

EXTRACTION OF DATE PALM SEED OIL (PHOENIX DACTYLIFERA) BY SOXHLET APPARATUS

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ABSTRACT

Oil extraction from date palm seeds (Iraqi date palm) is done by standard solvent extraction method using a Soxhlet apparatus. This work is aiming to investigate the extraction of palm seed oil as a cheap feedstock for producing bio-oil and determine the fatty acid composition of bio-oil. Parameters such as particle size, extraction time and type of solvent are optimized in order to enhance the yield of bio oil production. The bio-oil is characterized using Fourier Transform Infrared Spectroscopy (FT-IR) and Gas Chromatography Mass Spectrometry. Some of the basic fuel properties such as iodine value, saponification value, acid value, density, refractive index and kinematic viscosity are investigated to characterize fuel quality of the bio-oil. The extraction process was carried out on a laboratory scale with particle size 2mm, 1mm and 0.425mm for different time 1h, 2h, 4h and 6h. Particle size of 2 mm was chosen in order to study the effect of solvent type. The optimal conditions to obtain the highest oil yield of 8.5 % (w/w) were 120 min, 0.425 mm and n-hexane extracted time, particle size of grounded seed and type of solvent, respectively. The physical properties viscosity, density and refractive index were 29 CP, 0.925 g/cm² and 1.444 respectively.

KEYWORDS: Date seed, Phoenix dactylifera, Soxhlet extraction, Bio-oil, Fatty acids, GC/MS.

I. INTRODUCTION

The increasing demand for energy has led to exhaustion of the ordinary sources of fossil fuel [1]. In addition, the use of the ordinary sources of energy has led to many negative effects including climate change, receding of glaciers, rise in sea level, loss of biodiversity, etc. [2]. Increasing energy demand also leads to an increase in crude oil price, directly affected to global economic activity [3]. Progressive depletion of conventional fossil fuels with increasing energy consumption and GHG emissions have led to a move towards alternative, renewable, sustainable, efficient and cost-effective energy sources with lesser emissions [4,5]. Among many energy alternatives, biofuels, hydrogen, natural gas and syngas (synthesis gas) are likely to emerge as the four strategically important sustainable fuel sources in the foreseeable future. Within these four, biofuels are the most environment friendly energy source [6].

Previously, biofuel used to be produced from soybeans, canola oil, animal fat, palm oil, corn oil, waste cooking oil and jatropha oil [7], etc. However, attempts are now made towards biofuel production from non-food crops, microalgae [8, 9] (i.e. phytoplankton), macro algae (i.e. sea wood) etc. Recent studies indicate that the aromatic content and type, sulphur content, extraction temperature, and density are important factors for emission control [10].

Bio oil from date palm (Phoenix dactylifera) seeds is a promising new source for production of biofuel. The Phoenix dactylifera (date palm) is one of the member of the genus Phoenix, widely cultivated for its edible fruit. Dates have been a staple diet in the Middle East for thousands of years. They are believed to have originated around the Arabian Gulf, and have been cultivated since ancient

times from Mesopotamia to prehistoric Egypt, possibly as early as 4000 B.C. [11]. The world production of dates has increased considerably during the last 30 years. Indeed, the production has tripled from 2,289,511 tons in 1974 to 6,772,068 tons in 2004 [12]. According to FAO statistical data, 7.85 million tons of date fruits have been produced in 2010 [13]. The top ten countries producing date (Table 1) are Egypt, Iran, Saudi Arabia, United Arab Emirates (U.A.E), Pakistan, Algeria, Sudan, Iraq, Oman and Libya. They produce about 91% of the world's dates [12]. Date seeds constitute approximately 10% of the fruit and they are considered a waste by-product; date processing plants produce pitted dates, date powders, date syrup, date juice, chocolate-coated dates and date confectionery [14].

Table 1: World date production by region and selected countries (FAO statistics, 2010).

Country	Production(tons)	% world
• Egypt	1,352,950	17.2
• Saudi Arabia	1,078,300	13.7
• Iran	1,023,130	13
• U.A.E.	775,000	9.8
• Pakistan	759,200	9.6
• Algeria	710,000	9
• Iraq	566,829	7.2
• Sudan	431,000	5.4
• Oman	276,400	3.5
• Libya	161,000	2
• Asia	4,804,126	61.1
• Africa	3,011,205	38.3
• America	26,003	0.3
• Europe	16,121	0.2
World	7,857,455	–

Date seeds are discarded or used mainly as animal feeds for cattle, sheep, camel and poultry [15]. The reported composition was 3.1–7.1% moisture, 2.3–6.4% protein, 5.0–13.2% fat, 0.9–1.8% ash and 22.5–80.2% dietary fiber. In addition, the seed contains high level of phenolic compounds (3102–4430 mg gallic acid equivalents/100 g of seed powder) and has high amounts of antioxidants (580–929 μ mol trolox equivalents/g) and dietary fiber (78–80 g/100 g) [16].

Solvent extraction is one of the traditional techniques of extracting vegetable oil from oilseeds. In this method, pretreated (if necessary) oil seeds are put in contact with a suitable solvent, in its pure form, for extracting the oil from the solid matrix to the liquid phase. Solvent extraction technique [17, 18] is one of the cheapest and most efficient processes, applied to produce oil from seeds. Jojoba oil, soybean oil, palm oil, jatropha oil and many other oils are produced by this method.

The main objective of this research work is to produce a bio-oil from Date Palm seeds with the help of a conventional Soxhlet apparatus. The yield of the production is optimized with respect to particle size of the crushed seeds, extraction time, and choice of solvents. Later this bio-oil is physico-chemically characterized using Fourier Transform Infrared Spectroscopy (FT-IR) for the existence of surface functional groups and Gas Chromatography-Mass Spectrum for the identification of the fatty acids composition. Some of the basic fuel properties like iodine value, saponification value, and kinematic viscosity are determined to characterize fuel quality of the bio-oil.

II. MATERIALS AND METHODS

2.1. Materials

2.1.1 Date palm seed:

The date fruits (from zahidi trees) which were collected from farmlands in Karbala city, South Iraq in the late summer of 2013. After collected the palm fruit (Zahidi), seeds were separate manual and then washed in order to remove the peels and dry under the sun as shown in figure (1). The cleaning and washing seed was grinding using mechanical grinder model (RRH-AS500). Then, the ground palm date seed was separated by using a sieve shaker model 160 into three types of particle size, 0.425

mm and below and 1 mm and below and 1.2 mm and below to determine the optimum size and to obtain the highest oil yield.



Figure 1: palm fruit and palm seed

2.1.2. Choice of solvents:

In order to find out the best solvent that would give the maximum yield, extraction is carried out for predetermined period of time (4 h) using a variety of solvents (n-Hexane, Methanol, 2-Propanol, Chloroform, Toluene), for a fixed weight of palm seed (50 g) and fixed particle size of grounded seed (2 mm) in soxhlet apparatus. Leaching is carried out at the boiling point of each solvent. The physical properties of the solvents are indicated in Table (2).

Table (2): Physical properties of various solvents used.

Solvents	Boiling point (C°)	Density (kg/l)	Refractive index (25 °C)
Methanol	79	0.789	1.3616
2-opropanol	82	0.785	1.3772
Chloroform	61	1.498	1.4459
Hexane	69	0.655	1.3723
Toluene	111	0.867	1.4941

2.2. Methods:

2.2.1 Determination of moisture content:

Moisture and volatile matter content were determined using a procedure described by the British Standards Institution (BS EN ISO 665:2000).

2.2.2 Determination of Seed Oil Content:

The oil content of palm seed was determined according to the procedure described by the British Standards Institution (BS EN ISO 659:2009).

2.2.3 Apparatus:

Solvent extraction is done in a soxhlet apparatus to extract the palm oil from its seeds. This particular soxhlet apparatus consists of a glass extractor, fitted in between a round bottom flask at the bottom and a bulb condenser at the top. Inside the glass thimble holder, solid matrix of seeds is placed within thimble. The round-bottom distillation flask initially contained an extracting solvent and it is heated up by electrothermal heating mantle 450 C° maximum temperature, 1L max capacity and power 300W. As the solvent vapor goes up to the condenser, it condenses and accumulates inside the extractor. Here, the solvent comes in contact with the seeds and oil is leached out of the seeds. When the condensate moves down through the bed of seeds, mass transfer takes place. However, major amount of mass transfer of oil from the seeds to solvent occurs when the accumulated solvent moves up purely due to the hydrostatic pressure head so, surface area offered by the bed and the seed-solvent contact time are the two major factors for the yield of the oil production.

2.2.4 Oil extraction:

To obtain highest oil yield by selects appropriate time and particle size for oil extraction from date seed. 50 g of ground seed were weighed and transferred to a 30 mm × 200 mm cellulose thimble. It placed in the extraction chamber of a 250 mL soxhlet apparatus fitted with a condenser, which was placed on a 500-mL distillation flask containing 250 mL of solvents n-hexane. Date seeds oil was then extracted under reflux with n-hexane for 1, 2, 4 and 6 h (10– 12cycles/h). After that, hexane was then

removed by using a heated rotary evaporator (Stuart, England), under vacuum conditions. All extractions process were performed in triplicate, and the mean values were reported. The yield of oil extracts was expressed as a percentage of the weight of extracts obtained from extraction relative to the weight of date seeds used for extraction.

$$\text{Yield of oil extraction} = \frac{(\text{weight of oil extracted})}{(\text{weight of date seeds used})} \times 100\% \quad (1)$$

2.3 Analysis of bio-oil:

2.3.1. GC/MS analysis:

Fatty acid composition of the oil is determined by GC/MS analysis. A Gas Chromatograph (Make: Agilent Technologies; Model: 7890A;) with HP column, 30 m long x 0.32 mm ID x 0.25 μm film thickness (Make: SGE, Australia). The GC is equipped a flame ionization detector (FID) (Make: Hewlett Packard, USA). Helium gas of 99.99% purity is used as carrier gas. Data analysis is performed using GC/MSD CHEMSTATION software. The GC/MS instrument conditions are given in Table (3).

To obtain the fatty acid composition of oil must be converted TG to FAME to decreases the boiling point. After that, 0.2ml of biodiesel added to 1ml of mixture from hexane and 2-propanol (4/5 volume ratio). Before charging the oil, the sample is purified by filtration using 0.2 μm polytetrafluoroethylene syringe micro filter. 1- μL of the sample was then manual injected into the GC/MS using a 5- μL micro syringe (SGE, Australia).

Table (3): Operating conditions for the GC/MS

GC	
Carrier gas	Helium
Operation mode	Constant flow
Inlet temperature	250 °C
Oven temperature	240 °C
Injection volume	1 μl
Split ratio	1:50
Run time	20 min
Operation mod	constant flow
Average velocity	37.789 cm/sec
Pressure	17.04 psi
MS	
Transfer line temperature (°C)	240 °C
Source temperature (°C)	220 °C
Solvent delay	2 min
Scan mass range	45–500

2.3.2. FTIR analysis:

FTIR can be utilized to identify some of the functional groups present in a solid, liquid or gaseous sample. In the present study, the functional groups of the oil sample are analyzed by using Fourier transform infrared spectroscopy (FTIR) (Make: Bruker, Germany). The absorption frequency spectra are recorded and plotted as transmittance versus wave number. The standard IR spectra of hydrocarbons are used to identify the functional groups of the bio oil.

2.3.3. Iodine values:

Iodine value is a measure of the degree of unsaturation of the fuel. The unsaturation in the fatty acid chain is the main source of thermal instability and reason for causing carbon deposits due to burning. Iodine value (IV) of oil was calculated from fatty acid methyl ester compositions of oil with the help of Eqs. (2) [19].

$$IV = (\sum(254 \times D \times A_i)) / MW_i \quad (2)$$

Where A_i is the percentage of each fatty acid, D is the number of double bonds, and MW_i is the molecular mass of each component.

2.3.4. Saponification values:

Saponification value is used as an indicator of fatty acid chain length. The value is simply given as a measurement of the ml of KOH required to complete the hydrolysis of one gram of oil. The high saponification value is associated with corrosion problems to the critical parts of a diesel engine. The range of saponification values for various types of bio oils is 160– 190 mg KOH/g. Saponification value (IV) of oil was calculated from fatty acid methyl ester compositions of oil with the help of Eqs. (3) [19]:

$$SV = (\sum(560 \times A_i)) / MW_i \quad (3)$$

Where A_i is the percentage of each fatty acid, D is the number of double bonds, and MW_i is the molecular mass of each component.

2.3.5. Acid Value and Acidity:

Acid value (or "neutralization number" or "acid number") is the mass of potassium hydroxide (KOH) in milligrams that is required to neutralize one gram of chemical substance. The acid number is a measure of the amount of carboxylic acid groups in a chemical compound, such as a fatty acid, or in a mixture of compounds. Acidity is a percentage of free fatty acids in oil. The Determination of acid value and acidity were carried out based on a titration method. The procedure used in the experiments is described by the British Standards Institution (BS EN ISO 660: 2009).

2.3.6. Viscosity and density:

The viscosity and density of palm seed oil were predicated by using Cone- Plate test (DV-III ultra) and four digital high precision density testers, respectively.

III. RESULTS AND DISCUSSION

3.1. Yield of oil extraction:

3.1.1. Effect of use of different solvents:

Figure (2) indicates the variation of the yields of palm seed oil using various extraction solvents. The highest oil yield was found for toluene, a non- polar solvent. This result shows that palm seed oil yield is better with non-polar solvents (like chloroform, n-hexane and toluene) as compared to those with polar solvents (like methanol and 2-propanol). Toluene has a high boiling point, so it needs more heat to form vapor in any distillation recovery process compared to other solvents also, it produce higher amount of aromatic component than n-hexane solvent. The high boiling point of toluene can explained its high yield as compared to n-hexane. The explanation of this phenomenon can be due to the contact time between seed and solvent which is long, where the major amount of mass transfer of oil from the seeds to solvent occurs when the accumulated solvent moves up purely due to the hydrostatic pressure head [20]. Chloroform is not appropriate, because of the high concentration of aromatic components that is observed in FTIR test and low oil yield. Methanol and 2-propanol are not very suitable for the recovery of oil probably because of its polar character. n-hexane was used as a solvent in this research since it is less costly and its yield is comparable with that of the toluene.

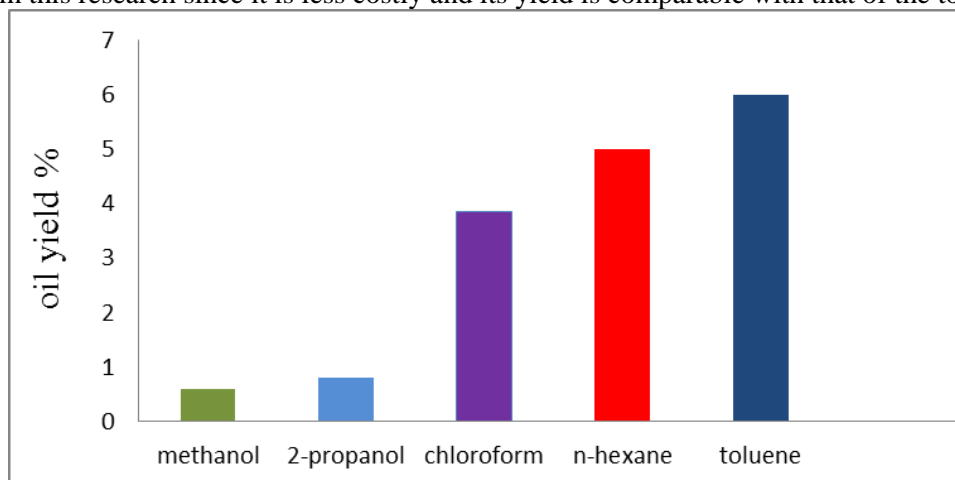


Figure 2: Effect of solvent type on oil yield.

3.1.2. The effect of particle size:

Figure (3) shows the effect of particle size on the yield of oil. It has been noticed that the decrease in particle size leads to increase of oil yield. This manner is expected because of the increased surface area of grounded seed. For this reason, the contacted area between seed and solvent increased, and the mass transfer of oil from the solid phase to the liquid phase increased accordingly. Also, the time needed for the solvent to diffuse inside the small particle seed is lower than large particle [21].

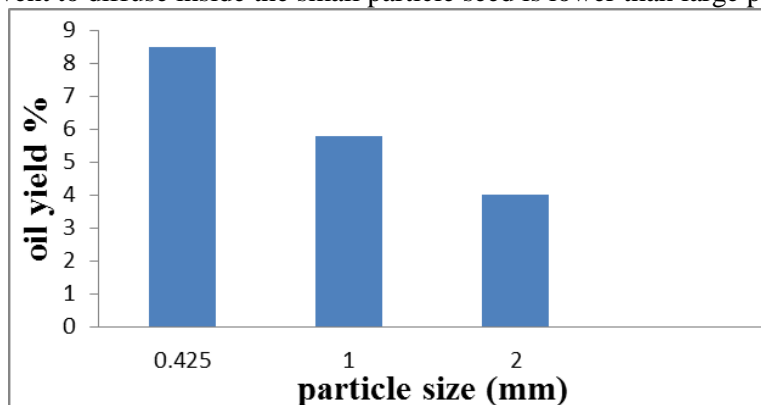


Figure 3: The effect of particle size on oil yield at 120 min.

3.1.3 The effect of extraction time:

Figure (4) shows the impact of extraction time on oil yield for different particle size. It has been observed that the oil extraction rate was quick at the starting of the extraction process before reaching the steady state. This is because the driving force for transfer of oil from the solid phase to the liquid phase is higher in start of the process. In the other word, the difference of oil concentration between the solid phase and solvent phase is greater in the initial extraction process. Therefore, the oil diffuses rapidly from date seed to the solvent and the maximum amount of extractable oil was transferred. The oil yield unchanged even after prolongation the time of extraction process. The optimum extraction time is about 2 h for all particle size [22].

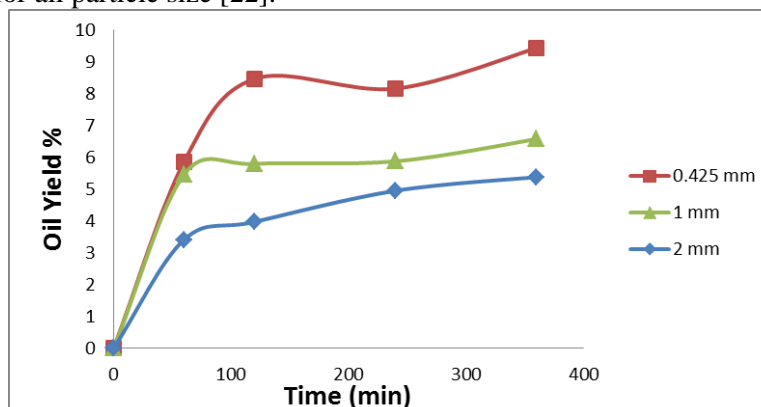


Figure 4: The oil yield vs. time for different particle size.

3.2. FTIR analysis:

Oil extracted by n-hexane:

Figure (7) shows the FTIR spectrum of the PSO extracted by n-hexane. The functional groups identified from FTIR spectrum of the oil as follow. The peaks at 584 cm^{-1} represent various inorganic compounds. The peak at 721 cm^{-1} is owing to the aromatic compounds [23]. The peaks in the $852\text{--}1114\text{ cm}^{-1}$ region are represented to the stretching vibration of C-O ester groups and the CH_2 wag. The peak at 1157 cm^{-1} attributed to the C-O stretching alcohols groups. The peaks in the $1200\text{--}1400\text{ cm}^{-1}$ region are mostly assigned to the bending vibrations of CH_2 and CH_3 aliphatic groups like symmetric HCH bending at 1376 cm^{-1} and CH_2 scissoring at 1457 cm^{-1} . The peak at 1743 cm^{-1} is assigned to the C=O stretching vibration of carboxylic acids of the ester. The peaks centered at 2921 cm^{-1} and 2852 cm^{-1} are assigned to the stretching vibrations of aliphatic C-H in CH_2 and terminal CH_3 groups

respectively. The two small peaks at 1652 cm^{-1} and 3648 cm^{-1} corresponding to the bending and stretching vibration of O-H bonds of the H_2O molecule in the oil [24].

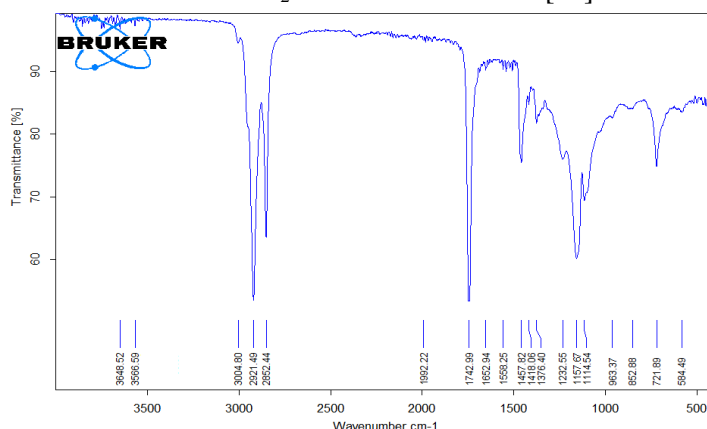


Figure 7: The FTIR spectra of PSO.

Oil extracted by other solvent:

Figure (8) show the FTIR spectra of PSO that extracted by toluene, chloroform compared with PSO that extracted by n-hexane. The functional groups of the oil extracted by toluene and chloroform are the same as for oil extracted by n-hexane. However, it was observed that the intensity of peak at 720 cm^{-1} of the first group is higher than that of oil extracted by n-hexane. This difference can be explained by the higher concentration of aromatic components in oil extracted by toluene and chloroform than that extracted by n-hexane.

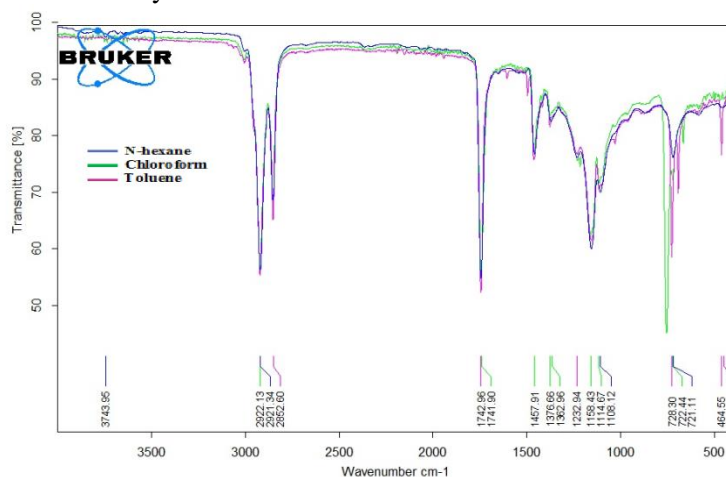


Figure 8: The FTIR spectra of PSO extracted by toluene and chloroform compared by n-hexane.

Figure (9) show the FTIR spectra of PSO that extracted by methanol and 2-propanol compared with PSO that extracted by n-hexane. When the FTIR spectra of oil extracted by methanol are compared with that of n-hexane, similarity is noticed in overall wave shape but with significant difference in the intensity of peaks (amplitude). This is because the methanol is a less efficient solvent for PSO extraction, which is returned to characteristic of methanol and oil. In FTIR spectrum of oil extracted by 2-propanol there are some of new peaks. The peak at 3303 cm^{-1} can be representing O-H stretch of alcohols or phenols. The peaks at 1609 cm^{-1} and 1520 cm^{-1} assigned to the $\text{C}=\text{C}$ stretching of alkenes and to the aromatic $\text{C}=\text{C}$ stretching, respectively [23].

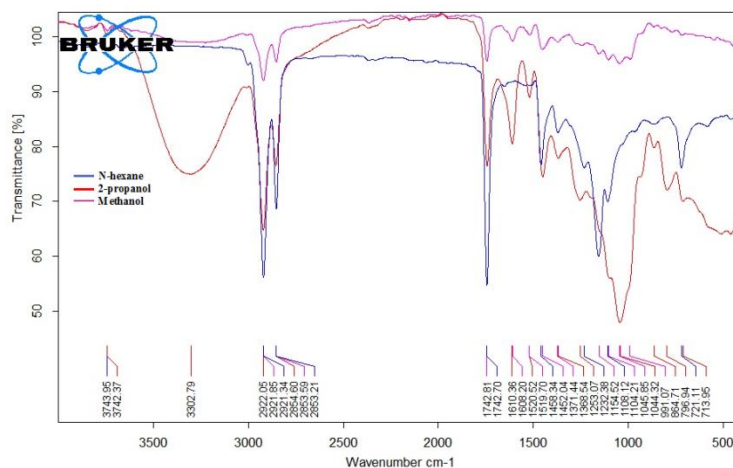


Figure 9: The FTIR spectra of PSO extracted by methanol and 2-propanol compared by n-hexane.

3.3. Fatty acid composition of palm seed oil:

The identification of fatty acid composition of palm seed oil extracted by the Soxhlet method was performed by Gas Chromatography Mass Spectrometry (GC/MS). The result is shown in figure (6).

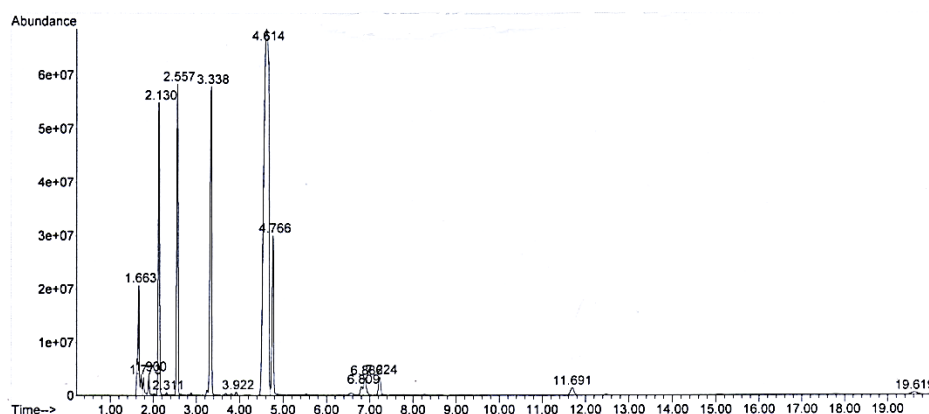


Figure 6: GC/MS diagram of PSO methyl ester.

The fatty acid compositions of the PSO that detected through the study are listed in the table (5). The results show that the oleic acid had the highest amount among unsaturated fatty acid while palmitate acid had the highest amount among saturated fatty acid. The high amount of oleic acid is in agreement with other research but for another kind of date palm like Akbari et al. [25] where they found that the percentage of oleic acid are 37.6%, 31.47%, and 31.79% in Kabkab, Shekar, and Shahabi respectively.

Table 5: The fatty acid compositions of the PSO.

Retention time (min)	Identified compounds	Saturation	Weight (%)
1.773	Caprylic acid	C8:0	0.7
1.9	Capric acid	C10:0	0.713
2.13	Lauric acid	C12:0	11.363
2.311	Tridecanoic acid	C13:0	0.103
2.557	Myristic acid	C14:0	11.447
2.883	Pentadecenoic acid	C15:0	0.068
3.338	Palmitate acid	C16:0	13.848
3.922	Heptadecanoic acid	C17:0	0.14
4.613	Oleic acid	C18:1	51.456
4.766	Stearate acid	C18:0	6.56
6.886	Cis-11 eicosenoic acid	C20:1	1.297

7.224	Eicesnoic acid	C20:0	1.175
11.691	Heneicosanoic	C22:0	0.75
19.620	Lignocerate acid	C24:0	0.397

3.4. Date Palm seed characterization:

The average weight of a palm seed is around 10–15% of the palm fruit weight. The oil content of date palm seed was about 10% that was determined according to the procedure described by the British Standards Institution (BS EN ISO 659:2009). The moisture and volatile matter content of date palm seed were 11.6 % that determined using the procedure described by the British Standards Institution (BS EN ISO 665:2000). This value is higher than that reported by other workers 3.1–7.1% reported by Amani et al. [13]. The high content of moisture can be returned to the fact that the used seeds were fresh and didn't dry enough.

3.5. Basic fuel properties:

Table (4) shows some properties of palm seed oil like viscosity (CP) at 40 °C , density(g/cm³), refractive index (n), saponification value(mg KOH /g) (SV), acid value (mg KOH/g)(AV) and iodine value (mg/g) (IV) compared with other researchers. At room temperature, PSO is a yellow liquid as shown in figure (5) having refractive index of 1.446. The percent of FFA in PSO is about 0.9 %. The free fatty acid percent and acid value of the PSO are little in general [26]. The PSO has little iodine value 45 due to its high content of saturated fatty acids. The high saponification value of 206 of the *P.dactylifera* seed oil indicates very high content of low molecular weight triacylglycerol [27].

Table 4: Physico-chemical properties of palm seed oil compared with other researchers:

Properties of Palm seed oil	ρ (g/cm ³)	μ (CP)	n	SV	IV	AV	Refer.
Zahidi	0.925	29	1.446	206	45	1.85	Current study
multiple trees	-	-	-	224	46	-	[13]
Kabkab	-	-	1.462	-	-	1.79	[25]
Shekar	-	-	1.462	-	-	1.33	[25]
Shahabi	-	-	1.461	-	-	1.07	[25]



Figure 5: palm seed oil

IV. CONCLUSIONS

The bio oil extracted from palm seeds is very much similar to other bio oils in chemical composition and basic fuel properties. It could be inferred from the present study generally, that date seed oil is rich in oleic acid. It has high viscosity compared with other type of vegetable oil. FTIR analysis shows that the palm seed oil is highly dominant with oxygenated species. GC–MS analysis of the oil indicates the presence of low molecular weight fatty acids with no unsaturation. The best oil yield was

satisfactory at 2h extraction time and 0.425 mm particle size by using Soxhlet extractor and n-hexane as solvent.

V. FUTURE WORK

Palm seed oil may become economically competitive in addition to being environmentally desirable. Additional research in this area is therefore recommended as follows:

- Distillation and thermo gravimetric analysis (TGA) of the Palm Seed oil to confirm the contents of low-boiling and high-boiling compounds. This analysis will help us to characterize the evaporation, decomposition and combustion kinetics.
- Determination of the thermal stability of the Palm Seed oil by observing the change of viscosity and water content of the oil with temperature.
- Determination of pour point, cloud point temperature and surface tension of the Palm Seed oil are necessary to categorize it as fuel.
- Improving oil extraction operations as well as finding other ways more efficient and less polluting to the environment.

ACKNOWLEDGEMENTS

The collaboration with the staff of Electrochemical Engineering Department/ College of Engineering / University of Babylon for the project is gratefully acknowledged.

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