



STUDY ON THE FATTY ACID EXTRACTION FROM IRAQI DATE PALM (*Phoenix dactylifera* L.) ZAHDI VARIETY, SEED OIL AND INTRODUCTION OF FATTY ACIDS INTO CERTAIN TYPES OF THERAPEUTIC AND FOOD RATION

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ABSTRACT

This study was carried out in order to extract oil from date palm (*Phoenix dactylifera* L.).

zahdi variety, using Soxhlet extraction method. The content of the oil extracted from saturated and unsaturated fatty acids was examined and

diagnostic tests were run with GC/MS (Gas Chromatography / Mass Spectrometry).

Key words: dates, zahdi variety ,palm seeds, date seed oil, oil extraction, fatty acids, mayonnaise product.

INTRODUCTION

The date palm (or *Phoenix dactylifera* L.) is associated with Plantae vegetable reign, Magnoliophyta section, Monocotyledones class (Liliopsida according to Cronquist system), Palme order, Arecaceae family, Phoenix Date Palm generation, Dactylifera Date Palm type, and it is one of the oldest fruit trees in the world. This family incorporates nearly 220 generations and Palme (Palmes) order is one of the biggest and most important order of plants ever known to people^[3,1].

The palm tree cultivation is very common in tropical and subtropical regions and the date palm cultivation is historically connected with the Arab East. Iraq is considered to be ancestral homeland of the date palms, especially the region of Shatt al Arab river (formed by the merging of the Tigris and Euphrates rivers) and northern coast of the Persian Gulf, and from there it spread its distribution range to the regions with favorable and suitable conditions and climate^[3]. Date tree is one of the most significant evergreen fruit trees that are

associated with monocotyledon and dicotyledon plants.

Palm trees hold a prominent place in the human life, especially in hot and dry regions, because their fruit is a high-energy food source and also an industrial, commercial and agricultural resource.

The humanity has known the palm trees since 4000 B.C. Babylonians and Assyrians attributed much importance to it, these trees were sacred for Sumerians and are considered to be one of the core industries of economy for many inhabitants of the Middle East^[4]. Palm tree fruit contains high percentage of sugar (44-88%), fat (0.2-0.5%), protein (2.3-5.6%), 15 types of mineral salts and vitamins and a large amount of fibre (6.4-11.5%)^[4].

Iraq holds a prominent place in the dates production among countries which produce dates, in terms of the number of the palm trees and date varieties (which reach 600 varieties), with Zahdi variety as the leader which accounts for 70% of the total production of dates^[1]. In addition to the economic importance of the dates and their products, date seeds are used as feed, food and fuel^[3].

The growth and maturity of the fruit is observed at five different stages: Al Khababouk (set), Al Jamri (green phase), Al Khillal (splitting), Ar Rutab (wetting) and the stage of generation of the date bean itself, and depending on the color, freshness, humidity

and sugar content of more than 30.5% the dates are considered to be fully matured^[3].

Dates are the most important fruit cultures in Iraq and on the Arab Peninsula, and you may hardly find a house without the dates, they are a fruit, a medicine, a drink and sweets for the rich and the poor, being an exemplary and sufficient food product for the people. Dates rank fifth in the list of tropical and subtropical fruit after citrus, mango, banana and pineapple, and are also at the head of the dry fruit list, together with raisins, fig and plum^[3]. The fruit consists of two principal parts: the flesh, which is its edible portion and accounts for 85-87% of the fruit weight, and the core pit, which is its second part and accounts for 13-15% of mass of an entire date, yet the date seeds (pits) are not always used properly although the date seeds contain proteins, fats, fibre, mineral salts and carbohydrates in the range from 5-7.7 to 10-20, 1-2, 80-75% respectively.

The date seeds are also called pits, stones, kernels, seeds and are wastes in many branches of industry that are related to the processing of dates. A large number of seeds (pits) may be gathered from the dates production or from the process fragments and wastes.

The proteins of the dates contain many amino acids (clotamic acid, asparaginic acid and arginine which represent some amino acids in the dates). The dates also

contain other acids (isoleucine, lysine, leucine) and due to the fact that these components are present in the date seeds, dates may be introduced to the animal and human diets.

The date seed oil is used in many branches of the food industry, like mayonnaise production.

[3] and [5] mentioned the use of date seed (pits) oil in many areas, including coal preparation for jewelry production and use for fuels in conventional copper-smelting furnaces. Seeds (pits) are also used as animal feed after grinding or water steeping as a source of carbohydrates, fats and proteins. A part of poor population uses them as food, they boil them like bean cultures to benefit from their high content of main nutritional substances, as well as from the fibre which protects the body from symptoms of poor digestion, constipations and large intestine dysfunction.

The oil extracted from the pits accounting for 8% of human consumption and soap production was used as a medicine for the treatment of some kidney and urinary tract diseases after roasting and further grinding and boiling in water. The oil extracted from the date pits was also used as a remedy against rheumatism, uratic arthritis and joint pains. The grinded pits mixed up with rose water were used for eyes treatment and after softening were also used as an alternative to antimonine (eye tinting antimony).

[9] mentioned that the dates were also used in many branches of the industry forming the basis of the industrial sector in Iraq, for the production of molasses, alcohol and other products, which led to the accumulation of a large number of date seeds which is estimated to be about 18000 tons. This is a colossal rich resource which was only used for animal feeding.

[3] mentioned that date seeds (pits) represent a certain percentage of the fruit weight and it should not be neglected if it contains a significant part suitable for oil extraction.

Some part of Arab nomads use seeds (pits) after roasting and grinding to produce flour mass similar to coffee and those nomads are convinced that both products (date seed (pits) oil and roasted pits mass drink) are an alternative to coffee beans and consider it panacea against uratic arthritis and joint pains, because such oil is used as a treatment cream to massage painful areas, in addition to the use of pits powder solution as a drink alternative to coffee.

Poor communities living in the desert or isolated areas use the date pits (cores) as food. They leave them in water for two days and then they cook them in the water, adding salt and some spices. Due to the increased stimulation of date production it is expected that the accumulation of secondary wastes at the date production

plants will increase, which makes their disposal an environmental and economic problem. We may therefore summarize from the aforementioned that the studies of date seeds were rare and were concentrated on the chemical analysis, while some physical properties of those components did not refer to the oil content in the seeds or to their applicability as a low-cost and accessible resource.

OBJECTIVE OF THE STUDY

1. Use local Iraqi dates as a food source by extracting oil from the seeds of the following local Iraqi date types: zahdi variety.
2. Make quantitative determination and gain knowledge about types of short-chain fatty acids and saturated / unsaturated fatty acids using Gas Chromatography / Mass Spectrometry.
3. Introduce extracted oil (date pits oil) into food product systems, prepare a sample of the mayonnaise product.

MATERIALS AND METHODS

MATERIALS ZAHDI IRAQI DATES

It is considered a semi-dry date and a popular commercial variety; it occupies the third place in the southern region in terms of prevalence in the Shatt al-Arab City - Basra Province, but in the central region it occupies

the first place in terms of the number and quantity of crops, and this is due to the difference in growing from soils. Finished products are manufactured and packaged in boxes and bags, and partially in wooden boxes zahdi Iraqi dates are considered the most voluminous Iraqi variety in terms of production and the consumption of zahdi dates is divided into two parts: the first part is for direct consumption, and the second part is for the production of products based on different types of dates, including liquid sugar, molasses, alcohol, vinegar and yeast[9].

GRINDED DATE SEED AND SELECTED VARIETIES

Variety of the date fruit (Phoenix dactylifera L.) were selected for the study (zahdi variety) at the bean stage. zahdi date variety was collected from one of the gardens of (Al-Maraa city in the southern part of Missan Province). These varieties were selected from vigorous trees based on their maturity, freshness, uniformity and disease clearance, similar sizes, low cost and availability in the market. The seeds (pits) were cleaned and isolated manually, thoroughly washed with distilled water and were left for drying up for 10 hours at the temperature of 25 °C. The dried pits (seeds) were grinded in a Chinese grinding machine (High-Speed Grinder SIZE 750 G). To get the date seeds grinded powder, the contents were stored in sealed plastic bags at 4-5 °C prior to the start of their usage.

METHODS OF THE STUDY

OIL EXTRACTION

The oil was extracted from grinded date seeds (pits) variety of the date fruit (zahdi variety) by ether oil solvent with boiling temperature of 40-60 °C in Soxhlet unit, and according to the data provided in^[8], 10 grams of grinded dry date seeds were used (as a powder-like mass). The moisture was removed prior to the extraction process to maintain high efficiency of the solvent during the extraction and to avoid its mixing with the moisture. The components were then placed into a thimble and a defatted piece of absorbent cotton was placed above the sample inside the thimble and inside the retort, and the retort was filled with sufficient amount of solvent (250 ml).

The extraction lasts for 6 hours until the solvent in the retort becomes colorless and no yellow color is observed in the retort at the end of the extraction.

A measuring tube is taken and the solvent is boiled off, then it is dried in the air oven until the weight is stabilized. After boiling off of the solvent, the oil is collected into the dry glass containers and is preserved under cooling conditions. The oil obtained after such process is called Crude Oil and then it is handed over to the expert (oven man) to evaluate its properties.

$$\% \text{ of oil} = \frac{\text{Oil weight}}{\text{Sample weight (in grams)}} \times 100$$

DETERMINATION OF THE OIL CONTENT IN FATTY ACIDS

ESTERIFICATION OF FATTY ACIDS

Esterification of fatty acids of the date seed oil was carried out to run GS-MS using Kang and Wang method (2005), and an amount of 50 ml of extracted oil was taken and placed into a screw tube and 1 ml of regular hexane 1 was added. After that 1 ml of esterification solution was added to the tube and the solution was generated (14 g of BF₃ boron trifluoride in 100 ml of methanol). After thoroughly mixing the mixture with electrode at the maximum speed for 1 minute, it was placed into the hot oven at 90-100 °C for one hour. Then the solution was left to cool off until the room temperature. Then 1 ml of distilled water was added and stirred well, and two layers appeared in the solution. Fatty esterified acids were extracted from the dissolved organic layer, dissolved hexane, and placed into the tube after reaching concentration of 2 ml in to run GS-MS by taking 1 ml.

SEPARATION AND DIAGNOSTICS OF DATE SAAD OIL FATTY ACIDS BY GAS CHROMATOGRAPHY / MASS SPECTROSCOPY

Date seed oil from zahdi variety

was analysed to determine types of fatty acids and evaluate the percentage of the peak area of each acid and its storage time in GS-MS using Gas Chromatography Mass Spectroscopy (GS-MS), type Shimadzu-Japan QP2010 Ultra connected with the computer and containing Library NIST08-LIB with separation column Column DB-1 ms, length 30 m, internal diameter 0.25mm, fixed phase layer thickness 0.25 mm, with helium as the carrier gas with the flow rate of 1ml / min and injection temp of 280 °C. Column Oven Temperature Program was

adopted, with initial temperature of 50°C for one minute and further increase to 150 °C during one minute, with gradual and slow increase each minute by 4 °C, until it finally reaches 280 °C. The mass spectrometer was used in the Quadrupole Ions analyzer at the Ion source temperature of 200 °C and eV70.

MAYONNAISE PRODUCTION

The Mayonnaise was prepared according to^[7]. And an explanation is provided in Table 3.4 with regard to the percentage ratio of different ingredients used for mayonnaise production.

Table 1- Percentage ratio of ingredients used for mayonnaise production

Quantity (in grams)	Components (ingredients)
66,66	Date seed oil
26,16	Whole egg
1,20	Edible salt
0,80	Sugar
0,33	Mustard flour
2,02	Vinegar
0,50	Pepper
2,33	Mustard oil

EXPERIMENTAL SECTION

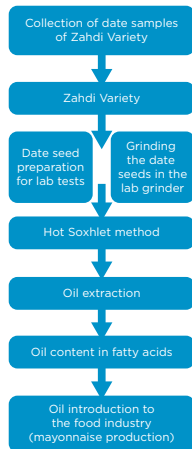


Picture 1 - Photo of the seeds preparation for grinding



Picture 2 - Photo of the laboratory grinder

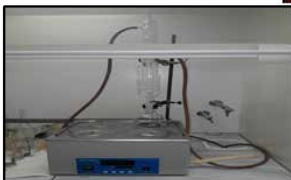
Figure 1- Summary plan for date seed grinding and oil extraction - sequential procedure consisting of different stages



Picture 3 - Photo of iraqi dates powder (powder of zahdi date seeds)



Picture 4 - Date seed oil extraction



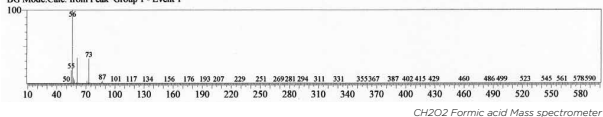
Picture 5 - Soxhlet extraction device



Picture 6 - Date seed oil

Figure 3 - Mass / charge spectra (m / e e) for fatty acids of zahdi seed oil

Line#:1 R.Time:3.133(Scan#:17) Retention Index:896! MasaPeaks:248
RawMode:Averaged 3.125-3.142(16-18) BasePeak:56.05(542818)
BG Mode:Calc. from Peak Group 1 - Event 1



Hit#:1 Entry:2255 Library:NIST08.LIB

SI:86 Formula:C5H10O2 CAS:592-84-7 MolWeight:102 RetIndex:783

CompName:Formic acid, butyl ester \$\$ n-Butyl formate \$\$ Butyl formate \$\$ HCOO(CH2)3CH3 \$\$ n-Butyl methanoate \$\$ Butyl methanoate \$\$ Butylester

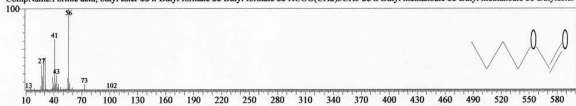


Figure 2 - Total - ion chromatogram for fatty acids in seed oil dates Zahdi

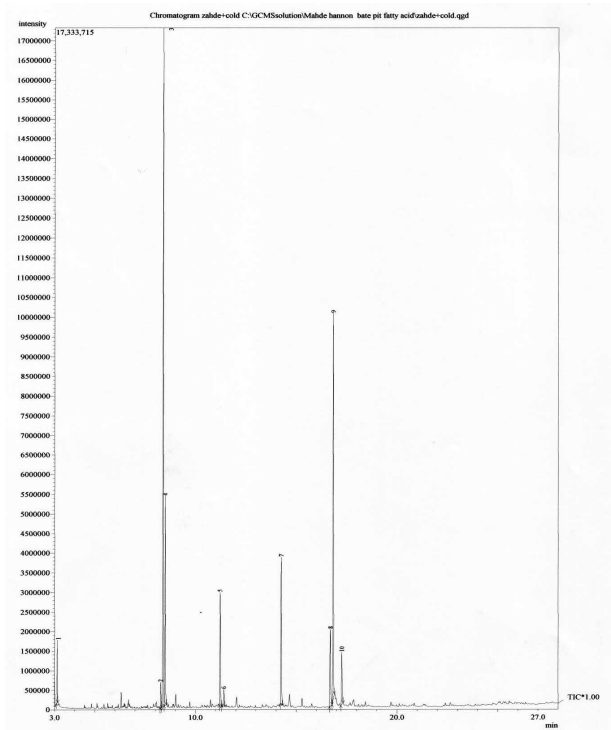
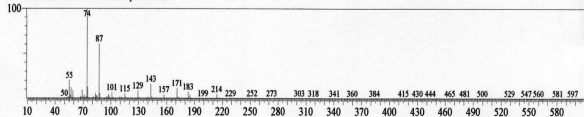


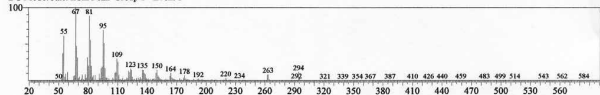
Figure 4 - Mass / charge spectra (m/e) for fatty acids of zahdi seed oil

Line# 4 R.Time:8.500(Scan#:661) Retention Index:1484 MassPeaks:337
RawMode:Averaged 8.492-8.508(660-662) BasePeak:74.00(1263590)
BG Mode:Calc. from Peak Group 1 - Event 1



C12H24O2 acid Lauric Mass spectrometer

Line# 8 R.Time:16.692(Scan#:1644) Retention Index:2354 MassPeaks:390
RawMode:Averaged 16.683-16.700(1643-1645) BasePeak:67.05(152051)
BG Mode:Calc. from Peak Group 1 - Event 1



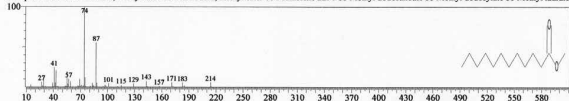
Library Lauric acid

Figure 5 - Mass / charge spectra (m/e) for fatty acids of zahdi seed oil

Hit# 1 Entry:32035 Library:NIST08.LIB

SI:95 Formula:C18H32O2 CAS:111-82-0 MolWeight:284 RetIndex:1481

CompName:Dodecanoic acid, methyl ester \$\$ Lauric acid, methyl ester \$\$ Metholene 2296 \$\$ Methyl dodecanoate \$\$ Methyl dodecylate \$\$ Methyl laurate

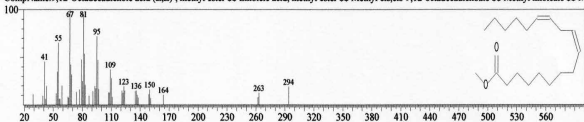


C18H32O2 acid Linoleic Mass spectrometer

Hit# 1 Entry:107904 Library:NIST08.LIB

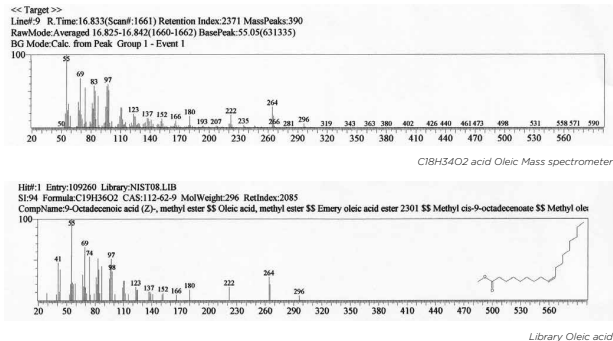
SI:96 Formula:C19H34O2 CAS:112-63-0 MolWeight:294 RetIndex:2093

CompName:9,12-Octadecadienoic acid (Z,Z), methyl ester \$\$ Linoleic acid, methyl ester \$\$ Methyl cis,cis-9,12-octadecadienoate \$\$ Methyl linoleate \$\$ M



Library Linoleic acid

Figure 6 - Mass / charge spectra (m / e e) for fatty acids of zahdi seed oil



RESULTS AND DISCUSSION

CONTENT OF FATTY ACIDS IN THE DATE SEED OIL

Forms 2 contain fatty acid chromatograms of date seed samples (Zahdi variety) obtained from Gas Chromatography/Mass Spectrometry (GC/MS). As for the forms 3,4,5 and 6, they illustrate mass-energy ranges for the analysis of fatty acids of date seed oil (from Zahdi variety). The specific diagnostics of separate fatty acids were performed through the integration of separate

maximum values and obtaining of relevant data like delay time, square, growth and percentage ratio of maximum values, with further Similarity Search using existing tools via software linked with the device, as shown in Tables 2, which explain the contents of date seed oil which was obtained (Zahdi variety) and which includes nine acids, of which seven are saturated acids (caprylic, capric, lauric, myristic, palmitic, stearic, arachidonic acids). As for the oleic and linolic fatty acids, these are unsaturated fatty acids which have significant importance. Oleic acid is represented as one of monounsaturated fatty acids called omega-9, which is

necessary for the body, yet it is not the most important one, because the body can generate it, too. Oleic acid has many features useful for health, it reduces the cholesterol level by decreasing the amount of low-density lipoproteins (LDL) and this type of protein contains 25% of protein and 45% of cholesterol and it distributes across different parts of the body and sometimes it deposits itself on the blood vessel walls. Although it does not affect the number of high-density lipoproteins (HDL), this type of protein contains about 50% of protein and 20% of cholesterol. HDL contributes to the removal of the excessive amounts of cholesterol from the body

(Alvarez & Rodriguez, 2000). Table 2 shows the content of fatty acids in the Zahdi variety date seed oil, and it was noted that it contains nice fatty acids, of which seven are saturated acids (caprylic 0.62%, capric 0.75%, lauric 31.62%, myristic

14.65%, palmitic 12.59%, stearic 3.93%, arachidonic 0.37%, respectively). It was noted that the content of lauric acid is higher than the content of other saturated fatty acids. As for the unsaturated fatty acids, the oleic and linolic

acids are one of the most important unsaturated fatty acids examined by Gas Chromatography/Mass Spectrometry (GC/MS), their percentage content in the oil was 46.12% and 6.96%, respectively.

Table 2 - Specific analysis of fatty acids contained in zahdi date variety seed oil, Using Gas Chromatography/Mass Spectrometry (GC/MS)

Peak	R.Time \ min	Area	Area%	Height	Height%	Scientific name	Trivial name
2	5.113	260244	0.29	0.29	0.62	Octanoic acid	Caprylic acid
3	6.462	325358	0.36	0.36	0.75	Docanoic acid	Capric acid
4	8.510	18019821	19.94	19.94	31.62	Dodecanoic acid	Lauric acid
5	11.241	11329457	12.54	12.54	14.65	Tetradecanoic acid	Myristic acid
6	14.269	10553973	11.68	11.68	12.59	Hexadecanoic acid	Palmitic acid
10	17.276	3073686	3.40	3.40	3.93	Octadecanoic acid	Stearic acid
12	19.712	264852	0.29	0.29	0.37	11-Eicosenoic acid	Arachidonic acid
Unsaturated acids with one conjugated double bond							
8	16.850	33823764	37.43	13973851	46.12	9-Octadecenoic acid	Linolic acid
Unsaturated acids with several conjugated double bonds							
7	16.702	5840953	6.46	2399073	6.96	9,12-Octadecadienoic acid	Linolenic acid

ORGANOLEPTIC EVALUATION OF THE MAYONNAISE PRODUCED FROM THE DATE SEED OIL OF THE STUDIED DATE VARIETIES

The mayonnaise was produced from the date seed oil of the studied date varieties (Zahdi variety) instead of other vegetable seeds (soybean oil used in the commercial mayonnaise is being currently studied), as well as from corn oil and sunflower oil. The mentioned percentages were incorporated into the mayonnaise preparation methods, also chemical starch was used instead of the natural one (as a bonding agent, as well as to correct textures and to prepare the filler or the filling substance)^[10]. When tests were carried out with regard to the commercial mayonnaise, the product was of better quality compared to the commercial trade mark mayonnaise in terms of its organoleptic properties (taste, flavor, color, texture, general acceptability). After running the tests, the product obtained good evaluation score from the tasting experts. Zahdi date seed oil gained the highest score. The use of nucleic acid in the mayonnaise production is caused by the high content of unsaturated fatty acids and nutritious substances, like tocopherols, which play an important role in heart problem risk mitigation. Date seed oil may be used as unconventional oil in several branches of the food industry, like mayonnaise production^[7].

CONCLUSION

1. The composition of the seed oil includes fatty acids: unsaturated fatty acids (oleic and linolic) and saturated fatty acids (caprylic, capric, lauric, myristic, palmitic, stearic, arachidonic).
2. Organoleptic tests of the mayonnaise produced with the date seed oil demonstrated significant advantage as compared to the commercial mayonnaise, especially in terms of general characteristics of public positive acceptability.
3. Use local Iraqi dates as a food source by extracting oil from the seeds of the following local Iraqi date types: zahdi variety.

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