



Faculty of Resource Science and Technology

**CHEMICAL AND MORPHOLOGICAL PROPERTIES OF STEM
COMPONENTS FROM *HIBISCUS SABDARIFFA* L.**

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FROM *Hibiscus sabdariffa* L.

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ABSTRACT

This study was performed to determine the chemical and morphological properties for stem components of *Hibiscus sabdariffa* L. (commonly called as roselle). The collected roselle samples were prepared into bast, core and the whole plant. For chemical analysis, the samples were in the form of grounded samples that retained on a BS60 or 250 μ m mesh sieve. *Acacia mangium* was also prepared as a control sample. Chemical compositions determined were cold water solubility, hot water solubility, alkali solubility, ethanol-acetone solubility, lignin, holocellulose, alpha-cellulose and ash content. Meanwhile, for morphological determination, the length, width and single wall thickness of the fibres for each specified components of the roselle stems were measured and calculated. The results showed that all of the chemical compositions for the roselle stem components and *Acacia mangium* were significantly different at confidence level of 95% except for holocellulose and alpha-cellulose. Correspondingly, morphological analysis stated that all of the fibre properties were significantly different at confidence level of 95%.

Keywords: *Hibiscus sabdariffa* L.; *Acacia mangium*; chemical composition; morphological determination

ABSTRAK

Kajian ini telah dijalankan bagi menentukan kandungan kimia dan ciri-ciri morfologi bagi komponen-komponen batang *Hibiscus sabdariffa* L. (biasanya dipanggil sebagai rozel). Sampel rozel yang diambil diasingkan kepada kulit, teras dan keseluruhan tumbuhan. Untuk analisis kimia, sampel-sampel yang digunakan adalah dalam bentuk serbuk yang diperolehi dari pengayak BS60 atau bersaiz 250 μ m. *Acacia mangium* juga disediakan sebagai sampel kawalan. Komposisi kimia yang telah ditentukan adalah keterlarutan air sejuk, keterlarutan air panas, keterlarutan alkali, keterlarutan etanol-aseton, kandungan lignin, holoselulosa, alfa-selulosa dan abu. Manakala, bagi analisis morfologi pula, kepanjangan, lebar serta ketebalan gentian bagi setiap komponen batang rozel yang telah ditetapkan, diukur dan dikira. Keputusan yang telah diperolehi menunjukkan kesemua kandungan bahan kimia bagi komponen-komponen batang rozel tersebut dan *A. mangium* adalah berbeza secara signifikan pada tahap keyakinan 95%, kecuali bagi holoselulosa dan alfa-selulosa. Analisis morfologi turut menyatakan bahawa ciri-ciri morfologi gentian kesemua sampel adalah berbeza secara signifikan pada tahap keyakinan 95%.

Kata kunci: *Hibiscus sabdariffa* L.; *Acacia mangium*; komposisi kimia; penentuan morfologi

INTRODUCTION

Lignocellulosic material is composed principally of carbon, hydrogen and oxygen (Haygreen and Bowyer, 1996). It is also a composite material consisting of three elements, that is cellulose, hemicellulose and lignin (Schniewind, 1989). Among the methods exist to evaluate the materials are the chemical analysis and morphological determination.

Chemical analysis is capable of differentiating the softwood (gymnosperm) and hardwood (angiosperm) even though the chemical composition is basically differ depending on the part of the tree (root, trunk or branch), types of wood, geographical climate and soil. However, Roger (1984) stated that this technique is less effective to classify each of the tree species because there is vary within the species and overlapping between most of the species.

Morphological determination through analysis of fibre length, width and thickness, enable us to classify the stem component according to their application. According to Foulger (1969), long fibres make certain species of plants desirable for papermaking, especially where strength is needed. Shorter and flatter ones are good for writing papers where a smooth surface is more necessary than strength.

OBJECTIVES

With increasing interest in non-wood pulping in this country, chemical and morphological analysis are able to provide data or information on the chemical composition and fibre properties especially on roselle since there is deficiency in information about this plant species. Therefore the objectives of this study were:

1. To determine the chemical compositions of different roselle stem components (whole plants, core and bast).

2. To determine the morphological properties of different roselle stem components (whole plants, core and bast).

LITERATURE REVIEW

Hibiscus sabdariffa L

Hibiscus sabdariffa L. (roselle) is an erect, glabrous, annual shrub of the Malvaceae family and is believed to have its origin from tropical Africa. It has been grown in India, Africa, and America since eighteenth century. It is currently being cultivated in Egypt, Sri Lanka, and tropical areas of Mexico. This plant also known as Jamaica sorrel, roselle, red Sorrel, Guinea sorrel and locally as Asam Susur (George and Roger, 1998).

The shrub grows to 3 metres tall and has glabrous leaves, which are palmately lobed with serrated margins and which are carried on long petioles. The flowers are showy (yellow or red in colour), and are borne singly on very short peduncles in the axils of the upper leaves. The calyces of the flowers are reddish and fleshy, and can be harvested and made into a red syrup for cold drinks. The seeds contain 17% oil similar in properties to cotton seed oil. The shrub is propagated by seeds (George and Roger, 1998).

Chemical components

The carbohydrate portion of the vast majority of plants is composed of cellulose and hemicellulose polymers with minor amounts of other sugar polymers such as starch and pectins. The combination of cellulose and the hemicelluloses are called holocellulose and usually accounts for 65–70 percent of the plant dry weight. These polymers are made up of simple sugars, mainly, D-glucose, D-mannose, D-galactose, D-xylose, L-arabinose, D-glucuronic acid, and lesser amounts of other sugars such as L-rhamnose and D-fucose. These

polymers are rich in hydroxyl groups, which are responsible for moisture sorption through hydrogen bonding.

Cellulose

Cellulose is the most abundant organic chemical on the face of the earth. It is a glucan polymer of D-glucopyranose units, which are linked together by β - (1—4)-glycosidic bonds. Actually the building block for cellulose is cellobiose, since the repeating unit in cellulose is a two-sugar unit (Han & Rowell, 1997). A polysaccharide composed solely of glucose units linked by β (1-4) glycosidic bonds. It is most abundant in cell wall and structural polysaccharide in the plant kingdom and probably the most abundant of all compounds found in living organisms. In the plant cell walls it is present in highly organised microfibriles, the formation and organisation of which is not fully understood at present (Elizabeth, 1984).

Synthesis of cellulose is thought to take place in the golgi apparatus. Enzymes capable of cellulose formation from primary molecules and nucleotide diphosphate derivatives of glucose (e.g. UDP and GDP-glucose) have been found in particulate cell fractions arising from the golgi apparatus. Study of cellulose synthetic enzymes has proved difficult because the enzymes are tightly membrane bound (Elizabeth, 1984). According to Schniewind (1989), normal softwoods and hardwoods contain on the average $42 \pm 2\%$ cellulose. Cellulose is divided into two parts which comprises of α -cellulose and β -cellulose. α -cellulose is the cellulose fraction insoluble in room temperature 17.5% sodium hydroxide wash water, while β -Cellulose is the cellulose fraction in the alkaline wash water which precipitates upon neutralisation.

Hemicellulose

Hemicellulose is any of variety of polysaccharide found in plant cell walls often in close association with cellulose. Hemicelluloses differ from cellulose in being composed of pentose sugars (arabinose or xylose) or hexose sugars other than glucose (e.g. mannose and galactose). They can be broken down by enzymes and so act as a nutrient reserve. Cellulose by contrast is metabolically inactive once it has been incorporated into the cell wall (Elizabeth, 1984). According to Haygreen and Bowyer (1996), softwoods contain 20-32% hemicellulose and hardwoods contain 15-35% hemicellulose (on dry weight basis).

Extractives

The extractives are a group of cell wall chemicals mainly consisting of fats, fatty acids, fatty alcohols, phenols, terpenes, steroids, resin acids, rosin, waxes, and so on. These chemicals exist as monomers, dimers, and polymers. They derive their name as chemicals that are removed by one of several extraction procedures.

Lignin

Lignins are amorphous, highly complex, mainly aromatic, polymers of phenylpropane units. Lignins can be classified in several ways but they are usually divided according to their structural elements (Sjöström, 1981). All plant lignins consist mainly of three basic building blocks of guaiacyl, syringyl, and *p*-hydroxyphenyl moieties. It is also described as a hard substance found in the thickened cell walls of xylem and sclerenchyma, a complex carbohydrate polymer making up about 25% of wood of trees and also found in the cell walls of sclerenchyma tissues and vessels, fibres and tracheids at maturity. It increases them more resistant to compression and tension.

Lignin is formed by condensation of the phenolic compound coniferyl alcohol. Its distribution in tissues can be shown by staining with acidified phloroglucin, which turns lignin red (Elizabeth, 1984). Lignin in wood is distributed throughout the secondary cell wall with the highest concentration in the middle lamella. Because of the difference in the volume of middle lamella to secondary cell wall, about 70% of the lignin is located in the secondary wall (Han & Rowell, 1997). According to Schniewind (1989), most softwoods contain $30 \pm 4\%$ lignin and hardwoods of the temperate zone $25 \pm 3\%$. Determination of lignin content in wood and pulp provides information for evaluation and application of the processes. Lignin content is interrelated with pulp properties such as colour, hardness, bleachability and other.

Ash

Ash is an inorganic content of plants that remain after high-temperature combustion in the presence of abundant oxygen. It is traceable to the occurrence of fireproof compounds that contain several constituents such as calcium, potassium, manganese, magnesium and silicon (Haygreen and Bowyer, 1996). This inorganic compound is usually referred to as ash content, which is an approximate measure of the mineral salts and other inorganic matter in the fibre after combustion at a temperature of 575°C . The inorganic content can be quite high in plants containing large amounts of silica. Ash is specifically defined as the residue remaining after ignition at 575°C for three hours or longer to burn off all the carbon. . It is a measure of mineral salts in the wood, but it is not necessarily quantitatively equal to them.

Fibre, like wood and pulp, is ashed at a lower temperature than paper (925°C) to minimise the volatilisation of inorganic compounds (Han & Rowell, 1997). It is important from the perspective of utilisation, which imply to low ash content which also mean low silica content in domestic wood, for the reason that woods with a silica content of more than 0.3% (on a dry weight basis) blunt cutting tools extremely (Haygreen and Bowyer, 1996). The

inorganic constituents (ash) generally amount to not more than 0.1-0.5% of the wood (Schniewind, 1989).

Morphological Properties

Fibre is generally described as long, narrow cell of wood or bark, other than vessels or parenchyma. The wood fibres include both the tracheids of softwoods and the fibres of hardwoods (Foulger, 1969). Fibre is also defined as a type of cell in which the wall has been thickened to perform a structural role. Typically, fibres are elongated sclerenchyma cells with tapered ends and have fewer cavities in the thickenings than do other types of thickened cells, though neither feature is diagnostic. Such cells can be found in the cortex, phloem and xylem (Allaby, 1998).

In general, fibres can be classified into wood and non-wood categories. The term “non-wood” was coined to distinguish plant fibres from the two main sources of wood fibres, hardwoods and softwoods. Non-wood and agro-based fibres are derived from selected tissues of various mono- or dicotyledonous plants (Parham & Kausftinen, 1974) and are categorised botanically as grass, bast, leaf, or fruit fibres. Some non-wood fibres are classified by means of production as byproducts. Other non-wood fibres are grouped as “fibre plants,” plants with high cellulose content that are cultivated primarily for the sake of their fibres. Non-wood fibres can be used to make paper, although the quality varies a great deal depending on the source of the fibres. The high cellulose content of cotton linter (85% to 90%) compared to that of wood (35% to 49% cellulose) and the low lignin content of hemp (3%) make these non-wood fibres valuable for papermaking. Although there are some drawbacks to non-wood pulping, most non-wood fibres are bulky and vulnerable to biological deterioration during storage (Han, 1998).

Roselle is grouped as non-wood fibres, which are formed in aggregates or bundles. Non-wood fibres have great variations in chemical and morphological properties. Morphological properties are important to the understanding of non-wood fibres. Fibre length is the most important of these properties for pulping. The low lignin content indicates that non-wood fibres will require very mild pulping conditions.

MATERIALS AND METHODS

Collection and preparation of roselle stem components

Roselle plants were collected from roselle plantation and prepared for chemical analysis and fibre morphological determination according to their stem components; bast (outer layer), core (inner layer) and the whole plant (bast and core). For the chemical analysis, the stem of roselle were grinded with Wiley Mill to get grounded sample which pass a BS 40 mesh sieve and retained on a BS 60 mesh sieve. The grounded sample was air dried for at least one day before chemical analysis. Besides that, some of the stem components were reduced to matchstick-size sample for morphological determination. As a comparison, *Acacia mangium* was used.

Chemical analysis

Determination of cold-water solubility (TAPPI Standard T207)

The analysis was carried out in duplicate. About 2g of air-dried grounded sample was weighed out accurately to 0.001g in a weighing bottle. The sample was transferred into a 400mL beaker and 300mL of distilled water was slowly added, to make sure that the grounded sample was well wetted initially to avoid tendency to float. The mixture was

allowed to digest at room temperature, with constant stirring, for 48 hours. The material was transferred to a tared filtering crucible, and washed with cold distilled water, and dried to constant weight at 105°C. The cold-water solubles was calculated on oven dry basis.

Calculation:

$$\text{Weight of weighing bottle} = W_1$$

$$\text{Weight of weighing bottle + air dried grounded sample} = W_2$$

$$\therefore \text{Weight of air dried grounded sample} = W_2 - W_1 = W_3$$

$$\text{Weight of oven dried grounded sample} = W_3X$$

$$\text{Weight of crucible} = Y_1$$

$$\text{Weight of crucible + oven dried extract} = Y_2$$

$$\therefore \text{Weight of oven dried extract} = Y_2 - Y_1 = Y_3$$

$$\% \text{ Cold Water Solubility} = \frac{(W_3X - Y_3)100}{W_3X}$$

Determination of hot-water solubility (TAPPI Standard T207)

The experiment was performed in duplicate. About 2g of air-dried grounded sample was weighed out accurately to 0.001g in a weighing bottle. The grounded sample was transferred to a 250mL Erlenmeyer flask, submerged in a boiling water bath, added with 100mL of distilled water and let boil for 3 hours. The contents of the flask was filtered to a tared filtering crucible, washed with hot water and dried to constant weight at 105°C. The hot water solubles was calculated on oven dry basis.

Calculation:

$$\text{Weight of weighing bottle} = W_1$$

$$\text{Weight of weighing bottle + air dried grounded sample} = W_2$$

$$\therefore \text{Weight of air dried grounded sample} = W_2 - W_1 = W_3$$

$$\text{Weight of oven dried grounded sample} = W_3X$$

$$\text{Weight of crucible} = Y_1$$

$$\text{Weight of crucible + oven dried extract} = Y_2$$

$$\therefore \text{Weight of oven dried extract} = Y_2 - Y_1 = Y_3$$

$$\% \text{ Hot Water Solubility} = \frac{(W_3X - Y_3)100}{W_3X}$$

Determination of ethanol-acetone solubility (TAPPI Standard T204)

The experiment was performed in duplicate. About 2g of air-dried grounded sample was weighed out accurately into a tibble. A clean dry extraction flask (250mL capacity) was weighed. The tibble and sample was placed in a position in the Soxhlet apparatus, and extracted with 200mL Ethanol-Acetone solution for 4 to 5 hours, keeping the liquid boiling briskly by heating on an electric heating mantle. The solvent was evaporated from the extraction flask to ensure the residue was not to be char (Best over a water bath). The flask and the contents were dried to constant weight in the oven at 105°C. The result was expressed as percentage ethanol-acetone solubility on oven dry sample.

Calculation:

$$\text{Weight of fritted glass crucible} = W_1$$

$$\text{Weight of fritted glass crucible + air dried sample} = W_2$$

$$\therefore \text{Weight of air dried sample} = W_2 - W_1 = W_3$$

$$\text{Weight of oven dried sample} = W_3X$$

$$\text{Weight of extraction flask} = Y_1$$

$$\text{Weight of extraction flask + oven dried residue} = Y_2$$

$$\therefore \text{Weight of ethanol-acetone solubles} = Y_2 - Y_1 = Y_3$$

$$\% \text{ Solubles} = \frac{100Y_3}{W_3X}$$

Determination of 1% NaOH solubility (TAPPI Standard T212)

The test was performed in duplicate. About 2g of air-dried grounded sample was weighed out accurately into a 200 mL tall-form beaker. 100 mL of 1% NaOH solution was added and the mixture was stirred with a glass rod. The beaker was covered with a watch glass, and placed in a water bath for an hour. The water in the bath should be kept boiling and its level above that of the alkali solution in the beaker. The solution was stirred with a rod about 5 seconds at 10, 15 and 25 minutes after placing the beaker in the bath. At the end of 1 hour, the material was transferred to a tared filtering crucible (medium porosity) and washed with 100 mL of hot water. 25 mL acetic acid was then added and allowed to soak for 1 minute before removal. This step was repeated with a second 25 mL portion of 10% acetic acid. The material was finally washed with hot water until acid free. The crucible and the contents were dried in an oven at 105°C to a constant weight, cooled in a desiccator, and weighed.

Calculation:

$$\text{Weight of weighing bottle} = W_1$$

$$\text{Weight of weighing bottle} + \text{air dried grounded sample} = W_2$$

$$\therefore \text{Weight of air dried grounded sample} = W_2 - W_1 = W_3$$

$$\text{Weight of oven dried grounded sample} = W_3X$$

$$\text{Weight of crucible} = Y_1$$

$$\text{Weight of crucible} + \text{oven dried residue} = Y_2$$

$$\therefore \text{Weight of oven dried extract} = Y_2 - Y_1 = Y_3$$

$$\% \text{ Alkali Solubility} = \frac{(W_3X - Y_3)100}{W_3X}$$

Determination of lignin (TAPPI Standard T222)

This analysis was carried out in duplicate. About 1g of air-dried extractive-free, grounded sample was weighed out accurately in a weighing bottle into a 50 mL beaker. 10 mL 72% sulphuric acid was carefully added with a pipette and with a small glass rod, the mixture was stirred. Subsequently the mixture was leave be with irregular stirring at room temperature for 2 hours. The mixture was next transferred to 500 mL conical flask, and dilute with water until the final volume was 300 mL and covered with reversed smaller conical flask. The mixture was heated on the hotplate for an hour.

This process was continued for extra 2 hours at the beginning of the next laboratory period. In the meantime, a crucible (fine or medium porosity) was oven dried for an hour at 105°C, and then allows cooling in desiccator and weighed accurately. The insoluble lignin was recovered, when the heating was completed, by filtration through the crucible after allowing the lignin to settle to facilitate filtration. The lignin was washed free from acid with 250 mL of hot distilled water. The crucible containing the lignin was dried at 105°C for a day, cooled in desiccator and weighed. The lignin content was reported as percentage by weight of the dry sample.

Calculation:

Weight of bottle = W_1

Weight of bottle + air dried sample = W_2

∴ Weight of air dried sample (extractive free) = $W_2 - W_1 = W_3$

Weight of oven dried sample = W_3X

Weight of crucible = Y_1

$$\text{Weight of crucible + lignin} = Y_2$$

$$\therefore \text{Weight of lignin} = Y_2 - Y_1 = Y_3$$

$$\% \text{ Lignin, based on extractive free grounded sample} = \frac{100 Y_3}{W_3 X}$$

Determination of holocellulose (Wise et al., 1946)

About 2g of air-dried, extractive-free grounded sample was weighed out accurately. The grounded sample was transferred quantitatively to a 250 mL conical flask. 100 mL water, 1.5 g sodium chlorite and 5 mL of 10% acetic acid, were added and the flask was placed in a water bath maintained at 70°C. The content of the flask was stirred at least once every five minutes. The flask was kept close with a small, inverted Erlenmeyer flask. 5 mL of 10% acetic acid was put in after 30 minutes. 1.5 g sodium chlorite was added subsequent to further 30 minutes, which means an hour after starting the experiment. Continuance additional of another acetic acid and sodium chlorite was performed at 30 minutes intervals until 6 g sodium chlorite has been added. The mixture was heated for 30 minutes after last addition of sodium chlorite. The suspension was set aside to cool in an ice bath. It was filtered into a weighed fritted glass crucible (medium or coarse porosity), followed by cleanse with iced distilled water and finally washed with acetone. With a punctured aluminium foil covering it, the residue was air dried by placing it to expose in the open laboratory for a day or two until it was free of acetone. Then it was transferred to a desiccator and weighed at daily intervals until the sample reaches constant weight.

Calculation:

From ethanol-acetone solubility, weight of extractive-free, oven dried wood = $W_3 X$

$$\text{Weight of crucible} = Y_1$$

$$\text{Weight of crucible + air-dried holocellulose} = Y_2$$

$$\text{Weight of air-dried holocellulose} = Y_2 - Y_1 = Y_3$$

From moisture content determination,

$$\text{Weight of oven-dried holocellulose} = Y_3 X_1$$

$$\% \text{ Holocellulose} = \frac{100 Y_3 X_1}{W_3 X}$$

Determination of alpha-cellulose (TAPPI Standard T203)

Air-dried holocellulose was used. The experiment was conducted in water bath at 20°C. About 2g of air-dried grounded sample was weighed out accurately into a 250 mL beaker which was placed in a water bath at 20°C. 15 mL of 17.5% NaOH was added and gently macerated with a flattened glass rod for 1 minute. 10 mL more NaOH was added and mixed for 45 seconds, followed by 10 mL more and mixed up for 15 seconds, so that at the end of 2 minutes, 35 mL of the NaOH have been added. The mixture was stirred and allowed to stand for another 3 minutes. Subsequently 10 mL additional NaOH was added and mixed with a stirring rod for 2 ½ minutes repeatedly for 4 times. The mixture was place in the water bath for 30 minutes more. Then 100 mL distilled water was added at 20°C. The diluted mixture was quickly and thoroughly mixed and leaved in the water bath for a further 30 minutes.

The liquid was filtered into a weighed fritted glass crucible (coarse porosity). The beaker and the residue was rinsed with 25 mL of 8.3% NaOH solution at 20°C and all the fibres were quantitatively transferred to the crucible. The crucible was washed with 500 mL distilled water at 20°C. The suction tube was disconnected and the crucible was filled with 2N acetic acid at 20°C and the residue was allowed to soak for 5 minutes. Suction was reapplied to remove acetic acid. The residue was washed with distilled water until it was free of acid as indicated by the litmus paper. The bottom and sides of the crucible was wiped with a dry towel and placed in the oven at 105°C. It was afterward dried to constant weight and then set

aside to cool and weighed for one more time. The α -cellulose was calculated as a percentage, based on oven-dried sample.

Calculation:

$$\text{Weight of oven-dried holocellulose} = W_3X$$

$$\text{Weight of crucible} = Y_1$$

$$\text{Weight of crucible + oven-dried } \alpha\text{-cellulose} = Y_2$$

$$\text{Weight of oven-dried } \alpha\text{-cellulose} = Y_2 - Y_1 = Y_3$$

$$\begin{aligned} \text{\% } \alpha\text{-Cellulose on oven-dried sample} &= \frac{Y_3X100}{W_3X} \\ &= \text{\% of oven-dried sample} \end{aligned}$$

Determination of ash (TAPPI Standard T15)

The experiment was performed in duplicate. The empty silica crucible and cover was cleansed and dried to constant weight in the oven at 105°C. Subsequently, the instrument was slightly cooled and placed in a desiccator. When cooled to room temperature the crucible was weighed on the analytical balance to the nearest 0.1 mg. About 2g of the air-dried grounded sample was placed in the crucible and weighed again. The crucible was placed in the furnace at 575°C for a period of at least 3 hours or longer if required, to burn off all the carbon. When ignition was complete, as indicated by the absence of black particles, the crucible was removed from the furnace; the cover was replaced and allowed to cool. Then, it was placed in a desiccator and cooled to room temperature. The crucible with the ash was reweighed to nearest 0.1mg and the percentage was calculated based on the oven-dried weight of grounded sample.

Calculation:

Weight of crucible	= W_1
Weight of crucible + air-dried grounded sample	= W_2
∴ Weight of air-dried grounded sample	= $W_2 - W_1 = W_3$
Weight of oven-dried grounded sample	= $Y_2 - Y_1 = W_3X$
Weight of crucible + ash	= W_4
∴ Weight of oven-dried ash	= $W_4 - W_1 = W_5$
% Ash in oven-dried grounded sample	= $\frac{W_5 100}{W_3 X}$

Morphological determination

Maceration was done for the roselle stem components using the prepared matchstick-size samples. The maceration technique suggested by Spearin and Isenberg (1947) was used by using (1:2) sodium chlorite and acetic acid as applied in this study. The mixture was heated gently at 70°C for 48 hours or longer if necessary to allow the bundles of cells or fibres to become separated from one another, to become individual cells or fibres.

The macerated fibre suspension was placed on a slide by means of a needle or medicine dropper. The slide was later covered with a glass cover slip placed over the fibres. One hundred fibres were randomly selected and measured in this study for their fibre length, width and thickness using a projector microscope.

Statistical Analysis

Statistical analysis using the one-way ANOVA and Least Significance Difference (LSD) was performed to evaluate the significance of roselle stem components as compared to *A. mangium*. Confidence level of 95% was used.

RESULTS AND DISCUSSION

Chemical analysis

The determination of the percentage of lignin, holocellulose and other chemical composition in grounded samples used in the experiments were on an oven dry basis. But the samples used were almost at equilibrium with the moisture of the air. Thus, it is important to perform an experiment to determine the moisture content of each roselle components and *A. mangium* in order to provide factor to convert air dried to oven-dried equivalent.

Table 1 shows variation in the different chemical compositions between roselle stem components and *A. mangium* with their percentages values and standard deviation. The determination of cold water and hot water solubility provides a measure of extraneous components such as gums, sugar, tannins and colouring matter, (and in addition, starches for hot water determination) in lignocellulosic material. In Table 1, it appears that cold-water solubility for whole plant was 6.63%, 4.36% in core, 10.65% for bast and 4.50% in *A. mangium*. This value was significantly different ($\alpha=0.05$) for bast, suggesting that the bast contain higher amount of cold water solubles.

Hot water solubility were higher in roselle stem components; 18.38% for whole plant, 5.35% for core and 12.82% for bast, compare to *A. mangium* with only 3.54%. The values of hot water solubility for all the samples were significantly different ($\alpha=0.05$). The high percentage of cold water or hot water solubility of certain lignocellulosic species directly indicates that the high soluble content of the extraneous components in the lignocellulosic material. The results attained pointed out that cold water and hot water solubles for roselle stem components are higher than *A. mangium*, suggesting that stem components of roselle consist of higher soluble content of the extraneous components than *A. mangium*.

Table 1: Comparison of chemical properties between stem components of roselle and *A. mangium*

Chemical Components (%)	Roselle stem components			<i>A. mangium</i>
	Whole Plant	Core	Bast	
Cold water solubility	6.63 ^b (±1.25)	4.36 ^b (±2.64)	10.65 ^a (±0.36)	4.50 ^b (±0.30)
Hot water solubility	18.38 ^a (±6.02)	5.35 ^b (±1.21)	12.82 ^a (±2.10)	3.54 ^b (±0.89)
Alkali solubility	32.46 ^a (±0.72)	27.06 ^b (±0.91)	14.14 ^d (±1.86)	16.72 ^c (±0.80)
Ethanol-acetone solubility	2.61 ^b (±0.16)	2.49 ^b (±0.58)	2.47 ^b (±0.40)	6.38 ^a (±0.66)
Lignin	21.39 ^b (±0.77)	20.65 ^b (±0.66)	20.66 ^b (±1.62)	31.08 ^a (±5.18)
Holocellulose	76.69 ^a (±1.63)	80.70 ^a (±1.02)	75.82 ^a (±3.07)	73.88 ^a (±5.81)
Alpha-cellulose	51.75 ^a (±2.25)	44.81 ^a (±7.93)	37.70 ^a (±17.14)	60.87 ^a (±1.60)
Ash	4.32 ^a (±0.36)	1.12 ^b (±0.31))	4.68 ^a (±0.86)	0.12 ^c (±0.04)

Notes: Values are means of three determinations. Value in parentheses is the standard deviation. Means followed by the same letter (a,b,c) in the same row are not significantly different at $\alpha=0.05$ according to Least Significance Difference (LSD).

Hot alkali solution extracts low molecular weight carbohydrates comprising mostly of hemicellulose and degraded cellulose in wood. The alkali solubility of wood may possibly specify the degree of a fungus decay, or of degradation by light, heat, oxidation and so forth. As the wood degrades or decays, the percentage of the alkali-soluble material increases. Alkali soluble extracted during determination of 1% NaOH solubility for *A. mangium* and roselle stem components was significantly different ($\alpha=0.05$). The whole plant of roselle

possess higher value of alkali soluble which was 32.46% compared to its other components, core (27.06%) and bast (14.14%). *A. mangium* showed 16.72% of alkali soluble.

Neutral solvents, water, toluene or ethanol, or combinations of solvents are employed to remove extractives in lignocellulosic material. In the implement of this experiments, the ethanol-acetone soluble content most probably testify a measure of waxes, resins, fats, and several other ether-insoluble components. The proportion of wood formed by extraneous substances varies widely with the species and normally, it is between 2-10% (Negi, 1997). High alcohol-benzene soluble of wood is usually associated with relatively high degree of durability (Takakashi & Kishima, 1973). Therefore, we can assume that the same things allied to ethanol-acetone soluble. The results gained indicate that ethanol-acetone solubility for *A. mangium* was 6.38%, the highest among the four samples and this value was significantly different ($\alpha=0.05$). Ethanol-acetone solubility for the components of *H. sabdariffa* was not significantly different ($\alpha=0.05$), with percentage of 2.61%, 2.49% and 2.47% for whole plant, core and bast, consecutively. The high content of extractive of *A. mangium* was shown indirectly through its brownish ethanol-acetone soluble.

Thus, this condition corresponds to Haygreen and Bowyer (1996) finding that hardwoods exhibit a wide range of colouration due to their extractives content. Roselle stem components showed less coloured extractives compared to *A. mangium*. High content of ethanol-acetone solubility in lignocellulosic material is usually related with their hardness. This is without doubt associated with the strength as extractives could stiffen and strengthen individual cells within woody materials (Panshin and de Zeeuw, 1970). The wood with higher ethanol-acetone solubility is more difficult to be infiltrated with liquids including chemicals consumed to preserve it.

Determination of lignin content generated the percentage values for roselle stem components and *A. mangium* such as the following; 21.39% in whole plant, 20.65% in core, 20.66% for bast and 31.08% in *A. mangium*. In general, the higher the lignin contents, the lower the cellulose content (Han, 1998). Consequently, *A. mangium* with the highest lignin content was supposed to retain the lowest cellulose content. The whole values in lignin content, except for *A. mangium* were not significantly different at confidence level of 95%. The function of lignin in plants is as an encrusting agent in the cellulose/hemicellulose matrix. It is often referred to as the plant cell wall adhesive. Both lignin and extractives in plants reduce the digestibility of grasses to animals (Iiyama et al., 1993). Lignins are also associated with the hemicelluloses, in some cases forming lignin-carbohydrate complexes that are resistant to hydrolysis even under pulping conditions.

According to Kollman and Cote (1984), the role of lignin in plant is to provide inflexibility to the plant thus making the upright growth possible and also enhances plant toxicity making the plant more resilient.

Holocellulose is lignin-free fibrous material comprising all of the hemicellulose and cellulose in lignocellulosic material. Usually it can be identify as white, cream or straw-coloured, depending on the kind of lignocellulosic material and accounts for 65–70 percent of the plant dry weight (Han and Rowell, 1997). The holocellulose content in the roselle stem components were slightly higher than in the *A. mangium*, which hold 76.68%, 80.70% and 75.82% for whole plant, core and bast consecutively. *A. mangium* holocellulose content was only 73.88%. These figures were not significantly different ($\alpha=0.05$), however between core and *A. mangium*, they were significantly different at $\alpha=0.05$.

Alpha-cellulose is the alpha fraction of high molecular weight of cellulose, which remains when a mixture of pulp and 8.3% NaOH solution was filtered after the fibres have

been previously swollen in a 17.5% NaOH solution. The result of alpha-cellulose determination specify that *A. mangium* with percentage of 60.87%, exhibits the highest alpha-cellulose content compare to the rest of the roselle stem components; whole plant (51.75%), core (44.81%) and bast (37.70%). These figures were not significantly different at confidence level of 95%. Similar to holocellulose the bast and *A. mangium*, showed significant different at $\alpha=0.05$.

Ash content is a measure of mineral salts; calcium, potassium, manganese, magnesium and silicon (Haygreen and Bowyer, 1996) in the wood, but it is not necessarily quantitatively equal to them. Ash content in roselle stem components were higher with bast represents the highest ash content (4.68%), followed by whole plant (4.32%) and core (1.12%). *A. mangium* showed only 0.12%. The percentage of ash contents in samples were significantly different at $\alpha=0.05$. However, whole plant and bast were not significantly different ($\alpha=0.05$). Haygreen and Bowyer (1996) stated that ash content could possibly portrayed silica content, which imply low ash content could be a sign of low silica content.

Morphological properties

Table 2 shows variation in the different fibre morphological properties between roselle stem components and *A. mangium* with their means values respectively. A notable physical difference between wood and non-wood fibre is that non-wood fibres are formed in aggregates or bundles. This is why non-wood fibres like cotton and flax can be used to make rope and textile. The fibre aggregates are polymers, with a single fibre unit representing the basic building block of the polymer (Han, 1998).