THE EFFECT OF BLANCHING AND STORAGE ON THE CHEMICAL COMPOSITION OF DATES

by

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ABSTRACT

The changes during storage of dates (*Phoenix dactylifera* L.) at room temperature and at -13^{o} were studied. The effect of blanching in boiling water and by microwave heating before storage was also studied. Moisture, pH, acidity, total soluble solids, sugar, colour, soluble leucoanthocyanidin tannin, insoluble leucoanthocyanidin tannin, total soluble phenolic compounds and the activities of polyphenolase and peroxidase enzymes were investigated.

The dates stored at room temperature showed darkening, and decrease in the amount of insoluble leucoanthocyanidin tannin. The dates stored at -13^{o} showed darkening, decrease in the total soluble phenolic compounds, increase in the insoluble leucoanthocyanidin tannin and increase in the total soluble solids.

The dates blanched in boiling water showed a dull colour, soft texture, significant increase in the moisture content, decrease in the total soluble solids and sugar, decrease in the total soluble phenolic compounds and decrease in the insoluble leucoanthocyanidin tannin. The dates blanched by microwave heating showed no changes in colour, texture and flavour, but loss in the moisture and decrease in insoluble leucoanthocyanidin tannin in fully

ripe dates. In green dates there was a significant loss in the moisture, decrease in the total soluble phenolic compounds and an increase in the insoluble leucoanthocyanidin tannin proportional to the decrease in the soluble leucoanthocyanidin tannin.

Two enzyme complexes were investigated, polyphenolase and peroxidase. Polyphenolase was more active in the fully ripe dates than the green dates whereas peroxidase was more active in the green dates than in the fully ripe dates. Polyphenolase was completely inactivated in the fully ripe dates by blanching dates in boiling water for 4 minutes. Microwave heating for 1 minute was sufficient for complete inactivation in both fully ripe and green dates. Peroxidase was completely inactivated in the fully ripe dates by blanching in boiling water for 3 minutes or by microwave heating for less than 1 minute.

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1. INTRODUCTION

1.1 General Introduction

The date palm Phoenix dactylifera L. is a native tree of Iraq and its cultivation, handling and processing provides a living for a considerable part of the population of that country.[1] Iraq leads the list of date producing countries and accounts for about 35% of the total world production followed by Iran, Egypt and Algeria.[2]

Many hundreds of different varieties of date are grown, however, these are often classified into three groups on the basis of the texture of the date fruit; these groups are soft, semi-dry and dry (hard).[3] In general the soft varieties are considered to be of highest "quality"; "quality" being defined according to the factors given in Table 1. In general low acidity, high sugar content and "good" sensory properties appear to be the most important factors.

The most commercially important soft varieties are Sayer, Hillawi and Khadrawi. The only commercially important semi-dry variety is Zahdi, which accounts for about 43% of

Table 1 Factors which relate to the quality of dates

Factor	High quality	Low quality	References
pH	6-6.9	less than 5	4,5
acidity	low acidity	high acidity	4,5
sugar	more than 60%	less than 60%	5
colour	light brown colour	black colour	6,7,8,9
taste	no astringent taste, no soluble tannin	astringent taste, presence of soluble tannin	10,11
texture	smooth and tender	tough, fibrous or rubbery, very soft	9,12
sugar spots	no sugar spots	sugar spots	6,13
syrup	no syrup exudate	syrup exudate	7
flavour	"good" flavour	off-flavour	8,9

the total number of date palm in Iraq. The dry varieties are not in general of commercial importance.[2,3]

The most important constituent of ripe dates is sugar which accounts for up to 70% of the wet weight.[3] Most of the sugar in ripe dry and semi-dry varieties of dates is sucrose, the remainder is invert sugar (a mixture of equal parts of glucose and fructose.)[14] All or nearly all of the sugar in ripe soft varieties is invert sugar. Crude fibre can be up to 10% of the wet weight.[14,15] Protein accounts for about 2% of the wet weight,[14,16,17] fat about 1%[14] and ash 1-2%.[1,15]

Dates contain many active enzymes such as invertase, cellulase, peroxidase and polyphenolase.[18,19,20,21,22] The polyphenolase is of particular importance with respect to quality, since it is involved in the darkening of the dates and hence loss of value.

Various products such as dibbis (thick date juice) alcohol (Arak), pure alcohol, vinegar, liquid sugar are produced on a commercial scale from dates.[3,23] Investigations have been launched into the manufacture of glucose and other products such as jam and baby food. The manufacture of paper, cellulose, lignin and furfural from date palm trees is also being investigated.[3] However, the majority of dates are eaten ripe as a fruit rather than in these processed forms.

1.2 Stages of Ripening

There are four stages in the ripening of dates, these are classified as kimri, khalal and tamar (fully ripe) stages.[3,14]

Kimri stage

The colour of the fruit in this stage is green and it has an astringent taste due to the presence of large amount of soluble tannin. During this stage there is a rapid increase in size and weight, rapid accumulation of total sugar (especially sucrose), high acidity and high moisture content about 80%. The fruit contains a small amount of protein and pectin.

Khalal stage

Dates in this stage may be yellow, pink, red or scarlet or yellow spotted with red, depending upon variety. The rate of gain in size and weight decreases, the weight may even decrease slightly. Invert sugar accumulates slowly but sucrose accumulates at a rapidly increasing rate. Acidity decreases and moisture content drops to about 60%. Protein and pectin decrease.

Rutab stage

In this stage the colour of the dates changes from the yellow-red colours of the khalal stage to brown or to nearly black and the fruits begin to soften. Little or no sugar accumulates during this stage but the dates continue to lose water and the water content drops to about 30-40%. Sucrose decreases and the invert sugars increase. The amount of protein and pectin also continues to decrease. The soluble tannin is converted into insoluble tannin and the astringent taste is lost.

Tamar stage (fully ripe)

Dates in this stage have dried to a fairly firm consistency with moisture contents of about 20-25%. The water activity is low enough to prevent the dates undergoing fermentation. Nearly all the sucrose has been converted into invert sugar in this stage in soft dates, while in semi-dry dates a small amount of sucrose remains.

Changes in the percentage of invert sugar, sucrose, total sugar and total solids are given in Table 2 for a soft variety, Khadrawi, and a semi-dry variety, Zahdi.

Table 2 Changes in the sugar content and total solids of two varieties of dates during ripening[24]

Variety of dates	Stage of ripening	% invert sugar	% sucrose	% total sugar	% total solids
Khadrawi	kimri	-	-	6.9	16.1
	khalal	4.3	24.2	28.5	37.3
	rutab	19.9	24.8	44.7	54.0
	tamar	63.6	0	63.6	75.4
Zahdi	kimri	-	-	6.1	16.6
	khalal	4.5	24.5	29.0	38.0
	rutab	19.5	40.2	59.7	70.8
	tamar	57.5	9.6	67.1	77.8

1.3 Chemical Composition of Dates

1.3.1 Variation in Composition During Growth and Ripening

The chemical changes during development and ripening of six varieties of Egyptian dates have been studied.[25] It was found that in the early green stage, a slow rate of accumulation of sugars mainly invert sugar (reducing) occurred which became rapid during the late green stage. In the khalal stage it was found that there was a slow increase in the total sugar and the ratio of sucrose to invert sugar becomes approximately one, a very rapid and complete inversion of sucrose occurred in the rutab stage.

It was found that sucrose was the predominant sugar during the kimri and khalal stages during development of twelve varieties of Basra dates in Iraq.[24] During the rutab stage inversion took place and the percentage of sucrose gradually decreased. In ten varieties it was found that the sucrose was completely inverted in the tamar stage, but in Zahdi dates an appreciable amount of sucrose was found to be present in the tamar stage.

A small amount of starch appears in young dates for a short time after pollination, but this soon disappears in most varieties.[14] Fibre decreases as the fruit develops.[14]

Soluble pectins, protopectin and total pectic substances have been reported to decrease during ripening of dates.[14]

Protein is found in considerable amounts in the kimri stage but this decreases during ripening.[17] Acidity in dates is highest during the period of most rapid growth (kimri stage); it decreases in the later part of the growing season and continues to decrease as the fruit ripens.[14] The mineral content of dates decreases as the fruit ripens.[27]

The changes in the phenolic fractions of dates for Deglet Noor variety with growth, maturation and storage are given in Table 3.

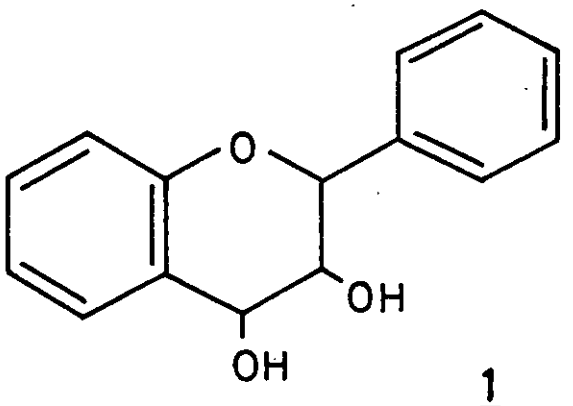
Simple phenolics decrease during growth, maturation and post harvest deterioration in Deglet Noor dates, while the soluble tannin undergo increase from the green to the khalal stage, followed by a decrease from the khalal to the ripe stage.[28] It was found that at the same time an increase occurred in the amount of insoluble tannin. It appears that during ripening, soluble tannin is converted into insoluble tannin.[28] Of the simple phenolics, it was found that the flavans such as flavan-3,4-diol, 1, and derivatives such as dactylifric acid (3-O-caffeoylshikimic acid), 2, underwent the greatest decrease during maturation.[29,30,31] Several simple phenolics not present in immature fruit were formed during post harvest deterioration; they appeared to be cinnamic acid derivatives.[29]

Table 3 Quantitative changes in the phenolic fractions of dates during growth, maturation and storage[28]

	mg(+) catechin equiv/average date			
Colour of fruit	green (kimri stage)	pink (khalal stage)	amber (rutab/tamar stage)	dark[(a)] (tamar stage)
Simple phenolics	3.01	2.67	1.74	1.18
Soluble tannin	37.5	85.0	10.7[(b)]	14.0[(b)]
	mg cyanidin chloride/average date			
Insoluble tannin	5.56	12.6	39.2	21.9

(a) A portion of the dry, ripe fruit was hydrated and held at 38°C for 23 days to accelerate darkening.

(b) This value is high because of the presence of interfering substances.



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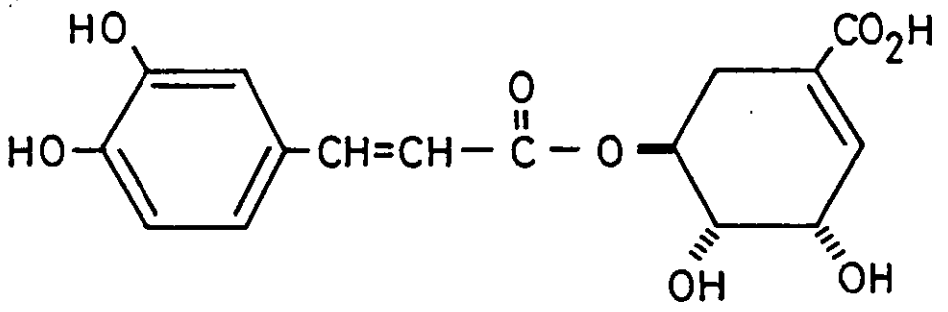

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1.3.2 The Chemical Constituents of Ripe Dates

The main constituents of ripe dates (tamar stage) are invert sugar (equal amounts of D-glucose and D-fructose) and sucrose.[14] For ripe Zahdi dates it has been reported that the invert sugar content is between 60.80% and 81.26% (average of five reported values is 69.44% as dry weight) and the sucrose content between 3.05 and 13.11% (average of five reported value is 7.83%).[1,15,24,26,32] In Khadrawi dates it has been reported that the invert sugar content is 74.20%[32] and 84.35%[24] (dry weight) and the sucrose content is 0%. Starch is not usually found in ripening or ripe fruits.[14] Zahdi dates were found to contain 0.5-1.35% pectin[1,15] and 4.5-10.1% crude fibre.[14,15,16] The same dates contain 1.9-2.4% protein as dry weight[16,17] and the presence of seventeen free amino acids has been reported.[16,17] The flesh of the dates contains between 0.06% and 1.9% fat (average of six reported value is 0.62%)[14] and 1.1-2.04% ash.[1,15] Zahdi dates contain between 11.4% and 22.2% moisture (average of five reported value is 15.7),[1,15,24,26,32] while Khadrawi dates were found to contain 13.9%[32] and 24.6%[24] moisture.

Ripe Deglet Noor dates, a semi-dry variety, contain about 0.5% total phenolic compounds (dry weight).[28] These total phenolic compounds are mainly insoluble tannin plus a small amount of simple phenolics, but no soluble tannin. The soluble leucoanthocyanidin tannin which is responsible for the astringent taste in all dates in the kimri stage, has also been shown to be absent from ripe khadrawi and Zahdi dates.[32]

Dates have been shown to contain several enzyme systems such as, invertase, peroxidase, polyphenolase and cellulase.[18,19,20,21,22] Invertase has been studied more than any other enzyme in dates.[18,19,20] Peroxidase occurs in dates, but its function is not known;[21] it does not appear to be involved in the reactions associated with darkening. Peroxidase in dates has an optimum pH of 4.7 and it is more tolerant to heat than invertase and polyphenolase. Polyphenolase is responsible for the enzymatic oxidative portion of date darkening.[21] It is more sensitive than peroxidase to acid but is more tolerant to alkalinity (optimum pH 5). Its tolerance to heat is intermediate between that of invertase and that of peroxidase.

1.4 Processing and Storage of Dates

After harvesting, the dates are transported to the packing houses in either boxes or bins. At the packing house the dates are fumigated as soon as possible after arrival in order to kill insects and micro-organisms. Methyl bromide is the most commonly used fumigating agent, usually at a level of about 1 kg methyl bromide per 60 m^3 of volume in the fumigation house, at a temperature above 16^{o}C and with an exposure time of 12 hours. Methyl bromide is toxic to humans and its odourless nature requires that it is handled with utmost caution. Carbon disulphide, hydrocyanic acid and ethylene oxide have also been used for fumigation.[3,33,34]

Dates are then cleaned, normally by using water sprays and roller brushes, and dryed by a warm air current. Sorting is necessary to separate the fruits into lots of uniform ripeness, colour, texture, size and moisture content. Any calyxes that remain on the dates are removed during the sorting. For some varieties of dates the moisture content is adjusted after sorting.[33,34] The dates are then packed and stored until distributed.

Several investigations have been carried out in order to determine the optimum storage conditions for dates.[35] A comparative study was undertaken in order to investigate the

effect of different temperatures (-3°C, 0°C, 5°C and room temperature) and different types of containers on the storage of three commercial varieties of dates: Zahdi (semi-dry), Hillawi (soft) and Sayer (soft). The following results were obtained from the experiment.

i) The quality of the date fruits stored at room temperature was poor and not suitable for human consumption.

ii) Zahdi and Hillawi varieties stored well at -3° 0° and 5°. Storage at 5° is therefore recommended as being the most economical.

iii) No increase in the percentage of insect infestation was noted in the samples stored at 5°, 0° or -3°. At room temperature the percentage of infestation increased up to 100% during the 9 months period of storage.

iv) A decrease in the moisture content of dates stored at room temperature from 11 to 9% was observed in loose dates at -3°, 0° and 5°. No significant changes in moisture content were noticed in dates stored in polyethylene bags or cellophane windo— cartons and small plastic containers.

v) A drop in the pH of the dates stored at room temperature from 6.2 to 5.2 was observed, while in dates stored at lower temperatures no change occurred.

vi) The external appearance of dates stored in polyethylene bags, plastic containers and windo-cartons remained good throughout the storage period boxed dates suffered some deterioration in appearance.

The flavours of apples, onions and potatoes were noted in dates stored with these products at ordinary cold storage temperatures and the flavour of meat was very marked in dates stored in frozen food locker plants in the same room with meat.[34]

The relative humidity required to maintain constant moisture in the dates was shown to depend on the initial moisture content and on the temperature of the storage space.[34]

1.5 Artificial Maturation

Artificial maturation (picking of date fruits in the khalal or early rutab stage and ripening them to maturity under controlled conditions) can be used when the dates do not ripen properly on the palm, or where damage from rain could occur if the dates were left on the palm until they ripen. The latter occurs in seasons when the dates are late in ripening.[3,36] Much research work has been carried out in attempts to find the most suitable conditions for artificial maturation. Three different general procedures have been used, these are: use of heat with high relative humidity, use of freezing and thawing followed by heating and use of chemical methods.

When heat with high relative humidity is used to mature dates artificially, then maturation rooms are used which must have a system to circulate air uniformly to all parts of the room and to introduce fresh air as needed.[34] The temperature and humidity must be controlled, and in general less mature fruits need higher temperature and higher humidity than more mature fruits. Temperatures of up to 54^{o}C may be used for maturation,but some varieties become too dark, and some of the enzymatic processes associated with maturation are interfered with leading to a lowering

of the final quality of the dates including loss of flavour. Artificial maturation of soft varieties is usually carried out at temperatures higher than those used for semi-dry varieties, e.g. Khadrawi dates (soft) are matured at 40-43°C whereas Deglet Noor dates (semi-dry) are commonly matured at 35°C, and Zahdi dates (semi-dry) at 35-38°C. The time of maturation should not normally be more than 18-24 hours, but Zahdi dates require 2-4 days or more because of their fibrous nature. The relative humidity is usually maintained at 80-90% for Zahdi dates in order to increase the moisture content whereas for most other varieties lower relative humidities are used in order to lower the moisture content of the dates.[34]

Successful maturation of Daire (semi-dry), Khadrawi (soft) and Khastawi (soft) dates can be achieved by freezing and thawing followed by heating. Dates in the khalal stage are completely frozen at -27°C and at -18°C for 15 hours, spread out to thaw and to dry for 2 hours and then put in a warm ripening chamber at 32°C for eight hours. The dates were then soft and syrupy and were stored in a refrigerator. Dates were found to darken rapidly during and after thawing. The reason given for this darkening is that the very low

temperatures caused the cells to break down and to release the enzymes which are responsible for the darkening.[14] Frozen dates at khalal stage gave perfectly normal, ripe fruits of good quality, with the exception of a very slight bitterness, which disappeared during storage. Artificial maturation by this procedure removes the astringency of the dates by converting the soluble tannin into insoluble tannin.[33,34]

Chemical methods have been used to hasten the ripening of dates and to improve the quality of the final product. Experiments have been carried out using acetic acid, carbon dioxide, ethylene, hot lye and salt.[14,36] The major problem with these methods is the effect the chemicals have on the organoleptic properties of the dates.

1.6 Deterioration of Dates

The deterioration of dates in storage is a major problem and results in undesirable changes in appearance, taste and food value of the fruit. There are several types of deterioration during storage. The most common types of deterioration are darkening, insect infestation, sugaring and microbiological spoilage.[6]

1.6.1 Darkening of Dates

Three types of reaction are involved in the darkening of dates:-

i) Enzymatic oxidative browning of simple phenolics such as derivatives of flavan-3,4-diol, 1, and dactylifric acid (3-O-caffeoylshikimic acid), 2.[29,30]

ii) Non-enzymatic oxidative browning of tannin material such as the polymers formed from flavan-3,4-diol derivatives. This polymeric material, which is normally referred to as leucoanthocyanidin tannin, can be either soluble tannin, normally 2 to 8 units in the polymer, or insoluble tannin, more than 8 units in the polymer. The soluble tannin is responsible for astringency. The insoluble tannin

appears to be the material involved in the non-enzymatic oxidative browning.[28,29,37]

iii) Non-enzymatic, non-oxidative browning of reducing sugars.[38]

At room temperature, enzymatic browning of simple phenolics and non-enzymatic oxidative browning of tannin occur far more rapidly than non-enzymatic, non-oxidative browning of sugar. Above 38°C, however, non-enzymatic, non-oxidative browning of sugar was found to predominate over the other two browning reactions.[39]

The enzymatic oxidative browning of simple phenolics is due entirely to the action of polyphenolase enzymes. Peroxidase enzymes, which are present in dates, have been shown to play no role in the oxidation and subsequent darkening of the dates. It was expected that the oxidation of soluble tannin by polyphenolase might explain the decrease in the amount of soluble tannin during ripening.[21] However, it was shown that soluble tannin was not oxidized by polyphenolase but was converted into insoluble tannin during growth and maturation.[28] Polyphenolase activity has been shown to increase during storage of dates.[39]

The non-enzymatic oxidative browning of the insoluble tannin in dates occurs during heating and during storage of the ripe dates. The rate of this reaction appears to be far higher than the rate of degradation of the tannin to give new enzymatic oxidative browning substrates.[9,29]

The presence of high concentrations of reducing sugars and of free amino acids indicates that non-enzymatic, non-oxidative browning is likely to occur, particularly at elevated temperatures.[38]

1.6.2 Deterioration other than Darkening

Insect infestation is a major problem in stored dates since fumigation is not capable of killing all kinds of insects in dates before storage. The storage room should not contain any kinds of insect or their larvas and eggs to avoid infestation by insects after fumigation. The mushroom mite Tyrophagus lintaeri osborn sometimes survives fumigation and this can lead to losses in stored dates. The mite's small size and ability to survive long periods of drying and absence of food make complete control difficult. Temperatures of $4^{o}C$ or lower prevent insect activity, but do not kill all forms of insect life; temperatures considerably below $0^{o}C$ are required to effect a complete kill.[34]

Sugar spotting is a form of deterioration of dates in which the surface of the dates is disfigured by white spots of sugars. Several of the semi-dry varieties and nearly all of the dry varieties contain a large amount of sucrose and are much less subject to sugar spotting than the invert sugar type. In general, lowering the temperature below ordinary room temperature reduces the rate of formation of sugar spots. Spotting does not normally occur in dates with a moisture content higher than about 33% and spotting decreases as the moisture falls below 22%.[13]

The fermentation of dates is usually caused by species of yeast belonging to the genus Zygosaccharomyces which are more tolerant of high sugar concentrations than others found in dates. Yeast growth leads to the gradual development of undesirable colours and flavours. Moulds are of normally little consequence in the spoilage of commercially packed dates except, but in moist batches, species of Aspergillus, Alternaria and Penicillium are commonly found. The role of bacteria in date spoilage is uncertain but Acetobacter is known to convert the alcohol produced by yeast into acetic acid, the odour of vinegar being sometimes pronounced in spoiled dates. It has been suggested that lactic acid bacteria also contribute to the gradual deterioration of dates.[6,34]

1.7 Enzyme Inactivation in Foodstuffs

Enzymes, such as polyphenolase and peroxidase, are often responsible for the production of undesirable colours, flavours and textures during storage of foodstuffs. Blanching is frequently used to destroy the enzymes in foodstuffs; care has to be taken when using water or steam blanching in order to prevent leaching of nutrients and flavour compounds.

A comparison of the use of microwave heating with boiling water for blanching whole potatoes has been undertaken.[40]

The following results were obtained from the experiment.

i) Microwave heating showed a more rapid inactivation of the enzymes than did boiling water.

ii) Polyphenolase was more heat-labile than peroxidase.

iii) Polyphenolase was inactivated in 6 to 7.5 minutes by boiling water while it was inactivated in 3 to 3.5 minutes by microwave heating.

iv) Peroxidase was inactivated in 13 minutes by boiling water while it was inactivated in 4.7 minutes by microwave heating.

v) No significant differences in texture were found between potatoes cooked in boiling water and by microwave heating.

The results suggested that microwave heating could be effectively utilized for blanching.

Another study has been carried out using microwave blanching of peaches before freezing. The peaches were heated for 6, 8 or 10 minutes in a domestic microwave oven, halved, stoned, deep frozen in liquid nitrogen, packed in cardboard boxes lined with thermoweldable plastics, stored for 1 month at ($-18^{\circ}C$), and thawed either at room temperature or in a microwave oven. Both polyphenolase and peroxidase were almost entirely inactivated by microwave heating for 10 minutes. Microwave blanching reduced browning of the flesh of the fruit, but the peel became brown for both blanched and non-blanched fruit. The flavour of the microwave-blanched samples was superior to that of non-blanched samples.[41]

1.8 The Present Investigation

Dates artificially matured by freezing are in general of good quality except that they are dark in colour and the darkening reactions produce some slight off-flavour. The darkening occurs only slightly during the freezing period but rapidly during thawing period. If the darkening and consequent production of off-flavours could be prevented, then the use of freezing for artificial maturation and storage could become of great commercial importance.

This investigation was therefore concerned with studying the chemical changes related to the darkening that occurs during freezing and storage of dates, particularly the changes in total soluble phenolic compounds, insoluble leucoanthocyanidin tannin and total soluble solids (mainly sugars). The use of blanching both by water and microwave in order to prevent the darkening of dates was also investigated.

2. EXPERIMENTAL

2.1 Materials

The following dates were used in this investigation:

A - Zahdi variety at the green stage. The dates were obtained by special delivery from Kerbala City, Iraq, packed in perforated plastic film (HDPE) and were immediately heated in a microwave oven and then analysed.

B - Zahdi variety at the khalal stage (¼ ripe). The dates were obtained from Kerbala City, Iraq, packed in perforated plastic film (HDPE) and were stored in a refrigerator at $0^{o}C$.

C - Zahdi variety at the tamar stage (fully ripe).

D - Khadrawi variety at the tamar stage (fully ripe).

For C and D the dates were obtained by special delivery from Kerbala City, Iraq, packed in perforated plastic film (HDPE) and were analysed immediately they were received.

2.2 Storage Procedures

The fresh material, Zahdi variety (khalal ½ ripe) was removed from the refrigerator (0°C) and frozen in a blast freezer for one hour at -25°C. After freezing the fruit was packed in LDPE bags, sealed and subjected to blast freezing at -30°C for 3 hours. The frozen samples were stored at -13°C. The relative humidity in the freezer was measured using hygrometer and was found to be 70%. All the analyses on frozen dates were carried out on the dates while they were still frozen. During thawing enzymatic browning and other changes might have occurred which could affect the results. Analyses were repeated, as detailed later in section 2.4, every month over a period of eight months.

The Khadrawi dates (tamar stage - fully ripe) were stored at room temperature (18°C ± 1°) and relative humidity (48 - 62%). Samples were packed in low-density polyethylene bags. One sample date was packed in the bag in the presence of air; one sample in a partial vacuum; and a third bag was flushed with oxygen from a cylinder.

The Zahdi dates (tamar stage - fully ripe) were either blanched in boiling water or treated with microwave heating. The samples were packed in low-density polyethylene bags and stored for two months at room temperature with the above samples of Khadrawi dates.

2.3 Extraction Procedures

The method of Maier and Metzler[28] was used to extract phenolic compounds from dates. In the extraction, 50g were used instead of 100g as stated in this method and half the amount of all reagents was used.

2.4 Analytical Methods

Determination of moisture

The Bidwell and Sterling method was used for moisture determination.[14] Between 3 and 10g of sample of dates was used for the determination depending on stage of ripeness of the dates.

Titratable acidity

The titratable acidity, expressed as grams of tartaric acid/100g dates, was determined by titrating the extract against 0.1M NaOH using phenolphthalein as indicator.[1]

pH

The pH reading was measured using a standard pH meter and glass electrode.

Total soluble solids

The total soluble solids (TSS) were measured using a hand refractometer.

Soluble phenolic compounds

The total soluble phenolic compounds and soluble leucoanthocyanidin tannin were determined according to the method of Swain and Hillis.[42] The Folin-Denis and sodium carbonate reagents were prepared according to the published procedure.[43] Calibration curves were prepared for the determination in dates the amount of total soluble phenolic compounds and soluble leucoanthocyanidin tannin expressed as (+) catechin or as tannic acid.

Insoluble leucoanthocyanidin tannin

The insoluble leucoanthocyanidin tannin was determined according to the method of Maier and Metzler.[28] The published extinction coefficient[44] of 29270 was used to calculate the amount of insoluble leucoanthocyanidin tannin present.

Colour

The colour intensity was measured by taking 20 cm^3 from the extract and the pH was adjusted to 6.3 by adding an appropriate amount of 4M acetate citrate buffer. 1 cm^3 of this extract was diluted to 100 cm^3 with distilled water and the absorbance was measured using a UV/visible spectrophotometer at 270 nm (1 cm path cell).

Sugar

The sugar was determined according to Lane and Eynon method.[45]

A new method, as described by Levy and Zucker[46] but with some modifications, was also used to determine the amount of sugar present in dates. This method determined sugar as glucose by using a periodate/thiobarbituric acid colorimetric procedure. A calibration curve was prepared using D-glucose.

The modified procedure was as follows:-

10g dates were dissolved in 250 cm^3 warm distilled water. After clarifying with lead acetate and potassium oxalate, 25 cm^3 of the clarified solution were placed in a 100 cm^3 volumetric flask and diluted to 100 cm^3; 4 cm^3 of this

solution were diluted to 50 cm^3. 1 cm^3 was then taken and placed in a stoppered tube. Sodium hydroxide (4N, 1cm^3) was added to the sample. The tubes were placed in a boiling water bath for 10 minutes. Then neutralized by adding HCl (4N, 1cm^3). 2 cm^3 of the buffer (p-toluene sulphonic acid 19.4 cm^3 + sodium p-toluene sulphonate 19.0g were dried in the oven at 104°C for 3 hours then dissolved in distilled water and made up to 100 cm^3 with distilled water, the pH = 1.8) were added, 0.25 cm^3 of 0.025 $NaIO_4$ in 0.125N H_2SO_4 was added. The solution was shaken and the reaction allowed to continue at room temperature for 10 minutes. The excess periodate was then removed by the addition of 0.5 cm^3 of 2% w/v sodium arsenite solution in 0.5M Hcl. Then the tubes were shaken vigorously for a few seconds and after 2 minutes, 3 cm^3 of 0.2% solution of 2-thiobarbituric acid were added. After being heated in boiling water bath for 20 minutes, the tubes were cooled in cold water to stop the reaction and the solution diluted to volume 25 cm^3 with distilled water. The pink colour intensity of the solution was read at 532 nm (1 cm cell). All the reagents without the sample were used as a blank.

2.5 Procedures to inactivate the enzymes in dates

Zahdi variety (green stage and tamar stage - fully ripe) and Khadrawi variety (tamar stage - fully ripe) were used for this investigation. The enzymes, polyphenolase and peroxidase, were inactivated by treating dates in a microwave oven (AKB104 - 8105 - 1.6 Kw). Usually 7 dates were treated in the microwave oven, for varying lengths of time of 0, 10, 15, 20, 25, 30, 40, 50, 60 seconds. The temperature was measured inside dates before and immediately after taking the dates out of the oven using thermocouples. The dates were allowed to cool to room temperature and then packed in LDPE bags.

Dates were also blanched in boiling water for 0, 1, 2 and 3 minutes. The variety of dates used for this investigation was Zahdi dates (tamar stage - fully ripe). The temperature was measured inside the dates before and during immersion in boiling water using thermocouples.

Measurement of enzyme activity in dates

Polyphenolase and peroxidase activities were assayed by a spectrophotometric method according to the Maier and Schiller method.[47] The absorbance measurements were made at 20 second intervals on a recording spectrophotometer.

Catechol was used as a substrate for determining polyphenolase activity. Guaiacol and hydrogen peroxide were used to determine peroxidase activity. For measuring polyphenolase activity, absorbancy measurements were carried out at 410 nm (1 cm path cell). For measuring peroxidase activity, measurements were taken at 400 nm (1 cm path cell).

3. RESULTS

3.1 Calculations

The total soluble phenolic compounds and soluble tannin calculated as (+) catechin.

$$\text{mg (+) catechin/100g dates (wet-weight)} = \frac{W \times L \times V \times M}{S \times 1000} \times 2$$

W = weight of (+) catechin (μmole) can be obtained from the calibration curve according to the optical density.

V = the final volume of the solution (75 cm^3).

M = Molecular weight of (+) catechin (290.3).

S = volume of sample taken (0.2 cm^3).

L = path cell (0.5 cm path cell was used).

Total soluble phenolic compounds and soluble tannin can be calculated as mg (+) catechin/100g dates (dry weight) according to the % moisture in dates.

The total soluble phenolic compounds and soluble tannin calculated as tannic acid.

$$\text{mg tannic acid/100g dates (wet-weight)} = \frac{W \times L \times V \times M}{S \times 1000} \times 2$$

W = weight of tannic acid (μmole) can be obtained from the calibration curve according to the optical density.

V = the final volume of the solution (75 cm^3).

M = molecular weight of tannic acid (170.12).

S = volume of sample taken (0.2 cm^3).

L = path cell (0.5 cm cell was used).

The soluble phenolic compounds and soluble tannin can be calculated as mg tannic acid/100g dates (dry weight) according to the % moisture in dates.

Insoluble leucoanthocyanidin tannin calculated as cyanidin chloride.

mg cyanidin chloride/100g dates (wet-weight) =

$$\frac{O.D \times V \times M \times W}{E \times L \times S} \times 2$$

O.D. = optical density.

E = molar extinction coefficient (29270).

L = path cell (0.1 cm).

V = final volume of the reagents (5 cm^3).

M = molecular weight of cyanidin chloride (322.5) (calculated from its chemical structure[48]).

S = weight of sample taken (0.01g).

W = weight of tissue residue after drying.

Insoluble leucoanthocyanidin tannin can be calculated as mg cyanidin chloride/100g dates (dry weight) according to the % moisture in dates.

3.2 Calibration Curve Tables and Graphs

Table 4 Calibration curve for the determination of total soluble phenolic compounds and soluble tannin in dates (as (+) catechin).

mg (+) catechin/100 cm^3 distilled water	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	µmole (+) catechin/ 1 cm^3 solution[a]	molar extinction coefficient
10	0.455, 0.466 0.468, 0.453	0.460	4	0.34	26744
8	0.365, 0.360 0.375, 0.374	0.368	4	0.27	26764
6	0.265, 0.270 0.278	0.271	3	0.20	26184
4	0.192, 0.185 0.190, 0.195 0.190	0.190	5	0.14	27536
2	0.094, 0.092 0.088, 0.090	0.091	4	0.07	26376

(a) Concentrations of standard solutions before addition of reagents. The solutions used to determine O.D., after addition of reagents, are 10 times more dilute.[42]

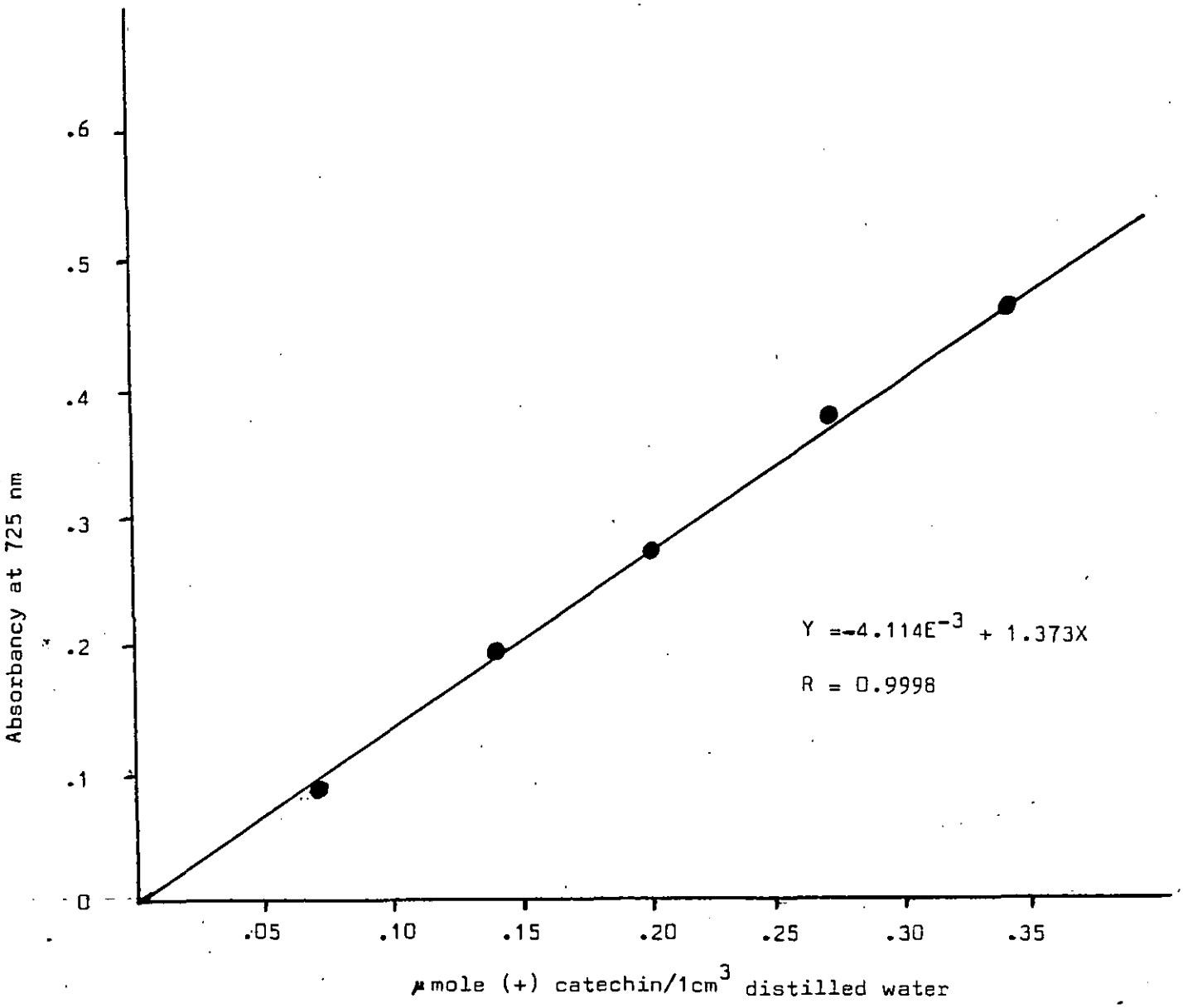

Figure 1 Standard curve for the determination of the amount of total soluble phenolic compounds and soluble tannin in dates.

Table 5 Calibration curve for the determination of total soluble phenolic compounds and soluble tannin in dates (as tannic acid).

mg tannic acid/100 cm^3 distilled water	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	μmole tannic acid/1 cm^3 solution[a]	molar extinction coefficient
14	0.525, 0.515, 0.520, 0.520, 0.531	0.522	5	0.82	12686
10	0.380, 0.380, 0.385, 0.382	0.382	4	0.59	12992
8	0.310, 0.302 0.305, 0.300	0.304	4	0.47	12936
6	0.230, 0.225 0.231, 0.234	0.230	4	0.35	13030
4	0.150, 0.152 0.151, 0.150	0.151	4	0.23	12850

(a) Concentrations of standard solutions before addition of reagents. The solutions used to determine O.D., after addition of reagents, are 10 times more dilute.[42]

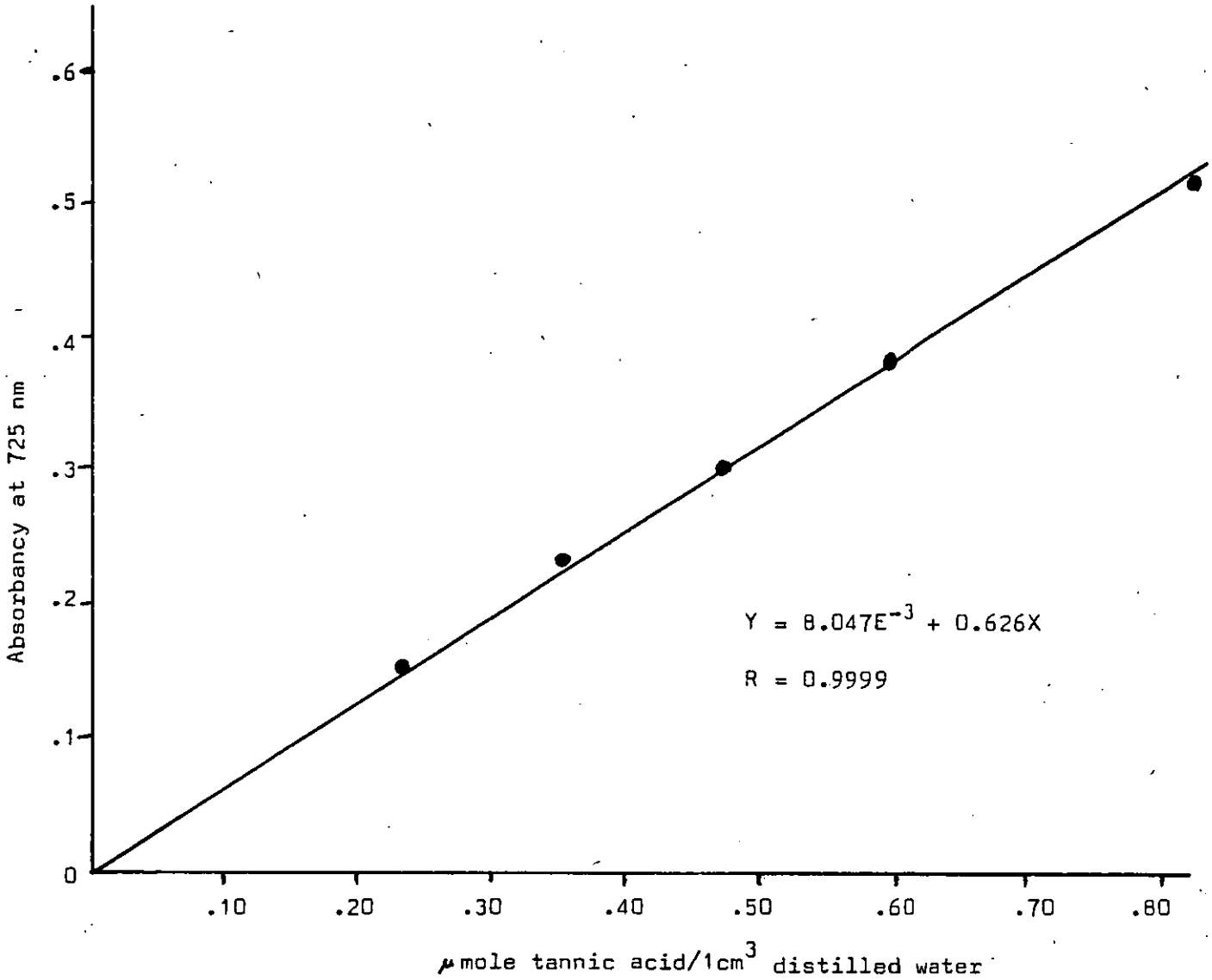

Figure 2 Standard curve for the determination of the amount of total soluble phenolic compounds and soluble tannin in dates.

Table 6 Calibration curve for the determination of sugars in dates (as glucose).

mg glucose/100 cm^3 distilled water	O.D at 532 nm 1 cm cell range	O.D at 532 nm 1 cm cell mean	number of replicates	μmole glucose/ 1 cm^3 solution [a]	molar extinction coefficient
50	0.300, 0.320 0.318, 0.295	0.308	4	2.7	2772
30	0.180, 0.185 0.176, 0.180	0.180	4	1.6	2699
20	0.115, 0.120 0.114, 0.132	0.120	4	1.1	2703
15	0.080, 0.095 0.100	0.092	3	0.8	2763
10	0.060, 0.062 0.058, 0.060	0.060	4	0.5	2703

(a) Concentrations of standard solutions before addition of reagents. The solutions used to determine O.D., after addition of reagents, are 25 times more dilute.

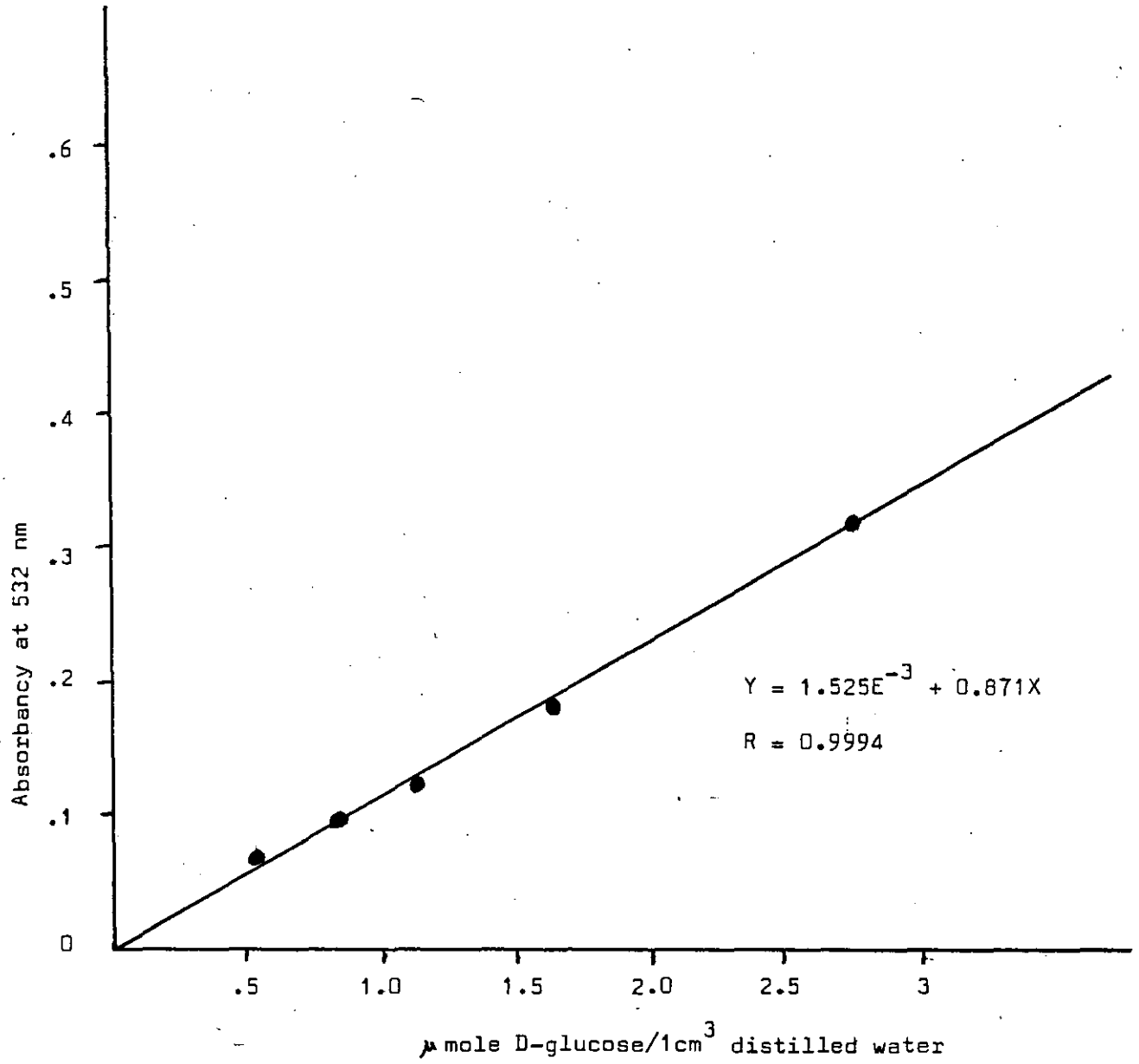

Figure 3 Standard curve for the determination of the amount of sugars in dates as (glucose) by the periodate and thiobarbituric acid reagents.

3.3 Effects of Variation in Storage Conditions at Room Temperature on the Chemical Composition of Dates

Table 7 The changes in pH, moisture, acidity, colour and total soluble solids during storage of Khadrawi dates (fully ripe) at room temperature.

type of treatment	% moisture	pH	% acidity	% TSS	colour O.D at 270 nm (1 cm cell)
Initial	18.5	5.4	0.180	39.6	0.554
Packed in vacuum	17.3	5.3	0.190	39.5	0.562
Packed in air	16.2	5.3	0.195	38.5	0.686
Flushed with pure oxygen	16.8	5.3	0.193	38.3	0.720

Table 8 The changes in total soluble phenolic compounds during storage of Khadrawi dates (fully ripe) at room temperature.

type of treatment	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	μmole (+) catechin/ $1cm^3$ solution	total phenol/100g dates [(a)]	μmole tannic acid/ $1cm^3$ solution	total phenol/100g dates [(b)]
initial	0.495, 0.495 0.490, 0.500 0.500	0.496	5	$4.2x10^{-3}$	0.36	192	0.77	241
packed in vacuum	0.500, 0.500 0.500, 0.495 0.500	0.499	5	$2.2x10^{-3}$	0.36	190	0.78	241
packed in air	0.500, 0.490 0.495, 0.495 0.495	0.495	5	$3.5x10^{-3}$	0.36	187	0.77	234
flushed with pure oxygen	0.485, 0.495 0.495, 0.500 0.490	0.493	5	$5.7x10^{-3}$	0.36	188	0.77	236

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

Table 9 The changes in insoluble leucoanthocyanidin tannin during storage of Khadrawi dates (fully ripe dates) at room temperature.

type of treatment	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
Initial	0.520, 0.525 0.555, 0.550, 0.560	0.542	5	0.018	6.434	29270	471
packed in vacuum	0.530, 0.550 0.540, 0.500, 0.525	0.529	5	0.019	5.644	29270	398
packed in air	0.240, 0.235 0.235, 0.260 0.255	0.245	5	0.012	6.262	29270	202
flushed with pure oxygen	0.152, 0.155 0.150, 0.148, 0.149	0.151	5	2.8×10^{-3}	6.068	29270	121

(a) Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

3.4 Effects of Freezing and Storage on the Chemical Composition of Dates

Table 10 The effect of freezing and storage on Zahdi dates (khalal ¼ ripe), stored at -13°C.

date of analysis	% moisture	pH	% acidity	% TSS	colour O.D at 270 nm 1 cm cell
7.9.1979	-	6.1	0.080	18	-
15.10.1979	42	6.2	0.062	21	0.291
10.12.1979	41	6.5	0.041	27	0.323
23.1.1980	40	6.3	0.055	30	0.340
25.2.1980	40	6.3	0.063	30	0.382
21.4.1980	39	6.2	0.070	30	0.454

Table 11 The effect of freezing and storage on the changes in total soluble phenolic compounds in Zahdi dates (khalal $\frac{1}{4}$ ripe), stored at -13°C.

date of analysis	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	µmole (+) catechin/1cm^3 solution	total phenol/100g dates (a)	µmole tannic acid/ 1cm^3 solution	total phenol/100g dates (b)
15.10.1979	0.413, 0.420 0.422, 0.430 0.431	0.423	5	7.5×10^{-3}	0.31	233	0.66	290
10.12.1979	0.403, 0.391 0.400, 0.390 0.381	0.393	5	8.7×10^{-3}	0.29	214	0.61	264
23.1.1980	0.371, 0.375 0.370, 0.381 0.365	0.372	5	6.0×10^{-3}	0.27	196	0.58	247
25.2.1980	0.352, 0.353 0.360, 0.350 0.355	0.354	5	3.8×10^{-3}	0.26	189	0.55	234
21.4.1980	0.320, 0.320 0.330, 0.325 0.330	0.325	5	5.0×10^{-3}	0.24	171	0.50	209

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

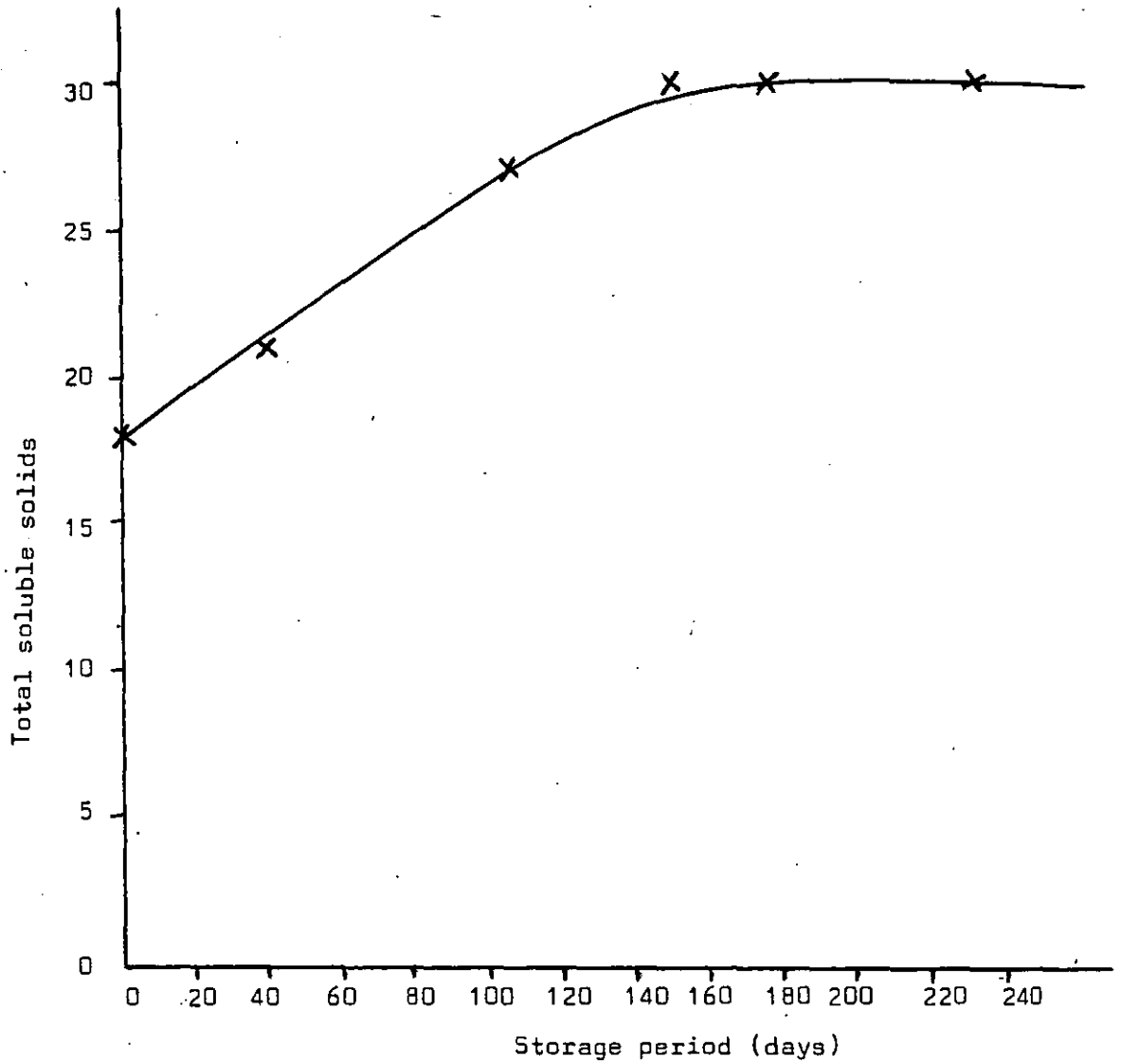

Figure 4 Effect of freezing and storage on total soluble solids changes in Zahdi dates (Khalal ¼ ripe), stored at -13°C.

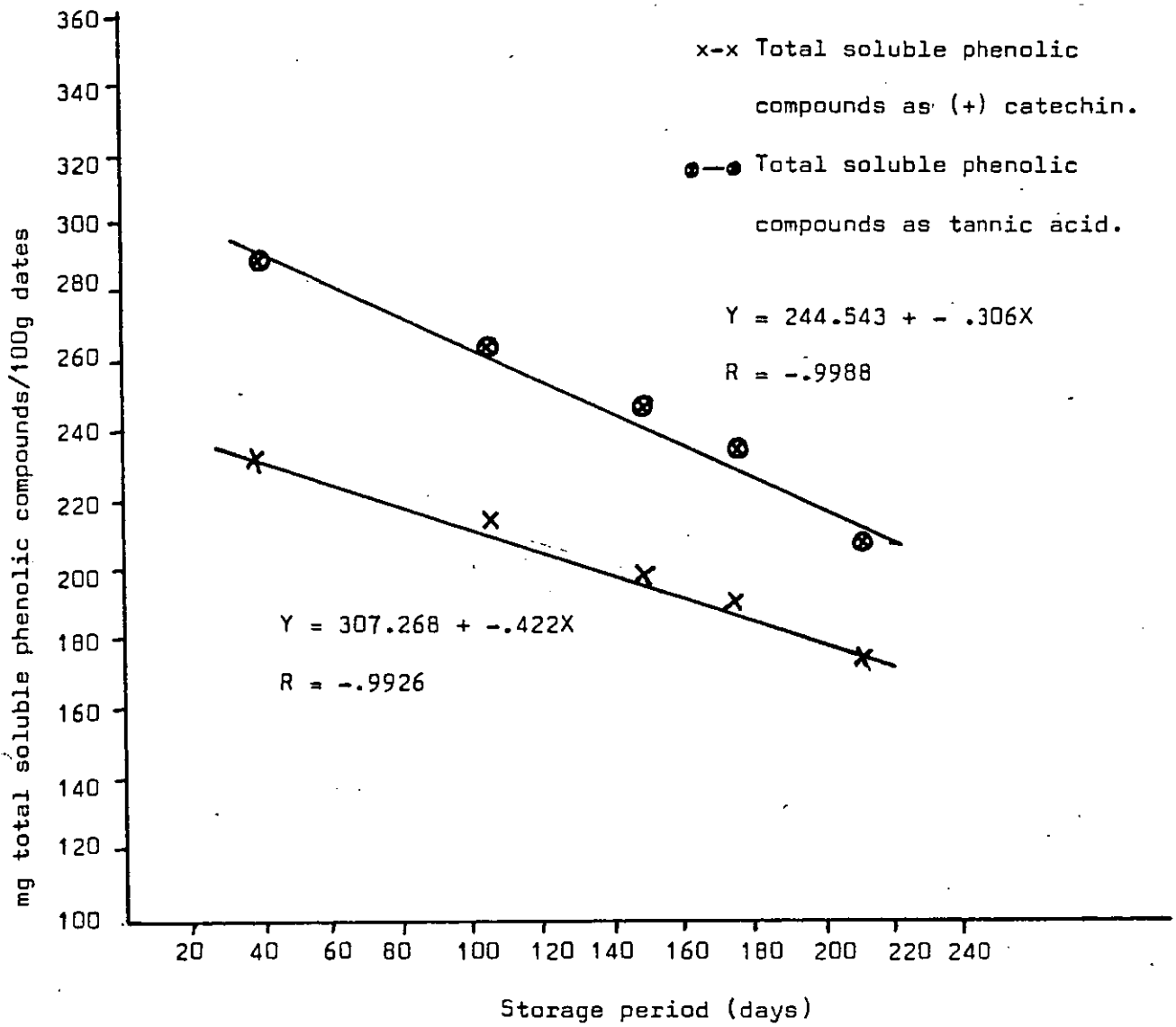

Figure 5 Effect of freezing and storage on changes in total soluble phenolic compounds in Zahdi dates (Khalal stage ½ ripe), stored at -13°C.

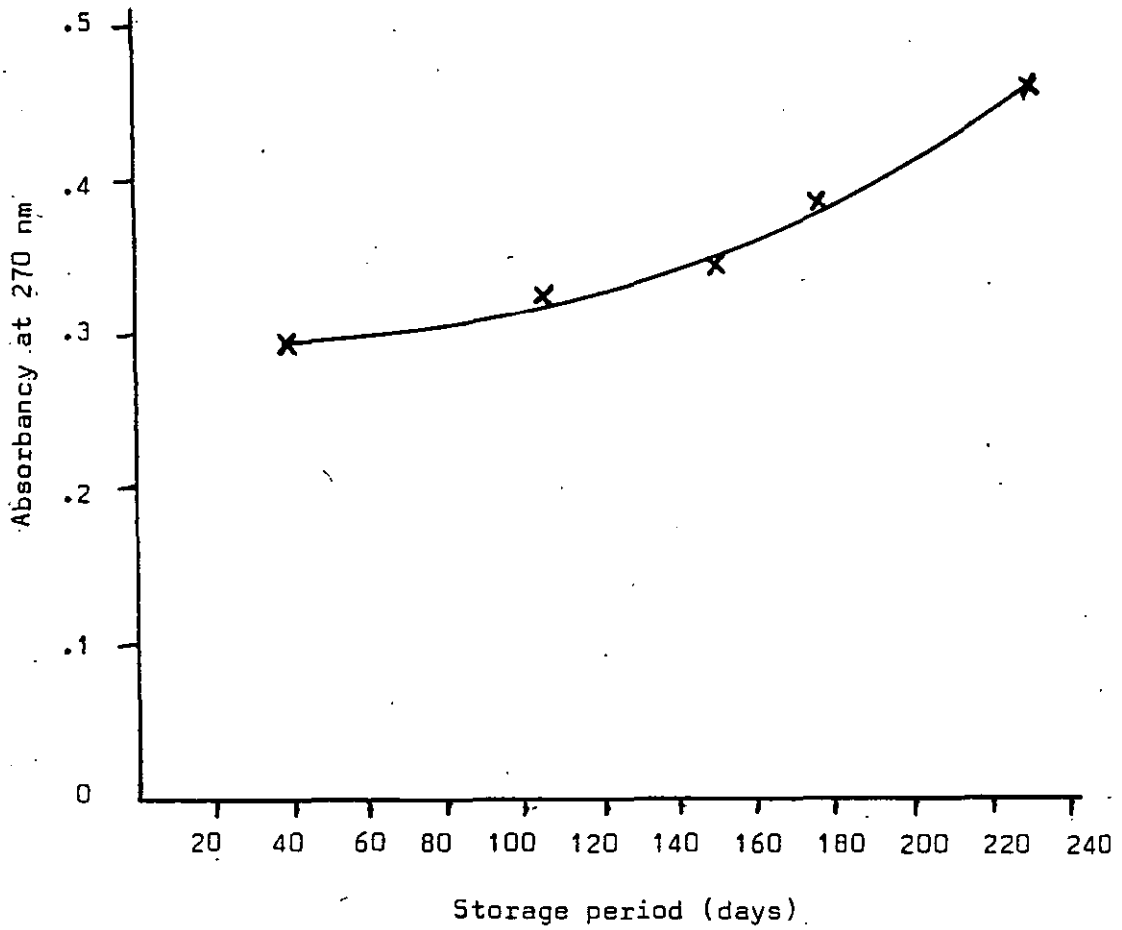

Figure 6 Effect of freezing and storage on colour changes in Zahdi dates (khalal ½ ripe), stored at -13°C.

Table 12 The effect of freezing and storage on the changes in insoluble leucoanthocyanidin tannin in Zahdi dates (khalal ¼ ripe), stored at -13°C.

date of analysis	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
15.10.1979	0.220, 0.235 0.206, 0.205 0.240	0.221	5	0.016	5.752	29270	241
10.12.1979	0.248, 0.256 0.292, 0.301 0.266	0.273	5	0.023	6.394	29270	326
23.1.1980	0.322, 0.320 0.345, 0.350 0.332	0.334	5	0.013	5.661	29270	353
25.2.1980	0.550, 0.532 0.523, 0.514 0.545	0.533	5	0.015	4.252	29270	416
24.4.1980	0.820, 0.830 0.832, 0.840 0.840	0.832	5	8.3×10^{-3}	4.375	29270	657

(a) Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

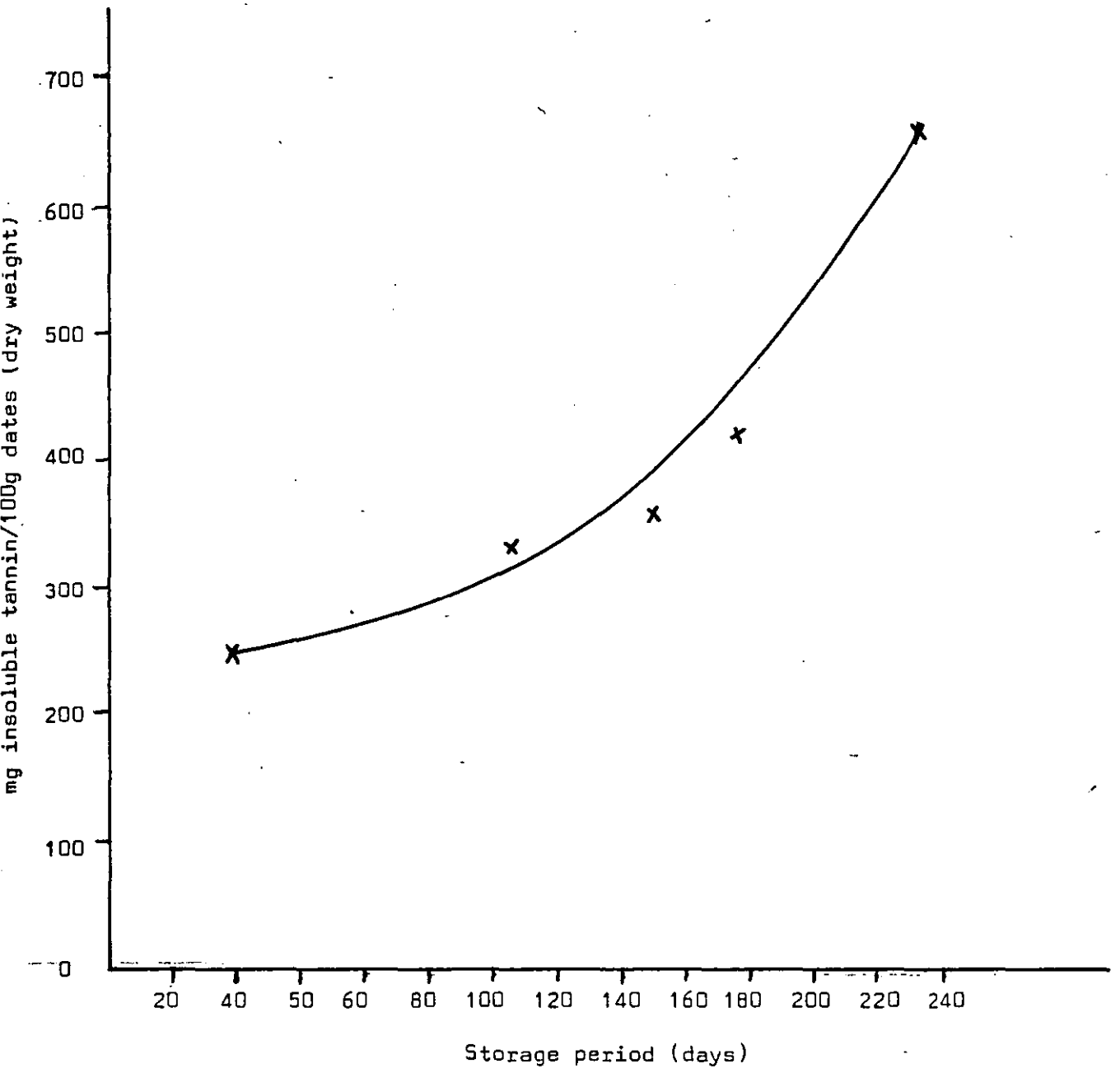

Figure 7 Effect of freezing and storage on the insoluble leucoanthocyanidin tannin changes in Zahdi dates (khalal ¼ ripe), stored at -13°C.

Table 13 The effect of storage at -13°C and storage at room temperature (18°C ± 1) on the colour changes of the tissue residue,[a] stored for 1 month exposed to air.

variety of dates	type of treatment of the tissue residue	O.D. at 400 nm[b] (1 cm cell)	colour
Zahdi fully ripe dates	initial	0.053	reddish-brown
	storage at room temperature	0.120	dark
	storage at -13°C	0.052	reddish-brown
Khadrawi fully ripe dates	initial	0.078	slightly dark
	storage at room temperature	0.218	dark
	storage at -13°C	0.085	slightly dark

(a) 160g dates were extracted with 750 cm^3 boiling water (distilled water) for 45 minutes. Then the juice was filtered by using muslin. The residue was used for this investigation.

(b) The extraction of the colour of the tissue residue was done according to procedure published by other workers.[49] All the measurements were carried out on a recording spectrophotometer SP800. 65% methanol was used as a blank.

3.5 Effects of Blanching and Storage on the Chemical Composition of Dates

Table 14 The effect of blanching and storage on Zahdi dates (tamar stage-dates halved), stored at room temperature.

period of blanching (minutes)	% moisture	% increase in weight after blanching	pH	% acidity	% TSS	colour O.D at 270 nm (1 cm cell)
Initial	14.3	-	5.1	0.170	40.0	0.472
0	13.3	-	5.0	0.175	39.5	0.434
1	20.1	4.86	5.0	0.173	37.2	0.343
2	22.9	8.78	5.0	0.176	35.3	0.281
3[a]	24.6	9.73	5.0	0.178	33.5	0.237

(a) Over this period of treatment the texture will be broken down and the dates become soft.

Table 15 The effect of blanching and storage on the changes in total soluble phenolic compounds in Zahdi dates (tamar stage - dates halved), stored at room temperature.

period of blanching (minutes)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	μmole (+) catechin/1cm^3 solution	total phenol/[a] 100g dates	μ mole tannic acid/ 1cm^3 solution	total phenol/[b] 100g dates
initial	0.480, 0.480 0.480, 0.460 0.470	0.474	5	8.9×10^{-3}	0.35	178	0.73	217
0	0.410, 0.405 0.400, 0.410 0.410	0.407	5	4.5×10^{-3}	0.30	151	0.63	185
1	0.365, 0.370 0.375, 0.375 0.380	0.373	5	5.7×10^{-3}	0.27	147	0.58	185
2	0.340, 0.340 0.345, 0.340 0.345	0.342	5	2.7×10^{-3}	0.25	141	0.53	175
3	0.285, 0.285 0.290, 0.300 0.300	0.292	5	7.6×10^{-3}	0.21	121	0.45	152

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

Table 16 The effect of blanching and storage on the changes in insoluble leucoanthocyanidin tannin in Zahdi dates (tamar stage), stored at room temperature.

period of blanching (minutes)	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	Insoluble tannin[a]/100g dates
initial	0.610, 0.620 0.590, 0.580 0.600	0.600	5	0.0158	5.716	29270	441
0	0.550, 0.495 0.570, 0.530 0.520	0.533	5	0.0286	6.438	29270	436
1	0.560, 0.580 0.550, 0.570 0.540	0.560	5	0.0158	5.588	29270	431
2	0.510, 0.520 0.540, 0.530 0.500	0.520	5	0.0158	5.536	29270	411
3	0.490, 0.465 0.450, 0.450 0.460	0.463	5	0.0164	5.330	29270	361

(a) Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

Table 17 The effect of blanching and storage on the changes in sugars in Zahdi dates (tamar stage), stored at room temperature.

	Lane & Eynon method					thiobarbituric acid method						
period of blanching (minutes)	titre reading[a] before inversion	titre reading[a] after inversion	% invert sugar	% sucrose	% total sugar	O.D at 532 nm before inversion	O.D at 532 nm after inversion	μ mole glucose/ $1cm^3$ before inversion	μ mole glucose/ $1cm^3$ after inversion	% invert sugar	% sucrose	% total sugar
initial	20.3	18.9	71.5	4.6	76.1	0.310, 0.320, 0.314	0.345, 0.350, 0.370	2.7	3.1	70.9	10.0	80.9
0	18.8	18.1	75.7	3.2	78.9	0.327, 0.333	0.360, 0.352	2.8	3.0	72.7	4.9	77.6
1	23.0	22.0	67.4	2.8	70.2	0.280, 0.275	0.300, 0.285	2.4	2.5	67.6	2.7	70.3
2	23.6	23.4	68.1	0.5	68.6	0.275, 0.265	0.270, 0.270	2.3	2.3	67.1	0.00	67.1
3	24.2	24.1	67.9	0.3	68.2	0.245, 0.250	0.255, 0.240	2.1	2.1	62.4	0.00	62.4

The amount of sugars calculated (as dry weight).

(a) Each reading was repeated three times and the mean of the reading was taken.

Table 18 The effect of blanching on the polyphenolase activity in Zahdi dates[a] (tamar stage - dates halved).

period of blanching (minutes)	temperature inside dates before and during blanching ^{o}C	polyphenolase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.1	0.280	100
1	86.8	0.086	30.7
2	94.4	0.042	15.0
3	100.0	0.021	7.5
4	101.2	0.000	0.0

(a) The thickness, diameter, length and weight of Zahdi dates before blanching or treated by microwave heating, were measured for eighty-four fruit. The average was taken. Thickness ranged from 4.05-4.40 mm (mean 4.23 mm), diameter ranged from 18.12-19.43 mm (mean 18.68 mm), weight ranged from 7.023-7.512g (mean 7.386g) and length ranged from 2.95-3.33 cm (mean 3.09 cm).

Table 19 The effect of blanching on the peroxidase activity in Zahdi dates[a] (tamar stage - dates halved).

period of blanching (minutes)	temperature inside dates before and during blanching °C	peroxidase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.1	0.040	100
1	86.8	0.021	52.5
2	94.4	0.012	30.0
3	100.0	0.000	0.0
4	101.2	0.000	0.0

(a) Divisions of the fruit are as given in Table 18.

3.6 Effects of Microwave Heating and Storage on the Chemical Composition of Dates

Table 20. The effect of microwave heating on Zahdi dates (fully ripe-whole dates).

microwave heating time (seconds)	% moisture	pH	% acidity	% TSS
0	13.8	5.0	0.170	41.0
15	13.7	5.0	0.172	40.5
20	13.6	5.0	0.175	40.2
25	13.6	5.0	0.176	40.0
30	13.3	4.9	0.183	40.0
40	13.1	4.9	0.185	40.0
50	13.0	4.9	0.187	39.8
60	12.8	4.8	0.188	39.6

Table 21 The effect of microwave heating and storage on Zahdi dates (fully ripe-whole dates), stored at room temperature.

microwave heating time (seconds)	% moisture	pH	% acidity	% TSS	colour O.D at 270 nm 1 cm cell
0	12.7	5.0	0.170	41.0	0.590
15	12.7	5.1	0.165	41.0	0.538
20	12.5	5.1	0.168	41.4	0.545
25	12.4	5.0	0.168	41.5	0.576
30	12.2	5.0	0.171	42.0	0.614
40	12.0	5.0	0.174	42.0	0.620
50	11.9	5.0	0.175	42.2	0.632
60	11.7	5.0	0.176	42.5	0.640

Table 22 The effect of microwave heating on the changes in the total soluble phenolic compounds in Zahdi dates (fully ripe - whole dates).

microwave heating time (seconds)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	µmole (+) catechin/1cm^3 solution	total phenol/100g dates[a]	µmole tannic acid/ 1cm^3 solution	total phenol/100g dates[b]
initial	0.555, 0.550 0.555, 0.555 0.555	0.591	5	2.2×10^{-3}	0.40	202	0.86	254
15	0.500, 0.500 0.505, 0.505 0.515	0.505	5	6.1×10^{-3}	0.37	187	0.79	234
20	0.495, 0.500 0.505, 0.500 0.500	0.500	5	3.5×10^{-3}	0.37	186	0.78	230
25	0.500, 0.500 0.505, 0.505 0.500	0.502	5	2.7×10^{-3}	0.37	186	0.78	230
30	0.515, 0.520 0.520, 0.520 0.520	0.519	5	2.2×10^{-3}	0.38	191	0.82	241
40	0.554, 0.552 0.555, 0.555 0.560	0.555	5	2.9×10^{-3}	0.41	205	0.85	250
50	0.555, 0.560 0.554, 0.546 0.556	0.554	5	5.1×10^{-3}	0.40	200	0.85	249
60	0.560, 0.555 0.556, 0.565 0.560	0.559	5	4.0×10^{-3}	0.41	205	0.88	257

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

Table 23 The effect of microwave heating and storage on the changes in total soluble phenolic compounds in Zahdi dates (fully ripe - whole dates), stored at room temperature.

microwave heating time (seconds)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	μmole (+) catechin/1cm^3 solution	total phenol/[a] 100g dates	μ mole tannic acid/ 1cm^3 solution	total phenol/[b] 100g dates
0	0.520, 0.525 0.525, 0.530 0.530	0.526	5	4.2×10^{-3}	0.38	190	0.82	240
15	0.450, 0.455 0.460, 0.440 0.445	0.450	5	7.9×10^{-3}	0.33	165	0.70	205
20	0.445, 0.440 0.437, 0.435 0.443	0.440	5	4.1×10^{-3}	0.32	159	0.69	201
25	0.486, 0.485 0.490, 0.495 0.490	0.489	5	4.0×10^{-3}	0.36	179	0.76	221
30	0.505, 0.502 0.510, 0.513 0.515	0.509	5	5.4×10^{-3}	0.37	183	0.80	232
40	0.533, 0.531 0.528, 0.529 0.528	0.530	5	2.2×10^{-3}	0.39	193	0.83	241
50	0.559, 0.564 0.554, 0.556 0.569	0.560	5	6.1×10^{-3}	0.41	203	0.88	255
60	0.572, 0.565 0.575, 0.570 0.560	0.568	5	5.9×10^{-3}	0.42	207	0.89	257

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

Table 24 The effect of microwave heating on the changes in insoluble leucoanthocyanidin tannin in Zahdi dates (fully ripe - whole dates).

microwave heating time (seconds)	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
Initial	0.675, 0.660 0.685, 0.690 0.700	0.682	5	0.015	5.444	29270	475
15	0.612, 0.625 0.636, 0.650 0.640	0.633	5	0.014	5.182	29270	419
20	0.625, 0.600 0.610, 0.565 0.585	0.597	5	0.023	5.364	29270	408
25	0.535, 0.505 0.560, 0.555 0.550	0.541	5	0.022	5.416	29270	374
30	0.595, 0.565 0.570, 0.560 0.580	0.574	5	0.014	5.014	29270	366
40	0.445, 0.440 0.470, 0.460 0.455	0.454	5	0.012	5.876	29270	338
50	0.465, 0.480 0.500, 0.500 0.475	0.484	5	0.015	5.463	29270	335
60	0.462, 0.475 0.505, 0.480 0.485	0.481	5	0.016	5.324	29270	323

[a] Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

Table 25 The effect of microwave heating and storage on the changes in insoluble leucoanthocyanidin tannin in Zahdi dates (fully ripe - whole dates), stored at room temperature.

microwave heating time (seconds)	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
0	0.550, 0.560 0.575, 0.620 0.600	0.581	5	0.029	5.920	29270	434
15	0.610, 0.615 0.625, 0.670 0.645	0.633	5	0.025	5.094	29270	407
20	0.620, 0.605 0.615, 0.625 0.600	0.613	5	0.010	5.038	29270	389
25	0.510, 0.515 0.520, 0.495 0.490	0.506	5	0.013	5.566	29270	354
30	0.440, 0.415 0.394, 0.445 0.443	0.427	5	0.022	5.946	29270	319
40	0.420, 0.430 0.390, 0.400 0.380	0.404	5	0.021	5.882	29270	297
50	0.495, 0.480 0.442, 0.400 0.425	0.448	5	0.039	5.262	29270	295
60	0.330, 0.350 0.375, 0.345 0.365	0.353	5	0.017	5.867	29270	258

(a) Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

Table 26 The effect of microwave heating on the polyphenolase activity in Zahdi dates[a] (tamar stage - whole dates).

microwave heating time (seconds)	temperature inside dates before and after microwave heating (°C)	polyphenolase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.1	0.280	100
15	58.4	0.242	86.4
20	78.2	0.212	75.7
25	90.2	0.170	60.7
30	100.1	0.125	44.6
40	105.2	0.075	26.8
50	111.2	0.012	4.3
60	112.1	0.000	0.0

(a) Divisions of the fruit are as given in Table 18.

Table 27 The effect of microwave heating on the peroxidase activity in Zahdi dates[a] (tamar stage - whole dates).

microwave heating time (seconds)	temperature inside dates before and after microwave heating (°C)	peroxidase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.1	0.040	100
15	58.4	0.021	68.5
20	78.2	0.015	37.5
25	90.2	0.012	30.0
30	100.1	0.004	10.0
40	105.2	0.000	0.0
50	111.2	0.000	0.0
60	112.1	0.000	0.0

(a) Divisions of the fruit are as given in Table 18.

Figure 8 The effect of microwave heating on Zahdi dates (fully ripe). Peroxidase test was carried out*.

1 - Untreated date. 2 - Treated for 15 seconds.*

3 - Treated for 20 seconds.* 4 - Treated for 30 seconds.*

5 - Treated for 40 seconds.* (See p. 110)

Figure 9 The effect of microwave heating on Khadrawi dates (fully ripe). Peroxidase test was carried out*

1 - Untreated date. 2 - Treated for 15 seconds.*

3 - Treated for 20 seconds.* 4 - Treated for 25 seconds.*

5 - Treated for 30 seconds.* (See p. 110)

Table 28 The effect of microwave heating on Khadrawi dates (fully ripe-whole dates).

microwave heating time (seconds)	% moisture	pH	% acidity	% TSS
0	16.1	5.2	0.170	40.5
10	15.8	5.2	0.174	40.3
15	15.5	5.1	0.180	40.0
20	15.4	5.1	0.183	39.8
25	15.2	5.1	0.185	39.5
30[a]	15.0	5.0	0.190	39.0
40	15.0	5.0	0.192	38.7
50	14.7	5.0	0.195	38.5
60	14.5	4.9	0.198	38.0

(a) At longer periods than 30 seconds the dates were damaged by excess heating by the microwave heating.

Table 29 The effect of microwave heating on the changes in total soluble phenolic compounds in Khadrawi dates (fully ripe - whole dates).

microwave heating time (seconds)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	µmole (+) catechin/1cm^3 solution	total phenol/[a] 100g dates	µmole tannic acid/ 1 cm^3 solution	total phenol/[b] 100g dates
0	0.620, 0.620 0.630, 0.620 0.635	0.625	5	7.1×10^{-3}	0.46	239	0.97	295
10	0.595, 0.595 0.600, 0.605 0.610	0.601	5	6.5×10^{-3}	0.44	227	0.94	285
15	0.575, 0.575 0.580, 0.585 0.585	0.580	5	5.0×10^{-3}	0.42	216	0.91	275
20	0.600, 0.600 0.595, 0.595 0.600	0.598	5	2.7×10^{-3}	0.44	226	0.93	280
25	0.610, 0.610 0.615, 0.600 0.600	0.607	5	6.7×10^{-3}	0.45	231	0.95	286
30	0.621, 0.625 0.623, 0.622 0.623	0.623	5	1.5×10^{-3}	0.45	236	0.97	291
40	0.620, 0.630 0.625, 0.628 0.625	0.626	5	3.8×10^{-3}	0.46	236	0.97	291
50	0.635, 0.625 0.630, 0.620 0.625	0.627	5	5.7×10^{-3}	0.46	235	0.97	290
60	0.628, 0.630 0.625, 0.628 0.635	0.629	5	3.7×10^{-3}	0.46	234	0.98	292

(a) Total soluble phenolic compounds calculated as mg (+) catechin/100g dates (dry weight).

(b) Total soluble phenolic compounds calculated as mg tannic acid/100g dates (dry weight).

Table 30 The effect of microwave heating on the changes in insoluble leucoanthocyanidin tannin in Khadrawi dates (fully ripe - whole dates).

microwave heating time (seconds)	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
0	0.520, 0.525 0.555, 0.550 0.560	0.542	5	0.018	6.434	29270	458
10	0.576, 0.590 0.635, 0.610 0.600	0.602	5	0.022	5.776	29270	455
15	0.575, 0.565 0.564, 0.540 0.560	0.561	5	0.013	5.852	29270	428
20	0.545, 0.485 0.552, 0.495 0.480	0.511	5	0.034	6.214	29270	414
25	0.525, 0.555 0.535, 0.500 0.530	0.529	5	0.020	5.462	29270	375
30	0.410, 0.450 0.470, 0.465	0.448	5	0.024	5.848	29270	340
40	0.380, 0.388 0.400, 0.405 0.375	0.390	5	0.013	5.623	29270	284
50	0.385, 0.395 0.375, 0.370 0.350	0.375	5	0.017	5.745	29270	278
60	0.395, 0.380 0.405, 0.370 0.355	0.381	5	0.020	5.472	29270	269

(a) Insoluble leucoanthocyanidin tannin calculated as mg cyanidin chloride/100g dates (dry weight).

Table 31 The effect of microwave heating on the polyphenolase activity in Khadrawi dates[a] (tamar stage - whole dates).

microwave heating time (seconds)	temperature inside dates before and after microwave heating (°C)	polyphenolase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.2	0.484	100
10	48.0	0.405	83.7
15	60.0	0.375	77.5
20	78.0	0.322	66.5
25	92.0	0.273	56.4
30	108.0	0.186	38.4
40	112.4	0.146	30.2
50	118.4	0.110	22.7
60	122.6	0.040	8.3

(a) The thickness, diameter, length and weight of Khadrawi dates before microwave heating, were measured for forty-eight fruit . The average was taken. The thickness ranged from 4.20-4.62 nm (mean 4.46), diameter ranged from 19.05-20.90 mm (mean 19.26 mm), weight ranged from 7.803-8.826g (mean 8.363g) and length ranged from 3.52-4.20 cm (mean 3.65 cm).

Table 32 The effect of microwave heating on the peroxidase activity in Khadrawi dates[a] (tamar stage - whole dates).

microwave heating time (seconds)	temperature inside dates before and after microwave heating (°C)	peroxidase activity (Δ absorbance/minute)	residual enzyme activity %
0	21.2	0.150	100
10	48.0	0.148	98.7
15	60.0	0.142	94.7
20	78.0	0.120	80.0
25	92.0	0.092	61.3
30	108.0	0.044	29.3
40	112.4	0.021	14.0
50	118.4	0.000	0.0
60	122.6	0.000	0.0

(a) Divisions of the fruit are as given in table 31.

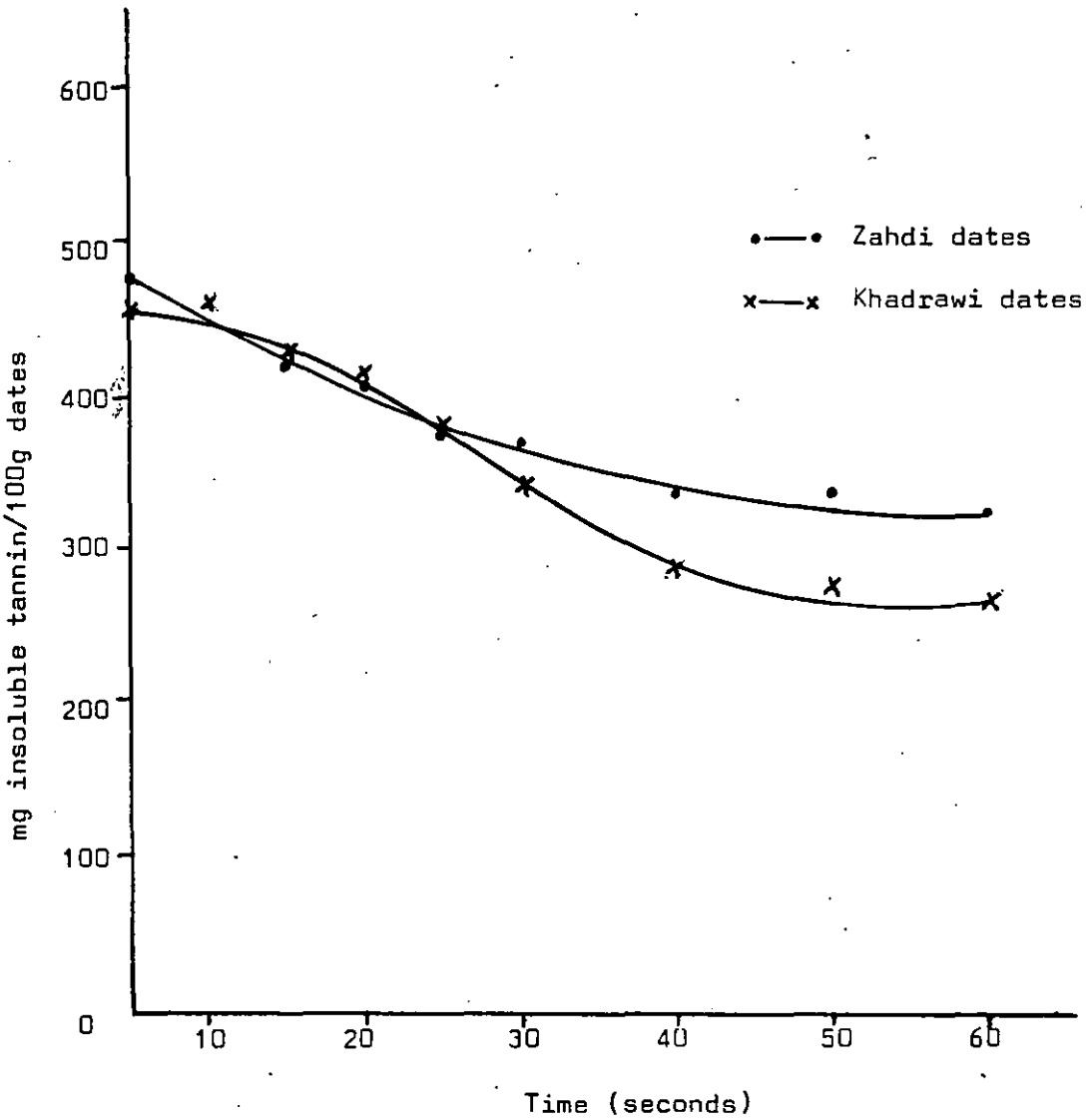

Figure 10 Effect of time on the changes in insoluble leucoanthocyanidin tannin in the fully ripe dates during treatment by microwave heating.

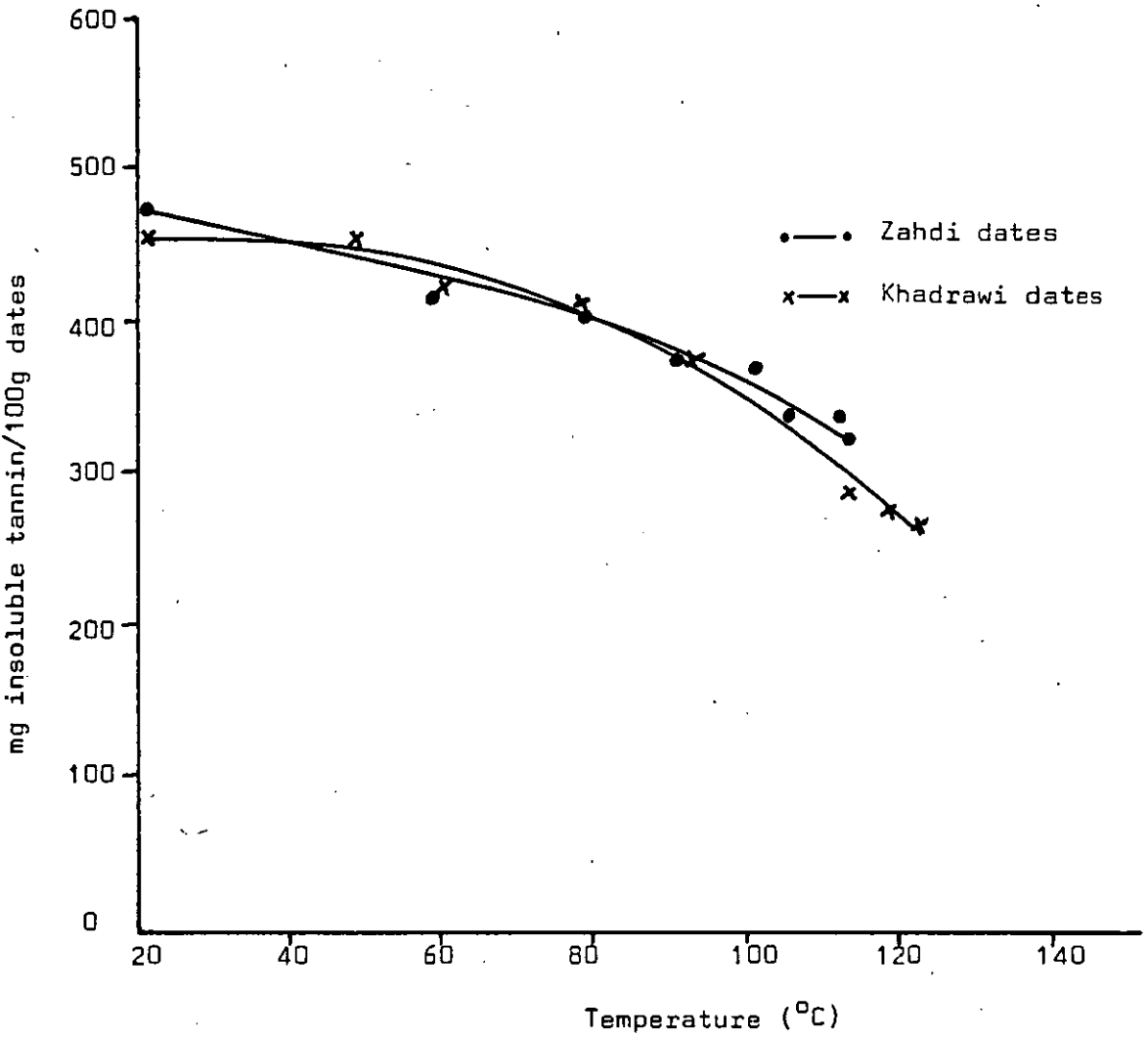

Figure 11 Effect of temperature on the changes in insoluble leucoanthocyanidin tannin in the fully ripe dates during treatment by microwave heating.

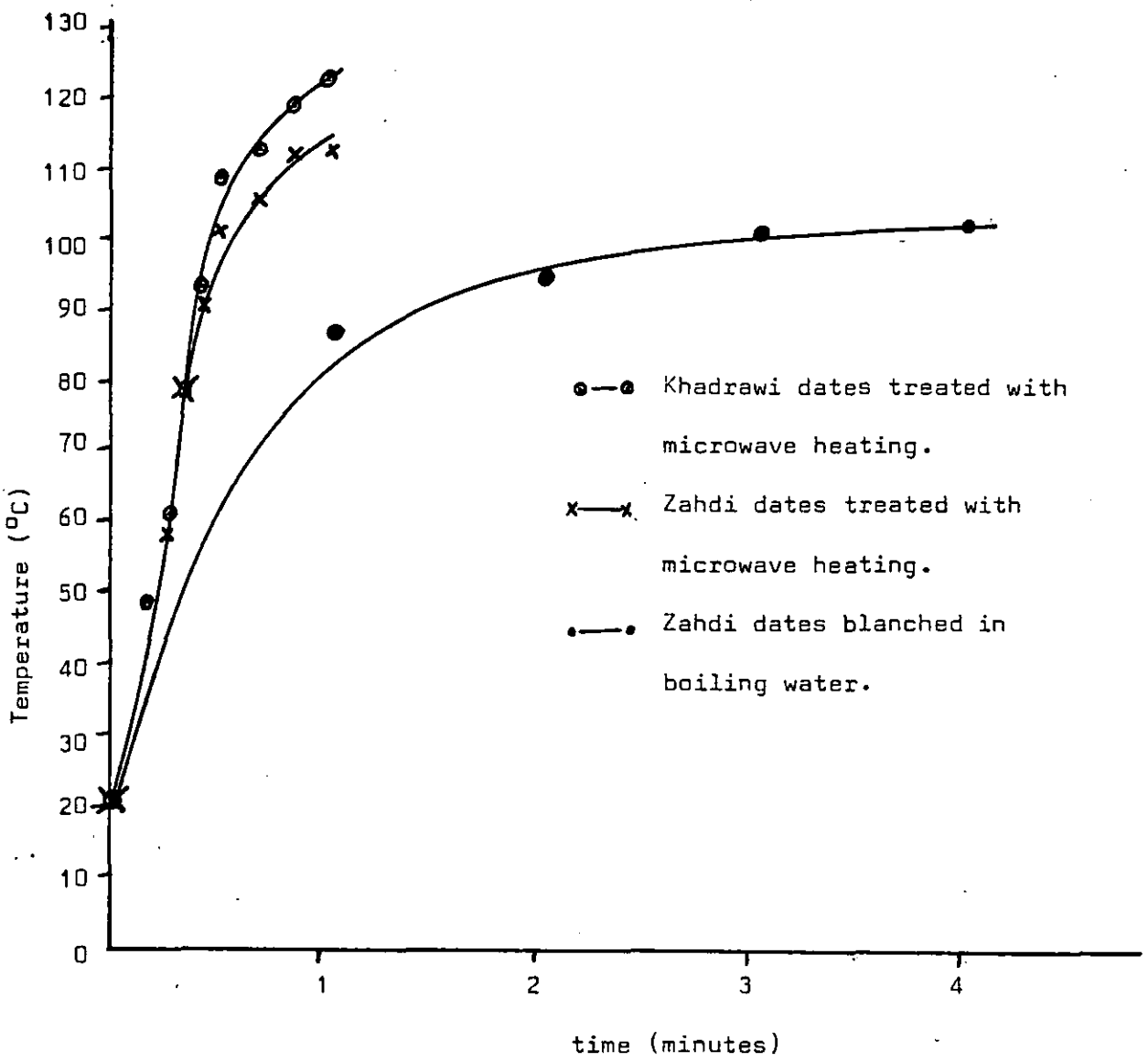

Figure 12 The relationship between time and temperature in inactivation of polyphenolase and peroxidase in dates.

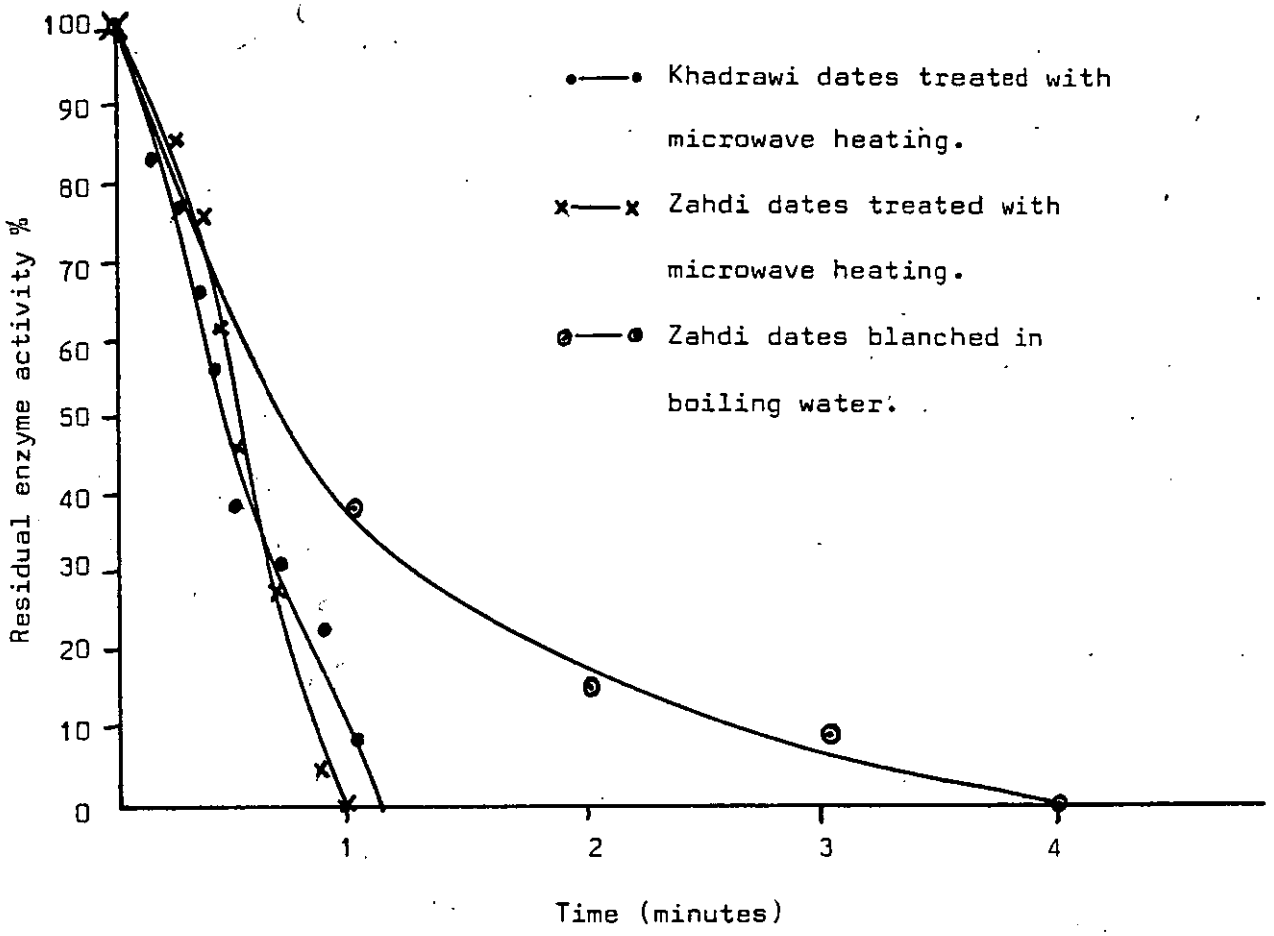

Figure 13 Effect of time of heating on polyphenolase activity in dates.

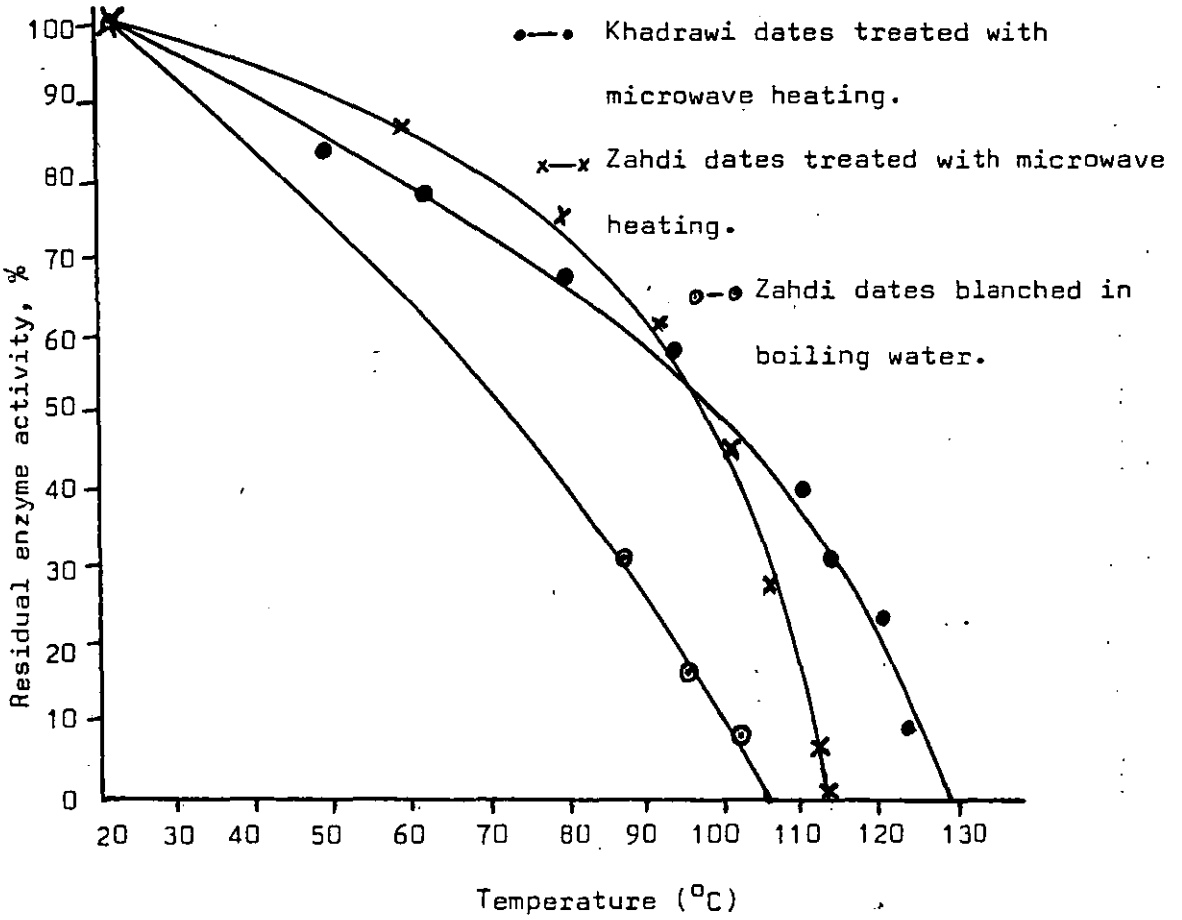

Figure 14 Effect of temperature on polyphenolase activity in dates.

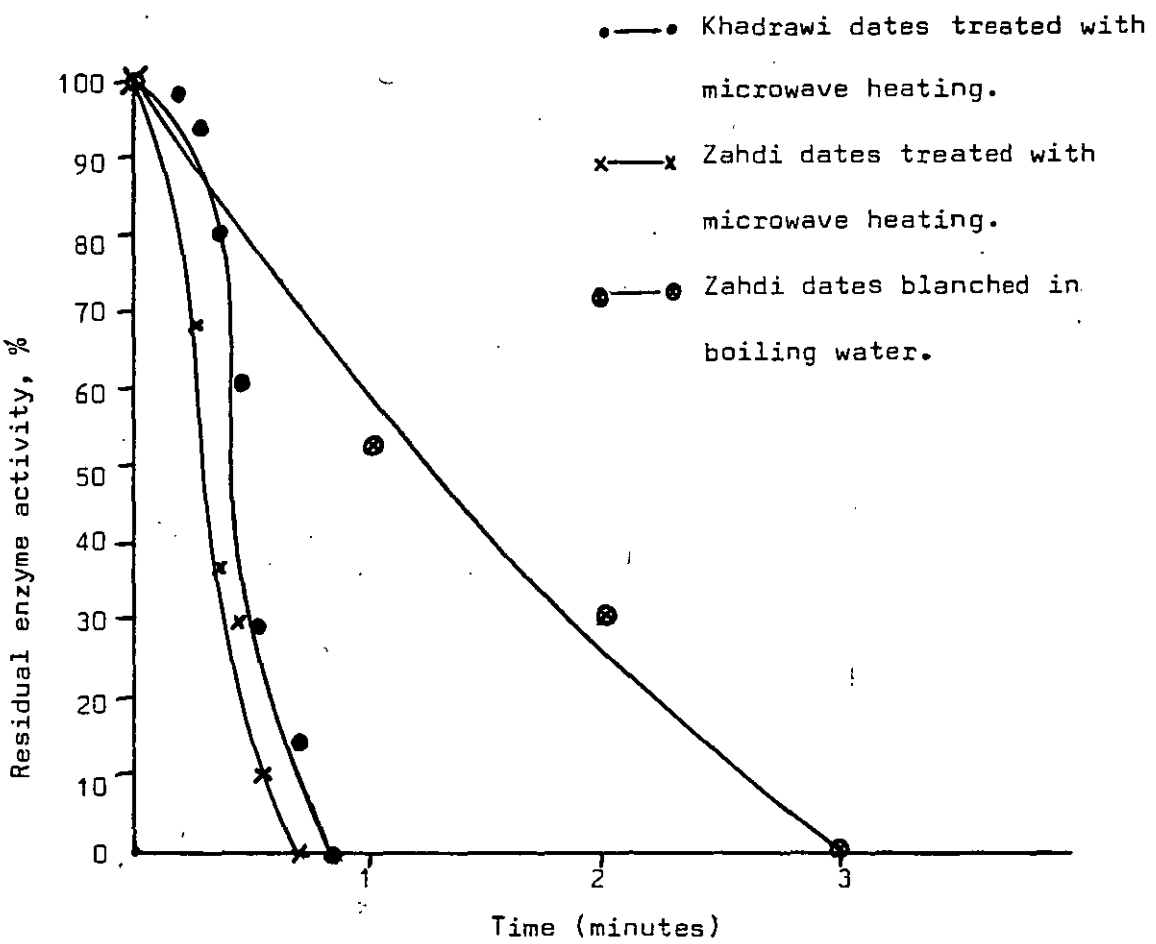

Figure 15 Effect of time of heating on peroxidase activity in dates.

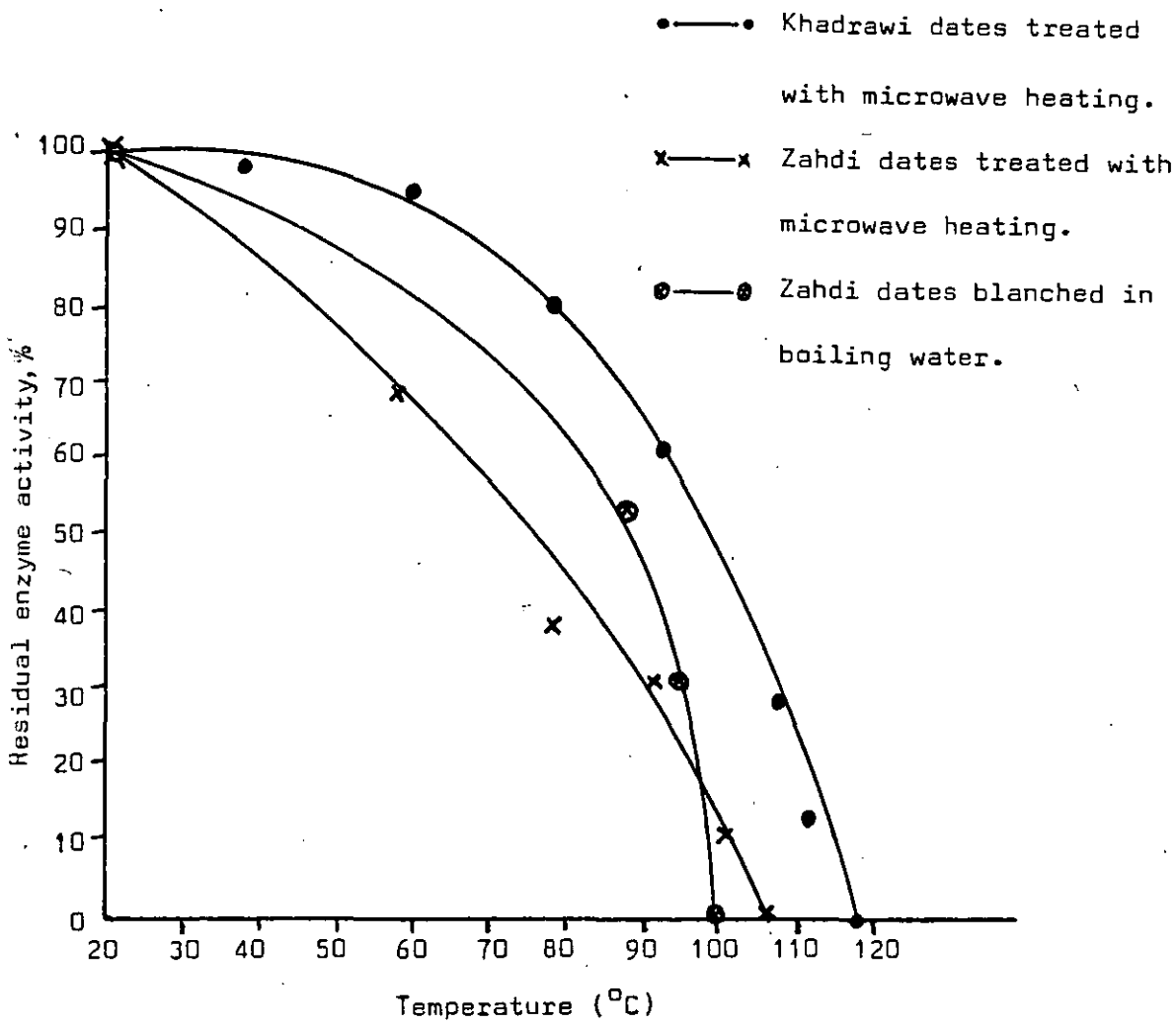

Figure 16 Effect of temperature on peroxidase activity in dates.

Table 33 The effect of microwave heating on Zahdi dates (green stage-whole dates).

microwave heating time (seconds)	% moisture	pH	% acidity	% TSS	colour O.D at 270 nm (1 cm cell)
0	74.1	5.6	0.160	6.0	0.294
(a)	73.4	5.2	0.171	6.5	0.335
20	72.8	5.5	0.165	6.0	0.365
30	70.2	5.5	0.167	6.3	0.445
40	67.7	5.3	0.168	6.5	0.612
50	64.3	5.1	0.170	7.0	0.712
60	60.8	5.0	0.171	8.0	0.745
(a)	72.9	4.9	0.170	9.0	0.746

(a) The samples were stored in the freezer (-13^{o}C) for 2 months, packed in LDPE bags.

Table 34 The effect of microwave heating on the changes in the total soluble phenolic compounds in Zahdi dates (green stage - whole dates).

microwave heating time (seconds)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	µmole (+) catechin/1cm^3 solution	total phenol/[a] 100g dates	µmole tannic acid/ 1cm^3 solution	total phenol/[b] 100g dates
0	0.668, 0.665 0.670, 0.670 0.672	0.669	5	2.64×10^{-3}	0.50	0.84	1.03	1.01
(c)	0.560, 0.560 0.565, 0.570 0.755	0.566	5	6.52×10^{-3}	0.42	0.69	0.88	0.84
20	0.710, 0.715 0.715, 0.720 0.725	0.717	5	5.70×10^{-3}	0.53	0.85	1.12	1.05
30	0.695, 0.695 0.690, 0.685 0.695	0.692	5	4.47×10^{-3}	0.51	0.74	1.08	0.92
40	0.720, 0.725 0.725, 0.730 0.735	0.727	5	5.70×10^{-3}	0.53	0.71	1.13	0.89
50	0.725, 0.740 0.735, 0.740 0.745	0.737	5	7.58×10^{-3}	0.54	0.66	1.15	0.82
60	0.765, 0.760 0.750, 0.752 0.768	0.769	5	7.90×10^{-3}	0.56	0.62	1.20	0.78
(c)	0.650, 0.650 0.640, 0.635 0.638	0.643	5	6.98×10^{-3}	0.47	0.75	1.00	0.94

(a) Soluble phenolic compounds calculated as g (+) catechin/100g dates (dry weight).

(b) Soluble phenolic compounds calculated as g tannic acid/100g dates (dry weight).

(c) The samples were stored in the freezer (-13°C) for 2 months, packed in LDPE bags.

Table 35 The effect of microwave heating on the changes in soluble leucoanthocyanidin tannin in Zahdi dates (green stage - whole dates).

microwave heating time (seconds)	O.D at 725 nm 0.5 cm cell range	O.D at 725 nm 0.5 cm cell mean	number of replicates	standard deviation	µmole (+) catechin/1cm^3 solution	tannin/(a) 100g dates	µmole tannic acid/ 1cm^3 solution	tannin/(b) 100g dates
0	0.579, 0.577 0.580, 0.580 0.585	0.580	5	2.95×10^{-3}	0.43	0.72	0.90	0.89
(c)	0.505, 0.510 0.510, 0.515 0.515	0.511	5	4.18×10^{-3}	0.37	0.60	0.80	0.77
20	0.572, 0.577 0.577, 0.582 0.587	0.579	5	5.70×10^{-3}	0.43	0.69	0.90	0.84
30	0.569, 0.571 0.571, 0.581 0.583	0.575	5	6.48×10^{-3}	0.42	0.61	0.90	0.77
40	0.580, 0.580 0.570, 0.575 0.572	0.575	5	4.56×10^{-3}	0.42	0.57	0.89	0.70
50	0.563, 0.563 0.568, 0.573 0.574	0.568	5	2.26×10^{-3}	0.42	0.51	0.89	0.64
60	0.560, 0.565 0.570, 0.574 0.565	0.567	5	5.36×10^{-3}	0.41	0.46	0.88	0.57
(c)	0.625, 0.630 0.640, 0.645 0.645	0.637	5	9.08×10^{-3}	0.47	0.75	0.99	0.93

(a) Soluble leucoanthocyanidin tannin calculated as g (+) catechin/100g dates (dry weight).

(b) Soluble leucoanthocyanidin tannin calculated as g tannic acid/100g dates (dry weight).

(c) The samples were stored in the freezer (-13°C) for 2 months, packed in LDPE bags.

Table 36 The effect of microwave heating on the changes in insoluble leucoanthocyanidin tannin in Zahdi dates (green stage - whole dates).

microwave heating time (seconds)	O.D at 550 nm 1 mm cell range	O.D at 550 nm 1 mm cell mean	number of replicates	standard deviation	wt. of tissue residue after drying	molar extinction coefficient	insoluble tannin[a]/100g dates
0	0.250, 0.240 0.270, 0.300 0.280	0.268	5	0.024	6.182	29270	0.70
(b)	0.305, 0.295 0.295, 0.278 0.270	0.289	5	0.014	6.583	29270	0.79
20	0.230, 0.230 0.232, 0.260 0.245	0.239	5	0.013	7.032	29270	0.68
30	0.350, 0.360 0.362, 0.364 0.375	0.362	5	8.95×10^{-3}	5.672	29270	0.76
40	0.398, 0.395 0.410, 0.410 0.420	0.407	5	0.010	5.842	29270	0.81
50	0.425, 0.435 0.440, 0.445 0.460	0.441	5	0.013	6.206	29270	0.84
60	0.485, 0.485 0.480, 0.470 0.430	0.470	5	0.023	6.984	29270	0.92
(b)	0.290, 0.260 0.275, 0.302 0.265	0.278	5	0.017	7.008	29270	0.79

(a) Insoluble leucoanthocyanidin tannin calculated as g cyanidin chloride/100g dates (dry weight).

(b) The samples were stored in the freezer (-13°C) for 2 months, packed in LDPE bags.

Table 37 The effect of microwave heating on the total phenolic compounds in Zahdi dates (green stage - whole dates).

microwave heating time (seconds)	% soluble tannin (a)	(b)	% soluble phenolic compounds (a)	(b)	% insoluble tannin (c)	% total phenolic compounds (a^1)	(b^1)
0	0.72	0.89	0.84	1.01	0.70	1.54	1.71
(E)	0.61	0.77	0.69	0.84	0.79	1.48	1.63
20	0.68	0.84	0.85	1.05	0.68	1.53	1.73
30	0.62	0.77	0.74	0.92	0.76	1.50	1.68
40	0.57	0.70	0.71	0.89	0.81	1.52	1.70
50	0.51	0.64	0.66	0.82	0.84	1.50	1.66
60	0.46	0.57	0.62	0.78	0.92	1.54	1.70
(E)	0.75	0.93	0.75	0.94	0.79	1.54	1.73

(a) Calculated as g (+) catechin/100g dates (dry weight).

(b) Calculated as g tannic acid/100g dates (dry weight).

(c) Calculated as g cyanidin chloride/100g dates (dry weight).

(a^1) Total phenolic compounds calculated as (a + c).

(b^1) Total phenolic compounds calculated as (b + c).

(E) The samples were stored in the freezer ($-13^{o}C$) for 2 months, packed in LDPE bags.

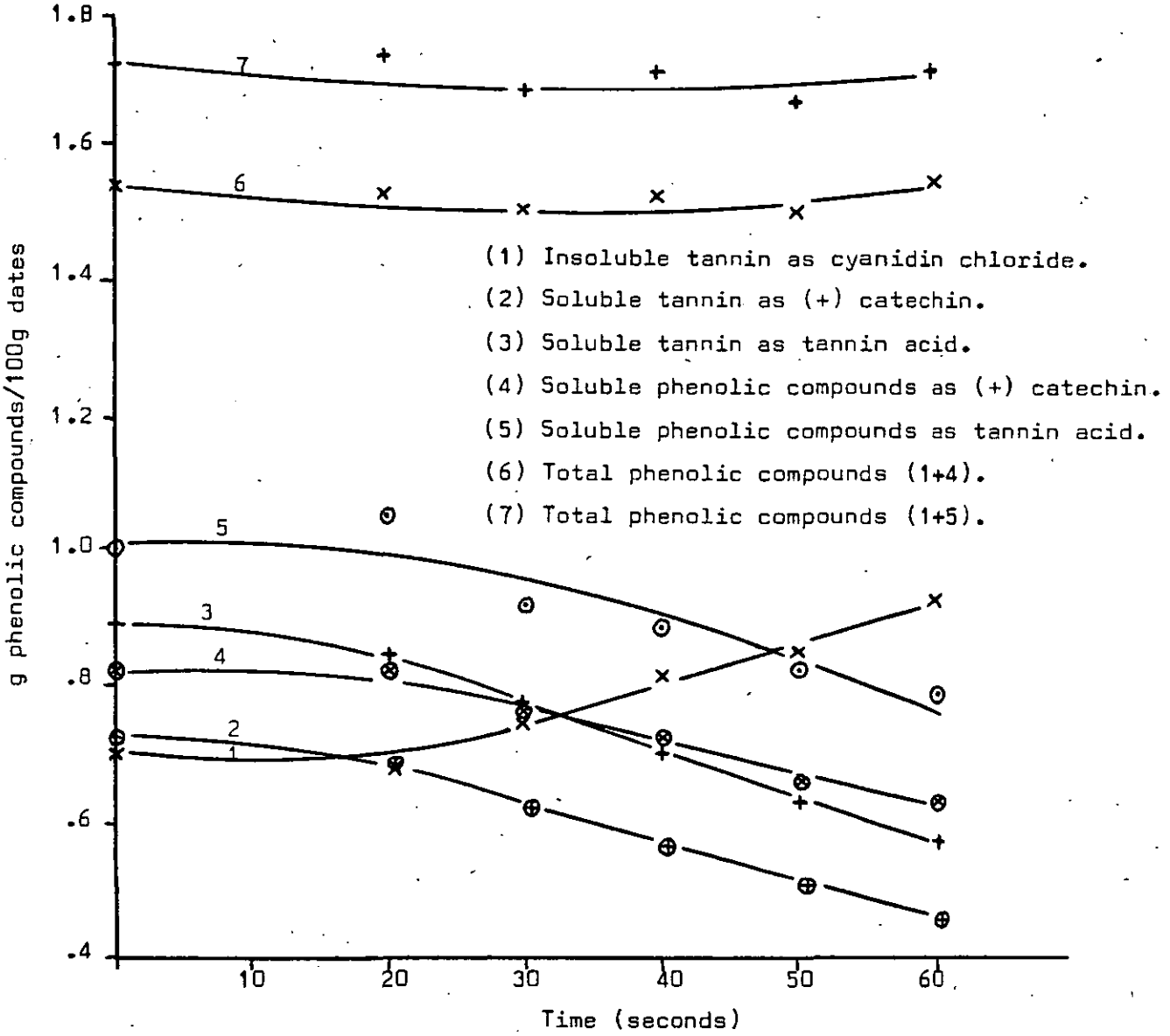

Figure 17 Effect of duration of treatment by microwave on the phenolic compounds in Zahdi dates (green stage).

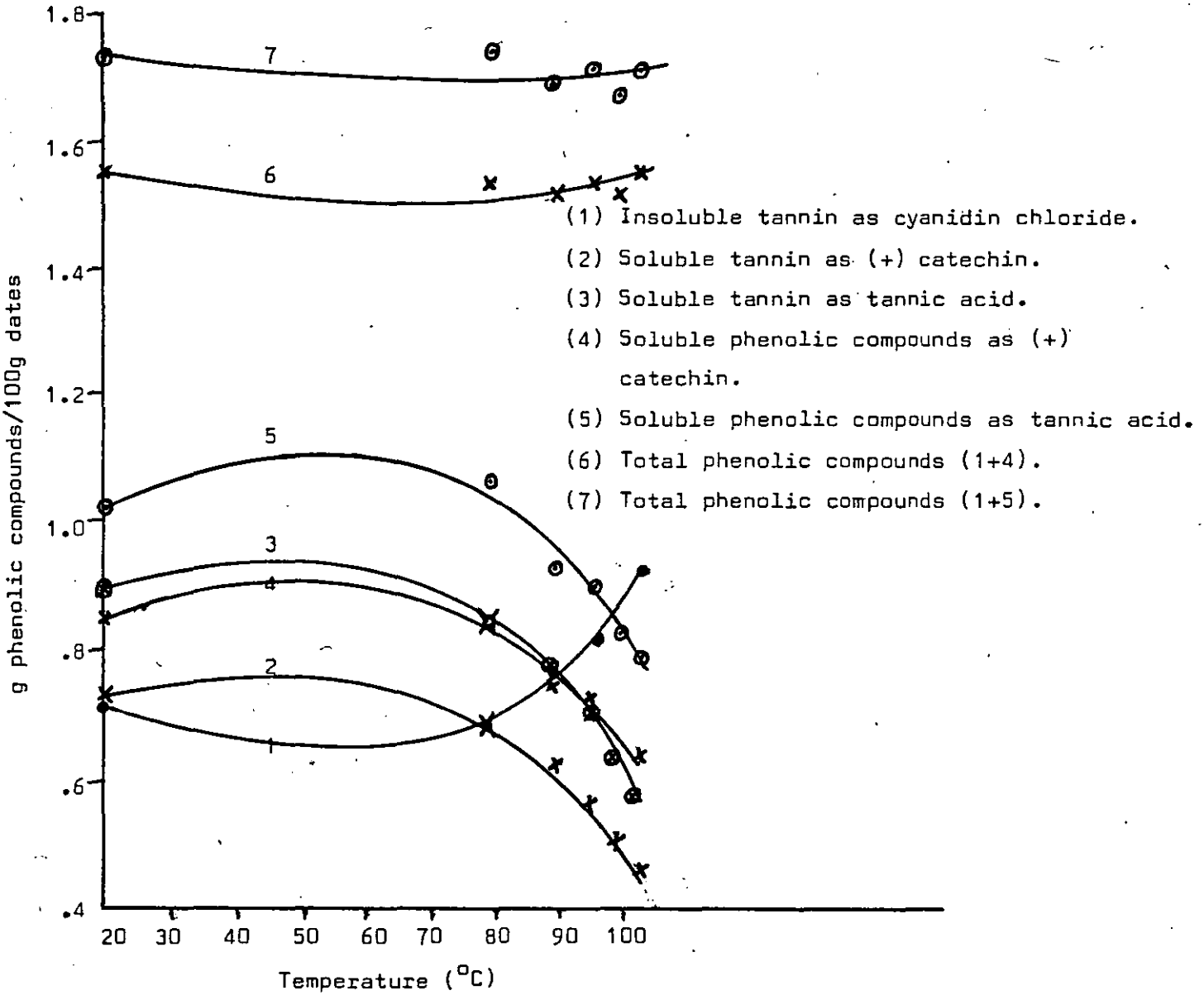

Figure 18 Effect of temperature achieved during microwave treatment on the phenolic compounds in Zahdi dates (green stage).

Table 38 The relationship between time and temperature in the inactivation of polyphenolase and peroxidase[a] in Zahdi dates (green stage - whole dates)

microwave heating time (seconds)	temperature inside dates before and after microwave heating (°C)
0	19.1
10	52.3
20	78.4
30	88.0
40	94.2
50	97.9
60	100.1

(a) The presence of polyphenolase was detected by flooding the dates with catechol solution (used as a substrate). Polyphenolase reacted with catechol and produced a dark colour immediately when the enzyme was still active in dates. Peroxidase was detected by flooding the dates with 1% freshly prepared guaiacol solution mixed with an equal volume of hydrogen peroxide (5 volume). Peroxidase reacted and produced a reddish-brown colour when still active.

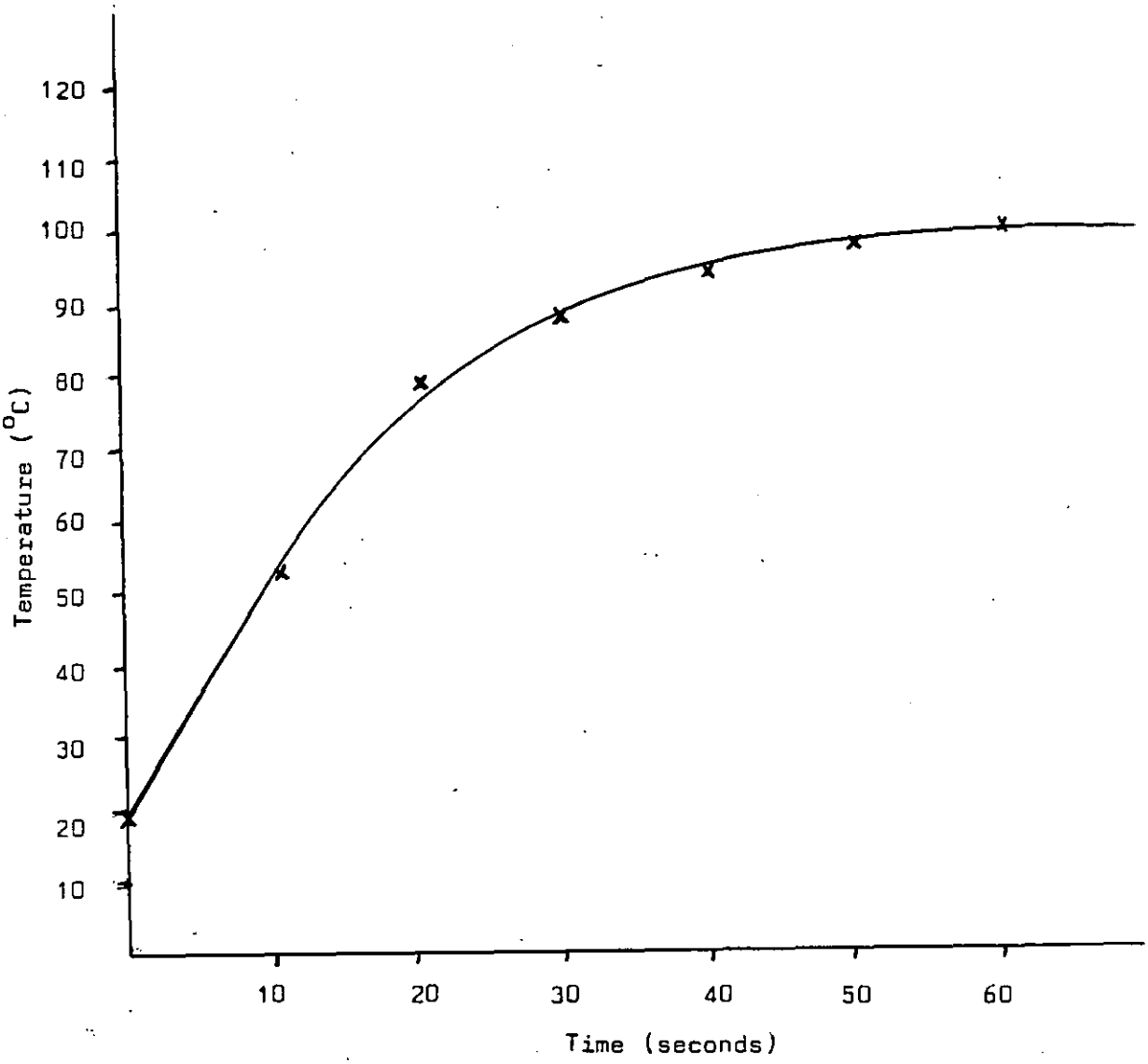

Figure 19 The relationship between time and temperature in inactivation of polyphenolase and peroxidase in Zahdi dates (green stage) by using microwave heating.

3.7 Determination of sugars in dates by periodate and thiobarbituric acid reagents

Table 39 Determination of the amount of invert sugar, sucrose and total sugar in Zahdi and Khadrawi dates (fully ripe) by Lane & Eynon and thiobarbituric acid methods.

		Lane & Eynon method					thiobarbituric acid method								
cultivar	variety of dates	reading titre (cm^3) mean (a)		% invert sugar	% sucrose	% total sugar	O.D at 532 nm range		O.D at 532 nm mean		μmole glucose/$1cm^3$ solution		% invert sugar	% sucrose	% total sugar
		B	A				B	A	B	A	B	A			
1979	Zahdi	20.3	18.9	71.5	4.60	76.1	0.310 0.320 0.314	0.345 0.350 0.370	0.315	0.355	2.7	3.1	70.9	9.98	80.9
1979	Khadrawi	17.6	17.3	86.2	1.79	87.9	0.355 0.350 0.343	0.365 0.370 0.355	0.349	0.363	3.0	3.1	82.8	2.61	85.4

(a) Mean of 3 readings. Duplicate samples were used for this investigation.

(B) Before inversion.

(A) After inversion.

Sugar calculated as g D-glucose/100g dates (dry weight) as follows:-

$$\text{g sugar/100g dates (wet weight)} = \frac{W \times M \times D}{10^6} \times 10$$

W = weight of D-glucose (μmole) can be obtained from the calibration curve according to the optical density.

M = molecular weight of D-glucose (180).

D = dilution factor $(50 \times \frac{100}{4} \times \frac{250}{25})$.

Sugars can be calculated as % dry weight in dates according to % moisture in dates.

3.8 Summary of Results

Effects of Variation in Storage Conditions at Room Temperature on the Chemical Composition of Dates

The following results were obtained:-

1. No significant changes in the moisture content.
2. Slight decrease in the pH, slight increase in the titratable acidity.
3. Slight decrease in the total soluble solids.
4. Increase in the colour intensity.
5. Decrease in the total soluble phenolic compounds.
6. Significant decrease in insoluble leucoanthocyanidin tannin in the samples stored in the presence of oxygen but a smaller decrease in the samples stored in the absence of oxygen.

Effects of Freezing and Storage on the Chemical Composition of Dates

The following results were obtained:-

1. No significant changes in the moisture content during storage.
2. Significant changes in the pH, significant changes in the titratable acidity.
3. Increase in the total soluble solids.

4. Significant decrease in the total soluble phenolic compounds.
5. Increase in the colour intensity.
6. Increase in the amount of insoluble leucoanthocyanidin tannin.
7. Disappearance of the astringent taste.
8. Some damage in texture at the end of storage.
9. Darkening of dates occurred rapidly during and after thawing.

Effects of Blanching and Storage on the Chemical Composition of Dates

The following results were obtained:-

1. Increase in the moisture content.
2. Slight fall in pH, slight increase in the titratable acidity.
3. Decrease in the total soluble solids (sugars).
4. Decrease in the colour intensity.
5. Decrease in the total soluble phenolic compounds.
6. Decrease in the amount of insoluble leucoanthocyanidin tannin.
7. Polyphenolase inactivated for 4 minutes in boiling water.

8. Peroxidase inactivated for 3 minutes in boiling water.
9. Soft texture due to the high moisture content.
10. Dull colour, shrinkage and cooked flavour.

Effects of Microwave Heating on the Chemical Composition of Dates

The following results were obtained:-

1. Slight decrease in the moisture in the tamar stage and significant decrease in the moisture in the green stage.
2. No significant changes in the pH, no significant changes in the titratable acidity.
3. Slight decrease in the total soluble solids.
4. Increase in the colour intensity, improved colour of dates.
5. Significant decrease in the total soluble phenolic compounds in treated dates which the enzyme was not inactivated in the tamar stage.
6. No significant changes in the total soluble phenolic compounds between the untreated dates and treated dates which the enzyme was completely inactivated.
7. Significant decrease in the total soluble phenolic compounds in the green dates.

8. Significant decrease in the soluble leucoanthocyanidin tannin in the green dates.
9. Significant increase in the insoluble leucoanthocyanidin tannin in the green dates.
10. Significant decrease in the insoluble leucoanthocyanidin tannin in the fully ripe dates.
11. The polyphenolase was completely inactivated for 1 minute in Zahdi dates (green and tamar stages).
12. The peroxidase was completely inactivated at less than 1 minute in the tamar stage. This period was not enough to inactivate the enzyme in the green dates.
13. No shrinkage, good flavour and taste in the treated dates.

4. DISCUSSION

4.1 Effects of Variation in Storage at Room Temperature on the Chemical Composition of Dates

As the dates were efficiently packed in low density poly-ethylene bags during storage, there were obviously no significant changes in their moisture content. However there was a slight increase in acidity and a corresponding fall in pH. This change in acidity was more marked in samples which had been flushed with oxygen. Oxidation of insoluble leucoanthocyanidin tannin and their subsequent break down rather than non-enzymatic, non-oxidative reactions might have been the cause of the pH change.[9,39] (See table 7).

Similar total soluble solids of the dates decreased slightly during storage. This was probably due to non-enzymatic, non-oxidative browning reactions between sugars and amino acids.[38] Evacuated samples showed greater resistance to this change than other samples.

Changes in colour of dates were more noticeable in samples stored in air and excess oxygen. This change of colour was usually a darkening of the red/brown colour and was probably caused by oxidation of insoluble leucoanthocyanidin tannin. It was found that as the brown colour became darker the

corresponding amount of insoluble leucoanthocyanidin tannin decreased in the date samples. (See tables 7 and 9).

In a similar manner the total soluble phenolic compounds decreased when the dates were stored in the presence of oxygen. The soluble phenolic compounds are substrates for polyphenolase in enzymatic browning reactions. Therefore, any action of polyphenolase will cause a reduction in the concentration of soluble phenolic compounds in producing brown pigment. Any reduction in moisture content during the storage of dates will slow down the reactions.[7] (See table 8).

A large decrease occurred in the insoluble leucoanthocyanidin tannin content of the dates particularly those stored in the bags flushed with oxygen. This decrease was most likely related to the oxidation of the insoluble leucoanthocyanidin tannin.[28] The dark colour of Khadrawi dates during storage could be related to considerable oxidation of insoluble leucoanthocyanidin tannin, whereas, with Zahdi dates, which are red-brown in colour, less oxidation had obviously occurred. The insoluble leucoanthocyanidin tannin in Zahdi dates appears to be less susceptible to oxidation than in Khadrawi dates, probably because of the presence of natural anti-oxidants. (See table 9).

4.2 Effects of Freezing and Storage at -13°C on the Chemical Composition of Dates

During storage of frozen dates at -13°C the pH rise slowly to a peak at pH 6.5 after 104 days of storage. The pH then decreased as darkening caused by polyphenolase action in the dates (see table 10).

A high solids content is responsible for the good keeping quality of mature dates.[28] The storage temperature of -13°C greatly retarded any non-enzymatic, non-oxidative browning reactions, and therefore, total soluble solids which are high throughout the frozen storage period, reaching a maximum after 148 days of storage. (See table 10 and figure 4).

Some darkening of dates did occur during frozen storage due to enzymatic browning. A lower storage temperature of -29°C could have probably retarded the reactions still further. On thawing, the dates darkened rapidly as the polyphenolase became active. The relatively slow freezing process had probably caused the cell to break down, this releasing the polyphenolase which caused rapid darkening on thawing the dates (see table 10 and figure 6).

The total soluble phenolic compounds decreased markedly during storage of dates at -13°C. This decrease could probably

be attributed to polyphenolase activity[21] in the enzymatic browning reactions. Any off-flavours produced could also have been caused by the polyphenolase (See table 11 and figure 5).

The amount of insoluble leucoanthocyanidin tannin increased during storage of dates at -13°C. This increase could have been connected to the disappearance of the astringent taste of soluble tannin in this kind of date.[36] Soluble tannin was probably converted into insoluble tannin during frozen stage. The disappearance of the astringent taste from dates is indicative of good quality. The changes in insoluble leucoanthocyanidin tannin during storage of dates at -13°C can be seen in table 12 and figure 7. The cause of darkening of frozen dates during storage, is difficult to attribute to the simple oxidation of insoluble leucoanthocyanidin tannin, as this compound actually increased during storage. However, the darkening of dates could be attributed probably to enzyme activity as the amount of soluble phenolic compounds decreased. The evidence that insoluble leucoanthocyanidin tannin might not have been oxidized during storage at low temperature (-13°C) is shown in table 13. The rate of oxidation of insoluble leucoanthocyanidin tannin would be slow at low temperatures (-13°C).

4.3 Effects of Blanching and Storage on the Chemical Composition of Dates

Blanching by immersion of dates in boiling water obviously caused an increase in moisture content of dates. High moisture content of dates leads to rapid spoilage[7] (see table 14).

The pH decreased slightly during blanching and subsequent storage. This decrease in pH could have been due to the break down of insoluble leucoanthocyanidin tannin by heat and non-enzymatic, non-oxidative browning reactions.[9,39] (See table 14).

The total soluble solids decreased during blanching. Sugars from the dates were leached out into the blanching water. The loss in total soluble solids as seen in table 14, was obviously higher in dates blanched for longer periods (see table 17). The decrease in sucrose could also have been due to the inversion of sucrose to invert sugar. This inversion was perhaps related to the heat treatment, and the results showed that sucrose was nearly or completely converted into invert sugar.

The colour intensity decreased proportionally to the time of blanching. This probably also could have been

related to the leaching of the pigments into the blanching water. (See table 14).

Soluble phenolic compounds decreased during blanching, similarly it was probably due to the leaching of soluble phenolic compounds into the blanching water. Soluble phenolic compounds could be measured in the blanching water after the treatment and a high amount of soluble phenolic compounds was found. (See table 15).

The amount of insoluble leucoanthocyanidin tannin decreased in all samples during blanching and storage. The decrease may have been related to the leakage of insoluble leucoanthocyanidin tannin into the blanching water. This was investigated by treating the blanching water after the blanching with insoluble leucoanthocyanidin tannin reagent. The slightly astringent taste in the blanched dates could be attributed to the breakdown of insoluble leucoanthocyanidin tannin at high temperature.[9] (See table 16).

Effect of blanching on polyphenolase activity

The results show that the polyphenolase was completely inactivated by blanching dates in boiling water for at least 4 minutes. The changes in total soluble phenolic compounds in blanched dates, when the polyphenolase was still active,

could have been related to the high moisture content which permitted enzyme activity during storage. With low moisture content no significant changes in total soluble phenolic compounds during storage of dates (tamar stage - fully ripe) were noticed. The extent of darkening of dates which could be caused by direct enzymatic browning was probably limited by the availability of substrate.[28] In dates with a low moisture content the activity of the enzyme was inhibited by this factor. It has been reported that the polyphenolase was inactivated at 100°C for 1.5 minute.[21] However, the inactivation reported was carried out on extract of dates not directly on dates. The effect of time and temperature on polyphenolase activity can be seen in table 18 and figures 13, 14.

Effect of blanching on peroxidase activity

The results show that peroxidase was completely inactivated by blanching dates (tamar stage - fully ripe) in boiling water for 3 minutes. The peroxidase was probably not involved in the darkening of dates.[21] It has been reported that the destruction of peroxidase can be accomplished in about ten times the period required for polyphenolase inhibition.[21] Peroxidase has been inactivated at 100°C for

14 minutes.[21] But the inactivation procedure reported was carried out on the extract not directly on dates. The effect of time and temperature on peroxidase activity can be seen in table 19 and figures 15, 16.

4.4 Effects of Microwave Heating and Storage on the Chemical Composition of Dates

As expected the moisture content of dates was found to decrease after microwave heating. However, the decrease was slight in the tamar stage but considerable in the green stage, this reflecting the higher initial amount of moisture in the green stage which exist as free water. (See tables 20, 28 and 33). The moisture content of dates continued to decrease during storage at room temperature (see table 21). Green dates could not be stored at room temperature because at this temperature the dates become dry and shrunk.

There were no significant changes in pH following microwave treatment of the dates. When the temperature increased to more than 90°C, there was a slight decrease in pH. (See tables 20, 28 and 33). The decrease in pH might have been due to the coagulation of protein which might serve as a buffer, or the breakdown of insoluble leucoanthocyanidin tannin might also cause a change in pH.

The total soluble solids slightly decreased as the temperature increased. This decrease might have been due to the non-enzymatic, non-oxidative browning reactions between sugars and amino acids.[38] This decrease can be seen in tables 20 and 28. There was an increase in total soluble solids during storage of dates as in table 21 which might have been due to the breakdown of pectic substances and fibres during sotrage. The increase in total soluble solids in the green dates may have been due to the breakdown of pectic substances and cellulose during microwave heating. (see table 33). The results given in table 21 show that the colour intensity increased as the temperature increased during the treatment. This could have been due to the non-enzymatic, non-oxidative browning of sugar[38] and non-enzymatic oxidative browning of insoluble leucoanthocyanidin tannin. The increase in the colour intensity in the green dates as in table 33 could have been due to the breakdown of chlorophyll during microwave heating treatment.

The results show that the total soluble phenolic compounds decreased as the temperature increased and reached a minimum at a particular temperature depending on the

variety of dates (see tables 22, 23 and 24). Then the amount of phenolic compounds increased as the temperature increased and reached a maximum. The amount of soluble phenolic compounds, however, continued to decrease during storage in the samples in which the polyphenolase was not completely inactivated, while there was no significant changes in the amount of total soluble phenolic compounds in the samples which the polyphenolase was completely inactivated. The changes in soluble phenolic compounds were probably due to the activity of polyphenolase during the microwave treatment as well as during storage. When the green dates were treated by microwave heating, the amount of soluble phenolic compounds decreased as the temperature increased. The decrease could have been due to the changes in soluble leucoanthocyanidin tannin. The results in table 34 show that the treated dates for 1 minute had shown no significant changes in the total phenolic compounds between treated and untreated dates. This could have been due to the loss of polyphenolase activity in the treated dates. The amount of soluble phenolic compounds increased in the dates stored at $-13^{o}C$ after the treatment by microwave heating for 1 minute. This could have been due to the reversible conversion of tannin.

protein + tannin $\longrightarrow$ soluble tannin $\rightleftharpoons$ insoluble tannin.

The decrease in total soluble phenolic compounds in the untreated dates stored for 2 months at -13°C could have been due to the conversion of soluble tannin into insoluble tannin. The increase in the colour intensity in the untreated dates and the insignificant changes in the colour of treated dates, could explain the reason why the untreated dates became darker whereas there was no change in colour in the treated dates. This colour change was related to the oxidation of phenolic compounds by the polyphenolase. (See table 34 and figures 17 and 18).

The soluble tannin was found to decrease as the temperature increased. This decrease could be related to the conversion of soluble tannin into insoluble tannin. The amount of soluble tannin, however, increased in the samples stored for 2 months at -13°C after treatment by microwave heating for 1 minute as in table 35. This increase in soluble tannin might have been due to the reversible conversion of tannin, as follows:-

protein + tannin $\longrightarrow$ soluble tannin $\rightleftharpoons$ insoluble tannin.

When the protein was denaturated by microwave heating, the soluble tannin was separated and converted to insoluble tannin. The insolubility of tannin could be due to its large molecular size or to interaction with other insoluble tissue fractions such as cellulose, pectin, hemicellulose or protein.[29] The

slight decrease in the total soluble phenolic compounds in the fully ripe dates was more likely caused by the activity of the polyphenolase rather than by the conversion of soluble tannin into insoluble tannin. The Folin-Denis method gave a high value in measuring the amount of soluble tannin in the ripe and fully ripe dates. This high value was probably due to the presence of new compounds in the ripe and stored dates and the absence of these compounds in the green dates.[29] The decrease in soluble tannin can be seen in table 35 and figures 17 and 18. The conversion of soluble tannin into insoluble tannin was more likely than the use of soluble tannin as a substrate for the polyphenolase.

The amount of insoluble leucoanthocyanidin tannin decreased as a result of the microwave heating. This decrease was probably due to breakdown of insoluble leucoanthocyanidin tannin during the treatment. The amount of insoluble leucoanthocyanidin tannin continued to decrease during storage at room temperature. This decrease was most likely related to the oxidation of insoluble leucoanthocyanidin tannin. The decrease in insoluble leucoanthocyanidin tannin in Khadrawi dates as the results show in table 30 and figures 10 and 11 was greater than in Zahdi dates (table 24 and figures 10 and 11). Therefore it appeared that insoluble leucoanthocyanidin tannin

of Khadrawi dates was more susceptible to breakdown than in Zahdi dates. This might explain the reason why Khadrawi dates become dark after ripening and during storage and Zahdi dates become a reddish-brown colour. This could be noticed in the tissue residue after extraction and drying. The insoluble leucoanthocyanidin tannin tended to breakdown during storage in all treatments. This could have been due to the oxidation of insoluble tannin by oxygen. (See table 25). These results might explain the reason why the insoluble leucoanthocyanidin tannin appeared to be more susceptible to oxidation than to acting as a substrate for the polyphenolases. The results in table 36 show that the amount of insoluble tannin increased during the microwave treatment as the temperature increased. This increase was most likely related to the conversion of soluble tannin into insoluble tannin. No significant oxidation of insoluble leucoanthocyanidin tannin in the green dates was noticed. It would appear therefore, that insoluble leucoanthocyanidin tannin was more susceptible to oxidation in the fully ripe dates and during storage than in the green dates. In the stored dates at $-13^{o}C$ for 2 months, after the treatment by microwaves for 1 minute, the amount of insoluble leucoanthocyanidin tannin decreased. This decrease

might explain that the conversion of soluble tannin into insoluble tannin at low temperature was reversible. The same treatment with untreated dates, indicated that soluble tannin was converted into insoluble tannin because the amount of insoluble tannin increased during storage, while the amount of soluble tannin decreased during storage.

Effect of microwave heating on polyphenolase activity

The increase in temperature during microwave treatment caused an increase in the velocity of reaction between the enzyme and the substrate.[49] This can be seen in the changes in soluble phenolic compounds given in tables 21, 29 and 34. The results show that when the temperature increased further the enzyme was inactivated. The thermal destruction of polyphenolase by microwave heating can be seen in figure 14. As with other enzymes the thermal inactivation of polyphenolase, is a function of both temperature and the destruction of the heat treatment. The relationship between time and temperature in inactivation of polyphenolase in dates can be seen in figures 12 and 19. The polyphenolase of Zahdi dates in both stages (green and tamar) was completely inactivated by microwave heating for 1 minute. Polyphenolase has been reported to have

been inactivated by blanching in hot water at 100°C for 1.5 minute.[21] The polyphenolase test was carried out with green dates by using catechol as a substrate. A dark colour was produced if polyphenolase was still active. To make sure the polyphenolase was inactivated, samples of green dates in which the enzyme was very active were stored at -13°C for 2 months, after half samples were treated with microwave heating for 1 minute. It was found that the untreated dates were darker in colour, but there was no change in the colour of the treated dates. The untreated dates gave darker colour with the polyphenolase test, while the treated dates gave no change in the colour. (See table 33). However, the results in table 31 show that polyphenolase was not completely inactivated for 1 minute in Khadrawi dates. This obviously indicated greater polyphenolase activity in Khadrawi dates than in Zahdi dates. Hence, this could explain the darker colour in Khadrawi dates and reddish-brown colour in Zahdi dates in the fully ripe dates. According to the results the activity of polyphenolase increased during storage. This agrees with the results of other workers.[47]

Effect of microwave heating on peroxidase activity

The activity of peroxidase decreased with time of microwave heating of Zahdi and Khadrawi dates (tamar stage) and the activity was completely lost in less than 1 minute. This is shown in tables 27 and 32 and graphically in figures 15 and 16. The results of the peroxidase test are shown in the photographs (figures 8 and 9) for Zahdi and Khadrawi dates (tamar stage). In both sets of photographs, samples 1 are untreated (no microwave heating and no peroxidase test), whereas all the other samples have been subjected to microwave heating for the given periods of time followed by addition of the peroxidase test reagents (1% guaiacol mixed with an equal volume of "5 volume" hydrogen peroxide). Peroxidase activity is indicated by a dark brown colour. It can be seen that the peroxidase activity decreases as the microwave heating time increases.

Peroxidase apparently is less active than polyphenolase during storage or after ripening of dates.[47] Peroxidase apparently, therefore, has no effect on the darkening of dates.[21] Inactivation of polyphenolase is important to prevent excess darkening but complete inactivation of peroxidase may not be essential.

4.5 Determination of Sugars in Dates by Periodate and Thiobarbituric acid Reagents

The results in table 39 show that the amount in dates of

invert sugar and total sugar after inversion of sucrose, could be determined by using a sensitive colourimetric method, e.g. periodate and thiobarbituric acid reagents. This method was applied to standard solutions of glucose, fructose, galactose, sucrose, lactose, starch and pectin. All the sugars mentioned here gave a pink colour with the reagents at a wavelength 532 nm but sucrose showed a lower optical density. So, sucrose should be inverted before determination by this method as in the Lane and Eynon method. The time, pH and temperature were studied to modify the method in order to make it more suitable for sugar measurement in dates. The thiobarbituric acid and periodate method could be used in dates in all stages of ripening without interference from for example quinic acid, which is not found in dates.[29] The method failed to give pink colour with shikimic acid, a part of caffeoylshikimic acid, a common phenolic substrate in dates. Different concentration of shikimic acid were used for this investigation. The results obtained by this method can be compared with Lane and Eynon method for reducing sugar, sucrose and total sugar in dates. Tables 17 and 39 summarise the results obtained in the different date samples.

5. CONCLUSIONS

Factors which contribute to the lowering of quality of dates include darkening, off-flavour and high acidity. This work has established a number of factors which contribute to the maintenance of high quality in dates.

Storing dates at low temperatures and particularly in a frozen state, has been shown to limit darkening and the development of high acidity. Frozen dates ($-13^{o}C$) were shown to ripen slowly during frozen storage, thus making dates available throughout the year. Gradual ripening under frozen conditions is also useful in the prevention of infestation by insects and variations caused by climatic changes in the normal dates ripening process. During frozen storage the pH remained at an acceptable level. The total soluble solids (mainly sugars) increased and the total soluble phenolic compounds decreased during storage of frozen dates. The insoluble leucoanthocyanidin tannin increased during the frozen storage period. However, enzymatic browning of polyphenolics occurred during frozen storage ($-13^{o}C$) and was the main cause of colour darkening in the dates. It was established that non-enzymatic oxidative browning of tannin and non-enzymatic, non-oxidative browning of sugar were not responsible for colour change during frozen storage.

In contrast to this browning in frozen dates, it was established that non-enzymatic oxidative browning of tannin was probably the predominant means of browning in dates (fully ripe), stored at room temperature, and enzymatic browning of polyphenolics and non-enzymatic, non-oxidative browning of sugar had little effect.

The darkening of Khadrawi dates, however, was found to be caused probably by the non-enzymatic oxidation of insoluble leucoanthocyanidin tannin. This compound was found to be more susceptible to oxidation in this variety of dates than in the Zahdi variety. The insoluble leucoanthocyanidin tannin appeared generally to be more susceptible to oxidation than to acting as a substrate for polyphenolase. It was established that dates need to be packed in the absence of oxygen to avoid oxidation of insoluble leucoanthocyanidin tannin and consequent loss of quality.

The main enzymes system responsible for darkening in dates is the group of polyphenolases. These enzymes were found to be more active in Khadrawi dates than in Zahdi dates. Peroxidase appeared to have no effect on the darkening of dates. The darkening of dates could be controlled by inactivation of the polyphenolase.

Blanching dates in boiling water was found to lower the quality as it resulted in loss of sugar, dull colour due to

loss of pigments, shrinkage and a cooked flavour.

Microwave treatment of dates, however, was found to be efficient in the inactivation of polyphenolase and peroxidase. Only short periods of treatment were necessary compared with long periods of blanching in boiling water to inactivate the enzymes. The darkening of dates was reduced when the polyphenolases were completely inactivated by the microwave treatment. However, some browning did occur during the treatment and consequent storage caused by non-enzymatic oxidative browning of tannin and non-enzymatic, non-oxidative browning of sugar. The undesirable changes which occurred during blanching dates in boiling water did not occur in microwave treatment for short period. In fact, the treatment may improve colour, texture and flavour of dates.

The microwave treatment caused some loss in moisture, which facilitates longer storage and a decrease in insoluble leucoanthocyanidin tannin in fully ripe dates. In green dates, however, the treatment caused a significant loss in moisture, decrease in the total soluble phenolic compounds and an increase in the insoluble leucoanthocyanidin tannin proportional to the decrease in the soluble leucoanthocyanidin tannin.

Thus microwave heating in the correct dosage, appears to be a very useful method in controlling deteriorative changes

in dates during subsequent storage. A high quality product of good colour, flavour and keeping quality is produced by this treatment. It would appear, therefore, that this method of treatment of dates is worth further investigation and application on an industrial scale. Although initial cost of plant could be high this could be justified by the production of a high quality product, which after cold storage would be available throughout the year.

Determination of sugars in dates by a new, and relatively simple, method using periodate and thiobarbituric acid gave results that were in reasonable agreement with those obtained by the standard Lane-Eynon method.

6. FURTHER WORK

1. The method of microwave blanching of the dates requires further development and, in particular, application to dates in the khalal stage before freezing and thawing.

2. A more accurate method than the Folin-Denis method for the determination of soluble phenolic compounds in dates needs to be developed in order to avoid interference from other materials in the ripe and stored dates.

3. An investigation should be carried out on the periodate and thiobarbituric acid method of determining sugars in dates as a possible replacement for the Lane&Eynon method.

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