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GCMS and FTIR Characterisation of Date Seed Oil

Sadisu Girigisu[1*], Sulaiman Adamu I.[2], Adebara Oladele O.[3], Yahaya Idris H.[4], Ayeni Adeyemi A.[5]

[1*,] Department of Biochemical and Chemical Science, Federal Polytechnic Offa, P.M.B 420, Offa Nigeria.
[2,3,4,5]Department of Science Laboratory Technology, Federal Polytechnic Offa, P.M.B 420, Offa Nigeria.
[*]Corresponding author email: sadisu.girigisu@fedpoffaonline.edu.ng (S. Girigisu) Tel.: +2348062852237

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Abstract

Date palm is an important agricultural product in many Arabian countries, and millions of people consume its fruit. The oil was extracted using a soxhlet apparatus with toluene as the extractant. The physicochemical parameters obtained for the oil include free fatty acid value (1.4±0.05 mg NaOH/g), acid value (2.8±0.05 mg KOH/g), Saponification value (396.91±0.045 mg KOH/g), Iodine value (38.07±0.02 gI_2/w), Peroxide value (29.5± 0.07 mEq/kg), and pH value (5.1). The Fourier transform infrared spectroscopy also reveals the functional group present in the oil to include C=O of the Ester group at around 1114.5cm^{-1}, C-H of Alkanes (2922.2 & 2855.1cm^{-1}). The peak at 1461.1 cm^{-1} is attributed to C-H bending of alkane while the peak at 1744 cm^{-1} is attributed to the C=O bending of carbonyl groups. The Gas Chromatography-Mass Spectroscopy reveals the major fatty acid composition of the oil to include oleic acid as well as palmitic acid which are important raw materials for pharmaceuticals, beverages, and the cosmetics industry, it also contains myristic acid which is used for food additives. The percent composition of the Oleic and lauric acid found in this oil is relatively higher as compared to those of other regions, also the presence of cis-11-eicosenoic and heneicosanoic which are the essential raw material for foam and paint industries makes this oil richer as compared from those of literature.

1. Introduction

Date palm fruit (Phoenix dactylifera L.) is globally marketed as a high-value commodity. It is produced in many arid and semi-arid regions of the world and has always played a crucial role in the economic and social life of the people of these regions [1, 2]. Date seeds, often considered waste, are a valuable byproduct of the date palm industry. They have drawn increasing attention recently due to their potential use in various applications, including the food, pharmaceutical, and cosmetic industries [3]. One crucial step in harnessing the beneficial properties of date seeds is the extraction of bioactive compounds from them. This process typically involves the use of solvents like N-hexane, followed by the characterization of the extracted compounds [4]. Date seeds, obtained from date palm trees, have a rich history dating back thousands of years. Originating in the Middle East, particularly in regions like Iraq and the Persian Gulf, date palms have been cultivated for their sweet, nutritious fruits. There are numerous types and species

of date palms, each characterized by distinct flavor profiles, sizes, and colors [4]. The Major types include Medjool, Deglet Noor, and Zahidi. Date seeds, often considered by-products, exhibit potential as a sustainable resource. While primarily used for propagation to grow new date palm trees, ongoing research explores alternative applications, such as animal feed or as a potential source of antioxidants. The multifaceted nature of date seeds underscores their significance beyond the fruit they sprout. Beyond propagation, date seeds have been considered very nutritive. They are rich in fiber, protein, and various micronutrients. The antioxidant properties have been explored by different researchers and it was found useful for different medicinal purposes. Additionally, date seeds can be converted to powdery form which can be incorporated into various food products, contributing to sustainability by minimizing waste [5]. The most important component of dates is carbohydrates, which may constitute about 70%. Its natural constituents have been screened for different medicinal activities to minimize the side effects of synthetic drugs that are harmful to the body system [6]. Date seed oil is a source of saturated (lauric, myristic, and palmitic acids), mono-unsaturated (palmitoleic and oleic acids), and polyunsaturated (linoleic and linolenic acids) fatty acids at about 50, 43, and 8%, respectively [7]. Date seeds are highly recommended for use in foods and dietary supplements. Because it is a very good source of dietary fiber. The total mineral content found in single seed is comparable to the mineral content of barley. The minerals contained in date seed include sodium, potassium, calcium, iron, copper, magnesium, manganese, zinc, phosphorus, lead, and cadmium. The quantity of dietary fiber found in date seed is about 58 % of which 53 % is soluble dietary fiber and exists as hemicellulose, cellulose, and lignin [8,9]. Their nutritional value with high antioxidant content may reduce the risk of many diseases related to oxidative stress [10-11]. The main products based on date fruits are jam, syrups, vinegar, date powder, and date paste. In most cases, date fruit seeds are regarded as by-products, they are disposed of or sometimes used as animal feeds [12]. Considering the high nutritional content of date seeds, few research has been carried out on the use of date seeds as a supplement for the human diet [13].

In recent times, there has been little or no research on date seed oil obtained from Northern Nigeria, specifically Kwara state, this research is thus carried out in other to compare the basic fatty acid component, physicochemical properties, and functional groups with date seed obtained from another region.

2.0 Experimental

2.1 Materials and Methods

2.2 Sample Collection and Preparation

The sample (dried date fruits) was collected from Offa, Kwara State. The dried date fruits were washed with distilled water and busted open to remove the seeds. The seeds were collected and sun-dried for some hours. It was then pulverized with mortar and pestle into smaller lumps and was grated into powdered form. The samples were packed in an air-tight container and stored at 25 ℃ for further analysis.

2.3 Apparatus/Equipment

The equipment used for this research work includes Fourier Transform Infrared Spectroscopy FTIR (Agilent, carry 630 model), Gas chromatography-mass spectroscopy GC-MS (Agilent 7890A GC/5975 MSD). Soxhlet apparatus (Model IC-122), and heating mantle.

2.4 Extraction of Oil

67.5g of crushed date seeds were weighed and transferred to a 30 mm × 200 mm cellulose thimble. It was then placed in a Soxhlet apparatus fitted with a condenser and placed in a distillation flask containing 250 ml of solvents toluene. The Soxhlet apparatus was heated to reflux at a constant temperature of 95 - 100℃ (below the boiling point of the solvent) and the vapor moved up through the distillation arm into the chamber holding the thimble of solid where the vapor is condensed and warm solvent flow down gravity and percolate through the bed of the crushed date seed to extract oil. Date seed oil was then extracted under reflux with toluene for 5 hours. After that, the oil mixed with the solvent was poured into a round bottom flask setup in a rotary evaporator at 50°C for 30 minutes to ensure complete removal of the residual solvent after which it was stored in a sample bottle for further analysis [14].

3.0 Results and Discussion

3.1 Physico-Chemical Parameters of the Date Seed Oil

The physicochemical properties of the date seed oil sample obtained in this study in comparison with those of other regions are presented in Table 1.

Table 1: Physicochemical properties of oil obtained from this study as compared to other regions.

Parameters	**This study**	[15].	[16].	[17].
Color	Yellowish – brown	Red Yellow	-	-
Free Fatty Acid (mgNaOH/g)	1.4± 0.05	-	0.6	-
Peroxide Value (mEq/kg)	29.5 ± 0.07	4.8	3.6	-
Acid Value (mgKOH/g)	2.8± 0.05	2.55	-	1.35-1.38
Saponification Value (mgKOH/g)	396.91± 0.045	0.255mg/g	191.3	204.84-215.87
Iodine Value (gI_2/w)	38.07 ± 0.02	71.12	76.7	67.22 – 74.80
pH Value	5.1			

The extracted oil was yellow-brownish in color; this may be due to the presence of carotene pigment in the date oil. The free fatty acid (FFA) content of oil is an indication that the oil will be useful in soap production [13]. The FFA and acid values obtained in this research are 1.4 mg NaOH/g and 2.8 mg/KOH/g respectively. However, according to [18], Free fatty acid (FFA) and Acid Value (AV) should be found in smaller amounts for oil in general, as low FFA and AV give high-quality oil which makes it acceptable for edible purposes. The FFA value of this oil is a bit higher in comparison to the findings of [16], [17], and [15] with 0.6%, 0%, and 0% respectively as highlighted in the table. This may be a result of differences in climatic conditions of their geographical locations.

Also, the acid value obtained in this study is a little bit higher than the one obtained by [15], [17], [16] as indicated in Table 1 above where the acid values obtained are 2.55, 1.35-1.38, and 0% respectively. Therefore, the relatively high FFA and AV obtained in this study is an indication that the oil will be useful in soap production.

The saponification value provides the required information on the average molecular weight of all the fatty acids present in the oil sample. This implies that when the saponification value of the oil is high, then the molecular weight of all fatty acids present in the oil will be low, and vice versa. The saponification value (396.91 mg KOH/g) obtained in this study was found to be higher than that of [17] [16] [15]**.** Thus, the higher saponification value is an indication that the oil will be suitable for soap production.

Peroxide value is a valuable measure of oil quality as it indicates the stability of the oil and the level of deterioration of fats. It is a measure of the extent of oxidation of fat or oil. It measures the oxidative rancidity of oil. The peroxide value obtained in this work is higher than that of the findings of [19], [16], and [15]. A low peroxide value that is less than 30 mEq/kg is safe to use to avoid rancidity [20].

The iodine value is a measure of the amount of unsaturation in fats and oils. The iodine value of this date oil is 38.07 gI_2/w, oils characterized with iodine value of less than 100 $gI_2/100g$ are regarded as non-drying oils. The iodine value obtained in this study is lower in comparison with the iodine value obtained from other research by [16], [15] and [17], where the IV values were obtained to be 71.2, 76.2, and 67.22 respectively, this is an indication that the oil is a non-drying type which makes it fit in for soap, paint and cosmetics industries.

3.2 Fatty Acid Composition

The identification of the fatty acid composition of date seed oil extracted by the Soxhlet method was performed by the Gas Chromatography-Mass Spectrometry (GCMS) model Agilent 7890 A GC/5975 MSD.

The GCMS analysis revealed that the date seed oil is predominantly rich in saturated acids except Oleic acid, which is the only unsaturated acid present in the sample. The most abundant ones of these acids include Oleic acid, Palmitic acid Myristic acid as well as Lauric acid, this is in agreement with the fatty acid components of date seed oil obtained by [14], [21], and [22]. The presence of oleic acid alongside Palmitic acid indicates that the oil can serve as a very important precursor in the Food, beverages, pharmaceuticals, and cosmetics industries, Oleic acid is recognized as one of the most important unsaturated fatty acids because of its potential for reducing cholesterol in the blood, as well as its high oxidative stability [23]. It is widely accepted that dietary oil which is rich in unsaturated fatty acids prevents cardiovascular and inflammatory diseases [24].

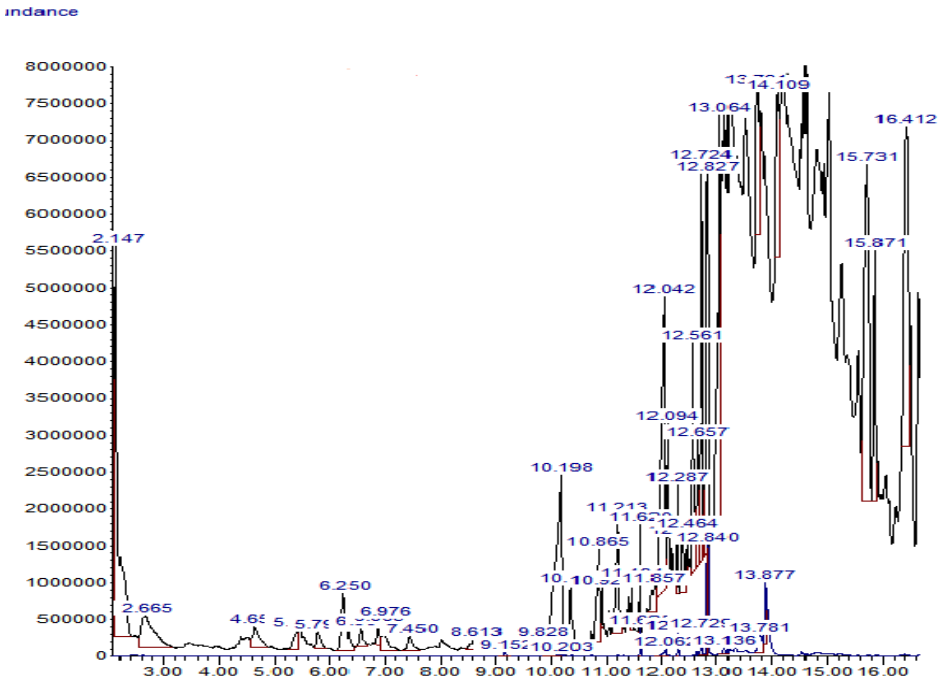

Figure 1: GC-MS chromatogram of toluene fraction of date seeds

Also, Myristic acid can serve as an important agent in the production of food additives, as compared to those of other research in Table 2, the myristic acid obtained in this study is higher, making it a potential ingredient for use in the food additives industries. The Lauric acid in the extracted oil serves as an active component used for soap production. The effectiveness of lauric acid on prostatic hyperplasia development has been pointed out by different findings [25], it has better health benefits compared to trans-fatty acids [26], also, its antimicrobial properties have been reported to inhibit the growth of microbes and production of toxins [24-26]. In comparison with oils extracted from date seeds of other regions, as highlighted in Table 3, it could be observed that the percent oleic and myristic acid present in the oil of this study is higher than those of other studies except that of [15] where the percent oleic acid yield is the same, thus, it serves as an indication that the oil will be of great importance to food, beverages, and pharmaceutical industries. Also, the presence of other fatty acid components of the oil such as Cis-11-eicosenoic acid which is used in preparing food supplements, and Heneicosanoic which is used as a precursor in foam and paint production makes the oil richer than oils obtained from date seed of other regions.

Table 2: Fatty acid content of oil from different regions compared with this study

	This study	[27]	[15]	[28]	[29]	[30]	[31]
Caprylic acid	**0.7**	0.80	0.71	Nd	0.88	0.48-0.56	0.25
Capric acid	**1.9**	Nd	Nd	Nd	0.41	Nd	Nd
Lauric acid	**11.36**	17.8	10.36	16.7-20.3	21.8	25.7-30.8	35.31
Mystriric	***11.45**	9.84	10.44	10.2-12.3	10.80	6.9-16.7	0.04
Palmitic acid	**3.38**	10.9	12.83	9.8-10.9	8.21	11.9-13.1	12.58
Stearic acid	**4.76**	5.67	5.56	2.9-3.7	2.98	1.8-2.3	3.3
Oleic acid	***51.45**	41.3	51.45	44.9-48.4	46.03	31.5-37.6	39.5
Cis-11 eicosenoic acid	***1.29**	-	-	-	-	-	-
Heneicosanoic	***0.75**	-	-	-	-	-	-

Table 2: Fatty acid content of oil from this study

S/N	Retention time	Identified compounds	Saturation	Weight percent	Molecular Formulae
1	1.773	Caprylic acid	C8:0	0.7	$C_8H_{16}O_2$
2	1.9	Capric acid	C10:0	0.713	$C_6H_{12}O_2$
3	2.13	Lauric acid	C12:0	11.363	$C_{12}H_{24}O_2$
4	2.311	Tridecanoic acid	C13:0	0.103	$CH_3(CH_2)11C00H$
5	2.557	Myristic acid	C14:0	11.447	$C_{14}H_{28}O_2$
6	2.883	Pentadeanoic acid	C15:0	0.068	$C_{15}H_{30}O_2$
7	3.388	Palmitic acid	C16:0	13.848	$CH_3(CH_2)14COOH$
8	3.922	Heptadecanoic acid	C17:0	0.14	$C_{17}H_{34}O_2$
9	4.613	Oleic acid	C18:1	51.456	$C_{18}H_{34}O_2$
10	4.766	Stearate acid	C18:0	6.56	$C_{18}H_{36}O_2$
11	6.886	Cis-11 eicosenoic acid	C20:0	1.297	$C_{20}H_{38}O_2$
12	11.691	Heneicosanoic	C22:0	0.75	$C_{21}H_{42}O_2$

3.3 FTIR Analysis

The FTIR analysis was done using Fourier Transform Infrared Spectroscopy FTIR (Agilent, carry 630 models).

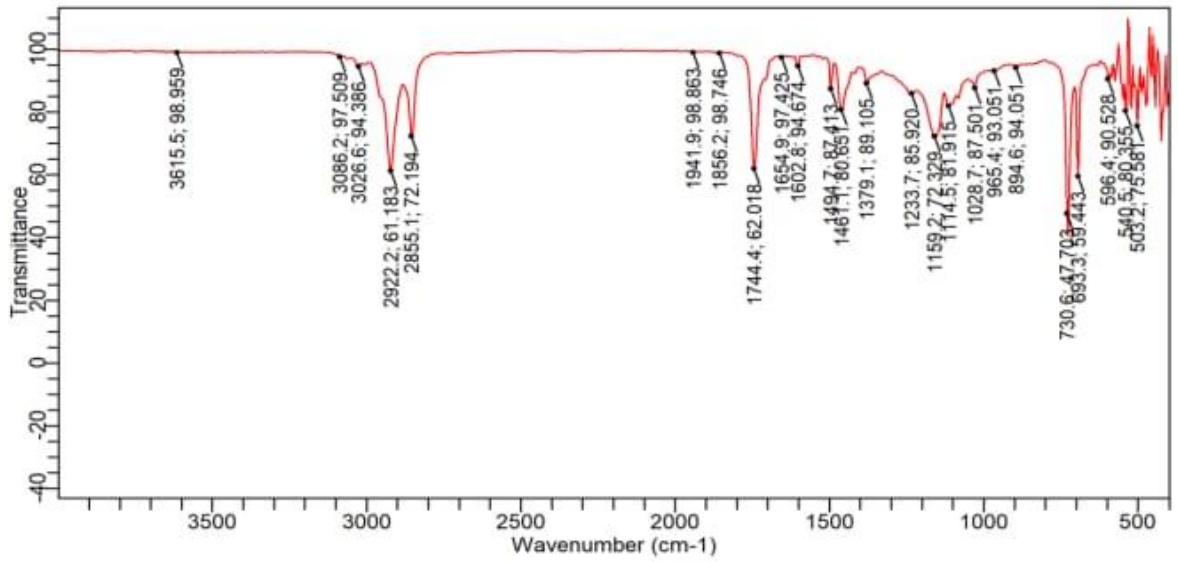

Figure 2: FTIR spectral of different functional groups of the oil

The major functional groups identified from the FTIR spectrum of the oil are as follows. The peak at 730.6 cm^{-1} is owing to C-H bending of alkynes. The peak at 894.6 can be attributed to C-H of aromatics, while the peak at 1114.5 cm^{-1} region is a characteristic stretching vibration of ester groups. The peak at 1461.1 is attributed to the C-H bend of alkane while the peak at 1744 cm^{-1} is attributed to the C=O bending of carbonyl groups. The peaks centered at 2922.2 cm^{-1} and 2855.1 cm^{-1} are assigned to stretching vibrations of aliphatic C-H of Alkane. Similar absorption bands were obtained by [32] for avocado seed oil. The peaks at 3026.6 and 3086.2 cm^{-1} are attributed to the C-H stretching of alkenes, and the peaks at 1744.62 cm^{-1} are attributed to the ester and ketone stretching, [33].

4.0. Conclusion

Date seed oil is important for its contents of saturated and unsaturated fatty acids which can be used for different applications in industries. The presence of the oleic acid alongside Palmitic acid found in the extracted oil indicates that the oil can serve as a very important precursor in Food, beverages, pharmaceuticals, and cosmetics industry, more so, the Myristic acid can serve as an important agent in the production of food additives. Lauric acid serves as an active component used in soap production. With the increasing availability of date seeds coupled with the useful chemical component found in them, their economic value cannot be over-emphasized as it can serve as a better replacement to be used in pharmaceuticals, cosmetics, and food beverage companies. Therefore, more research is needed in the purification process to enhance the quality of the extracted oil.

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