytical results is equal to zero. All the duplicate results, for both fibre and fines of dry leaf in Table 4.17 were thus examined. The variance, standard deviation and standard error of the mean are shown in Table 4.22. The data for green leaf, top (where only fibre data are available), rind and stalk given in Tables 4.18 – 4.21 were similarly examined and the statistical results are also shown in Table 4.22.

The results from the Student's t-test show that the duplicate results are not different.

Table 4.22. One sample Student t-test results for duplicate analyses of Brix-free water.

Dry leaf					
Sample	Size	Mean	Variance	Standard deviation	Standard error of mean
BFW1-BFW2	72	-0.2383	0.6064	0.7787	0.09177
		95% confidence interval for mean: (-0.4213, -0.05535)			
Green leaf					
Sample	Size	Mean	Variance	Standard deviation	Standard error of mean
BFW1-BFW2	72	-0.2092	0.7129	0.8443	0.09951
		95% confidence interval for mean: (-0.4076, -0.01076)			
Top					
Sample	Size	Mean	Variance	Standard deviation	Standard error of mean
BFW1-BFW2	36	-0.3603	0.715	0.8456	0.1409
		95% confidence interval for mean: (-0.6464, -0.07418)			
Rind					
Sample	Size	Mean	Variance	Standard deviation	Standard error of mean
BFW1-BFW2	72	-0.2322	0.5892	0.7676	0.09046
		95% confidence interval for mean: (-0.4126, -0.05185)			
Stalk					
Sample	Size	Mean	Variance	Standard deviation	Standard error of mean
BFW1-BFW2	72	-0.1849	0.8944	0.9457	0.1115
		95% confidence interval for mean: (-0.4071, 0.03737)			

The analytical results shown in Tables 4.17-4.21 were analysed statistically by making use of GENSTAT for Windows (Version 8.0). The objective of this analysis was to determine the effect of replication in the field, cane variety, size of cane component (fibre or fines/pith), age, replication of their interactions, cane component parts and location of crop growth, on the Brix-free water content.

The results of the analyses of variance are shown in Table 4.23. Statistical significance refers to $P < 0.05$(*), $P < 0.01$ (**) and $P < 0.001$ (***) levels.

4.7.3.2 *Replication*

The three replicates in the field did not differ significantly in the Brix-free water content of dry leaf fibre and fines, green leaf fibre and fines, top fibre and rind fibre and fines, whereas in the case of stalk fibre and pith, the difference was highly significant ($P < 0.001$). This is because the Brix-free water values of pith in certain replicates of all cane varieties at 52 weeks were high, notably the 3rd replicate of R 579, the 1st replicate of R 570, the 2nd replicate of M 1557/70 and the 1st replicate of M 1400/86. These replicates were in a flowering state and had probably developed pithiness which led to higher Brix-free water values.

4.7.3.3 *Cane variety*

When the fibre and fines of each component part as well as all the three ages were considered together, there was a highly significant main effect of cane variety ($P < 0.001$) for all component parts except stalk indicating that the Brix-free water in dry leaf is different in the four cane varieties, similarly for green leaf, top and rind. However in stalk, when the combined effect of fibre and pith and the three ages were considered, the four cane varieties were not different.

4.7.3.4 *Size: fibre or fines*

When all the four cane varieties and all the three ages were considered together, there was a highly significant ($P < 0.001$) main effect of size (whether fibre or fines) on the Brix-free water values in all components examined. The fines of a cane component part generally have about a unit higher Brix-free water value than the corresponding fibres except in stalk where the difference is much larger.

Table 4.23. Analysis of variance (Brix-free water of five component fibres, four varieties and three ages).

Variate: BFW (dry leaf)					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	4.871	2.435	2.14	**NS**
Variety	3	21.015	7.005	6.15	* * *
Age	2	6.968	3.484	3.06	*
Size	1	28.676	28.676	25.17	* * *
Variety.Age	6	33.859	5.643	4.95	* * *
Variety.Size	3	8.215	2.738	2.4	**NS**
Age.Size	2	2.604	1.302	1.14	**NS**
Variety.Age.Size	6	18.047	3.008	2.64	*
Residual	118	134.458	1.139		
Total	143	258.713			
Variate: BFW (green leaf)					
Source of variation	d.f.	s.s.	m.s.	v.r.	**F pr.**
Rep stratum	2	1.0407	0.5203	0.53	**NS**
Variety	3	63.9123	21.3041	21.5	* * *
Age	2	11.1698	5.5849	5.64	* *
Size	1	52.1043	52.1043	52.57	* * *
Variety.Age	6	53.3321	8.8887	8.97	* * *
Variety.Size	3	21.6693	7.2231	7.29	* * *
Age.Size	2	3.2411	1.6205	1.64	**NS**
Variety.Age.Size	6	34.8555	5.8093	5.86	* * *
Residual	118	116.9474	0.9911		
Total	143	358.2726			
Variate: BFW (top)					
Source of variation	d.f.	s.s.	m.s.	v.r.	**F pr.**
Rep stratum	2	2.6806	1.3403	1.44	**NS**
Variety	3	34.2414	11.4138	12.23	* * *
Age	2	23.5971	11.7985	12.64	* * *
Variety.Age	6	7.2579	1.2097	1.3	**NS**
Residual	58	54.1214	0.9331		
Total	71	121.8983			
Variate: BFW (rind)					
Source of variation	d.f.	s.s.	m.s.	v.r.	**F pr.**
Rep stratum	2	0.212	0.106	0.09	**NS**
Variety	3	102.602	34.201	27.76	* * *
Age	2	46.892	23.446	19.03	* * *
Size	1	98.903	98.903	80.27	* * *
Variety.Age	6	67.59	11.265	9.14	* * *
Variety.Size	3	43.531	14.51	11.78	* * *
Age.Size	2	36.73	18.365	14.91	* * *
Variety.Age.Size	6	14.66	2.443	1.98	**NS**
Residual	118	145.387	1.232		
Total	143	556.505			
Variate: BFW (stalk)					
Source of variation	d.f.	s.s.	m.s.	v.r.	**F pr.**
Rep stratum	2	23.368	11.684	7.92	* * *
Variety	3	10.488	3.496	2.37	**NS**
Age	2	102.388	51.194	34.72	* * *
Size	1	1905.104	1905.104	1292.17	* * *
Variety.Age	6	16.044	2.674	1.81	**NS**
Variety.Size	3	97.102	32.367	21.95	* * *
Age.Size	2	235.477	117.739	79.86	* * *
Variety.Age.Size	6	38.068	6.345	4.3	* * *
Residual	118	173.973	1.474		
Total	143	2602.012			

NS	Not significant
*	$P < 0.05$
**	$P < 0.01$
***	$P < 0.001$

4.7.3.5 *Age*

When the fibre and fines of each component part, as well as the four cane varieties were considered together, there was a slightly significant effect ($P < 0.05$) of age on the Brix-free water in dry leaf, slightly more significant ($P < 0.01$) in green leaf and highly significant ($P < 0.001$) in top, rind and stalk.

4.7.3.6 *Interaction effects*

The interaction effects of variety x age, variety x size, age x size and variety x age x size are examined below.

4.7.3.6.1 Variety x age

When the fibre and fines of a cane component part were considered together, the interaction effect of variety x age was highly significant ($P < 0.001$) for the Brix-free water in dry leaf, green leaf and rind, but not significant in top and stalk, indicating that genotypes were affected differently with age in respect of dry leaf, green leaf and rind, but not in top and stalk.

4.7.3.6.2 Variety x size

When all three ages were considered together, the interaction effect of variety x size was not significant for the Brix-free water in dry leaf, but highly significant ($P < 0.001$) in green leaf, rind and stalk.

4.7.3.6.3 Age x size

When all four cane varieties were considered together, the interaction effect of age x size was not significant for the Brix-free water in both dry and green leaves, but was highly significant ($P < 0.001$) in rind and stalk.

4.7.3.6.4 Variety x age x size

The interaction effect of variety x age x size was highly significant ($P < 0.001$) for the Brix-free water in green leaf and stalk, slightly significant ($P < 0.05$) for dry leaf and not significant in rind.

At 52 weeks old, the stalk fibre of M 1557/70 had the highest Brix-free water value of 13.08%, and R 579 had the lowest (10.56%); the opposite was true for stalk pith, M 1557/70 had the lowest (20.79%) and R 579 had the highest (24.29%). Also, the rind fibre of M 1557/70 had the highest Brix-free water value of 12.94%. It is also worth

pointing out that at 52 weeks old, dry leaf fibre of R 579 had the highest Brix-free water value of 15.26%, and dry leaf fines of this cane variety also had high Brix-free water (17.02%).

4.7.3.7 *Cane component parts*

The analytical data given in Tables 4.17, 4.18, 4.20 and 4.21 for dry leaf, green leaf, rind and stalk were examined to answer the question whether the fibres from these cane component parts are different for the four cane varieties and three ages and the same question applies to their fines. Cane top was not included in this study, since only its fibre was analysed. A highly significant difference ($P < 0.001$) was found for both fibre and fines in the main effects of variety, age and component parts, as well as in the interaction effects of variety x age, variety x component part, age x component part and variety x age x component part.

When dry leaf was excluded in the statistical analysis, since there was insufficient material from the four stalks sampled and additional material had to be collected from the same replication in the field (Section 3.2), the results (Table 4.24) showed highly significant differences in all the above cited parameters except that the interaction effect of variety x age x component part was highly significant ($P < 0.001$) for fibres of green leaf, rind and stalk, and significant ($P < 0.01$) for fines of green leaf, rind and stalk.

4.7.3.8 *Crop location*

The statistical analysis of the data presented in Tables 4.17 – 4.21 for the nine cane components of triplicate cane samples of the variety R 570 of three ages obtained in 2001 and 2003 is shown in Table 4.25.

It is most relevant that the stalks of R 570 grown in different locations, i.e. Mon Trésor sampled in 2001 and Nouvelle Industrie sampled in 2003 show a highly significant difference ($P < 0.001$) in the main effects of crop and size, i.e. fibre or pith; and in the interaction effects of crop x age, crop x size, age x size and crop x age x size. The difference in the main effect of age is significant ($P < 0.01$). The difference in the replications in the field was not significant.

It is worth mentioning that the rind also showed a highly significant difference ($P < 0.001$) in all the effects except that of crop x size and crop x age x size.

Table 4.24. Analysis of variance (green leaf, rind and stalk).

Fibre – Green leaf, rind and stalk					
Variate: BFW					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	8.0882	4.0441	5.73	* *
Variety	3	52.9153	17.6384	25	* * *
Age	2	18.5154	9.2577	13.12	* * *
Component part	2	52.9668	26.4834	37.53	* * *
Variety.Age	6	16.4209	2.7368	3.88	* * *
Variety.Component part	6	26.8301	4.4717	6.34	* * *
Age.Component part	4	72.9782	18.2446	25.85	* * *
Variety.Age.Component part	12	47.0724	3.9227	5.56	* * *
Residual	178	125.608	0.7057		
Total	215	421.3953			
Fines – Green leaf, rind and stalk					
Variate: BFW					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	1.311	0.656	0.36	**NS**
Variety	3	45.571	15.19	8.3	* * *
Age	2	146.743	73.372	40.07	* * *
Component part	2	1670.723	835.361	456.23	* * *
Variety.Age	6	102.215	17.036	9.3	* * *
Variety.Component part	6	213.988	35.665	19.48	* * *
Age.Component part	4	197.661	49.415	26.99	* * *
Variety.Age.Component part	12	58.841	4.903	2.68	* *
Residual	178	325.919	1.831		
Total	215	2762.973			

NS Not significant
* $P < 0.05$
** $P < 0.01$
*** $P < 0.001$

Average rainfall, maximum, minimum and mean temperatures during the growth cycle of R 570 aged 52 weeks were 115 mm, 27.3, 20.6 and 23.9 °C for samples harvested in 2001 and 106 mm, 27.8, 19.5 and 23.6 °C for samples harvested in 2003 respectively. For R 570 aged 44 weeks, they were 131 mm, 27.6, 21.0 and 24.3 °C for samples harvested in 2001 at 113 mm, 28.4, 19.9 and 24.1 °C; and for R 570 aged 36 weeks, they were 145 mm, 28.3, 21.6 and 25.0 °C for samples harvested in 2001 and 131 mm, 28.7, 20.4 and 24.5 °C. The growing conditions of the two crops of R 570 with respect to rainfall, maximum, minimum and mean temperatures are therefore comparable.

The crop of R 570 sampled in 2001 was grown at Mon Trésor (see Fig 1.1) on a Latosolic Reddish Prairie P3 (slightly weathered and shallow) soil, while that sampled in 2003 was grown at Nouvelle Industrie on a Low Humic Latosol L2 soil. Classifications of mineral resources and soil types in Mauritius have been described by Simpson (1951) and Parish and Feillafé (1965).

Table 4.25. Analysis of variance (two crops).

Variate: BFW (dry leaf)					
Source of variation	d.f.(mv.)	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	1.67	0.835	0.7	**NS**
Crop	1	15.929	15.929	13.37	* * *
Age	2	16.27	8.135	6.83	* *
Size	1	3.927	3.927	3.3	**NS**
Crop.Age	2	10.104	5.052	4.24	*
Crop.Size	1	14.692	14.692	12.33	* * *
Age.Size	2	6.769	3.385	2.84	**NS**
Crop.Age.Size	2	1.943	0.972	0.82	**NS**
Residual	56(2)	66.729	1.192		
Total	69(2)	137.174			
Variate: BFW (green leaf)					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	5.4476	2.7238	3.22	*
Crop	1	25.5732	25.5732	30.24	* * *
Age	2	15.7134	7.8567	9.29	* * *
Size	1	2.5051	2.5051	2.96	**NS**
Crop.Age	2	10.8045	5.4023	6.39	* *
Crop.Size	1	7.3408	7.3408	8.68	* *
Age.Size	2	8.6919	4.346	5.14	* *
Crop.Age.Size	2	15.3934	7.6967	9.1	* * *
Residual	58	49.0449	0.8456		
Total	71	140.5148			
Variate: BFW (rind)					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	2.4945	1.2473	1.9	**NS**
Crop	1	87.186	87.186	132.82	* * *
Age	2	36.3639	18.1819	27.7	* * *
Size	1	4.7381	4.7381	7.22	* *
Crop.Age	2	34.6596	17.3298	26.4	* * *
Crop.Size	1	0.0961	0.0961	0.15	**NS**
Age.Size	2	22.8319	11.4159	17.39	* * *
Crop.Age.Size	2	0.055	0.0275	0.04	**NS**
Residual	58	38.0724	0.6564		
Total	71	226.4974			
Variate: BFW (stalk)					
Source of variation	d.f.	s.s.	m.s.	v.r.	F pr.
Rep stratum	2	1.29	0.645	0.38	**NS**
Crop	1	134.152	134.152	78.95	* * *
Age	2	24.448	12.224	7.19	* *
Size	1	712.657	712.657	419.41	* * *
Crop.Age	2	111.784	55.892	32.89	* * *
Crop.Size	1	108.241	108.241	63.7	* * *
Age.Size	2	72.331	36.165	21.28	* * *
Crop.Age.Size	2	37.294	18.647	10.97	* * *
Residual	58	98.554	1.699		
Total	71	1300.752			

NS Not significant
* $P < 0.05$
** $P < 0.01$
*** $P < 0.001$

4.7.4. Estimated Brix-free water content of reconstituted cane leaves and cane stalk

Igathinathane *et al.* (2005), after measuring the equilibrium moisture content (EMC) of corn components, namely, stalk skin and stalk pith, proposed that EMC of reconstituted corn stalk could be estimated from the sum of the dry mass fraction and the measured EMC of each of the two components.

Thus:

$M_{st} = D'_{ss}\, M_{ss} + D'_{sp}\, M_{sp}$ where M_{st} is the estimated EMC of the stalk (% db), D'_{ss} and D'_{sp} are the dry matter mass fraction of stalk skin and stalk pith components, M_{ss} and M_{sp} are the observed EMC of stalk skin and stalk pith component (% db) respectively.

Similarly for sugar cane component parts, it would be possible to estimate Brix-free water value of reconstituted dry leaf from the dry mass fraction of dry leaf fibre and dry leaf fines, and their measured constituents Brix-free water values. In so doing, it is assumed that the Brix-free water content is additive, and that the values in the separated components represented the values of these components in the intact state. The same can be done to estimate Brix-free water value of green leaf from the dry mass fraction of green leaf fibre and green leaf fines, and their measured constituents Brix-free water values.

Thus:

$$m_{dl} = D'_{dF}\, m_{dF} + D'_{df}\, m_{df}$$

and $$m_{gl} = D'_{gF}\, m_{gF} + D'_{gf}\, m_{gf}$$

where D'_{dF}, D'_{df}, D'_{gF} and D'_{gf} are the dry mass fractions of dry leaf fibre, dry leaf fines, green leaf fibre and green leaf fines, m_{dl} and m_{gl} are the estimated Brix-free water values of reconstituted dry leaf and reconstituted green leaf, m_{lF}, m_{df}, m_{gF} and m_{gf} are the measured Brix-free water values of dry leaf fibre, dry leaf fines, green leaf fibre and green leaf fines.

In a similar way, it would be possible to reconstitute cane stalk from the dry mass fraction of cane stalk fibre, stalk pith, rind fibre and rind fines, and their observed Brix-free water values.

Thus:

$$m_{st} = D'_{sF}\, m_{sF} + D'_{sp}\, m_{sp} + D'_{rF}\, m_{rF} + D'_{rf}\, m_{rf}$$

where D'_{sF} , D'_{sp} , D'_{rF} and D'_{rf} are the dry mass fractions of stalk fibre, stalk pith, rind fibre and rind fines, m_{st} is the estimated Brix-free water value of the sugar cane stalk, m_{sF} , m_{sp}, m_{rF} and m_{rf} are the measured Brix-free water of stalk fibre, stalk pith, rind fibre and rind fines.

The two mass fractions (fibre and fines) in dry leaf and green leaf, and the four mass fractions in cane stalk, i.e. rind fibre and fines, and stalk fibre and pith, were calculated for the four cane varieties harvested in 2003 and the R 570 harvested in 2001, from Tables 3.8 and 3.11 for cane aged 52 weeks. With the corresponding component Brix-free water values obtained experimentally and tabulated in Tables 4.17, 4.18, 4.20 and 4.21, those of the reconstituted dry leaf, green leaf and cane stalk aged 52 weeks can be predicted (Table 4.26).

Similarly, mass fractions data for samples aged 44 and 36 weeks were calculated using data in Tables 3.9, 3.10 and 3.11, and with the corresponding constituent Brix-free water values obtained experimentally and tabulated in Tables 4.17, 4.18, 4.20 and 4.21 respectively, those of the reconstituted dry cane leaf, green cane leaf and cane stalk aged 44 and 36 weeks can be predicted (Tables 4.27 and 4.28).

In Tables 4.26 – 4.28, all the measured Brix-free water values had 1.1 units added in order to correct for the residual moisture content in the test fibre samples prior to the Brix-free water determination. (Refer to Section 4.4.6 for a discussion of this point.)

Results from Tables 4.26 – 4.28 show that:

- If the reconstituted cane stalk aged 52 weeks is considered, the Brix-free water values of the four cane varieties (Table 4.26) average 16.03% and vary from 15.13 to 16.95%, which is much lower than the traditionally accepted value of 25% for Brix-free water of cane stalk (Section 2.2).
- Brix-free water values for reconstituted dry leaves of four cane varieties investigated aged 52 weeks, i.e. R 579, R 570 harvested in 2003 and in 2001, M 1557/70 and M 1400/86 are 17.10, 16.99, 15.99, 15.08 and 15.61% respectively. From Chapter 2 Section 2.2.2.6, Brix-free water values of dry leaves of the four main cane varieties cultivated in Mauritius, i.e. M 695/69, M 3035/66, R 570 and M 1658/78 were found to be 25.6, 28.5, 28.7 and 27.8% respectively.

The main difference in the two sets of results are that the former were obtained by the devised method of Brix-free water determination with a blank incorporated, while the latter, by Mangion and Player's (1991) method. The other difference being in the state of the dry leaves used. In the former, Brix-free water values were determined on the fibre and fines fractions of dry leaves and values were used to estimate those of the dry leaves reconstituted form these fractions while in the latter, Brix-free water values were determined on the intact whole dry leaves.

- For the reconstituted dry leaf, green leaf and cane stalk, the Brix-free water values (corrected for residual moisture) do not vary much with age nor with variety. At 52, 44 and 36 weeks, the dry leaf of the four cane varieties has an average Brix-free water value of 16.15, 16.57 and 16.41%, green leaf, 15.26, 14.89 and 14.95% and cane stalk 16.03, 15.40 and 15.66%, respectively. If these values are taken together with the corresponding Brix-free water values of 16.06, 16.42 and 15.66% (corrected for residual moisture content) shown in Table 4.19 for intact cane tops aged 52, 44 and 36 weeks respectively, it would mean that the Brix-free water values of these different parts of the sugar cane plant, i.e. dry leaf, green leaf, cane tops and cane stalk, are all about 15-16%. Only those of fibres differ from those of fines or pith as seen previously.

When the moisture content of any of these parts of sugar cane plant is below the Brix-free water value of 15%, the press juice in contact with it will have inflated analyte results, which will affect the cane payment system. However, of these four different parts of sugar cane plant, only dry leaves can have moisture level below 15%.

Re-examination of the results obtained from the survey of moisture in dry trash (see Section 2.2.3), most of the dry trash samples had a low moisture content below the Brix-free water value of 15%. Tables 2.16 – 2.19 show that 51.6, 64.3, 26.7 and 59.1% of the 192, 98, 60 and 22 samples respectively contain less than 15% moisture.

Table 4.26. Predicted Brix-free water of reconstituted dry leaf, green leaf and cane stalk aged 52 weeks.

Sample*		Mass fraction								Experimental Brix-free water/%								Predicted Brix-free water/% of		
		Dry leaf		Green leaf		Rind		Stalk		Dry leaf		Green leaf		Rind		Stalk		reconstituted		
		fibre	fines	fibre	fines	fibre	fines	fibre	pith	fibre	fines	fibre	fines	fibre	fines	fibre	pith	Dry leaf	Green leaf	Cane stalk
R 579	1	0.552	0.448	0.688	0.312	0.377	0.135	0.229	0.259	14.92	16.86	14.00	14.41	11.58	15.76	10.55	23.94	16.88	15.23	16.21
	2	0.578	0.422	0.685	0.315	0.336	0.188	0.176	0.300	15.11	17.16	14.16	12.43	12.10	16.66	10.25	22.74	17.08	14.71	16.92
	3	0.605	0.395	0.649	0.351	0.380	0.195	0.128	0.296	15.77	17.03	13.83	13.55	11.95	14.89	10.88	26.18	17.36	14.83	17.70
	Mean	0.578	0.422	0.674	0.326	0.365	0.172	0.178	0.285	15.26	17.02	14.00	13.46	11.87	15.77	10.56	24.29	**17.10**	**14.92**	**16.95**
R 570	1	0.589	0.411	0.616	0.384	0.363	0.179	0.197	0.262	14.57	17.31	12.53	14.60	10.80	10.00	10.84	26.60	16.79	14.43	15.90
	2	0.622	0.378	0.700	0.300	0.293	0.315	0.164	0.228	15.73	16.55	13.53	13.24	10.82	10.84	11.09	23.13	17.13	14.54	14.78
	3	0.528	0.472	0.697	0.303	0.385	0.227	0.146	0.242	14.79	17.36	11.96	13.23	11.15	10.27	11.89	22.13	17.10	13.45	14.81
	Mean	0.579	0.421	0.671	0.329	0.347	0.240	0.169	0.244	15.03	17.07	12.67	13.69	10.92	10.37	11.27	23.95	**16.99**	**14.11**	**15.13**
M 1557/70	1	0.550	0.450	0.710	0.290	0.364	0.245	0.138	0.253	15.52	13.94	14.85	16.10	11.09	12.48	14.19	18.59	15.91	16.31	14.85
	2	0.564	0.436	0.700	0.300	0.395	0.180	0.183	0.241	15.50	14.64	15.28	17.87	15.37	16.42	13.38	23.33	16.22	17.15	18.21
	3	0.583	0.417	0.701	0.299	0.385	0.188	0.164	0.264	14.43	15.07	15.03	18.09	12.38	13.92	11.66	20.46	15.79	17.04	15.78
	Mean	0.566	0.434	0.704	0.296	0.381	0.204	0.162	0.253	15.15	14.55	15.05	17.35	12.94	14.27	13.08	20.79	**15.99**	**16.83**	**16.32**
M 1400/86	1	0.586	0.414	0.754	0.246	0.294	0.223	0.171	0.312	14.39	12.85	14.48	14.90	13.31	16.35	14.24	23.30	14.85	15.68	18.36
	2	0.577	0.423	0.689	0.311	0.369	0.222	0.144	0.265	13.43	13.28	14.79	14.94	12.01	13.28	12.73	20.42	14.46	15.94	15.73
	3	0.555	0.445	0.727	0.273	0.429	0.219	0.126	0.226	13.65	16.45	14.01	14.75	12.41	13.89	10.55	21.90	15.99	15.31	15.74
	Mean	0.572	0.428	0.723	0.277	0.364	0.221	0.147	0.268	13.82	14.19	14.43	14.86	12.57	14.50	12.51	21.87	**15.08**	**15.64**	**16.58**
R 570 (2001)	1	0.433	0.567	0.691	0.309	0.360	0.251	0.129	0.260	14.28	15.13	14.16	13.74	12.58	13.49	10.79	17.86	15.86	15.13	15.05
	2	0.395	0.605	0.665	0.335	0.363	0.222	0.134	0.281	14.55	14.58	14.48	12.19	13.38	13.30	10.82	16.22	15.67	14.81	14.92
	3	0.345	0.655	0.701	0.299	0.363	0.185	0.133	0.319	14.42	14.00	14.06	11.82	14.56	12.32	12.27	16.49	15.24	14.49	15.56
	Mean	0.391	0.609	0.685	0.315	0.362	0.220	0.132	0.287	14.41	14.57	14.23	12.58	13.51	13.03	11.29	16.86	**15.61**	**14.81**	**15.17**
																		16.15	15.26	16.03

* Samples were harvested in 2003 except where indicated.

Table 4.27. Predicted Brix-free water of reconstituted dry leaf, green leaf and cane stalk aged 44 weeks.

Sample*		Mass fraction								Experimental Brix-free water/%								Predicted Brix-free water/% of reconstituted		
		Dry leaf		Green leaf		Rind		Stalk		Dry leaf		Green leaf		Rind		Stalk				
		fibre	fines	fibre	fines	fibre	fines	fibre	pith	fibre	fines	fibre	fines	fibre	fines	fibre	pith	Dry leaf	Green leaf	Cane stalk
R 579	1	0.508	0.492	0.686	0.314	0.337	0.162	0.203	0.298	15.81	15.63	12.95	12.07	11.87	13.02	10.67	18.09	16.82	13.77	14.76
	2	0.539	0.461	0.718	0.282	0.389	0.224	0.151	0.236	15.50	15.43	14.60	13.97	13.24	11.54	11.29	16.98	16.57	15.52	14.54
	3	0.541	0.459	0.726	0.274	0.317	0.164	0.188	0.331	14.94	17.79	12.70	14.02	12.04	15.47	10.84	19.43	17.35	14.16	15.92
	Mean	0.529	0.471	0.710	0.290	0.348	0.183	0.181	0.288	15.42	16.28	13.42	13.35	12.38	13.34	10.93	18.16	**16.92**	**14.50**	**15.06**
R 570	1	0.556	0.444	0.727	0.273	0.424	0.192	0.149	0.235	14.52	16.27	14.39	15.00	11.66	11.01	12.81	18.73	16.39	15.65	14.47
	2	0.530	0.470	0.718	0.282	0.438	0.187	0.151	0.224	17.10	14.42	12.12	11.11	11.14	11.18	13.72	18.01	16.94	12.93	14.17
	3	0.520	0.480	0.670	0.330	0.376	0.239	0.154	0.231	15.28	17.40	12.35	11.07	10.92	11.37	12.26	18.36	17.40	13.02	14.05
	Mean	0.535	0.465	0.705	0.295	0.413	0.206	0.151	0.230	15.63	16.03	12.95	12.39	11.24	11.19	12.93	18.37	**16.92**	**13.88**	**14.22**
M 1557/70	1	0.502	0.498	0.696	0.304	0.386	0.262	0.123	0.229	15.41	14.26	13.43	15.55	14.55	13.61	11.52	17.07	15.94	15.17	15.60
	2	0.436	0.564	0.705	0.295	0.379	0.204	0.148	0.269	14.28	13.45	12.34	17.64	15.12	15.43	13.12	19.69	14.91	15.00	17.22
	3	0.468	0.532	0.689	0.311	0.403	0.214	0.137	0.246	14.05	17.02	13.64	17.90	13.79	13.80	12.79	16.81	16.73	16.06	15.49
	Mean	0.469	0.531	0.697	0.303	0.389	0.227	0.136	0.248	14.58	14.91	13.13	17.03	14.48	14.28	12.47	17.85	**15.85**	**15.42**	**16.10**
M 1400/86	1	0.480	0.520	0.697	0.303	0.347	0.240	0.140	0.273	14.51	15.08	13.18	15.42	11.13	13.17	14.31	19.62	15.90	14.96	15.48
	2	0.551	0.449	0.691	0.309	0.333	0.246	0.146	0.275	15.17	14.07	12.64	14.70	10.85	10.90	13.25	18.97	15.78	14.37	14.55
	3	0.496	0.504	0.705	0.295	0.323	0.233	0.149	0.295	13.97	16.49	13.91	15.15	10.60	11.50	12.59	16.96	16.34	15.37	14.08
	Mean	0.509	0.491	0.697	0.303	0.334	0.240	0.145	0.281	14.55	15.21	13.24	15.09	10.86	11.86	13.38	18.51	**15.97**	**14.90**	**14.72**
R 570 (2001)	1	0.677	0.323	0.614	0.386	0.422	0.113	0.180	0.285	16.46	17.66	14.18	15.03	15.05	16.68	13.01	17.82	17.94	15.60	16.75
	2	0.710	0.290	0.666	0.334	0.396	0.147	0.171	0.286	16.93	14.76	13.95	14.60	15.07	12.93	14.07	18.22	17.40	15.27	16.58
	3	0.553	0.447	0.676	0.324	0.389	0.171	0.146	0.294	15.95	13.67	15.08	15.92	14.47	15.14	14.90	19.74	16.03	16.45	17.30
	Mean	0.647	0.353	0.652	0.348	0.402	0.144	0.166	0.288	16.45	15.36	14.40	15.18	14.86	14.92	13.99	18.59	**17.16**	**15.77**	**16.90**
																		16.57	14.89	15.40

Samples were harvested in 2003 except where indicated.

Table 4.28. Predicted Brix-free water of reconstituted dry leaf, green leaf and cane stalk aged 36 weeks.

Sample*		Mass fraction								Experimental Brix-free water/%								Predicted Brix-free water/% of reconstituted		
		Dry leaf		Green leaf		Rind		Stalk		Dry leaf		Green leaf		Rind		Stalk				
		fibre	fines	fibre	fines	fibre	fines	fibre	pith	fibre	fines	fibre	fines	fibre	fines	fibre	pith	Dry leaf	Green leaf	Cane stalk
R 579	1	0.601	0.399	0.745	0.255	0.386	0.158	0.172	0.285	15.57	16.37	13.77	13.17	12.24	15.53	14.42	20.41	16.99	14.72	16.56
	2	0.592	0.408	0.685	0.315	0.376	0.158	0.159	0.306	14.77	15.45	14.18	16.04	12.08	17.05	14.36	21.65	16.14	15.86	17.26
	3	0.565	0.435	0.699	0.301	0.419	0.224	0.122	0.235	14.36	15.94	13.39	17.44	11.29	19.08	14.05	18.80	16.14	15.70	16.24
	Mean	0.586	0.414	0.709	0.291	0.393	0.180	0.151	0.276	14.90	15.92	13.78	15.55	11.87	17.22	14.28	20.29	**16.42**	**15.39**	**16.61**
R 570	1	0.651	0.349	0.719	0.281	0.383	0.224	0.150	0.243	14.44	17.78	12.44	14.88	12.32	16.16	13.50	22.74	16.70	14.23	16.99
	2	0.605	0.395	0.673	0.327	0.414	0.266	0.114	0.206	14.85	16.75	12.29	14.55	12.63	13.66	13.23	20.82	16.70	14.13	15.76
	3	0.635	0.365	0.711	0.289	0.400	0.304	0.096	0.200	14.51	15.11	12.85	15.87	12.71	13.62	13.07	20.60	15.83	14.82	15.69
	Mean	0.631	0.369	0.701	0.299	0.399	0.265	0.120	0.216	14.60	16.54	12.53	15.10	12.55	14.48	13.27	21.38	**16.42**	**14.39**	**16.16**
M 1557/70	1	0.700	0.300	0.742	0.258	0.417	0.241	0.110	0.232	15.30	19.76	13.45	13.41	12.25	16.90	16.36	17.49	17.73	14.54	16.13
	2	0.699	0.301	0.689	0.311	0.431	0.261	0.093	0.215	15.27	16.84	13.55	14.67	12.03	13.93	15.90	18.25	16.84	14.99	15.32
	3	0.638	0.362	0.674	0.326	0.427	0.247	0.100	0.226	14.69	15.40	13.02	15.68	13.61	13.62	13.04	16.45	16.05	14.98	15.29
	Mean	0.679	0.321	0.702	0.298	0.425	0.250	0.101	0.224	15.09	17.33	13.34	14.59	12.63	14.81	15.10	17.40	**16.91**	**14.81**	**15.59**
M 1400/86	1	0.700	0.300	0.691	0.309	0.391	0.236	0.112	0.260	16.07	17.58	13.45	12.86	12.15	14.81	14.53	20.26	17.62	14.37	16.25
	2	0.632	0.368	0.719	0.281	0.382	0.262	0.100	0.256	14.87	15.00	12.35	13.13	12.24	13.92	14.99	19.13	16.01	13.67	15.82
	3	0.629	0.371	0.717	0.283	0.345	0.214	0.150	0.291	17.06	14.45	12.59	13.94	12.98	15.00	14.02	16.72	17.19	14.07	15.75
	Mean	0.654	0.346	0.709	0.291	0.373	0.238	0.121	0.269	16.00	15.67	12.80	13.31	12.45	14.58	14.51	18.70	**16.99**	**14.04**	**15.99**
R 570 (2001)	1	0.582	0.418	0.645	0.355	0.371	0.185	0.179	0.265	13.64	14.79	14.97	14.35	11.77	15.84	9.37	14.09	15.22	15.85	13.80
	2	0.548	0.452	0.646	0.354	0.356	0.169	0.201	0.274	15.12	12.80	15.16	15.15	13.68	16.04	14.30	11.76	15.17	16.25	14.77
	3	0.496	0.504	0.629	0.371	0.382	0.162	0.207	0.249	14.75	14.07	14.89	15.75	12.75	13.01	10.38	12.27	15.50	16.30	13.28
	Mean	0.542	0.458	0.640	0.360	0.370	0.172	0.196	0.263	14.50	13.88	15.01	15.08	12.73	14.96	11.35	12.70	**15.32**	**16.13**	**13.94**
																		16.41	14.95	15.66

* Samples were harvested in 2003 except where indicated.

4.8 SUMMARY AND CONCLUSIONS

An analytical method has been developed to determine the Brix-free water in fibres obtained from various component parts of sugar cane plant. Prior to analysis, the fibre sample is dried in an air oven at 65 °C for one hour with occasional stirring, followed by vacuum oven drying at 65 °C under 875 mbar vacuum for 16 hours. The method involves contacting the sample with a 10° Brix sucrose solution for one and a half hours, during which time the sample is shaken every 10 minutes. The Brix-change in the contact solution gives a measure of the Brix-free water capacity of the sample. The ratio of the contact solution to sample size is kept at 20. Good separation of fibre and fines in the sample is essential to obtain reproducible results. The method makes use of a distilled water blank to compensate for any residual sucrose in the sample, since residual sucrose in the fibre sample was found to inflate the results. This method showed that:

- An increased concentration of the sucrose contacting solution increased the Brix-free water values of the fibre samples, contrary to the findings of previous workers, who found a decrease in the Brix-free water value.
- A ratio (14-26) of the volume of the contact solution to the mass of sample used did not affect the Brix-free water value of a stalk fibre sample. However, if the ratio became much higher, the analytical errors became large as the Brix-change after equilibrium would be less than 0.1 unit, and at low ratio, e.g. 5, pith samples would not be completely wetted.
- Sample fineness did not affect Brix-free water value of sample analysed. When some fibre samples were reduced to a finer state (about 3 mm long) by cutting with a pair of scissors, there was no change in the Brix-free water value obtained, contrary to the findings of Mangion and Player (1991).
- For 21 Brix-free water values of various cane component parts (Table 4.16), a standard error of the mean of 0.547 was obtained for a mean of 12.82 on the original samples, and 0.500 for a mean of 12.72 on finely cut samples.
- A halogen thermogravimetric method was developed to determine residual moisture in dried fibre samples. The method requires about 0.6 g of sample and takes a few minutes to execute instead of the three hours required by the standard drying method. Statistical analyses showed that the halogen method gave results comparable to that

obtained by the standard drying method; for 45 data, the standard error of mean was 0.104% for a mean of 2.42% moisture.

- The drying method described at the beginning of this Section 4.8 prior to the devised method of Brix-free water determination left residual moisture in the cane component parts. It varied from 0.68% in cane top fibre to 1.42% for dry leaf fines, and averaged 1.12% for all cane component parts. Brix-free water results obtained by using this drying method are therefore under-estimated. A correction for the residual moisture must therefore be made to the Brix-free water results obtained. To simplify matters, all Brix-free water values obtained in this work should have 1.1 units added to correct for this but that in the tables of data this correction has not been implemented.

- The Brix-free water values (corrected for residual moisture) of the reconstituted cane stalk of the four cane varieties aged 52 weeks were calculated. The average value is 16.03%, which is much lower than the traditionally accepted value of 25% for Brix-free water of cane stalk. However, these two quantities are not comparable. The reconstituted cane does not include nodes, and strictly speaking, if Brix-free water value of cane is required, fibres should be obtained from cane stalk including rind, stalk and nodes, and Brix-free water determined on the fibres under specified conditions.

- For reconstituted dry leaf, green leaf and cane stalk, the Brix-free water values do not vary much with age nor with variety. The reconstituted dry leaf, green leaf and cane stalk of the four cane varieties each has an average of Brix-free water value of 15-16%. If these values are taken together with the corresponding 15-16% Brix-free water value of intact cane tops, it would appear that the Brix-free water values of the different parts of the sugar cane plant, i.e. dry leaf, green leaf, cane tops and cane stalk, are all about 15-16%. Only those of fibres differ from those of fines or pith.

Since the Brix-free water of cane fibre is characterised as the water strongly bound to the fibre and unavailable for the solution of the soluble components present in sugar cane, and various types of bound water can be determined from a study of the adsorption properties of a material, it would be of interest to study the adsorption behaviour of the sugar cane

component parts, and verify the Brix-free water value of 15-16% obtained for the different cane components.

CHAPTER 5. ADSORPTION ISOTHERMS OF SUGAR CANE FIBRES

In the previous chapter the Brix-free water content of the cane components are determined by means of a contact method. This chapter describes the experiments performed to determine the equilibrium moisture content, and hence adsorption properties, by means of a vapour sorption method of these cane component parts which enabled the determination of a number of thermodynamic parameters that provide insight into the microstructure of the fibre-water interface.

5.1 THE CONCEPT OF BOUND WATER IN FIBRE

All biological systems have the ability to retain molecular hydration as a fundamental defensive mechanism against dehydration (Quioco *et al.*, 1989). The structure, mobility and function of biological molecules are affected by the water molecules bound to the ionic, polar and hydrophilic sites of macromolecules (Vertucci and Leopold, 1987). Rascio *et al.* (1992) demonstrated the important role played by bound water in the plant's adaptation to the moderate stress of dehydration and related its tolerance towards dehydration to the quantity of bound water, the strength of binding and its ability to tolerate the removal of bound water without damage. However, the relationship between the quantity of bound water and water binding strength of plant tissues was not elucidated.

In their review of moisture sorption isotherm characteristics of food products, Al-Muhtaseb *et al.* (2002) quoted that food preservation consisted of controlling the moisture content during the processing of foods, achieved either by removing it or binding it such that the food becomes stable to both microbial and chemical deterioration (Labuza, 1980). Later, the concept of water activity was introduced to indicate the ‘quality’ of the water content of food. It describes the degree of ‘boundness’ of water and hence, its availability to participate in physical, chemical and microbiological reactions. In a biological system, three aspects of water can be distinguished (Rizvi and Benato, 1984):

1) Structural, the position and orientation of water molecules in relation to each other and to macromolecules.
2) Dynamic, molecular motions of water molecules and their contribution to the hydrodynamic properties of the system.

3) Thermodynamic, water in equilibrium with its surroundings, at a certain relative humidity and temperature.

Moreover, in a biological system, water is believed to exist with either unhindered or hindered mobility, referred to as free or bound water respectively. 'Bound water' is considered as that portion of water held in the material which exhibits physical properties significantly different from those of free water or bulk water (Berlin, 1981), through stronger hydrogen bonding than liquid water. Some of the characteristics of bound water are lower vapour pressure, high binding energy as measured during dehydration, reduced mobility, unfreezability at low temperature and unavailability as a solvent such as in the definition of Brix-free water (Labuza and Busk, 1979). Although each of these characteristics has been used to define bound water, each gives a different value for the amount of water which is bound. As a result of this, as well as the complexities and interactions of the binding forces involved, no universal definition of bound water has been adopted.

5.2 TYPES OF ADSORPTION AND ADSORPTION ISOTHERMS

In this study the interaction of sugar cane fibres with water was studied. When a solid surface (in this case the sugar cane fibre) is exposed to a fluid (i.e. gas or liquid, and in this case water) adsorption occurs. It is understood to mean the increase in the density of the fluid in the vicinity of an interface. With certain systems, e.g. some metals exposed to hydrogen, oxygen or water, the adsorption process is accompanied by absorption, i.e. the penetration of the fluid into the solid phase. In such a case the term sorption is used and, in particular, when the adsorption and absorption processes cannot be distinguished experimentally.

Distinction was made in the early 1930s between physical adsorption (physisorption) in which weak Van der Waals interactions are involved and chemical adsorption (chemisorption) in which the adsorbed molecules are attached by strong chemical bonding.

The characteristic features distinguishing between the two types of adsorption may be summarised as follows:

(a) Physisorption is a general phenomenon with a relatively low degree of specificity, whereas chemisorption is dependent on the reactivity of the adsorbent (solid

material on which adsorption occurs) and the adsorbate (adsorbable substance in the fluid phase).

(b) Physisorption generally occurs as a multilayer at high relative pressures whereas chemisorbed molecules are linked to reactive parts of the surface and the adsorption is confined to a monolayer.

(c) A physisorbed molecule keeps its identity and on desorption returns to the fluid phase in its original form, whereas a chemisorbed molecule undergoes reaction, it loses its identity and cannot be recovered by desorption.

(d) Physisorption is always exothermic, and the energy involved is not much larger than the energy of condensation of the adsorbate, whereas the energy of chemisorption is the same order of magnitude as the energy change in a comparable chemical reaction.

(e) Physisorption generally attains equilibrium fairly rapidly, in chemisorption, an activation energy is often involved and at low temperature, the system may not have sufficient energy to reach thermodynamic equilibrium.

The terms adsorption and desorption are used to indicate the direction from which the equilibrium states have been approached. The former occurs when the material reaches equilibrium by wetting and the latter by drying. Adsorption hysteresis arises when the amount adsorbed is not brought to the same level by the adsorption and desorption approach to a given 'equilibrium' pressure or bulk concentration.

The term ‘adsorption isotherm’ was first introduced by Freundlich (1907) to describe the relationship, at constant temperature, between the amount adsorbed by a substrate (adsorbent) and the equilibrium pressure, or concentrations of a fluid (adsorbate).

He proposed a general mathematical relationship for the isotherm, which is now universally referred to as the Freundlich adsorption equation (1926).

$$a = kp^s$$

where a is the fraction of the total surface area covered by an adsorbate. p is the pressure, and k and s are constants. A plot of $ln\ (a)$ against $ln\ (p)$ is linear.

The above Freundlich equation only fits adsorption data taken over a small pressure range, and breaks down at high pressures and low temperatures (Brunauer, 1945).

Langmuir (1916, 1917, 1918) proposed that adsorption on both liquid and solid surfaces normally involves the formation of a monomolecular layer (the monolayer). To support this concept, he collected evidence of isotherms in which the amount of gas adsorbed increased with increasing pressure and then saturated to a plateau, i.e. a monolayer coverage. This type of isotherm was later referred to as a type I isotherm (see Fig 5.1). His work paved the way to subsequent progress in the interpretation of adsorption data.

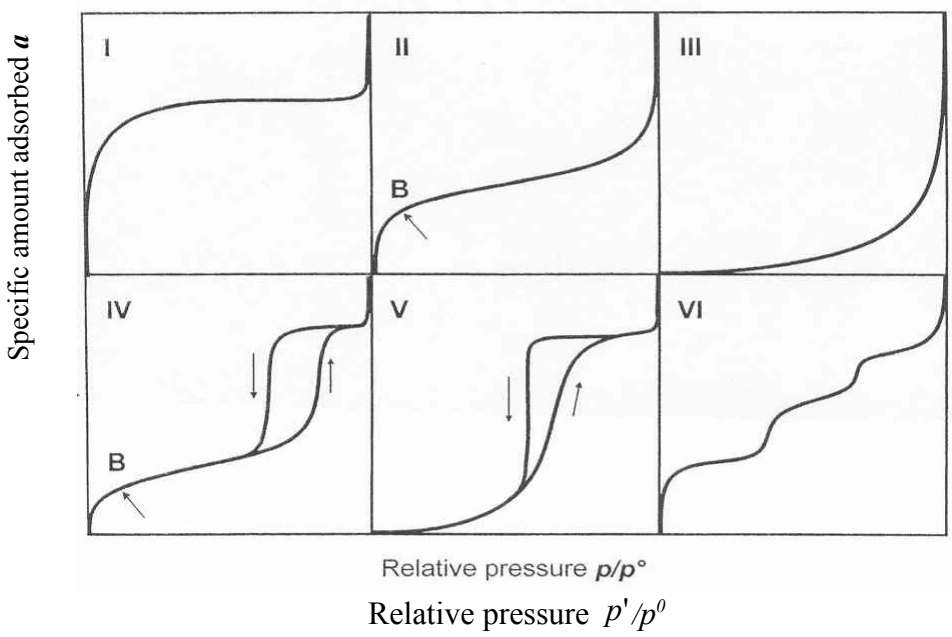


Figure 5.1. The six main types of gas physisorption isotherms, as per the IUPAC classification (Sing *et al.*, 1985).

In his 1916 paper, Langmuir stated that with highly porous adsorbents such as charcoal, "it is impossible to know definitely the area on which the adsorption takes place" and that "there are some spaces in which a molecule would be closely surrounded by carbon atoms on nearly all sides". He concluded that equations derived for plane surfaces were not applicable to adsorption by charcoal. Dubinin (1960) later showed that the mechanism of physisorption in very narrow pores is not the same as that in wider pores, or on open surfaces. He identified three groups of pores of different width: micropores, transitional pores (now termed mesopores), and macropores, of internal width less than 2 nm, between 2 and 50 nm, and greater than 50 nm respectively.

Emmett and Brunauer (1934) used low temperature adsorption of nitrogen to determine the surface area of a synthetic iron catalyst for the production of ammonia. They noted that

the adsorption isotherms of a number of gases, measured at temperatures at, or near, their respective boiling points were all S-shaped with certain distinctive features. This prompted the recognition that adsorption was not always restricted to monolayer coverage and to the emergence of the Brunauer-Emmett-Teller (BET) theory (1938) of multilayer adsorption. This type of isotherm shows the amount adsorbed increases with increasing pressure up to a point B, then levels off and starts to increase again at higher pressures; it was later referred to as a type II isotherm. Point B is usually considered to represent the completion of the monolayer and the beginning of the formation of the multilayer.

In addition to the type I and II isotherms described above, there are type III isotherms, where initially there is very little adsorption, then once a small droplet of adsorbate nucleates on the surface, additional adsorption occurs more easily because of strong adsorbate-adsorbate interactions. Type IV and type V isotherms occur when multilayers of gas adsorb onto the surface of the pores in a porous solid. Initially, the adsorption resembles that of type II or type III adsorption, then the adsorbed layer gets so thick that it fills up the pores, no more gas can adsorb, and the isotherm saturates. Both type IV and V isotherms exhibit a hyteresis loop, the lower curve of which represents measurements obtained by progressive addition of gas to the adsorbent, and the upper curve by progressive removal.

These five classifications of isotherms were proposed by S. Brunauer, L.S. Deming, W.S. Deming and E. Teller and are termed the BDDT classification (1940), and are sometimes also referred to as the Brunauer classification (1945).

The classification proposed by the International Union of Pure and Applied Chemistry (IUPAC) in 1985 (Sing *et al.*, 1985), shown in Fig 5.1, includes a type VI isotherm which has been observed more recently. It appears as a stepped isotherm, associated with layer-by-layer adsorption on a highly uniform surface. This type of isotherm is relatively rare (Rouquerol *et al.*, 1999).

5.3 ADSORPTION OF MOISTURE

When a hygroscopic material is maintained in contact with air at constant temperature and humidity until equilibrium is reached, the material will attain a definite moisture content. This moisture is termed the equilibrium moisture content (EMC) under the specified conditions. In order to characterise this water sorption mechanism, it is common practice

to determine the moisture sorption isotherm, which describes the EMC of the material and the water activity or relative humidity at a certain temperature.

The water in a material is generally measured in terms of water activity, a_w, which by definition is given by :

$$\frac{\text{water vapour pressure at the solid-gas interface}}{\text{vapour pressure of liquid at the same temperature}}$$

Thus, when a moist material is in equilibrium with its surroundings, the water vapour pressure of the material is equal to the partial pressure of water vapour in the atmosphere, and so, the water activity in the material is equal to the equilibrium relative humidity of the air. Knowledge of the water adsorption characteristics is needed for shelf life predictions of products that deteriorate mainly by moisture gain and is important in drying, packaging and storage.

Thermodynamic properties of the material relate the concentration of water in the material to its partial pressure, which is crucial in the analyses of heat and mass transport phenomena during drying. The EMC determines the end-point to which the material must be dehydrated in order to achieve a stable product with optimal moisture content, and yield a theoretical minimum amount of energy required to remove a given amount of water from the material. The properties also provide an insight into the microstructure associated with the material as well as the theoretical interpretation of physical phenomena occurring at the material-water interface.

The control of moisture content is particularly important in foods as well as in materials, such as woody straw fibres, during processing and storage because water has many roles in substrate reactions and keeping quality. In this respect the moisture sorption isotherm is an extremely important tool as it can be used to predict changes in substrate stability and to select appropriate packaging material and ingredients. There are many works on moisture sorption isotherms of substrates over the last two decades; some deal with the determination of moisture sorption isotherms, others, with the development of mathematical models to represent the moisture sorption isotherms.

5.3.1 Moisture sorption isotherm models

The models available in the literature to describe water sorption isotherms can be divided into several categories: kinetic models based on a multilayer adsorption mechanism (BET model), kinetic models based on a multilayer and condensed film mechanism

(Guggenheim-Anderson-de Boer GAB model), semi-empirical (Henderson and Halsey models) and empirical models (Smith and Oswin models).

Most of these equations predict EMC values from known water activity data, others from relative humidity values. If the latter are expressed as a fraction instead of a percentage value, they are equivalent to water activity as shown below.

The water activity a_w of a substrate is defined as the ratio of the equilibrium vapour pressure of water (p') in the substrate to the vapour pressure of pure water (p^0) at the same temperature, i.e. $a_w = \frac{p'}{p^0} = \frac{R_H}{100} = H_R$, where R_H and H_R are the equilibrium relative humidity expressed respectively as a percentage and fraction.

Some commonly used moisture adsorption isotherm models are compiled in Table 5.1. All are two-parameter equations, except for the GAB and Hailwood-Horrobin equations which are three-parameter and Peleg which is a four-parameter equation.

Some isotherm models had been modified to take into account the effect of temperature as shown in Table 5.2 (e.g. the modified Chung-Pfost, the modified GAB, the modified Halsey, the modified Henderson and the modified Oswin models). All of them are four-parameter equations including a temperature term.

Table 5.1. Some commonly used isotherm models to predict equilibrium moisture content (m) from the known water activity (a_w) of agricultural products.

Model	Equation*
Bradley (Bradley, 1936)	$\ell n\left(\frac{1}{a_w}\right) = bc^m$
Caurie I (Caurie, 1981)	$\ell n\frac{1}{m} = -\ell n\frac{1}{bm_o} + \frac{2b}{m_o}\ell n\left(\frac{1-a_w}{a_w}\right)$
Caurie II (Caurie, 1970)	$m = e^{(b+ca_w)}$
Halsey (Halsey, 1948)	$m = \left(\frac{c}{\ell n\, a_w}\right)^{\frac{1}{b}}$
Henderson (Henderson, 1952)	$\ell n\left[\ell n\frac{1}{1-a_w}\right] = \ell n\, b + c\,\ell n\, m$
Kuhn (Kuhn, 1964)	$m = \left(\frac{b}{\ell n\, a_w}\right) + c$
Smith (Smith, 1947)	$m = b\left[\ell n(1-a_w)\right] + c$
GAB (Guggenheim, 1966; Anderson, 1946 and de Boer, 1953)	$m = \frac{m_o\, b\, c\, a_w}{(1-c\,a_w)(1-c\,a_w+b\,c\,a_w)}$
BET (Brunauer *et al*, 1938)	$m = \frac{m_o\, b\, a_w}{(1-a_w)(1-a_w+ba_w)}$
Modified BET (Aguerre *et al.*, 1938)	$m = \frac{m_o\, b\, a_w}{(1-a_w)\left[1-b\,\ell n(1-a_w)\right]}$
Day-Nelson (Day and Nelson, 1965)	$1-a_w = e^{\left(-b/m^c\right)}$
Hailwood-Horrobin (Hailwood and Horrobin, 1946)	$\frac{a_w}{m} = b + c\,a_w + d\,(a_w)^2$
Iglesias-Chirife (Iglesias and Chirife, 1981)	$m = b\left(\frac{a_w}{1-a_w}\right) + c$
Mizrahi (Mizrahi *et al.*, 1970; Ayranci and Duman, 2005)	$a_w = \frac{b+m}{c+m}$
Oswin (Oswin, 1946)	$m = b\left[\frac{a_w}{1-a_w}\right]^c$
Harkins-Jura (Erbas *et al.*, 2005)	$\frac{1}{m^2} = \frac{c}{b} - \frac{1}{b}\ell n\, a_w$
Peleg (Peleg, 1993)	$m = b\,(a_w)^d + c\,(a_w)^f$

* where m_o is the moisture content of the sample on a dry basis when each sorption site contains one water molecule (monolayer), a_w is the water activity, and *b, c, d, f* are constants in the sorption models.

Table 5.2. Some commonly used three-parameter isotherm equations with a temperature term for the calculation of the equilibrium moisture content (m) of agricultural products (Jayas and Mazza, 1993).

Model	Equation*
Modified Chung-Pfost (Jayas & Mazza, 1993; Nilsson *et al.*, 2005)	$m = -\frac{1}{d}\ell n\left[\frac{(T+c)\,\ell n(a_w)}{-b}\right]$
Modified GAB (Weisser, 1986; Nilsson *et al.*, 2005)	$m = \frac{b\,c\,a_w\,(d/T)}{(1-c\,a_w)[1-c\,a_w+c\,a_w(d/T)]}$
Modified Halsey (Nilsson *et al.*, 2005)	$m = \left[\frac{-e^{(b+cT)}}{\ell n\,H_R}\right]^{\frac{1}{d}}$
Modified Henderson (Nilsson *et al.*, 2005)	$m = \left[\frac{\ell n(1-H_R)}{-b(T+d)}\right]^{\frac{1}{c}}$
Modified Oswin (Nilsson *et al.*, 2005)	$m = \frac{b+cT}{\left(\frac{1}{H_R}-1\right)^{\frac{1}{d}}}$

* where a_w is the water activity, H_R is the relative humidity expressed as a fraction, b, c and d are constants in the sorption models and T is the temperature in °C.

5.3.2 Applicability of different adsorption isotherm models

Many researchers have developed equations and models to describe experimental EMC results obtained from agricultural products. Chirife and Iglesias (1978) have compiled 23 such mathematical equations, and their use for fitting sorption isotherms of foodstuffs. They pointed out that no one equation, however, was found to give accurate results universally for all types of food materials and throughout the whole range of relative humidities. Labuza (1975) attributed this to the fact that water is associated with the food matrix by different mechanisms in different activity regions.

In 1981, Van den Berg and Bruin identified 77 isotherm models to predict the EMC of substrates. Of these, the most widely utilised and versatile model is recognised to be the GAB equation, particularly in the case of food and food products.

Lomauro *et al.* (1985) found that with 163 food materials including fruits, vegetables, spices and starchy foods, the three-parameter GAB equation fits better for most of these food materials than some two-parameter equations.

The BET model (Brunauer *et al.*, 1938) represents a fundamental milestone in the interpretation of multilayer sorption isotherms, particularly for type II and III adsorption. It is the most popular isotherm but it is valid only from 10 – 50% relative humidity (Labuza, 1968). Many research workers modified the BET equation to give a good fit up to 90% relative humidity (Dincer & Esin, 1996).

The semi-empirical Halsey equation (Halsey, 1948) has also demonstrated its suitability to describe the experimental isotherms of foods, and is representative of 69 different products (Bosquet *et al.*, 1978).

The Smith model (Smith, 1947) was found good for describing the sorption isotherm of biological materials such as starch and cellulose; and the Henderson model (Henderson, 1952) for cereal grain. Day and Nelson (1965) modified the Henderson equation to describe the sorption behaviour of wheat up to 70% relative humidity. The Chung-Pfost model was good for grain over the 20 – 90% relative humidity range.

In general, the GAB, BET, Halsey, Henderson and Oswin models had been applied with success to high sugar-containing foods (Tsami *et al.*, 1990, Maskan and Göğüs, 1997, and Kaymak-Ertekin and Gedik, 2004).

In this study, 14 of the isotherm models listed in Table 5.1 (all except the last two) and the modified Chung-Pfost and modified GAB models with a temperature term incorporated in their respective original model (Table 5.2) have been applied to predict the EMC of the fibre components derived from the sugar cane plant.

5.3.3 Choice of isotherm models

The usefulness of a sorption model depends on the predetermined objectives set by the user. For instance, if the user is interested in predicting shelf life, a model with good agreement to the experimental data as opposed to one which fits well with theoretical considerations will be required. The simplicity of a model can also influence the choice, since a model with a lower number of parameters will have an improved usability in that computation times will be lower (Bosquet *et al.*, 1978).

In this work the model which best described the experimental data was determined in order to extract thermodynamic parameters that provide information on the bound water content.

5.3.4 Fitting of sorption data to adsorption isotherm models

The criteria adopted to evaluate whether the isotherm model used was a good fit of the experimental data were: 1) the coefficient of determination, R^2, 2) the mean relative deviation modulus P, and 3) the standard error of the estimate, E_s.

The mean relative deviation modulus P is defined as: $$P = \frac{100}{N}\sum_{n=1}^{n}\left|\frac{m - \hat{m}}{m}\right|$$

and E_s is given by: $$E_s = \sqrt{\frac{\sum (m - \hat{m})^2}{df}}$$

where m and $\hat{m}$ are the measured and predicted EMC (on a dry basis), N is the number of data points and df is the number of degrees of freedom which equals N minus the number of parameters.

The criteria for accepting the fit of a model to the experimental data are that R^2 should approach 1 and that the value of E_s should be as small as possible. In addition if P is ≤ 5, the fit is considered to be excellent. If $5 \leq P \leq 10$, the fit is considered reasonably good, but if $P > 10$, the fit is deemed poor (Lomauro *et al.*, 1985).

In addition, Chen and Morey (1989) showed that the residuals (i.e. measured EMC – predicted EMC) can be plotted against the predicted moisture content, and the plots evaluated visually for randomness or pattern. If the plots have a clear pattern, the model is not accepted.

In the present study, the above criteria were adopted to decide whether a model is a good fit of the experimental data.

5.4 PREVIOUS RESEARCH PERFORMED TO MEASURE MOISTURE SORPTION ISOTHERMS ON SUGAR CANE FIBRE AND SOME WOODY FIBRES

While work on the determination of adsorption isotherms of cane fibre is relatively rare, the literature abounds with adsorption studies on fruits such as apple (Prothon and Ahrné, 2004) and pineapple (Hossain *et al.*, 2001); on foodstuff, e.g. macaroni (Arslan and Toğrul, 2005), semolina (Erbas *et al.*, 2005) and alligator meat (Lopes Filho *et al.*, 2002);

and on agricultural products such as potatoes (McMinn and Magee, 2003; McLaughlin and Magee, 1998), starch (Al-Muhtaseb *et al.*, 2004a) and cowpea (Ayranci and Duman, 2005). Fibres from eucalyptus (Moreira *et al.*, 2001) and fibres from flax, hemp and reed canary grass (Nilsson *et al.*, 2005) have also been the subjects of study. The above list is by no means exhaustive; many more publications exist on other materials.

5.4.1 Sugar cane fibre

One of the few works on the determination of adsorption isotherms involving cane fibre dates back to the late 1950s, when Kelly (1957) studied the water adsorption of sugar cane fibre in an atmosphere of constant water vapour pressure provided by sulfuric acid solutions of appropriate concentrations at two temperatures. Sugar cane fibre samples after fibration were washed free of sucrose and other water-soluble materials. The dry samples were then exposed to an atmosphere of constant water vapour pressure in a desiccator placed in a thermostatically-controlled oven for at least 40 hours. After which period, it was assumed that the condition of equilibrium had been reached. The tests were carried out at 27.2 °C and 51.0 °C. Approximately one gram of fibre was spread as a thin layer on a flat silica dish and allowed to remain in the prepared atmosphere for the required time. To determine the equilibrium moisture content, the sample was quickly transferred to a stoppered weighing bottle and weighed after cooling before and after drying to constant mass at 105 °C. The technique was found to operate satisfactorily even when the samples were in an atmosphere of 100% humidity, when water was the air-conditioning reagent. He estimated a value of 35% Brix-free water at 100% relative humidity.

When the log of adsorbed water per 100 g dry fibre was plotted as ordinate against the log of water vapour pressure as abscissa, for each temperature, there exist two equilibrium conditions for which straight lines obeying a Freundlich type of equation were shown above and below a transition point occurring at an EMC value of 14.5% of fibre (Fig 5.2).

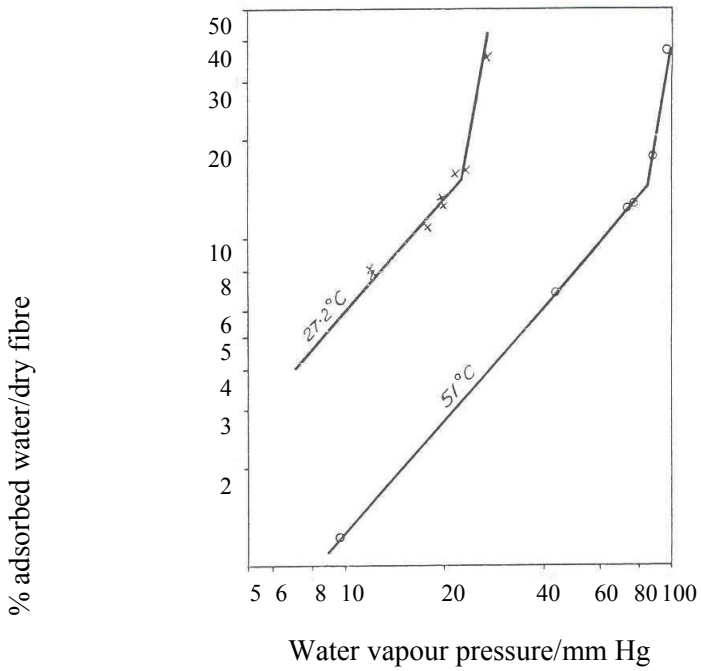


Figure 5.2. Adsorption isotherms of cane fibre (Kelly, 1957).

According to Kelly (1957), below the transition point, a primary monolayer adsorption process represented by the first part of the curve took place, and above the transition point, a secondary process involving the condensation of moisture on top of the primary adsorbed layer (multilayer adsorption) represented by the second part of the curve, occurred. For the isotherms at 27.2 °C and 51 °C, he found a similar slope (1.14) for the isotherms below the transition point and a similar, but much steeper, slope (5.55) above the transition point. Based on these similar slopes, he calculated that the heat of adsorption involved in the primary process to be 43 cal g^{-1} (i.e. 180 J g^{-1}) or 775 cal mol^{-1} (i.e. 3240 J mol^{-1}), and in the secondary process to be 19 cal g^{-1} (i.e. 80 J g^{-1}) or 342 cal mol^{-1} (i.e. 1430 J mol^{-1}).

Kelly concluded that the adsorbed water is firmly attached to the sugar cane fibre, and part of this water represents the so-called 'hygroscopic water' of Behne (1937), which is also known as Brix-free water.

Foster (1962) conducted vapour sorption experiments on pre-dried cane fibre of variety Q 50 at 20 °C for 35 – 95% relative humidity. He subsequently extrapolated his results to 100% relative humidity and quoted a value of 25% for Brix-free water.

Bruijn (1963) studied the mass increase of initially dry cane fibre exposed to humid air; the water adsorbed was reported to be as high as about 40 g per 100 g fibre at room temperature.

A recent publication on a sorption study of cane rind fibre by Han and Wu (2004) described the division of sugar cane rind stem with a thickness of 1.0 to 2.0 mm into segments of 10 to 25 cm in length separated by nodes. The rind stem consisted, on the cross-section, of an outer wax layer, rind fibres and inner pith. Samples were conditioned to reach equilibrium at relative humidity, R_H, values of 32.5, 66, 76, 81 and 93% over different saturated salt solutions in desiccators. Experimental data of the EMC at various R_H values were fitted to the sorption isotherm model proposed by Nelson (1983)

$$\text{EMC} = m_v\left\{1.0 - \frac{1}{A}\ell n\left[\left(-\frac{RT}{m_w}\right)\ell n(R_H)\right]\right\}$$

where m_v is a material constant which approximates the fibre saturation point for desorption (%), m_w is the molar mass of water (18.02 g mol^{-1}), R is the universal gas constant (8.314 J mol^{-1} K^{-1}), T is the absolute temperature (K), and A is the natural logarithm of the Gibbs free energy per gram of sorbed water as R_H approaches zero.

They found that Nelson's sorption isotherm accurately reproduced the experimental data of sugar cane rind.

5.4.2 Woody fibre from eucalyptus

In order to design and simulate the pneumatic drying operation of medium density fibreboard, Moreira *et al.* (2001) studied eucalyptus fibre to determine the equilibrium conditions of the fibre with respect to the relative humidity of the air during desorption. Triplicate samples of fresh fibre (about 0.3 g) were placed in petri-dishes in bottles containing saturated salt solutions providing a range of relative humidities from 0.07 to 0.91. The bottles were placed in an oven at 70, 55, 40 and 25 °C until equilibrium was reached (3 – 4 weeks). The equilibrium moisture content was then determined in a vacuum oven at 70 °C and 0.1 bar.

The sorption isotherms of eucalyptus were found to be of the type II isotherm, and the three-parameter GAB model was found to fit the experimental data adequately.

5.4.3 Other woody fibre from flax, hemp and reed canary grass

Increased awareness of sustainable production has led to the search for new environmentally friendly products. Natural fibre from flax, hemp and reed canary grass are alternatives that can replace synthetic fibres (e.g. carbon, glass and polyester fibres) in many industrial applications; in addition, they are produced from renewable materials and can be degraded biologically after use. A deeper knowledge of the adsorption characteristics of fibre from flax, hemp and reed canary grass was therefore essential in the management of harvest and post-harvest operations, as well as in the improvement of quality control during storage.

Fibre from flax, hemp and reed canary grass was studied by Nilsson *et al.* (2005). The plant material was cut in lengths of about 200 mm and placed on perforated trays in an oven at 50 °C to dry to about 5 – 7% moisture. The trays were then placed in climate chambers connected to air-conditioning units, in which the temperature and relative humidity were controlled. Experiments were carried out at 5, 15 and 25 °C over a range of relative humidities from 35 – 90%. When equilibrium was reached, the EMC of the samples was determined by oven drying at 105 °C for 24 hours.

Five commonly used isotherm models, namely the modified Henderson, the modified Chung-Pfost, the modified Halsey, the modified Oswin and the modified GAB models were examined. The modified Halsey model was best for predicting the EMC of un-retted flax and spring-harvested reed canary grass, the modified Oswin model for dew-retted flax and un-retted hemp, while the modified Chung-Pfost was best for frost-retted hemp.

5.4.4 Fibre from corn stover components

Igathinathane *et al.* (2005) determined the EMC and equilibrium relative humidity of corn leaf, stalk skin and stalk pith of corn stover, which is the aboveground components of the corn plant minus grain and cob. They used a static gravimetric method at 10, 20, 25, 30, 35 and 40 °C and at ten equilibrium relative humidity values ranging from 0.11 to 0.98 obtained from saturated salt solutions. The results showed that corn stover components followed a type II isotherm. The experimental data were fitted with the isotherm models of Henderson, modified Henderson, Chung-Pfost, modified Chung-Pfost, Halsey, modified Halsey, Oswin, modified Oswin and GAB. The modified Oswin followed by the modified Halsey model produced the best fit for the corn stover components.

After measuring the EMC of dry corn stalk skin and stalk pith separately, Igathinathane *et al.* (2005) proposed that the corn stalk EMC could be estimated from the sum of the dry mass fraction and the measured EMC of each of the components, stalk skin and stalk pith, as mentioned in Section 4.7.4.

5.5 METHODS FOR MEASURING EQUILIBRIUM MOISTURE CONTENT

Rouquerol *et al.* (1999) described methods to determine the equilibrium moisture content of dry material by making use of one of three physical properties: pressure, gas flow or mass in an environment of constant temperature and constant relative humidity.

In gas adsorption manometry, the pressure of the gas is measured in a calibrated, constant volume environment, at a known temperature.

In gas flow techniques, a gas flowmeter is used to determine the amount adsorbed. The flowmeter can be of a differential type (Nelsen and Eggertsen, 1958) or a simple form with either a sonic nozzle (Rouquerol, 1972; and Grillet *et al.*, 1977) or a thermal detector, which emits a signal depending on the heat capacity, thermal conductivity or the mass flow of the gas (Pieters and Gates, 1984).

In gas adsorption gravimetry as used by McBain and Bakr (1926), a spring balance is utilised to measure the mass adsorbed. The apparatus consists of an adsorbent bucket attached to the lower end of a fused silica spring, suspended within a vertical glass tube, connected to a mercury manometer as shown below in Fig 5.3.

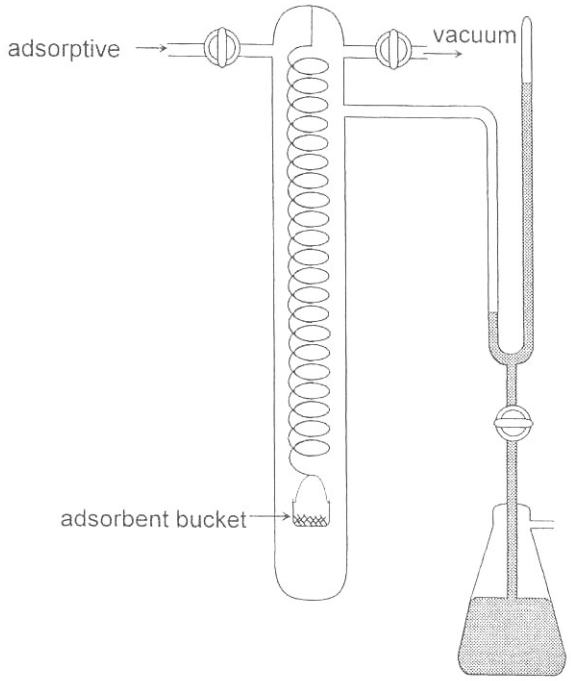


Figure 5.3. The McBain spring adsorption balance (McBain and Bakr, 1926).

The most commonly used modern methods for determining the equilibrium moisture content can be classified into three categories: gravimetric, either static or dynamic; manometric and hygrometric (Gal, 1981).

5.5.1 Gravimetric method

The gravimetric method involves the measurement of mass changes, which can be determined either continuously or discontinuously in static or dynamic systems. Continuous methods employ the use of electro-balances or quartz spring balances. In the discontinuous systems, salt or sulfuric acid solutions are placed in vacuum or atmospheric systems with the test sample, to give a measure of the equilibrium relative humidity.

A simple static procedure is to place a number of pre-dried samples in ordinary laboratory desiccators containing saturated salt solutions or sulfuric acid solutions of known concentrations which produce atmospheres of known relative humidity. The desiccators are maintained at constant temperature. The sample in each desiccator is weighed periodically until a constant mass is reached. The moisture content at this constant mass represents the EMC under those particular conditions of relative humidity and temperature. The EMC value of many materials depends on the direction in which the equilibrium is

approached, that is, either by adsorption to constant mass or desorption of the moisture at 105 °C after adsorption. A higher value is obtained when a wet material loses moisture by desorption than when a dry material gains it by adsorption (McMinn and Magee, 2003; Al-Muhtaseb *et al.*, 2004b; Arslan and Toğrul, 2005), in which case, the material is said to exhibit hysteresis.

In the dynamic method, air is moved mechanically and is often moistened by air conditioning units (Brooker *et al.*, 1992; Viswanathan *et al.*, 2003). The pre-dried sample may be placed in a U-tube through which is drawn a continuous flow of controlled-humidity air. The equipment is maintained at constant temperature. Again the sample is weighed periodically until a constant mass is reached.

5.5.2 Manometric method

The manometric method measures the vapour pressure of water in the vapour space surrounding the test sample. The whole system is maintained at constant temperature and the test sample will lose water to equilibrate with the vapour space. This will be indicated by the difference in height on the manometer (Gal, 1981).

5.5.3 Hygrometric method

The hygrometric method measures the equilibrium relative humidity of air in contact with the test sample, at a given moisture content. Dew-point hygrometers detect the condensation of cooling water vapour. Electric hygrometers measure the change in conductance or capacitance of hygrosensors. Most hygrosensors are coated with a hygrocopic salt, such as lithium chloride, which absorbs moisture from the test sample (Gal, 1981).

5.6 DETERMINATION OF ADSORPTION ISOTHERMS FOR COMPONENT PARTS OF CANE VARIETY R 570

The aims of the work described in this chapter were (i) to determine the water adsorption for the fibres of nine component parts of variety R 570 of two ages (52 and 36 weeks) at four temperatures (30, 45, 55 and 60 °C) by means of a static method (ii) to model the sorption behaviour by testing the goodness of fit of 17 commonly used models and (iii) to evaluate the constants of the isotherm models.

5.6.1 Materials

Water sorption isotherms were determined for sugar cane variety R 570. Since the water activity of a material can be greatly affected by maturity, two ages were examined: 52 and 36 weeks. For each age, nine cane component parts, namely: stalk fibre and pith, rind fibre and fines, top fibre, dry leaf fibre and fines, and green leaf fibre and fines obtained as described in Section 3.4.3.2 were studied.

The three replicates of stalk fibre aged 52 weeks were well mixed separately, a representative sample (30 g) was taken from each replicate to obtain a mixture of 90 g. Subsamples of one gram of this mixture were weighed out in plastic bags, and the bags sealed ready for use. The other eight cane component parts of R 570 aged 52 weeks were treated in the same way. Subsamples of the nine component parts of R 570 aged 36 weeks were similarly obtained.

An atmosphere of constant relative humidity can be created with sulfuric acid solutions of varying concentrations. Published data exist for the various concentrations of sulfuric acid solutions corresponding to particular water vapour pressures, e.g. Perry and Chilton (1973) and Perry *et al.* (1984); or to water activity (Rüegg, 1980). An extract of relevant data from the former is reproduced in Table 5.3, and from the latter in Table 5.4.

Table 5.3. Vapour pressure of aqueous sulfuric acid solutions at selected concentrations and temperatures (Perry and Chilton, 1973).

Concentration of sulfuric acid solution/% (m/m)	Vapour pressure/mm Hg				Latent heat of pure water /kJ mol^{-1}
	30 °C	45 °C	55 °C	60 °C	
5*	32.0	72.0	118.0	146.0	-
10	30.0	68.1	113.0	143.0	43.24
20	27.8	63.3	106.0	133.0	43.40
30	23.8	54.7	91.0	116.0	43.47
40	18.0	41.0	69.0	87.3	44.00
50	11.3	26.7	45.5	58.0	45.05
60	5.4	13.0	22.7	29.3	47.01

* The vapour pressure values for 5% sulfuric acid at all temperatures have been extrapolated from the higher concentration data.

Table 5.4. Water activity (a_w) of aqueous sulfuric acid solutions at selected concentrations and temperatures (Rüegg, 1980).

Concentration of sulfuric acid solution/% (m/m)	Water activity a_w			
	30 °C	45 °C	55 °C*	60 °C
5	0.9808	0.9812	0.9816	0.9818
10*	0.9746	0.9751	0.9760	0.9764
20	0.8814	0.8839	0.8868	0.8882
30	0.7549	0.7629	0.7684	0.7711
40	0.5711	0.5866	0.5948	0.5989
50	0.3574	0.3765	0.3879	0.3936
60	0.1677	0.0548	0.1937	0.1988

* The water activity values for all sulfuric acid concentrations at 55 °C and for the 10% (m/m) sulfuric acid solution at all temperatures have been interpolated from the other data.

Bulk solutions of 2 kg sulfuric acid reagent were prepared from BDH laboratory reagent grade sulfuric acid of 96% purity and kept in winchesters ready for use. Since the purity of the acid was only 96%, the mass of acid used per 2 kg solution was as indicated in Table 5.5.

Table 5.5. Masses of reagents required to prepare 2 kg sulfuric acid solutions of various concentrations.

Concentration of sulfuric acid solution/% (m/m)	Mass of 96% H_2SO_4/g	Mass of distilled water/g
5	104.2	1895.8
10	208.3	1791.7
20	416.7	1583.3
30	625.0	1375.0
40	833.3	1166.7
50	1041.7	958.3
60	1250.0	750.0

5.6.2 Equipment

Instead of the flat silica dishes used by Kelly (1957) to spread sugar cane fibre before exposure to a prepared atmosphere, alternative options were evaluated in this work to avoid loss of sample and the risk of desorption of the adsorbed water from taking place. Glass bottles of 60 mm diameter and 70 mm height with snap-on metallic caps were found

convenient to hold one gram of cane fibre sample. Nine of these glass bottles could easily be accommodated in a desiccator of 210 mm diameter and 190 mm height (Fig 5.4), and two of these desiccators could easily be placed inside a standard air oven (make Memmert) (Fig 5.5).

Figure 5.4.

Desiccator containing nine cane components in glass bottles above an aqueous solution of sulfuric acid.

Figure 5.5.

Two desiccators accommodated inside a standard air oven.

The cane fibre samples were dried prior to subjecting them to a constant relative humidity environment. This was particularly important when the sulfuric acid solution was of high concentration and the temperature was low. Drying was effected in a vacuum oven of Gallenkamp make with a power rating of 1000 W and a capacity of 31 L connected to a Fisons single stage vacuum pump of 50 Hz and 185 W. Dishes containing silica gel were placed in the oven to absorb moisture.

A four-decimal place analytical balance (Mettler AE200) was used for all mass determinations. The balance was calibrated against a standard mass of 100 g each day before use.

5.6.3 Procedure to determine equilibrium moisture content of sugar cane components

The nine cane component parts of the same age (1 g) were placed in separate pre-weighed empty glass bottles, and dried in a vacuum oven (without applying vacuum) at 65 °C for about two hours. During this period, the samples were stirred two or three times by using a spatula. The vacuum was then applied and drying continued at 65 °C and 900 mbar overnight. These conditions were used since it has been reported that drying under a harsh regime could cause irreversible changes in the fibre which could lose its adsorptive power (Nilsson *et al.*, 2005). The next day, the glass bottles were removed from the oven, stoppered, cooled in desiccators and weighed.

Aqueous sulfuric acid solution (500 mL) of known concentration (e.g. 5%) was poured into a desiccator. The nine glass bottles containing the dried samples were placed with the covers off onto a support with perforated holes above the sulfuric acid solution, the desiccator was closed and placed in an air oven heated at 30 °C. Since the air oven could accommodate two desiccators at a time, a second series of nine dried samples could be examined in an atmosphere of 10% sulfuric acid at 30 °C. The masses of the samples were checked daily and weighing was accomplished as fast as possible. The samples were allowed to reach equilibrium which could take from one to five days depending on the acid concentration and temperature used. Preliminary tests showed that the speed at which the nine components reached equilibrium did not differ much, but it varied tremendously with sulfuric acid concentration and temperature. The slowest was at low acid concentration and high temperature, e.g. 5 days in 5% sulfuric acid at 60 °C, and the fastest was at high acid strength and low temperature, about one day in 60% sulfuric acid at 30 °C (Table 5.6).

Equilibrium was considered to be attained when no discernible mass change (of < 0.005 g) was detected between two consecutive measurements. Typical equilibration times are indicated in Table 5.6.

After equilibrium was achieved, the glass bottles were removed from the desiccator, stoppered firmly, cooled in desiccators and weighed. The moisture adsorbed was then determined by drying at 105 °C in an air oven to constant mass (about three hours), stoppering, cooling in desiccator and weighing. The sulfuric acid solution and the samples were discarded after each determination.

Determinations were effected for 5, 10, 20, 30, 40, 50 and 60% (m/m) sulfuric acid at each of 30, 45, 55 and 60 °C. Duplicate determinations were carried out for each acid concentration and temperature.

The whole experiment was repeated for the nine cane components of variety R 570 aged 36 weeks.

A number of precautions were taken:

- A mercury thermometer was placed in the middle of the air oven to ensure that the required temperature was reached, although there was a dial gauge thermometer on the oven.
- Half-way through the adsorption process, the samples were gently shaken to break up any lumps formed. This was necessary especially with fine samples, e.g. pith, which had a tendency to cake.
- At high temperature and high humidity, any condensation collected after equilibrium on or in the bottle, was wiped dry before weighing.
- During drying at 105 °C, the samples were shaken gently to allow all moisture to escape.

Table 5.6. Number of days to reach equilibrium in aqueous sulfuric acid at various temperatures.

Concentration of sulfuric acid solution/% (m/m)	30 °C	45 °C	55 °C	60 °C
5	4	4	4	5
10	3	3	4	4
20	2	2	2	2
30		1	1	
40	1		1	
50				
60	1	1		1

5.6.4 Results and discussion

An example of a typical set of EMC results is presented in Table 5.7, and calculations are shown for the amount of water adsorbed per 100 g sample both after vacuum oven drying at 65 °C under 900 mbar vacuum and after air oven drying at 105 °C. It can be seen that vacuum oven drying at 65 °C did not completely drive out all the moisture in the samples. The residual moisture in the nine types of samples was calculated and shown in Table 5.7.

The raw data used to calculate the EMC values for the cane components aged 52 weeks and 36 weeks are presented on the compact disc (files: EMC 52 weeks.xls and EMC 36 weeks.xls).

5.6.4.1 *EMC results*

The EMC results for different water activities for the nine cane component parts aged 52 weeks and 36 weeks are summarised in Tables 5.8 – 5.16.

Table 5.7. Typical EMC results of nine sugar cane components at 30 °C under atmosphere of 10 and 20% sulfuric acid.

10% sulfuric acid at 30ºC

	Mixture of triplicates R570 aged 52 weeks	Mass of empty bottle/g	Mass of bottle + "dry" sample/g	Mass of bottle + sample after equilibrium/g	Mass of bottle + sample/g after drying at 105°C	Residual moisture/% after vacuum oven drying	Mass/g of dry sample (m_1)	Mass of moisture/g gained at equilibrium (m_2)	Equilibrium moisture content/% = $100m_2/m_1$
1	Stalk fibre	92.8647	93.8976	94.0860	93.8779	2.73	1.0132	0.2081	20.54
2	Stalk pith	93.2474	94.2588	94.4947	94.2358	4.00	0.9884	0.2589	26.19
3	Rind fibre	91.6727	92.5348	92.6840	92.5172	4.66	0.8445	0.1668	19.75
4	Rind fines	93.5350	94.4518	94.6163	94.4360	3.12	0.9010	0.1803	20.01
5	Top	91.9143	92.7810	92.9717	92.7385	2.65	0.8242	0.2332	28.29
6	Dry leaves fibre	91.3431	92.3068	92.5004	92.2631	3.33	0.9200	0.2373	25.79
7	Dry leaves fines	91.9515	92.8675	93.0607	92.8495	3.78	0.8980	0.2112	23.52
8	Green leaves fibre	92.2999	93.2865	93.4892	93.2696	4.42	0.9697	0.2196	22.65
9	Green leaves fines	92.9738	93.9486	94.1517	93.9051	3.79	0.9313	0.2466	26.48

20% sulfuric acid at 30ºC

	Mixture of triplicates R570 aged 52 weeks	Mass of empty bottle/g	Mass of bottle + "dry" sample/g	Mass of bottle + sample after equilibrium/g	Mass of bottle + sample/g after drying at 105°C	Residual moisture/% after vacuum oven drying	Mass/g of dry sample (m_1)	Mass of moisture/g gained at equilibrium (m_2)	Equilibrium moisture content/% = $100m_2/m_1$
1	Stalk fibre	90.0370	90.9952	91.1157	90.9784	2.94	0.9414	0.1373	14.58
2	Stalk pith	93.3375	94.3522	94.5116	94.3310	3.96	0.9935	0.1806	18.18
3	Rind fibre	90.4363	91.4232	91.5451	91.4077	3.43	0.9714	0.1374	14.14
4	Rind fines	96.4609	97.4557	97.5802	97.4392	2.70	0.9783	0.1410	14.41
5	Top	97.9062	98.8040	98.9341	98.7588	1.75	0.8526	0.1753	20.56
6	Dry leaves fibre	96.8486	97.8598	97.9987	97.8401	3.33	0.9915	0.1586	16.00
7	Dry leaves fines	90.0554	91.0211	91.1571	90.9997	3.41	0.9443	0.1574	16.67
8	Green leaves fibre	93.8058	94.7114	94.8389	94.6946	3.10	0.8888	0.1443	16.24
9	Green leaves fines	90.8766	91.8400	91.9772	91.8224	3.55	0.9458	0.1548	16.37

Table 5.8. Equilibrium moisture content (on a dry basis) of **stalk fibre** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C			45 °C			55 °C			60 °C		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	23.60	24.29	0.9812	25.21	25.79	0.9816	26.26	26.79	0.9818	31.04	26.52
		26.08	24.10		27.46	27.47		30.71	27.32		33.40	21.18
mean		24.84	24.20		26.34	26.63		28.49	27.06		32.22	23.85
10%	0.9746	20.54	25.63	0.9751	25.46	25.98	0.9760	30.66	27.94	0.9764	32.82	27.11
		19.81	22.30		23.76	29.53		24.81	26.44		24.42	27.76
mean		20.18	23.97		24.61	27.76		27.74	27.19		28.62	27.44
20%	0.8814	15.26	16.03	0.8839	16.54	15.38	0.8868	16.02	16.04	0.8882	16.06	15.19
		17.28	14.94		15.30	15.55		14.67	13.12		14.39	15.58
mean		16.27	15.49		15.92	15.47		15.35	14.58		15.23	15.39
30%	0.7549	13.79	12.41	0.7629	14.45	12.26	0.7684	12.66	10.32	0.7711	11.15	11.83
		15.20	10.66		11.24	12.70		10.53	11.63		10.88	10.32
mean		14.50	11.54		12.85	12.48		11.60	10.98		11.02	11.08
40%	0.5711	9.65	8.21	0.5866	9.60	7.66	0.5948	7.76	8.18	0.5989	7.89	7.49
		7.62	7.65		11.05	7.79		7.85	7.86		8.04	7.59
mean		8.64	7.93		10.33	7.73		7.81	8.02		7.97	7.54
50%	0.3574	5.40	4.69	0.3765	5.16	5.40	0.3879	5.33	5.55	0.3936	5.32	5.10
		5.30	6.89		5.34	4.53		4.83	5.59		4.57	4.04
mean		5.35	5.79		5.25	4.97		5.08	5.57		4.95	4.57
60%	0.1677	4.93	4.15	0.1834	3.47	3.38	0.1937	3.35	3.56	0.1988	3.21	2.45
		4.33	3.92		3.51	3.26		2.80	3.16		3.32	2.76
mean		4.63	4.04		3.49	3.32		3.08	3.36		3.27	2.61

Table 5.9. Equilibrium moisture content (on a dry basis) of **stalk pith** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 ºC			45 ºC			55 ºC			60 ºC		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	35.71	31.99	0.9812	31.66	37.04	0.9816	35.01	36.50	0.9818	42.88	38.91
		34.17	31.29		38.31	34.10		39.26	36.83		40.19	27.25
mean		34.94	31.64		34.99	35.57		37.14	36.67		41.54	33.08
10%	0.9746	26.19	27.24	0.9751	40.32	37.62	0.9760	46.67	37.94	0.9764	40.56	37.94
		28.33	23.79		39.51	33.04		42.34	36.79		41.96	42.55
mean		27.26	25.52		39.92	35.33		44.51	37.37		41.26	40.25
20%	0.8814	22.11	19.45	0.8839	24.08	18.05	0.8868	19.55	19.80	0.8882	21.87	18.60
		20.91	18.55		21.13	17.87		22.42	18.32		18.19	17.19
mean		21.51	19.00		22.61	17.96		20.99	19.06		20.03	17.90
30%	0.7549	15.05	14.30	0.7629	13.80	15.44	0.7684	12.97	16.10	0.7711	12.66	12.53
		13.97	12.93		13.20	16.64		12.71	13.25		14.22	14.17
mean		14.51	13.62		13.50	16.04		12.84	14.68		13.44	13.35
40%	0.5711	12.92	11.76	0.5866	9.51	9.15	0.5948	9.05	7.15	0.5989	9.31	7.69
		9.23	10.96		9.70	9.75		8.92	8.53		8.90	6.41
mean		11.08	11.36		9.61	9.45		8.99	7.84		9.11	7.05
50%	0.3574	6.62	5.73	0.3765	8.23	5.76	0.3879	6.54	4.01	0.3936	5.89	7.89
		8.12	6.28		7.87	6.62		6.31	5.28		3.79	3.57
mean		7.37	6.01		8.05	6.19		6.43	4.65		4.84	5.73
60%	0.1677	8.08	5.22	0.1834	6.68	4.09	0.1937	3.61	3.90	0.1988	2.42	4.78
		4.85	5.00		4.76	4.05		3.44	3.81		3.36	4.88
mean		6.47	5.11		5.72	4.07		3.53	3.86		2.89	4.83

Table 5.10. Equilibrium moisture content (on a dry basis) of **rind fibre** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C			45 °C			55 °C			60 °C		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	21.73	21.66	0.9812	23.74	22.21	0.9816	22.89	24.78	0.9818	29.61	24.19
		22.30	21.60		24.44	24.31		30.26	30.40		44.89	21.41
mean		22.02	21.63		24.09	23.26		26.58	27.59		37.25	22.80
10%	0.9746	19.75	19.78	0.9751	24.24	21.46	0.9760	26.55	22.91	0.9764	32.97	24.97
		22.06	20.19		21.44	23.66		23.69	21.37		19.00	20.62
mean		20.91	19.99		22.84	22.56		25.12	22.14		25.99	22.80
20%	0.8814	16.64	18.21	0.8839	16.33	14.99	0.8868	15.52	15.58	0.8882	15.38	12.99
		14.34	14.57		15.06	15.28		14.22	14.68		14.21	14.77
mean		15.49	16.39		15.70	15.14		14.87	15.13		14.80	13.88
30%	0.7549	11.41	11.85	0.7629	13.84	10.65	0.7684	13.60	10.92	0.7711	9.08	11.17
		14.65	12.49		10.42	12.22		9.76	9.61		10.14	9.54
mean		13.03	12.17		12.13	11.44		11.68	10.27		9.61	10.36
40%	0.5711	9.57	7.91	0.5866	8.96	7.39	0.5948	5.80	7.71	0.5989	7.13	10.02
		7.63	8.97		10.86	7.65		7.19	7.47		8.71	7.00
mean		8.60	8.44		9.91	7.52		6.50	7.59		7.92	8.51
50%	0.3574	5.07	4.54	0.3765	4.95	5.15	0.3879	5.14	4.78	0.3936	4.51	6.65
		6.97	4.98		5.98	5.14		3.98	5.10		4.43	4.58
mean		6.02	4.76		5.47	5.15		4.56	4.94		4.47	5.62
60%	0.1677	3.52	4.04	0.1834	2.66	3.08	0.1937	3.17	3.34	0.1988	3.07	2.01
		4.82	7.06		3.15	2.07		1.84	3.05		2.79	2.83
mean		4.17	5.55		2.91	2.58		2.51	3.20		2.93	2.42

Table 5.11. Equilibrium moisture content (on a dry basis) of **rind fines** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C			45 °C			55 °C			60 °C		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	23.55	23.35	0.9812	24.78	24.73	0.9816	24.34	23.00	0.9818	22.28	24.80
		24.07	23.00		25.53	26.26		24.84	24.95		24.78	17.79
mean		23.81	23.18		25.16	25.50		24.59	23.98		23.53	21.30
10%	0.9746	20.01	19.77	0.9751	25.15	20.13	0.9760	22.38	18.39	0.9764	28.52	17.83
		19.45	18.60		20.22	21.24		21.75	17.77		18.40	17.90
mean		19.73	19.19		22.69	20.69		22.07	18.08		23.46	17.87
20%	0.8814	15.19	15.28	0.8839	16.80	14.29	0.8868	16.46	14.51	0.8882	16.31	13.53
		14.77	15.09		15.18	14.92		14.77	13.29		14.47	11.81
mean		14.98	15.19		15.99	14.61		15.62	13.90		15.39	12.67
30%	0.7549	11.64	12.07	0.7629	10.84	11.89	0.7684	10.53	8.81	0.7711	10.22	10.25
		11.39	10.77		12.63	13.64		7.71	9.85		10.74	11.00
mean		11.52	11.42		11.74	12.77		9.12	9.33		10.48	10.63
40%	0.5711	9.50	8.15	0.5866	8.02	7.60	0.5948	6.77	5.25	0.5989	6.76	7.20
		7.51	7.73		9.22	7.72		5.25	7.49		5.26	7.82
mean		8.51	7.94		8.62	7.66		6.01	6.37		6.01	7.51
50%	0.3574	5.42	4.86	0.3765	6.57	5.24	0.3879	4.99	4.80	0.3936	4.45	4.94
		5.53	5.29		5.17	5.32		4.54	5.07		4.50	5.83
mean		5.48	5.08		5.87	5.28		4.77	4.94		4.48	5.39
60%	0.1677	4.25	4.17	0.1834	3.16	3.28	0.1937	3.01	3.36	0.1988	3.13	3.16
		3.91	4.08		3.13	3.15		2.76	1.72		3.04	2.90
mean		4.08	4.13		3.15	3.22		2.89	2.54		3.09	3.03

Table 5.12. Equilibrium moisture content (on a dry basis) of **top fibre** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C			45 °C			55 °C			60 °C		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	29.12	30.45	0.9812	32.40	38.38	0.9816	46.16	41.71	0.9818	53.30	43.83
		32.11	29.61		33.79	35.46		43.96	46.82		39.60	34.81
mean		30.62	30.03		33.10	36.92		45.06	44.27		46.45	39.32
10%	0.9746	28.29	26.81	0.9751	30.20	27.81	0.9760	26.52	28.12	0.9764	27.98	28.69
		24.34	23.54		27.45	25.68		24.14	24.38		31.35	24.44
mean		26.32	25.18		28.83	26.75		25.33	26.25		29.67	26.57
20%	0.8814	22.32	17.95	0.8839	19.19	16.83	0.8868	18.98	18.52	0.8882	21.38	15.45
		17.22	18.79		17.84	17.99		16.82	17.94		19.95	15.45
mean		19.77	18.37		18.52	17.41		17.90	18.23		20.67	15.45
30%	0.7549	14.74	16.07	0.7629	12.55	14.35	0.7684	15.35	11.42	0.7711	11.62	12.71
		16.65	13.83		12.84	15.72		9.64	12.16		12.78	11.15
mean		15.70	14.95		12.70	15.04		12.50	11.79		12.20	11.93
40%	0.5711	11.57	9.25	0.5866	9.29	8.79	0.5948	9.41	7.50	0.5989	8.24	8.60
		8.58	10.48		8.14	8.49		8.42	8.78		8.07	6.68
mean		10.08	9.87		8.72	8.64		8.92	8.14		8.16	7.64
50%	0.3574	6.18	6.14	0.3765	5.79	5.27	0.3879	5.80	5.63	0.3936	5.40	5.53
		6.42	5.99		8.74	9.13		5.39	4.50		3.45	5.09
mean		6.30	6.07		7.27	7.20		5.60	5.07		4.43	5.31
60%	0.1677	4.23	4.81	0.1834	3.43	3.72	0.1937	3.76	4.08	0.1988	3.55	5.14
		7.21	4.41		3.88	3.74		3.64	2.96		3.70	2.07
mean		5.72	4.61		3.66	3.73		3.70	3.52		3.63	3.61

Table 5.13. Equilibrium moisture content (on a dry basis) of **dry leaf fibre** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C			45 °C			55 °C			60 °C		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	26.21	28.02	0.9812	31.59	33.21	0.9816	31.65	33.21	0.9818	48.23	64.56
		27.59	27.02		27.58	28.35		36.35	26.59		40.56	54.97
mean		26.90	27.52		29.59	30.78		34.00	29.90		44.40	59.77
10%	0.9746	25.49	25.58	0.9751	24.61	27.74	0.9760	26.14	25.09	0.9764	26.03	19.24
		22.04	24.34		24.28	25.27		19.67	22.33		33.18	21.42
mean		23.77	24.96		24.45	26.51		22.91	23.71		29.61	20.33
20%	0.8814	16.75	17.12	0.8839	18.02	16.91	0.8868	16.94	17.13	0.8882	17.21	13.91
		16.35	16.40		16.44	15.43		15.85	16.38		15.82	14.74
mean		16.55	16.76		17.23	16.17		16.40	16.76		16.52	14.33
30%	0.7549	17.51	13.29	0.7629	12.51	13.18	0.7684	14.44	12.50	0.7711	10.77	10.93
		15.10	14.01		10.75	12.91		12.37	12.01		9.41	11.99
mean		16.31	13.65		11.63	13.05		13.41	12.26		10.09	11.46
40%	0.5711	10.80	8.92	0.5866	8.97	8.35	0.5948	8.58	9.73	0.5989	7.77	7.43
		8.49	8.56		8.37	8.56		8.08	8.37		9.14	4.33
mean		9.65	8.74		8.67	8.46		8.33	9.05		8.46	5.88
50%	0.3574	5.98	5.97	0.3765	5.54	8.80	0.3879	5.29	5.49	0.3936	5.26	5.45
		6.20	5.77		5.86	8.21		5.27	8.59		5.06	5.07
mean		6.09	5.87		5.70	8.51		5.28	7.04		5.16	5.26
60%	0.1677	4.11	4.83	0.1834	3.79	3.70	0.1937	3.73	4.08	0.1988	3.59	3.71
		7.36	4.59		3.89	3.72		2.64	3.71		3.66	3.53
mean		5.74	4.71		3.84	3.71		3.19	3.90		3.63	3.62

Table 5.14. Equilibrium moisture content (on a dry basis) of **dry leaf fines** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 ºC			45 ºC			55 ºC			60 ºC		
	Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)		Water activity, a_w	water adsorbed (g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	27.36	28.12	0.9812	27.76	32.63	0.9816	28.61	30.82	0.9818	34.04	31.06
		28.61	27.84		28.51	31.65		26.61	25.10		34.39	26.39
mean		27.99	27.98		28.14	32.14		27.61	27.96		34.22	28.73
10%	0.9746	23.52	23.32	0.9751	27.11	26.78	0.9760	30.08	27.78	0.9764	24.12	23.74
		22.92	21.81		25.78	27.50		24.00	26.39		32.15	27.48
mean		23.22	22.57		26.45	27.14		27.04	27.09		28.14	25.61
20%	0.8814	22.38	18.09	0.8839	18.68	17.46	0.8868	17.75	17.44	0.8882	16.89	15.52
		19.43	16.81		16.33	16.71		16.73	16.70		16.19	14.00
mean		20.91	17.45		17.51	17.09		17.24	17.07		16.54	14.76
30%	0.7549	18.51	12.94	0.7629	12.25	11.93	0.7684	11.64	12.49	0.7711	11.36	13.23
		13.52	12.14		11.72	12.25		9.32	13.87		11.32	10.98
mean		16.02	12.54		11.99	12.09		10.48	13.18		11.34	12.11
40%	0.5711	11.22	9.38	0.5866	8.79	11.60	0.5948	8.28	9.81	0.5989	8.19	8.39
		9.83	10.65		9.19	8.87		6.26	10.67		7.84	8.83
mean		10.53	10.02		8.99	10.24		7.27	10.24		8.02	8.61
50%	0.3574	7.40	6.44	0.3765	5.44	6.74	0.3879	9.48	5.60	0.3936	5.46	5.58
		8.57	6.21		6.59	6.48		4.21	6.87		4.38	7.12
mean		7.99	6.33		6.02	6.61		6.85	6.24		4.92	6.35
60%	0.1677	4.42	5.02	0.1834	3.56	3.87	0.1937	3.87	4.24	0.1988	4.04	3.40
		4.84	4.99		4.98	2.54		3.23	3.10		2.77	3.17
mean		4.63	5.01		4.27	3.21		3.55	3.67		3.41	3.29

Table 5.15. Equilibrium moisture content (on a dry basis) of **green leaf fibre** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

	30 ºC		45 ºC		55 ºC		60 ºC	
	Water	water adsorbed	Water	water adsorbed	Water	water adsorbed	Water	water adsorbed

m/m of 96% H_2SO_4 soln	activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	27.77	27.00	0.9812	27.69	30.04	0.9816	30.30	27.63	0.9818	39.92	58.83
		27.86	26.94		29.52	29.62		31.98	29.05		28.15	57.83
mean		27.82	26.97		28.61	29.83		31.14	28.34		34.04	58.33
10%	0.9746	22.65	24.31	0.9751	30.79	29.03	0.9760	33.52	31.54	0.9764	26.06	27.84
		23.70	21.73		26.86	29.97		28.90	23.49		28.02	32.58
mean		23.18	23.02		28.83	29.50		31.21	27.52		27.04	30.21
20%	0.8814	16.65	17.16	0.8839	18.56	17.53	0.8868	18.66	17.69	0.8882	18.27	16.85
		16.61	17.14		17.09	17.11		16.84	17.03		19.51	15.56
mean		16.63	17.15		17.83	17.32		17.75	17.36		18.89	16.21
30%	0.7549	14.57	12.20	0.7629	12.35	11.90	0.7684	12.77	12.49	0.7711	12.09	12.01
		14.89	12.48		11.55	12.12		9.92	11.59		9.35	11.37
mean		14.73	12.34		11.95	12.01		11.35	12.04		10.72	11.69
40%	0.5711	10.72	8.73	0.5866	10.25	8.26	0.5948	6.92	6.54	0.5989	8.39	9.16
		8.35	8.34		9.21	8.42		8.30	8.39		7.45	7.14
mean		9.54	8.54		9.73	8.34		7.61	7.47		7.92	8.15
50%	0.3574	5.80	6.05	0.3765	5.54	6.94	0.3879	5.83	5.43	0.3936	5.45	5.66
		6.00	5.92		5.78	4.99		5.32	6.52		5.17	5.29
mean		5.90	5.99		5.66	5.97		5.58	5.98		5.31	5.48
60%	0.1677	3.96	4.68	0.1834	3.84	3.64	0.1937	3.39	2.74	0.1988	3.52	3.64
		3.95	4.55		3.66	3.46		3.25	3.73		4.48	3.11
mean		3.96	4.62		3.75	3.55		3.32	3.24		4.00	3.38

Table 5.16. Equilibrium moisture content (on a dry basis) of **green leaf fines** from R 570 aged 52 weeks and 36 weeks at various temperatures and water activities.

	30 °C		45 °C		55 °C		60 °C	
	Water	water adsorbed	Water	water adsorbed	Water	water adsorbed	Water	water adsorbed

m/m of 96% H_2SO_4 soln	activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)		activity, a_w	(g/100g dry fibre)	
		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks		52 weeks	36 weeks
5%	0.9808	27.39	28.14	0.9812	30.49	29.52	0.9816	37.52	37.46	0.9818	31.34	41.77
		28.51	27.73		38.55	33.33		48.10	36.58		39.07	51.82
mean		27.95	27.94		34.52	31.43		42.81	37.02		35.21	46.80
10%	0.9746	26.48	21.92	0.9751	28.51	25.80	0.9760	30.49	23.71	0.9764	30.69	24.79
		24.77	23.70		27.57	26.06		25.76	25.33		25.82	25.80
mean		25.63	22.81		28.04	25.93		28.13	24.52		28.26	25.30
20%	0.8814	18.10	17.25	0.8839	18.03	17.01	0.8868	18.39	17.00	0.8882	16.39	16.33
		16.46	16.51		16.70	16.57		16.70	16.12		17.59	17.01
mean		17.28	16.88		17.37	16.79		17.55	16.56		16.99	16.67
30%	0.7549	17.40	12.26	0.7629	13.24	11.65	0.7684	12.05	12.02	0.7711	11.67	12.87
		12.90	11.75		11.24	11.74		13.16	11.90		11.92	11.47
mean		15.15	12.01		12.24	11.70		12.61	11.96		11.80	12.17
40%	0.5711	9.11	8.94	0.5866	8.66	8.14	0.5948	8.31	7.24	0.5989	8.60	7.26
		8.42	8.51		9.68	8.37		8.06	8.12		7.72	9.04
mean		8.77	8.73		9.17	8.26		8.19	7.68		8.16	8.15
50%	0.3574	5.99	6.52	0.3765	5.67	8.86	0.3879	5.86	5.37	0.3936	5.49	4.98
		6.22	5.97		5.86	6.21		5.37	6.07		4.96	5.93
mean		6.11	6.25		5.77	7.54		5.62	5.72		5.23	5.46
60%	0.1677	4.31	4.86	0.1834	3.97	2.66	0.1937	3.87	4.06	0.1988	3.54	3.26
		4.90	4.80		3.96	3.64		3.18	3.87		3.73	3.42
mean		4.61	4.83		3.97	3.15		3.53	3.97		3.64	3.34

5.6.4.2 *Kelly's type of two-equilibria isotherms*

When the *ln* of EMC results obtained in Tables 5.8 – 5.16 were plotted against *ln* of vapour pressure (Table 5.3), as previously described for Kelly's work (1957) in Section 5.4.1, two-equilibria isotherms were obtained for all nine cane components of R 570 aged 52 weeks (Fig 5.6) and 36 weeks (Fig 5.7). The transition point of the primary and secondary equilibria occurred at *ln* (EMC/% db) = 2.5, i.e. at EMC/% db = 12.5, compared to the value of 14.5 found by Kelly. The regression coefficient R^2 and the Freundlich constants *s* and k are compiled for 52 weeks samples (Table 5.17) and for 36 weeks samples (Table 5.18). All the slopes *s* of the primary equilibria for 30, 45, 55 and 60 °C isotherms are different for all cane components, the same applied to the slopes of the secondary equilibria for 30, 45, 55 and 60 °C isotherms. Therefore, it was not possible to calculate the heat of adsorption involved in the primary and secondary equilibria as did Kelly, since his calculations were based on the identical slope of 1.14 for the primary equilibria of both 27.2 °C and 51 °C isotherms, and 5.55 for their secondary equilibria.

When Kelly's EMC data (1957) were plotted similarly, the gradients of the primary equilibria at 27.2 °C and 51 °C were found respectively: 0.854 and 1.137, and of the secondary equilibria at 27.2 °C and 51 °C were respectively 3.305 and 4.551.

5.6.4.3 *Adsorption isotherms*

The adsorption isotherms for the nine sugar cane component parts aged 52 weeks are shown in Fig 5.8. Typical S-shape curves referred to as type II isotherms were found. According to Van den Berg and Bruin (1981), type II isotherms can be divided into three different regions: in the first region at low water activity, there is monolayer adsorption of water held by strong hydrophilic bonds on polar sites by Van der Waal forces. In the second region, called the multilayer region, water is more loosely held by hydrogen bonds and is under transition to the natural properties of free water. The least firmly bound water occurs when a_w is above 0.6. In the third region, the isotherm rises steeply as practically free water becomes mechanically entrapped in the void spaces of the material, mainly as a result of capillary condensation. Water uptake in the first region is normally rapid, slows down in the second region and is accelerated in the third region. The adsorbed water can be classified as monolayer, multilayer or condensed capillary water. The enthalpy of

vaporisation generally decreases from the first to the third region. The nine sugar cane components aged 36 weeks also exhibit type II isotherms (Fig 5.9).

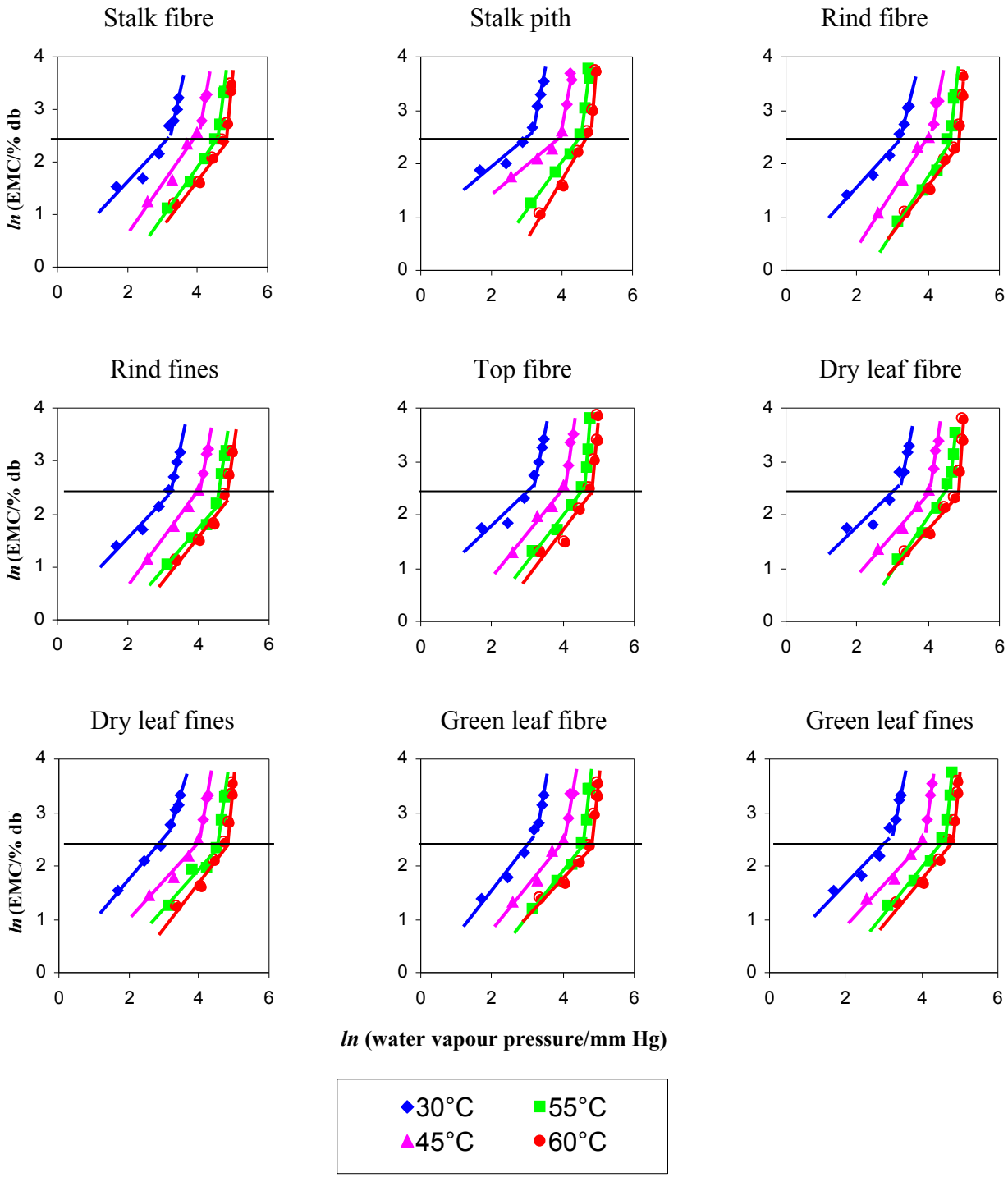


Figure 5.6. Adsorption isotherms of nine cane components aged 52 weeks (as per Kelly's method, 1957).

Note the similar behaviour of all the nine cane components.

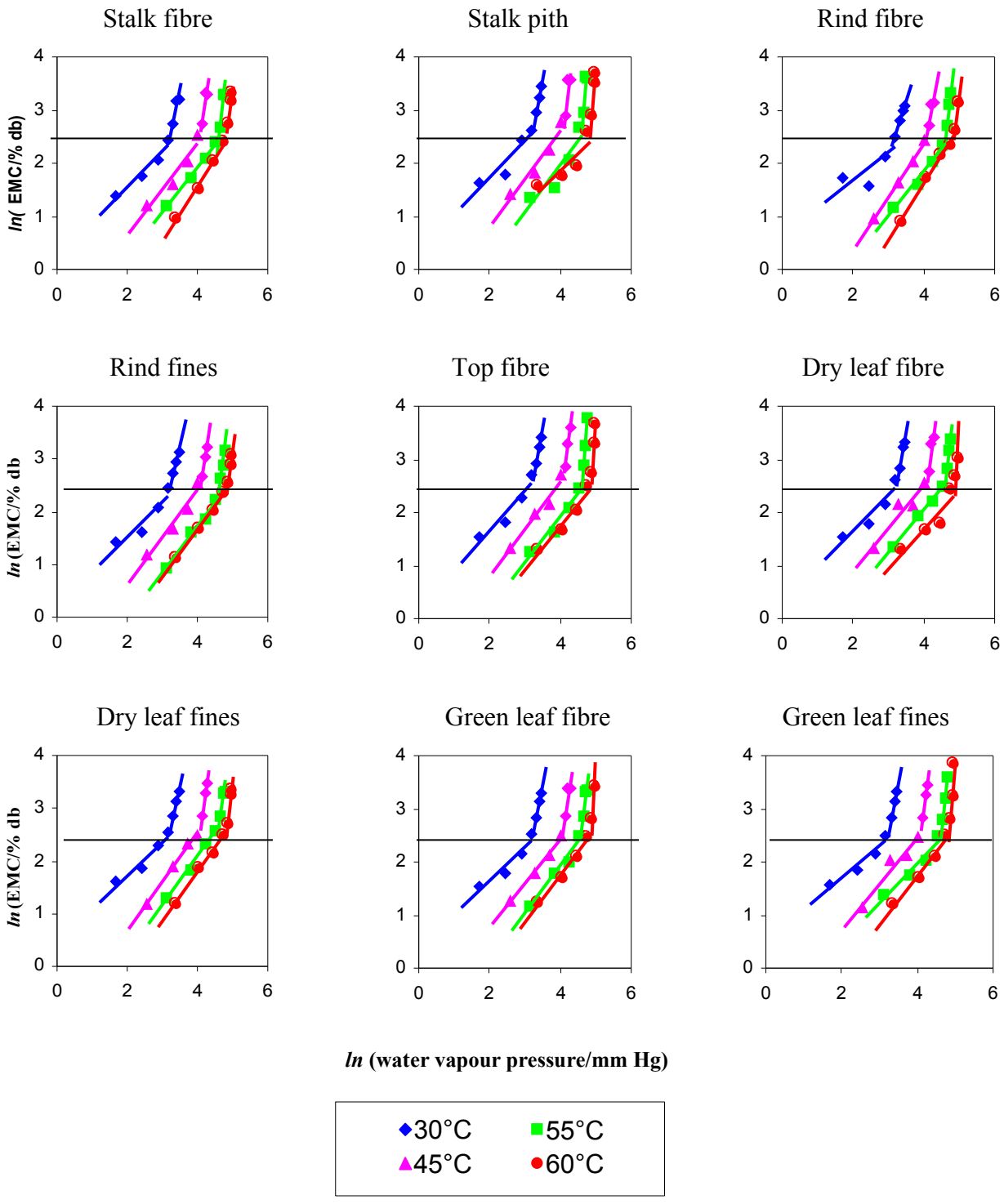


Figure 5.7. Adsorption isotherms of nine cane components aged 36 weeks (as per Kelly's method, 1957).

Note the similar behaviour of all the nine cane components.

Table 5.17. Freundlich constants for primary and secondary equilibria of adsorption isotherms of cane components aged 52 weeks.

	R^2	*s*	*ln k*	*k*	R^2	*s*	*ln k*	*k*	R^2	*s*	*ln k*	*k*
	Stalk fibre				**Stalk pith**				**Rind fibre**			
30 °C isotherm												
Primary equilibria	0.83	0.726	0.164	1.17821	0.88	0.538	0.869	2.38453	0.95	0.732	0.126	1.13428
Secondary equilibria	1.00	3.002	-7.197	7.49E-04	1.00	3.438	-8.370	2.32E-04	0.89	2.543	-5.682	3.41E-03
45 °C isotherm												
Primary equilibria	0.95	0.941	-1.243	0.288517	0.95	0.560	0.276	1.31785	0.99	1.024	-1.585	0.204948
Secondary equilibria	0.90	4.014	-13.840	9.76E-07	0.62	3.614	- 11.78	7.66E-06	0.89	3.416	-11.38	1.14E-05
55 °C isotherm												
Primary equilibria	0.98	0.933	-1.844	0.158183	0.99	0.906	-1.585	0.204948	0.96	1.044	-2.401	0.0906273
Secondary equilibria	0.87	6.030	-25.330	9.98E-12	0.64	5.807	-23.93	4.05E-11	0.90	5.626	-23.49	6.29E-11
60 °C isotherm												
Primary equilibria	0.97	0.881	-1.853	0.156766	0.97	1.121	-2.807	0.0603859	0.97	0.894	-1.999	0.135471
Secondary equilibria	1.00	8.209	-37.410	5.66E-17	0.96	8.381	-37.97	3.23E-17	0.97	9.342	-43.01	2.09E-19
	Rind fines				**Top fibre**				**Dry leaf fibre**			
30 °C isotherm												
Primary equilibria	0.95	0.691	0.166	1.18057	0.82	0.649	0.512	1.66863	0.77	0.657	0.483	1.62093
Secondary equilibria	1.00	3.303	-8.269	2.56E-04	0.98	3.129	-7.404	6.09E-04	0.95	3.491	-8.771	1.55E-04
45 °C isotherm												
Primary equilibria	1.00	0.907	-1.191	0.303917	0.98	0.825	-0.802	0.448431	0.98	0.766	-0.673	0.510176
Secondary equilibria	0.95	3.584	-12.07	5.73E-06	0.95	4.588	-16.08	1.04E-07	0.99	4.229	-14.68	4.21E-07
55 °C isotherm												
Primary equilibria	0.97	0.781	-1.406	0.245122	0.97	0.868	-1.468	0.230386	0.96	1.000	-2.037	0.130419
Secondary equilibria	0.97	4.320	-17.38	2.83E-08	0.94	8.368	-36.19	1.92E-16	0.98	6.680	-28.38	4.73E-13
60 °C isotherm												
Primary equilibria	0.91	0.824	-1.74	0.17552	0.88	0.877	-1.809	0.163818	0.97	0.768	-1.355	0.257947
Secondary equilibria	0.96	4.882	-21.13	6.66E-10	0.87	7.719	-34.75	8.10E-16	0.96	9.936	-45.81	1.27E-20
	Dry leaf fines				**Green leaf fibre**				**Green leaf fines**			
30 °C isotherm												
Primary equilibria	0.97	0.792	0.170	1.1853	0.95	0.858	-0.158	0.85385	0.88	0.742	0.169	1.18412
Secondary equilibria	0.96	2.052	-3.799	0.0223932	0.99	3.678	-9.402	8.26E-05	0.91	3.470	-8.647	1.76E-04
45 °C isotherm												
Primary equilibria	0.97	0.712	-0.433	0.64856	0.97	0.829	-0.860	0.423162	0.97	0.784	-0.694	0.499574
Secondary equilibria	0.90	3.784	-12.79	2.79E-06	0.80	3.820	-12.90	2.50E-06	0.98	5.395	-19.50	3.40E-09
55 °C isotherm												
Primary equilibria	0.95	0.726	-0.965	0.380983	0.98	0.850	-1.490	0.225373	0.97	0.883	-1.554	0.211401
Secondary equilibria	0.87	4.591	-18.52	9.05E-09	0.84	5.512	-22.77	1.29E-10	0.99	8.244	-35.60	3.46E-16
60 °C isotherm												
Primary equilibria	0.96	0.868	-1.782	0.168301	0.95	0.708	-1.077	0.340616	0.96	0.841	-1.623	0.197306
Secondary equilibria	1.00	7.675	-34.73	8.26E-16	0.97	5.958	-26.21	4.14E-12	0.99	7.607	-34.37	1.18E-15

Table 5.18. Freundlich constants for primary and secondary equilibria of adsorption isotherms of cane components aged 36 weeks.

	R^2	*s*	*ln k*	*k*	R^2	*s*	*ln k*	*k*	R^2	*s*	*ln k*	*k*
	Stalk fibre				**Stalk pith**				**Rind fibre**			
30 °C isotherm												
Primary equilibria	0.95	0.674	0.204	1.2263	0.88	0.692	0.356	1.42761	0.63	0.520	0.654	1.92322
Secondary equilibria	0.81	3.247	-7.997	3.36E-04	1.00	3.632	-9.126	1.09E-04	0.96	1.991	-3.807	0.0222147
45 °C isotherm												
Primary equilibria	0.94	0.887	-1.163	0.312547	0.94	0.909	-1.020	0.360595	0.99	1.008	-1.659	0.190329
Secondary equilibria	0.76	4.410	-15.47	1.91E-07	0.82	5.507	-19.87	2.35E-09	0.86	3.442	-11.52	9.93E-06
55 °C isotherm												
Primary equilibria	0.99	0.839	-1.437	0.23764	0.83	0.905	-1.639	0.194174	0.98	0.834	-1.491	0.225147
Secondary equilibria	0.83	6.607	-25.55	8.01E-12	0.82	6.436	-26.99	1.90E-12	1.00	5.628	-23.53	6.04E-11
60 °C isotherm												
Primary equilibria	0.99	1.042	-2.613	0.0733143	0.76	0.650	-0.740	0.477114	0.99	1.074	-2.703	0.0670042
Secondary equilibria	0.81	5.553	-24.39	2.56E-11	0.81	7.783	-35.13	5.54E-16	0.96	5.719	-25.32	1.01E-11
	Rind fines				**Top fibre**				**Dry leaf fibre**			
30 °C isotherm												
Primary equilibria	0.90	0.666	0.193	1.21288	0.92	0.769	0.126	1.13428	0.88	0.679	0.300	1.34986
Secondary equilibria	1.00	3.006	-7.273	6.94E-04	0.99	3.512	-8.753	1.58E-04	0.92	3.575	-9.030	1.20E-04
45 °C isotherm												
Primary equilibria	0.96	0.912	-1.239	0.289674	0.95	0.899	-1.008	0.364948	0.92	0.807	-0.699	0.497082
Secondary equilibria	1.00	4.347	-15.34	2.18E-07	1.00	5.839	-21.36	5.29E-10	0.95	5.088	-18.29	1.14E-08
55 °C isotherm												
Primary equilibria	0.99	0.901	-1.879	0.152743	0.95	0.856	-1.496	0.224024	0.99	0.802	-1.139	0.320139
Secondary equilibria	0.98	5.011	-20.75	9.74E-10	0.96	8.079	-34.81	7.62E-16	1.00	5.400	-22.36	1.95E-10
60 °C isotherm												
Primary equilibria	0.99	0.892	-1.921	0.14646	0.95	0.833	-1.601	0.201695	0.84	0.735	-1.272	0.280271
Secondary equilibria	0.98	5.355	-23.66	5.30E-11	0.95	9.355	-43.04	2.03E-19	0.68	12.580	-58.96	2.48E-26
	Dry leaf fines				**Green leaf fibre**				**Green leaf fines**			
30 °C isotherm												
Primary equilibria	0.94	0.625	0.483	1.62093	0.92	0.633	0.384	1.46815	0.94	0.592	0.510	1.66529
Secondary equilibria	1.00	3.356	-8.300	2.49E-04	0.98	3.237	-7.906	3.69E-04	1.00	3.593	-9.111	1.10E-04
45 °C isotherm												
Primary equilibria	0.99	0.945	-1.237	0.290254	0.99	0.823	-0.877	0.416029	0.95	0.870	-1.016	0.36204
Secondary equilibria	0.97	4.977	-17.78	1.90E-08	0.83	4.377	-15.24	2.41E-07	0.98	4.923	-17.58	2.32E-08
55 °C isotherm												
Primary equilibria	0.99	0.932	-1.645	0.193013	0.97	0.888	-1.616	0.198692	0.94	0.748	-1.023	0.359515
Secondary equilibria	0.88	4.800	-19.50	3.40E-09	0.88	4.769	-19.34	3.99E-09	0.98	7.398	-31.72	1.68E-14
60 °C isotherm												
Primary equilibria	1.00	0.926	-1.934	0.144569	0.98	0.885	-1.818	0.16235	0.98	0.916	-1.942	0.143417
Secondary equilibria	1.00	7.262	-32.82	5.58E-15	0.90	12.390	-57.85	7.52E-26	0.83	9.683	-44.59	4.31E-20

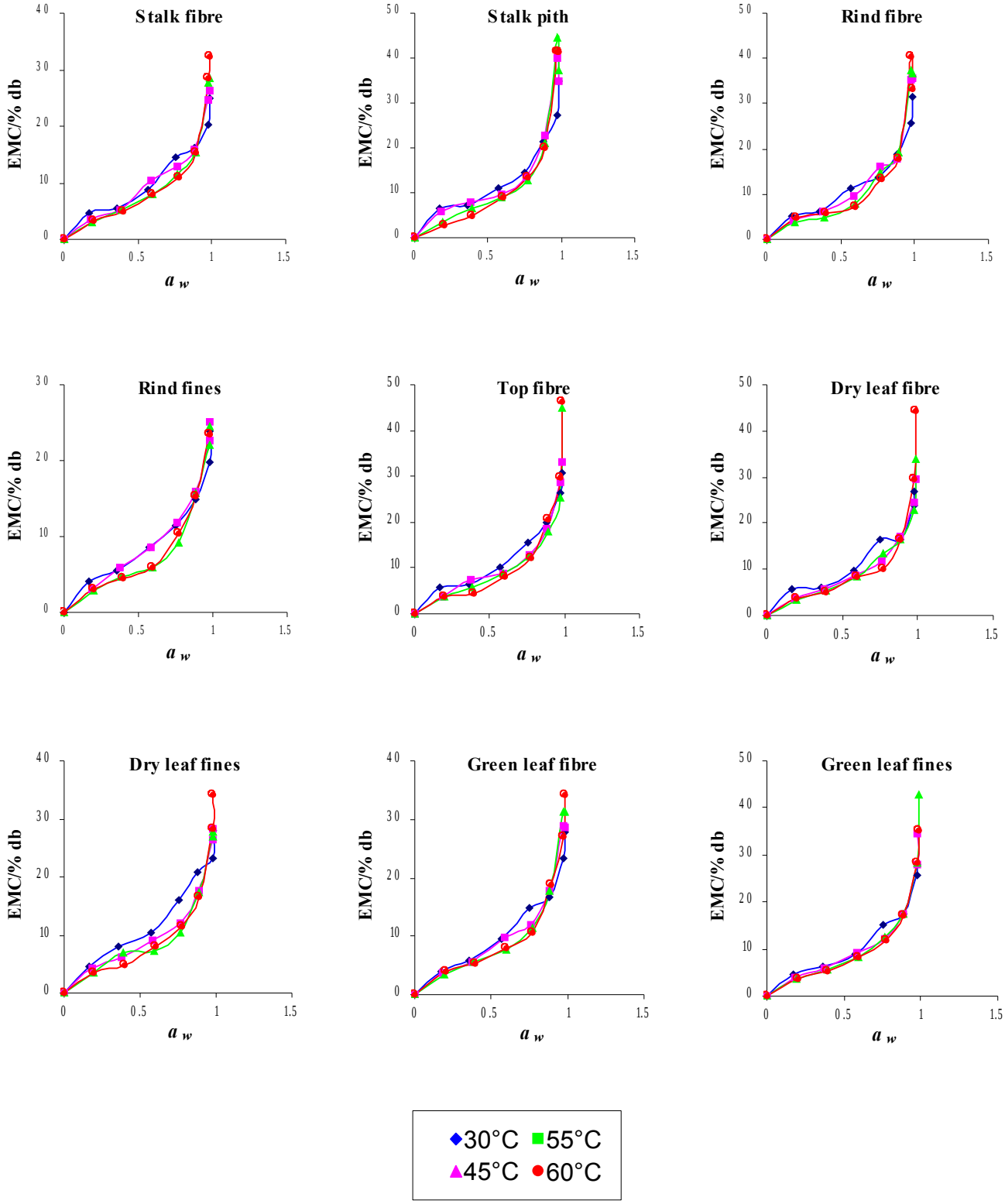


Figure 5.8. Experimental sorption isotherms of the nine sugar cane components of R 570 aged 52 weeks.

Note the similar behaviour of all the nine cane components.

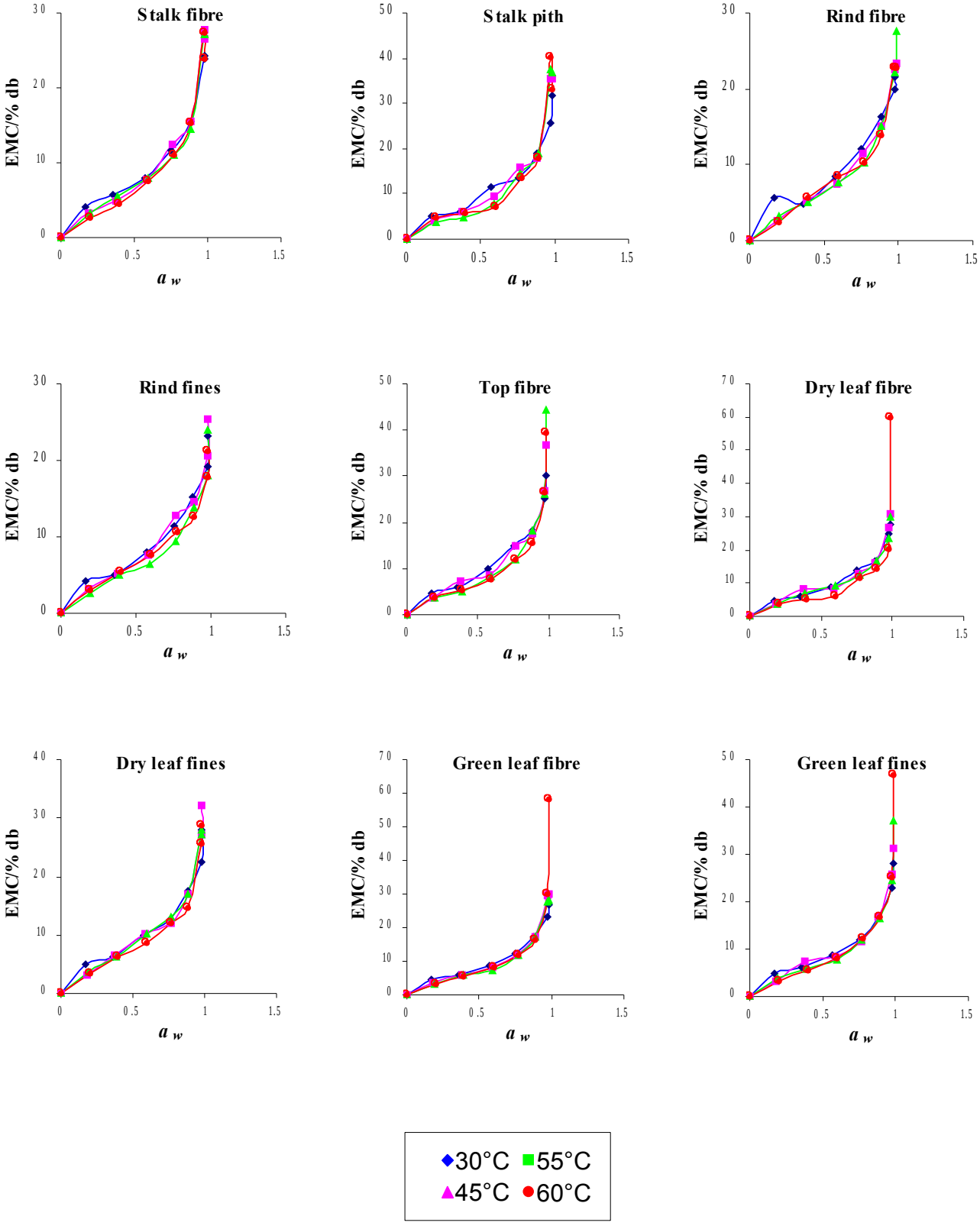


Figure 5.9. Experimental sorption isotherms of the nine sugar cane components of R 570 aged 36 weeks.

Note the similar behaviour of the nine cane components except those of dry leaf fibre and green leaf fibre.

5.6.4.4 *Fitting of sorption models to the experimental EMC data*

In order to determine which isotherm model best described the experimental EMC data obtained, fifteen of the isotherm models listed in Table 5.1 (all except the last two) and the modified Chung-Pfost and modified GAB models in Table 5.2 were fitted to the data. The parameters of the first seven models in Table 5.1 (namely, the Bradley, Caurie I, Caurie II, Halsey, Henderson, Kuhn and Smith models) were estimated by linear regression with Microsoft Excel software whereas those of the GAB model were estimated with the aid of the WATER ANALYSER PROGRAM (Webbtech@bigpond.com). Those of the remaining nine models (namely, BET, modified BET, Day-Nelson, Hailwood-Horrobin, Iglesias-Chirife, Mizrahi, Oswin, modified Chung-Pfost and modified GAB) were estimated by making use of the non-linear regression procedure of SigmaPlot (SPSS Inc.). The values of these parameters together with the calculated regression criteria, i.e. the coefficient of determination R^2, the mean deviation modulus P and the standard error of the estimate E_s, for each model and for each of the nine sugar cane components aged 52 and 36 weeks are shown in Tables 5.19 – 5.27. The BET, Day-Nelson and modified Chung-Pfost models yielded spurious results; they would undoubtedly give poor fit of the experimental results and were not included in the Tables.

From Tables 5.19 – 5.27, it can be seen that for the first ten isotherm models, namely the GAB, Hailwood-Horrobin, Henderson, Bradley, Caurie I, Smith, Oswin, Halsey, Caurie II and modified GAB models, the coefficient of determination R^2 approaches one for most temperatures studied (30, 45, 55 and 60 °C) and for all the nine cane component parts of both ages. However, for the last four isotherm models, namely the Kuhn, Iglesias-Chirife, Mizrahi and modified BET models, this was not the case, R^2 was low and mostly below 0.90, and the mean relative deviation modulus P was much greater than 10. They therefore gave a poor fit of the experimental results. As mentioned earlier in Section 5.3.4, for a good fit, R^2 must approach one, the P value must be between 5 and 10, and the standard error of the estimate, E_s, must be as small as possible.

The poor fit of the last four isotherm models was confirmed from inspection of the isotherm plots for the EMC of stalk fibre of variety R 570 aged 52 weeks shown in Fig 5.10. The complete set of isotherm plots for all the nine components is included on the CD (file: Isotherm plots.xls).

The isotherm plots show that in general, apart from the bad fit of the four models mentioned above, the Bradley and Smith models are only applicable within the activity

range of 0.4 to 0.6, the Halsey model up to 0.4, the Caurie I, Caurie II and Oswin models up to 0.6, and the modified GAB model up to 0.95; whereas the GAB, Hailwood-Horrobin and Henderson models fit for the whole range of water activity data.

In addition to the above criteria for deciding whether a model is a good fit to the experimental data, Chen and Morey (1989) and Soysal and Öztekin (1999) showed that the residuals (i.e. measured EMC – predicted EMC) could be plotted against the predicted EMC as abscissa. If the residuals were uniformly scattered about the x-axis (independent variable) and showed no systematic distribution or clear pattern in the positive or negative directions of the y-axis (dependent variable residuals), the model showed a good fit to the experimental values.

The residual plots for the ten candidate isotherm models were plotted for the experimental EMC data obtained for the nine cane components aged 52 and 36 weeks. A typical plot for stalk fibre aged 52 weeks is shown in Fig 5.11. The whole series of 18 plots is on the CD (file: Fig 5.11.1–5.11.18 Residuals.xls). In this study, the model residual plots have the EMC residuals (measured EMC – predicted EMC) plotted on the y-axis and the predicted EMC on the x-axis; other workers have used measured EMC (Arslan and Toğrul, 2005) on the x-axis, and equilibrium relative humidity (Igathinathane *et al.*, 2005) on the y-axis. The use of either the predicted or the measured EMC on the x-axis has been checked, and it was established that either practice did not affect the eventual randomness or pattern of the residual plot.

All the residual plots are examined for randomness (R) or systematic pattern (S), and the results are compiled in Table 5.28. In general, the Halsey and the two Caurie models give the poorest fit to the experimental data; while the residuals of the Bradley, Smith and Oswin models exhibit two distinct regions: at low water activity up to 0.97, a pattern is evident, at high water activity from 0.97 to 0.98, the residual distribution does not have a pattern, but the differences between the predicted EMC and the measured EMC are large, particularly in the case of stalk pith, rind fibre aged 52 weeks, top fibre, dry fibre, green leaf fibre aged 36 weeks and green leaf fines. However, these large differences are no worse than those predicted by the GAB, Hailwood-Horrobin, modified GAB and Henderson models. The residual patterns of these last four models for all the cane components, except dry and green leaf fibre aged 36 weeks at 60 °C, showed a random distribution for the whole range of water activity studied.

Table 5.19. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **stalk fibre** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Stalk fibre							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.43	5.18	3.68	3.22	4.17	3.99	3.54	4.00
	b	15.350	6.519	8.583	12.42	27.85	10.40	12.66	5.146
	c	0.7817	0.8193	0.8892	0.9140	0.8462	0.8733	0.8886	0.8660
	R^2	0.9601	0.9846	0.9949	0.9920	0.9981	0.9899	0.9909	0.9798
	P	9.579	6.705	6.099	8.681	2.359	9.083	7.773	5.550
	E_s	1.874	1.367	0.9519	1.337	0.4523	3.371	1.208	1.663
Hailwood Horrobin	*b*	0.021	0.022	0.014	-0.001	0.006	0.019	0.002	0.041
	c	0.143	0.172	0.266	0.336	0.234	0.225	0.301	0.192
	d	-0.123	-0.158	-0.250	-0.308	-0.204	-0.212	-0.272	-0.199
	R^2	0.960	0.985	0.995	0.992	0.998	0.990	0.992	0.980
	P	10.37	7.759	5.923	7.857	2.986	5.137	8.152	5.460
	E_s	1.866	1.330	0.8654	1.257	0.6292	1.213	1.112	1.656
Henderson	*b*	0.01600	0.03501	0.05738	0.06064	0.02252	0.04735	0.04559	0.07212
	c	1.761	1.456	1.292	1.262	1.649	1.358	1.386	1.240
	R^2	0.9590	0.9893	0.9904	0.9779	0.9830	0.9859	0.9843	0.9939
	P	11.01	5.444	7.283	11.16	7.458	7.790	8.294	5.156
	E_s	1.610	0.7998	1.545	3.004	1.239	1.548	1.923	1.656
Bradley	*b*	4.975	3.285	2.164	1.890	3.539	2.448	2.362	2.244
	c	0.7909	0.8209	0.8444	0.8571	0.8085	0.8376	0.8355	0.8309
	R^2	0.9590	0.9943	0.9878	0.9732	0.9948	0.9852	0.9815	0.9737
	P	10.61	5.577	11.86	17.83	5.588	10.36	12.15	9.862
	E_s	1.695	0.731	1.252	2.053	0.6541	1.318	1.458	1.693
Caurie I	*b*	0.1401	0.1592	0.1812	0.1839	0.1511	0.1737	0.1720	0.1923
	m_o	0.9180	0.8833	0.8865	0.8752	0.9139	0.8843	0.8984	0.9204
	R^2	0.9378	0.9532	0.9876	0.9958	0.9892	0.9807	0.9915	0.9665
	P	11.98	13.90	7.526	4.801	5.425	8.631	5.840	13.05
	E_s	2.479	2.290	1.644	0.8024	1.339	2.165	1.273	2.997
Smith	*b*	-4.900	-5.816	-6.815	-7.494	-5.457	-6.517	-6.422	-6.187
	c	4.642	3.436	1.531	0.7796	3.430	2.119	1.918	1.674
	R^2	0.9432	0.9863	0.9972	0.9903	0.9965	0.9899	0.9926	0.9745
	P	13.14	11.06	2.799	9.100	3.434	4.745	4.769	6.834
	E_s	1.996	1.135	0.6048	1.231	0.5348	1.088	0.9196	1.667
Oswin	*b*	8.662	8.177	6.951	6.559	7.783	7.375	6.943	6.821
	c	0.260	0.301	0.364	0.398	0.300	0.343	0.354	0.344
	R^2	0.936	0.978	0.992	0.999	0.987	0.978	0.992	0.955
	P	16.35	16.08	10.18	5.247	8.611	13.67	6.616	19.10
	E_s	2.122	1.430	1.000	0.4352	1.029	1.611	0.9704	2.224
Halsey	*b*	2.816	2.412	2.121	2.057	2.584	2.196	2.260	2.090
	c	200.2	71.92	30.65	27.16	102.7	38.40	43.06	23.92
	R^2	0.9046	0.9119	0.9613	0.9785	0.9653	0.9534	0.9688	0.9314
	P	16.42	19.08	13.25	10.54	10.37	13.99	10.90	18.35
	E_s	2.997	2.944	2.462	1.758	2.026	2.914	1.963	3.626
Caurie II	*b*	1.067	0.7904	0.5304	0.5116	0.9534	0.6279	0.6674	0.3633
	c	2.043	2.423	2.703	2.751	2.154	2.594	2.509	2.824
	R^2	0.9733	0.9811	0.9695	0.9492	0.9718	0.9749	0.9556	0.9822
	P	9.357	8.451	12.60	15.83	10.31	10.64	14.20	8.790
	E_s	1.779	1.951	3.169	4.391	2.120	2.725	3.387	2.507
Modified GAB	*b*	5.705	4.784	3.440	3.007	4.080	3.864	3.282	3.798
	c	0.771	0.832	0.897	0.920	0.850	0.877	0.897	0.872
	d	322.894	503.164	1296.622	200000000	1364.002	694.361	1047.207	443.567
	R^2	0.960	0.985	0.995	0.992	0.998	0.990	0.992	0.980
	P	10.3181	7.6235	5.9209	7.7407	2.7014	5.071	8.349	5.437
	E_s	1.8652	1.3230	0.8616	1.2204	0.4367	1.215	1.203	1.650
Kuhn	*b*	-0.3184	-0.3773	-0.4441	-0.4953	-0.3627	-0.4264	-0.4204	-0.3849
	c	8.700	8.312	7.140	6.813	7.826	7.495	7.174	7.039
	R^2	0.7719	0.8408	0.8986	0.9396	0.8533	0.8587	0.9027	0.8194
	P	33.75	41.20	37.63	32.25	31.00	40.19	32.20	47.78
	E_s	4.000	3.869	3.611	3.080	3.475	4.077	3.341	4.434
Iglesias - Chirife	*b*	0.319	0.378	0.444	0.496	0.363	0.427	0.421	0.385
	c	8.842	8.481	7.339	7.033	7.987	7.685	7.361	7.212
	R^2	0.770	0.840	0.898	0.939	0.852	0.858	0.902	0.818
	P	33.89	41.39	37.85	32.49	31.15	40.40	32.41	48.00
	E_s	4.012	3.885	3.627	3.099	3.489	4.092	3.357	4.447
Mizrahi	*b*	-8.842	-8.481	-7.339	-7.033	-7.987	-7.685	-7.361	-7.212
	c	-8.523	-8.103	-6.894	-6.538	-7.624	-7.258	-6.941	-6.827
	R^2	0.770	0.840	0.898	0.939	0.852	0.858	0.902	0.818
	P	33.89	41.39	37.85	32.50	31.15	40.42	32.41	48.00
	E_s	4.012	3.885	3.627	3.099	3.489	4.093	3.357	4.447
Modified BET	m_o	2.164	2.336	2.453	2.608	2.234	2.433	2.366	2.220
	b	30000000	70000000	50000000	70000000	60000000	40000000	50000000	40000000
	R^2	0.354	0.607	0.819	0.893	0.619	0.749	0.799	0.718
	P	43.43	36.75	30.12	26.05	39.39	33.36	33.33	33.08
	E_s	6.730	6.082	4.830	4.091	5.597	5.429	4.799	5.535

Note: m_o, *b*, *c* and *d* are constants.

Table 5.20. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **stalk pith** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Stalk pith							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.39	4.74	4.20	4.05	5.36	4.62	3.91	3.56
	b	364.8	200.0	10.22	4.933	17.32	10.65	17.66	200.0
	c	0.8465	0.8924	0.9166	0.9229	0.8330	0.8894	0.9138	0.9210
	R^2	0.9659	0.9784	0.9690	0.9964	0.9687	0.9844	0.9925	0.9613
	P	5.862	6.668	7.611	6.116	8.924	7.829	7.302	9.887
	E_s	2.427	2.461	3.435	1.243	2.158	2.029	1.513	3.387
Hailwood Horrobin	*b*	-0.002	-0.003	0.016	0.028	0.006	0.008	0.022	-0.007
	c	0.191	0.221	0.214	0.210	0.185	0.215	0.211	0.301
	d	-0.162	-0.195	-0.211	-0.219	-0.160	-0.199	-0.211	-0.272
	R^2	0.966	0.979	0.969	0.997	0.969	0.985	0.993	0.962
	P	5.940	6.428	8.880	6.597	7.947	7.272	7.878	8.197
	E_s	2.654	2.477	3.613	1.160	2.145	1.972	1.521	3.378
Henderson	*b*	0.009239	0.01985	0.05688	0.08503	0.01592	0.03755	0.06046	0.04232
	c	1.796	1.510	1.178	1.053	1.661	1.331	1.188	1.322
	R^2	0.9490	0.9366	0.9719	0.9877	0.9663	0.9796	0.9590	0.9133
	P	11.87	14.42	11.68	9.883	9.603	10.36	14.71	19.67
	E_s	2.921	4.221	4.590	2.737	2.045	2.543	2.903	5.143
Bradley	*b*	3.949	2.491	1.719	1.522	3.726	2.249	1.770	1.835
	c	0.8465	0.8775	0.8933	0.8974	0.8359	0.8736	0.8836	0.8793
	R^2	0.9649	0.9520	0.9412	0.9733	0.9763	0.9780	0.9753	0.9261
	P	10.63	18.35	27.04	26.26	9.800	12.88	20.83	26.48
	E_s	2.202	3.280	4.227	2.918	1.671	2.114	2.452	4.187
Caurie I	*b*	0.1235	0.1384	0.1742	0.1957	0.1363	0.1584	0.1800	0.1662
	m_o	0.8043	0.7638	0.7706	0.7847	0.8336	0.7868	0.8008	0.8116
	R^2	0.9771	0.9806	0.9856	0.9872	0.9664	0.9832	0.9785	0.9699
	P	7.394	8.728	8.550	9.859	9.933	6.954	10.58	12.52
	E_s	2.191	3.154	4.204	2.961	2.082	2.260	2.635	3.672
Smith	*b*	-6.973	-8.893	-10.27	-10.67	-6.463	-8.570	-9.340	-9.005
	c	5.005	2.916	0.1763	-0.8895	4.369	2.109	0.4190	0.6628
	R^2	0.9706	0.9675	0.9579	0.9905	0.9756	0.9880	0.9900	0.9472
	P	6.136	11.40	15.24	12.66	9.088	5.577	11.32	17.83
	E_s	2.012	2.699	3.575	1.736	1.698	1.566	1.555	3.538
Oswin	*b*	10.443	9.946	8.470	7.698	9.476	8.860	7.945	7.786
	c	0.293	0.347	0.407	0.436	0.296	0.363	0.400	0.398
	R^2	0.967	0.957	0.950	0.990	0.972	0.985	0.982	0.944
	P	7.479	9.835	13.88	13.74	11.88	11.51	15.31	12.51
	E_s	2.139	3.112	3.896	1.755	1.809	1.758	2.085	3.648
Halsey	*b*	2.766	2.353	1.910	1.740	2.616	2.139	1.916	2.089
	c	364.2	122.6	29.24	16.58	182.4	53.99	25.76	41.16
	R^2	0.9651	0.9763	0.9666	0.9628	0.9406	0.9591	0.9646	0.9730
	P	9.558	8.529	14.45	16.70	14.73	12.26	13.06	11.73
	E_s	2.684	3.696	5.033	4.307	2.727	3.214	3.762	4.031
Caurie II	*b*	1.367	1.151	0.5972	0.2818	1.149	0.8200	0.5210	0.7578
	c	1.984	2.315	2.953	3.310	2.146	2.639	2.956	2.628
	R^2	0.9443	0.9130	0.9439	0.9672	0.9627	0.9629	0.9496	0.8887
	P	11.60	18.77	18.71	15.14	10.77	12.91	18.52	23.78
	E_s	3.708	5.470	6.386	5.512	2.949	4.277	4.750	6.591
Modified GAB	*b*	5.365	4.708	4.092	3.814	5.119	4.353	3.986	3.545
	c	0.847	0.893	0.919	0.928	0.841	0.896	0.912	0.921
	d	300000000	500000000	895.523	601.296	1087.733	1378.578	690.626	400000000
	R^2	0.966	0.978	0.969	0.997	0.969	0.985	0.993	0.961
	P	5.735	6.338	8.312	6.541	7.651	7.212	7.813	9.561
	E_s	2.425	2.456	3.427	1.157	2.138	1.972	1.506	3.386
Kuhn	*b*	-0.4732	-0.5842	-0.6671	-0.6980	-0.4362	-0.5725	-0.6093	-0.5824
	c	10.48	10.22	8.653	7.824	9.480	8.999	8.095	8.118
	R^2	0.8663	0.8459	0.8587	0.9201	0.8608	0.8934	0.8944	0.8605
	P	24.69	30.36	42.40	48.21	31.48	38.00	44.04	35.52
	E_s	4.295	5.877	6.551	5.047	4.052	4.662	5.069	5.752
Iglesias - Chirife	*b*	0.474	0.585	0.668	0.699	0.437	0.573	0.610	0.583
	c	10.686	10.477	8.950	8.135	9.673	9.253	8.367	8.377
	R^2	0.865	0.845	0.858	0.919	0.860	0.892	0.894	0.860
	P	24.80	30.52	42.67	48.55	31.65	38.21	44.28	35.70
	E_s	4.312	5.894	6.569	5.070	4.070	4.683	5.089	5.766
Mizrahi	*b*	-10.69	-10.48	-8.950	-8.135	-9.673	-9.253	-8.367	-8.377
	c	-10.21	-9.893	-8.283	-7.436	-9.236	-8.680	-7.757	-7.794
	R^2	0.865	0.845	0.858	0.919	0.860	0.892	0.894	0.860
	P	25.01	30.54	42.67	48.55	31.65	38.21	44.28	35.70
	E_s	4.316	5.894	6.569	5.070	4.070	4.683	5.089	5.766
Modified BET	m_o	2.939	3.329	3.468	3.492	2.690	3.147	3.186	3.086
	b	200000000	80000000	10000000	40000000	200000000	80000000	20000000	50000000
	R^2	0.596	0.743	0.841	0.916	0.608	0.814	0.867	0.828
	P	41.09	36.62	28.05	25.16	39.44	30.25	23.88	30.38
	E_s	7.468	7.595	6.948	5.180	6.799	6.151	5.696	6.379

Note: m_o, *b*, *c* and *d* are constants.

Table 5.21. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors the of estimate E_s for isotherms of **rind fibre** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Rind fibre							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.36	5.51	3.70	2.69	4.71	4.56	3.51	3.99
	b	13.34	5.232	6.060	10.94	78.44	5.527	12.06	7.411
	c	0.7724	0.7923	0.8787	0.9357	0.7948	0.8263	0.8796	0.8453
	R^2	0.9949	0.9893	0.9950	0.9496	0.9842	0.9981	0.9797	0.9863
	P	2.950	4.778	4.457	14.89	8.356	3.020	16.62	9.635
	E_s	0.6175	1.040	0.8479	3.542	1.061	0.4506	5.371	1.168
Hailwood Horrobin	*b*	0.018	0.032	0.038	-0.015	0.006	0.038	0.010	0.024
	c	0.159	0.144	0.215	0.435	0.200	0.166	0.283	0.227
	d	-0.134	-0.137	-0.220	-0.397	-0.162	-0.165	-0.259	-0.211
	R^2	0.995	0.990	0.995	0.950	0.984	0.998	0.978	0.987
	P	3.004	6.671	4.998	13.42	8.683	3.901	6.225	11.26
	E_s	0.6320	1.015	0.8455	3.460	1.061	0.4467	1.665	1.152
Henderson	*b*	0.01581	0.04070	0.07651	0.07569	0.01094	0.05442	0.05040	0.05858
	c	1.792	1.429	1.221	1.193	1.950	1.356	1.380	1.343
	R^2	0.9931	0.9930	0.9926	0.9619	0.9072	0.9988	0.9853	0.9878
	P	3.869	3.982	5.738	13.17	12.85	2.210	7.366	7.286
	E_s	0.5073	0.7156	0.8846	4.445	1.381	0.2803	1.828	0.7985
Bradley	*b*	5.356	3.540	2.114	1.623	5.837	2.916	2.421	2.878
	c	0.7779	0.8065	0.8345	0.8656	0.7698	0.8083	0.8243	0.8038
	R^2	0.9907	0.9953	0.9925	0.9177	0.9686	0.9986	0.9766	0.9936
	P	5.061	5.978	11.09	28.21	11.98	2.092	10.64	5.909
	E_s	0.7412	0.6118	0.9141	3.958	1.324	0.3406	1.529	0.6980
Caurie I	*b*	0.1402	0.1649	0.1976	0.1977	0.1348	0.1800	0.1776	0.1812
	m_o	0.9398	0.9084	0.9216	0.8854	0.9616	0.9335	0.9269	0.9496
	R^2	0.9618	0.9344	0.9742	0.9883	0.9180	0.9549	0.9861	0.9389
	P	10.16	16.11	10.42	7.637	13.57	14.64	7.573	16.37
	E_s	1.907	2.639	2.254	2.437	1.948	2.470	1.611	2.148
Smith	*b*	-4.574	-5.316	-6.359	-8.035	-4.408	-5.391	-5.962	-5.232
	c	4.635	3.559	1.330	-0.2736	4.747	2.651	1.928	2.585
	R^2	0.9732	0.9801	0.9971	0.9404	0.9589	0.9904	0.9844	0.9890
	P	10.12	14.71	4.389	20.31	10.32	9.394	3.807	11.91
	E_s	1.261	1.256	0.567	3.368	1.515	0.8793	1.246	0.9172
Oswin	*b*	8.398	7.940	6.509	5.636	8.364	7.106	6.613	6.823
	c	0.253	0.289	0.361	0.450	0.248	0.311	0.349	0.315
	R^2	0.966	0.970	0.985	0.969	0.945	0.974	0.983	0.981
	P	12.58	19.29	16.76	7.227	12.97	17.83	9.356	16.31
	E_s	1.422	1.546	1.311	2.435	1.753	1.445	1.319	1.206
Halsey	*b*	2.889	2.411	2.028	1.934	3.035	2.259	2.260	2.304
	c	217.2	62.08	20.13	18.40	303.1	35.69	37.39	37.28
	R^2	0.9215	0.8830	0.9420	0.9743	0.9030	0.9101	0.9616	0.8928
	P	14.69	21.55	16.50	11.77	14.94	20.33	12.90	21.88
	E_s	2.467	3.196	3.015	2.581	2.430	3.086	2.191	2.654
Caurie II	*b*	1.067	0.7017	0.3174	0.3618	1.152	0.5257	0.5897	0.5016
	c	1.993	2.465	2.871	2.899	1.858	2.599	2.531	2.592
	R^2	0.9931	0.9827	0.9795	0.9274	0.9332	0.9881	0.9652	0.9638
	P	4.091	8.578	11.05	17.61	11.25	7.484	11.16	13.48
	E_s	0.9692	1.418	2.364	6.175	1.346	1.274	3.019	1.962
Modified GAB	*b*	5.354	5.100	3.548	2.532	4.793	4.349	3.318	3.736
	c	0.773	0.806	0.883	0.940	0.791	0.833	0.886	0.855
	d	403.533	339.774	467.083	500000000	1410.154	323.645	1825.450	786.362
	R^2	0.995	0.990	0.995	0.950	0.984	0.998	0.978	0.987
	P	2.9429	6.448	4.9819	11.640	8.637	3.9557	6.087	11.209
	E_s	0.6180	1.006	0.8315	3.461	1.057	0.4224	1.654	1.131
Kuhn	*b*	-0.2939	-0.3392	-0.4093	-0.5481	-0.2816	-0.3450	-0.3911	-0.3296
	c	8.473	8.078	6.643	5.922	8.471	7.217	6.794	7.056
	R^2	0.7786	0.8084	0.8771	0.9503	0.7583	0.8218	0.8992	0.8529
	P	32.38	45.45	45.81	33.05	30.22	45.05	31.94	44.05
	E_s	3.621	3.892	3.710	3.074	3.673	3.787	3.170	3.359
Iglesias - Chirife	*b*	0.294	0.339	0.410	0.549	0.282	0.345	0.391	0.330
	c	8.604	8.230	6.826	6.165	8.596	7.372	6.968	7.204
	R^2	0.777	0.807	0.876	0.950	0.757	0.820	0.898	0.852
	P	32.51	45.64	46.05	33.33	30.33	45.26	32.11	44.25
	E_s	3.633	3.906	3.726	3.091	3.684	3.800	3.184	3.371
Mizrahi	*b*	-8.604	-8.230	-6.826	-6.165	-8.596	-7.372	-6.968	-7.204
	c	-8.310	-7.891	-6.416	-5.617	-8.315	-7.027	-6.577	-6.874
	R^2	0.777	0.807	0.876	0.950	0.757	0.820	0.898	0.852
	P	32.51	45.64	46.05	33.34	30.33	45.26	32.11	44.25
	E_s	3.633	3.906	3.726	3.091	3.684	3.800	3.184	3.371
Modified BET	m_o	2.049	2.168	2.269	2.708	2.003	2.096	2.213	2.007
	b	30000000	100000000	20000000	30000000	70000000	30000000	50000000	30000000
	R^2	0.300	0.530	0.800	0.931	0.239	0.634	0.784	0.657
	P	43.98	37.38	28.07	22.20	45.12	34.72	33.18	35.27
	E_s	6.438	6.094	4.738	3.622	6.516	5.424	4.640	5.132

Note: m_o, *b*, *c* and *d* are constants.

Table 5.22. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **rind fines** aged 52 and 36 weeks at 30, 45, 55 and 60 °C.

Model	Parameter	Rind fines							
		52 weeks				36 weeks			
		30 °C	45 °C	55 °C	60 °C	30 °C	45 °C	55 °C	60 °C
GAB	m_o	4.55	4.88	3.20	3.35	4.39	4.52	3.47	3.77
	b	17.52	6.444	20.06	20.04	21.760	7.798	7.663	16.47
	c	0.8116	0.8205	0.8834	0.8773	0.8140	0.8269	0.8577	0.8259
	R^2	0.9782	0.9931	0.9929	0.9975	0.9795	0.9684	0.9600	0.9743
	P	5.236	4.894	6.060	4.619	5.167	6.635	6.667	8.309
	E_s	1.333	0.8785	0.8988	0.5528	1.266	1.788	1.905	1.303
Hailwood-Horrobin	*b*	0.010	0.023	0.025	0.035	0.013	0.025	0.023	0.019
	c	0.211	0.185	0.260	0.221	0.208	0.192	0.268	0.235
	d	-0.179	-0.170	-0.249	-0.219	-0.177	-0.177	-0.250	-0.207
	R^2	0.978	0.994	0.993	0.998	0.980	0.969	0.960	0.974
	P	5.043	5.380	5.466	3.916	5.553	6.304	8.067	8.378
	E_s	1.325	0.8125	0.9497	0.4967	5.317	1.817	1.891	1.305
Henderson	*b*	0.01900	0.03734	0.06157	0.06117	0.02018	0.03892	0.06090	0.03846
	c	1.742	1.463	1.341	1.337	1.736	1.471	1.378	1.559
	R^2	0.9856	0.9988	0.9746	0.9755	0.9750	0.9897	0.9857	0.9948
	P	6.009	1.979	10.52	9.651	8.180	6.629	7.792	3.996
	E_s	1.193	0.4743	1.496	1.194	1.238	1.376	1.695	0.9154
Bradley	*b*	4.367	3.268	2.302	2.364	4.312	3.202	2.631	3.760
	c	0.7859	0.8116	0.8169	0.8168	0.7823	0.8063	0.7951	0.7675
	R^2	0.9853	0.9982	0.9827	0.9884	0.9812	0.9786	0.9642	0.9869
	P	5.033	1.515	14.52	11.52	7.759	7.204	10.50	4.080
	E_s	0.9748	0.3892	1.249	1.019	1.086	1.312	1.602	0.8311
Caurie I	*b*	0.1468	0.1632	0.1895	0.1875	0.1493	0.1663	0.1885	0.1668
	m_o	0.9462	0.9089	0.9586	0.9533	0.9569	0.9281	0.9926	1.007
	R^2	0.9758	0.9631	0.9806	0.9766	0.9693	0.9614	0.9658	0.9598
	P	8.837	12.52	8.270	9.719	9.254	10.97	11.67	11.37
	E_s	1.551	2.073	1.861	2.080	1.720	2.177	2.022	1.568
Smith	*b*	-4.795	-5.501	-5.694	-5.671	-4.705	-5.338	-5.011	-4.313
	c	3.931	3.243	1.598	1.774	3.811	3.045	2.017	3.157
	R^2	0.9794	0.9913	0.9898	0.9920	0.9744	0.9728	0.9661	0.9794
	P	8.628	8.492	6.574	5.363	8.831	9.462	9.107	9.394
	E_s	1.155	0.8523	0.9583	0.8452	1.264	1.477	1.559	1.042
Oswin	*b*	7.764	7.711	6.214	6.450	7.607	7.341	6.013	6.611
	c	0.277	0.301	0.349	0.339	0.277	0.306	0.332	0.286
	R^2	0.975	0.981	0.976	0.972	0.966	0.966	0.960	0.976
	P	10.36	12.96	12.26	15.53	11.75	15.27	13.93	11.98
	E_s	1.271	1.253	1.487	1.583	1.455	1.651	1.685	1.131
Halsey	*b*	2.764	2.422	2.187	2.204	2.744	2.421	2.291	2.643
	c	145.1	64.57	26.36	28.27	128.7	58.28	29.65	71.69
	R^2	0.9447	0.9211	0.9596	0.9550	0.9417	0.9237	0.9323	0.9200
	P	13.51	17.80	12.15	13.23	13.87	16.16	17.13	15.92
	E_s	2.083	2.708	2.488	2.739	2.239	2.718	2.451	1.988
Caurie II	*b*	0.9958	0.7484	0.4554	0.4580	0.9588	0.7107	0.4534	0.6972
	c	2.044	2.403	2.607	2.623	2.060	2.398	2.536	2.240
	R^2	0.9798	0.9838	0.9569	0.9663	0.9778	0.9814	0.9657	0.9771
	P	6.699	7.578	14.09	12.19	7.798	7.141	11.10	7.575
	E_s	1.858	1.668	2.477	2.106	1.705	2.141	2.473	1.733
Modified GAB	*b*	4.395	4.476	3.279	3.551	4.383	4.300	3.242	3.761
	c	0.818	0.834	0.881	0.870	0.814	0.835	0.867	0.826
	d	818.179	524.661	748.214	559.941	665.128	510.614	836.686	1022.867
	R^2	0.978	0.994	0.993	0.998	0.980	0.969	0.960	0.974
	P	4.996	5.3905	5.3594	3.8818	5.139	6.557	8.073	8.377
	E_s	1.323	0.8023	0.8924	0.4908	5.064	1.776	1.884	1.303
Kuhn	*b*	-0.3195	-0.3560	-0.3650	-0.3543	-0.3106	-0.3497	-0.3254	-0.2728
	c	7.783	7.843	6.380	6.668	7.635	7.441	6.159	6.826
	R^2	0.8426	0.8412	0.8635	0.8409	0.8229	0.8462	0.8648	0.8512
	P	28.96	37.23	35.94	40.28	30.03	35.80	36.40	31.82
	E_s	3.190	3.646	3.514	3.780	3.328	3.514	3.115	2.798
Iglesias - Chirife	*b*	0.320	0.356	0.365	0.355	0.311	0.350	0.326	0.273
	c	7.925	8.002	6.543	6.827	7.773	7.597	6.305	6.948
	R^2	0.841	0.840	0.863	0.840	0.822	0.845	0.864	0.850
	P	29.11	37.41	36.13	40.46	30.17	36.82	36.59	31.95
	E_s	3.203	3.661	3.526	3.792	3.341	3.528	3.127	2.809
Mizrahi	*b*	-7.925	-8.002	-6.543	-6.827	-7.773	-7.597	-6.305	-6.948
	c	-7.606	-7.646	-6.177	-6.473	-7.462	-7.247	-5.979	-6.675
	R^2	0.841	0.840	0.863	0.840	0.822	0.845	0.864	0.850
	P	29.06	37.41	36.13	40.45	30.17	36.82	36.59	31.95
	E_s	3.203	3.661	3.526	3.792	3.341	3.528	3.127	2.809
Modified BET	m_o	2.065	2.204	2.073	2.062	2.016	2.134	1.895	1.763
	b	60000000	40000000	30000000	100000000	20000000	30000000	60000000	70000000
	R^2	0.504	0.608	0.762	0.721	0.488	0.628	0.718	0.530
	P	41.85	37.25	31.93	33.84	41.97	37.20	34.80	40.87
	E_s	5.665	5.728	4.642	5.003	5.663	5.463	4.498	4.973

Note: m_o, *b*, *c* and *d* are constants.

Table 5.23. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **top fibre** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Top fibre							
		52 weeks				36 weeks			
		30º C	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.60	4.44	3.24	3.44	5.51	4.55	3.24	2.94
	b	29.86	11.49	14.64	16.26	13.10	9.561	14.64	22.14
	c	0.8238	0.8774	0.9298	0.9309	0.8217	0.8783	0.9298	0.9302
	R^2	0.9840	0.9900	0.8979	0.9262	0.9797	0.9397	0.8979	0.9361
	P	6.571	5.944	18.51	10.99	6.556	12.00	13.78	15.54
	E_s	1.508	1.427	6.411	5.219	1.676	3.596	5.706	4.090
Hailwood Horrobin	*b*	0.008	0.007	-0.011	0.008	0.014	0.005	-0.007	-0.017
	c	0.164	0.228	0.342	0.284	0.161	0.233	0.345	0.396
	d	-0.140	-0.207	-0.310	-0.272	-0.143	-0.211	-0.317	-0.356
	R^2	0.984	0.991	0.871	0.926	0.980	0.941	0.898	0.936
	P	6.658	7.678	16.61	10.74	6.208	13.32	13.25	14.13
	E_s	1.514	1.362	6.377	5.214	1.736	3.535	5.676	4.012
Henderson	*b*	0.01278	0.03459	0.04999	0.07052	0.01916	0.03405	0.05952	0.05529
	c	1.725	1.401	1.272	1.143	1.611	1.396	1.221	1.274
	R^2	0.9588	0.9824	0.9514	0.9494	0.9825	0.9739	0.9529	0.9550
	P	9.959	8.522	14.50	17.15	7.384	10.27	15.29	14.03
	E_s	1.642	1.935	6.544	5.719	1.423	3.347	6.083	4.913
Bradley	*b*	4.118	2.520	1.811	1.564	3.716	2.491	1.690	1.766
	c	0.8337	0.8553	0.8777	0.8899	0.8317	0.8592	0.8796	0.8688
	R^2	0.9843	0.9854	0.8546	0.9068	0.9859	0.9451	0.8773	0.9099
	P	9.774	12.23	26.45	28.12	7.306	15.22	28.56	26.16
	E_s	1.336	1.484	6.028	5.241	1.248	3.030	5.561	4.265
Caurie I	*b*	0.1309	0.1576	0.1721	0.1884	0.1410	0.1562	0.1815	0.1793
	m_o	0.8293	0.8263	0.8200	0.8084	0.8422	0.8176	0.8279	0.8541
	R^2	0.9630	0.9810	0.9714	0.9750	0.9687	0.9692	0.9783	0.9887
	P	9.682	8.656	11.10	12.32	10.46	10.89	9.873	6.142
	E_s	2.180	1.619	5.010	4.215	2.251	2.708	4.455	2.911
Smith	*b*	-6.359	-7.402	-8.891	-9.927	-6.269	-7.628	-9.049	-8.239
	c	4.879	2.561	0.520	-0.6243	4.265	2.560	-0.0208	0.3250
	R^2	0.9799	0.9941	0.8734	0.9262	0.9798	0.9538	0.8974	0.9317
	P	8.223	6.175	17.63	16.87	9.233	10.99	17.89	16.51
	E_s	1.511	0.9422	5.627	4.663	1.493	2.778	5.085	3.715
Oswin	*b*	10.004	8.355	6.957	6.981	9.314	8.384	6.638	6.347
	c	0.281	0.346	0.428	0.446	0.292	0.354	0.441	0.430
	R^2	0.970	0.992	0.900	0.941	0.972	0.962	0.921	0.958
	P	12.09	8.867	10.72	12.41	12.90	12.08	9.742	6.382
	E_s	1.849	1.092	4.998	4.177	1.748	2.532	4.452	2.895
Halsey	*b*	2.705	2.262	2.054	1.850	2.563	2.260	1.964	2.053
	c	250.1	63.19	35.22	20.54	145.8	65.81	26.01	29.73
	R^2	0.9411	0.9534	0.9550	0.9636	0.9366	0.9401	0.9648	0.9785
	P	13.44	14.31	15.76	15.39	15.44	14.57	14.61	9.986
	E_s	2.909	2.472	5.026	4.627	2.977	3.180	4.552	2.949
Caurie II	*b*	1.225	0.8475	0.6543	0.4202	1.067	0.8597	0.5358	0.5839
	c	2.079	2.492	2.733	3.056	2.217	2.504	2.851	2.717
	R^2	0.9673	0.9545	0.9239	0.9327	0.9826	0.9486	0.9282	0.9221
	P	9.744	13.99	16.58	18.88	7.016	13.12	17.26	18.02
	E_s	2.261	3.535	7.973	7.416	2.247	4.691	7.598	6.538
Modified GAB	*b*	5.639	4.161	3.202	3.347	5.415	4.146	3.068	2.870
	c	0.822	0.885	0.930	0.933	0.825	0.890	0.934	0.933
	d	786.808	1767.520	200000000	2359.258	468.075	2573.757	300000000	700000000
	R^2	0.984	0.991	0.871	0.926	0.980	0.941	0.898	0.935
	P	6.709	7.702	14.775	10.720	6.218	13.414	12.203	12.708
	E_s	1.508	1.340	6.357	5.214	1.673	3.527	5.671	4.035
Kuhn	*b*	-0.4199	-0.4952	-0.6169	-0.6673	-0.4168	-0.5218	-0.6240	-0.5621
	c	10.05	8.499	7.244	7.188	9.316	8.504	6.884	6.678
	R^2	0.8276	0.9017	0.8925	0.9091	0.8392	0.9045	0.9058	0.9418
	P	30.79	32.89	35.09	39.23	33.18	34.38	34.82	29.20
	E_s	4.427	3.854	5.185	5.177	4.215	3.997	4.872	3.429
Iglesias - Chirife	*b*	0.420	0.496	0.618	0.668	0.417	0.522	0.625	0.563
	c	10.233	8.719	7.517	7.484	9.501	8.735	7.160	6.927
	R^2	0.826	0.901	0.892	0.908	0.838	0.904	0.905	0.941
	P	30.89	33.09	35.35	39.49	33.32	34.58	35.09	29.41
	E_s	4.444	3.873	5.198	5.194	4.232	4.016	4.887	3.446
Mizrahi	*b*	-10.23	-8.719	-7.517	-7.484	-9.501	-8.735	-7.160	-6.927
	c	-9.813	-8.224	-6.900	-6.816	-9.084	-8.213	-6.536	-6.364
	R^2	0.826	0.901	0.892	0.908	0.838	0.904	0.905	0.941
	P	30.85	33.09	35.35	39.49	33.32	34.58	35.10	29.41
	E_s	4.445	3.873	5.198	5.194	4.232	4.016	4.887	3.446
Modified BET	m_o	2.695	2.799	3.106	3.298	2.601	2.894	3.096	2.841
	b	40000000	50000000	30000000	20000000	20000000	100000000	30000000	60000000
	R^2	0.516	0.786	0.857	0.895	0.578	0.797	0.882	0.909
	P	41.15	32.25	28.72	20.94	39.30	32.10	25.12	25.91
	E_s	7.415	5.688	5.973	5.569	6.830	5.833	5.457	4.292

Note: m_o, *b, c* and *d* are constants.

Table 5.24. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **dry leaf fibre** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Dry leaf fibre							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.41	4.17	4.24	2.68	4.68	4.47	4.47	0.88
	b	27.85	15.30	6.343	32.97	28.03	12.47	11.88	200.0
	c	0.8069	0.8665	0.8734	0.9484	0.8411	0.8635	0.8533	1.000
	R^2	0.9628	0.9835	0.9212	0.9423	0.9915	0.9709	0.9669	0.7976
	P	9.266	5.112	11.69	15.45	4.628	9.043	8.325	59.97
	E_s	1.988	1.539	3.749	4.507	1.023	2.174	2.129	11.75
Hailwood Horrobin	b	0.007	0.009	0.017	-0.028	0.006	0.000	0.004	-2.953
	c	0.167	0.233	0.230	0.457	0.207	0.248	0.236	10.811
	d	-0.138	-0.210	-0.216	-0.410	-0.179	-0.217	-0.208	-7.931
	R^2	0.963	0.984	0.922	0.942	0.992	0.972	0.968	0.871
	P	9.627	4.888	10.98	14.22	4.144	12.71	9.589	54.50
	E_s	1.985	1.522	3.752	4.429	1.019	2.064	2.114	8.669
Henderson	b	0.009977	0.03352	0.05206	0.06142	0.01839	0.02598	0.02807	0.06554
	c	1.865	1.466	1.306	1.216	1.666	1.525	1.524	1.221
	R^2	0.9443	0.9845	0.9811	0.9378	0.9733	0.9615	0.9857	0.8355
	P	11.91	7.280	9.018	16.99	8.763	11.09	6.854	27.80
	E_s	1.738	1.773	3.241	6.290	1.398	2.225	1.982	14.27
Bradley	b	5.221	2.756	2.306	1.536	3.644	3.036	3.088	1.286
	c	0.8073	0.8347	0.8468	0.8845	0.8226	0.8380	0.8288	0.8961
	R^2	0.9692	0.9833	0.9310	0.8933	0.9931	0.9745	0.9745	0.6517
	P	10.56	8.485	12.88	34.47	7.613	11.14	8.144	62.43
	E_s	1.603	1.377	3.123	5.367	0.8198	1.748	1.645	12.71
Caurie I	b	0.1278	0.1596	0.1743	0.1845	0.1429	0.1493	0.1513	0.1917
	m_o	0.8776	0.8746	0.8693	0.8321	0.8730	0.8525	0.8762	0.8572
	R^2	0.9439	0.9861	0.9636	0.9875	0.9809	0.9582	0.9773	0.9028
	P	9.523	7.359	12.66	7.986	6.933	10.38	8.795	21.70
	E_s	2.157	1.706	3.204	3.440	1.492	1.429	1.754	11.82
Smith	b	-5.388	-6.397	-6.919	-9.471	-5.934	-6.548	-6.125	-10.67
	c	5.275	2.732	1.963	-0.8228	3.889	3.322	3.306	-2.652
	R^2	0.9602	0.9887	0.9361	0.9208	0.9936	0.9826	0.9782	0.6796
	P	9.422	4.095	8.434	25.82	9.918	10.86	7.048	50.40
	E_s	1.820	1.134	3.005	4.624	2.561	1.442	1.521	12.19
Oswin	b	9.610	7.780	7.365	5.928	8.611	8.332	8.084	3.345
	c	0.260	0.331	0.358	0.478	0.297	0.325	0.316	0.656
	R^2	0.955	0.984	0.939	0.960	0.986	0.988	0.979	0.759
	P	11.63	8.960	15.92	10.71	9.335	11.16	8.530	42.23
	E_s	1.926	1.362	2.933	3.273	1.172	1.174	1.496	10.56
Halsey	b	2.935	2.360	2.169	1.937	2.607	2.467	2.514	1.910
	c	378.8	65.31	38.11	23.71	139.7	101.8	102.2	19.64
	R^2	0.9202	0.9613	0.9314	0.9839	0.9589	0.9286	0.9476	0.9090
	P	12.87	12.59	17.26	9.198	11.60	14.43	13.66	20.17
	E_s	2.698	2.390	3.635	3.193	2.229	1.995	2.273	11.23
Caurie II	b	1.265	0.8212	0.5930	0.5346	1.059	0.9763	0.9243	0.4858
	c	1.925	2.396	2.682	2.832	2.142	2.276	2.283	2.812
	R^2	0.9546	0.9687	0.9667	0.8968	0.9709	0.9226	0.9581	0.7937
	P	11.47	10.17	10.56	21.82	10.19	17.14	10.97	31.48
	E_s	2.204	2.942	4.296	8.234	2.284	3.632	3.185	15.69
Modified GAB	b	5.587	4.014	3.852	2.627	4.599	4.047	4.103	0.208
	c	0.798	0.872	0.885	0.950	0.844	0.877	0.866	1.015
	d	923.420	1364.860	950.206	500000000	1211.634	100000000	3505.082	200000000
	R^2	0.963	0.984	0.922	0.941	0.992	0.972	0.968	0.857
	P	9.458	4.803	11.126	13.780	4.241	12.747	9.428	66.94
	E_s	1.966	1.521	3.713	4.472	1.017	2.034	2.061	9.11
Kuhn	b	-0.3552	-0.4251	-0.4596	-0.6546	-0.3965	-0.4431	-0.4012	-0.7827
	c	9.660	7.908	7.519	6.343	8.640	8.500	8.314	4.686
	R^2	0.8088	0.8847	0.8770	0.9554	0.8595	0.9115	0.8909	0.7951
	P	29.38	30.80	39.89	27.45	30.61	29.19	28.42	30.39
	E_s	3.990	3.617	4.168	3.469	3.704	3.253	3.400	9.746
Iglesias - Chirife	b	0.355	0.425	0.460	0.655	0.397	0.443	0.402	0.784
	c	9.819	8.097	7.723	6.633	8.816	8.697	8.493	5.030
	R^2	0.807	0.884	0.876	0.955	0.858	0.911	0.890	0.795
	P	29.51	30.94	40.09	27.74	30.77	29.37	28.59	30.67
	E_s	4.004	3.634	4.183	3.486	3.720	3.271	3.415	9.748
Mizrahi	b	-9.819	-8.097	-7.723	-6.633	-8.816	-8.697	-8.493	-5.030
	c	-9.463	-7.672	-7.263	-5.977	-8.419	-8.253	-8.092	-4.246
	R^2	0.807	0.884	0.876	0.955	0.858	0.911	0.890	0.795
	P	29.51	30.94	40.09	27.73	30.77	29.37	28.56	30.67
	E_s	4.004	3.634	4.183	3.486	3.720	3.271	3.415	9.748
Modified BET	m_o	2.408	2.469	2.547	3.155	2.450	2.599	2.413	3.447
	b	20000000	60000000	80000000	50000000	40000000	50000000	100000000	1072586
	R^2	0.372	0.735	0.779	0.943	0.612	0.733	0.685	0.780
	P	43.35	35.17	31.61	20.53	39.09	35.35	37.96	25.63
	E_s	7.230	5.486	5.585	3.930	6.156	5.647	5.774	10.10

Note: m_o, b, c and d are constants.

Table 5.25. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **dry leaf fines** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Dry leaf fines							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	7.62	4.32	3.71	3.36	4.98	4.44	5.15	4.03
	b	8.604	22.56	29.23	13.66	27.94	9.307	6.598	7.402
	c	0.7344	0.8615	0.8836	0.9123	0.8227	0.8711	0.8344	0.8713
	R^2	0.9761	0.9984	0.9940	0.9853	0.9713	0.9779	0.9910	0.9804
	P	5.004	2.863	6.683	7.368	6.033	9.909	5.731	10.75
	E_s	1.630	0.4905	0.9424	1.792	1.777	2.002	1.164	1.755
Hailwood Horrobin	*b*	0.023	0.007	0.004	0.003	0.004	0.009	0.017	0.004
	c	0.094	0.226	0.268	0.309	0.202	0.228	0.186	0.274
	d	-0.081	-0.201	-0.241	-0.286	-0.170	-0.208	-0.170	-0.246
	R^2	0.976	0.998	0.994	0.986	0.971	0.978	0.992	0.982
	P	4.648	2.597	7.735	6.526	5.204	12.21	6.067	11.60
	E_s	1.642	0.4658	0.9575	1.743	1.771	1.924	1.064	1.570
Henderson	*b*	0.01211	0.02689	0.03865	0.05962	0.01301	0.03958	0.03398	0.04245
	c	1.752	1.532	1.430	1.254	1.789	1.370	1.439	1.396
	R^2	0.9874	0.9830	0.9638	0.9762	0.9748	0.9884	0.9974	0.9887
	P	5.872	7.808	12.20	11.12	7.925	7.016	3.301	6.448
	E_s	1.646	1.416	2.002	2.720	1.789	1.782	0.7004	1.640
Bradley	*b*	5.804	2.997	2.521	1.883	4.360	2.601	3.063	2.575
	c	0.8117	0.8333	0.8366	0.8614	0.8120	0.8483	0.8335	0.8337
	R^2	0.9584	0.9942	0.9803	0.9699	0.9777	0.9823	0.9978	0.9847
	P	9.734	6.667	14.26	17.90	7.057	9.699	3.346	8.837
	E_s	1.921	0.7999	1.517	2.252	1.394	1.554	0.4958	1.307
Caurie I	*b*	0.1273	0.1519	0.1648	0.1822	0.1344	0.1623	0.1555	0.1662
	m_o	0.8455	0.8673	0.8855	0.8619	0.8834	0.8408	0.8596	0.8879
	R^2	0.9315	0.9895	0.9761	0.9936	0.9786	0.9664	0.9671	0.9774
	P	13.73	6.214	9.174	5.811	7.571	11.75	11.12	9.094
	E_s	3.252	1.460	1.692	1.353	1.695	1.890	2.135	1.295
Smith	*b*	-5.477	-6.333	-6.467	-7.743	-5.561	-7.020	-6.289	-6.319
	c	6.012	3.171	2.304	0.7886	4.519	2.649	3.406	2.424
	R^2	0.9319	0.9987	0.9902	0.9861	0.9763	0.9875	0.9940	0.9916
	P	14.54	2.396	8.223	8.810	7.473	9.976	7.497	7.389
	E_s	2.457	0.3837	1.068	1.529	1.439	1.310	0.8115	0.9669
Oswin	*b*	10.593	8.210	7.436	6.787	8.912	8.118	8.485	7.341
	c	0.243	0.318	0.341	0.398	0.280	0.342	0.309	0.340
	R^2	0.926	0.990	0.982	0.993	0.973	0.990	0.985	0.995
	P	17.24	7.441	11.35	7.091	9.081	11.14	13.10	7.556
	E_s	2.560	1.065	1.438	1.107	1.526	1.182	1.272	0.7685
Halsey	*b*	2.883	2.456	2.321	2.045	2.809	2.248	2.412	2.327
	c	387.4	90.93	55.08	27.92	245.6	55.59	81.96	54.95
	R^2	0.8803	0.9672	0.9564	0.9768	0.9540	0.9288	0.9289	0.9466
	P	18.26	11.23	12.36	11.51	12.03	17.60	16.24	14.43
	E_s	3.839	2.242	2.343	2.158	2.234	2.603	2.836	1.967
Caurie II	*b*	1.243	0.9291	0.7654	0.5239	1.183	0.7696	0.8364	0.7195
	c	2.038	2.295	2.428	2.774	1.988	2.547	2.432	2.483
	R^2	0.9873	0.9679	0.9324	0.9518	0.9671	0.9590	0.9809	0.9576
	P	5.593	10.63	16.91	15.48	8.347	11.59	8.427	12.96
	E_s	1.485	2.624	3.266	4.492	2.532	3.404	2.242	3.239
Modified GAB	*b*	7.843	4.207	3.626	3.179	4.804	4.098	4.660	3.561
	c	0.728	0.865	0.886	0.918	0.829	0.881	0.849	0.887
	d	227.477	1879.811	3847.253	6821.804	1974.738	1340.801	833.710	5015.572
	R^2	0.976	0.998	0.994	0.986	0.971	0.978	0.992	0.982
	P	4.814	2.6126	7.5006	6.543	5.478	12.075	5.920	11.643
	E_s	1.627	0.4596	0.9299	1.746	1.763	1.922	1.049	1.569
Kuhn	*b*	-0.3465	-0.4154	-0.4176	-0.5113	-0.3740	-0.4723	-0.4003	-0.4116
	c	10.689	8.381	7.687	7.029	8.934	8.241	8.733	7.609
	R^2	0.7229	0.8704	0.8765	0.9340	0.8557	0.9057	0.8548	0.9137
	P	35.41	30.57	31.89	32.08	26.91	36.96	37.90	32.09
	E_s	4.956	3.778	3.796	3.334	3.548	3.593	3.994	3.103
Iglesias - Chirife	*b*	0.347	0.416	0.418	0.512	0.374	0.473	0.401	0.412
	c	10.844	8.566	7.873	7.257	9.100	8.451	8.913	7.793
	R^2	0.721	0.869	0.876	0.933	0.854	0.905	0.854	0.913
	P	35.54	30.73	32.06	32.30	27.04	37.20	34.88	32.28
	E_s	4.970	3.793	3.810	3.353	3.563	3.612	4.215	3.119
Mizrahi	*b*	-10.84	-8.566	-7.873	-7.257	-9.100	-8.451	-8.913	-7.793
	c	-10.50	-8.150	-7.455	-6.745	-8.726	-7.978	-8.512	-7.381
	R^2	0.721	0.869	0.876	0.933	0.854	0.905	0.854	0.913
	P	35.20	30.73	32.06	32.30	27.04	37.20	38.08	32.28
	E_s	4.974	3.793	3.810	3.353	3.563	3.612	4.010	3.119
Modified BET	m_o	2.494	2.487	2.412	2.691	2.396	2.682	2.459	2.374
	b	50000000	40000000	40000000	60000000	60000000	50000000	50000000	50000000
	R^2	0.194	0.686	0.750	0.887	0.526	0.778	0.639	0.773
	P	45.15	36.85	34.53	26.30	42.02	31.82	37.10	33.74
	E_s	8.456	5.884	5.405	4.355	6.429	5.506	6.302	5.037

Note: m_o, *b*, *c* and *d* are constants.

Table 5.26. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **green leaf fibre** aged 52 and 36 weeks at 30, 45, 55 and 60 °C.

Model	Parameter	Green leaf fibre							
		52 weeks				36 weeks			
		30 °C	45 °C	55 °C	60 °C	30 °C	45 °C	55 °C	60 °C
GAB	m_o	5.71	4.54	3.68	3.52	4.53	4.09	4.09	1.99
	b	8.388	10.13	13.82	62.30	37.02	12.67	10.10	15.78
	c	0.7995	0.8627	0.9018	0.9048	0.8389	0.8825	0.8740	0.9770
	R^2	0.9712	0.9946	0.9982	0.9775	0.9882	0.9977	0.9986	0.8813
	P	7.108	4.986	3.008	5.315	2.796	3.918	3.402	33.12
	E_s	1.840	0.9516	0.6234	2.131	1.146	0.6741	0.4737	8.604
Hailwood Horrobin	*b*	0.021	0.014	0.014	0.004	0.006	0.011	0.020	-0.077
	c	0.151	0.207	0.253	0.277	0.213	0.237	0.215	0.746
	d	-0.135	-0.190	-0.240	-0.254	-0.183	-0.218	-0.205	-0.660
	R^2	0.971	0.995	0.998	0.977	0.988	0.998	0.999	0.876
	P	6.499	4.862	3.593	5.491	2.755	5.162	5.266	31.93
	E_s	1.899	0.9462	0.6223	2.131	1.154	0.9407	0.7823	8.485
Henderson	*b*	0.02271	0.03599	0.05576	0.04672	0.01763	0.04087	0.04901	0.07174
	c	1.588	1.414	1.268	1.335	1.702	1.372	1.333	1.130
	R^2	0.9891	0.9884	0.9800	0.9574	0.9748	0.9867	0.9878	0.9238
	P	6.489	6.520	9.573	13.74	8.357	7.624	6.951	19.83
	E_s	1.346	1.370	2.165	3.041	1.519	1.716	1.199	10.73
Bradley	*b*	3.903	2.689	1.994	2.059	3.796	2.389	2.390	1.306
	c	0.8180	0.8436	0.8606	0.8580	0.8144	0.8504	0.8425	0.9049
	R^2	0.9813	0.9908	0.9815	0.9631	0.9879	0.9885	0.9924	0.7986
	P	6.030	8.049	17.14	17.44	7.459	11.61	10.19	51.71
	E_s	1.322	1.083	1.743	2.438	1.038	1.271	0.9734	9.580
Caurie I	*b*	0.1466	0.1602	0.1787	0.1712	0.1429	0.1659	0.1725	0.1909
	m_o	0.8707	0.8507	0.8522	0.8568	0.8912	0.8500	0.8736	0.7991
	R^2	0.9572	0.9798	0.9910	0.9859	0.9837	0.9897	0.9790	0.9741
	P	12.14	9.141	5.970	6.446	6.481	6.403	9.917	9.662
	E_s	2.419	2.115	1.927	2.059	1.553	1.736	2.113	7.478
Smith	*b*	-5.735	-6.784	-7.697	-7.537	-5.645	-7.141	-6.713	-11.67
	c	4.184	2.771	1.154	1.363	3.899	2.140	2.121	-2.703
	R^2	0.970	0.994	0.995	0.978	0.9883	0.9973	0.9971	0.8285
	P	11.71	5.841	7.227	10.02	5.170	2.881	5.268	39.14
	E_s	1.672	0.8948	0.9478	1.892	1.019	0.6128	0.6027	8.840
Oswin	*b*	8.827	8.233	7.311	7.278	8.391	7.817	7.562	4.935
	c	0.285	0.329	0.378	0.376	0.293	0.351	0.343	0.575
	R^2	0.965	0.982	0.987	0.976	0.980	0.990	0.984	0.895
	P	14.56	12.00	12.00	9.661	9.059	9.274	12.88	22.64
	E_s	1.812	1.496	1.496	1.952	1.318	1.211	1.410	6.904
Halsey	*b*	2.563	2.293	2.060	2.156	2.661	2.207	2.197	1.798
	c	121.8	60.82	30.47	39.59	148.3	47.26	40.63	18.47
	R^2	0.9160	0.9505	0.9712	0.9753	0.9621	0.9650	0.9506	0.9702
	P	17.23	14.60	11.54	9.524	11.08	12.25	14.66	13.26
	E_s	3.045	2.923	2.819	2.660	2.197	2.602	2.910	6.927
Caurie II	*b*	0.9786	0.8001	0.5659	0.6730	1.063	0.7374	0.6307	0.4440
	c	2.246	2.486	2.750	2.610	2.093	2.554	2.623	3.042
	R^2	0.9858	0.9734	0.9564	0.9355	0.9701	0.9657	0.9688	0.8786
	P	5.227	9.771	16.01	16.25	9.358	13.23	12.71	24.27
	E_s	2.064	2.701	3.809	4.414	2.268	3.209	2.622	12.85
Modified GAB	*b*	5.455	4.320	3.596	3.509	4.491	3.914	3.984	1.932
	c	0.808	0.869	0.904	0.905	0.840	0.888	0.877	0.978
	d	336.758	836.416	1196.953	4404.242	1369.123	1217.131	770.973	-2000000000
	R^2	0.971	0.995	0.998	0.977	0.988	0.998	0.999	0.872
	P	6.857	4.9012	3.6396	5.370	2.604	4.5640	4.2421	30.740
	E_s	1.830	0.9038	0.6028	2.130	1.145	0.6073	0.4505	8.533
Kuhn	*b*	-0.3804	-0.4428	-0.5005	-0.4916	-0.3771	-0.4724	-0.4295	-0.8330
	c	8.819	8.385	7.506	7.534	8.421	7.954	7.770	5.708
	R^2	0.8268	0.8578	0.8927	0.9037	0.8544	0.8841	0.8667	0.9165
	P	35.45	37.41	37.50	30.00	28.29	35.73	39.09	29.52
	E_s	4.023	4.250	4.202	3.937	3.596	4.032	4.080	6.170
Iglesias - Chirife	*b*	0.381	0.443	0.501	0.492	0.377	0.473	0.430	0.834
	c	8.988	8.583	7.729	7.754	8.588	8.164	7.962	6.075
	R^2	0.825	0.857	0.892	0.903	0.853	0.883	0.866	0.916
	P	35.62	37.61	37.72	30.18	28.40	35.93	39.28	29.89
	E_s	4.038	4.267	4.219	3.953	3.611	4.049	4.095	6.179
Mizrahi	*b*	-8.988	-8.583	-7.729	-7.754	-8.588	-8.164	-7.962	-6.075
	c	-8.607	-8.140	-7.228	-7.262	-8.211	-7.691	-7.533	-5.241
	R^2	0.825	0.857	0.892	0.903	0.853	0.883	0.866	0.916
	P	35.62	37.61	37.72	30.18	28.40	35.93	39.28	29.89
	E_s	4.038	4.267	4.219	3.953	3.611	4.049	4.096	6.179
Modified BET	m_o	2.409	2.593	2.709	2.672	2.353	2.656	2.468	3.755
	b	80000000	30000000	40000000	200000000	50000000	70000000	40000000	5000857
	R^2	0.539	0.714	0.837	0.835	0.589	0.785	0.751	0.908
	P	39.47	35.10	28.70	30.19	40.18	32.41	32.70	21.91
	E_s	6.559	6.026	5.178	5.155	6.045	5.489	5.571	6.459

Note: m_o, *b, c* and *d* are constants.

Table 5.27. Parameters of the sorption isotherm models, coefficient of determination R^2, mean relative deviation modulus P, and standard errors of the estimate E_s for isotherms of **green leaf fines** aged 52 and 36 weeks at 30, 45, 55 and 60 ºC.

Model	Parameter	Green leaf fines							
		52 weeks				36 weeks			
		30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
GAB	m_o	5.15	3.94	3.37	3.46	4.37	4.26	3.33	2.94
	b	18.45	16.40	13.06	16.49	71.79	8.156	40.08	10.78
	c	0.8277	0.8946	0.9263	0.9109	0.8474	0.8731	0.9122	0.9401
	R^2	0.9863	0.9803	0.9301	0.9808	0.9788	0.9747	0.9263	0.8628
	P	5.734	8.054	14.12	7.671	4.187	9.364	11.41	21.66
	E_s	1.324	2.029	4.628	2.070	1.561	2.103	3.979	6.968
Hailwood Horrobin	*b*	0.013	0.001	-0.007	0.001	0.000	0.005	-0.010	-0.021
	c	0.173	0.269	0.335	0.304	0.234	0.254	0.336	0.420
	d	-0.152	-0.243	-0.306	-0.279	-0.199	-0.229	-0.300	-0.380
	R^2	0.986	0.981	0.930	0.981	0.979	0.976	0.926	0.863
	P	5.695	7.460	13.40	6.942	4.106	12.09	11.24	20.40
	E_s	1.328	1.985	4.583	2.051	1.554	1.956	3.949	6.912
Henderson	*b*	0.01895	0.03670	0.05536	0.05337	0.01547	0.03831	0.04168	0.06207
	c	1.635	1.392	1.238	1.282	1.746	1.401	1.376	1.210
	R^2	0.9764	0.9768	0.9642	0.9739	0.9684	0.9715	0.9526	0.9476
	P	8.903	9.912	12.89	11.15	8.970	10.11	13.45	15.31
	E_s	1.290	2.718	5.189	2.962	1.992	2.136	4.448	7.318
Bradley	*b*	3.751	2.300	1.754	1.936	3.792	2.593	2.072	1.614
	c	0.8260	0.8581	0.8788	0.8631	0.8158	0.8435	0.8579	0.8819
	R^2	0.9893	0.9721	0.9093	0.9663	0.9794	0.9743	0.9129	0.8342
	P	8.043	13.67	26.53	17.85	7.955	12.90	21.41	33.24
	E_s	1.045	2.114	4.662	2.418	1.371	1.820	3.843	6.761
Caurie I	*b*	0.1412	0.1616	0.1771	0.1762	0.1400	0.1629	0.1675	0.1829
	m_o	0.8578	0.8342	0.8200	0.8507	0.8906	0.8576	0.8583	0.8318
	R^2	0.9700	0.9932	0.9869	0.9938	0.9884	0.9618	0.9840	0.9715
	P	8.919	5.561	7.324	5.473	4.998	11.07	11.07	10.43
	E_s	2.060	1.363	3.389	1.489	1.403	1.701	1.701	5.499
Smith	*b*	-6.047	-7.583	-8.980	-7.842	-5.708	-6.801	-7.560	-9.227
	c	4.155	1.983	0.275	0.9887	3.904	2.519	1.334	-0.3661
	R^2	0.9843	0.9858	0.9294	0.9825	0.9844	0.9832	0.9309	0.8558
	P	7.540	6.417	15.929	8.571	3.598	10.13	12.84	22.54
	E_s	1.266	1.508	4.114	1.744	1.193	1.474	3.423	6.305
Oswin	*b*	9.036	7.789	6.911	7.038	8.360	7.766	6.950	6.169
	c	0.291	0.367	0.430	0.393	0.297	0.345	0.392	0.464
	R^2	0.975	0.991	0.952	0.990	0.981	0.987	0.947	0.892
	P	12.37	6.748	7.425	6.913	7.021	11.96	7.392	12.44
	E_s	1.608	1.187	3.388	1.330	1.310	1.299	2.985	5.444
Halsey	*b*	2.586	2.217	1.995	2.085	2.707	2.277	2.203	1.964
	c	142.8	53.18	29.59	33.13	172.3	55.76	45.00	25.58
	R^2	0.9417	0.9743	0.9714	0.9782	0.9713	0.9295	0.9731	0.9567
	P	13.05	10.96	12.10	10.76	9.294	17.02	10.21	15.51
	E_s	2.788	2.111	3.586	2.213	1.938	2.343	3.107	5.403
Caurie II	*b*	1.058	0.8054	0.5908	0.5997	1.115	0.7822	0.7367	0.5199
	c	2.185	2.515	2.808	2.714	2.035	2.482	2.527	2.860
	R^2	0.9769	0.9545	0.9355	0.9490	0.9579	0.9357	0.9253	0.9143
	P	8.604	13.10	16.52	15.43	10.42	16.54	16.06	18.98
	E_s	2.074	4.258	6.873	4.673	2.784	3.598	5.745	8.894
Modified GAB	*b*	5.156	3.701	3.170	3.281	4.268	3.813	3.251	2.764
	c	0.828	0.902	0.931	0.916	0.851	0.887	0.915	0.944
	d	547.665	21327.257	200000000	38350.749	97910.576	2746.192	200000000	300000000
	R^2	0.986	0.981	0.930	0.981	0.979	0.976	0.926	0.862
	P	5.782	7.533	7.533	6.992	4.115	12.134	9.966	17.332
	E_s	1.325	1.968	1.968	2.027	1.553	1.952	3.960	6.898
Kuhn	*b*	-0.3997	-0.5163	-0.6166	-0.5186	-0.3872	-0.4598	-0.5151	-0.6409
	c	9.063	7.930	7.167	7.298	8.386	7.902	7.200	6.566
	R^2	0.8331	0.9257	0.9302	0.9331	0.8778	0.9104	0.9175	0.8966
	P	33.98	29.99	32.96	31.06	25.34	32.62	29.55	34.69
	E_s	4.132	3.447	4.090	3.405	3.339	3.399	3.740	5.339
Iglesias - Chirife	*b*	0.400	0.517	0.617	0.519	0.388	0.460	0.516	0.642
	c	9.240	8.159	7.441	7.529	8.557	8.106	7.428	6.850
	R^2	0.832	0.925	0.930	0.932	0.877	0.909	0.917	0.896
	P	34.13	30.21	33.21	31.27	25.49	32.81	29.74	34.97
	E_s	4.149	3.467	4.108	3.424	3.354	3.417	3.755	5.351
Mizrahi	*b*	-9.240	-8.159	-7.441	-7.529	-8.557	-8.106	-7.428	-6.850
	c	-8.840	-7.643	-6.823	-7.010	-8.170	-7.646	-6.913	-6.208
	R^2	0.832	0.925	0.930	0.932	0.877	0.909	0.917	0.896
	P	34.13	30.18	33.21	31.27	25.45	32.81	29.74	34.97
	E_s	4.149	3.467	4.108	3.424	3.354	3.417	3.755	5.351
Modified BET	m_o	2.510	2.813	3.100	2.747	2.384	2.597	2.720	3.123
	b	40000000	50000000	60000000	100000000	70000000	40000000	60000000	6140520
	R^2	0.566	0.842	0.901	0.880	0.622	0.788	0.855	0.875
	P	39.28	30.66	24.78	27.56	40.23	32.07	30.97	26.82
	E_s	6.661	5.034	4.875	4.560	5.872	5.234	4.955	5.868

Note: m_o, *b, c* and *d* are constants.

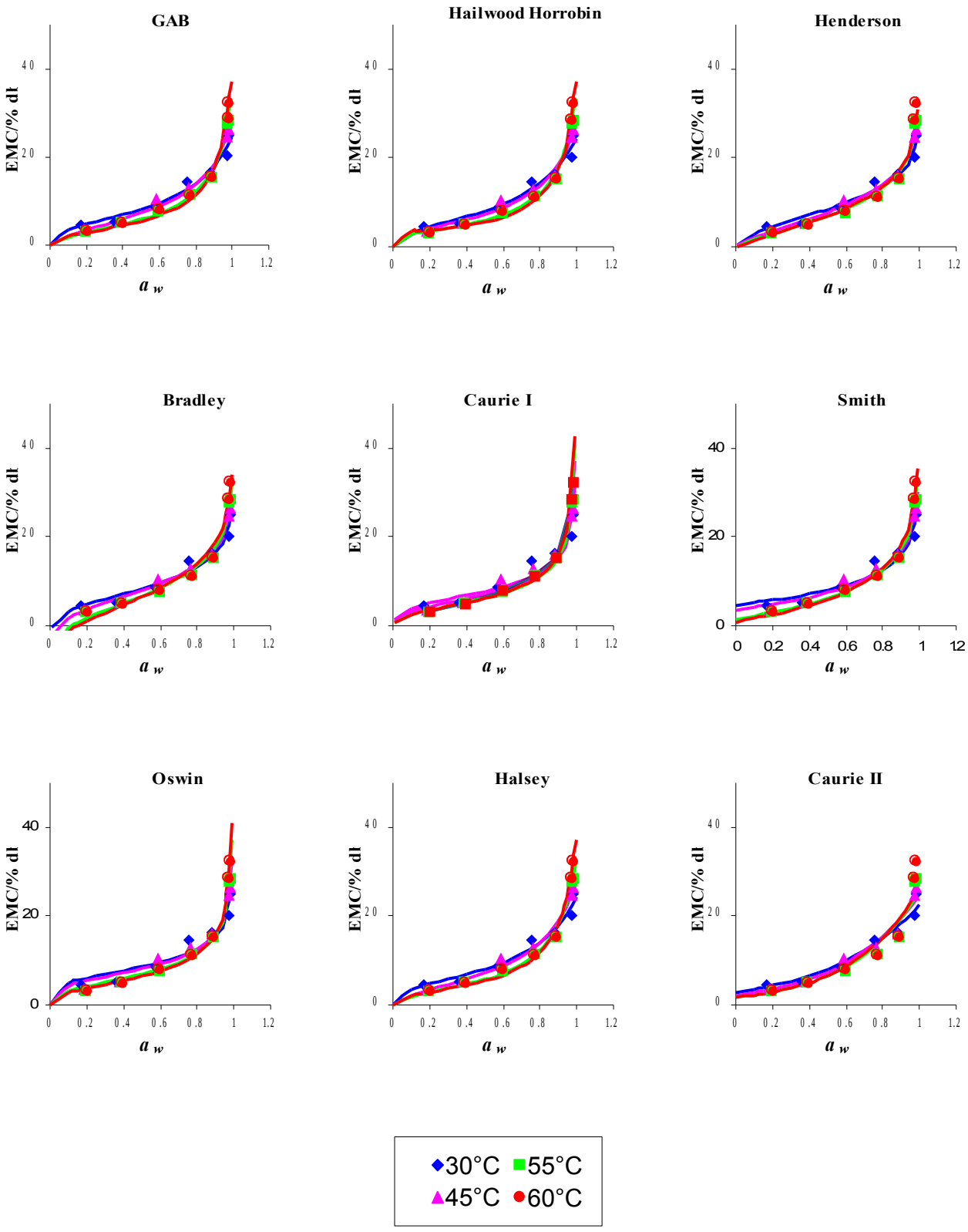


Figure 5.10. Comparison of the experimental and predicted EMC of stalk fibre of R 570 aged 52 weeks by different sorption models (Lines represent the predicted values).

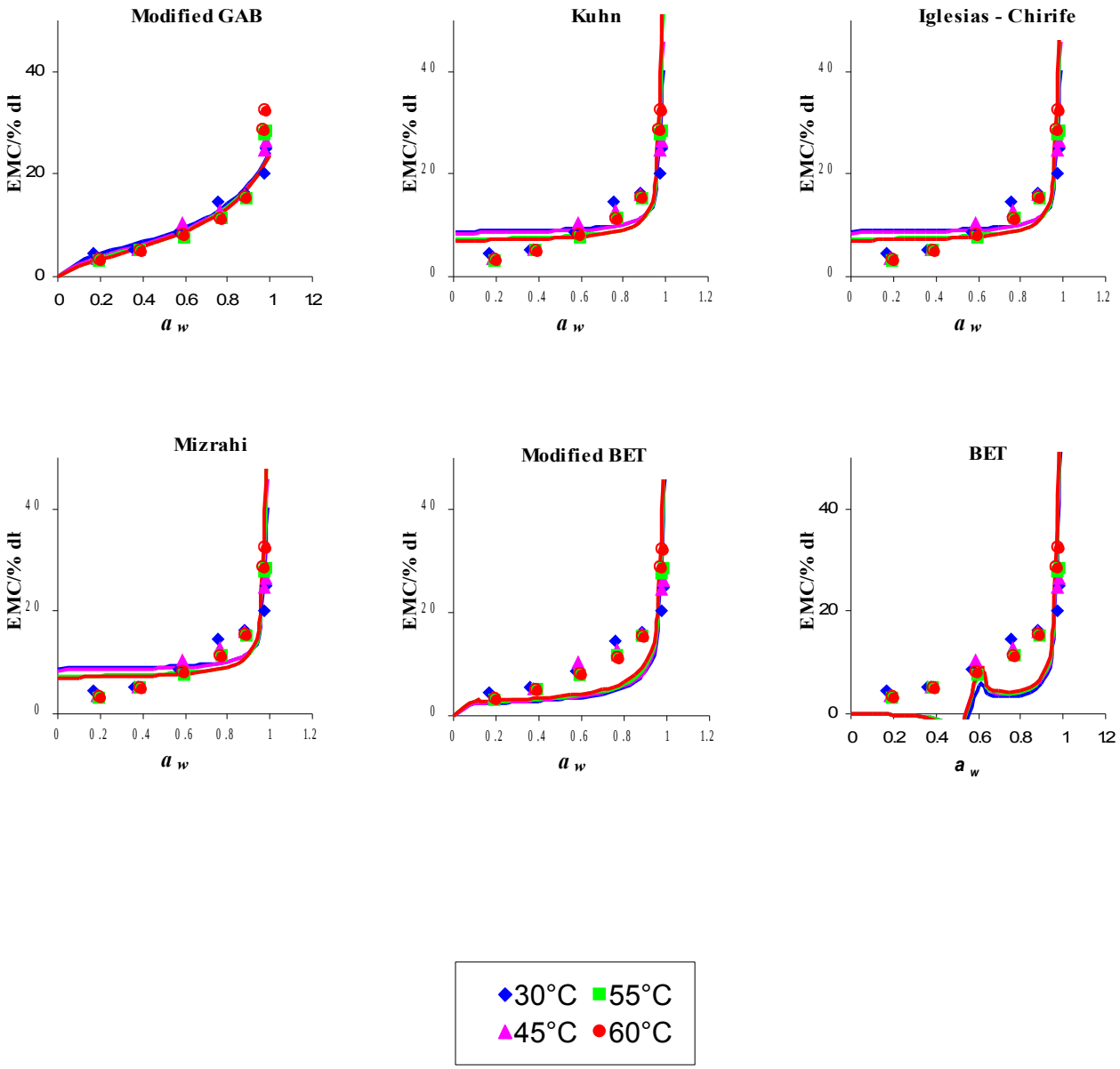


Figure 5.10. (Contd.)

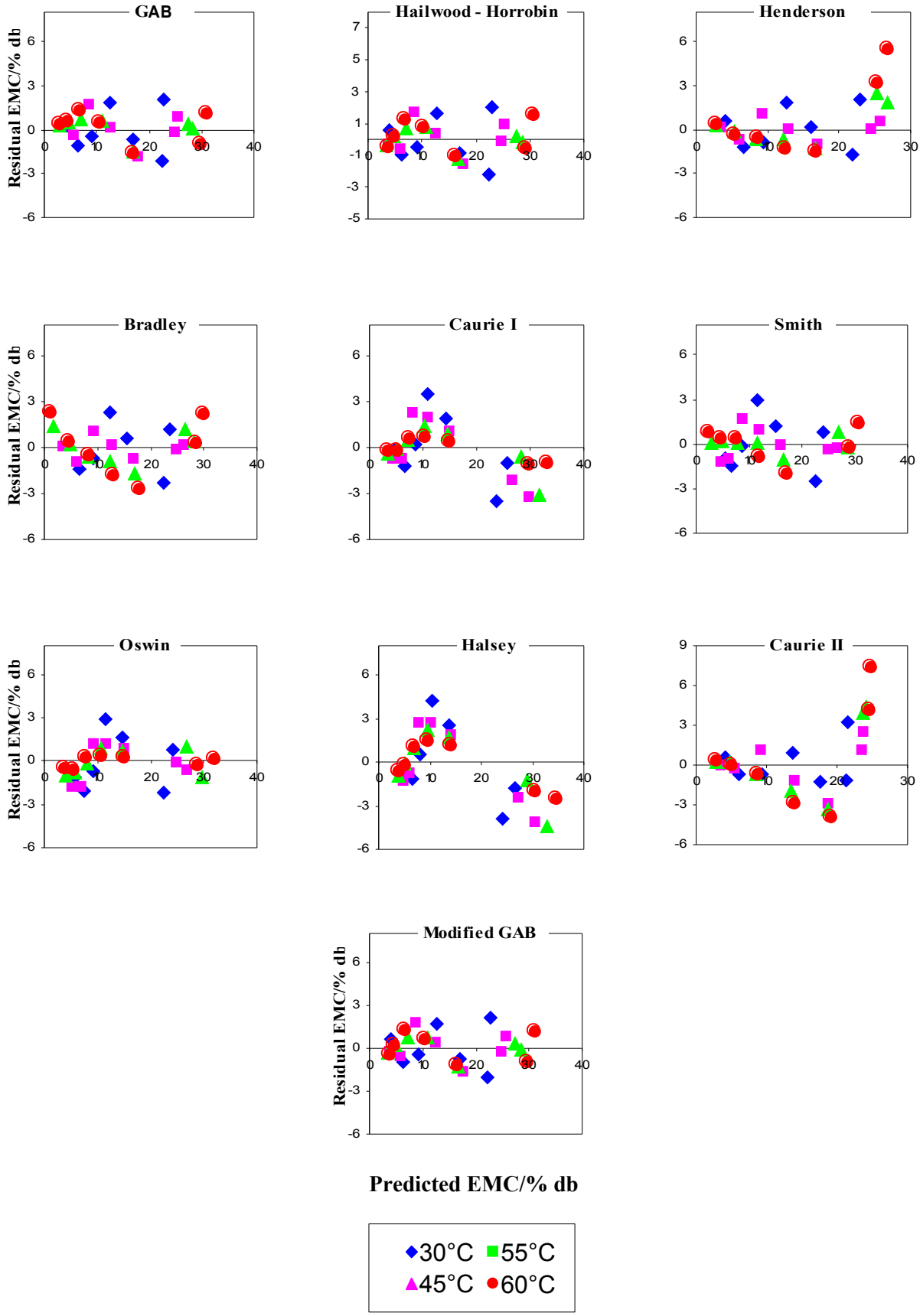


Figure 5.11. Residual plots for the different isotherm models of the EMC data of stalk fibre of R 570 aged 52 weeks at temperatures of 30, 45, 55 and 60 ºC.

Table 5.28. Classification of residual plots for various isotherm models applied to the nine cane components of R 570 aged 52 and 36 weeks.

	Stalk fibre		Stalk pith		Rind fibre		Rind fines		Top fibre		Dry leaf fibre		Dry leaf fines		Green leaf fibre		Green leaf fines	
Isotherm model	52	36	52	36	52	36	52	36	52	36	52	36	52	36	52	36	52	36
GAB	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Hailwood Horrobin	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Henderson	R	R	S	S	R	R	R	R	S	S	S	R	S	S	S	R	S	S
Bradley	R	R	R	R	R	R	R	R	R	R	S	R	R	R	R	R	S	R
Caurie I	S	S	S	S	S	S	S	S	S	S	S	R	S	S	S	S	S	S
Smith	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R
Oswin	R	R	R	R	S	R	S	R	S	R	R	R	S	R	S	R	S	R
Halsey	S	S	S	S	S	S	S	S	S	S	S	R	S	S	S	S	S	R
Caurie II	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Modified GAB	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R

R = random, S = systematic

For the four remaining models, a comparison of the performance parameters R^2, P and E_s listed in Tables 5.19 – 5.27 showed that, in general, the GAB, Hailwood-Horrobin and modified GAB models all yield comparable R^2 values, however, the modified GAB model gave a slightly lower value of P and E_s than the Hailwood-Horrobin, GAB and Henderson models, in that order. Hence, it has been shown that the modified GAB model provides an acceptable description of the isotherms of sugar cane component parts in the range of water activity and temperature studied, with only the exception mentioned above, with GAB and Hailwood-Horrobin models rank a close second followed by Henderson models. It is to be noted that none of the models studied gave a satisfactory fit to the data obtained at 60 °C for dry leaf fibre and green leaf fibre, both aged 36 weeks. This is due to the data obtained as seen in Fig 5.9, both components gave isotherms which were in general less steep than those of the other cane components.

The experimental EMC data of the nine cane components aged 52 weeks were compared with the data predicted by the modified GAB, Hailwood-Horrobin and GAB models (see Figs 5.12 – 5.14). Although the modified GAB model was found best to describe the isotherms of the sugar cane component parts at temperatures of 30, 45, 55 and 60 °C, it did not extend beyond water activity of 0.95, whereas the other two did, up to a water activity of one. So, in conclusion, the models which best describe the sorption characteristics of the sugar cane component parts are the Hailwood-Horrobin and the GAB isotherms.

In contrast, as mentioned in Section 5.4.1, Han and Wu (2004) found that Nelson's sorption isotherm model was a good fit for their experimental adsorption data of sugar cane rind. As Nelson's model involves desorption which was not performed on samples investigated, comparison could not be made.

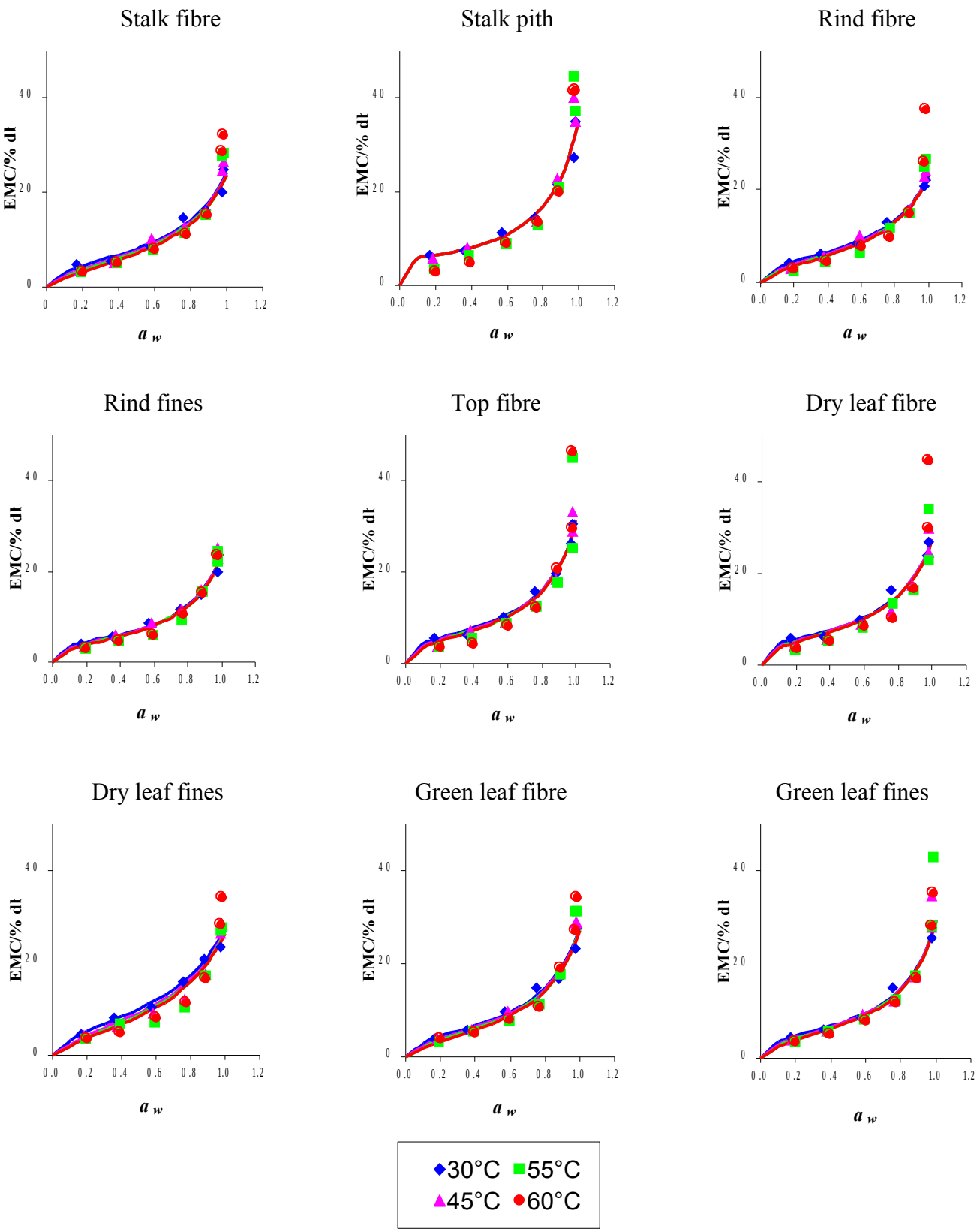


Figure 5.12. Comparison of the experimental and predicted EMC of the nine cane components of R 570 aged 52 weeks by the modified GAB model (Lines represent the predicted values).

Note the similar behaviour of all the nine cane components and the temperature correction by the isotherm model.

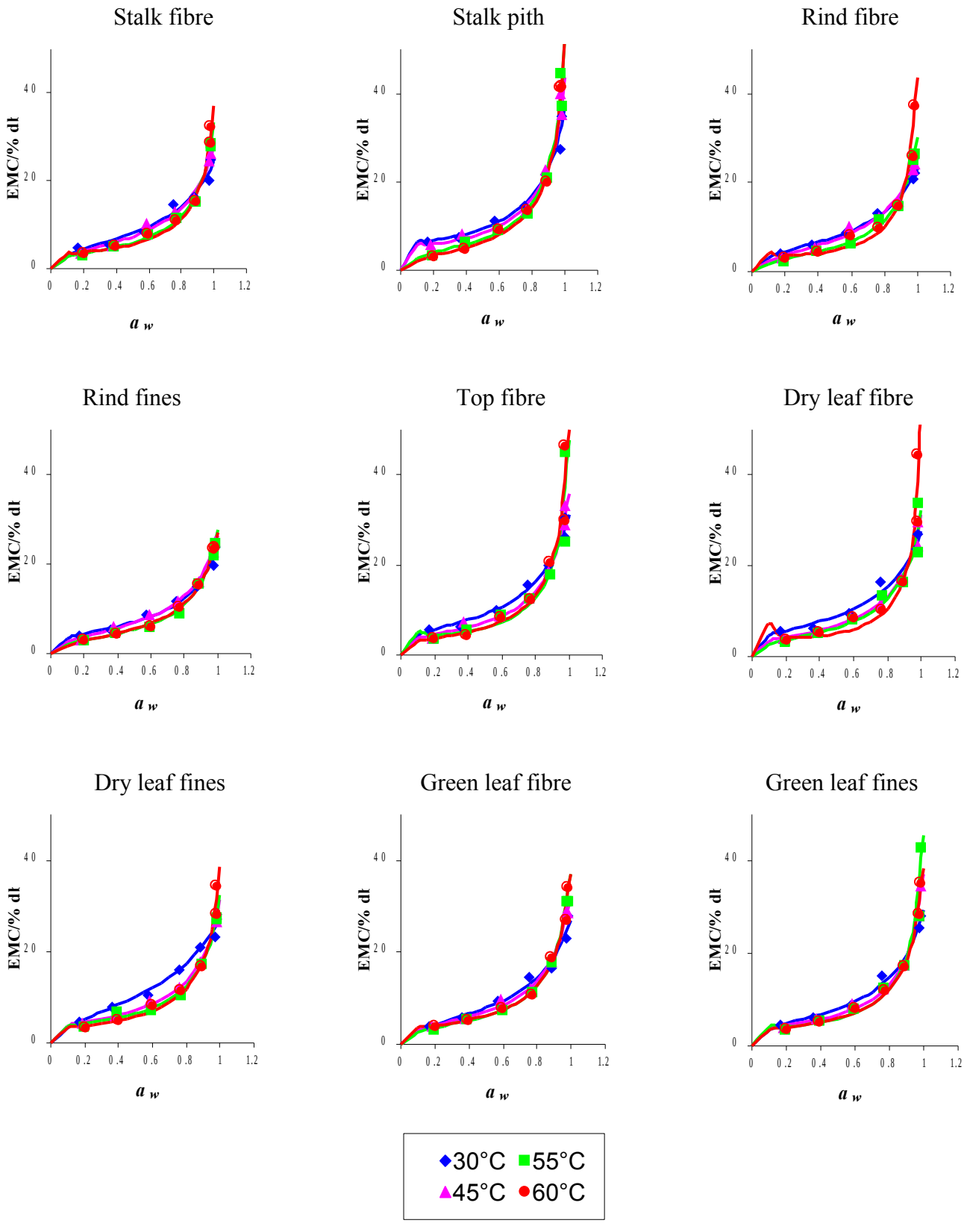


Figure 5.13. Comparison of the experimental and predicted EMC of the nine cane components of R 570 aged 52 weeks by the Hailwood-Horrobin model (Lines represent the predicted values). Note the similar behaviour of all the nine cane components.

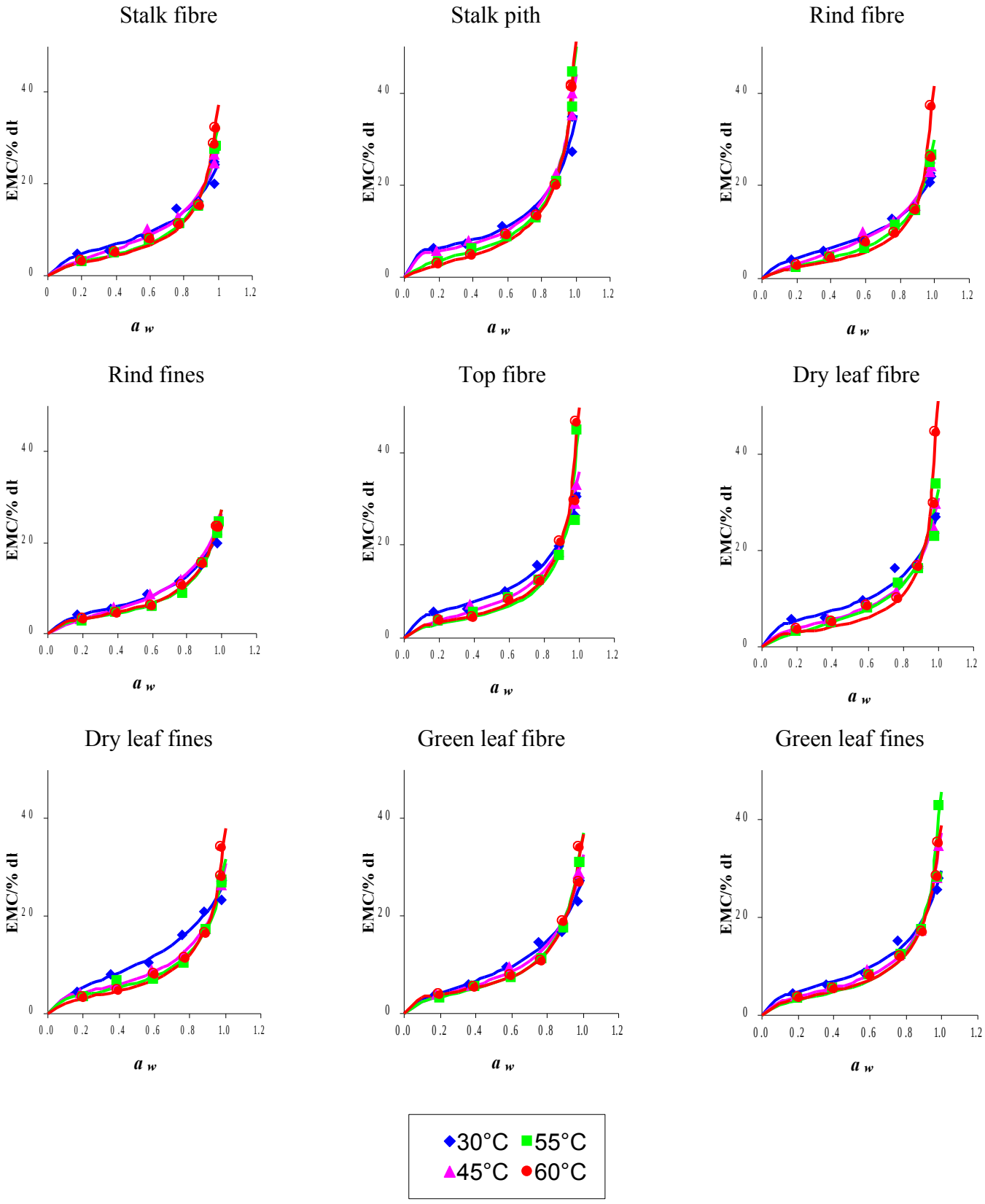


Figure 5.14. Comparison of the experimental and predicted EMC of the nine cane components of R 570 aged 52 weeks by the GAB model (Lines represent the predicted values).

Note the similar behaviour of all the nine cane components.

5.6.4.5 *Calculated EMC values of reconstituted R 570*

In a similar way to the work done on corn stover by Igathinathane *et al.* (2005), as described in 4.7.4, the EMC of sugar cane stalk could be estimated from the dry mass fraction of cane stalk fibre, stalk pith, rind fibre and rind fines, and their observed EMC. Thus:

$$M_{st} = D'_{sF} M_{sF} + D'_{sp} M_{sp} + D'_{rF} M_{rF} + D'_{rf} M_{rf}$$

where D'_{sF}, D'_{sp}, D'_{rF} and D'_{rf} are the dry mass fractions of stalk fibre, stalk pith, rind fibre and rind fines, M_{st} is the estimated EMC of the sugar cane stalk, and M_{sF}, M_{sp}, M_{rF} and M_{rf} are the measured EMC of stalk fibre, stalk pith, rind fibre and rind fines respectively.

From Table 4.25, the average mass fractions of these four components from the three replicates of R 570 aged 52 weeks were calculated to be 0.169, 0.244, 0.347 and 0.240 respectively. Their respective measured EMC's at various water activities at 30, 45, 55 and 60 °C were extracted from Tables 5.8 – 5.11 to enable the calculation of the EMC values of reconstituted R 570 cane stalk aged 52 weeks (Table 5.29).

Similarly, from Table 4.27, the average mass fractions of the four components from the three replicates of R 570 aged 36 weeks were found to be 0.120, 0.216, 0.399 and 0.265 respectively for stalk fibre, stalk pith, rind fibre and rind fines. Their respective measured EMC's at various water activities at 30, 45, 55 and 60 °C are extracted from Tables 5.8 – 5.11. EMC values of reconstituted R 570 aged 36 weeks were thus calculated (Table 5.30).

In a similar manner, EMC of reconstituted dry leaf and green leaf aged 52 and 36 weeks were predicted from their respective mass fractions (fibre and fines) and their experimental EMC values (Tables 5.31 – 5.32). From Table 4.25, the mass fractions of dry leaf fibre and fines were calculated to be 0.579 and 0.421 for 52 weeks sample, and those of the green leaf fibre and fines were 0.371 and 0.329.

For 36 weeks sample, the mass fractions of dry leaf fibre and fines were 0.631 and 0.369, and of the green leaf fibre and fines, 0.701 and 0.299 (Table 4.27).

Since the Hailwood-Horrobin isotherm and GAB models were found to describe the sorption behaviour of the fibres of sugar cane components well, these models were fitted to the calculated EMC data for reconstituted cane stalk, dry leaf and green leaf to determine whether they fitted these data too.

Table 5.29. Calculated equilibrium moisture content (db) for reconstituted cane stalk of variety R 570 aged 52 weeks at various temperatures and water activities.

m/m of H_2SO_4 soln	30 °C						45 °C						55 °C						60 °C					
	Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db				
		Experimental				Predicted		Experimental				Predicted		Experimental				Predicted		Experimental				Predicted
		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted
		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane
5%	0.9808	23.6	35.7	21.7	23.5	25.89	0.9812	25.2	31.6	23.7	24.7	26.17	0.9816	26.2	35.0	22.8	24.3	26.76	0.9818	31.0	42.8	29.6	22.2	31.33
		26.0	34.1	22.3	24.0	26.26		27.4	38.3	24.4	25.5	28.60		30.7	39.2	30.2	24.8	31.23		33.4	40.1	44.8	24.7	36.97
mean		24.8	34.9	22.0	23.8	**26.08**		26.3	34.9	24.0	25.1	**27.38**		28.4	37.1	26.5	24.5	**29.00**		32.2	41.5	37.2	23.5	**34.15**
10%	0.9746	20.5	26.1	19.7	20.0	21.52	0.9751	25.4	40.3	24.2	25.1	28.59	0.9760	30.6	46.6	26.5	22.3	31.15	0.9764	32.8	40.5	32.9	28.5	33.73
		19.8	28.3	22.0	19.4	22.58		23.7	39.5	21.4	20.2	25.95		24.8	42.3	23.6	21.7	27.96		24.4	41.9	19.0	18.4	25.37
mean		20.1	27.2	20.9	19.7	**22.05**		24.6	39.9	22.8	22.6	**27.27**		27.7	44.5	25.1	22.0	**29.56**		28.6	41.2	25.9	23.4	**29.55**
20%	0.8814	15.2	22.1	16.6	15.1	17.39	0.8839	16.5	24.0	16.3	16.8	18.37	0.8868	16.0	19.5	15.5	16.4	16.81	0.8882	16.0	21.8	15.3	16.3	17.30
		17.2	20.9	14.3	14.7	16.54		15.3	21.1	15.0	15.1	16.61		14.6	22.4	14.2	14.7	16.43		14.3	18.1	14.2	14.4	15.27
mean		16.2	21.5	15.4	14.9	**16.97**		15.9	22.6	15.7	15.9	**17.49**		15.3	20.9	14.8	15.6	**16.62**		15.2	20.0	14.8	15.3	**16.29**
30%	0.7549	13.7	15.0	11.4	11.6	12.76	0.7629	14.4	13.8	13.8	10.8	13.21	0.7684	12.6	12.9	13.6	10.5	12.55	0.7711	11.1	12.6	9.08	10.2	10.58
		15.2	13.9	14.6	11.3	13.79		11.2	13.2	10.4	12.6	11.77		10.5	12.7	9.76	7.71	10.12		10.8	14.2	10.1	10.7	11.40
mean		14.5	14.5	13.0	11.5	**13.28**		12.8	13.5	12.1	11.7	**12.49**		11.6	12.8	11.6	9.12	**11.33**		11.0	13.4	9.61	10.4	**10.99**
40%	0.5711	9.65	12.9	9.57	9.50	10.38	0.5866	9.60	9.51	8.96	8.02	8.98	0.5948	7.76	9.05	5.80	6.77	7.16	0.5989	7.89	9.31	7.13	6.76	7.70
		7.62	9.23	7.63	7.51	7.99		11.0	9.70	10.8	9.22	10.22		7.85	8.92	7.19	5.25	7.26		8.04	8.90	8.71	5.26	7.82
mean		8.64	11.0	8.60	8.51	**9.19**		10.3	9.61	9.91	8.62	**9.60**		7.81	8.99	6.50	6.01	**7.21**		7.97	9.11	7.92	6.01	**7.76**
50%	0.3574	5.40	6.62	5.07	5.42	5.59	0.3765	5.16	8.23	4.95	6.57	6.17	0.3879	5.33	6.54	5.14	4.99	5.48	0.3936	5.32	5.89	4.51	4.45	4.97
		5.30	8.12	6.97	5.53	6.62		5.34	7.87	5.98	5.17	6.14		4.83	6.31	3.98	4.54	4.83		4.57	3.79	4.43	4.50	4.31
mean		5.35	7.37	6.02	5.48	**6.11**		5.25	8.05	5.47	5.87	**6.16**		5.08	6.43	4.56	4.77	**5.15**		4.95	4.84	4.47	4.48	**4.64**
60%	0.1677	4.93	8.08	3.52	4.25	5.05	0.1834	3.47	6.68	2.66	3.16	3.90	0.1937	3.35	3.61	3.17	3.01	3.27	0.1988	3.21	2.42	3.07	3.13	2.95
		4.33	4.85	4.82	3.91	4.53		3.51	4.76	3.15	3.13	3.60		2.80	3.44	1.84	2.76	2.61		3.32	3.36	2.79	3.04	3.08
mean		4.63	6.47	4.17	4.08	**4.79**		3.49	5.72	2.91	3.15	**3.75**		3.08	3.53	2.51	2.89	**2.94**		3.27	2.89	2.93	3.09	**3.01**

Table 5.30. Calculated equilibrium moisture content (db) for reconstituted cane stalk of variety R 570 aged 36 weeks at various temperatures and water activities.

m/m of H_2SO_4 soln	30 °C						45 °C						55 °C						60 °C					
	Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db					Water a_w	Water adsorbed/% db				
		Experimental				Predicted		Experimental				Predicted		Experimental				Predicted		Experimental				Predicted
		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted		Stalk		Rind		Reconstituted
		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane		fibre	pith	fibre	fines	cane
5%	0.9808	24.20	31.90	21.60	23.35	24.65	0.9812	25.70	37.04	22.21	24.73	26.51	0.9816	26.70	36.50	24.78	23.00	27.08	0.9818	26.52	36.50	24.10	24.80	27.29
		24.10	31.20	21.60	23.00	24.36		27.40	34.10	24.30	26.20	27.32		27.30	36.80	30.40	24.90	29.98		21.18	36.80	21.40	17.70	23.75
mean		24.20	31.60	21.60	23.18	**24.51**		26.60	35.50	23.20	25.50	**26.92**		27.00	36.60	27.50	23.90	**28.53**		23.85	36.60	22.80	21.30	**25.52**
10%	0.9746	25.62	27.24	19.70	19.77	22.09	0.9751	25.90	37.62	21.40	20.13	25.14	0.9760	27.90	37.94	22.91	18.30	25.56	0.9764	27.11	37.94	24.97	17.82	26.14
		22.30	23.70	20.10	18.60	20.80		29.50	33.00	23.60	21.20	25.75		26.40	36.70	21.30	17.70	24.36		27.76	36.70	20.62	17.90	24.25
mean		23.96	25.50	19.90	19.18	**21.45**		27.76	35.30	22.50	20.66	**25.44**		27.10	37.32	22.10	18.00	**24.96**		27.44	37.32	22.80	17.86	**25.19**
20%	0.8814	16.02	19.45	18.21	15.28	17.44	0.8839	15.38	18.06	14.90	14.20	15.51	0.8868	16.04	19.80	15.58	14.51	16.26	0.8882	15.10	19.80	12.90	13.52	14.87
		14.94	18.50	14.57	15.00	15.61		15.55	17.80	15.20	14.90	15.78		13.12	18.32	14.68	13.20	14.91		15.58	18.32	14.70	11.80	14.85
mean		15.48	19.00	16.39	15.14	**16.53**		15.47	17.93	15.14	14.61	**15.64**		14.58	19.06	15.13	13.86	**15.59**		15.34	19.06	13.80	12.66	**14.86**
30%	0.7549	12.41	14.30	11.85	12.07	12.50	0.7629	12.26	15.44	10.65	11.80	12.21	0.7684	10.32	16.10	10.92	8.81	11.41	0.7711	11.83	16.10	11.17	10.25	12.07
		10.66	12.90	12.40	10.77	11.91		12.70	16.64	12.20	13.64	13.61		11.60	13.20	9.61	9.85	10.70		10.32	13.20	9.54	11.00	10.82
mean		11.54	13.60	12.13	11.42	**12.21**		12.48	16.04	11.42	12.72	**12.91**		10.96	14.60	10.27	9.33	**11.05**		11.08	14.60	10.36	10.63	**11.45**
40%	0.5711	8.21	11.76	7.91	8.15	8.84	0.5866	7.66	9.15	7.39	7.60	7.86	0.5948	8.18	7.15	7.71	5.25	6.99	0.5989	7.49	7.15	10.02	7.20	8.35
		7.65	10.96	8.97	7.73	8.91		7.79	9.75	7.65	7.72	8.14		7.86	8.53	7.47	7.49	7.75		7.59	8.53	7.00	7.82	7.62
mean		7.93	11.36	8.44	7.94	**8.88**		7.73	9.45	7.52	7.66	**8.00**		8.02	7.84	7.59	6.37	**7.37**		7.54	7.84	8.51	7.51	**7.98**
50%	0.3574	4.69	5.73	4.54	4.86	4.90	0.3765	5.40	5.76	5.15	5.24	5.34	0.3879	5.55	4.01	4.78	4.80	4.71	0.3936	5.10	4.01	6.65	4.94	5.44
		6.89	6.28	4.98	5.29	5.57		4.53	6.62	5.14	5.32	5.43		5.59	5.28	5.10	5.07	5.19		4.04	5.28	4.58	5.83	5.00
mean		5.79	6.01	4.76	5.08	**5.24**		4.97	6.19	5.15	5.28	**5.38**		5.57	4.65	4.94	4.94	**4.95**		4.57	4.65	5.62	5.39	**5.22**
60%	0.1677	4.15	5.22	4.04	4.17	4.34	0.1834	3.38	4.09	3.08	3.28	3.39	0.1937	3.56	3.90	3.34	3.36	3.49	0.1988	2.45	3.90	2.01	3.16	2.78
		3.92	5.00	7.06	4.08	5.45		3.26	4.05	2.07	3.15	2.93		3.16	3.81	3.05	1.72	2.87		2.76	3.81	2.83	2.90	3.05
mean		4.04	5.11	5.55	4.13	**4.90**		3.32	4.07	2.58	3.22	**3.16**		3.36	3.86	3.20	2.54	**3.18**		2.61	3.86	2.42	3.03	**2.91**

Table 5.31. Calculated equilibrium moisture content (db) for reconstituted dry leaf and green leaf of variety R 570 aged 52 weeks at various temperatures and water activities.

m/m of 96% H_2SO_4 soln	30 °C							45 °C							55 °C							60 °C						
	Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db					
		Experimental				Predicted			Experimental				Predicted			Experimental				Predicted			Experimental				Predicted	
		Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted	
		fibre	fines	fibre	fines	dry leaf	green leaf		fibre	fines	fibre	fines	dry leaf	green leaf		fibre	fines	fibre	fines	dry	green		fibre	fines	fibre	fines	dry	green leaf
5%	0.9808	26.2 27.5	27.3 28.6	27.7 27.8	27.3 28.5	26.69 28.02	27.64 28.07	0.9812	31.5 27.5	27.7 28.5	27.6 29.5	30.4 38.5	29.98 27.97	28.61 32.49	0.9816	31.6 36.3	28.61 26.61	30.3 31.9	37.5 48.1	30.37 32.25	32.68 37.28	0.9818	48.2 40.5	34.0 34.3	39.9 28.1	31.3 39.0	42.26 37.96	40.69 31.38
Mean		26.9	27.9	27.8	27.9	**27.36**	**27.86**		29.5	28.1	28.6	34.5	**28.97**	**30.55**		34.0	27.61	31.1	42.8	**31.31**	**34.98**		44.4	34.2	34.0	35.2	**40.11**	**36.03**
10%	0.9746	25.4 22.0	23.5 22.9	22.6 23.7	26.4 24.7	24.66 22.41	23.91 24.05	0.9751	24.6 24.2	27.1 25.7	30.7 26.8	28.5 27.5	25.66 24.91	30.04 27.09	0.9760	26.1 19.6	30.08 24.00	33.5 28.9	30.4 25.7	27.80 21.49	32.52 27.87	0.9764	26.0 33.1	24.1 32.1	26.0 28.0	30.6 25.8	25.23 32.75	25.79 29.57
Mean		23.7	23.2	23.1	25.6	**23.54**	**23.98**		24.4	26.4	28.8	28.0	**25.29**	**28.57**		22.9	27.04	31.2	28.1	**24.65**	**30.20**		29.6	28.1	27.0	28.2	**28.99**	**27.68**
20%	0.8814	16.7 16.3	22.3 19.4	16.6 16.6	18.1 16.4	19.12 17.65	17.13 16.56	0.8839	18.0 16.4	18.6 16.3	18.5 17.0	18.0 16.7	18.30 16.39	18.39 16.96	0.8868	16.9 15.8	17.75 16.73	18.6 16.8	18.3 16.7	17.28 16.22	18.57 16.79	0.8882	17.2 15.8	16.8 16.1	18.2 19.5	16.3 17.5	17.08 15.98	17.88 18.35
Mean		16.5	20.9	16.6	17.2	**18.38**	**16.84**		17.2	17.5	17.8	17.3	**17.35**	**17.67**		16.4	17.24	17.7	17.5	**16.75**	**17.68**		16.5	16.5	18.8	16.9	**16.53**	**18.11**
30%	0.7549	17.5 15.1	18.5 13.5	14.5 14.8	17.4 12.9	17.93 14.43	15.50 14.24	0.7629	12.5 10.7	12.2 11.7	12.3 11.5	13.2 11.2	12.40 11.16	12.64 11.45	0.7684	14.4 12.3	11.64 9.32	12.7 9.92	12.0 13.1	13.26 11.09	12.53 10.99	0.7711	10.7 9.41	11.3 11.3	12.0 9.35	11.6 11.9	11.02 10.21	11.74 9.63
Mean		16.3	16.0	14.7	15.1	**16.18**	**14.87**		11.6	11.9	11.9	12.2	**11.78**	**12.05**		13.4	10.48	11.3	12.6	**12.17**	**11.76**		10.0	11.3	10.7	11.8	**10.62**	**10.69**
40%	0.5711	10.8 8.49	11.2 9.83	10.7 8.35	9.11 8.42	10.98 9.05	10.19 8.37	0.5866	8.97 8.37	8.79 9.19	10.2 9.21	8.66 9.68	8.89 8.72	9.73 9.36	0.5948	8.58 8.08	8.28 6.26	6.92 8.30	8.31 8.06	8.45 7.31	7.38 8.22	0.5989	7.77 9.14	8.19 7.84	8.39 7.45	8.60 7.72	7.95 8.59	8.24 7.83
Mean		9.65	10.5	9.54	8.77	**10.02**	**9.28**		8.67	8.99	9.73	9.17	**8.80**	**9.55**		8.33	7.27	7.61	8.19	**7.88**	**7.80**		8.46	8.02	7.92	8.16	**8.27**	**8.04**
50%	0.3574	5.98 6.20	7.40 8.57	5.80 6.00	5.99 6.22	6.58 7.20	5.86 6.07	0.3765	5.54 5.86	5.44 6.59	5.54 5.78	5.67 5.86	5.50 6.17	5.58 5.81	0.3879	5.29 5.27	9.48 4.21	5.83 5.32	5.86 5.37	7.05 4.82	5.84 5.34	0.3936	5.26 5.06	5.46 4.38	5.45 5.17	5.49 4.96	5.34 4.77	5.42 5.04
Mean		6.09	7.99	5.90	6.11	**6.89**	**5.97**		5.70	6.02	5.66	5.77	**5.83**	**5.69**		5.28	6.85	5.58	5.62	**5.94**	**5.59**		5.16	4.92	5.31	5.23	**5.06**	**5.23**
60%	0.1677	4.11 7.36	4.42 4.84	3.96 3.95	4.31 4.90	4.24 6.30	4.08 4.26	0.1834	3.79 3.89	3.56 4.98	3.84 3.66	3.97 3.96	3.69 4.35	3.88 3.76	0.1937	3.73 2.64	3.87 3.23	3.39 3.25	3.87 3.18	3.79 2.89	3.55 3.23	0.1988	3.59 3.66	4.04 2.77	3.52 4.48	3.54 3.73	3.78 3.29	3.61 4.09
Mean		5.74	4.63	3.96	4.61	**5.27**	**4.17**		3.84	4.27	3.75	3.97	**4.02**	**3.82**		3.19	3.55	3.32	3.53	**3.34**	**3.39**		3.63	3.41	4.00	3.64	**3.53**	**3.85**

Table 5.32. Calculated equilibrium moisture content (db) for reconstituted dry leaf and green leaf of variety R 570 aged 36 weeks at various temperatures and water activities.

m/m of H_2SO_4 soln	30 °C							45 °C							55 °C							60 °C						
	Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db						Water a_w	Water adsorbed/% db					
		Experimental				Predicted			Experimental				Predicted			Experimental				Predicted			Experimental				Predicted	
		Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted			Dry leaf		Green leaf		Reconstituted	
		fibre	fines	fibre	fines	dry	green		fibre	fines	fibre	fines	dry	green		fibre	fines	fibre	fines	dry	green		fibre	fines	fibre	fines	dry	green leaf
5%	0.9808	28.0	28.1	27.0	28.1	28.06	27.34	0.9812	33.2	32.6	30.0	29.5	33.00	29.88	0.9816	33.2	30.8	27.6	37.4	32.33	30.57	0.9818	64.5	31.0	58.8	41.7	52.20	53.73
		27.0	27.8	26.9	27.7	27.32	27.18		28.3	31.6	29.6	33.3	29.57	30.73		26.5	25.1	29.0	36.5	26.04	31.30		54.9	26.3	57.8	51.8	44.42	56.03
Mean		27.5	27.9	26.9	27.9	**27.69**	**27.26**		30.7	32.1	29.8	31.4	**31.28**	**30.31**		29.9	27.9	28.3	37.0	**29.18**	**30.94**		59.7	28.7	58.3	46.8	**48.31**	**54.88**
10%	0.9746	25.5	23.3	24.3	21.9	24.75	23.60	0.9751	27.7	26.7	29.0	25.8	27.39	28.06	0.9760	25.0	27.7	31.5	23.7	26.08	29.20	0.9764	19.2	23.7	27.8	24.7	20.90	26.93
		24.3	21.8	21.7	23.7	23.41	22.32		25.2	27.5	29.9	26.0	26.09	28.80		22.3	26.3	23.4	25.3	23.83	24.04		21.4	27.4	32.5	25.8	23.66	30.55
Mean		24.9	22.5	23.0	22.8	**24.08**	**22.96**		26.5	27.1	29.5	25.9	**26.74**	**28.43**		23.7	27.0	27.5	24.5	**24.96**	**26.62**		20.3	25.6	30.2	25.3	**22.28**	**28.74**
20%	0.8814	17.1	18.0	17.1	17.2	17.48	17.19	0.8839	16.9	17.4	17.5	17.0	17.11	17.37	0.8868	17.1	17.4	17.6	17.0	17.24	17.48	0.8882	13.9	15.5	16.8	16.3	14.50	16.69
		16.4	16.8	17.1	16.5	16.55	16.95		15.4	16.7	17.1	16.5	15.90	16.95		16.3	16.7	17.0	16.1	16.50	16.76		14.7	14.0	15.5	17.0	14.47	15.99
Mean		16.7	17.4	17.1	16.8	**17.01**	**17.07**		16.1	17.0	17.3	16.7	**16.51**	**17.16**		16.7	17.0	17.3	16.5	**16.87**	**17.12**		14.3	14.7	16.2	16.6	**14.49**	**16.34**
30%	0.7549	13.2	12.9	12.2	12.2	13.16	12.22	0.7629	13.1	11.9	11.9	11.6	12.72	11.83	0.7684	12.5	12.4	12.4	12.0	12.50	12.35	0.7711	10.9	13.2	12.0	12.8	11.78	12.27
		14.0	12.1	12.4	11.7	13.32	12.26		12.9	12.2	12.1	11.7	12.67	12.01		12.0	13.8	11.5	11.9	12.70	11.68		11.9	10.9	11.3	11.4	11.62	11.40
Mean		13.6	12.5	12.3	12.0	**13.24**	**12.24**		13.0	12.0	12.0	11.7	**12.69**	**11.92**		12.2	13.1	12.0	11.9	**12.60**	**12.02**		11.4	12.1	11.6	12.1	**11.70**	**11.83**
40%	0.5711	8.92	9.38	8.73	8.94	9.09	8.79	0.5866	8.35	11.6	8.26	8.14	9.55	8.22	0.5948	9.73	9.81	6.54	7.24	9.76	6.75	0.5989	7.43	8.39	9.16	7.26	7.78	8.59
		8.56	10.6	8.34	8.51	9.33	8.39		8.56	8.87	8.42	8.37	8.67	8.41		8.37	10.6	8.39	8.12	9.22	8.31		4.33	8.83	7.14	9.04	5.99	7.71
Mean		8.74	10.0	8.54	8.73	**9.21**	**8.59**		8.46	10.2	8.34	8.26	**9.11**	**8.31**		9.05	10.2	7.47	7.68	**9.49**	**7.53**		5.88	8.61	8.15	8.15	**6.89**	**8.15**
50%	0.3574	5.97	6.44	6.05	6.52	6.14	6.19	0.3765	8.80	6.74	6.94	8.86	8.04	7.51	0.3879	5.49	5.60	5.43	5.37	5.53	5.41	0.3936	5.45	5.58	5.66	4.98	5.50	5.46
		5.77	6.21	5.92	5.97	5.93	5.93		8.21	6.48	4.99	6.21	7.57	5.35		8.59	6.87	6.52	6.07	7.96	6.39		5.07	7.12	5.29	5.93	5.83	5.48
Mean		5.87	6.33	5.99	6.25	**6.04**	**6.06**		8.51	6.61	5.97	7.54	**7.81**	**6.43**		7.04	6.24	5.98	5.72	**6.74**	**5.90**		5.26	6.35	5.48	5.46	**5.66**	**5.47**
60%	0.1677	4.83	5.02	4.68	4.86	4.90	4.73	0.1834	3.70	3.87	3.64	2.66	3.76	3.35	0.1937	4.08	4.24	2.74	4.06	4.14	3.13	0.1988	3.71	3.40	3.64	3.26	3.60	3.53
		4.59	4.99	4.55	4.80	4.74	4.62		3.72	2.54	3.46	3.64	3.28	3.51		3.71	3.10	3.73	3.87	3.48	3.77		3.53	3.17	3.11	3.42	3.40	3.20
Mean		4.71	5.01	4.62	4.83	**4.82**	**4.68**		3.71	3.21	3.55	3.15	**3.52**	**3.43**		3.90	3.67	3.24	3.97	**3.81**	**3.45**		3.62	3.29	3.38	3.34	**3.50**	**3.36**

The isotherm parameters of the Hailwood-Horrobin and GAB models were estimated by the non-linear regression procedure of SigmaPlot (SPSS Inc.) for the calculated EMC data of reconstituted R 570 cane stalk, dry leaf and green leaf aged 52 weeks (Tables 5.29 and 5.31) and aged 36 weeks (Tables 5.30 and 5.32). The values of the isotherm parameters, together with the calculated regression criteria: coefficient of determination R^2, the mean deviation modulus P and the standard error of the estimate E_s, for each model and for the reconstituted cane stalk, dry leaf and green leaf aged 52 and 36 weeks are shown in Table 5.33. All R^2 values approach one and the P values are less than 10, except for reconstituted green leaf aged 36 weeks as predicted by the Hailwood-Horrobin model, and the E_s values are also low. The good-fit of the Hailwood-Horrobin and GAB models to the calculated EMC values of reconstituted cane and leaves is confirmed by inspection of the isotherm plots (Fig 5.15).

5.7 CONCLUSIONS

The EMC of cane components of variety R 570 aged 52 and 36 weeks were determined at 30, 45, 55 and 60 °C for water activities ranging from 0.17 to 0.98. The resulting sorption isotherms exhibit a type II sigmoid pattern. Three models were found to provide a good-fit to the experimental data: the modified GAB, Hailwood-Horrobin and GAB models in this order. However, the modified GAB model did not extend to water activity values greater than 0.95, whereas the other two models covered the whole range of water activities studied.

The EMC of sugar cane stalk of variety R 570 aged 36 and 52 weeks was estimated from the dry mass fractions of cane stalk fibre, stalk pith, rind fibre and rind fines, and the respective individual observed EMC values. Similarly, the EMC of dry leaf and green leaf was calculated from the dry mass fractions of fibre and fines and their constituent experimental EMC.

The GAB model was found to fit the calculated EMC values of the reconstituted cane stalk, dry leaf and green leaf of R 570 aged 36 and 52 weeks well; similarly for the Hailwood-Horrobin model except for green leaf aged 36 weeks.

The models of the sorption characteristics of the sugar cane component parts could now be used to determine a number of thermodynamic parameters that enable the bound water to be characterised. This work is described in Chapter 6.

Table 5.33. Parameters of the Hailwood Horrobin and GAB sorption isotherm models, the coefficient of determination R^2, mean relative deviation modulus P, and the standard error of the estimate E_s for **reconstituted R 570** of two ages and at various temperatures.

Reconstituted R 570	Model	Parameter	52 weeks				36 weeks			
			30 ºC	45 ºC	55 ºC	60 ºC	30 ºC	45 ºC	55 ºC	60 ºC
Cane stalk	Hailwood Horrobin	*b*	0.01	0.02	0.02	0.01	0.01	0.02	0.01	0.02
		c	0.18	0.19	0.24	0.31	0.20	0.20	0.27	0.24
		d	-0.15	-0.18	-0.24	-0.29	-0.17	-0.19	-0.25	-0.22
		R^2	0.98	1.00	1.00	0.99	0.99	0.99	0.99	0.99
		P	4.397	11.79	3.916	6.224	6.340	4.846	4.616	6.604
		E_s	1.288	2.228	0.7431	1.243	1.060	0.9785	1.167	0.8600
	GAB	m_o	5.05	4.58	3.55	3.07	4.67	3.75	3.39	3.66
		b	25.90	16.67	13.79	40.23	34.06	-300000000	24.38	14.98
		c	0.81	0.85	0.90	0.92	0.82	0.88	0.89	0.87
		R^2	0.98	1.00	1.00	0.99	0.99	0.99	0.99	0.99
		P	4.530	4.349	3.934	6.217	5.988	9.930	4.656	6.407
		E_s	1.288	0.694	0.737	1.244	1.032	1.170	0.9805	0.8417
Dry leaf	Hailwood Horrobin	*b*	0.015	0.008	0.012	-0.015	0.005	0.003	0.009	-0.057
		c	0.133	0.230	0.246	0.392	0.205	0.242	0.218	0.628
		d	-0.111	-0.206	-0.227	-0.356	-0.176	-0.215	-0.194	-0.554
		R^2	0.977	0.992	0.973	0.961	0.988	0.978	0.984	0.808
		P	6.584	3.922	8.018	11.12	4.638	12.60	8.067	28.32
		E_s	1.567	1.038	2.082	3.348	1.216	1.875	1.479	8.345
	GAB	m_o	6.424	3.933	3.581	2.846	4.669	4.055	4.094	2.166
		b	13.807	-30000000	-10000000	40000000	48.238	96.221	-50000000	8362848
		c	0.770	0.874	0.891	0.938	0.839	0.879	0.867	0.960
		R^2	0.977	0.992	0.972	0.960	0.988	0.978	0.983	0.804
		P	6.707	5.114	5.114	9.505	4.412	12.61	9.801	27.90
		E_s	1.550	1.073	1.073	3.325	1.165	1.874	1.508	8.425
Green leaf	Hailwood Horrobin	*b*	0.018	0.010	0.007	-0.002	0.219	0.243	0.251	0.597
		c	0.158	0.227	0.279	0.312	0.004	0.009	0.011	-0.052
		d	-0.141	-0.208	-0.261	-0.285	-0.188	-0.222	-0.232	-0.532
		R^2	0.978	0.996	0.994	0.973	0.986	0.997	0.991	0.870
		P	5.844	4.361	5.758	6.633	44.17	45.78	44.41	63.69
		E_s	1.622	0.8335	1.213	2.471	5.869	6.288	5.976	14.93
	GAB	m_o	4.664	4.095	3.429	3.257	4.419	3.878	3.698	2.218
		b	-200000000	28.513	45.999	4013274	66.576	33.752	27.781	10000000
		c	0.839	0.881	0.915	0.918	0.844	0.888	0.891	0.969
		R^2	0.973	0.996	0.994	0.973	0.986	0.997	0.991	2.218
		P	11.18	4.443	5.768	5.768	2.882	6.269	5.576	25.22
		E_s	1.789	0.8266	1.217	1.217	1.263	0.7331	1.217	8.099

Note: m_o, *b, c* and *d* are constants.

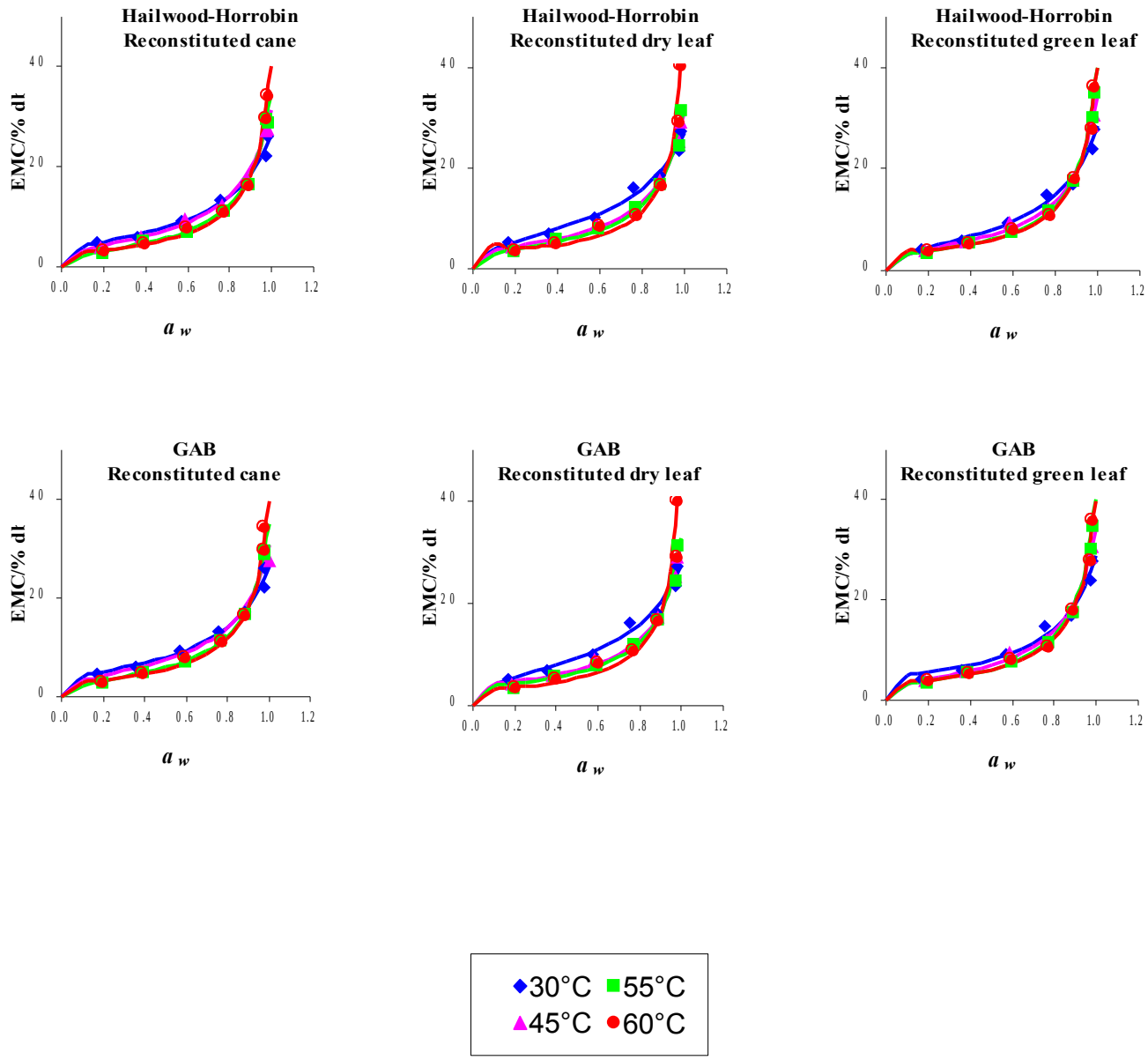


Figure 5.15. Comparison of the calculated and predicted EMC data for reconstituted cane stalk, dry leaf and green leaf of variety R 570 aged 52 weeks by the Hailwood-Horrobin and GAB models at 30, 45, 55 and 60 ºC (Lines represent data predicted by the isotherm models).

CHAPTER 6. PROPERTIES OF THE SORBED WATER IN SUGAR CANE FIBRES

In a review on moisture sorption isotherm characteristics of food products, Al-Muhtaseb *et al.* (2002) quoted that all types of water in a biological system exist as free or bound water.

With type II isotherm as exhibited by the sugar cane component parts (Section 5.6.4.3), most authors (Singh *et al.*, 2006, Arslan and Toğrul, 2005) divide the isotherm into three regions. The first, low water activity a_w region (0.05 – 0.20) is indicative of strongly bound water, at the intermediate water activity region (0.20 – 0.60), water molecules which are less firmly bound, and in the region of high water activity ($a_w \geq 0.6$) excess water is present in macro-capillaries or as part of the fluid phase in high moisture materials. This water exhibits nearly all the properties of bulk water and thus is capable of acting as a solvent.

The water in these three regions has also been termed non-freezable bound water, freezable bound water and free water. The water in the first region is strongly held by hydrogen bonds formed with the hydrophilic groups on the surface of the fibre and is not removable by drying in a vacuum. This was observed in this work that despite stringent drying, some moisture still remained in the fibres. This water is termed 'non-freezable' because it is frequently impossible to observe crystallisation or melting. The second type of bound water is less strongly held and does exhibit crystallisation and melting. Because of the bipolar nature of the water molecule, these water molecules form the multilayers by aligning themselves above those bonded to the hydroxyl groups of the fibres and form hydrogen bonds to produce hydrated layers. The water in the third region exists in the larger voids and capillaries, and essentially acts as bulk water.

Water binds to the amorphous and not the crystalline part of the cellulose, it is known that water molecules directly attached to the hydroxyl group in the amorphous region are non-freezing (Hatakeyama *et al.*, 2000). In the case of cotton cellulose, the maximum amount of bound water of cellulose fibre corresponds to that where one hydroxyl group in the amorphous region attracts about one water molecule. Hailwood and Horrobin (1946) called the fraction of a polymer molecule which is inaccessible to water molecule to form a monohydrate the "crystalline" portion of the fibres.

It has been shown in Chapter 5 that the best-fit model for the sorption isotherms of sugar cane component parts is the Hailwood-Horrobin followed by the GAB model. The work described in this chapter is aimed at investigating some properties of the sorbed water and the sorbent, and the thermodynamic properties of sorption, that can be obtained from the isotherm models and will assist in better defining the bound water in the above described three regions.

6.1 THE MONOLAYER MOISTURE CONTENT

Three of the sorption isotherm models tested in Chapter 5 have the monolayer moisture content, m_o, as one of their parameters. These are the GAB, Caurie I and the modified BET models. The m_o value gives an indication of the total number of polar groups on the sorbent binding water. The prediction of m_o values is important since the deterioration of a food material or fibre is very small below the m_o value, where water is strongly bound to the material, and is not involved in any deteriorative reaction either as a solvent or as one of the substrates.

The GAB equation was derived independently by Guggenheim (1966), Anderson (1946) and de Boer (1953). It is written as:

$$m = \frac{m_o\, b\, c\, a_w}{(1 - c\, a_w)(1 - c\, a_w + b\, c\, a_w)} ,$$

where m is the equilibrium moisture content (kg/kg dry solid, i.e. decimal), m_o is the moisture content corresponding to saturation of all primary adsorption sites by one water molecule, i.e. monolayer moisture content (kg/kg dry solid, i.e. decimal), a_w is the water activity, and b and c are constants related to the energies of interaction between the monolayer and multilayer molecules at the individual sorption sites.

Caurie I, BET and modified BET models (Table 5.1) also contain a constant term of m_o in their equation. However, since the BET equation did not fit the data well, not much credence can be given to the m_o values derived from that equation.

Caurie I $$\ell n \frac{1}{m} = - \ell n \frac{1}{b m_o} + \frac{2b}{m_o} \ell n \left(\frac{1 - a_w}{a_w} \right)$$

and modified BET $$m = \frac{m_o\, b\, a_w}{(1 - a_w)\,[1 - b\, \ell n (1 - a_w)]}$$

where m is the equilibrium moisture content (% dry solid), m_o is the monolayer moisture content (% dry solid), a_w is the water activity, b in the Caurie I equation is the density of the bound water, and b in the modified BET equation is a constant.

The monolayer moisture content, m_o, of each of these models was determined by fitting the experimental EMC data to these sorption equations (see Section 5.6.4.4). Tables 5.19 – 5.27 show the m_o values for the nine cane component parts of R 570 aged 52 and 36 weeks.

For stalk fibre aged 52 weeks, the m_o values at 30, 45, 55 and 60 °C calculated from the GAB model decrease from 5.43, 5.18, 3.68 to 3.22 g/100 g dry fibre, and with stalk fibre aged 36 weeks, the respective values were 4.17, 3.99, 3.54 and 4.00 g/100 g dry fibre. In general, the m_o values determined from the GAB equation range from 3 – 5, whereas those from the Caurie I are less than one, and those from the modified BET model were between 2 and 3.

The change of m_o values with temperature is best illustrated in Figs 6.1 to 6.3 for the GAB, Caurie I and modified BET models respectively. In general, the m_o values of the GAB and Caurie I models tend to decrease with increased temperature while that of the modified BET tend to do the opposite. The decrease in monolayer moisture content with increase in temperature at a given water activity can be explained by considering the structural changes in the fibres of the sugar cane components at increased temperatures. The degree of hydrogen bonding in such materials is reduced with increased temperature, thereby decreasing the availability of active sites for water binding and thus, the monolayer moisture content (Westgate *et al.*, 1992).

As described in Section 5.6.4.5, the calculated EMC for reconstituted R 570 cane stalk, dry leaf and green leaf aged 52 weeks and 36 weeks (Tables 5.29 to 5.32), were fitted to the Caurie I sorption model by linear regression with Microsoft Excel software, and to the modified BET model by the non-linear regression procedure of SigmaPlot (SPSS Inc.). The values of the isotherm parameters, together with the calculated regression coefficient of determination R^2, the mean deviation modulus P, and the standard error of the estimate E_s, are shown in Table 6.1.

The magnitudes of R^2, P and E_s for reconstituted cane stalk, dry leaf and green leaf were similar to those for the nine cane component parts, therefore, the previous conclusion that the Caurie I and modified BET models do not fit the data adequately also applies to reconstituted cane stalk, dry leaf and green leaf.

As seen in Section 5.6.4.5, the Hailwood-Horrobin and GAB models describe well the sorption behaviour of reconstituted cane stalk, dry leaf and green leaf.

The change of m_o values of reconstituted cane stalk and dry and green leaves with temperature as obtained from the GAB, Caurie I and modified BET models is shown in Fig 6.4.

Table 6.1. Parameters of the Caurie I and modified BET sorption models, the coefficient of determination R^2, the mean relative deviation modulus P, and the standard errors of the estimate E_s for the isotherms of **reconstituted R 570** of two ages and at various temperatures.

Reconstituted R 570	Model	Parameter	52 weeks				36 weeks			
			30 °C	45 °C	55 °C	60 °C	30 °C	45 °C	55 °C	60 °C
Cane stalk	Caurie I	*b*	0.1368	0.1551	0.1869	0.1932	0.1405	0.1705	0.1806	0.1792
		m_o	0.8979	0.8607	0.8769	0.8656	0.9212	0.8869	0.9029	0.9154
		R^2	0.9734	0.9767	0.9866	0.9939	0.9628	0.9731	0.9895	0.9747
		P	14.01	15.41	8.150	5.748	8.114	9.082	6.873	14.58
		E_s	2.774	2.923	2.408	2.000	1.046	1.978	1.533	2.622
	Modified BET	m_o	0.564	0.615	0.632	0.681	0.538	0.589	0.585	0.545
		b	10000000	9738113	10000000	8725001	20000000	8909343	10000000	20000000
		R^2	0.009	0.304	0.602	0.741	0.019	0.411	0.573	0.451
		P	42.60	36.05	26.57	24.04	42.26	32.33	28.77	34.50
		E_s	6.546	6.281	5.245	5.175	6.394	5.784	5.498	8.952
Dry leaf	Caurie I	*b*	0.1271	0.1563	0.1700	0.1837	0.1397	0.1536	0.1528	0.1830
		m_o	0.8635	0.8714	0.8741	0.8435	0.8763	0.8474	0.8695	0.8591
		R^2	0.9543	0.9883	0.9809	0.9930	0.9827	0.9670	0.9770	0.9486
		P	10.34	7.654	13.41	7.977	7.506	10.51	9.019	21.09
		E_s	2.386	1.668	3.044	3.765	1.340	1.752	1.964	12.20
	Modified BET	m_o	0.599	0.614	0.618	0.743	0.602	0.654	0.598	0.774
		b	8402048	40000000	30000000	20000000	10000000	20000000	10000000	8571450
		R^2	-	0.407	0.536	0.801	0.194	0.460	0.339	0.779
		P	44.33	35.50	31.60	21.04	40.25	34.38	36.87	26.13
		E_s	7.705	5.707	5.528	4.105	6.235	5.562	6.346	6.706
Green leaf	Caurie I	*b*	0.1450	0.1607	0.1785	0.1753	0.1421	0.1648	0.1711	0.1888
		m_o	0.8663	0.8447	0.8401	0.8515	0.8910	0.8519	0.8679	0.8078
		R^2	0.9637	0.9867	0.9947	0.9903	0.9854	0.9861	0.9871	0.9744
		P	12.48	9.387	6.512	6.808	6.691	6.249	10.23	9.299
		E_s	2.468	2.344	2.418	2.300	1.535	1.625	2.231	7.867
	Modified BET	m_o	0.604	0.663	0.709	0.689	0.585	0.657	0.631	0.907
		b	20000000	40000000	20000000	8474309	9600464	30000000	10000000	30000000
		R^2	0.159	0.489	0.693	0.696	0.220	0.526	0.559	0.846
		P	39.51	33.96	26.96	25.72	40.16	32.54	30.60	25.85
		E_s	6.592	5.660	4.725	5.501	5.991	5.356	5.326	7.158

Note: m_o, *b, c* and *d* are constants.

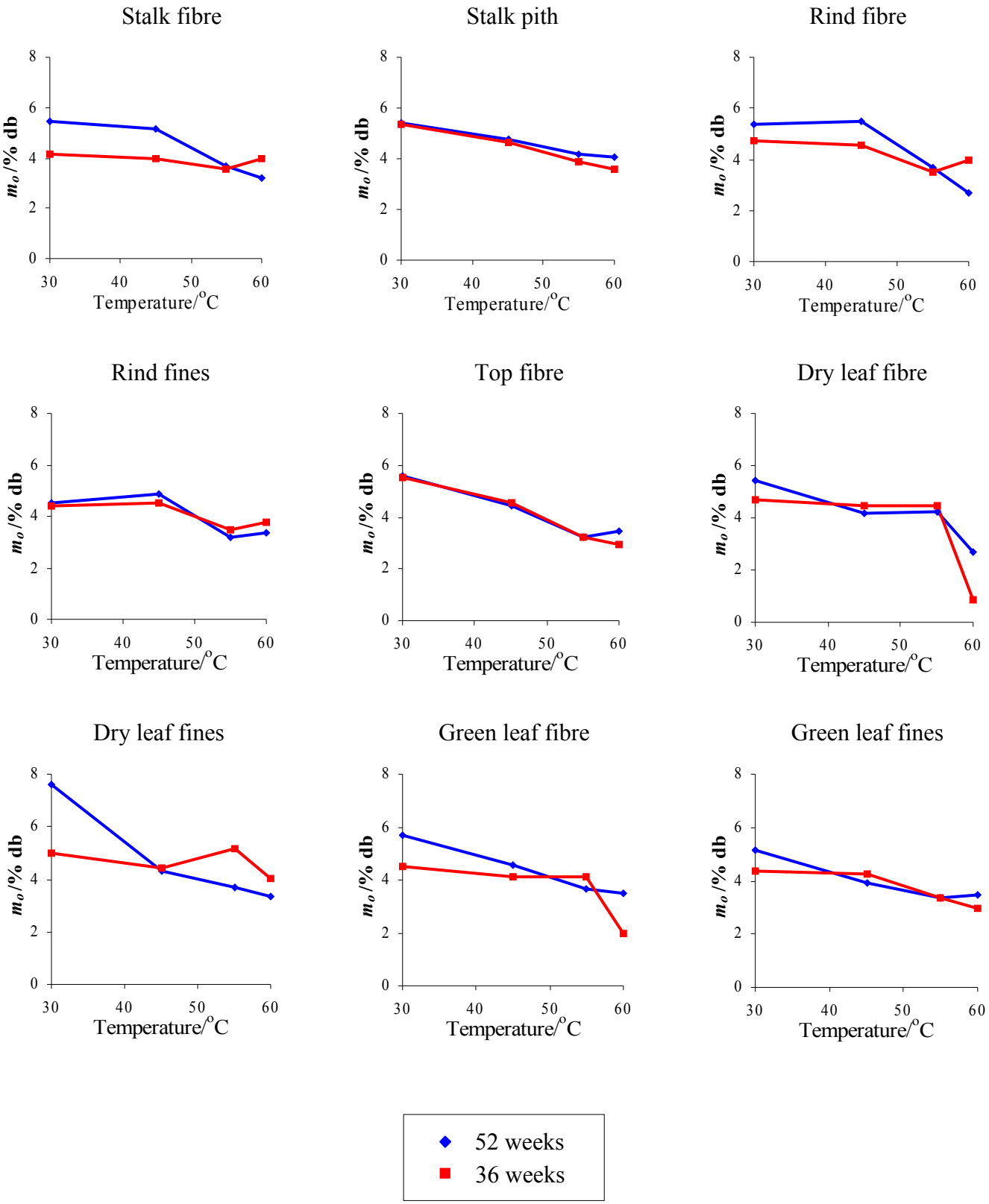


Figure 6.1. Variation of the GAB model monolayer moisture content with temperature for the nine cane components of R 570 aged 52 and 36 weeks.

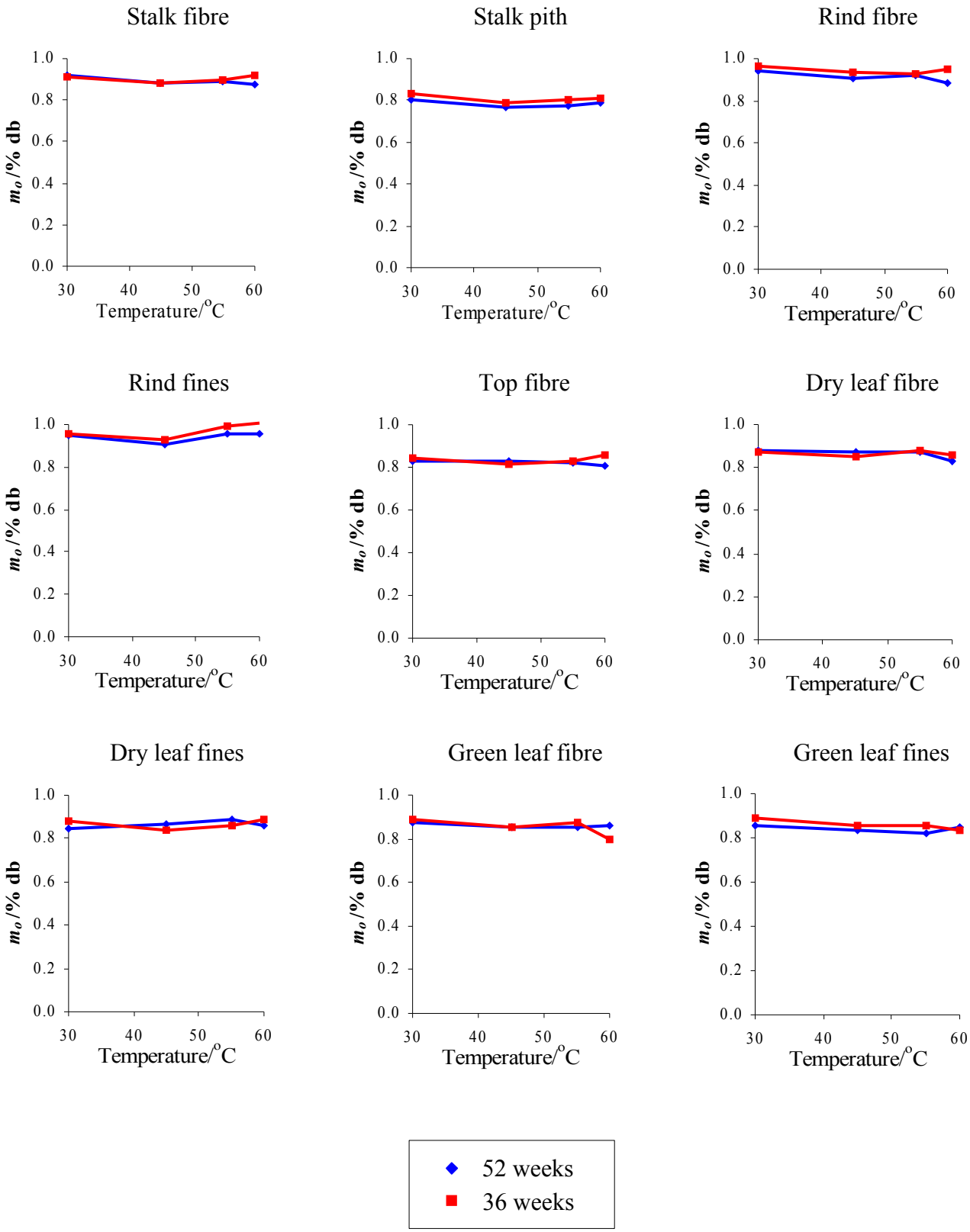


Figure 6.2. Variation of the Caurie I model monolayer moisture content with temperature for the nine cane components of R 570 aged 52 and 36 weeks.

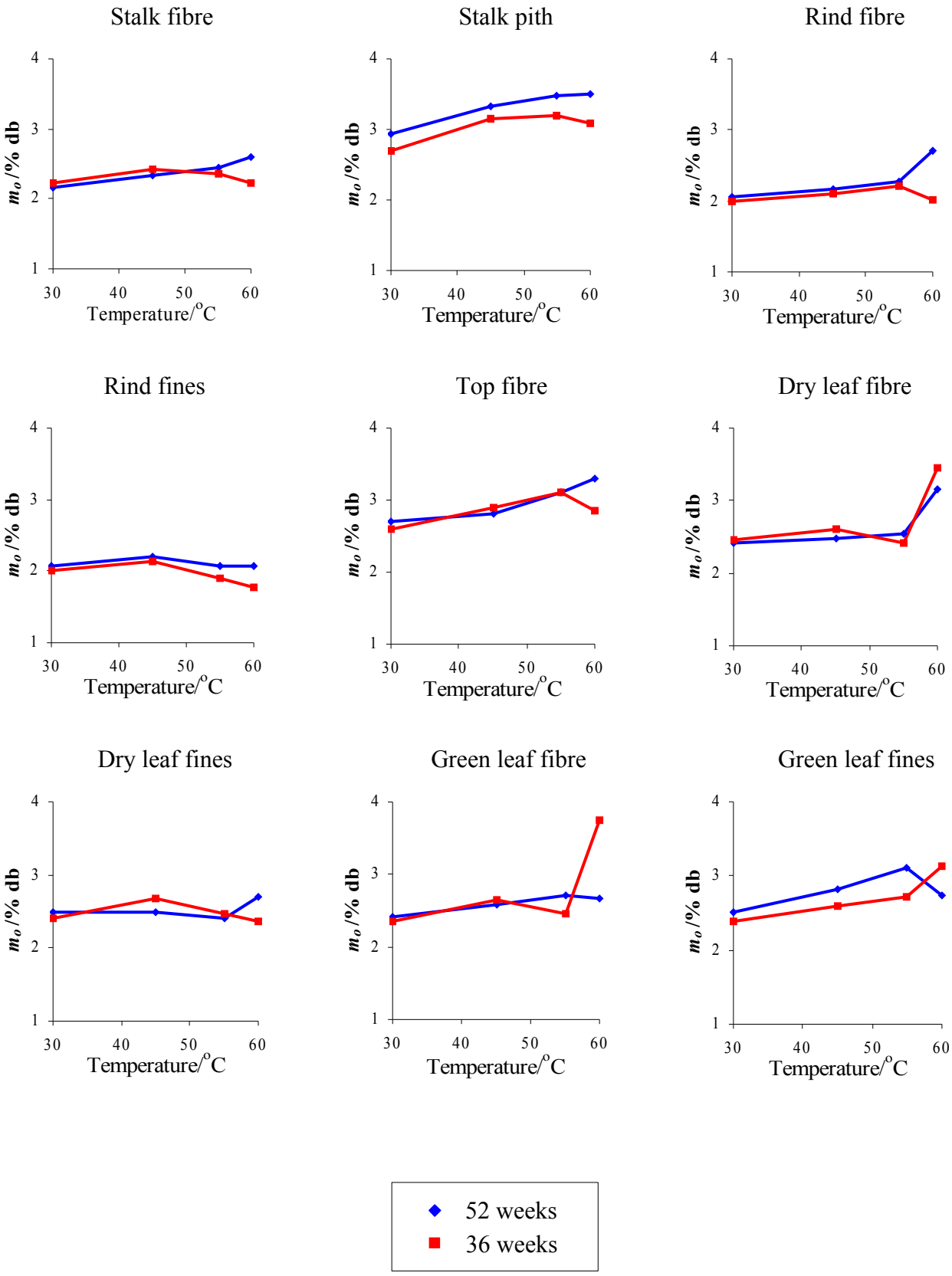


Figure 6.3. Variation of the modified BET model monolayer moisture content with temperature for the nine cane components of R 570 aged 52 and 36 weeks.

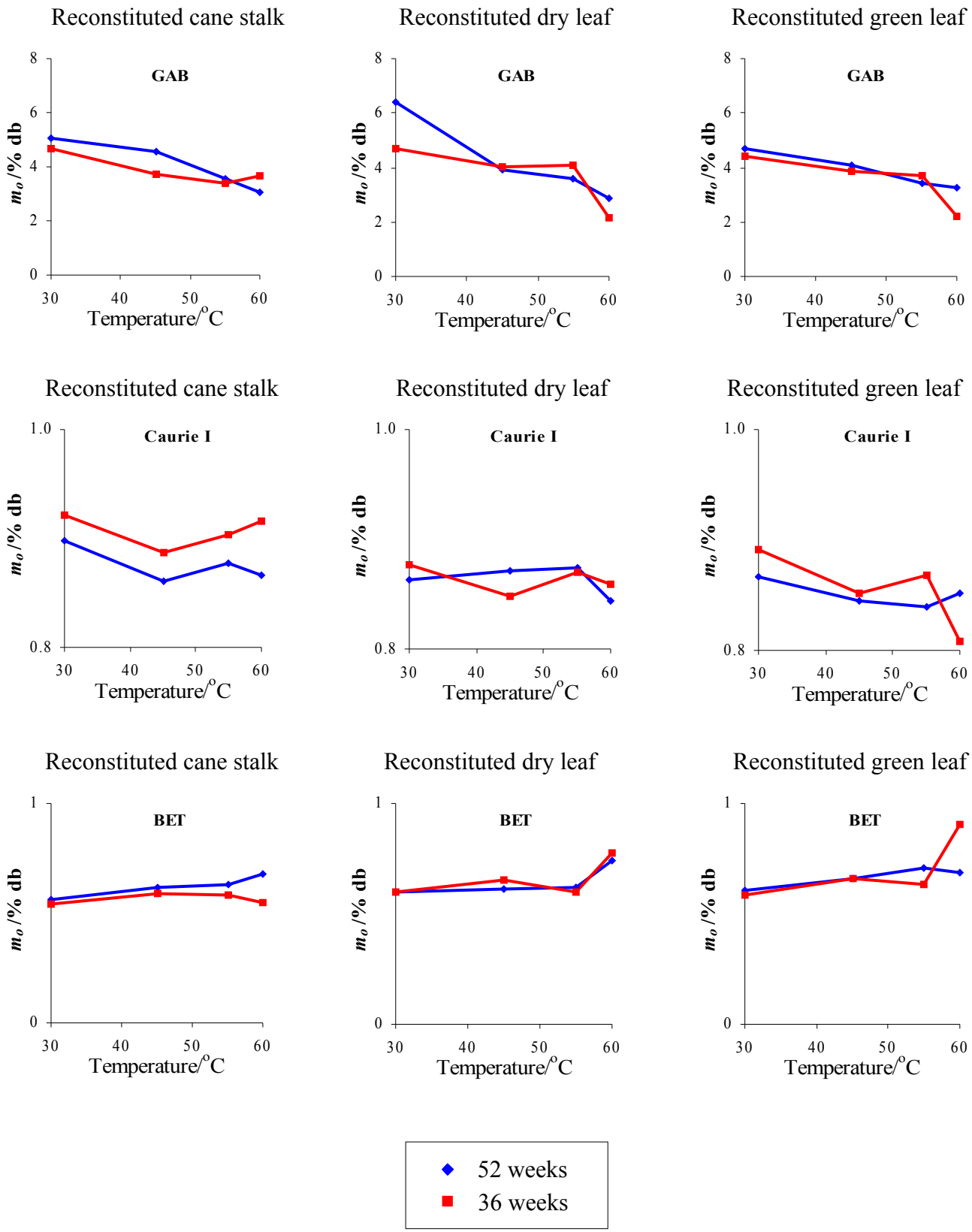


Figure 6.4. Variation of the GAB, Caurie I and modified BET models monolayer moisture content with temperature for the reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 and 36 weeks.

6.2 THE NUMBER OF ADSORBED MONOLAYERS N_o, THE DENSITY OF BOUND WATER AND THE PERCENTAGE OF BOUND OR NON-FREEZABLE WATER

A number of parameters can be derived from the Caurie I sorption model proposed by Caurie (1981). Although this model did not fit the experimental data best, it did give a passable fit to the data up to activity values of about 0.6. Hence it was used to give an indication of the magnitude of a number of properties of the sorbed water.

The number of adsorbed monolayers, N_o, can be obtained from $N_o = \frac{2}{s_c}$ where s_c is the slope of the Caurie I plot, and can be deduced to be $N_o = \frac{m_o}{b}$ since the slope of the plot is $\frac{2b}{m_o}$ (Rao *et al.*, 2006).

The density of the bound water is represented by the Caurie constant b as already mentioned in Section 6.1.

The percentage of bound water or non-freezable water is given by the product of the number of adsorbed monolayers N_o and the monolayer moisture content m_o, i.e. N_o x m_o.

The results of these calculations for the cane component parts of R 570 of two ages (including reconstituted cane stalk, dry leaf and green leaf) at various temperatures are shown in Table 6.2.

At 52 weeks, the number of monolayers N_o and the percentage of bound water decreased with increased temperature; samples at 36 weeks also showed this tendency but there were discrepancies, while the density of bound water increases with temperature at both ages, and did not differ much among the cane components. For cane components aged 52 weeks, the highest number of adsorbed monolayers was shown by dry leaf fibre at low temperature and by rind fines at high temperature. The highest bound water was exhibited by rind fibre and fines at low temperature and by rind fines at high temperature; this was true for these components of both ages.

Over the temperature range studied, the number of monolayers varies from about 7 to 4. This is similar to the number of hydration layers (5 or 6) estimated at the fibre saturation point in wood (Berry and Roderick, 2005).

Table 6.2. Properties of sorbed bound water and sorbent of cane components at different temperatures.

	Number of adsorbed monolayers, N_o				Density of bound water/g cm^{-3}				Bound or non-freezable water/%				Surface area/m^2 g $solids^{-1}$			
Temperature/°C	30	45	55	60	30	45	55	60	30	45	55	60	30	45	55	60
52 weeks																
Stalk fibre	6.55	5.55	4.89	4.76	0.14	0.16	0.18	0.18	6.02	4.90	4.34	4.17	192.3	183.5	130.3	114.1
Stalk pith	6.51	5.52	4.42	4.01	0.12	0.14	0.17	0.20	5.24	4.22	3.41	3.15	190.9	167.9	148.8	143.5
Rind fibre	6.70	5.51	4.66	4.48	0.14	0.16	0.20	0.20	6.30	5.00	4.30	3.97	189.9	195.2	131.1	95.3
Rind fines	6.45	5.57	5.06	5.08	0.15	0.16	0.19	0.19	6.10	5.06	4.85	4.85	161.2	172.8	113.3	118.7
Top fibre	6.34	5.24	4.76	4.29	0.13	0.16	0.17	0.19	5.25	4.33	3.91	3.47	198.4	157.3	114.8	121.8
Dry leaf fibre	6.87	5.48	4.99	4.51	0.13	0.16	0.17	0.18	6.03	4.79	4.34	3.75	191.6	147.7	150.2	94.9
Dry leaf fines	6.64	5.71	5.37	4.73	0.13	0.15	0.16	0.18	5.62	4.95	4.76	4.08	269.9	153.0	131.4	119.0
Green leaf fibre	5.94	5.31	4.77	5.00	0.15	0.16	0.18	0.17	5.17	4.52	4.06	4.29	202.2	160.8	130.3	124.7
Green leaf fines	6.08	5.16	4.63	4.83	0.14	0.16	0.18	0.18	5.21	4.31	3.80	4.11	182.4	139.6	119.4	122.6
Reconstituted cane stalk	6.57	5.55	4.69	4.48	0.14	0.16	0.19	0.19	5.90	4.78	4.11	3.88	178.9	162.2	125.7	108.7
Reconstituted dry leaf	6.79	5.57	5.14	4.59	0.13	0.16	0.17	0.18	5.87	4.86	4.49	3.87	227.5	139.3	126.8	100.8
Reconstituted green leaf	5.97	5.26	4.71	4.86	0.15	0.16	0.18	0.18	5.18	4.44	3.95	4.14	165.2	145.0	121.5	115.4
36 weeks																
Stalk fibre	6.05	5.09	5.22	4.79	0.15	0.17	0.17	0.19	5.53	4.50	4.69	4.41	147.7	141.3	125.4	141.7
Stalk pith	6.12	4.97	4.45	4.88	0.14	0.16	0.18	0.17	5.10	3.91	3.56	3.96	189.9	163.6	138.5	126.1
Rind fibre	7.13	5.19	5.22	5.24	0.14	0.18	0.18	0.18	6.86	4.84	4.84	4.98	166.8	161.5	124.3	141.3
Rind fines	6.41	5.58	5.27	6.04	0.15	0.17	0.19	0.17	6.13	5.18	5.23	6.08	155.5	160.1	122.9	133.5
Top fibre	5.97	5.23	4.56	4.76	0.14	0.16	0.18	0.18	5.03	4.28	3.78	4.07	195.2	161.2	114.8	104.1
Dry leaf fibre	6.10	5.71	5.79	4.47	0.14	0.15	0.15	0.19	5.33	4.87	5.07	3.83	165.8	158.3	158.3	31.2
Dry leaf fines	6.57	5.18	5.53	5.34	0.13	0.16	0.16	0.17	5.81	4.36	4.75	4.74	176.4	157.3	182.4	142.7
Green leaf fibre	6.24	5.12	5.06	4.19	0.14	0.17	0.17	0.19	5.56	4.36	4.42	3.35	160.5	144.9	144.9	70.5
Green leaf fines	6.36	5.26	5.12	4.55	0.14	0.16	0.17	0.18	5.67	4.51	4.40	3.78	154.8	150.9	117.9	104.1
Reconstituted cane stalk	6.56	5.20	5.00	5.11	0.14	0.17	0.18	0.18	6.04	4.61	4.51	4.68	165.4	132.8	120.1	129.6
Reconstituted dry leaf	6.27	5.52	5.69	4.69	0.14	0.15	0.15	0.18	5.50	4.67	4.95	4.03	165.4	143.6	145.0	76.7
Reconstituted green leaf	6.27	5.17	5.07	4.28	0.14	0.16	0.17	0.19	5.59	4.40	4.40	3.46	156.5	137.4	131.0	78.6

6.3 TOTAL SOLID SURFACE AREA AVAILABLE FOR HYDROPHILIC BINDING IN ADSORPTION

The solid surface area was determined from the monolayer moisture content by using the following equation (Mazza and Le Maguer, 1978):

$$A = m_o N_A A_m / m_w = 35.42\ m_o$$

where A is the solid surface area (m^2 g $solids^{-1}$), m_o is the monolayer moisture content (% db), m_w is the molar mass of water (18.02 g mol^{-1}), N_A is Avogadro's number (6.022 x 10^{23} molecules mol^{-1}), and A_m is the area of a water molecule (1.06 x 10^{-19} m^2 $molecule^{-1}$).

Since the GAB was found to be a good fit to the experimental adsorption data, the m_o value calculated from the GAB equation was used to estimate the total solid surface area A.

The results of the total solid surface area A calculated are also presented in Table 6.2. In general, A decreased with increased temperature. Stalk pith and dry leaf fines were among those components which had high surface area for hydrophilic binding.

Arslan and Toğrul (2005) found in their study of moisture adsorption of macaroni, the total available surface area for hydrophilic binding decreases with an increase in temperature. This is also true for most sugar cane component parts aged 52 and 36 weeks except for rind fibre and rind fines of both ages, top fibre, dry leaf fibre and green leaf fines aged 52 weeks and dry leaf fines aged 36 weeks.

The values found for the sugar cane fibres vary from 100 – 200 m^2 g $solids^{-1}$, which are much lower than the 200 – 300 m^2 g $solids^{-1}$ found for macaroni by Arslan and Toğrul (2005), and similarly for quinoa grains by Tolaba *et al.* (2004), but higher than in goat meat (Singh *et al.*, 2006), kheer (Kumar *et al.*, 2005) and chhana podo (Rao *et al.*, 2006), all about 80 m^2 g $solids^{-1}$.

As the temperature increased from 30 to 60 °C, the number of adsorbed monolayers, the percentage of bound water and the surface area of adsorbent decreased. This may be due to the reduction in sorption of water with increasing temperature in the low water activity range.

Noguchi (1981) reported that hydrophobic hydration of biopolymers melts off rapidly as the temperature increases.

6.4 HEATS OF SORPTION OF THE MONOLAYER AND MULTILAYER

In the GAB model, m_o is the monolayer moisture content, and b and c are constants related to monolayer and multilayer properties. The temperature dependence of the GAB constants b and c may be described with an Arrhenius form of equation (Sanni *et al.*, 1997):

$$b = b_o e^{[(H_1 - H_m)/RT]} \qquad (1)$$

$$c = c_o e^{[(H_L - H_m)/RT]} \qquad (2)$$

where b_o, c_o are adjusted constants for the temperature effect, H_1 and H_m (kJ mol^{-1}) are the heat of sorption of the monolayer and multilayer respectively, H_L (kJ mol^{-1}) is the heat of condensation of pure water vapour (43.53 kJ mol^{-1} at 35 °C), R is the universal gas constant (8.314 J mol^{-1} K^{-1}) and T is the Kelvin temperature (K).

The terms (H_1-H_m) and (H_L-H_m) represent the difference in enthalpy between monolayer and multilayer sorption and the difference between the heat of condensation of water and the heat of sorption of the multilayer respectively (van den Berg, 1984).

Arslan and Toğrul (2005) evaluated b_o, c_o, H_1 and H_m for the sorption isotherms of macaroni by using non-linear regression analysis after inserting b and c in equations (1) and (2) into the GAB equation.

An alternative way of solving for the constants b_o, c_o, H_1 and H_m is as follows:

From equation (1), $$ln\ b = ln\ b_o + \frac{(H_1 - H_m)}{R}\frac{1}{T}$$

and from equation (2), $$ln\ c = ln\ c_o + \frac{(H_L - H_m)}{R}\frac{1}{T}$$

By plotting $ln\ b$ against $\frac{1}{T}$, the intercept is $ln\ b_o$ and the slope is $\frac{(H_1 - H_m)}{R}$ and by similarly plotting $ln\ c$ against $\frac{1}{T}$, the intercept is $ln\ c_o$ and the slope is $\frac{(H_L - H_m)}{R}$.

The heat of vaporisation for the temperature range studied (30 to 60 °C) was taken as the value at 45 °C. This value was found by interpolation from data in Table 5.3 and was found to be 44.62 kJ mol^{-1}.

Thus, for each cane component of each age, the values of *ln b* and *ln c* found for the four temperatures were each plotted against 1/T, and the slopes and the intercepts of the two graphs provided solutions to the values of b_o, c_o, H_1 and H_m.

The data for reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 and 36 weeks were similarly treated. The results obtained are shown in Table 6.3.

The heat of sorption of multilayer H_m for each particular fibre is systematically higher at 52 weeks (48 – 49 kJ mol^{-1}) than at 36 weeks (46 – 47 kJ mol^{-1}), whereas the heat of sorption of monolayer H_1 is variable as is the constant b_o, the constant c_o is also systematically higher at 52 weeks than at 36 weeks for each particular fibre.

For values of b_o, c_o, $H_1 - H_m$ and $H_L - H_m$, Lopes Filho *et al.,* (2002) found for alligator's meat, 38980, 9.10, 5.42 kJ mol^{-1} and –5.96 kJ mol^{-1}, Arslan and Toğrul (2005) found for macaroni, values of 0.026, 0.617, 13.50 kJ mol^{-1} and 0.752 kJ mol^{-1}, and Al-Muhtaseb *et al*. (2004a) reported for amylopectin powder 0.014, 0.716, 16.9 kJ mol^{-1} and 617 kJ mol^{-1}. None of these authors reported individual values of H_1, H_L and H_m.

Table 6.3. Heat of sorption of the monolayer H_1(kJ mol^{-1}) and multilayer H_m(kJ mol^{-1}) for the nine cane components and reconstituted cane stalk and leaves of R 570 of two ages.

Sample	52 weeks				36 weeks			
	b_o	c_o	H_1	H_m	b_o	c_o	H_1	H_m
Stalk fibre	0.4418	4.518	57.42	49.07	4.771×10^{-6}	1.236	84.74	45.56
Stalk pith	1.73×10^{-19}	2.263	172.2	47.09	3.309×10^{9}	2.602	-1.969	47.48
Rind fibre	0.169653	5.963	60.17	49.83	2.395×10^{-9}	1.982	106.8	46.92
Rind fines	248.1	2.172	39.58	47.13	0.0486	1.150	60.17	45.48
Top fibre	0.0252	3.487	65.58	48.26	1609.2	3.557	35.72	48.31
Dry leaf fibre	0.1485	3.732	61.15	48.49	2864773.9	3.249	17.56	48.08
Dry leaf fines	38832.003	7.859	29.92	50.56	3.52×10^{-6}	1.284	85.44	45.72
Green leaf fibre	293386086	3.369	3.782	48.24	0.000331	1.284	74.52	45.72
Green leaf fines	1.688	2.714	53.58	47.59	2.216×10^{-5}	2.569	84.25	47.43
Reconstituted cane	54.10	3.411	45.88	48.26	0.0111	1.949	67.21	46.78
Reconstituted dry leaf	8.28×10^{72}	6.089	-366.7	49.81	8.87×10^{57}	2.598	-283.4	47.49
Reconstituted green leaf	4.512×10^{98}	2.394	-548.9	47.26	2.692×10^{39}	2.958	-175.8	47.80

b_o and c_o are constants.

6.5 THE NET ISOSTERIC HEAT OF SORPTION q_{st}, THE TOTAL ISOSTERIC HEAT OF SORPTION Q_{st} AND THE ENTROPY OF SORPTION S_d

The net isosteric heat of sorption q_{st} is defined as the total heat of sorption Q_{st} in the food or fibre minus the latent heat of vaporisation of water H_L, at the system temperature (Tsami *et al.*, 1990). Conventionally, q_{st} is a positive quantity when heat is evolved during adsorption and negative when heat is absorbed during desorption. It is indicative of the intermolecular attractive forces between the sorption sites and water vapour (Wang and Brennan, 1991).

The values of the net isosteric heat of sorption q_{st} are obtained from the slopes of $ln\ a_w$ versus 1/T plots by linear regression analysis with the assumption that they are constant over the temperature range studied. According to the differential form of the Clausius Clapeyron equation (Labuza, 1984):

$$\left[\frac{d(ln\ a_w)}{d(1/\mathrm{T})}\right]_m = -\frac{q_{st}}{\mathrm{R}} \qquad (3)$$

where a_w is the water activity, T is the Kelvin temperature (K), m is the equilibrium moisture content, q_{st} is the net isosteric heat of sorption (kJ mol^{-1}), and R is the universal gas constant (8.314 J mol^{-1} K^{-1}).

Since equation (3) holds only for constant moisture content, values of the water activity a_w at specified moisture contents need to be evaluated from the best-fit isotherm equation (Hailwood-Horrobin model in this case). In this study the water activity at 18 fixed moisture values (namely 0.01, 0.05, 0.1, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20, 25, 30 and 35 g/g db) were calculated. In order to do this, the Hailwood-Horrobin equation:

$$\frac{a_w}{m} = b + c\,a_w + d(a_w)^2$$

was written in the form of a quadratic equation:

$$d\,a_w^{\,2} + (c - \frac{1}{m})a_w + b = 0$$

and solved for a_w at the chosen values of m.

Only the positive values of a_w are taken. This was effected at four temperatures (i.e. 30, 45, 55 and 60 °C) for the same chosen values of the moisture content. For each moisture content, $ln\ a_w$ calculated for the four temperatures are plotted as ordinate against 1/T as

abscissa. For each moisture level (18 in all), a straight line was obtained, the slope of which equals $-q_{st}/R$, from which the net isosteric heat of sorption q_{st} could be calculated at that particular moisture level (Wang and Brennan, 1991). This procedure was repeated for each of the nine cane components of R 570 aged 52 and 36 weeks.

This approach assumes that q_{st} is independent of temperature; although this is not always true, it has been accepted as such (Iglesias *et al.*, 1989). The application of this method requires the measurement of sorption isotherms, at no less than three temperatures.

The total isosteric heat of sorption Q_{st}, is a measure of the interaction (binding energy) between water vapour and the adsorbent material; in this work, sugar cane fibre. It is the sum of the net isosteric heat of sorption q_{st} and the latent heat of vaporisation of pure water, H_L:

$$Q_{st} = q_{st} + H_L$$

The latent heat of vaporisation of pure water H_L at various temperatures can be interpolated from Table 5.3. Thus, H_L values at 30, 45, 55 and 60 °C are 43.47, 44.53, 46.03 and 47.01 kJ mol^{-1} respectively, but the average value of H_L for the temperature range may be used (Sanchez *et al.*, 1997), and can be taken as constant at 43 kJ mol^{-1}.

The relationship between the total isosteric heat of sorption Q_{st} and the entropy of sorption S_d is given by (Aguerre *et al.*,1986):

$$- ln\ a_w = \frac{Q_{st}}{RT} - \frac{S_d}{R} \qquad (4)$$

where a_w is the water activity, Q_{st} is the total isosteric heat of sorption (kJ mol^{-1}), R is the universal gas constant (J mol^{-1} K^{-1}), T is the Kelvin temperature (K) and S_d is the entropy of sorption (J mol^{-1} K^{-1}).

The entropy of sorption S_d is proportional to the number of available sorption sites at a specific energy level (Madamba *et al.*, 1996).

If $ln\ a_w$ is plotted against 1/T, for a constant moisture content, the intercept yields S_d/R.

An example of a Clausius Clapeyron plot for stalk fibre aged 52 weeks is shown in Fig 6.5. The calculated net isosteric heat of sorption q_{st} is plotted against moisture level for all nine cane components of R 570 aged 52 and 36 weeks in Fig 6.6, and the corresponding entropy of sorption is shown in Fig 6.7.

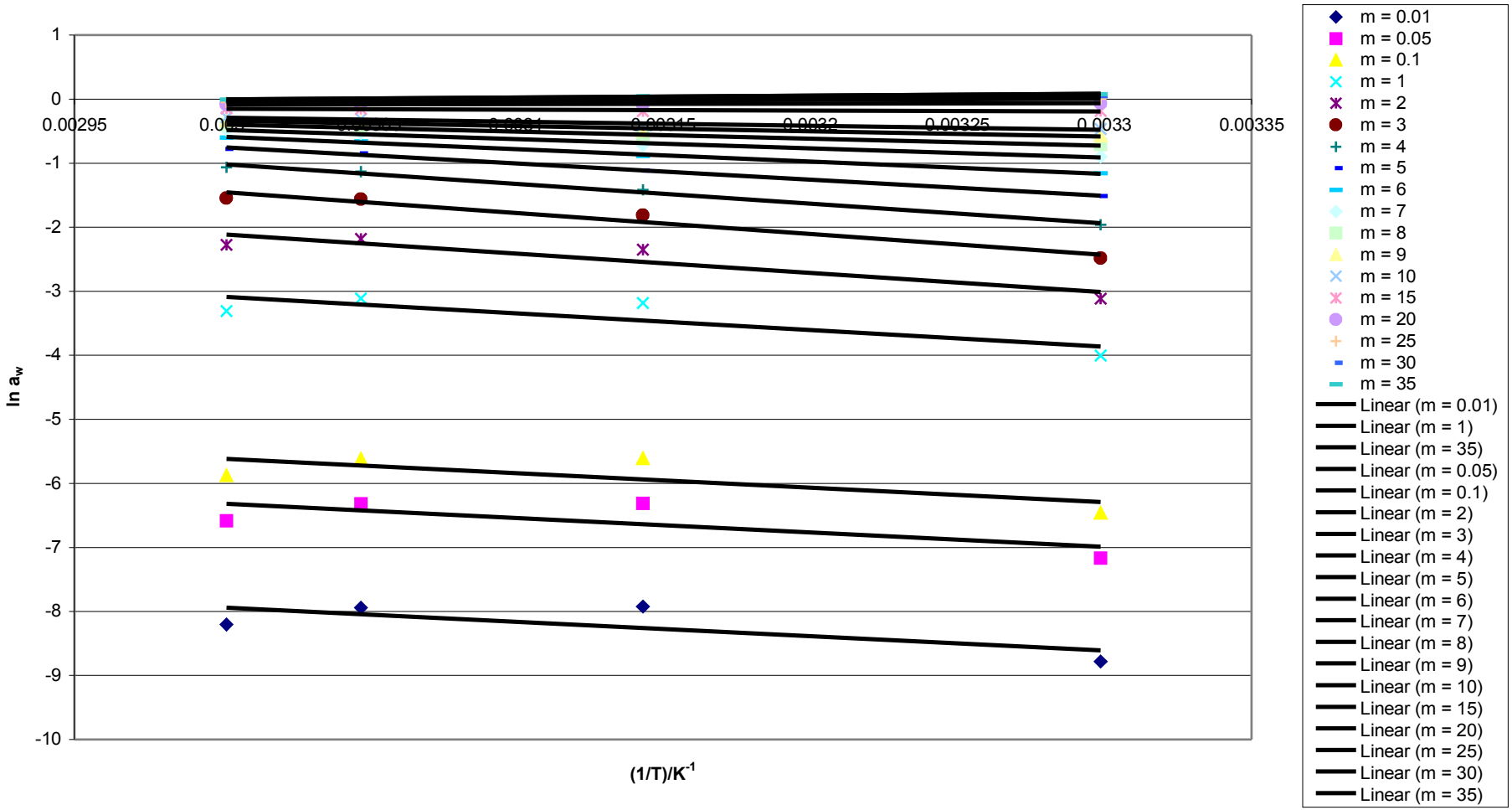


Figure 6.5. Typical Clausius Clapeyron plot for stalk fibre aged 52 weeks.

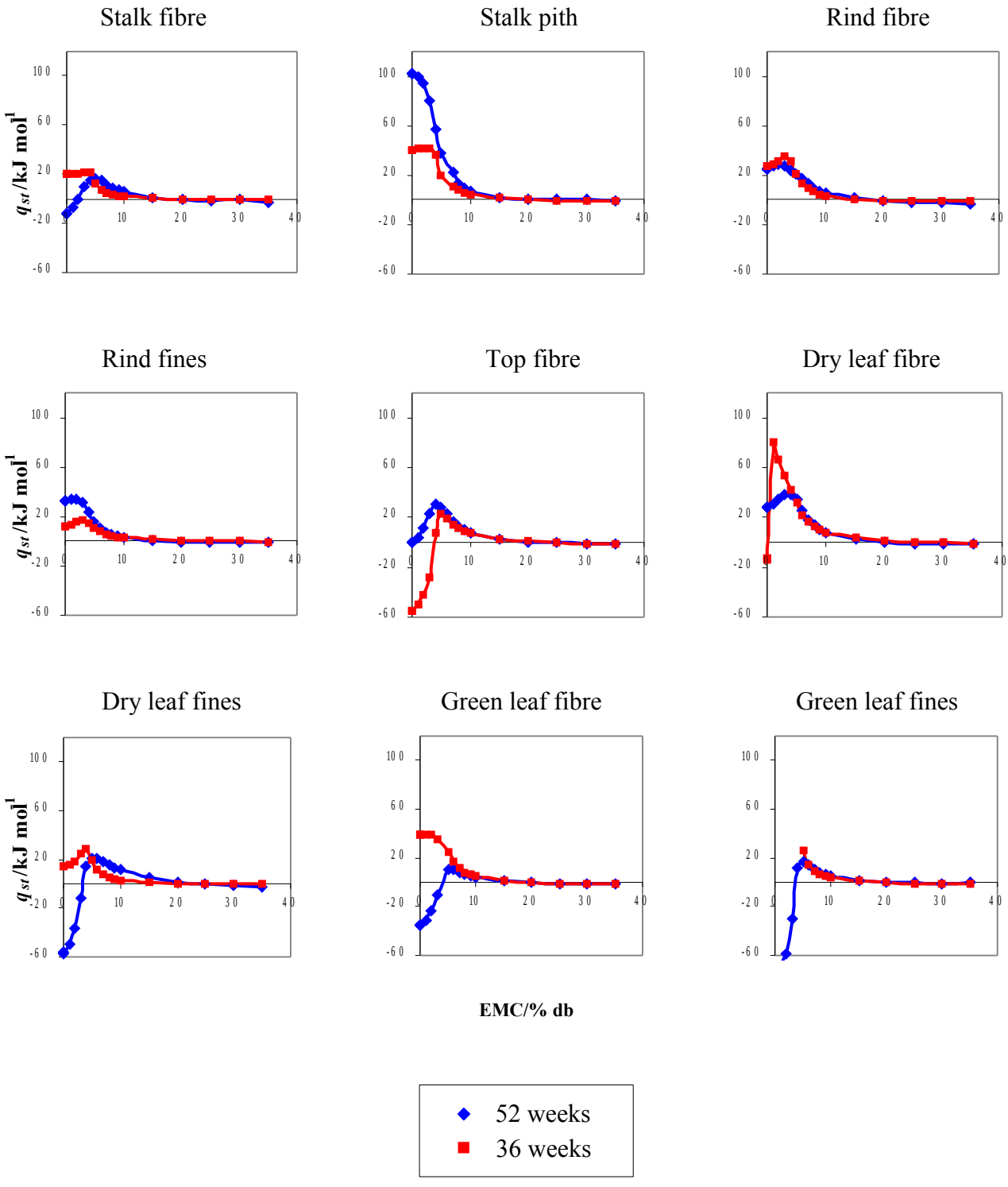


Figure 6.6. The variation of the net isosteric heat of adsorption calculated from the Hailwood-Horrobin model with moisture content for the nine cane components of R 570 aged 52 and 36 weeks.

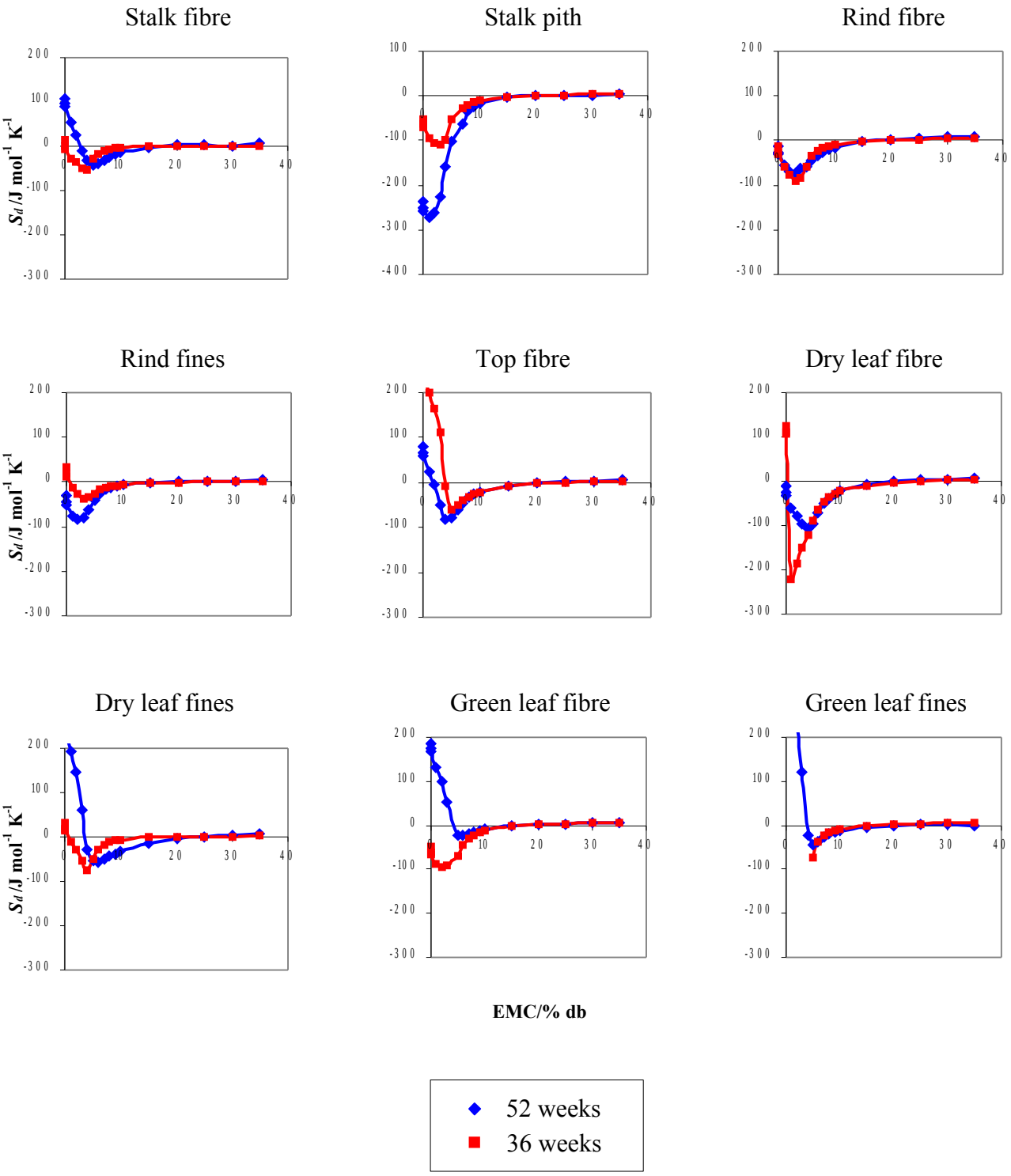


Figure 6.7. The variation of the entropy of sorption calculated from the Hailwood-Horrobin model with moisture content for the nine cane components of R 570 aged 52 and 36 weeks.

A similar procedure was followed to calculate the net isosteric heat of adsorption and the sorption entropy from the GAB model.

The GAB net isosteric heat of sorption q_{st} is plotted against moisture level for all nine cane components of R 570 aged 52 and 36 weeks in Fig 6.8, and corresponding entropy of sorption in Fig 6.9.

From Figs 6.6 and 6.8, it can be seen that q_{st} values for all cane components are large at low EMC, they reach a maximum between 0 – 5% EMC and then decrease sharply reaching a plateau at about a moisture content of 15%. The rapid increase in the net isosteric heat of sorption q_{st} at low moisture content is due to the existence of highly active polar sites on the surface of the sugar cane components, which are covered with water molecules forming a monomolecular layer (Hossain *et al.*, 2001). As the moisture content increases, the available sites for sorption of water decrease, resulting in lower values of the isosteric heats. The net isosteric heat of sorption approaches the latent heat of vaporisation of pure water (H_L) at the moisture content of 15% (on a dry basis). At moisture contents greater than 15% (on a dry basis), there is no significant difference between the latent heat of vaporisation of pure water and the net isosteric heat of sorption. Iglesias and Chirife (1976) explained that at this point, the existence of water in free form in the product is indicated. This moisture level has been termed the 'free water point', and seems to correspond numerically to the Brix-free water value determined in Chapter 4.

The maximum net heat of sorption and entropy of sorption for nine cane components aged 52 and 36 weeks as calculated by the GAB model are shown in Table 6.4.

Stalk pith of 52 weeks showed the highest net isosteric heat of sorption q_{st} value calculated from the GAB model (Table 6.4 and Fig 6.8), as well as from the Hailwood-Horrobin model (Fig 6.6); inversely, it had the lowest entropy of sorption S_d calculated from the GAB model (Table 6.4 and Fig 6.9), as well as from the Hailwood-Horrobin model (Fig 6.7).

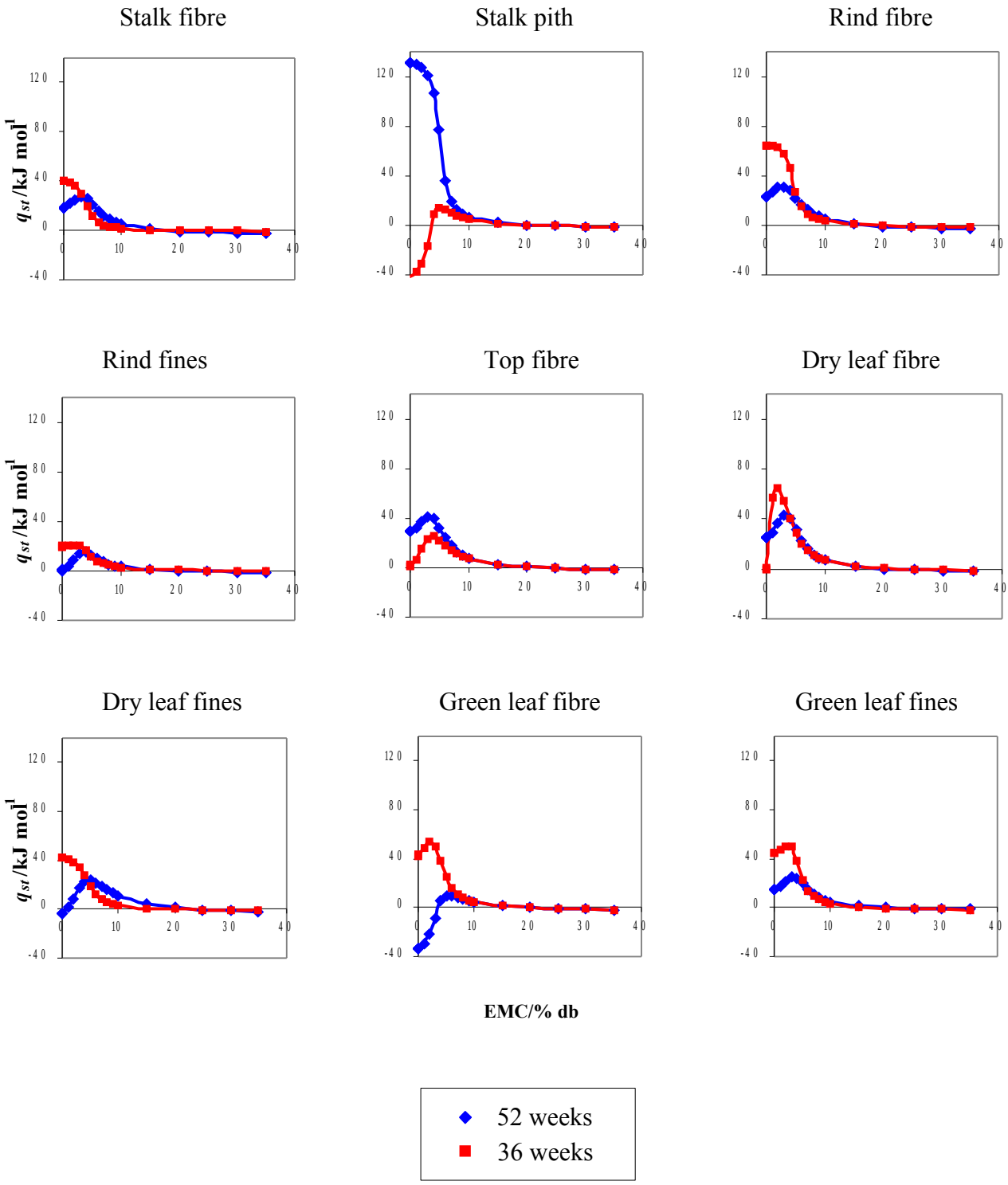


Figure 6.8. The variation of the GAB model net isosteric heat of adsorption with moisture content for the nine cane components of R 570 aged 52 and 36 weeks.

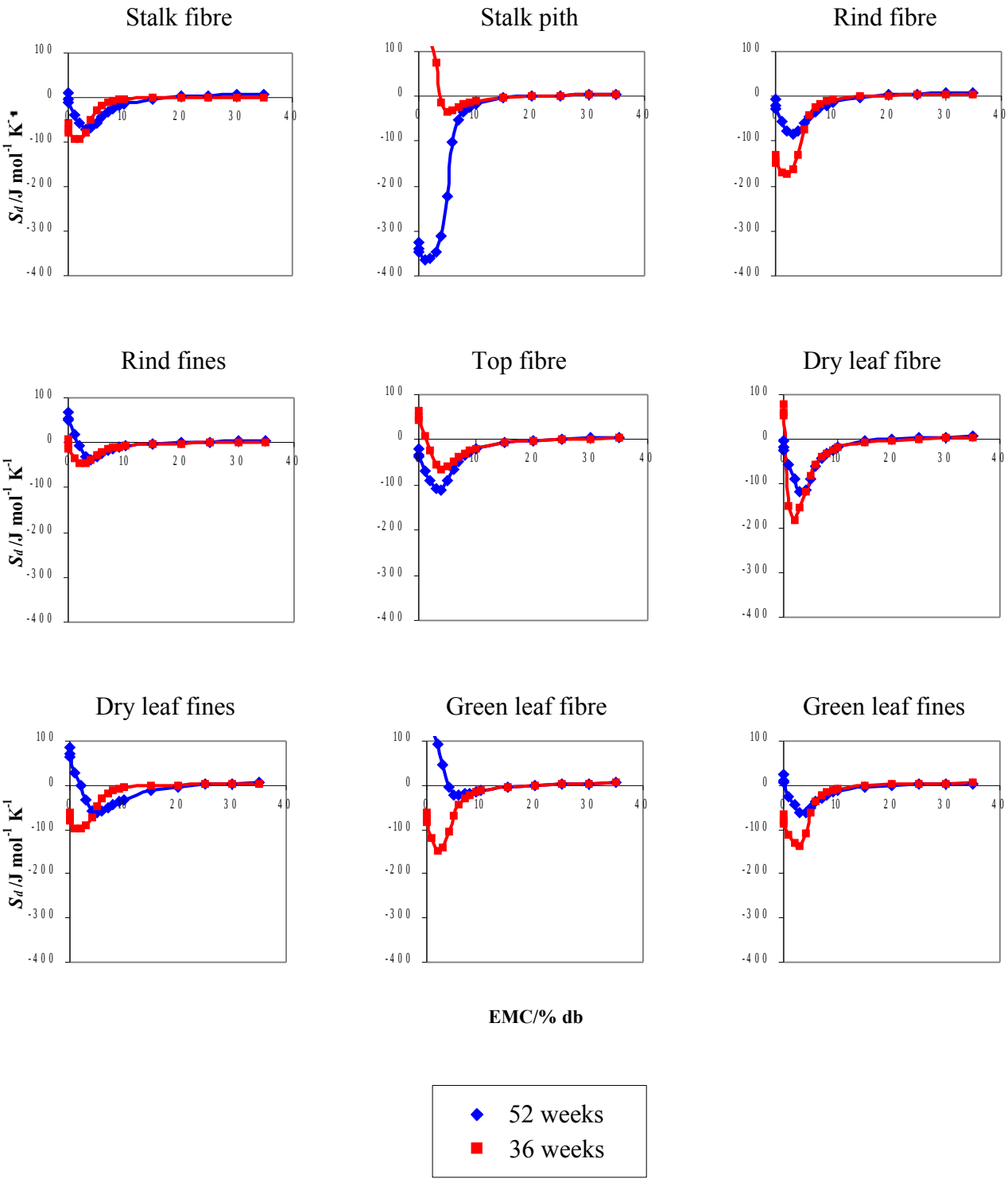


Figure 6.9. The variation of the GAB model entropy of sorption with moisture content for the nine cane components of R 570 aged 52 and 36 weeks.

Table 6.4. The GAB model maximum net isosteric heat of sorption, q_{st}, and entropy of sorption, S_d, and the corresponding EMC/% db for the nine cane components aged 52 and 36 weeks.

Sample	Maximum q_{st}/kJ mol^{-1}				Maximum S_d/kJ mol^{-1}			
	52 weeks	EMC/% db	36 weeks	EMC/% db	52 weeks	EMC/% db	36 weeks	EMC/% db
Stalk fibre	27.27	3	40.88	0	-69.77	3	-93.87	1
Stalk pith	130.77	0.01	14.08	5	-363.07	1	-33.51	5
Rind fibre	31.30	3	64.28	0.1	-83.93	3	-173.40	2
Rind fines	15.89	4	20.92	2	-38.49	4	-46.95	3
Top fibre	40.81	3	25.17	4	-109.80	4	-66.96	4
Dry leaf fibre	43.24	3	64.82	2	-117.87	3	-182.59	2
Dry leaf fines	23.59	5	41.95	0.01	-63.55	5	-98.93	2
Green leaf fibre	9.75	5	53.92	2	-22.97	6	-146.76	2
Green leaf fines	24.70	3	49.67	2	.62.47	4	-136.16	3

6.6 ENTHALPY-ENTROPY COMPENSATION

A promising theory that has been widely considered to investigate the physical and chemical phenomena involved in water sorption (Aguerre *et al.*, 1986, Madamba *et al.*, 1996) is the enthalpy-entropy compensation theory, or isokinetic relationship, as originally applied by Bell (1937). This theory states that compensation arises from changes in the nature of the interaction between the solute and the solvent causing the reaction, and that the relationship between enthalpy and entropy for a specific reaction is linear. When this theory is applied to a sorption process, the enthalpy corresponds to the net isosteric heat of sorption.

For a linear enthalpy and entropy relation, the isokinetic temperature (T_β) can be determined from the slope of the line and, if the theory is valid, should be constant at any point (Heyrovsky, 1970). It represents the temperature at which all the reactions in the series proceed at the same rate (Heyrovsky, 1970), and the free energy at T_β, ΔG, provides a criterion to evaluate whether the water sorption is a spontaneous ($-\Delta G$) or a non-spontaneous process ($+\Delta G$).

To test the validity of the compensation theory, the isokinetic temperature is compared with the harmonic mean temperature (T_{hm}), and $T_\beta \neq T_{hm}$. T_{hm} is defined as:

$$T_{hm} = \frac{n_i}{\sum_{i=1}^{n_i} 1/T}$$

where n_i is the total number of isotherms and, T is the Kelvin temperature (K).

Moreover, if $T_\beta > T_{hm}$ the process is enthalpy-driven, and if $T_\beta < T_{hm}$, the process is considered to be entropy-controlled (Telis *et al.*, 2000).

If the net isosteric heat of sorption q_{st} is plotted as ordinate against the entropy of sorption S_d as abscissa, the slope of the plot gives the isokinetic temperature T_β and the intercept gives the free energy ΔG at the isokinetic temperature. The harmonic mean temperature for this study can be calculated to be 320.23 K, since the isotherm was investigated at four different temperatures of 30, 45, 55 and 60 °C.

$$T_{hm} = \frac{4}{(303.15)^{-1} + (318.15)^{-1} + (328.15)^{-1} + (333.15)^{-1}}$$

$$= 320.23 \text{ K}$$

The plots of the net heat of sorption q_{st} (kJ mol^{-1}) against the entropy of sorption S_d (kJ mol^{-1} K^{-1}) for the nine cane components of R 570 aged 52 and 36 weeks are shown in Fig 6.10, the coefficient of determination R^2, the slope and the intercept for the enthalpy-entropy relationship are shown in Table 6.5. The slope corresponds to the isokinetic temperature T_β, and the intercept, ΔG.

It can be seen from Table 6.5 that T_β for all cane components aged 52 and 36 weeks is greater than the harmonic mean temperature T_{hm} of 320.23 K except in the case of stalk pith aged 36 weeks, hence, all processes except stalk pith aged 36 weeks are enthalpy-controlled. Hence the driving force for the adsorption of moisture on these fibres is the strength of binding of water molecules to the surface of the fibre. The positive sign of ΔG (except for the case of rind fines aged 36 weeks) indicates that the water sorption process in the cane components is non-spontaneous, and that the enthalpy-entropy compensation theory was satisfied.

Beristain *et al.* (1996) applied the enthalpy-entropy compensation theory to water adsorption in starchy materials. Two isokinetic temperatures were observed, suggesting that during the initial stages (at low water activity) the isotherm process was entropy-controlled, whereas in the later stage, the process was controlled by changes in the enthalpy of water. They also reported a spontaneous sorption isotherm for starch materials.

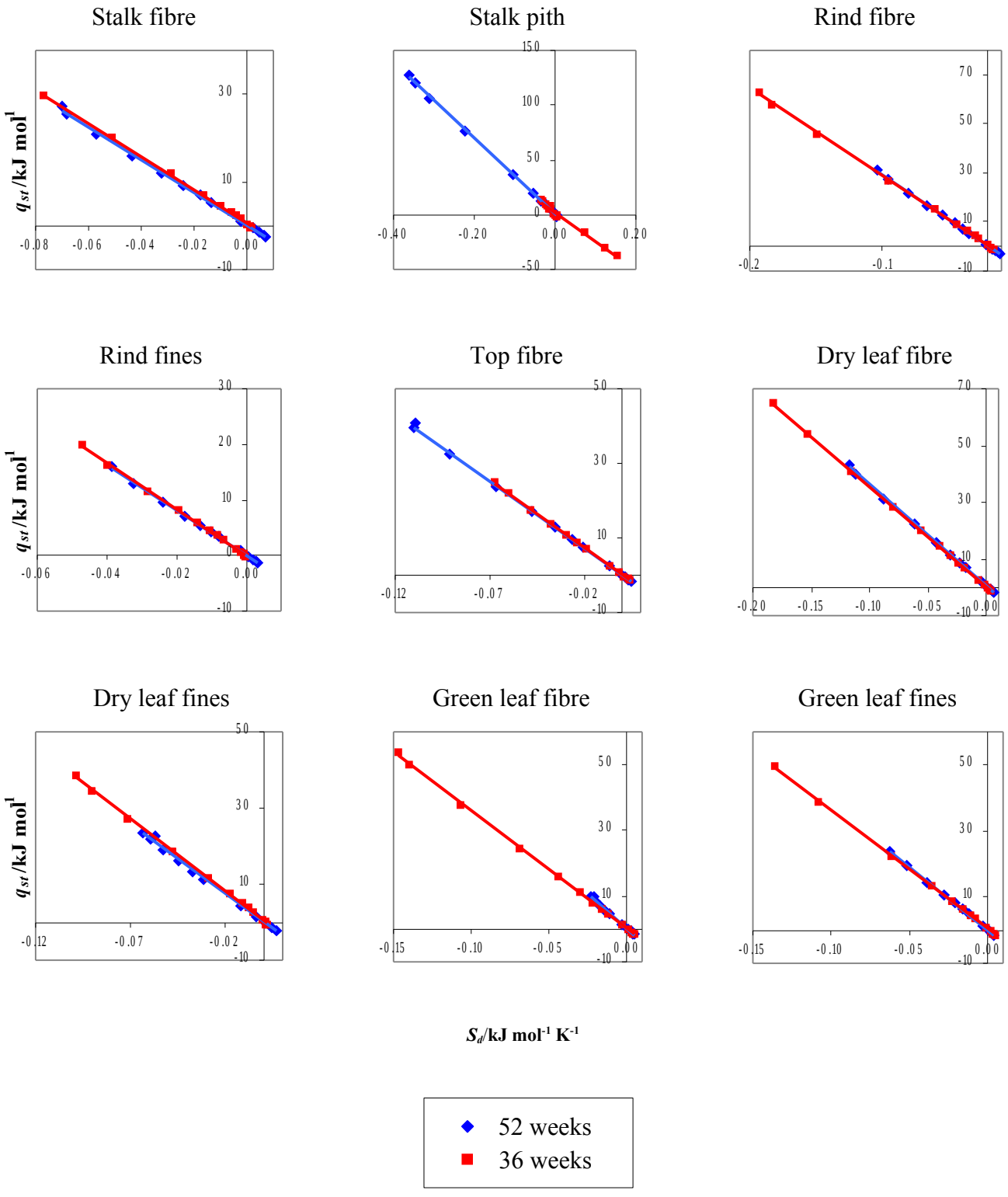


Figure 6.10. Net isosteric heat-entropy relationship for adsorption in the nine cane components of R 570 aged 52 and 36 weeks.

Table 6.5. Characteristic parameters obtained from the net isosteric heat-entropy relationship for the nine cane components of R 570 aged 52 and 36 weeks.

Sample	52 weeks				36 weeks			
	R^2	Slope	Intercept= ΔG/kJ mol^{-1}	T_β/K	R^2	Slope	Intercept= ΔG/kJ mol^{-1}	T_β/K
Stalk fibre	0.999	-373.80	0.2420	373.80	0.999	-380.96	0.5139	380.96
Stalk pith	1.00	-345.02	0.5352	345.02	0.985	-265.29	2.2753	265.29
Rind fibre	1.00	-363.57	0.2431	363.57	1.00	-355.28	0.3731	355.28
Rind fines	1.00	-401.55	0.1676	401.55	0.999	-419.85	-0.0243	419.85
Top fibre	1.00	-360.67	0.1874	360.67	1.00	-366.76	0.1579	366.76
Dry leaf fibre	1.00	-357.92	0.2092	357.92	1.00	-352.27	0.1640	352.27
Dry leaf fines	0.997	-367.35	0.0465	367.35	0.999	-380.49	0.4431	380.49
Green leaf fibre	0.995	-406.66	0.2946	406.66	1.00	-358.37	0.2794	358.37
Green leaf fines	1.00	-374.95	0.2617	374.95	1.00	.358.24	0.4324	358.24

6.7 PRIMARY, SECONDARY AND TERTIARY BOUND WATER

Kaleemullah and Kailappan (2007) determined the equilibrium moisture content of red chillies at various water activities within the temperature range of 25 – 45 °C, and obtained binding energies of three different types of water bound to the surface of the material studied.

They calculated the heat of sorption of the primary adsorbed layer H_1 by using the BET model which described the sorption behaviour of red chillies well. They then calculated the net isosteric heat of sorption q_{st}, and multiplied it by the Universal gas constant R (8.3143 J mol^{-1} K^{-1}) to obtain, what they called, the binding energy, which was then plotted against the moisture content. From this plot they determined the moisture content of the secondary layer from where the break in the curve occurred and they marked the end of the tertiary layer where the binding energy became zero. For the primary layer, the value of the binding energy H_1 was read off from the y-axis, a horizontal line was placed and from where it intersected the curve determined the corresponding moisture content was deemed to be the primary layer.

In a similar manner, calculations were carried out for cane components aged 52 and 36 weeks, and the results are shown in Figs 6.11 and 6.12. The moisture content of the primary, secondary and tertiary bound water layers were compared to the Brix-free water

value extracted from Tables 4.17 – 4.21 and corrected for residual moisture content. The H_1 values were extracted from Table 6.3. The results are shown in Table 6.6.

Table 6.6. Comparison of the calculated bound water (primary, secondary and tertiary) with the Brix-free water values of the nine cane components of R 570 of two ages.

Sample	52 weeks				36 weeks			
	Bound water at moisture content/%			Brix-free water/%	Bound water at moisture content/%			Brix-free water/%
	Primary	Secondary	Tertiary		Primary	Secondary	Tertiary	
Stalk fibre	8	10	22	12.37	6	9	20	14.37
Stalk pith	10	11	20	25.05	-	14	22	22.48
Rind fibre	9	10	21	12.02	7	10	21	13.65
Rind fines	8	10	22	11.47	7	13	30	15.58
Top fibre	10	13	23	16.76	12	14	25	15.82
Dry leaf fibre	10	13	20	16.13	13	14	25	15.70
Dry leaf fines	14	15	26	18.17	8	10	25	17.64
Green leaf fibre	-	15	21	13.77	8	10	25	13.63
Green leaf fines	9	10	25	14.79	7	9	20	16.20

It can be seen from Table 6.6 that the Brix-free water values of sugar cane component parts correspond most closely to the secondary bound water, except those of stalk pith with inherent high surface area, which indicate the similarity to the tertiary bound water, or the free water point, reported by some research workers (Kaleemullah and Kailappan, 2007; Arslan and Toğrul, 2005). Kaleemullah and Kailappan (2007) found the primary, secondary and tertiary bound waters of red chillies end at moisture contents of 1.5, 14 and 53.6% db respectively.

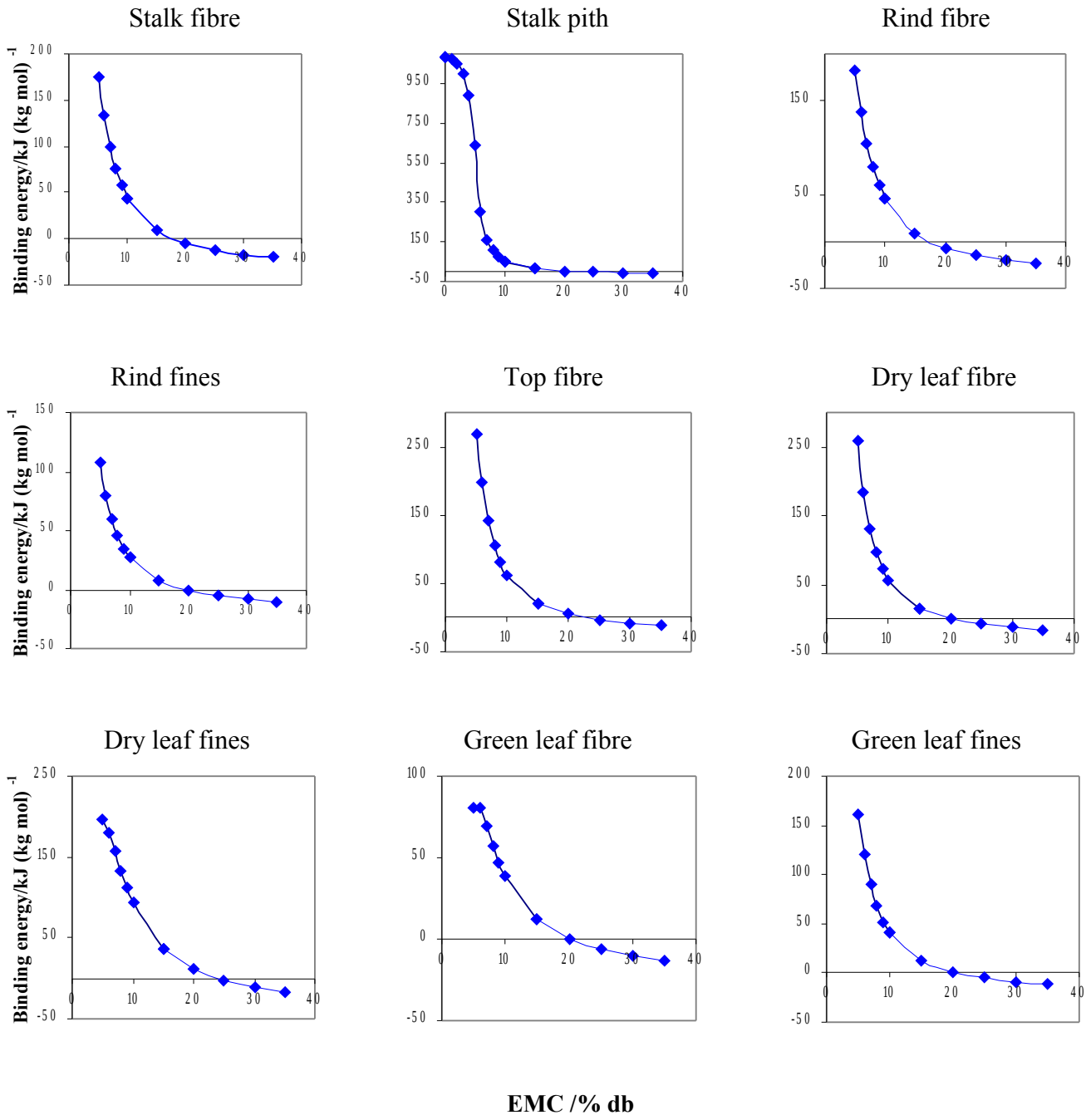


Figure 6.11. Average energy of water binding by sugar cane component parts of cane variety R 570 aged 52 weeks.

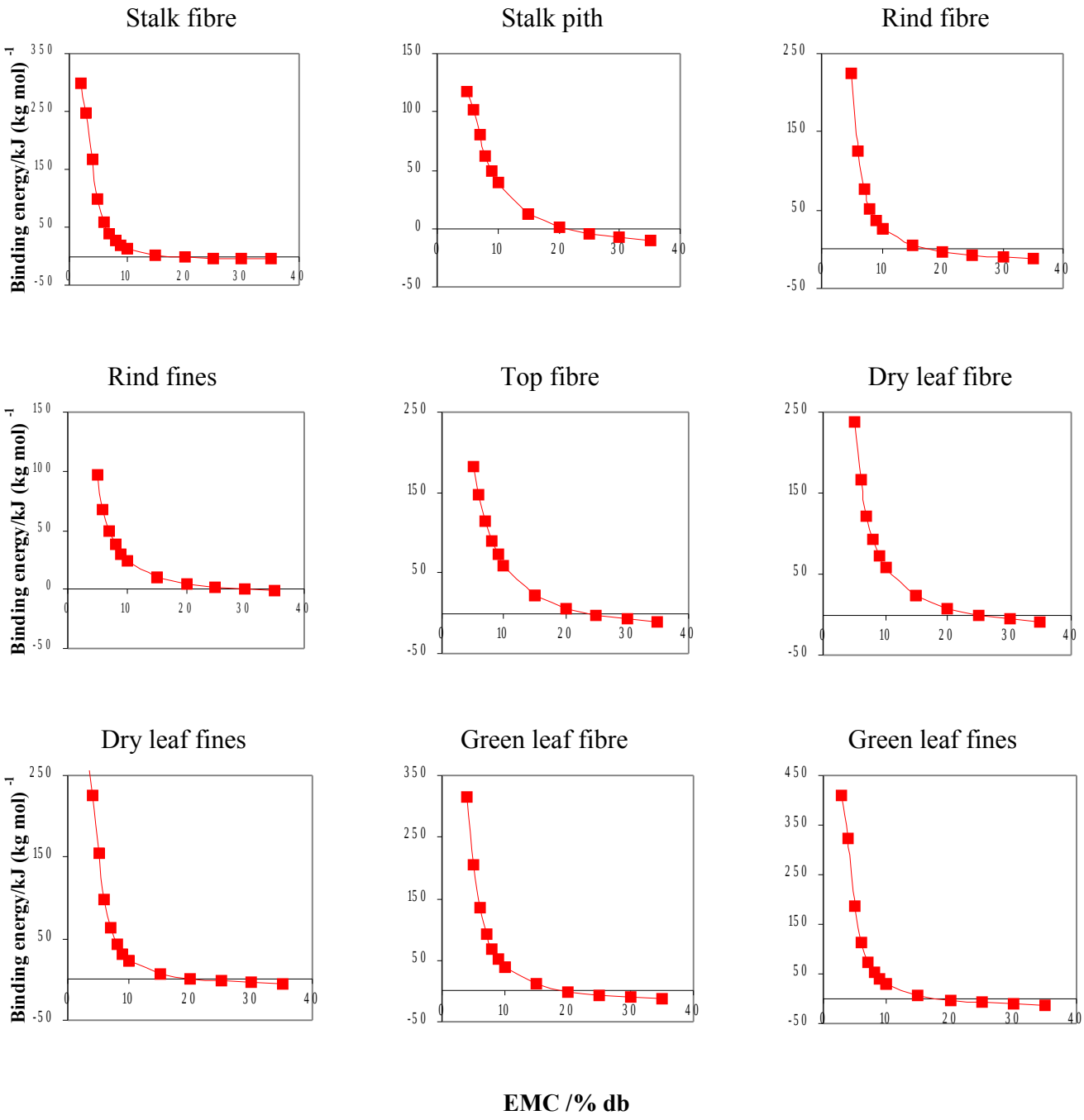


Figure 6.12. Average energy of water binding by sugar cane component parts of cane variety R 570 aged 36 weeks.

6.8 CALCULATION OF BOUND WATER AND DISSOLVED WATER FROM THE HAILWOOD-HORROBIN MODEL

The Hailwood-Horrobin sorption model is based on the assumption that the subject of study forms an ideal solid solution with three species present in the solid phase: dissolved water, hydrated molecules and unhydrated molecules. Hence the adsorbed water is either in simple solution or combined with a fibre molecule to form a hydrate. When the sorption equation of the model (Hailwood and Horrobin, 1946) is expressed as:

$$m\frac{W}{1800} = \frac{K_2 a_w}{1 - K_2 a_w} + \frac{K_1 K_2 a_w}{1 + K_1 K_2 a_w} \qquad (5)$$

The first term on the right hand side refers to dissolved water (m_s) and the second term refers to the hydrated water (m_h). In equation (5), m is the equilibrium moisture content (%), W is the molecular mass of the adsorbate substance necessary to bond one molecular mass of water (mol/mol), K is the equilibrium constant between the free dissolved water and the hydrated water, K_2 is the equilibrium constant between the dissolved water and the external vapour pressure and a_w is the water activity.

The amount of dissolved water can be calculated by:

$$m_s = \frac{K_2 a_w}{1 - K_2 a_w} \times \frac{1800}{W}$$

and the amount of hydrated water by:

$$m_h = \frac{K_1 K_2 a_w}{1 + K_1 K_2 a_w} \times \frac{1800}{W},$$

hence the total adsorbed water, m is given by:

$$m = m_s + m_h$$

In this work the experimental EMC data at various values of a_w were fitted to the following form of Hailwood-Horrobin sorption model:

$$\frac{a_w}{m} = b + c\,a_w + d\,(a_w)^2$$

and the 'best fit' values for the parameters b, c and d determined.

Equation (5) can be rearranged to be in the above form as written in Table 5.1.

$$\frac{mW}{1800} = \frac{K_2 a_w + K_1 K_2^2 (a_w)^2 + K_1 K_2 a_w - K_1 K_2^2 (a_w)^2}{(1 - K_2 a_w)(1 + K_1 K_2 a_w)}$$

$$\frac{a_w}{m} = \left[\frac{1 + (K_1 K_2 - K_2) a_w - K_1 K_2^2 (a_w)^2}{K_2 + K_1 K_2} \right] \frac{W}{1800}$$

$$= \frac{W}{1800 (K_2 + K_1 K_2)} + \frac{W (K_1 K_2 + K_2) a_w}{1800 (K_2 + K_1 K_2)} - \frac{W K_1 K_2^2 (a_w)^2}{1800 (K_2 + K_1 K_2)}$$

The parameters K_1, K_2 and W can then be calculated from their algebraic relationship to b, c and d.

hence $\frac{c}{b} = \frac{W(K_1 K_2 - K_2)}{1800(K_2 + K_1 K_2)} \times \frac{1800(K_2 + K_1 K_2)}{W} = K_1 K_2 - K_2$ (6)

and $\frac{d}{b} = \frac{-W K_1 K_2^2}{1800(K_2 + K_1 K_2)} \times \frac{1800(K_2 + K_1 K_2)}{W} = K_1 K_2^2$ (7)

from equation (6), $K_2 = \frac{c/b}{(K_1 - 1)}$,

substituting K_2 into equation (7),

$$\frac{K_1 (c/b)^2}{(K_1 - 1)} = d/b$$

$$\frac{K_1}{(K_1 - 1)^2} = \frac{d/b}{(c/b)^2} = z$$

$$z K_1^2 - (2z + 1) K_1 + z = 0$$

The quadratic equation K_1 $Ax^2 + Bx + C = 0$

was found to be $A = z$, $B = -2z - 1$ and $C = z$

and the root of the equation is $\frac{-B \pm \sqrt{B^2 - 4AC}}{2A}$

$$K_1 = \pm \frac{2z + 1 \pm \sqrt{(-2z - 1)^2 - 4z^2}}{2z}$$

The positive value is taken for K_1, and this value was used to calculate K_2 from the equation given above.

W is calculated from the equation $b = \frac{W}{1800(K_2 + K_1 K_2)}$

$$W = b \times 1800(K_2 + K_1 K_2)$$

The values of K_1, K_2 and W were used to calculate m_h, m_s and m for each experimental a_w value at each of the four temperatures for all the cane components. The results are compared with the experimental values of the equilibrium moisture content, and are shown in Tables 6.7 for all nine components aged 52 and 36 weeks.

Similarly the results for reconstituted cane stalk, dry leaf and green leaf are compared in Table 6.8. These results are plotted against water activity a_w in Figures 6.13.1 – 6.13.8 for nine cane components aged 52 and 36 weeks at 30, 45, 55 and 60 °C and are shown on the CD (File: Fig.6.13.1-6.13.8 Hydrated and dissolved water.xls). Typical plot for nine cane components aged 52 weeks at 30 °C is shown in Figure 6.13, and similar plots for reconstituted cane stalk, dry leaf and green leaf aged 52 and 36 weeks at four temperatures are presented in Figures 6.14 and 6.15 respectively.

From Tables 6.7 – 6.8, we observe that m_h decreases with decrease in water activity and with increase in temperature. The latter is in keeping with the fact that, in general, for a fixed a_w the EMC decreases with increase in temperature at the smaller values of water activity. At 52 weeks and at 30 °C, dry leaf fines has the highest m_h value of 6.47 and rind fines, the lowest value of 4.20. At 36 weeks and 30 °C, top fibre has the highest m_h value of 5.01 and stalk fibre, the lowest value of 3.99.

From Fig 6.13 it can be seen that the m_h curves exhibit a Langmuir-type monolayer isotherm, and become saturated in the high water activity region, in fact the m_h values obtained from the Hailwood-Horrobin model agree fairly well with the monolayer moisture content, m_o values derived from the GAB model. It therefore appears that this hydrated water corresponds to the initially bound water that binds directly to the polar groups on the surface of the fibre. This stronger binding is reflected in the larger heats of sorption observed when the EMC is between 0 and 5%.

The dissolved water, m_s, values decrease with decrease in water activity and increase with increase in temperature. This is in keeping with the fact that at large a_w values the EMC at given value of a_w increases with temperature. At 52 weeks and at 30 °C, stalk pith has the highest m_s value of 27.87 and rind fibre, the lowest value of 16.94. At 36 weeks and at 30 °C, stalk pith has the highest m_s value of 24.28 and rind fibre, the lowest value of 16.54.

The m_s curves depicted in Fig 6.13 increase sharply within the whole water activity region. This dissolved water therefore corresponds to multilayer adsorption where water molecules hydrogen bond to water molecules already attached to the surface of the fibre. The binding

is much weaker and is reflected in the heats of adsorption measured particularly at EMC values between 5 and 20% and the H_m values calculated from the temperature dependence of the GAB parameters.

The value of m_h/m generally decreases with increase of water activity and with increase of temperature. This observation can be easily rationalised from the foregoing. Since the hydrated water is that fraction of water that binds first to the active sites on the surface of the fibre, with increasing water activity the number of these sites which are unoccupied will diminish and this is reflected in the m_h/m ratio. The number of active sites decreases with increase in temperature as observed before and hence this ratio also decreases with increase in temperature. According to Norimoto and Yamada (1977), one hydrated water molecule is bonded to about four hydroxyl groups by hydrogen bonds, whereas one dissolved water molecule is bonded to not more than two hydroxyl groups. This further corroborates the difference observed in the heats of binding for these two types of water.

Figures 6.13 – 6.15 show the experimental or calculated EMC values correspond closely to the total water calculated as the sum of the hydrated and dissolved water (except in the case of dry leaf fibre aged 36 weeks at 45 and 60 °C, green leaf fines aged 36 weeks and at 30 °C and reconstituted green leaf aged 36 weeks at 30, 45, 55 and 60 °C).

What is apparent from here is that the Hailwood-Horrobin dissolved water encompasses both the multilayer water that has a heat of adsorption just somewhat larger than the heat of vaporisation of pure water and free water with the same properties of bulk water and probably situated in the voids in the fibre.

Table 6.7. Separation of the total adsorbed water (m) into hydrated water (m_h) and dissolved water (m_s) as given by the Hailwood-Horrobin model for the nine cane components of R 570 aged 52 and 36 weeks at various temperatures.

Sample	30 ºC						45 ºC						55 ºC						60 ºC					
	a_w	Exp. EMC	m_h	m_s	m	m_h/m	a_w	Exp. EMC	m_h	m_s	m	m_h/m	a_w	Exp. EMC	m_h	m_s	m	m_h/m	a_w	Exp. EMC	m_h	m_s	m	m_h/m
Stalk fibre	0.9808	24.84	5.02	17.82	22.85	0.22	0.9812	26.34	4.29	21.10	25.39	0.17	0.9816	28.49	3.27	25.42	28.68	0.11	0.9818	32.22	3.00	27.63	30.63	0.10
52 weeks	0.9746	20.18	5.02	17.37	22.39	0.22	0.9751	24.61	4.29	20.41	24.69	0.17	0.9760	27.74	3.27	24.25	27.51	0.12	0.9764	28.62	3.00	26.10	29.10	0.10
	0.8814	16.27	4.96	12.16	17.12	0.29	0.8839	15.92	4.24	13.23	17.47	0.24	0.8868	15.35	3.25	13.39	16.64	0.20	0.8882	15.23	3.00	13.19	16.20	0.19
	0.7549	14.50	4.85	7.97	12.83	0.38	0.7629	12.85	4.16	8.29	12.45	0.33	0.7684	11.60	3.22	7.63	10.86	0.30	0.7711	11.02	3.00	7.20	10.20	0.29
	0.5711	8.64	4.63	4.50	9.13	0.51	0.5866	10.33	4.01	4.55	8.56	0.47	0.5948	7.81	3.17	3.93	7.10	0.45	0.5989	7.97	3.01	3.61	6.62	0.45
	0.3574	5.35	4.16	2.17	6.34	0.66	0.3765	5.25	3.67	2.18	5.85	0.63	0.3879	5.08	3.04	1.83	4.88	0.62	0.3936	4.95	3.02	1.66	4.67	0.65
	0.1677	4.63	3.19	0.85	4.04	0.79	0.1834	3.49	2.94	0.86	3.80	0.77	0.1937	3.08	2.73	0.72	3.45	0.79	0.1988	3.27	3.04	0.65	3.69	0.82
Stalk pith	0.9808	34.94	5.39	27.87	33.25	0.16	0.9812	34.99	4.70	32.88	37.58	0.13	0.9816	37.14	3.83	39.30	43.14	0.09	0.9818	41.54	3.41	39.14	42.54	0.08
52 weeks	0.9746	27.26	5.39	26.80	32.19	0.17	0.9751	39.92	4.70	31.29	36.00	0.13	0.9760	44.51	3.83	37.06	40.89	0.09	0.9764	41.26	3.00	36.85	39.85	0.08
	0.8814	21.51	5.40	16.37	21.77	0.25	0.8839	22.61	4.71	17.39	22.10	0.21	0.8868	20.99	3.81	18.45	22.26	0.17	0.8882	20.03	3.00	17.91	20.91	0.14
	0.7549	14.51	5.41	9.73	15.14	0.36	0.7629	13.50	4.72	9.92	14.64	0.32	0.7684	12.84	3.76	9.99	13.76	0.27	0.7711	13.44	3.00	9.60	12.61	0.24
	0.5711	11.08	5.43	5.10	10.53	0.52	0.5866	9.61	4.75	5.10	9.85	0.48	0.5948	8.99	3.67	4.99	8.67	0.42	0.5989	9.11	3.01	4.78	7.78	0.39
	0.3574	7.37	5.49	2.35	7.84	0.70	0.3765	8.05	4.81	2.35	7.16	0.67	0.3879	6.43	3.48	2.29	5.77	0.60	0.3936	4.84	3.02	2.20	5.21	0.58
	0.1677	6.47	5.69	0.89	6.58	0.86	0.1834	5.72	5.01	0.91	5.92	0.85	0.1937	3.53	3.02	0.89	3.91	0.77	0.1988	2.89	3.04	0.86	3.90	0.78
Rind fibre	0.9808	22.02	4.84	16.94	21.77	0.22	0.9812	24.09	4.29	19.41	23.70	0.18	0.9812	26.58	3.07	23.34	26.41	0.12	0.9818	37.25	2.55	30.84	33.39	0.08
52 weeks	0.9746	20.91	4.83	16.50	21.33	0.23	0.9751	22.84	4.28	18.85	23.13	0.19	0.9751	25.12	3.06	22.29	25.35	0.12	0.9764	25.99	2.55	28.69	31.24	0.08
	0.8814	15.49	4.79	11.52	16.31	0.29	0.8839	15.70	4.21	12.70	16.92	0.25	0.8839	14.87	3.02	12.72	15.74	0.19	0.8882	14.80	2.56	12.71	15.27	0.17
	0.7549	13.03	4.70	7.54	12.24	0.38	0.7629	12.13	4.10	8.18	12.28	0.33	0.7629	11.68	2.95	7.36	10.31	0.29	0.7711	9.61	2.58	6.56	9.14	0.28
	0.5711	8.60	4.53	4.25	8.77	0.52	0.5866	9.91	3.87	4.59	8.46	0.46	0.5866	6.50	2.81	3.82	6.63	0.42	0.5989	7.92	2.61	3.19	5.81	0.45
	0.3574	6.02	4.14	2.05	6.19	0.67	0.3765	5.47	3.41	2.23	5.64	0.60	0.3765	4.56	2.52	1.77	4.29	0.59	0.3936	4.47	2.70	1.45	4.16	0.65
	0.1677	4.17	3.30	0.80	4.10	0.81	0.1834	2.91	2.52	0.89	3.41	0.74	0.1834	2.51	1.93	0.69	2.62	0.74	0.1988	2.93	3.00	0.57	3.56	0.84
Rind fines	0.9808	23.81	4.20	17.71	21.91	0.19	0.9812	25.16	4.02	20.00	24.02	0.17	0.9816	24.59	3.02	21.34	24.36	0.12	0.9818	23.53	3.11	20.91	24.02	0.13
52 weeks	0.9746	19.73	4.20	17.16	21.36	0.20	0.9751	22.69	4.01	19.34	23.35	0.17	0.9760	22.07	3.01	20.47	23.48	0.13	0.9764	23.46	3.10	20.14	23.25	0.13
	0.8814	14.98	4.18	11.30	15.49	0.27	0.8839	15.99	3.97	12.49	16.46	0.24	0.8868	15.62	2.99	11.85	14.84	0.20	0.8882	15.39	3.07	12.11	15.18	0.20
	0.7549	11.52	4.15	7.07	11.22	0.37	0.7629	11.74	3.90	7.80	11.70	0.33	0.7684	9.12	2.95	6.93	9.88	0.30	0.7711	10.48	3.00	7.25	10.25	0.29
	0.5711	8.51	4.07	3.85	7.92	0.51	0.5866	8.62	3.76	4.28	8.03	0.47	0.5948	6.01	2.86	3.64	6.50	0.44	0.5989	6.01	2.88	3.87	6.74	0.43
	0.3574	5.48	3.90	1.81	5.71	0.68	0.3765	5.87	3.45	2.05	5.49	0.63	0.3879	4.77	2.68	1.71	4.39	0.61	0.3936	4.48	2.62	1.85	4.47	0.59
	0.1677	4.08	3.46	0.70	4.16	0.83	0.1834	3.15	2.77	0.81	3.58	0.77	0.1937	2.89	2.26	0.68	2.93	0.77	0.1988	3.09	2.09	0.74	2.83	0.74
Top fibre	0.9808	30.62	5.39	23.31	28.70	0.19	0.9812	33.10	4.04	27.19	31.22	0.13	0.9816	45.06	3.22	34.52	37.74	0.09	0.9818	46.45	3.25	36.59	39.84	0.08
52 weeks	0.9746	26.32	5.39	22.57	27.96	0.19	0.9751	28.83	4.04	25.96	30.00	0.13	0.9760	25.33	3.22	32.28	35.50	0.09	0.9764	29.67	3.25	34.32	37.58	0.09
	0.8814	19.77	5.36	14.77	20.13	0.27	0.8839	18.52	4.02	14.86	18.88	0.21	0.8868	17.90	3.23	15.05	18.29	0.18	0.8882	20.67	3.24	16.20	19.45	0.17
	0.7549	15.70	5.32	9.20	14.51	0.37	0.7629	12.70	4.00	8.61	12.62	0.32	0.7684	12.50	3.25	7.92	11.18	0.29	0.7711	12.20	3.23	8.59	11.82	0.27
	0.5711	10.08	5.22	4.98	10.20	0.51	0.5866	8.72	3.96	4.48	8.44	0.47	0.5948	8.92	3.29	3.89	7.19	0.46	0.5989	8.16	3.20	4.24	7.44	0.43
	0.3574	6.30	4.99	2.34	7.33	0.68	0.3765	7.27	3.85	2.08	5.93	0.65	0.3879	5.60	3.40	1.77	5.17	0.66	0.3936	4.43	3.13	1.94	5.07	0.62
	0.1677	5.72	4.41	0.90	5.31	0.83	0.1834	3.66	3.58	0.80	4.38	0.82	0.1937	3.70	3.75	0.69	4.44	0.85	0.1988	3.63	2.94	0.76	3.70	0.79
Dry leaf fibre	0.9808	26.90	5.39	20.39	25.78	0.21	0.9812	29.59	3.87	23.81	27.69	0.14	0.9816	34.00	3.59	24.74	28.33	0.13	0.9818	44.40	2.65	35.89	38.55	0.07
52 weeks	0.9746	23.77	5.39	19.81	25.20	0.21	0.9751	24.45	3.87	22.82	26.70	0.15	0.9760	22.91	3.59	23.73	27.32	0.13	0.9764	29.61	2.65	33.06	35.72	0.07
	0.8814	16.55	5.37	13.39	18.76	0.29	0.8839	17.23	3.86	13.51	17.37	0.22	0.8868	16.40	3.57	13.79	17.35	0.21	0.8882	16.52	2.67	13.64	16.31	0.16
	0.7549	16.31	5.33	8.54	13.87	0.38	0.7629	11.63	3.83	7.99	11.82	0.32	0.7684	13.41	3.53	8.08	11.61	0.30	0.7711	10.09	2.71	6.86	9.57	0.28
	0.5711	9.65	5.24	4.72	9.96	0.53	0.5866	8.67	3.78	4.21	7.99	0.47	0.5948	8.33	3.44	4.24	7.69	0.45	0.5989	8.46	2.78	3.29	6.07	0.46
	0.3574	6.09	5.04	2.25	7.29	0.69	0.3765	5.70	3.66	1.97	5.62	0.65	0.3879	5.28	3.26	2.00	5.26	0.62	0.3936	5.16	2.97	1.49	4.45	0.67
	0.1677	5.74	4.52	0.87	5.39	0.84	0.1834	3.84	3.34	0.77	4.11	0.81	0.1937	3.19	2.83	0.79	3.62	0.78	0.1988	3.63	3.68	0.58	4.26	0.86

Table 6.7. (Contd.)

Sample	30 °C						45 °C						55 °C						60 °C					
	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m
Dry leaf fines	0.9808	27.99	6.47	19.85	26.31	0.25	0.9812	28.14	4.07	23.77	27.85	0.15	0.9816	27.61	3.58	24.58	28.16	0.13	0.9818	34.22	3.15	28.84	31.99	0.10
52 weeks	0.9746	23.22	6.46	19.41	25.87	0.25	0.9751	26.45	4.07	22.82	26.89	0.15	0.9760	27.04	3.58	23.53	27.11	0.13	0.9764	28.14	3.15	27.32	30.47	0.10
	0.8814	20.91	6.34	14.19	20.53	0.31	0.8839	17.51	4.06	13.72	17.78	0.23	0.8868	17.24	3.58	13.43	17.01	0.21	0.8882	16.54	3.15	13.99	17.14	0.18
	0.7549	16.02	6.15	9.65	15.79	0.39	0.7629	11.99	4.04	8.18	12.22	0.33	0.7684	10.48	3.57	7.79	11.36	0.31	0.7711	11.34	3.14	7.69	10.83	0.29
	0.5711	10.53	5.75	5.61	11.36	0.51	0.5866	8.99	3.99	4.34	8.33	0.48	0.5948	7.27	3.55	4.06	7.61	0.47	0.5989	8.02	3.13	3.88	7.01	0.45
	0.3574	7.99	4.96	2.77	7.73	0.64	0.3765	6.02	3.89	2.03	5.92	0.66	0.3879	6.85	3.50	1.91	5.41	0.65	0.3936	4.92	3.10	1.80	4.90	0.63
	0.1677	4.63	3.50	1.09	4.60	0.76	0.1834	4.27	3.61	0.79	4.40	0.82	0.1937	3.55	3.38	0.75	4.13	0.82	0.1988	3.41	3.03	0.71	3.74	0.81
Green leaf fibre	0.9808	27.82	4.80	20.20	25.00	0.19	0.9812	28.61	4.06	24.64	28.70	0.14	0.9816	31.14	3.41	28.16	31.57	0.11	0.9818	34.04	3.47	28.08	31.55	0.11
52 weeks	0.9746	23.18	4.80	19.61	24.40	0.20	0.9751	28.83	4.06	23.65	27.71	0.15	0.9760	31.21	3.41	26.80	30.21	0.11	0.9764	27.04	3.47	26.75	30.22	0.11
	0.8814	16.63	4.74	13.17	17.91	0.26	0.8839	17.83	4.03	14.18	18.22	0.22	0.8868	17.75	3.39	14.48	17.87	0.19	0.8882	18.89	3.46	14.43	17.89	0.19
	0.7549	14.73	4.64	8.36	13.00	0.36	0.7629	11.95	3.99	8.45	12.44	0.32	0.7684	11.35	3.36	8.16	11.52	0.29	0.7711	10.72	3.45	8.13	11.58	0.30
	0.5711	9.54	4.44	4.60	9.04	0.49	0.5866	9.73	3.90	4.48	8.37	0.47	0.5948	7.61	3.30	4.17	7.47	0.44	0.5989	7.92	3.44	4.16	7.60	0.45
	0.3574	5.90	4.01	2.18	6.19	0.65	0.3765	5.66	3.70	2.10	5.79	0.64	0.3879	5.58	3.16	1.94	5.10	0.62	0.3936	5.31	3.40	1.95	5.34	0.64
	0.1677	3.96	3.10	0.84	3.94	0.79	0.1834	3.75	3.21	0.82	4.02	0.80	0.1937	3.32	2.82	0.76	3.59	0.79	0.1988	4.00	3.28	0.77	4.05	0.81
Green leaf fines	0.9808	27.95	4.80	22.11	26.90	0.18	0.9812	34.52	3.68	27.98	31.66	0.12	0.9812	42.81	3.17	33.19	36.36	0.09	0.9818	35.21	3.26	28.90	32.16	0.10
52 weeks	0.9746	25.63	4.79	21.39	26.18	0.18	0.9751	28.04	3.68	26.55	30.23	0.12	0.9751	28.13	3.18	31.10	34.28	0.09	0.9764	28.26	3.26	27.41	30.67	0.11
	0.8814	17.28	4.76	13.84	18.60	0.26	0.8839	17.37	3.68	14.39	18.07	0.20	0.8839	17.55	3.18	14.76	17.94	0.18	0.8882	16.99	3.26	14.19	17.45	0.19
	0.7549	15.15	4.70	8.55	13.25	0.35	0.7629	12.24	3.67	8.10	11.77	0.31	0.7629	12.61	3.19	7.82	11.02	0.29	0.7711	11.8	3.26	7.84	11.09	0.29
	0.5711	8.77	4.57	4.60	9.18	0.50	0.5866	9.17	3.67	4.13	7.80	0.47	0.5866	8.19	3.22	3.86	7.08	0.45	0.5989	8.16	3.25	3.96	7.22	0.45
	0.3574	6.11	4.29	2.16	6.45	0.67	0.3765	5.77	3.66	1.89	5.55	0.66	0.3765	5.62	3.29	1.76	5.04	0.65	0.3936	5.23	3.24	1.84	5.08	0.64
	0.1677	4.61	3.62	0.83	4.44	0.81	0.1834	3.97	3.62	0.73	4.35	0.83	0.1834	3.53	3.49	0.68	4.17	0.84	0.1988	3.64	3.22	0.73	3.94	0.82
Stalk fibre	0.9808	24.20	3.99	20.99	24.98	0.16	0.9812	26.63	3.58	23.93	27.51	0.13	0.9816	27.06	3.26	24.48	27.75	0.12	0.9818	23.85	3.21	22.85	26.05	0.12
36 weeks	0.9746	23.97	3.99	20.20	24.19	0.17	0.9751	27.76	3.58	22.90	26.48	0.14	0.9760	27.19	3.26	23.35	26.61	0.12	0.9764	27.44	3.20	21.99	25.20	0.13
	0.8814	15.49	3.98	12.41	16.39	0.24	0.8839	15.47	3.55	13.36	16.92	0.21	0.8868	14.58	3.26	12.86	16.12	0.20	0.8882	15.39	3.15	13.13	16.28	0.19
	0.7549	11.54	3.96	7.41	11.37	0.35	0.7629	12.48	3.51	7.83	11.34	0.31	0.7684	10.98	3.26	7.32	10.57	0.31	0.7711	11.08	3.08	7.83	10.90	0.28
	0.5711	7.93	3.92	3.89	7.81	0.50	0.5866	7.73	3.41	4.10	7.52	0.45	0.5948	8.02	3.25	3.77	7.01	0.46	0.5989	7.54	2.92	4.16	7.08	0.41
	0.3574	5.79	3.83	1.80	5.62	0.68	0.3765	4.97	3.20	1.91	5.11	0.63	0.3879	5.57	3.23	1.76	4.98	0.65	0.3936	4.57	2.60	1.99	4.59	0.57
	0.1677	4.04	3.56	0.68	4.25	0.84	0.1834	3.32	2.71	0.74	3.45	0.78	0.1937	3.36	3.17	0.69	3.87	0.82	0.1988	2.61	1.99	0.80	2.79	0.71
Stalk pith	0.9808	31.64	4.97	24.28	29.25	0.17	0.9812	35.57	4.21	31.64	35.85	0.12	0.9816	36.67	3.63	34.40	38.03	0.10	0.9818	33.08	3.56	33.73	37.28	0.10
36 weeks	0.9746	25.52	4.96	23.43	28.39	0.17	0.9751	35.33	4.21	30.09	34.29	0.12	0.9760	37.37	3.63	32.60	36.23	0.10	0.9764	40.25	3.20	31.84	35.04	0.09
	0.8814	19.00	4.95	14.74	19.69	0.25	0.8839	17.96	4.19	16.58	20.77	0.20	0.8868	19.06	3.60	16.94	20.54	0.18	0.8882	17.9	3.15	15.84	18.99	0.17
	0.7549	13.62	4.92	8.94	13.86	0.36	0.7629	16.04	4.16	9.41	13.57	0.31	0.7684	14.68	3.54	9.36	12.90	0.27	0.7711	13.35	3.08	8.59	11.66	0.26
	0.5711	11.36	4.86	4.75	9.60	0.51	0.5866	9.45	4.11	4.83	8.94	0.46	0.5948	7.84	3.43	4.73	8.16	0.42	0.5989	7.05	2.92	4.30	7.21	0.40
	0.3574	6.01	4.71	2.21	6.92	0.68	0.3765	6.19	3.98	2.22	6.20	0.64	0.3879	4.65	3.20	2.18	5.38	0.59	0.3936	5.73	2.60	1.98	4.59	0.57
	0.1677	5.11	4.31	0.84	5.16	0.84	0.1834	4.07	3.64	0.86	4.50	0.81	0.1937	3.86	2.67	0.86	3.52	0.76	0.1988	4.83	1.99	0.78	2.77	0.72
Rind fibre	0.9808	21.63	4.63	16.54	21.17	0.22	0.9812	23.26	3.64	19.70	23.35	0.16	0.9816	27.59	3.21	22.46	25.67	0.13	0.9818	22.80	3.40	19.18	22.58	0.15
36 weeks	0.9746	19.99	4.63	16.08	20.72	0.22	0.9751	22.56	3.64	19.05	22.69	0.16	0.9760	22.14	3.21	21.50	24.71	0.13	0.9764	22.80	3.40	18.55	21.95	0.15
	0.8814	16.39	4.62	11.00	15.62	0.30	0.8839	15.14	3.58	12.26	15.84	0.23	0.8868	15.13	3.20	12.28	15.48	0.21	0.8882	13.88	3.36	11.65	15.01	0.22
	0.7549	12.17	4.60	7.08	11.67	0.39	0.7629	11.44	3.48	7.64	11.12	0.31	0.7684	10.27	3.18	7.13	10.31	0.31	0.7711	10.36	3.32	7.16	10.48	0.32
	0.5711	8.44	4.54	3.94	8.48	0.54	0.5866	7.52	3.28	4.18	7.46	0.44	0.5948	7.59	3.14	3.72	6.86	0.46	0.5989	8.51	3.21	3.89	7.11	0.45
	0.3574	4.76	4.41	1.88	6.29	0.70	0.3765	5.15	2.89	2.00	4.88	0.59	0.3879	4.94	3.06	1.75	4.80	0.64	0.3936	5.62	2.99	1.89	4.88	0.61
	0.1677	5.55	4.06	0.73	4.79	0.85	0.1834	2.58	2.13	0.79	2.92	0.73	0.1937	3.20	2.83	0.69	3.52	0.80	0.1988	2.42	2.51	0.76	3.27	0.77

Table 6.7. (Contd.)

Sample	30 °C						45 °C						55 °C						60 °C					
	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m	a_w	Expt. EMC	m_h	m_s	m	m_h/m
Rind fines	0.9808	23.18	4.12	16.87	20.99	0.20	0.9812	25.50	3.82	19.00	22.83	0.17	0.9816	23.98	3.00	18.72	21.72	0.14	0.9818	21.30	3.49	16.08	19.56	0.18
36 weeks	0.9746	19.19	4.11	16.36	20.48	0.20	0.9751	20.69	3.82	18.38	22.20	0.17	0.9760	18.08	3.00	18.02	21.02	0.14	0.9764	17.87	3.48	15.62	19.10	0.18
	0.8814	15.19	4.09	10.89	14.98	0.27	0.8839	14.61	3.78	11.89	15.67	0.24	0.8868	13.90	2.98	10.86	13.84	0.22	0.8882	12.67	3.46	10.33	13.79	0.25
	0.7549	11.42	4.05	6.87	10.92	0.37	0.7629	12.77	3.71	7.43	11.14	0.33	0.7684	9.33	2.94	6.51	9.45	0.31	0.7711	10.63	3.42	6.58	10.00	0.34
	0.5711	7.94	3.95	3.76	7.71	0.51	0.5866	7.66	3.57	4.08	7.65	0.47	0.5948	6.37	2.86	3.47	6.33	0.45	0.5989	7.51	3.33	3.67	7.01	0.48
	0.3574	5.08	3.74	1.78	5.52	0.68	0.3765	5.28	3.26	1.95	5.21	0.63	0.3879	4.94	2.69	1.65	4.34	0.62	0.3936	5.39	3.15	1.81	4.96	0.64
	0.1677	4.13	3.22	0.69	3.91	0.82	0.1834	3.22	2.61	0.77	3.38	0.77	0.1937	2.54	2.30	0.66	2.96	0.78	0.1988	3.03	2.72	0.74	3.46	0.79
Top fibre	0.9808	30.03	5.01	23.54	28.56	0.18	0.9812	36.92	4.05	28.15	32.19	0.13	0.9816	44.27	3.08	34.37	37.45	0.08	0.9818	39.32	2.88	31.41	34.29	0.08
36 weeks	0.9746	25.18	5.01	22.77	27.78	0.18	0.9751	26.75	4.05	26.83	30.88	0.13	0.9760	26.25	3.08	32.09	35.17	0.09	0.9764	26.57	2.88	29.39	32.27	0.09
	0.8814	18.37	4.97	14.70	19.67	0.25	0.8839	17.41	4.04	15.14	19.17	0.21	0.8868	18.23	3.09	14.78	17.86	0.17	0.8882	15.45	2.89	13.59	16.49	0.18
	0.7549	14.95	4.90	9.07	13.97	0.35	0.7629	15.04	4.02	8.70	12.73	0.32	0.7684	11.79	3.10	7.74	10.83	0.29	0.7711	11.93	2.92	7.14	10.06	0.29
	0.5711	9.87	4.75	4.88	9.63	0.49	0.5866	8.64	3.99	4.50	8.49	0.47	0.5948	8.14	3.12	3.79	6.91	0.45	0.5989	7.64	2.97	3.51	6.48	0.46
	0.3574	6.07	4.42	2.28	6.71	0.66	0.3765	7.20	3.92	2.08	5.99	0.65	0.3879	5.07	3.18	1.72	4.90	0.65	0.3936	5.31	3.10	1.60	4.70	0.66
	0.1677	4.61	3.66	0.88	4.54	0.81	0.1834	3.73	3.71	0.80	4.51	0.82	0.1937	3.52	3.37	0.67	4.04	0.83	0.1988	3.61	3.54	0.63	4.17	0.85
Dry leaf fibre	0.9808	27.52	4.48	22.15	26.63	0.17	0.9812	30.78	-	-	-	-	0.9812	29.90	4.05	23.81	27.85	0.15	0.9818	59.77	0.34	59.89	60.23	0.01
36 weeks	0.9746	24.96	4.48	21.36	25.84	0.17	0.9751	26.51	-	-	-	-	0.9751	23.71	4.05	22.91	26.96	0.15	0.9764	20.33	0.34	23.03	23.37	0.01
	0.8814	16.76	4.46	13.38	17.85	0.25	0.8839	16.17	-	-	-	-	0.8839	16.76	4.04	13.80	17.84	0.23	0.8882	14.33	0.36	1.90	2.26	0.16
	0.7549	13.65	4.44	8.09	12.53	0.35	0.7629	13.05	-	-	-	-	0.7629	12.26	4.03	8.26	12.29	0.33	0.7711	11.46	0.41	0.75	1.15	0.35
	0.5711	8.74	4.39	4.29	8.67	0.51	0.5866	8.46	-	-	-	-	0.5866	9.05	4.00	4.40	8.40	0.48	0.5989	5.88	0.56	0.32	0.88	0.64
	0.3574	5.87	4.27	1.99	6.26	0.68	0.3765	8.51	-	-	-	-	0.3765	7.04	3.95	2.09	6.04	0.65	0.3936	5.26	5.21	0.14	5.35	0.97
	0.1677	4.71	3.94	0.76	4.70	0.84	0.1834	3.71	-	-	-	-	0.1834	3.90	3.79	0.83	4.62	0.82	0.1988	3.62	-0.23	0.05	-0.18	1.29
Dry leaf fines	0.9808	27.98	4.70	20.72	25.42	0.18	0.9812	32.14	3.95	26.28	30.23	0.13	0.9816	27.96	4.28	23.15	27.44	0.16	0.9818	28.73	3.51	23.85	27.36	0.13
36 weeks	0.9746	22.57	4.70	20.04	24.74	0.19	0.9751	27.14	3.95	25.12	29.06	0.14	0.9760	27.09	4.28	22.39	26.67	0.16	0.9764	25.61	3.51	22.88	26.38	0.13
	0.8814	17.45	4.69	12.95	17.64	0.27	0.8839	17.09	3.93	14.47	18.41	0.21	0.8868	17.07	4.25	14.13	18.38	0.23	0.8882	14.76	3.50	13.16	16.66	0.21
	0.7549	12.54	4.67	7.99	12.66	0.37	0.7629	12.09	3.91	8.42	12.33	0.32	0.7684	13.18	4.19	8.71	12.90	0.32	0.7711	12.11	3.49	7.68	11.17	0.31
	0.5711	10.02	4.64	4.30	8.94	0.52	0.5866	10.24	3.85	4.39	8.24	0.47	0.5948	10.24	4.07	4.74	8.81	0.46	0.5989	8.61	3.47	4.03	7.50	0.46
	0.3574	6.33	4.55	2.01	6.56	0.69	0.3765	6.61	3.72	2.04	5.76	0.65	0.3879	6.24	3.82	2.28	6.10	0.63	0.3936	6.35	3.43	1.91	5.34	0.64
	0.1677	5.01	4.29	0.77	5.07	0.85	0.1834	3.21	3.39	0.79	4.19	0.81	0.1937	3.67	3.24	0.92	4.15	0.78	0.1988	3.29	3.32	0.76	4.08	0.81
Green leaf fibre	0.9808	26.97	4.36	20.87	25.23	0.17	0.9812	29.83	3.73	25.42	29.15	0.13	0.9816	28.34	3.67	25.61	29.29	0.13	0.9818	58.33	1.91	49.15	51.06	0.04
36 weeks	0.9746	23.02	4.36	20.15	24.51	0.18	0.9751	29.50	3.73	24.28	28.00	0.13	0.9760	27.52	3.67	24.57	28.24	0.13	0.9764	30.21	1.91	42.12	44.02	0.04
	0.8814	17.15	4.35	12.74	17.09	0.25	0.8839	17.32	3.71	13.91	17.62	0.21	0.8868	17.36	3.64	14.29	17.93	0.20	0.8882	16.21	1.93	11.75	13.68	0.14
	0.7549	12.34	4.33	7.75	12.08	0.36	0.7629	12.01	3.68	8.07	11.75	0.31	0.7684	12.04	3.59	8.38	11.98	0.30	0.7711	11.69	1.98	5.31	7.29	0.27
	0.5711	8.54	4.28	4.13	8.40	0.51	0.5866	8.34	3.62	4.20	7.82	0.46	0.5948	7.47	3.49	4.40	7.89	0.44	0.5989	8.15	2.08	2.42	4.50	0.46
	0.3574	5.99	4.16	1.92	6.08	0.68	0.3765	5.97	3.49	1.94	5.43	0.64	0.3879	5.98	3.27	2.08	5.35	0.61	0.3936	5.48	2.38	1.07	3.44	0.69
	0.1677	4.62	3.85	0.73	4.59	0.84	0.1834	3.55	3.14	0.75	3.89	0.81	0.1937	3.24	2.77	0.82	3.59	0.77	0.1988	3.38	3.99	0.41	4.40	0.91
Green leaf fines	0.9808	27.94	-	-	-	-	0.9812	31.43	3.73	25.34	29.07	0.13	0.9816	37.02	3.25	28.67	31.92	0.10	0.9818	46.80	2.78	36.40	39.17	0.07
36 weeks	0.9746	22.81	-	-	-	-	0.9751	25.93	3.73	24.18	27.91	0.13	0.9760	24.52	3.25	27.10	30.35	0.11	0.9764	25.30	2.78	33.64	36.42	0.08
	0.8814	16.88	-	-	-	-	0.8839	16.79	3.72	13.75	17.47	0.21	0.8868	16.56	3.26	13.78	17.04	0.19	0.8882	16.67	2.80	14.20	17.00	0.16
	0.7549	12.01	-	-	-	-	0.7629	11.70	3.71	7.94	11.65	0.32	0.7684	11.96	3.28	7.54	10.81	0.30	0.7711	12.17	2.82	7.20	10.03	0.28
	0.5711	8.73	-	-	-	-	0.5866	8.26	3.68	4.12	7.80	0.47	0.5948	7.68	3.32	3.79	7.10	0.47	0.5989	8.15	2.88	3.47	6.36	0.45
	0.3574	6.25	-	-	-	-	0.3765	7.54	3.62	1.90	5.52	0.66	0.3879	5.72	3.42	1.74	5.16	0.66	0.3936	5.46	3.04	1.57	4.61	0.66
	0.1677	4.83	-	-	-	-	0.1834	3.15	3.44	0.74	4.18	0.82	0.1937	3.97	3.74	0.68	4.42	0.85	0.1988	3.34	3.57	0.61	4.19	0.85

Table 6.8. Separation of the total adsorbed water (m) into hydrated water (m_h) and dissolved water (m_s) as given by the Hailwood-Horrobin model for the reconstituted cane stalk and dry leaf and green leaf of R 570 of two ages at various temperatures.

Sample	30 °C						45 °C						55 °C						60 °C					
	a_w	Predicted EMC	m_h	m_s	m	m_h/m	a_w	Predicted EMC	m_h	m_s	m	m_h/m	a_w	Predicted EMC	m_h	m_s	m	m_h/m	a_w	Predicted EMC	m_h	m_s	m	m_h/m
Reconstituted cane stalk	0.9808	26.08	4.83	19.48	24.31	0.20	0.9812	27.38	4.28	23.83	28.11	0.15	0.9816	29.00	3.25	26.37	29.63	0.11	0.9818	34.15	2.98	29.73	32.70	0.09
52 weeks	0.9746	22.05	4.83	18.90	23.73	0.20	0.9751	27.27	4.27	22.94	27.22	0.16	0.9760	29.56	3.25	25.16	28.40	0.11	0.9764	29.55	2.98	28.01	30.98	0.10
	0.8814	16.97	4.80	12.59	17.40	0.28	0.8839	17.49	4.24	14.18	18.42	0.23	0.8868	16.62	3.22	13.86	17.08	0.19	0.8882	16.29	2.97	13.86	16.83	0.18
	0.7549	13.28	4.76	7.95	12.71	0.37	0.7629	12.49	4.19	8.60	12.80	0.33	0.7684	11.33	3.18	7.89	11.07	0.29	0.7711	10.99	2.95	7.49	10.44	0.28
	0.5711	9.19	4.67	4.35	9.02	0.52	0.5866	9.60	4.08	4.62	8.70	0.47	0.5948	7.21	3.09	4.06	7.15	0.43	0.5989	7.76	2.92	3.73	6.66	0.44
	0.3574	6.11	4.47	2.06	6.53	0.68	0.3765	6.16	3.85	2.18	6.03	0.64	0.3879	5.15	2.89	1.89	4.78	0.60	0.3936	4.64	2.85	1.71	4.56	0.63
	0.1677	4.79	3.95	0.79	4.74	0.83	0.1834	3.75	3.28	0.85	4.14	0.79	0.1937	2.94	2.44	0.75	3.18	0.77	0.1988	3.01	2.67	0.67	3.34	0.80
Reconstituted dry leaf	0.9808	27.36	5.80	19.57	25.36	0.23	0.9812	28.97	3.96	23.79	27.76	0.14	0.9816	31.31	3.57	24.68	28.25	0.13	0.9818	40.11	2.87	33.90	36.76	0.08
52 weeks	0.9746	23.54	5.79	19.08	24.87	0.23	0.9751	25.29	3.96	22.82	26.79	0.15	0.9760	24.65	3.57	23.65	27.22	0.13	0.9764	28.99	2.87	31.57	34.44	0.08
	0.8814	18.38	5.73	13.43	19.16	0.30	0.8839	17.35	3.95	13.60	17.55	0.22	0.8868	16.75	3.55	13.62	17.17	0.21	0.8882	16.53	2.88	14.10	16.97	0.17
	0.7549	16.18	5.63	8.84	14.48	0.39	0.7629	11.78	3.93	8.07	12.00	0.33	0.7684	12.17	3.53	7.94	11.47	0.31	0.7711	10.62	2.90	7.30	10.20	0.28
	0.5711	10.02	5.42	5.01	10.43	0.52	0.5866	8.80	3.88	4.27	8.14	0.48	0.5948	7.88	3.47	4.16	7.62	0.46	0.5989	8.27	2.94	3.56	6.50	0.45
	0.3574	6.89	4.97	2.42	7.39	0.67	0.3765	5.83	3.76	1.99	5.76	0.65	0.3879	5.94	3.34	1.95	5.29	0.63	0.3936	5.06	3.06	1.62	4.68	0.65
	0.1677	5.27	3.96	0.95	4.91	0.81	0.1834	4.02	3.46	0.78	4.24	0.82	0.1937	3.34	3.01	0.77	3.79	0.80	0.1988	3.53	3.44	0.63	4.07	0.84
Reconstituted green leaf	0.9808	27.86	4.82	21.45	26.27	0.18	0.9812	30.55	3.92	26.29	30.21	0.13	0.9812	34.98	3.34	30.06	33.41	0.10	0.9818	36.03	3.26	29.90	33.17	0.10
52 weeks	0.9746	23.98	4.82	20.79	25.61	0.19	0.9751	28.57	3.92	25.12	29.04	0.13	0.9751	30.20	3.34	28.47	31.81	0.11	0.9764	27.68	3.26	28.30	31.57	0.10
	0.8814	16.84	4.77	13.70	18.47	0.26	0.8839	17.67	3.90	14.46	18.36	0.21	0.8839	17.68	3.33	14.70	18.04	0.18	0.8882	18.11	3.27	14.40	17.66	0.18
	0.7549	14.87	4.69	8.57	13.26	0.35	0.7629	12.05	3.87	8.41	12.28	0.32	0.7629	11.76	3.32	8.10	11.42	0.29	0.7711	10.69	3.27	7.88	11.16	0.29
	0.5711	9.28	4.51	4.66	9.17	0.49	0.5866	9.55	3.81	4.38	8.19	0.47	0.5866	7.80	3.29	4.09	7.38	0.45	0.5989	8.04	3.28	3.97	7.25	0.45
	0.3574	5.97	4.13	2.20	6.33	0.65	0.3765	5.69	3.67	2.03	5.71	0.64	0.3765	5.59	3.22	1.88	5.11	0.63	0.3936	5.23	3.30	1.84	5.13	0.64
	0.1677	4.17	3.29	0.85	4.14	0.80	0.1834	3.82	3.32	0.79	4.11	0.81	0.1834	3.39	3.04	0.74	3.78	0.80	0.1988	3.85	3.35	0.72	4.08	0.82
Reconstituted cane stalk	0.9808	24.51	4.48	18.56	23.03	0.19	0.9812	26.92	3.79	22.55	26.33	0.14	0.9816	28.53	3.22	23.17	26.39	0.12	0.9818	25.52	3.37	21.98	25.35	0.13
36 weeks	0.9746	21.45	4.47	17.98	22.46	0.20	0.9751	25.44	3.78	21.68	25.47	0.15	0.9760	24.96	3.22	22.17	25.39	0.13	0.9764	25.19	3.37	21.16	24.53	0.14
	0.8814	16.53	4.46	11.87	16.33	0.27	0.8839	15.64	3.75	13.25	16.99	0.22	0.8868	15.59	3.20	12.59	15.79	0.20	0.8882	14.86	3.34	12.64	15.98	0.21
	0.7549	12.21	4.43	7.44	11.87	0.37	0.7629	12.91	3.68	7.98	11.67	0.32	0.7684	11.05	3.18	7.29	10.46	0.30	0.7711	11.45	3.30	7.54	10.84	0.30
	0.5711	8.88	4.36	4.05	8.41	0.52	0.5866	8.00	3.56	4.26	7.82	0.45	0.5948	7.37	3.12	3.79	6.91	0.45	0.5989	7.98	3.21	4.01	7.22	0.44
	0.3574	5.24	4.20	1.91	6.11	0.69	0.3765	5.38	3.28	2.01	5.28	0.62	0.3879	4.95	3.00	1.78	4.78	0.63	0.3936	5.22	3.02	1.91	4.94	0.61
	0.1677	4.90	3.78	0.74	4.52	0.84	0.1834	3.16	2.66	0.78	3.45	0.77	0.1937	3.18	2.69	0.70	3.40	0.79	0.1988	2.91	2.58	0.77	3.35	0.77
Reconstituted dry leaf	0.9808	27.69	4.57	22.11	26.68	0.17	0.9812	31.28	3.99	25.33	29.33	0.14	0.9816	29.18	4.12	23.10	27.22	0.15	0.9818	48.31	2.15	36.27	38.42	0.06
36 weeks	0.9746	24.08	4.57	21.33	25.91	0.18	0.9751	26.74	3.99	24.23	28.22	0.14	0.9760	24.96	4.12	22.29	26.40	0.16	0.9764	22.28	2.15	32.70	34.85	0.06
	0.8814	17.01	4.56	13.44	18.00	0.25	0.8839	16.51	3.99	14.08	18.07	0.22	0.8868	16.87	4.10	13.72	17.82	0.23	0.8882	14.49	2.18	11.76	13.93	0.16
	0.7549	13.24	4.54	8.15	12.70	0.36	0.7629	12.69	3.98	8.23	12.21	0.33	0.7684	12.60	4.07	8.33	12.40	0.33	0.7711	11.70	2.22	5.66	7.88	0.28
	0.5711	9.21	4.50	4.33	8.83	0.51	0.5866	9.11	3.96	4.30	8.26	0.48	0.5948	9.49	4.01	4.48	8.49	0.47	0.5989	6.89	2.32	2.66	4.97	0.47
	0.3574	6.04	4.39	2.01	6.41	0.69	0.3765	7.81	3.92	2.00	5.92	0.66	0.3879	6.74	3.88	2.14	6.03	0.64	0.3936	5.66	2.58	1.19	3.77	0.69
	0.1677	4.82	4.10	0.77	4.87	0.84	0.1834	3.52	3.79	0.78	4.57	0.83	0.1937	3.81	3.55	0.86	4.41	0.81	0.1988	3.50	3.87	0.46	4.33	0.89
Reconstituted green leaf	0.9808	27.26	1.18	22.13	23.31	0.05	0.9812	30.31	1.05	24.70	25.75	0.04	0.9816	30.94	1.02	24.64	25.66	0.04	0.9818	54.88	0.44	6.82	7.26	0.06
36 weeks	0.9746	22.96	1.18	20.81	21.99	0.05	0.9751	28.43	1.05	22.91	23.96	0.04	0.9760	26.62	1.01	22.94	23.96	0.04	0.9764	28.74	0.44	6.51	6.95	0.06
	0.8814	17.07	1.11	10.41	11.53	0.10	0.8839	17.16	1.00	10.41	11.40	0.09	0.8868	17.12	0.97	10.36	11.32	0.09	0.8882	16.34	0.41	3.56	3.97	0.10
	0.7549	12.24	1.02	5.55	6.57	0.16	0.7629	11.92	0.92	5.40	6.32	0.15	0.7684	12.02	0.89	5.38	6.27	0.14	0.7711	11.83	0.38	2.02	2.40	0.16
	0.5711	8.59	0.86	2.71	3.57	0.24	0.5866	8.31	0.78	2.63	3.41	0.23	0.5948	7.53	0.76	2.63	3.39	0.23	0.5989	8.15	0.33	1.04	1.37	0.24
	0.3574	6.06	0.62	1.20	1.82	0.34	0.3765	6.43	0.58	1.17	1.75	0.33	0.3879	5.90	0.57	1.19	1.76	0.32	0.3936	5.47	0.25	0.49	0.74	0.34
	0.1677	4.68	0.33	0.45	0.78	0.43	0.1834	3.43	0.33	0.45	0.77	0.42	0.1937	3.45	0.33	0.46	0.79	0.42	0.1988	3.36	0.15	0.19	0.34	0.43

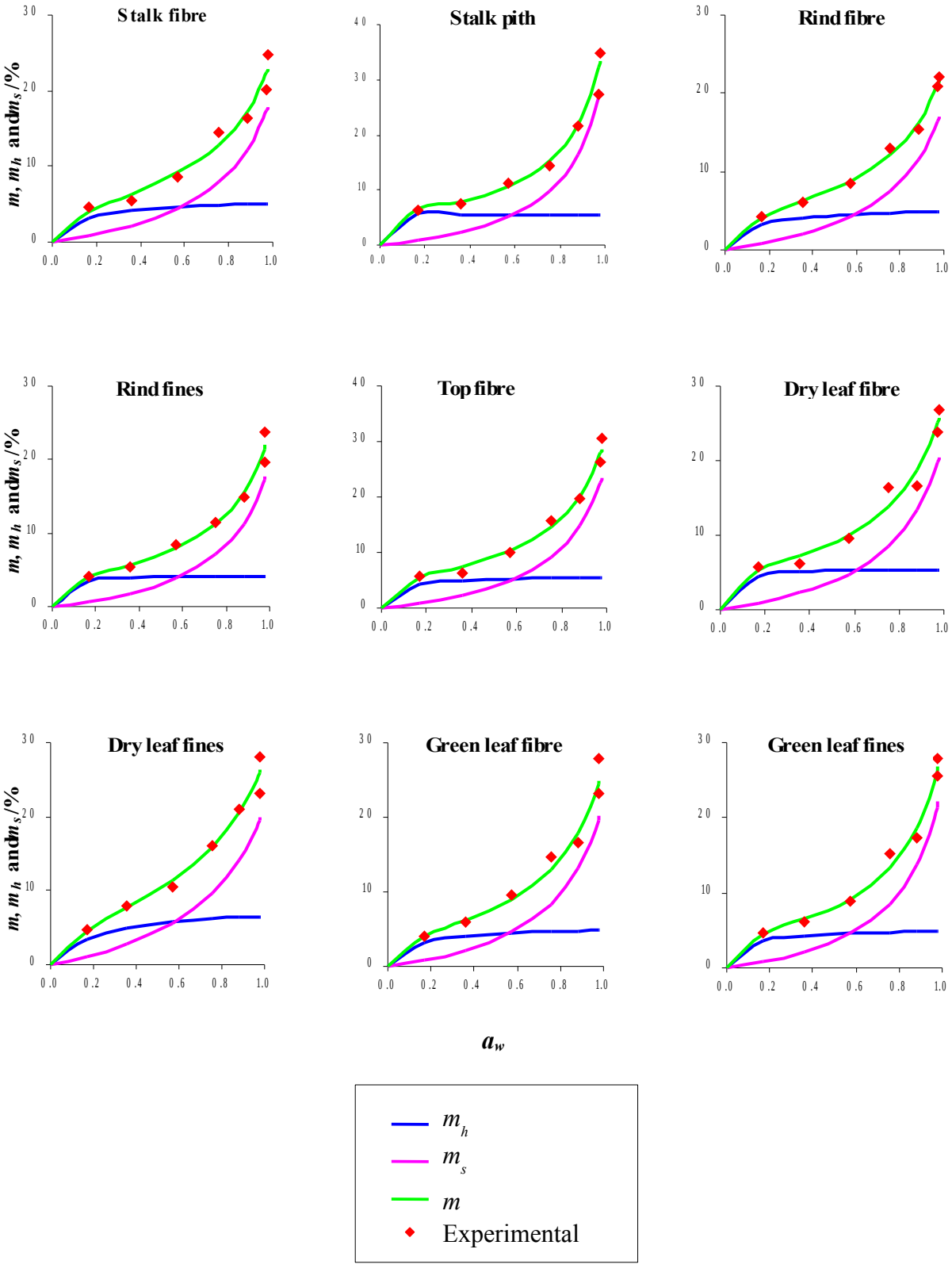


Figure 6.13. Amounts of adsorbed hydrated water (m_h), dissolved water (m_s), total adsorbed water (m) and the experimental equilibrium moisture content for the nine cane component parts of R 570 aged 52 weeks at 30 °C.

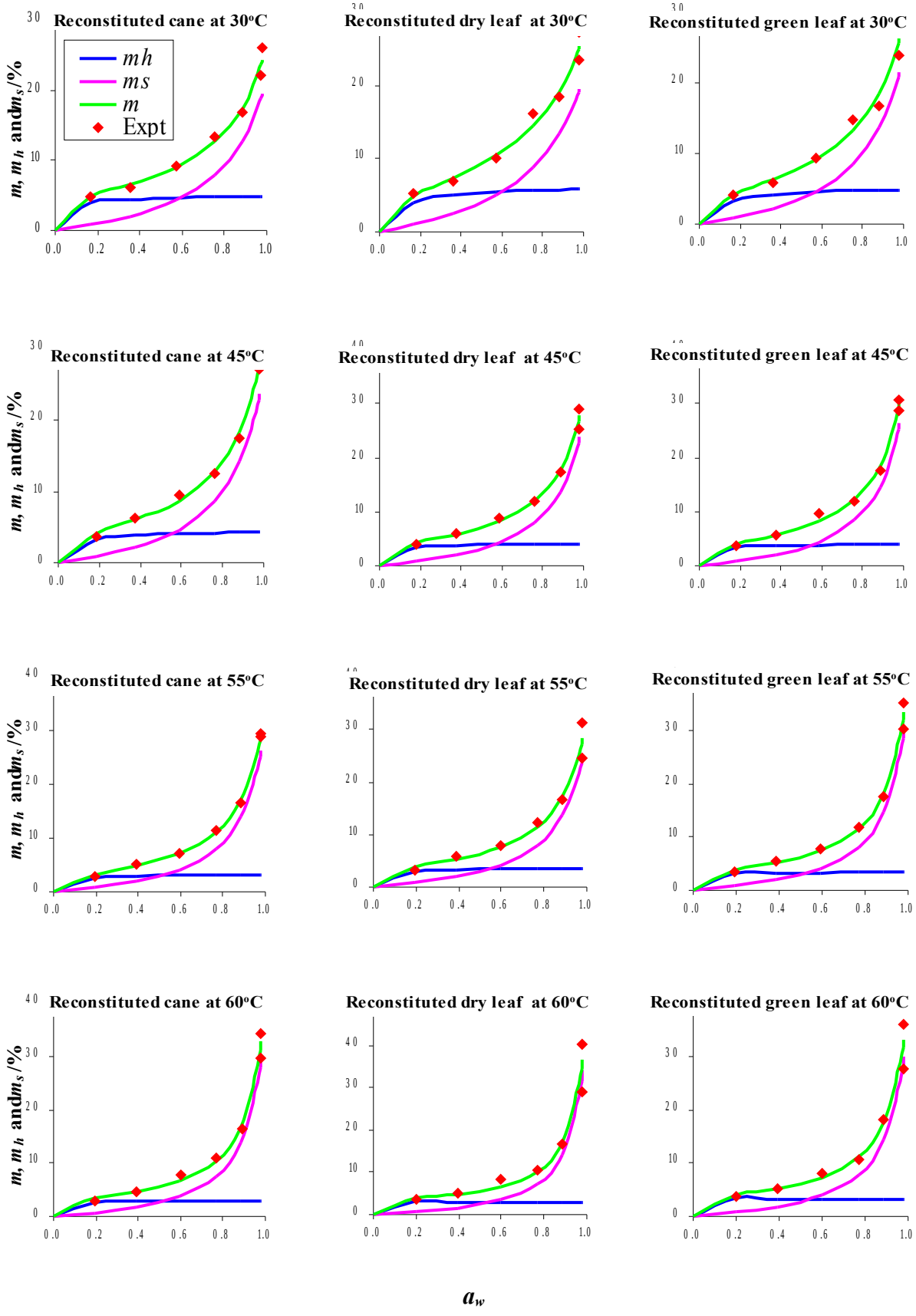


Figure 6.14. Amounts of adsorbed hydrated water (m_h), dissolved water (m_s), total adsorbed water (m) and the predicted equilibrium moisture content of reconstituted cane stalk, dry leaf and green leaf aged 52 weeks at various temperatures.

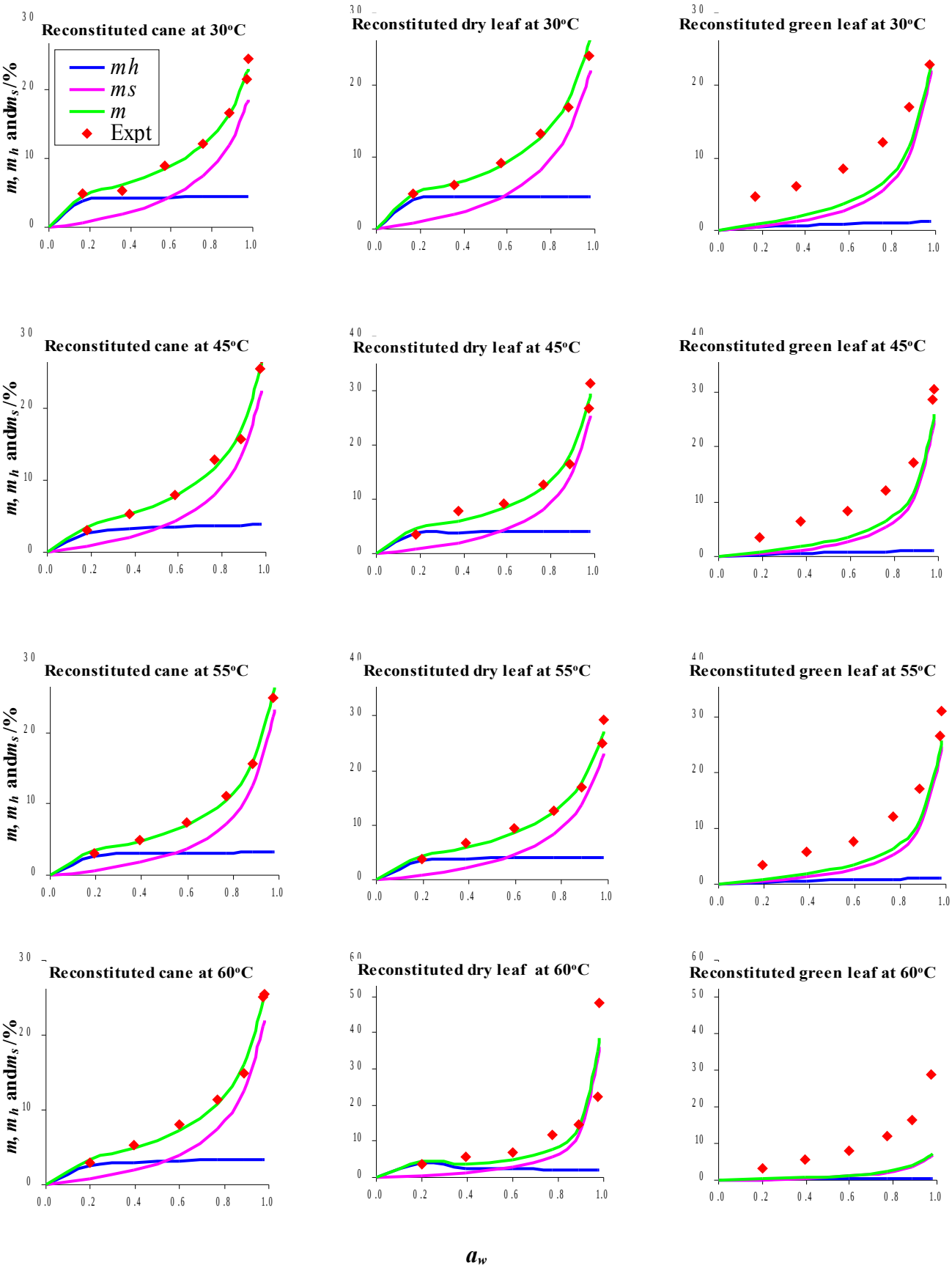


Figure 6.15. Amounts of adsorbed hydrated water (m_h), dissolved water (m_s), total adsorbed water (m) and the predicted equilibrium moisture content of reconstituted cane stalk, dry leaf and green leaf aged 36 weeks at various temperatures.

6.9 FIBRE SATURATION POINT

Berry and Roderick (2005) in their review on plant-water relations introduce the idea of fibre saturation point (FSP), well known and in routine practical use by engineers and material scientists for at least 50 years. Fibre saturation point is routinely used to estimate the volume fractions of solid, liquid and gas phases in bulk timber, it is based on the concept that a certain (and repeatable) amount of water is chemically bound to cellulose and other substances in wood. This water, also called bound water, exists in an integrated mixture of cell wall material and bound water, is recognized as a distinct phase called 'solid solution' by Stamm (1964), a separate phase from the adjacent water in either a pure liquid phase or a vapour phase.

The following illustrates well the concept of fibre saturation point. When a small volume of liquid water is poured onto oven-dried timber, observations show that when equilibrium is attained, the added water is not visible in the voids, but it must be located inside the cell wall matrix; which will swell as a result and the strength of the timber will progressively decline as the moisture content increases. If the addition of water is continued, the moisture content increases further, and the system will reach a new equilibrium state when liquid water begins to accumulate in the voids. The moisture content at which this occurs is called the fibre saturation point, which has been described as the moisture content at which the cell walls are fully saturated with liquid moisture but the cell cavities contain no water.

Water exists in timber in three phases: as vapour in the gas-filled voids, as 'free' or 'bulk' liquid water in the voids and as bound water in the cell wall matrix (Stamm, 1967a). The volume fraction of the three phases in timber can be estimated given its fresh volume, fresh mass, dry mass and the fibre saturation point. At the fibre saturation point, all of the liquid water is bound water, as it represents the maximum amount of water that can be taken up from the vapour phase by a unit mass of timber at a given temperature (Browning, 1963). If the fibre saturation point of timber is measured before and after pulverization to a larger surface area keeping the total mass the same, the results do not differ showing that there is a distinct number of binding sites for water in the timber, and the number of the binding sites is independent of the surface area of the sample (Stamm, 1964). The forces that hold the water preferentially in the cell walls are chemical bonding and/or capillary (i.e. surface) forces. The support of this statement comes from the fact that heat of wetting is evolved

when water is added to dry wood, which implies the existence of chemical reaction (Stamm, 1964).

From the point of view of equilibrium thermodynamic, if EMC is plotted against relative humidity in absorption study of timber, when the relative humidity approaches 100%, the moisture content of the timber will be about 30%, which is usually the fibre saturation point of timber. With increase of temperature, the fibre saturation point of timber is expected to decrease since a larger proportion of the water molecules should then have sufficient kinetic energy to enter an adjacent gas phase (Berry and Roderick, 2005).

The EMC data for the nine cane components of R 570 aged 52 weeks determined at 30, 45, 55 and 60 ºC (Tables 5.8 – 5.16) were plotted against relative humidity, taken as 100 multiplied by the water activity (Fig. 6.16). The fibre saturation point value of each component at each temperature was estimated and compared to the Brix-free water results determined at ambient temperature extracted from Tables 4.17 – 4.21 with 1.1 units added to correct for the residual moisture content (Table 6.9).

Table 6.9. Brix-free water and fibre saturation point bound water in fibres of cane components of R 570.

Sample	Brix-free water/%	Fibre saturation point value/%			
	Ambient temperature	30 ºC	45 ºC	55 ºC	60 ºC
Stalk fibre	12.37	20.18	24.61	27.74	28.62
Stalk pith	25.05	27.26	34.99	37.14	41.26
Rind fibre	12.02	22.02*	24.09*	26.58*	25.99
Rind fines	11.47	19.73	22.69	24.59	23.53*
Top fibre	16.76	26.32	28.83	25.33	29.67
Dry leaf fibre	16.13	23.77	24.45	22.91	29.61
Dry leaf fines	18.17	23.22	28.14	27.61*	28.14
Green leaf fibre	13.77	23.18	28.83*	31.21*	27.04
Green leaf fines	14.79	25.63	28.04	28.13	28.26
Reconstituted cane stalk	15.13	22.05	27.27*	29.56	29.55
Reconstituted dry leaf	16.99	23.54	25.29	24.65	28.99
Reconstituted green leaf	14.11	23.98	30.55	30.20	27.68

* Fibre saturation point not yet attained.

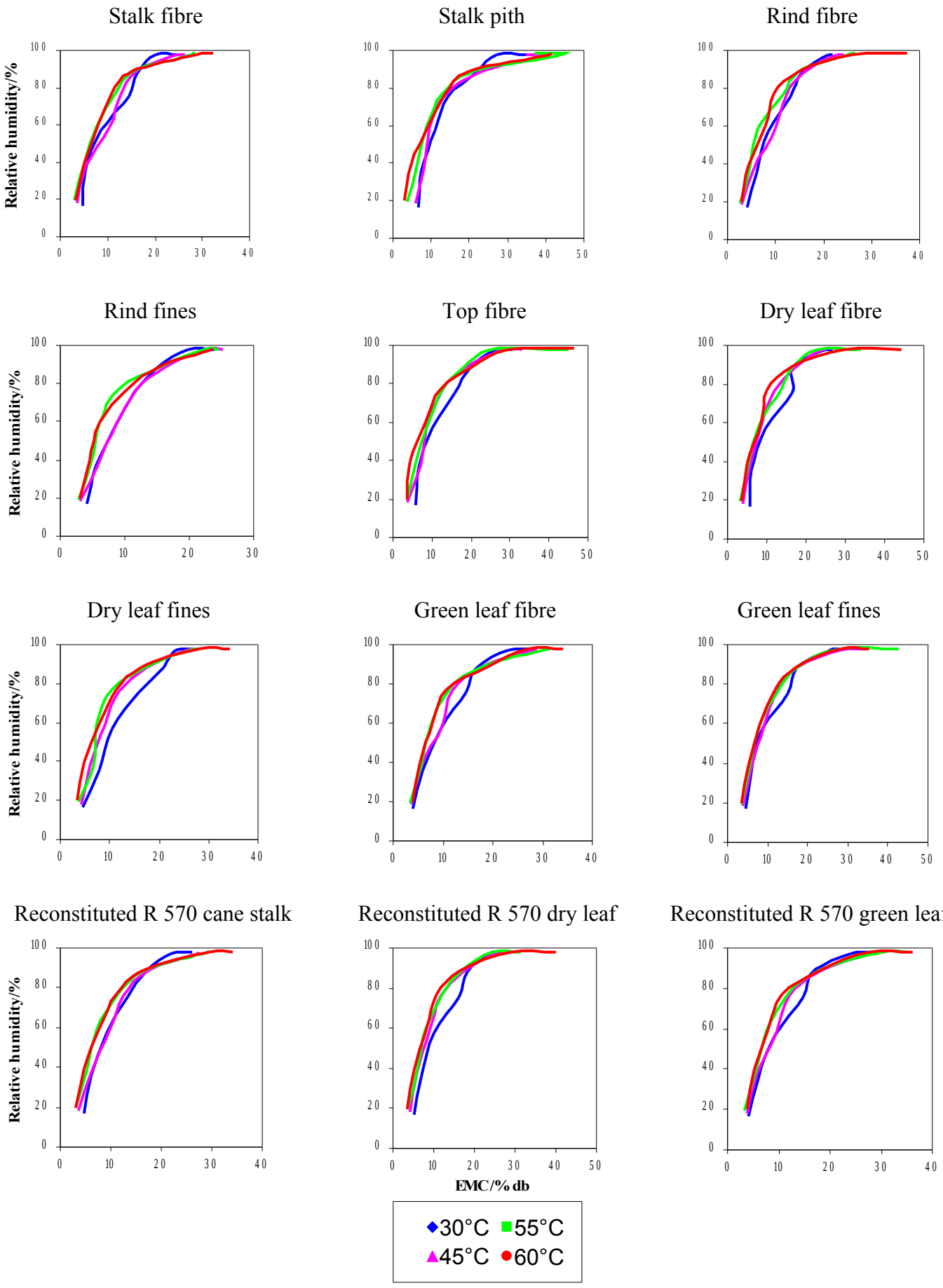


Figure 6.16. Fibre saturation point in nine cane components and reconstituted R 570 aged 52 weeks.

It is evident that in some cases marked with asterisks, the fibre saturation point is not yet reached. From Table 6.9, it can be seen that the bound water values estimated are much higher than the Brix-free water results obtained at ambient temperature, and that the FSP increases with increase in temperature from 30 ºC to 60 ºC in most cases, contrary to the finding of Berry and Roderick (2005). The reason why cane fibres behave differently from timber is not known.

As described in Section 5.6.4.5, the mass fraction of cane components (stalk fibre and pith, rind fibre and fines) and the measured component EMC can help to calculate the EMC of the reconstituted cane stalk (Igathinathane *et al*., 2005). By plotting the calculated EMC against relative humidity, the fibre saturation point at 30, 45, 55 and 60 ºC can be estimated. Similarly for the reconstituted dry leaf and green leaf of R 570 aged 52 weeks. These values are compared in Table 6.9 to the Brix-free water value (corrected for residual moisture) of the reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 weeks extracted from Table 4.26.

Stamm (1967b) provides a mathematical treatment of the possible types of diffusion in wood during drying, including diffusion of water vapour within the void structure, and diffusion of water in the 'solid solution'. If the water is chemically bound to materials in the cell walls during adsorption, then a diffusion process would involve a series of discrete jumps from one binding site to the next. The final equilibrium would equate to a thermodynamic state of maximum entropy.

The fibre saturation point values, in a number of cases are similar to the total water, m, as found with the Hailwood-Horrobin model, i.e. the sum of the dissolved and hydrated water.

6.9.1 The accepted Brix-free water value of 25% for cane

The traditionally accepted value of 25% Brix-free water on cane fibre has been quoted by Anon. (1970) and Anon. (1984), but no information is given on the source of the information. A literature search revealed that Foster (1962) mentioned he had obtained: "a value of 25% in an earlier work (Foster, 1956) on Q 50 cane fibre by measuring the water adsorbed at 20 ºC at relative humidities of 35 to 95% and extrapolating the values to 100% relative humidity. This gave a value of 25% for hygroscopic water, which is the value generally used in milling calculations and cane analysis in Queensland at the present time".

What was actually determined was not hygroscopic water but the fibre saturation point at 20 °C (see Section 6.9).

Table 6.9 compiles the values of fibre saturation point bound water of various cane components at 30, 45, 55 and 60 °C. Data at 20 °C were not available, but results calculated for 30 °C were comparable with the 25% value obtained by Foster (1956) at 20 °C, erroneously taken as Brix-free water value. Table 6.9 also shows the Brix-free water values determined in this work. These are in general lower than the fibre saturation point values. Hence, the generally accepted value of 25% is much too high for Brix-free water of cane.

6.10 SUMMARY AND CONCLUSIONS

The various parameters calculated have allowed for a better understanding of the nature of the water sorbed onto the sugar cane component parts.

As described in literature the bound water occurs in three regions.

From the monolayer moisture content and the amount of "hydrated water" as calculated from the Hailwood-Horrobin model it is clear that at EMC values between 0 and 5% the water is tightly bound to the surface of the fibre. The corresponding water activities are 0-0.3. This constitutes what has been termed non-freezable water.

The second region starts at EMC values from about 5% and ends at 10-15% depending on the cane components. The corresponding a_w values are 0.3 to 0.6-0.8. In this region the bound water has a heat of adsorption, H_m, which is 2 to 5 kJ mol^{-1} greater than the heat of vaporisation H_L of bulk pure water. This is termed the freezable water.

The third type of water is essentially free water, it exists after the second region and ends at EMC of approximately 25% (where the relative humidity is nearly 100%). This type of water has the same heat of adsorption as the latent heat of vaporisation of pure water.

From this study it is apparent that Brix-free water as measured in this work measures the amount of water bound in the first two regions.

- Treatment of the experimental sorption data according to the GAB, Caurie I and BET equations allowed the evaluation of monolayer moisture content in nine cane components aged 52 and 36 weeks, and in reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 and 36 weeks.

- The number of adsorbed monolayers, density of bound water, the percentage of bound water and the surface area available for hydrophilic binding in adsorption in nine cane components, and reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 and 36 weeks have been successfully calculated at four temperatures.
- The heats or sorption of monolayer and multilayer have been calculated for nine cane components and reconstituted cane stalk, dry leaf and green leaf of R 570 aged 52 and 36 weeks. The heat of sorption of multilayer for each particular fibre is systematically higher at 52 weeks (48-49 kJ mol^{-1}) than at 36 weeks (46-47 kJ mol^{-1}), whereas the heat of sorption of monolayer is very variable.
- The net isosteric heat of sorption and the entropy of sorption have been successfully calculated.
- The plots of the net isosteric heat of sorption versus the entropy of sorption for water adsorption in fibres of nine cane components of R 570 ages 52 and 36 weeks, within the water activity range 0.17 – 0.98, satisfy the enthalpy-entropy compensation theory. The adsorption process in the fibres of sugar cane plant is essentially enthalpy-controlled (isokinetic temperature > harmonic mean temperature) and non-spontaneous (+ΔG).
- The heat of sorption of monolayer and the plot of binding energy against moisture content lead to the primary, secondary and tertiary bound water, it was found that the secondary bound water corresponds to the Brix-free water values of sugar cane component parts, except in the case of stalk pith, which shows similarity to the tertiary bound water.
- The dissolved and hydrated waters adsorbed in fibres of sugar cane plant of R 570 aged 52 and 36 weeks during the adsorption process have been quantified. The same applied to reconstituted cane stalk, dry leaf and green leaf of R 570 of two ages.
- The values of fibre saturation point bound water have also been calculated for the nine cane components of R 570 aged 52 weeks and found to increase with increase of temperature. They vary from about 20% to 37%.
- The value of 25% traditionally accepted as Brix-free water value of cane was in fact found to be the fibre saturation point bound water determined at 20 °C.

CHAPTER 7. CONCLUSIONS

The trend in cane quality received at Mauritian sugar mills has been examined from 1960 to 2004, and compared to those in other sugar producing countries where milling data are available. The deleterious effects of extraneous matter such as soil, cane tops and trash on cane processing, and on Mauritian factory performance parameters, such as mill extraction, sucrose lost in filter cake % sucrose in cane, Clerget purity of molasses at 85° Brix % cane, mass of molasses at 85° Brix % cane and sugar quality have been examined.

The different effects of various kinds of extraneous matter, notably dry trash, green leaves and cane tops, have been clearly demonstrated by the addition of measured quantities of these extraneous matter to clean cane. Among the most important findings are: dry trash has by far a more adverse effect than green leaves or tops on juice extraction, sugar recovery, boiling house recovery and overall recovery. One unit of dry trash increases fibre % cane, mass of bagasse % cane and sucrose loss in bagasse % cane and in molasses % cane by 0.57, 1.17, 0.030 and 0.011 units respectively, and decreases juice extraction and sugar recovery by 0.22 and 0.23 units, respectively. The detrimental effect of green leaves is intermediate between those of dry trash and cane tops. The latter does not seem to affect fibre % cane, the mass of bagasse % cane and juice extraction. One unit of cane tops increases the non-sucrose level in mixed juice, which increases the mass of molasses at 85° Brix produced % cane by 0.041 unit, resulting in 0.015 unit sucrose loss in molasses % cane, a much higher loss than that produced by the same amount of dry trash.

The economic implication of the presence of extraneous matter in cane should be of great concern to both cane growers and millers, as besides a reduction in income, the following additional expenses should be considered: cost of harvest and transport of the extraneous matter, the increased cost of maintenance of the factory equipment, investment in new equipment to cope with the trash, soil and rocks, and the cost of lengthening of the crushing season.

During the controlled addition of extraneous matter to clean cane, it was found that when the moisture level of dry trash was below a certain critical level, the press juice obtained on crushing dry trash with clean cane had increased concentrations in sucrose, Brix, and pol, and above the critical moisture level, these concentrations decrease.

Data in Tables 2.9 and 2.11 show that dry trash raised the analytes (HPIC sucrose, Clerget sucrose, Brix and pol) of press juice. Unfortunately, moisture content of the dry trash used in the first case was not determined, whereas in the second case, the dry trash had 7.1 and 15.1% moisture, which indicates that the Brix-free water value of the dry trash used was about or greater than 15%. Experiment should have been performed with added moisture to the dry trash and determine at what moisture level, the analytes of the press juice cease to be raised, when the dry trash is crushed with clean cane. Data in Table 2.10 show that a dry trash sample of 29.8% moisture raised the Brix of press juice, this was probably due to experimental error, as only Brix was raised. In the fourth trial of dry trash addition to clean cane (Table 2.2), the dry trash sample had 17.88% moisture (Table 2.5), and it raised only the pol level in mixed juice, indicating that the Brix-free water value of the dry trash is below 17.88%. The fact that dry bagasse of 10.7 and 22.2% moisture (Table 2.12) raised the analytes of press juice would indicate that the Brix-free water of the bagasse sample used was about or greater than 22.2%. This was probably due to the presence, in the bagasse sample, a high proportion of pith, which has a higher Brix-free water value than other components of sugar cane plant. The above indicates that the Brix-free water in dry trash might be involved below a critical moisture level in raising the analytes of press juice and mixed juice.

To test this assertion, the sugar cane plant of four cane varieties and of three ages has been successfully separated into fibres of its component parts by means of a simple method specially developed for the purpose, for determination of Brix-free water capacity and sorption behaviour. The use of a 1.18 mm sieve ensures complete separation of fibres from pith, of which the ratio gives an indication of the milling quality of the cane variety. The four cane varieties under study show fibre/pith ratio approaching to one, indicating good millability.

An analytical method has been developed to determine the Brix-free water in fibres obtained from the various component parts of sugar cane plant. It involves contacting the dried fibre sample with a sucrose solution; the subsequent Brix-change in the contact solution gives a measure of the Brix-free water value of the sample. The method makes use of a distilled water blank to compensate for any residual sucrose in the sample, and good separation of fibre and fines in the sample is essential to obtain reproducible results.

The vacuum drying method of the fibre sample prior to the Brix-free water determination left residual moisture in the samples, which average 1.12% for the nine cane component

parts. Hence, the Brix-free water values determined by this method must have 1.12 units added to correct for the residual moisture.

The nine cane components (dry leaf fibre and fines, green leaf fibre and fines, top fibre, rind fibre and fines, and stalk fibre and pith) of triplicate cane samples of four cane varieties (R 579, R 570, M 1557/70 and M 1400/86) aged 52, 44 and 36 weeks were analysed in duplicate by the Brix-free water determination method devised. The Brix-free water results obtained were analysed statistically.

The Brix-free water values (corrected for residual moisture) of the reconstituted cane stalk of the four cane varieties aged 52 weeks were calculated, the average value is 16.03%, which is much lower than the traditionally accepted value of 25% for Brix-free water of cane stalk. For reconstituted dry leaf, green leaf and cane stalk, the Brix-free water values do not vary much with age nor with variety. The reconstituted dry leaf, green leaf and cane stalk of the four cane varieties each has an average of Brix-free water value of 15-16%. If these values are taken together with the corresponding 15-16% Brix-free water value of intact cane tops, it would mean that Brix-free water values of the different parts of the sugar cane plant, i.e. dry leaf, green leaf, cane tops and cane stalk, are all about 15-16%. Only those of fibres differ from those of fines or pith.

Equilibrium moisture content of the cane components of R 570 aged 52 and 36 weeks were determined by a static method at 30, 45, 55 and 60 °C at water activity from 0.1 – 0.9. The isotherms exhibit a type II sigmoid pattern. Of the 17 isotherm models fitted, the Hailwood-Horrobin and GAB models describe best the sorption behaviour of the sugar cane components.

Most authors divide the type II isotherm into three regions. The first low water activity region (0 – 0.3) is indicative of strongly bound water, at the intermediate water activity region (0.3 to 0.6 – 0.8), water molecules which are less firmly bound, and in the region of high water activity ($a_w \geq 0.6$), excess water is present in larger voids and capillaries, and essentially acts as bulk water.

Calculations were carried out to provide an insight in some properties of the sorbed water and the sorbent. The number of monolayers varies from about 7 to 4, which is similar to the number of hydration layers (5 or 6) estimated at the fibre saturation point in wood.

The heats of sorption of monolayer and multilayer have been calculated for the nine cane components of R 570 aged 52 and 36 weeks. The heat of sorption of multilayer for each

particular fibre is systematically higher at 52 weeks (48 – 49 kJ mol^{-1}) than at 36 weeks (46 – 47 kJ mol^{-1}), whereas the heat of sorption of monolayer is very variable.

The net isosteric heat of sorption-entropy relationship for water adsorption in fibres of nine cane components of R 570 aged 52 and 36 weeks, satisfy the enthalpy-entropy compensation theory. The adsorption process in the fibres of sugar cane plant is essentially enthalpy-controlled (isokinetic temperature > harmonic mean temperature) and non-spontaneous (+ΔG).

The various quantities calculated provide an insight into the nature of the water sorbed onto the sugar cane component parts.

As described in literature the bound water occurs in three regions.

From the monolayer moisture content and the amount of "hydrated water" as calculated from the Hailwood-Horrobin model it is clear that at EMC values between 0 and 5% the water is tightly bound to the surface of the fibre. The corresponding water activities are 0 – 0.3. This constitutes what has been termed non-freezable water.

The second region starts at EMC values from about 5% and ends at 10 – 15%. The corresponding a_w values are 0.3 to 0.6 – 0.8. In this region the bound water has a heat of adsorption, H_m, which is 2 to 5 kJ mol^{-1} greater than the heat of vaporisation H_L of bulk pure water. This is termed the freezable water.

The third type of water is essentially free water, it exists after the second region and ends at EMC of approximately 25% (where the relative humidity is nearly 100%). This type of water has the same heat of adsorption as the latent heat of vaporisation of pure water.

From this study it is apparent that Brix-free water as measured in this work measures the amount of water bound in the first two regions. The heat of sorption of monolayer and the plot of binding energy against moisture content lead to the primary, secondary and tertiary bound water, it was found that the secondary bound water corresponds to the Brix-free water values of sugar cane component parts, except in the case of stalk pith, which shows similarity to the tertiary bound water.

The value of 25% traditionally accepted as Brix-free water of cane was found to be the fibre saturation point bound water determined at 20 °C.

REFERENCES

Aguerre R.J., C. Suarez and P.E. Viollaz (1986). Enthalpy-entropy compensation in sorption phenomena: Application to the prediction of the effect of temperature on food isotherms. *Journal of Food Science.* 51:1547-1549.

Aguerre R.J., C. Suarez and P.E. Viollaz (1989). New BET type multilayer sorption isotherms. Part II: Modelling water sorption in foods. *Lebensmittel-Wissenchaft und Technologie.* 22:192-195.

Al-Muhtaseb A.H., W.A.M. McMinn and T.R.A. Magee (2002). Moisture sorption isotherm characteristics of food products: a review. *Transactions of Institution of Chemical Engineers,* vol 80, part C:118-128.

Al-Muhtaseb A.H., W.A.M. McMinn and T.R.A. Magee (2004a). Water sorption isotherms of starch powders. Part I: Mathematical description of experimental data. *Journal of Food Engineering.* 61:297-307.

Al-Muhtaseb A.H., W.A.M. McMinn and T.R.A. Magee (2004b). Thermodynamic characteristics. *Journal of Food Engineering.* 62:135-142.

Anderson B.B. (1946). Modification of the BET equation. *Journal of American Chemical Society.* 68:686-691.

Anon. (1858). Rapport à la chamber d'Agriculture de l'Ile Maurice, p 116.

Anon. (1970). Laboratory manual for Queensland Sugar Mills, 5th edition. Bureau of Sugar Experiment Stations, Division of Mill Technology, Brisbane, Queensland, pp 250.

Anon. (1977). Report of the Mechanization Committee (Factory) of the Mauritius Chamber of Agriculture. *Revue Agricole et Sucrière de l'Ile Maurice*. 56(3):146-166.

Anon. (1984). The standard laboratory manual for Australian sugar mills. *BSES.* Vol. 1, p 2-3.

Anon. (1985a). *Proceedings of South African Sugar Technologists' Association*. 59:27.

Anon. (1985b). Laboratory manual for South African Sugar Factories. *South African Sugar Technologists Association*: 372 p.

Anon. (1991). Laboratory manual for Mauritian sugar factories (official methods). *Société de Technologie Agricole et Sucrière de Maurice.* p1, p 24-27, p 30-35, p 113-114, p 115-121.

Anon. (1997). Sugar in Mauritius. *The Public Relations Office of the Sugar Industry.* 131 p.

Anon. (1999). Annual Report 1998, Mauritius Sugar Industry Research Institute, 101-104.

Anon. (2001a). The sugar sector strategic plan for the period 2001-2005 published by the Ministry of Agriculture, Food Technology and Natural Resources on 28 June 2001.

Anon. (2001b). Annual Report 2000, Mauritius Sugar Industry Research Institute, 23 and 92.

Anon. (2002a). Annual Report 2001, Mauritius Sugar Industry Research Institute, 25.

Anon. (2002b). *Proceedings of South African Sugar Technologists' Association*. 76:35.

Anon. (2003). Annual Report 2002, Mauritius Sugar Industry Research Institute, 29.

Anon. (2004). Annual Report 2003, Mauritius Sugar Industry Research Institute, 30.

Anon. (2005a). Annual Report 2004, Mauritius Sugar Industry Research Institute, 29 and 92.

Anon. (2005b). *Proceedings of South African Sugar Technologists' Association*. 79:23.

Anon. (2006). Annual Report 2005, Mauritius Sugar Industry Research Institute, 28.

Anon. (2007). Annual Report 2006, Mauritius Sugar Industry Research Institute, 2, 87-90.

Arceneaux G. and L.G. Davidson (1944). Some effects of trash in cane on milling results. *The Sugar Bulletin.* 22(18):151-158.

Arslan N. and H. Toğrul (2005). Modelling of water sorption isotherms of macaroni stored in a chamber under controlled humidity and thermodynamic approach. *Journal of Food Engineering.* 69:133-145.

Atchison J.E. (1971). Review of bagasse depithing. *Proceedings of International Society of Sugar Cane Technologists*. 14:1202-1217.

Atherton P.G., J.C. Dow, P.J. Mathew, A.G. Noble and D.R. Ridge (1992). The determination of soil in sugar cane. *Proceedings of Australian Society of Sugar Cane Technologists.* 14:18-23.

Ayranci E. and O. Duman (2005). Moisture sorption isotherms of cowpea (*Vigna unguiculata* L. Walps) and its protein isolate at 10, 20 and 30 °C. *Journal of Food Engineering.* 70:83-91.

Behne E.R. (1937). Milling tests. *Bureau of Sugar Experiment Stations Technical Communication.* No. 7, p 157.

Bell R.P. (1937). Relationships between the energy and entropy of solution and their significance. *Transaction of Faraday Society*. 33:496-501.

Bennett M.C, (1957). The physical nature and behaviour of cane sugar juice. *International Sugar Journal.* 208-212.

Bennett M.C. (1975). Flocculation technology in sugar manufacture. *Sugar Industry Technologists, paper* 381:22-32.

Beristain C.I., H.S. Garcia and E. Azuara (1996). Enthalpy-entropy compensation in food vapour adsorption. *Journal of Food Engineering.* 30:405-415.

Berlin E. (1981). Hydration of milk proteins, *water activity: Influences on Food Quality*, Rockland, L.B. and G.F. Steward (ed) (Academic Press, New York, USA). pp 467-488.

Bernhardt H.W. (1994). Dry cleaning of sugar cane – a review. *Proceedings of South African Sugar Technologists' Association*. 68:91-96.

Bernhardt W. (1998). Handling properties of sugar cane bagasse I. Characterisation of bagasse particles. *Zuckerindustrie* 123 No. 6, 428-432.

Berry S.L. and M.L. Roderick (2005). Plant-water relations and the fibre saturation point. *New Phytologist.* 168:25-37.

Blanchi R.H. and A.G. Keller (1952). Clean, fresh cane, how much is it worth. *Bulletin No. 28, Eng. Expt. St.* Louisiana State University.

Bosquet R., J. Chirife and H. Iglesias (1978). Equations for fitting water sorption isotherms of foods II. Evaluation of various two-parameter models. *Journal of Food Technology.* 13:319-327.

Bradley R.S. (1936). Polymolecular adsorbed films. Part I. The adsorption of argon on salt crystals at low temperatures and the determination of surface fields. *Journal of the American Chemical Society*. 77:1467-1474.

Brokensha M.A. and P. Mellet (1977). Sampling and analysis of prepared cane for its ash content with reference to estimating soil levels in cane. *Proceedings of South African Sugar Technologists' Association.* 51:97-100.

Brooker D.B., F.W. Bakker-Arkema and C.W. Hall (1992). Drying and storage of grains and oilseeds. *Avi, van Nostrand Reinhold*, New York.

Brotherton G.A. (1980). The influence of extraneous matter on CCS. *Proceedings Australian Society of Sugar Cane Technologists.* 2:7-12.

Brown Jr. C.A. and R.E. Blouin (1907). Chemistry of the sugar cane and its products in Louisiana. *Louisiana State University Experimental Station, Bulletin No. 91,* p 3.

Browning B.L. (1963). The wood-water relationship. In: Browning B.L., ed. *The chemistry of Wood.* New York, USA: *Interscience*. 405-439.

Brunauer S. (1945). The adsorption of gases and vapours. vol I. Princeton University Press, Princeton NJ.

Brunauer S., L.S. Deming, W.S. Deming and E. Teller (1940). *Journal of American Chemical Society.* 62:1723.

Brunauer S., P.H. Emmett and E. Teller (1938). Adsorption of gases in multimolecular layer. *Journal of American Chemical Society.* 60:309-319.

Bruijn J. (1963). Brix-free water in cane fibre. *Sugar Milling Research Institute Quarterly Bulletin* No. 25:43-47.

Cabrer A., F.A. Thillet and W. Clinchard (1965). Sugar cane washing at Central Roig and Central Cortada. *Proceedings of International Society of Sugar Cane Technologists*. 12:1529-1538.

Cargill J.M. (1976). Stop – Hulett's "stalk only please" performance. *Proceedings of South African Sugar Technologists Association*. 66:220-223.

Caurie M. (1970). A new model equation for predicting safe storage moisture levels for optimum stability of dehydrated foods. *Journal of Food Technology.* 5:301-307.

Caurie M. (1981). Derivation of full range moisture sorption isotherms. In L.B. Rockland and G.F. Stewart (ed). *Water activity: influences on food quality*. pp 63-87. Academic Press, New York, USA.

Chen C. and R.V. Morey (1989). Comparison of four EMC/ERH equations. *Transactions of the ASAE.* 32(3):983-990.

Chen J.C.P. and C.C. Chou (1993). Cane sugar handbook – a manual for cane sugar manufacturers and their chemists. 12th edition. John Wiley and Sons Inc. pp 1090.

Chinsamy S., S.B. Davis, P. Sahadeo and N. Kalidass (2004). The development of an automated pith-fibre separator for the cane sugar industry.

Chirife H.A. and J. Iglesias (1978). Equations for fitting water sorption isotherms of foods. 1. A review. *Journal of Food Technology.* 13:159-174.

Clarke M.A. and B.L. Legendre (1996). Sugar cane quality: Impact on sugar yield and quality factors. *Sugar Y. Azucar.* July:30-36.

Clarke S.J. (2003). Cane quality. *Sugar Journal*. May, 8.

Clayton J.E. and D.L. Roberts (1971). Methods of dry cleaning sugar cane in the handling process. *Proceedings of American Society of Sugarcane Technologists*. 1:164-169.

d'Espaignet J.T. (1974). Review of performance of sugar factories in 1973. *Revue Agricole et Sucrière de l'Ile Maurice*. 53(1&2):19-26.

d'Espaignet J.T. (1977). Review of performance of sugar factories in 1976. *Revue Agricole et Sucrière de l'Ile Maurice*. 56(1):27-33.

Day D.L. and G.L. Nelson (1965). Desorption isotherms for wheat. *Transactions of the ASAE.* 8(2):293-296.

de Beer A.G., M.M.W. Boast and B. Worlock (1989). The agricultural consequences of harvesting sugar cane containing various amounts of tops and trash. *Proceedings of South African Sugar Technologists' Association*. 63:107-110.

de Boer J.H. (1953). The Dynamical Character of Adsorption (Clarendon Press, Oxford, U.K).

Deerr N. (1921). Cane sugar – a textbook on the agriculture of the sugar cane, the manufacture of cane sugar, and the analysis of sugar-house products. 2nd edition. London: Norman Rodger. 644 p.

Dincer T.D. and A. Esin (1996). Sorption isotherms for macaroni. *Journal of Food Engineering.* 27:211-228.

Downing C.M and G.D. McBain (2000). Towards understanding the pressure dependence of hygroscopic water. *Proceedings of Australian Society of Sugar Cane Technologists.* 22:361-367.

Dubinin M.M. (1960). Chem. Rev., 60:235.

Emmett P.H. and S. Brunauer (1934). *Journal of American Chemical Society.* 56:35.

Erbas M., M.F. Ertugay and M. Certel (2005). Moisture sorption behaviours of semolina and farina. *Journal of Food Engineering.* 69:191-198.

Foster D.H. (1956). Analytical methods used in assessing mill tandem performance. *Proceedings of International Society of Sugar Cane Technologists.* 9(2):426-436.

Foster D.H. (1962). Fibre water and juice. *Proceedings of Queensland Society of Sugar Cane Technologists.* 29:179-184.

Foster D.H. (1963). A tentative method of analysis of cane. *Proceedings of Queensland Society of Sugar Cane Technologists.* 30:163-165.

Freundlich C. (1907). *Zeitschrift für physikalische chemie.* 57:385.

Freundlich C. (1926). Colloid and capillary chemistry. English translation from 3rd German edition by E.S. Hatfield, Methuen & Co, pp 111, 134.

Gal S. (1981). Techniques for obtaining complete sorption isotherms. In Rockland L.B. and G.F. Steward (ed). *Water activity: Influences on Food Quality*. pp 89-154. Academic Press, New York, USA.

Graham W.S. and J.R. Gunn (1971). Fibre – its effects on milling and processing efficiency. *Proceedings of South African Sugar Technologists Association.* 45:72-74.

Grillet Y., J. Rouquerol and F. Rouquerol (1977). Study of physical adsorption of gases by a continuous procedure. *Le Journal de Chimie Physique et de Physico-Chimie biologique.* 74(2):179.

Guggenheim E.A. (1966). Application of Statistical Mechanics (Clarendon Press, Oxford, U.K).

Hailwood A.J. and S. Horrobin (1946). Absorption of water by polymers: Analysis in terms of a simple model. Transactions of Faraday Society B42, 84-102.

Halsey G. (1948). Physical adsorption on non-uniform surfaces. *Journal of Chemical Physics.* 16:931-937.

Han G. and Q. Wu (2004). Comparative properties of sugar cane rind and wood strands for structural composite manufacturing. *Forest Products Journal.* 54(12):283-288.

Hatakeyama T., K. Nagamura and H. Hatakeyama (2000). Vaporisation of bound water associated with cellulose fibres. *Thermochimica Acta* 352-353, 233-239.

Hemaida S.E.A., G.E. Sayed and A.A. El-Badawi (1977). Effect of green and dry trash on cane and milling qualities. *Proceedings of International Society of Sugar Cane Technologists.* 16(3):2485-2491.

Henderson S.M. (1952). A basic concept of equilibrium moisture. *Agricultural Engineering.* 33(1):29-33.

Herbert L.P. (1973). Testing of sugar cane varieties for milling quality. *Sugar Journal*. 36:4.

Heyrovsky J. (1970). Determination of isokinetic temperature. *Nature.* 227:66-67.

Hossain M.D., B.K. Bala, M.A. Hossain and M.R.A. Mondol (2001). Sorption isotherms and heat of sorption of pineapple. *Journal of Food Engineering.* 48:103-107.

Igathinathane C., A.R. Womac, S. Sokhansanj and L.O. Pordesimo (2005). Sorption equilibrium moisture characteristics of selected corn stover components. *American Society of Agricultural Engineers.* 48(4):1449-1460.

Iglesias H.A. and J. Chirife (1976). Isosteric heats of water vapour sorption in dehydrated foods. Part 1. Analysis of differential heat curve. *Lebensmittel-Wissenschaft und Technologie.* 9:116-122.

Iglesias H.A. and J. Chirife (1981). An equation for fitting uncommon water sorption isotherms in foods. *Lebensmittel-Wissenschaff und Tecnolgie.* 14:111-117.

Iglesias H.A., J. Chirife and Ferro Fontán (1989). On the temperature dependence of isosteric heats of water sorption in dehydrated foods. *Journal of Food Science.* 54(6):1620-1631.

Jarman C. (1998). Small-scale textiles – plant fibre processing – A handbook. *Intermediate Technology Publications.* pp 52.

Jayas D.S. and G. Mazza (1993). Comparison of five three-parameter equations for the description of adsorption data of oats. *Transactions of the ASAE.* 36(1):119-125.

Jhingooree D., R. Jeetah and U. Kokil (2000). Degumming of sugar cane fibres from sugar cane stalk. *Revue Agricole et Sucrière de l'Ile Maurice*. 79(2):32-41.

Kaleemullah S. and R. Kailappan (2007). Monolayer moisture, free energy change and fractionation of bound water of red chillies. *Journal of Stored Products Research*. 43:104-110.

Kampen W.H. (1974). The effect of field soil on sugar cane clarification. *Sugar Y Azucar.* August 18-20.

Kaymak-Ertekin F. and A. Gedik (2004). Sorption isotherms and isosteric heat of sorption for grapes, apricots, apples and potatoes. *Lebensmittel-Wissenchaft und Technologie.* 37:429-438.

Keller A.G. (1966). Methods for depithing sugar cane bagasse – A review. *Sugar Journal*. 28(11)26-33.

Keller A.G. and F.C. Schaffer (1951). The effect of cane trash on the milling operation. *Bulletin No. 25, Eng. Expt. St.* Louisiana State University.

Kelly F.H.C. (1957). Water adsorption on sugar cane fibre. *International Sugar Journal.* 59:36-39.

Kelly F.H.C. and B.J. Rutherford (1957). Water adsorption on sugar cane fibre. Part II. *International Sugar Journal.* 59:152-155.

Kent G.A., C.P. Hoare, J.F. Miller and W.J. Allen (2003). Harvest and factory data to assist in evaluating the effect of extraneous matter on sugar production costs and revenues. *Proceedings of Australian Society of Sugar Cane Technologists* (CD-ROM), 25:15p.

Kent G.A., W.J. Allen, S.P. Hoare and T.F. Dixon (1999). The effect of extraneous matter on factory throughput and performance. *Proceedings of Australian Society of Sugar Cane Technologists*. 21:38-43.

King N.J. (1969). Historical review of sugar cane mechanization in Australia. *Proceedings of South African Sugar Technologists' Association*. 43:93-98.

Kollmann F.P. and A. Schneider (1963). Principles of wood science and technology, I, solid wood. Edited by F.P. Kollmann, W.A. Cote Jr (1968): 194-195, New York Springer-Verlag.

Kong Win Chang K.T.K.F. and L. Wong Sak Hoi (1999). Plant data of Mauritian sugar factories. *Mauritius Sugar Industry Research Institute Technical Circular, Sugar Technology Series.* No. 21, 31 p.

Kong Win Chang K.T.K.F., L.J.C. Autrey and L. Wong Sak Hoi (2001). Production of electricity from bagasse in Mauritius. *Proceedings of International Society of Sugar Cane Technologists*. 24(1):282-286.

Kuhn I.J. (1964). A new theoretical analysis of adsorption phenomena. Introductory part: The characteristic expression of the main regular types of adsorption isotherms by a single simple equation. *Journal of colloid science*. 19:685-698.

Kumar A.J., R.R.B. Singh, G.R. Patil and A.A. Patel (2005). Effect of temperature on moisture desorption isotherms of kheer. *LWT* 38, 303-310.

Labuza T.P. (1968). Sorption phenomenon in foods. *Food Technology.* 22, 263-272.

Labuza T.P. (1975). Interpretation of sorption data in relation to the state of constituent water. In R. Duckworth (Ed.) Water Relations in Foods (pp 155-172) New York. Academic Press.

Labuza T.P. (1980). The effect of water activity on reaction kinetics of food deterioration. *Food Technol.* April:36-59.

Labuza T.P. (1984). Moisture sorption : Practical aspects of isotherm measurement and use. Minneapolis, MN : *American Association of Cereal Chemists*.

Labuza T.P. and C.G. Busk (1979). An analysis of the water binding in gels. *Journal of Food Science.* 44:1379-1385.

Lamusse J.P. and S. Munsamy (1979). Extraneous matter in cane and its effect on the extraction plant. *Proceedings of South African Sugar Technologists Association.* 53:84-89.

Langmuir I. (1916). *Journal of American Chemical Society.* 38:2221.

Langmuir I. (1917). *Journal of American Chemical Society.* 39:1848.

Langmuir I. (1918). *Journal of American Chemical Society.* 40:1361.

Larrahondo J.E., C.O Briceno, C. Vikeros, J. Cock, A. Palma, A. Navarrete and O. Ospina (1998). Evaluation of cane trash. *International Sugar Journal.* Vol. 100 No. 1200, 587-591.

Lee E.F.T. and M. Donovan (1996). Filterability of raw sugars. *Proceedings of International Society of Sugar Cane Technologists.* 22(1):195-204.

Legendre B.L. (1991). Increasing sugar recovery: the case for improved sugar cane quality. *Sugar Journal.* 53(9):9-18.

Legendre B.L. and J.E. Irvine (1974). Some effects of cane trash on milling quality of sugar cane. *Proceedings of American Society of Sugar Cane Technologists.* 3:167-173.

Lionnet G.R.E. (1986). Post-harvest deterioration of whole stalk sugar cane. *Proceedings of South African Sugar Technologists Association.* 60:52-57.

Lionnet G.R.E. (1992a). A review of some aspects of cane quality in South Africa. *Revue Agricole et Sucrière de l'Ile Maurice.* 71(2 & 3):79-87.

Lionnet G.R.E. (1992b). The effect of tops and trash on cane diffusion based on trials at Gledhow. *Sugar Milling Research Institute Technical Report* No. 1554, 5 Oct 1989, 7 p.

Lionnet G.R.E. (1994). Sixty-ninth annual review of the milling season in South Africa (1993-1994). *Proceedings of South African Sugar Technologists' Association*. 68:xix-xxxix.

Lionnet G.R.E. (1996). A review of cane quality in South Africa and its effect on factory performance. *Proceedings of International Society of Sugar Cane Technologists.* 22(1):103-113.

Lionnet G.R.E. and K. Koster (1986). A boiling house recovery formula independent of juice quality. *Proceedings of South African Sugar Technologists Association.* 60:30-32.

Lionnet R. and P. Wagener (1976). Determination of sand in prepared cane by an ashing method. Internal report, Hullets Sugar Limited, Research and Development.

Lomauro C.J., A.S. Bakshi and T.P. Labuza (1985). Evaluation of food moisture sorption isotherm equations Part I. Fruit, vegetable and meat products. *Lebensmittel-Wissenschaff und Technologie.* 18(2):111-117.

Lopes Filho J.F., P.F. Romanelli, S.H.R. Barboza, A.L. Gabas and J. Felis-Romero (2002). Sorption isotherms of alligator's meat. *Journal of Food Engineering.* 52:201-206.

Madamba P.S., R.H. Driscoll and K.A. Buckle (1996). Enthalpy-entropy compensation models for sorption and browning of garlic. *Journal of Food Engineering.* 28:109-119.

Mangion M.J. and M.R. Player (1991). Hygroscopic water in sugar cane. *Proceedings of Australian Society of Sugar Cane Technologists.* 13:285-290.

Maskan M. and F. Göğüs (1997). The fitting of various models to water sorption isotherms of pistachio nut paste. *Journal of Food Engineering*. 33:227-237.

Mathew P.J., F.G. Fuelling, J.C. Dow, P.L. Phillips and A.G. Noble (1994). A soil monitor for sugar cane. *Proceedings of Australian Society of Sugar Cane Technologists*. 16:307-313.

Mazza G. and M. LeMaguer (1978). Water sorption properties of yellow globe onion (Allium cepa L.) *Canadian Institute of Food Science and Technology Journal*. 11(4):189-193.

McBain J.W. and A.M. Bakr (1926). *Journal of American Chemical Society.* 48:690.

McLaughlin C.P. and T.R.A. Magee (1998). The determination of sorption isotherm and isosteric heat of sorption for potatoes. *Journal of Food Engineering.* 35:267-280.

McMinn W.A.M. and T.R.A. Magee (2003). Thermodynamic properties of moisture sorption of potato. *Journal of Food Engineering.* 60:157-165.

Mizrahi S., T.P. Labuza and M. Karel (1970). Computer-aided predictions of extent of browning in dehydrated cabbage. *Journal of Food Science*. 35(6):799-803.

Moodley M. (1991). The pith and fibre contents of cane. *Sugar Milling Research Institute Technical Report* No. 17, Jan 1991, 7 p.

Moreira R., G. Vázquez, F. Chenlo and J. Carballal (2001). Desorption isotherms of eucalyptus globulus modelling using GAB equation. *Electronic Journal of Environmental, Agricultural and Food Chemistry.* ISSN:1519-4377.

Muller R.L., M.R. Player and M.B. Wise (1982). Input, disposition and effect of dirt in Queensland sugar mills. *The Sugar Journal*. 9-13.

Nelsen F.M. and F.T. Eggertsen (1958). Analytical chemistry 30:1387.

Nelson R.M. (1983). A model for sorption of water vapour by cellulosic materials. *Wood and Fibre Science.* 15(1):8-22.

Nilsson D., B. Svennerstedt and C. Wretfors (2005). Adsorption equilibrium moisture contents of flax straw, hemp stalks and reed canary grass. *Biosystems Engineering.* 91(1):35-43.

Noguchi H. (1981). Hydration around hydrophobic groups. In L.B. Rockland and G.F. Steward (Eds). *Water activity: Influences on Food Quality*. pp 281-293. Academic Press, New York, USA.

Norimoto M. and T. Yamada (1977). Dielectric behaviour of water adsorbed on MWL. *Mokuzai Gakkaishi.* 23(2):99-196.

Oguri (1932). *J. Soc. Chim. Ind. Japan.* 35:279

Olson B.C., M.P. Coleman and T.G. Fuelling (1999). A commercial monitor for soil in sugar cane. 21:443-448.

Oswin C.R. (1946). The kinetics of package life. III Isotherm. *Journal of the Society of Chemical Industry.* 65:419-421.

Parish D.H. and S.M. Feillafé (1965). Notes on the 1:100000 soil map of Mauritius. *Mauritius Sugar Industry Research Institute Occasional Paper No. 22*. 43 p.

Paturau J.M. (1989). By-products of the cane sugar industry, an introduction to their industrial utilization: *Elsevier Publishing Co., Amsterdam.* 37-42 pp 435.

Peleg M. (1993). Assessment of a semi-empirical four-parameter general model for sigmoid moisture sorption isotherms. *Journal of Food Process Engineering.* 16:21-37.

Perry R.H. and C.H. Chilton (1973). Chemical Engineers' Handbook, 5th edition, McGraw Hill Ltd. 3-64, 2545 p.

Perry R.H., D.W. Green and J.O. Maloney (1984). Perry's Chemical Engineers' Handbook, 6th edition, McGraw Hill Inc. 3-65, 3-69, 2719 p.

Pieters W.J.M. and W.E. Gates (1984). U S Patent 4489, 593.

Prinsen Geerligs, H.C. (1897). Studiën over Ampas, Archief voor de Java. *Suikerindustrie*. Vol. V. p 369.

Prinsen Geerligs, H.C. (1904). Chemical control in use in the Java factories, 437-444.

Prothon F. and L.M. Ahrné (2004). Application of the GAB model to correlate water activity and moisture content during osmotic dehydration of apples. *Journal of Food Engineering.* 61:467-470.

Qin L.J. and E.T. White (1991). A press method for evaluating hygroscopic water for cane fibres. *Proceedings of Australian Society of Sugar Cane Technologists.* 13:291-297.

Qojewska J., P. Miskowiec, T. Qojewski and L.M. Proniewicz (2005). Cellulose oxidative and hydrolytic degradation: *in situ* FTIR approach. *Polymer degradation and stability.* 88(2005):512-520.

Quioco F.A., D.K. Wilson and N.K. Vyas (1989). Substrate specificity and affinity of a protein modulated by bound water molecules. *Nature.* 340:404-407.

Rao K.J., P.H.A. Dhas, F.M.E. Emerald, B.C. Ghosh, B.V. Balasubramanyam and S. Kulkarni (2006). Moisture sorption characteristics of *chhana podo* at 5 °C and 35 °C. *Journal of Food Engineering.* 76:453-459.

Rascio A., C. Platani, G. Scalfati, A. Tonti and N.D. Fonzo (1994). The accumulation of solutes and water binding strength in durum wheat. *Physiologia Plantarum*. 90:715-721.

Rascio A., C. Platini, N. Di Fonzo and G. Wittmer (1992). Bound water in durum wheat under drought stress. *Plant Physiology.* 98:908-912.

Reid M.J. and G.R.E. Lionnet (1989). The effects of tops and trash on cane milling based on trials at Maidstone. *Proceedings of South African Sugar Technologists Association*. 63:3-6.

Rein P.W. (1975). A statistical analysis of the effect of cane quality on extraction performances. *Proceedings of South African Sugar Technologists Association*. 49:43-47.

Rein P.W. (2003). Private communication.

Rein P.W. and I.A. Smith (1981). Molasses exhaustibility studies based on sugar analysed by gas-liquid chromatography. *Proceedings of South African Sugar Technologists Association.* 55:85-90.

Richardson I.J. (1970). Automatic cane sampling and analysis by wet disintegrator. *CSR Internal Report*.

Rizvi S.S.H. and A.L. Benato (1984). Thermodynamic properties of dehydrated foods. *Food Technol*. 38:83-92.

Rouillard A. (2001). Three hundred years of sugar manufacture in Mauritius. *Revue Agricole et Sucrière de l'Ile Maurice.* 79(3):1-67.

Rouquerol F., J. Rouquerol and K. Sing (1999). Adsorption by powders and porous solids: Principles, methodology and applications. *Academic Press USA*. 467 p.

Rouquerol J. (1972). In: Thermochimie, Colloques Internationaux du CNRS, No. 201. Editions du CNS, Paris, p 537.

Rüegg M. (1980). Calculation of the activity of water in sulfuric acid solutions at various temperatures. *Lebensmittel-Wissenschaft und Technologie*. 13:22-24.

Sanchez E.S., R. Sanjuan, S. Simal and C. Rosello (1997). Calorimetric techniques applied to the determination of isosteric heat of desorption for potato. *Journal of Science Food and Agriculture.* 74:57-63.

Sanni L.O., A. Atere and A. Kuye (1997). Moisture sorption isotherms of fufu and tapioca at different temperatures. *Journal of Food Engineering*. 34:203-212.

Schäffler K.J. (Referee) (1994). Subject 8: Chromatographic techniques for sugars. *Proceedings ICUMSA.* 21:294-311.

Scott R.P. (1977). The limitations imposed on crushing rate by tops and trash. *Proceedings of South African Sugar Technologists Association*. 51:164-166.

Simpson E.S.W. (1951). The geology and mineral resources in Mauritius. Colonial Geology and mineral resources. I. 217-238.

Sing K.S.W., D.H. Everett, R.A.W. Haul, L. Moscou, R.A. Pierotti, J. Rouquerol and T. Siemieniewska (1985). *Pure Applied Chemistry.* 57:603.

Singh R.R.B., K.H. Rao, A.S.R. Anjaneyulu and G.R. Patil (2006). Water desorption characteristics of raw goat meat: effect of temperature. *Journal of Food Engineering.* 75:228-236.

Smith S.E. (1947). The sorption of water vapour by proteins and high polymers. *Journal of the American Chemical Society.* 69:646-651.

Smits J.H. and R.L. Blunt (1976). The implication of poor cane quality. *Proceedings of South African Sugar Technologists Association.* 50:227-230.

Snow J.T. (1974). Hard fibre and pith in sugar cane. A simple method for evaluating millability of new sugar cane varieties. *Proceedings of International Society of Sugar Cane Technologists.* 15:1169-1173.

Soysal Y. and S. Öztekin (1999). Equilibrium moisture content equations for some medicinal and aromatic plants. *Journal of Agricultural Engineering Research.* 74(3):317-324.

Spoelstra H.J. (1935). Verhandelingen voor leden van het proefstation. No. 7.

St Antoine J.D. de R. de (1977). Qualité de la matière et son influence sur le fonctionnement et l'efficience des sucreries. *Revue Agricole et Sucrière de l'Ile Maurice.* 56(3):141-145.

Stamm A.J. (1964). Wood and Cellulose Science. New York, USA: The Ronald Press Co.

Stamm A.J. (1967a). Movement of fluids in wood – Part I. Flow of fluids in wood. *Wood Science and Technology.* I:122-141.

Stamm A.J. (1967b). Movement of fluids in wood – Part II. *Diffusion Wood Science and Technology.* I:205-230.

Stamm J. and L.A. Hansen (1938). Surface-bound versus capillary-condensed water in wood. *Journal of Physical Chemistry.* 42:209-214.

Steuerwald L.G.L. (1912). Adsorpbeerend Vermogen van reitgom en reitvezelstof. Adsorptiewater in ampas. Mededeelingen van het Peoefstation Voor de Java – Suikerindustrie. 2:1325.

Sun J.X., X.F. Sun, H. Zhao and R.C. Sun (2004). Isolation and characterisaiton of cellulose from sugar cane bagasse. *Polymer degradation and stability.* 84:331-339.

Telis V.R.N., A.L. Gabas, F.C. Menegalli and J. Telis-Romero (2000). Water sorption thermodynamic properties applied to persimmon skin and pulp. *Thermochimica acta*. 343(1-2):49-56.

Tolaba, M.P., M. Peltzer, N. Enriquez and M.L. Pollio (2004). Grain sorption equilibria of quinoa grains. *Journal of Food Engineering.* 61:365-371.

Tsai Ming Chuin (1973). Sugar cane quality control and sugar yield. *Taiwan Sugar.* 20:18-20.

Tsai W.L., H.H. Ting and M.C. Tsai (1961). A report on the processing of sugar manufacture affected by cane trash. *Taiwan Sugar.* 24-27.

Tsami E., D. Marinous-Kouris and Z.B. Maroulis (1990). Water sorption isotherms of raisins currants, figs, prunes and apricots. *Journal of Food Science*. 55:1594-1597.

Tsami E., Z.B. Maroulis, D. Morunos-Kouris and G.D. Saravacos (1990). Heat of sorption of water in dried fruit. *International Journal of Food Science Technology.* 25:350-359.

Tulip J.R. and W.E. Moore (2007). Trash analysis in billet cane using spectral unmixing. *International Sugar Journal*. Vol. 109, No. 1304:513-520.

Valko E.I. (1943). Cellulose and cellulose derivatives. Ed. Ott E., New York: *Interscience Publishers Inc.*, 379-423.

Van den Berg C. (1984). Description of water activity of foods for engineering purpose by means of the GAB model of sorption. In B.M. McKemenne (Ed.). *Engineering and Food.* vol 1; pp 311-321. London; Elsevier Applied Science.

Van den Berg C. and S. Bruin (1981). Water activity and its estimation in food systems. In Rockland L.B. and F. Stewart (Eds.). Water activity : influence on food quality (pp 147-177). New York : Academic Press.

Van der Pol C., C.M. Young and K. Douwes Dekker. (1957). The determination of certain qualities of individual consignment of cane. *South African Sugar Journal.* 51:544-548.

Van Dillewijn C. (1952). Botany of sugar cane. Waltham, Mass. USA. *The Chronica Botanica Co: Book Department.* pp 371.

Van Hengel A. (1974). Proposal for the evaluation of cane and sugar in identical units at standardized factory efficiency. *Proceedings of International Society of Sugar Cane Technologists.* 15:1446-1455.

Van Soest P.J. (1963). Use of detergents in the analysis of fibrous feeds I. Preparation of fibre residues of low nitrogen content. *Journal of the Association of Official Agricultural Chemists (AOAC).* Vol 46 No. 6, 825-829.

Verma D.R. and D.K. Gupta (1988). Equilibrium moisture content isotherms of sugarcane bagasse. *Journal of Agricultural Engineering*. 25:49-56.

Vertucci C.W. and A.C. Leopold (1984). Bound water in soybean seed and its relation to respiration and imbibitional damage. *Plant Physiology.* 75:114-117.

Vertucci C.W. and C. Leopold (1987). The relationship between water binding and desiccation tolerance in tissues. *Plant Physiology.* 85:232-238.

Vignes E.C. (1980). The implications of cane washing. *Proceedings of International Society of Sugar Cane Technologists*. 21:2221-2230.

Villavicencio E.J. (1974). World market potential for bleached bagasse pulp – a technical review. *Proceedings of International Society of Sugar Cane Technologists.* 15:1783-1792.

Viswanathan R., D.S. Jayas and R.B. Hulasare (2003). Sorption isotherms of tomato slices and onion shreds. *Biosystems Engineering.* 86(4):465-472.

Wang N. and J.G. Brennan (1991). Moisture sorption isotherm characteristics of potatoes at four temperatures. *Journal of Food Engineering.* 14:269-282.

Weisser H. (1986). Influence of temperature on sorption isotherms. In *Food Engineering and Process Applications* Vol 1. Edited by M. LeMaguer and P. Jelen. Elsevier Applied Science Publishers. New York. 189-199.

Westgate P., J.Y. Lee and M.R. Ladisch (1992). Modelling of equilibrium sorption of water vapour on starchy materials. *American Society of Agricultural Engineers*. 35(1):213-219.

Whayman E. (1992). Cane juice purification – a review of techniques for clarification and filtration. *Revue Agricole et Sucrière de l'Ile Maurice*. 71(2&3):218-230.

Wong Sak Hoi L. (1997). Rapid moisture determination using halogen thermogravimetry. *International Sugar Journal*. Vol. 99, No. 1185:435-440.

Wong Sak Hoi L. (1998). Halogen thermogravimetric moisture determination in filter cake, bagasse and cane. *International Sugar Journal*. Vol. 100, No. 1191E:102-106.

Wong Sak Hoi L. (2001). A review of 2000 milling season. *Revue Agricole et Sucrière de l'Ile Maurice.* 80(1 & 2):32-39.

Wong Sak Hoi L. (2003). A review of 2001 milling season. *Revue Agricole et Sucrière de l'Ile Maurice.* 80(3), 81(1, 2 & 3):329.

Wong Sak Hoi L. and J. Chung (1996). Clarification studies leading to adoption of saccharate liming in a Mauritian sugar factory. *Proceedings of International Society of Sugar Cane Technologists*. 22(1):172-180.

Wong Sak Hoi L. and L.J.C. Autrey (1997). Trends in cane quality and its effect on some aspects of sugar processing in Mauritius. *Revue Agricole et Sucrière de l'Ile Maurice.* 76(3):1-8.

Wong Sak Hoi L. and L.J.C. Autrey (1998). Deterioration in cane quality and its impact on factory performance. *Revue Agricole et Sucrière de l'Ile Maurice.* 77(2 & 3):8-19.

Wong Sak Hoi L., J. Lim Shin Chong and L.J.C. Autrey (1999). A rapid method of estimating extraneous matter in a cane consignment. *Revue Agricole et Sucrière de l'Ile Maurice.* 78(3):8-11.