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Extraction, Characterization and Determination of Physicochemical Properties of Biodiesel obtained from Desert Date (*Balanites aegyptiaca)* Seed Oil

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ABSTRACT: Biodiesel is a domestically produced, clean-burning, renewable substitute for petroleum diesel. Using biodiesel as a vehicle fuel increases energy security, improves air quality and the environment, and provides safety benefits. . This study extracted, characterized and determined the physicochemical properties of biodiesel produced from Desert date (*B. aegyptiaca)* seed oil using Soxhlet extraction method with n-hexane as the extraction solvent. The oil obtained was characterized using FTIR and GC-MS analyses and its physicochemical properties determined. The oil obtained was subsequently transesterified using methanol and KOH catalyst. The obtained biodiesel was characterized and its physicochemical properties determined. The result showed that *B. aegyptiaca* seed oil had density of 0.9 g/cm^3, viscosity of 6 cSt at 40 ^{0}C and viscosity of 2.5 cSt at 100 ^{0}C. The cloud and pour points of the oil was 21 and 9 ^{0}C respectively. The oil comprised of eleven fatty acids; predominantly linoleic acid (21.88 %), vaccenic acid (13.90 %) and palmitic acid (10.96 %). The biodiesel had density of 0.88 g/cm^3, kinematic viscosity of 4.2 cSt, cloud and pour points of 16 ^{0}C and 7 ^{0}C respectively. The biodiesel also had free fatty acid (FFA) of 0.79 %, iodine value of 42 mgI$_2$/100g and saponification value of 176. The biodiesel primarily consisted of 9,12-Octadecadienoic acid methyl ester (45.46 %), Hexadecanoic acid methyl ester (20.12 %), Butyl-9,12-Octadecadienoate (12.43 %) and Heptadecanoic acid-16-methyl ester (10.43 %). The properties obtained for the biodiesel were comparable to those of biodiesels reported in literature and thus, shows remarkable potentials to be used as substitute to fossil-based diesel fuel.

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There is an increase in the demand for energy sources worldwide. The Energy Information Administration of the United States forecasts an estimate consumption of 100.6 million b/d of petroleum and liquid fuels in year 2022 and a global consumption of 102.2 million b/d in year 2023 (EIA, 2022). This demand is driven by advancement in technology, which has ensured that there are various equipment, tools and/or gadget capable of reproducing most manual and mundane activities. Nowadays, diesel has become one of the most important petroleum products due to its high and increasing demand globally (Kojima *et al.*, 2010). In 2004, approximately 700 litres (150 gallons) of diesel was sold every second in the UK. Furthermore, sales of diesel in UK have been steadily increasing for the last twenty years, with demand exceeding 27 billion litres in 2014 (Chilcott, 2006). In the U. S., sales of on-road diesel fuel rose from 32 billion gallons in 1999 to over 37 billion gallons in 2004, an increase of nearly three percent annually (Marketing, 2007). In 2018, diesel accounted for about 20 % of total U.S. petroleum consumption and about 22 % of total petroleum consumption by the transportation sector (Vandervell, 2015). Over the period to 2030, energy analysts forecast that diesel demand will continue to grow (IHS, 2013). Despite the glaring demand and

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need for diesel and other petroleum-based liquids, fossil fuel are non-renewable resources and are available in limited quantities on earth. Furthermore, diesel fuels have been reported to produce many harmful emissions when burned, and diesel-fuelled vehicles are major sources of harmful pollutants such as ground-level ozone and particulate matter. The U.S. Energy Information Administration (EIA) estimates that in 2017, diesel (distillate) fuel consumption in the U.S. transportation sector resulted in the emission of 451 million metric tons of carbon dioxide (CO_2), a greenhouse gas. This amount was equal to 24 % of total U.S. transportation sector CO_2 emissions and equal to 9 % of total U.S. energy-related CO_2 emissions in 2017 (EIA, 2022). Though diesel is reported to cost less than gas, its engines are less versatile than that of gas and thus in the long run, they are quite expensive. Diesel engines are also a huge source of noise pollution. Diesel driven plants have been reported to be quite loud while the fumes they produce are toxic and can lead to lung ailments such as lung cancer etc. (Uddin *et al*., 2016). Thus, there is a need for alternative energy sources to petroleum based fuels due to the depletion of the world's petroleum reserves, global warming and environmental concerns which are as a result of oil exploration. Possible substitutes or compliments of fossil fuel are fuels produced from oils of plant origin. Studies from literature have revealed that these oils are suitable for use in diesel engines (Kumar and Sharma, 2016). Vegetable oils have been hypothesized as potential substitutes for fossil fuels since the early 20th century. However, it was only recently that conscious science-based efforts have been made to investigate the potentials of plant-derived oils as substitutes for fossil fuels. Plant and animal-derived oils are technically feasible, environmentally friendly, and readily available. Therefore, the objective of this paper was to extract, characterize and determine the physicochemical properties of biodiesel produced from Desert date (*B. aegyptiaca*) seed oil.

MATERIALS AND METHOD

Study Material: Balanites aegyptiaca; known as 'Desert date or Thorn tree' is a member of *Zygophyllacceae*, one of the most common plant species of the dry land areas of Africa and South Asia. In Nigeria, it is found mostly in the Northern region. It is known as 'Aduwa' in Hausa, 'Utazi' in Igbo, and '*Teji'* in Yoruba. It is an evergreen, woody, spiny flowering tree about 10 m height and is highly resistant to stresses such as sandstorms and heat waves, and grows with minimal available moisture (Saed and Isam, 2018). *Balanites aegyptiaca* is multi-branched, spiny shrub or tree up to l0 m tall. Its crowns are spherical, in one or several distinct masses. It has short trunks and it often branches from near the base. Its stems are covered with dark brown to grey deeply fissured barks. Its branches are armed with stout yellow or green thorns up to 8 cm long. The leaves of *B. aegyptiaca* are composed of two separate leaflets; these leaflets are obovate, asymmetric, 2.5 to 6 cm long, bright green, leathery, with fine hairs when young. Flowers in fascicles in the leaf axils, and are fragrant, yellowish-green (Schmidt and Joker, 2001).

Fig 1: Balanites aegyptiaca plant showing its fruits

Sample collection and preparation: Balanites aegyptiaca were bought from Samaru market, Zaria, Kaduna state, Nigeria and were identified at the Herbarium, Department of Botany, Faculty of Life Sciences, Ahmadu Bello University Zaria. Healthy *Balanites aegyptiaca* seeds were selected, cleaned and sun dried. The seeds were then cracked and the shells carefully removed to obtain the kernels. The kernels were pulverized using mechanical method (mortar and pestle) and then used for extraction as reported by Shivani *et al*. (2011).

Oil Extraction: Oil from the pulverized *Balanites aegyptiaca* seeds was extracted using the Soxhlet extraction method with n-hexane as the extraction solvent as described by Jock *et al.* (2017). The percentage oil yield was subsequently calculated using the equation below:

$$\% \text{ yield of oil} = \frac{Weight\ of\ oil\ extracted}{Weight\ of\ the\ sample} \times 100$$

Characterization of oil extract: Fourier Transform Infrared Spectrometry (FTIR) was used to study the functional groups present in the oil extract. While Gas Chromatography - Mass Spectrometry (GC-MS) was used to evaluate the fatty acid components of the extracted oil. A Shimadzu QP2010 plus series gas chromatography coupled with Shimadzu QP2010 plus mass spectroscopy detector (GC-MS) system was used for the evaluation. The mass spectra were compared with the NIST05 mass spectral library (Warra and Abubakar, 2015). In carrying out the analysis, the

plates were thoroughly cleaned using acetone. Using a syringe pipette, a drop of the oil sample was placed between the two KCl plates. The chloride plates were then attached to a Fourier Transform Infrared Spectrometer coupled to a computer with print out system. The sample in the plate was irradiated by infrared lamp source at one end of the spectrometer and each sample was analyzed between ranges of 600 cm^{-1} to 4000 cm^{-1} (Dallatu *et al.,* 2017).

Production of biodiesel from B. aegyptiaca oil extract: Base-catalysed transesterification as described by Jock *et al.,* (2017) was adopted for the homogeneous catalyst in the study. For base-catalysed homogeneous transesterification, exactly 250 cm^3 methanol was transferred into a 500cm^3 conical flask and potassium hydroxide (0.25 g) was carefully added into the container and stoppered. The container was swirled round thoroughly for about 2 minutes until the potassium hydroxide becomes completely dissolved in the methanol to form potassium methoxide. *B. aegyptiaca* extract (100.0 g) was filtered into a clean dry conical flask and heated to 60 ^{O}C on a hot plate equipped with magnetic stirrer, with the magnetic stirrer switched off. The prepared potassium methoxide was carefully added to the flask. The hot plate and the magnetic stirrer was then switched on and mixture stirred at low speed of 100 rpm and temperature maintained at 60 ^{0}C for 90 minutes (Jock *et al.,* 2017).

Determination of saponification value: Oil (2.0 g) was weighed accurately into a 250 cm^3 conical flask containing 25 cm^3 of 0.5 M alcoholic KOH solution. A reflux condenser was fitted to the flask and heated in a water bath for an hour, swirling the flask frequently to ensure that the sample was fully dissolved. The excess KOH solution was titrated hot with 0.5 M HCl solution using 1 cm^3 of phenolphthalein indicator until a pink endpoint was reached (Onimisi *et al.,* 2021). A blank determination was also carried out. The saponification value was calculated using equation:

$$\text{Saponification value (mg KOH / g oil)} = \frac{5.6 \times C \times (V^{\circ} - V)}{M}$$

Where; V = Volume (cm^3) of the 0.5M HCl solution used in blank; V° = Volume (cm^3) of the 0.5 M HCl solution used in assay with oil; M = mass molar concentration of HCl solution used; 5.6 = Relative molar mass of KOH.

Acid value determination and free fatty acid (% FFA): The oil was filtered and heated to about 100 ^{0}C to remove traces of water. The oil sample (5.00 g) was then weighed into a clean dried 250 cm^3 conical flask, containing 25 cm^3 absolute ethanol. To this was added 2-3 drops of phenolphthalein indicator and the mixture heated with shaking in water bath for 10 minutes. It was allowed to cool and then titrated against 0.1 M alcoholic potassium hydroxide solution, shaking constantly until a pink colouration persisted for at least 10 seconds (Gonzaga and Sobral, 2012). The volume of KOH solution used was recorded and the acid value was determined according to equation:

$$\text{Acid value (mg KOH/g oil)} = \frac{5.6 \times C \times V}{Weight\ of\ the\ oil\ sample\ (g)}$$

Where; V = Volume of the potassium hydroxide used in cm^3; C = Molar concentration of potassium hydroxide used; 5.6 = Relative molar mass of KOH

The acid value determination was followed by the fatty acid (% FFA) determination as:
% Free Fatty Acid (% FFA) = Acid Value x 0.503

Determination of specific gravity: A clean and dry stoppered density bottle of 25 cm^3 capacity was weighed empty and the weight recorded as W_0. The density bottle was then filled with distilled water, stoppered and maintained in a water bath at 15 ^{0}C for 10-15 min for the water to assume the bath temperature. The outside of the bottle was wiped, dried and reweighed as W_1. The bottle was emptied, washed with water and dried. It was then filled with the oil and equilibrated to 15 ^{0}C in the water bath. The outside of the bottle was again wiped dry and weighed as W_2 (Onyezeka *et al.* 2020).

The specific gravity of each sample was calculated using the expression below:

$$\text{Specific gravity at 15 °C} = \frac{W_2 - W_0}{W_1 - W_0}$$

Where; (W_2-W_0) = mass of sample; (W_1-W_0) = mass of an equal volume of water.

Determination of iodine value: Oil (0.2 g) was weighed in a dry 250 cm^3 conical flask and 10 cm^3 of chloroform was added and swirled until it dissolved. Wijs' solution (20 cm^3) was added and the stopper previously moistened with potassium iodide solution was inserted immediately, then swirled and allowed to stand in the dark for 30 min with occasional swirling. Any free iodine on the stopper was washed down using 10 cm^3 15 % (w/v) potassium iodide solution and thoroughly mixed with 100 cm^3 of freshly boiled and cooled distilled water. The mixture was swirled again

and titrated against 0.1 M sodium thiosulphate solution using starch indicator. The titration was continued until the blue colour just disappeared after shaking. The titre value was recorded as V_1. A blank determination was carried out with 100 cm^3 of chloroform (titre value = V_2) (Onyezeka *et al.* 2020). Iodine value was determined using the formula.

$$\text{Iodine value} = \frac{V_2 - V_1 \times C \times 12.69}{Weight\ of\ sample\ (g)}$$

Where; C = concentration of sodium thiosulphate solution used; V1 = volume of sodium thiosulphate solution used for sample; V2 = volume of sodium thiosulphate solution used for blank

Determination of kinematic viscosity: A Redwood viscometer was used to measure the kinematic viscosity of the samples. The determination of kinematic viscosity was carried out by introducing about 50 cm^3 of the sample into a clean dried viscosity tube. This was done by inverting the tube thinner arm into the sample and then using suction force to draw up the oil to the upper timing mark of the viscometer. The instrument measured the time of gravity flow in seconds of fixed volume of the fluid (50 cm^3) through specified orifice made in an agate piece. The viscometer was placed into a holder and inserted into a constant temperature bath set at 40 ^{0}C temperature and allowed for 30 minutes for the sample to come to the bath temperature. The suction force was then applied to the thinner arm to draw the sample slightly above the upper timing mark. The afflux time was recorded by timing the flow of the sample as it flowed freely from the upper timing mark to the lower timing mark. The kinematic viscosity (ν) was calculated by means of the equation below adapted from Dallatu *et al.* (2017).

$$V = C \times t$$

Where; V = Kinematic viscosity (mm^2/s); t = Time for of 50 cm^3 sample in seconds; C = viscosity tube constant (0.09757)

Cloud point analysis: The sample was filtered and about 50 cm^3 was placed in a conical flask and closed tightly with a cork carrying a test thermometer. The flask was heated to 130 ^{0}C and maintained at this temperature for 5 minutes to remove all moisture in the sample. The flask was cooled in a water bath filled with ice and stirred thoroughly to ensure temperature uniformity and to prevent super cooling and solidification of fat crystals at the side and at the bottom of the sample bottle. The flask was continuously monitored for cloud formation. To achieve this, the flask was removed periodically without disturbing the sample in order to inspect if the sample had turned cloudy. The cloud point was taken as the temperature at which the first crystals began to form in the normally clear fuel. The test was repeated with the temperature of the water bath set at 5 ^{0}C below the cloud point of fossil diesel. The temperature that corresponds to the first formation of a cloud in the fuel sample was recorded as the cloud point (Onimisi *et al.,* 2021). The results of repeated tests were recorded and the average value determined and presented.

Determination of pour point: The ASTM D-97 standard method was adopted for the pour point test. The sample (50 cm^3) was filtered and poured into a test jar to the level mark. The sample was heated in a water bath until it was just sufficiently fluid to pour into the test jar. The heated sample in the test jar was kept at room temperature for 24 hours before testing. The test jar was covered with the cork carrying the high-pour thermometer with its bulb immersed completely into the test sample and the beginning of the capillary was 3 mm below the surface of the sample. The sample was cooled inside a cooling bath to allow the formation of wax crystals and for every subsequent 3 ^{0}C, the test jar was removed and tilted to check for surface movement or flow. When the oil failed to flow when tilted, the jar was held horizontally for 5 seconds. If it failed to flow again, 3 ^{0}C was added to the corresponding temperature of the oil (Dallatu *et al.* 2017).

RESULTS AND DISCUSSION

The results of findings are as presented in this section. The implications of these findings were further discussed. The oil yield obtained was 45.62% which was similar to findings by Ogala *et al*. (2018) and Zang *et al*. (2018) who reported yields of range 35 % to 46 %. Also, Chapagain and Wiesman (2011) reported that the general yield of *B. aegyptiaca* range from 30 % to 50 % regardless of the extraction method used. The physicochemical properties of *B. aegyptiaca* oil (BAO) are presented in Table 1 while its fatty acid composition is as presented in Figure 1. Linoleic acid was the most common fatty acid present in the extracted oil while vaccenic and palmitic acids were also present in significant quantities (> 10 %). Linoleic acid has been reported to show anticarcinogenic, antiobese, antidiabetic and antihypertensive properties (Koba and Yanagita, 2014). Vaccenic acid has been reported to supress intestinal inflammation while palmitic acid has cardiovascular and anti-carcinogenic activities (Jacome-Sosa *et al.,* 2016; Fattore and Fanelli, 2013). This highlights the potential diverse uses and importance of *B. aegyptiaca*. This importance was also reported by Chothani and Vaghasiya (2011), Yadav

and Panghal (2010) and Al-Thobaiti and Zeid (2018) respectively. FTIR analysis of the extracted oil (Figure 2) revealed wavelengths of 3008.0 cm^{-1} associated with C-H stretching, 2922.8cm^{-1} to -OH (acid), 1744.4 cm^{-1} to C=O, 1461.1 cm^{-1} to C=C stretching, 1237.6 cm^{-1} to C-O, 1159.2 cm^{-1} to C-C and 723.1 cm^{-1} to C-H bending.

Table 1: Physicochemical properties of *B. aegyptiaca* seed oil

Specifications	Values
Yield (%)	45.62
Density (g/cm^3)	0.9
Viscosity (at 40 ^{0}C) (cSt)	6
Viscosity (at 100 ^{0}C) (cSt)	2.5
Cloud point (^{0}C)	21
Pour point (^{0}C)	9
Acid value (mg KOH/g)	3.64
Free fatty acid (%)	1.82
Iodine value (mgI_2/100g)	94.97
Saponification value (mg KOH/g)	180.34
Viscosity index	228.32

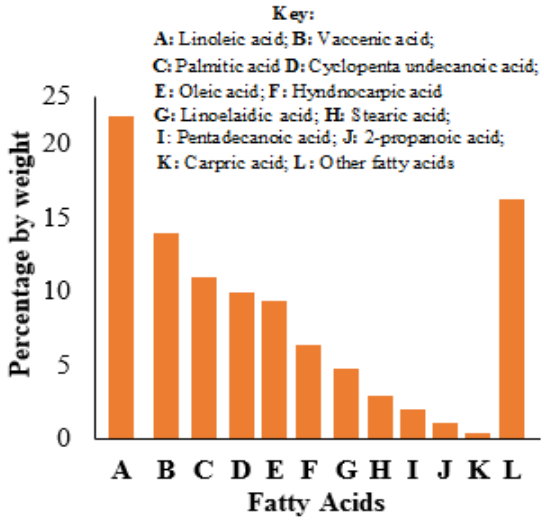

Fig 2: Fatty acid composition of *B. aegyptiaca* seed oil

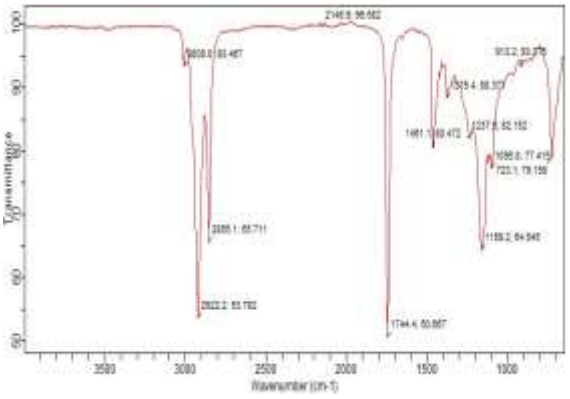

Fig 3: Fourier Transform Infrared Spectrum of *B. aegyptiaca seed* oil.

The physicochemical properties of the produced biodiesel are presented in Table 2. A 92 % biodiesel yield was obtained. The density of the biodiesel was 0.88 g/cm^3, this was observed to be comparable to that of fossil diesel fuel. The viscosity at 40 ^{0}C was also within the acceptable range for diesel fuels. The cloud and pour points were obtained to be 16 ^{0}C and 7 ^{0}C respectively. The acid value of the synthesized biodiesel was 1.57 mgKOH/g while the free fatty acid was 0.79 %. The iodine and saponification values of the biodiesel was 42 mg I_2 / 100 g and 176 mg KOH/g respectively. These values were similar to those of diesel fuels and similar to those reported in literature for biodiesel. For instance, Azeez *et al.* (2019) reported an acid value of 1.16 mgKOH/g, saponification value of 159.9 mg KOH/g and free fatty acid of 0.58 % for biodiesel prepared from *J. curcas* see oil. Similarly, Tutunea *et al.* (2019) reported values of 4.3 mm^2/s and 0.88g/cm^3 for the viscosity and density of biodiesel produced from sunflower seed oil. In addition, Dieng *et al.* (2019) synthesized biodiesel from rapeseed oil and reported a density of 0.91 g/cm^3, acid value of 2.7, iodine value of 113.5 mgI_2/100g and saponification value of 194.7 mg KOH/g respectively.

The methyl ester composition of *B. aegyptiaca* is presented on Figure 2. 9,12-Octadecadienoic acid methyl ester was the most predominant methyl ester accounting for over 40% of methyl esters. Hexadecanoic acid methyl ester, butyl-9,12-octadecadienoate and heptadecanoic acid-16-methyl ester were also present in significant quantities. Studies by Sokoto *et al.* (2011) revealed that diesel properties are directly related to the amount and composition of its methyl esters. Diesel properties such as oxidation stability, cetane number, iodine value and viscosity were correlated with the methyl ester composition and structural configuration. They also found that the cetane number and oxidation stability of the produced biodiesel is a function of the degree of unsaturation and long chain saturated factor.

The profile of methyl esters in the biodiesel produced indicates its likelihood to be a viable fuel source for internal combustion engines (Sokoto *et al.*, 2011). The diverse types and quantities of methyl esters present in the synthesized biodiesel suggests that it will be stable and have properties similar to those of standard biodiesels. The FTIR spectrum of the produced biodiesel is presented in figure 4. The FTIR analysis of *B. aegyptiaca* biodiesel (BAB) revealed wavelengths of 3008.0 cm^{-1} associated with C-H stretching, 2922.2cm^{-1} to -OH (acid), 1740.7 cm^{-1} to C=O, 1461.1 cm^{-1} to C=C stretching, 1244.9 cm^{-1} to C-O, 1196.5 cm^{-1} to C-C and 723.1 cm^{-1} to C-H bending. These wavelengths reemphasize the presence of methyl esters in the prepared biodiesel.

Table 2: Physicochemical properties of *B. aegyptiaca* biodiesel (BAB)

Specifications	BAB	ASTM VALUE	EN
Yield (%)	92	-	
Density (g/cm³)	0.88	0.86-0.9	0.86-0.90
Viscosity (at 40 ^{0}C)	4.2	1.9-6	3.5-5.0
Cloud point (^{0}C)	16	-	-
Pour point (^{0}C)	7	-	-
Acid value (mg KOH/g)	1.57	0.50	0.50
Free fatty acid (%)	0.79		
Iodine value (mgI_2/100g)	42	-	120
Saponification value (mg KOH/g)	176	-	-

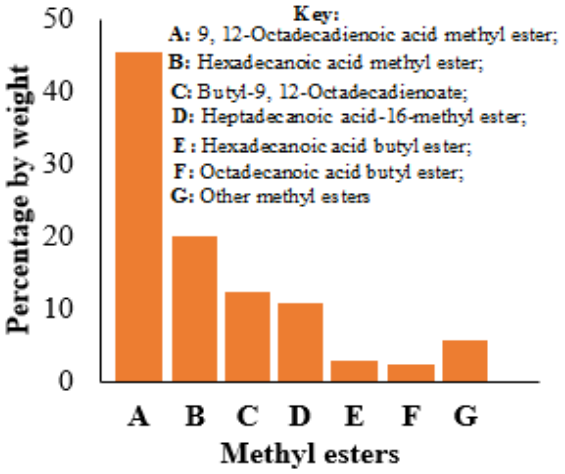

Fig 4: Methyl ester composition of *B. aegyptiaca* biodiesel

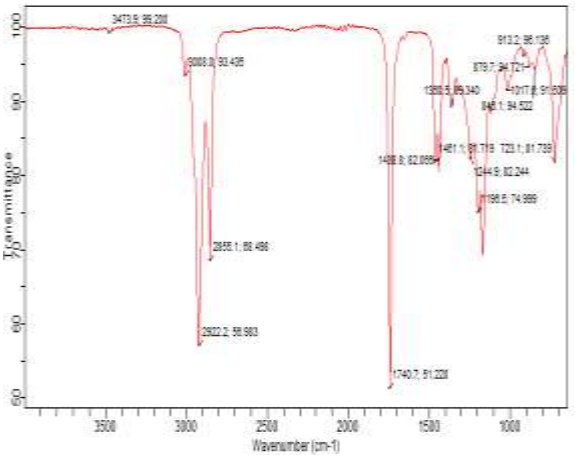

Fig 5: FTIR for *B. aegyptiaca* methyl ester

Conclusion: Fatty acid alkyl esters (biodiesel) was synthesized and characterized from Desert date (*B. aegyptiaca*) seed oil, and its physicochemical properties were determined. Its physicochemical properties were comparable to those of diesel and showed remarkable potentials to be used as an alternative to fossil-based diesel.

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