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VALORIZATION OF DATE KERNELS FOR THE PRODUCTION OF BIODIESEL

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Current Trends in Natural Sciences

Abstract
This work is part of the diversification of renewable energy sources, such as biomass for example. This research focuses on the recovery of date seeds which cannot be consumed by humans and are considered as waste. For this, the physicochemical and biochemical properties of these seeds were studied and determined. The results obtained allowed us to conclude that this waste product can be valorized for the production of biodiesel. Besides, the techniques adopted to achieve the best results in terms of quality were investigated as well. In addition, the process of extracting oils from date seeds was determined using the Soxhlet extraction procedure with a volatile solvent. The results obtained showed that the oil content of date seeds was estimated at around 4% to 5%, with a very high acid number (8.9776). This was achieved by means of the esterification and transesterification reactions, which allowed producing biodiesel. This method makes it possible to produce up to 91% of biodiesel with characteristics close to those reported in previous studies and recognized by international standards.

Keywords: biodiesel, date palms, date seeds, oil extraction, transesterification, valorization

1. INTRODUCTION

Over the last few, the increasingly damaging pollution of the environment and the growing consumption of energy across the world have been aggravated by gas emissions from the public transportation system and the growth of modernization and industrialization (Bibin et al., 2012). The use of fossil fuels is detrimental to the environment such as climate change and global warming (Dharma et al., 2016a). In addition, the accelerated warming of the Earth's core – from 1979 to 2016 – is evident through surface and satellite data. Alarmingly, as the rate of global warming seems to be accelerating with each passing decade, the future risks associated with climate change will be immense and, thus, will become more challenging to navigate (IPCC, 2021).
In order to remedy this problem, it was deemed necessary to diversify and increase the energy sources so as to achieve low levels of CO_2 emissions (Ajiskrishnan et al., 2015). On the other hand, it is widely acknowledged that the world may soon face the risk of depletion of fossil fuels due to their accelerated and irresponsible use. In addition, the probable depletion of petroleum resources in

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the near future and the increased socio-economic impacts of the environmental pollution have led to the development of sustainable and alternative fuels from cheap and environmentally friendly renewable energy sources. The growing demands for energy, the longer-term trend decline in the average oil production, and the growing concern over increasing environmental pollution have prompted researchers to find alternative renewable energy sources to use as a substitute for oil-based fuels (Azad et al., 2016; Bhuiya et al., 2016; Ong et al., 2020). These new energy sources must be sustainable and economically feasible for widespread adoption; they should also be able to meet the energy needs of the growing global population. The emissions resulting from the combustion of fossil fuels inside the engine are very harmful, human being and the environment (Siddartha,et al., 2022). Over the past few years, the production of biodiesel, which is derived from the transformation of biomass from non-fossil organic materials and is environmentally friendly, has attracted the attention of many researchers around the world (Cantin, 2010). Biodiesel, generally composed of mono-alkyl esters of fatty acids. It is non-toxic and biodegradable, making it the perfect fuel (Singaram, 2009). It should be noted that several factors must be taken into account when choosing the raw materials for biodiesel. These materials must be locally available, economically competitive, easily accessible and technically feasible (Anwar et al., 2018). It is worth recalling that the non-edible oil seeds have widely been recognized as an interesting raw material for the production on second generation biodiesel (Chen et al., 2018). Biodiesel synthesis from vegetable oil is usually accomplished via the most efficient technology called the transesterification process (Bhuiya et al., 2020). Transesterification is regarded as the best method among other approaches due to its low cost and simplicity (Atabani et al., 2012). Several researchers around the world are currently working on the diversification of renewable energy sources.

Over the years, the Algerian phoenicultural sector has made significant progress, with a potential that has reached 18 million palm trees planted over an area of 350 hectares. More than 11 million trees are already productive, with a very important harvest of around 492 thousand tons of dates. According to statistics from the Food and Agriculture Organization (FAO, 2015), Algeria is ranked fourth producer of dates worldwide. It is important to mention that the region of Adrar produces a significant quantity of dates, estimated at about 935 thousand quintals per year (DSA, 2020). This region is famous for its 305 varieties of dates (Benkhedda, 2014). In addition, the significant production of date seeds of low market value in the Adrar region is estimated at more than 1 177 quintals per year. This quantity is very important and is sufficient to produce a biomass that can be energetically valorized. The reasons for our choice for date pits for the production of biodiesel are based on the following criteria: 1) Date pits are non-recovered waste except as an animal feed source, and 2) They contain a high level of fat. It should be noted that dates are widely consumed in our society, which generates a huge amount of date seeds. Therefore, it would be urgent to launch a project for the valorization of this product to produce bioenergy, given the large amount of oil they contain. The present work aims to produce biodiesel as an alternative fuel by the direct transesterification process or through the esterification reaction of the oils extracted from date seeds by following a transesterification reaction, depending on the value of the acid number of the vegetable oils used.

2. MATERIALS AND METHODS

2.1. Sampling

The date seed samples were collected from the region of Mraguen, one of the ksours in the north of the Wilaya (Province) of Adrar (Fig. 1). Mraguen is located 12 km from the center of the city of

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Adrar. The production of dates in that region is very important. Unfortunately, these dates are of low market value.

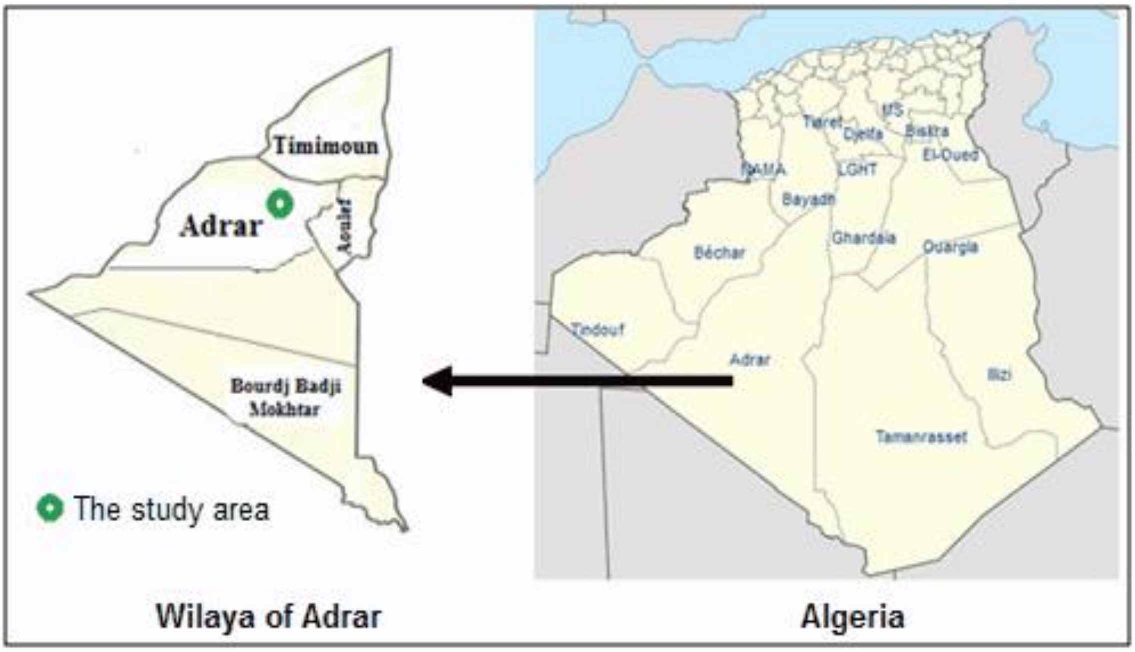

Figure 1. Presentation of the sampling site

2.2. Tonnage calculation

Determination of the weight of a date seed

The weight of a date seed was determined by considering 20 dates, taken at random, from each variety. The method for this operation was to find:

• the dimension of the dates and their seeds (length and width), using a caliper with an accuracy of ± 0.1 cm.

• the weight of the dates, their pulps and seeds, using a precision analytical balance with an accuracy of ± 0.001 g.

Tonnage of date seeds

According to data from the Directorate of Agricultural Services, the region of Adrar annually produces a significant tonnage of dates, about 935 thousand quintals per year. The tonnage of date seeds with low market value can then be calculated as follows:

$$\mathbf{WKi} = \frac{\text{The average weight of of 20 date kernels*100}}{\text{The average average weight of 20 dates}}$$

$$\mathbf{WK} = \frac{\sum_{i=1}^{5} \text{WKi}}{5} \qquad \mathbf{PTK} = \frac{\text{WK} \,.\, 100}{\text{PTD}}$$

WKi: The ratio of the average weight of 20 date kernels*100 over the average weight of 20 dates, for a given variety;
WK: Means of WKi;
PTK: Production tonnage of date kernels in quintals per year;
PTD: Production tonnage of dates in quintals per year.

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2.3. Preparation of date seeds

The various stages of this study were carried out in the laboratories of the University of Adrar and the laboratories of the Renewable Energy Research Unit (URER). The seeds under study came from five different varieties of dates. After pitting the dates (Fig. 2), the seeds were dried at 50°C, for a period of 48 hours, and then crushed using an electric grinder to obtain a powder of sufficiently fine particle size. Note that the preparation of date seeds included the following steps:

- ***separation of the pulp from the seed***: it is a simple and easy manual operation.
- ***washing:*** the seeds were washed with hot water to remove traces of pulp and all kinds of impurities that stick to them.
- ***drying***: after washing, these seeds were placed in an oven brought to the temperature of 50°C, for 48 hours, in order to facilitate grinding.
- ***grinding***: the grinding was carried out by means of an electric grinder in order to have small fragments that were subsequently crushed and blended using an electric mixer (Fig. 3).
- ***drying***: the powder obtained after grinding was dried in an oven at a temperature of 105°C for 24 hours, then stored in airtight plastic jars.

Figure 2. Pitting dates

Figure 3. Grinding of date seeds

Extracting date seed oil

A Soxhlet apparatus was utilized to extract the oil from the date seeds following a solid-liquid extraction procedure. In this operation, 300 ml of hexane were poured into a flask and 70 grams of ground material was introduced into the filter paper cartridge which was then placed inside the Soxhlet apparatus. Afterwards, the flask was heated and the solvent vaporized and passed through the adduction tube where it was condensed by the condenser and then flowed into the cartridge and dissolved the product which was supposed to be extracted from the solid. When the Soxhlet device was full, the solution siphoned and returned to the flask. The solvent was concentrated in the desired product, while the starting solid was depleted. This cycle was repeated for 6 hours, until exhaustion. This extraction process was based on the following principle. Non-polar compounds, such as fatty substances, are insoluble in polar compounds, such as water, but are soluble in non-polar solvents such as hexane. The evaporation temperature of hexane is lower than that of the fats to be extracted, so it is very easy to separate them through heating.

Biodiesel synthesis protocol

Due to the high acid number of their oil (IA = 8.97 mg KOH/g of oil), and given their low volatility, date seeds contain an excessive amount of polyunsaturated free fatty acids (FFA) which cause unwanted soap formation. Therefore, these polyunsaturated free fatty acids decrease the biodiesel yield during the transesterification process (Ong et al., 2014). Many researchers have used both

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esterification and transesterification procedures to produce methyl ester from crude oil with a high FFA content (Ajala et al., 2017). Moreover, several studies were carried out to optimize the process parameters, using different stocks of second generation biodiesel through esterification and transesterification reactions (Tshizanga et al., 2017; Yatish et al., 2018). In this work, the biodiesel production process was carried out in two stages.

Esterification

The esterification reaction was carried out in a 250 ml flask, equipped with a reflux condenser and a magnetic stirrer (Fig. 4). The mixture, containing 39.787 g of date seed oil, 19.893 g of methanol and 0.397 g of sulfuric acid, was stirred for 3 hours. In order to prevent the formation of unwanted products, like soap, several procedures, such as pretreatment with crude oil, refining the raw material, and carrying out the esterification process with an acid catalyst etc., were successfully adopted in order to decrease the percentage content of free fatty acids (Chattopadhyay and Sen, 2013). It is worth noting that the pre-treatment step is necessary to significantly reduce the FFA percentage content (less than 1%) in crude oil before the transesterification process is carried out (Dharma et al., 2016b; Boulal et al., 2016). After decantation of the product obtained, the glycerin was removed from the mixture. Then the remaining crude ester was neutralized and washed three times with distilled water. Phase separation tests were carried out by gravity in a separating funnel. Finally, the esterified oil was recovered after evaporation of methanol.

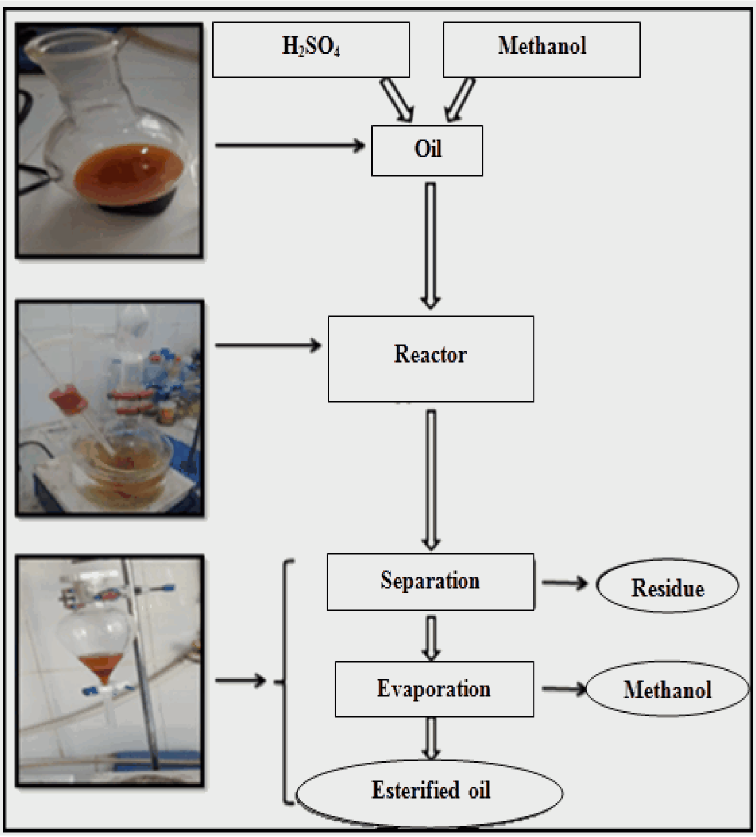

Figure 4: Steps in the esterification process of date seed oil

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Transesterification

In a 100 ml beaker, equipped with a condenser and a thermometer, we put 21.0839 g heated to 70° C. and subjected to magnetic stirring (Fig. 5). When the temperature is stable, we add a pre-prepared solution according to the molar mass of the oil containing 6.325 g of methanol and 0.212 KOH to the oil with stirring (Boulal et al., 2019). It is important to know that the production of biodiesel using alkaline catalyzed transesterification with a high FFA content is extremely difficult as there is a huge tendency to react with the free fatty acids (FFAs) in the presence of the alkaline catalysts (Dhar and Kevin, 2012).

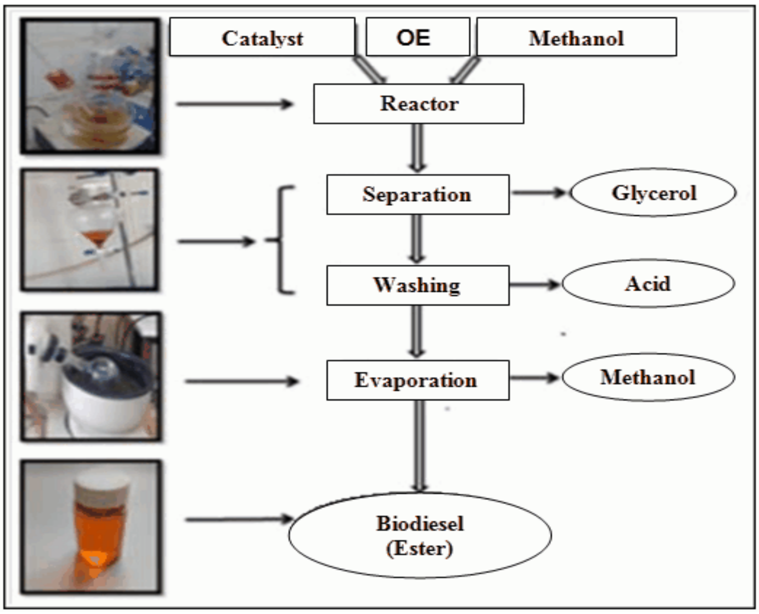

Figure 5. Steps in the oil transesterification of date stones

Biodiesel recovery

At the end of the reaction, the product of the transesterification reaction was transferred into a separating funnel where it remained for 24 hours in order to separate the glycerol from biodiesel. The dark brown glycerol was then collected in a bottle and stored, while the biodiesel was washed with distilled water.

Biodiesel yield

The reaction yield may be calculated using the following formula:

$$\%\ \text{yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

2.4. Methods of analysis

Biochemical analyses of date seeds

Determination of water content and oil content according to NF V 03-921, 1967 and respectively NF EN ISO 734-1, 2000.

Characterization of date seed oil

The extracted oil was characterized by measuring its refractive index, density, viscosity and acid index, using the infrared spectroscopy technique.

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- physical parameters
  - the refractive index (AFNOR T 60-212)
  - density (ASTM D941-55)
  - viscosity (ASTM D445)
  - infrared (IR) absorption spectroscopy (Type IR CARY 660 FTIR ATR)
- chemical parameters: acid index and acidity according to NF EN ISO 660, 1999

Characterization of biodiesel

The analyses carried out aimed to determine the parameters such as the flash point (using ASTM D92 method), pour point (using ASTM D97 method) and calorific value (according to ISO 1928: 2009). In addition, other properties, such as viscosity, density and refractive index, were also determined by the same methods mentioned above.

3. RESULTS AND DISCUSSIONS

Morphological characteristics

The detailed results concerning the morphological characteristics of the five varieties of dates studied, with their pulps and seeds, are given in the Table 1.

Table 1. Morphological characteristics of dates, pulps and seeds

Averages	Aghmmou	Hmira	Takerboucht	Tinaceur	Tegazza
Date weight (g)	6.9223	4.769035	10.16349	6.679955	9.79796
Pulp weight (g)	6.2636	4.102065	9.104165	5.878215	8.78389
Seed weight (g)	0.64651	0.664675	0.992085	0.79299	0.99343
Date diameter (cm)	1.9025	1.695	2.608	1.873	2.1975
Seed diameter (cm)	0.741	0.6995	0.857	0.7335	0.8165
Date length (cm)	3.93	3.144	3.529	3.922	4.33
Seed length (cm)	1.9605	1.9885	2.2425	2.2455	2.313

Table 1 shows us that the weight of the date kernel varies according to the variety. The production of tonnage of date seeds with low market value in Adrar is then 1.177.507 thousand quintals per year, this value is very important and sufficient for a biomass that will be transformed into energy

Biochemical composition of date seed powder

The biochemical characteristics of date seed powder are presented in the Table 2.

Water content

The water content of the date seed powder was 5.722%; it is very close to that found by Al-Farsi et al. (2007) for the date variety Um-sallah (5.19%).

Fat content

The yield of fat obtained by hot extraction was found equal to 4.773%, which is comparable to the value found by Hashim and Khalil (2015) in the interval (5 – 6%) in a study they conducted on Tunisian date varieties (Mabsili, Um-Sallah, Shahal), and the value of 4% reported by Idir (2016). However, it should be noted that this percentage is relatively low compared to that reported by Hamadaa et al. (2002) and which was within the range extending from 8.7 to 12.3%. Therefore, the

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value obtained can be considered as acceptable in comparison with the one found in the work done previously.

Table 2. Biochemical index values of date seed powder

Constituent	**Average values (%)**
Water content	5.722
Fat	4.773

Characteristics of date seed oil

Color and flavor of date seed oil

Visual observation (Fig. 6) revealed an orange color, with quite pleasant smell and flavor. Note also that this oil is quite fluid at room temperature.

Figure 6. Oil from the powder of date seeds

Physicochemical characteristics of date seed oil

Table 3 summarizes the physical and chemical indices of the oil extracted from the powder of the date seeds.

Table 3. The physicochemical characteristics of date seed oil

Parameters	**Average content**
Refraction index at 40°C	1.47
Density at 40°C	0.9132
Viscosity at 20°C	387.54
Viscosity at 40°C	82.878
Acid value	8.9776

Refractive index

The refractive index obtained at 40°C was 1.47. This value is very close to those stated by the Moroccan standard (NM 08.5.90) for which the refractive indices oscillate between 1.4630 and 1.4720 (Kouidri, 2008). Therefore, one may say that this is an acceptable value compared with those reported in works previously carried out.

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Density

The density found in the present study was of the order of 0.9132 g/m^3 which is comparable to the value of 0.9116 g/m^3 found by Rehab et al. (2012). The date seed oil had a density close to that of olive oil, as given by the Codex Alimentarius, in the range extending from 910 kg/m^3 to 916 kg/m^3.

Viscosity

The viscosity of the oil was measured at two temperatures, i.e. 20°C and 40°C. It was noted that this viscosity decreases with temperature (387.54 mm^2/s - 82.878 mm^2/s). The value found at 20°C (448.14 mm^2/s) was slightly lower than those found by Alloune et al. (2012). This finding is encouraging for the production of biodiesel.

Infrared absorption spectroscopy

Fourier transform infrared spectroscopy or FTIR spectroscopy is based on the absorption of energy in the infrared spectral range which extends from 400 cm^{-1} to 4000 cm^{-1}. Each absorption band is associated with a type of functional group (Fig. 7).

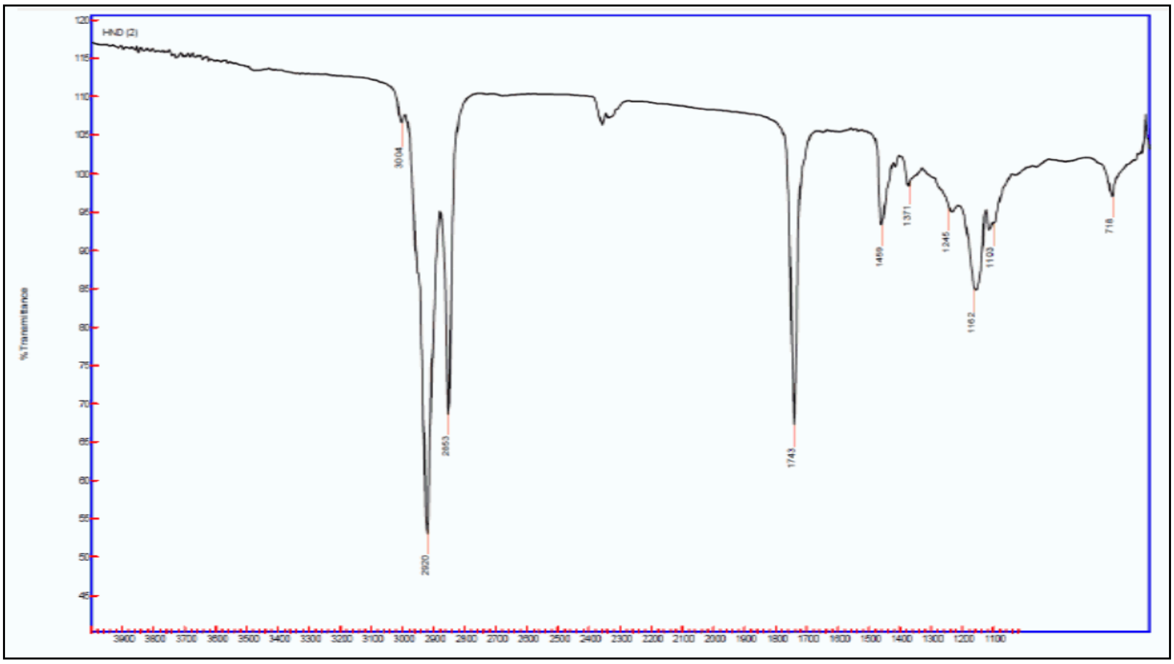

Figure 7. IR spectrum of oil from date seeds

Several absorption peaks, corresponding to various functional groups, were observed in the spectrum obtained. These functional groups are often responsible for the bonds between the adsorbent and the adsorbate.

- *C – H vibrational mode*. The bands observed in the region from 3050 cm^{-1} to 2800 cm^{-1} were attributable to the C – H bond stretching vibrations of the CH_2 and CH_3 groups. The three bands, observed at 2853 cm^{-1}, 2853 cm^{-1} and 3004 cm^{-1}, were attributed, respectively, to asymmetric and symmetrical stretching vibrations of the CH_2 group.
- *= C-H vibrational mode*. A low intensity band was observed at 1459 cm^{-1}. This band is due to the cis stretching vibration of the = C – H group.

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- *C = O vibrational mode*. The most intense band of the spectra was observed at 1743 cm^{-1}. This band is attributable to the stretching vibration of the C = O group, characteristic of esters.
- *C – O vibrational mode*. The low frequency region often contains low intensity bands. Three bands were clearly seen at 1103 cm^{-1}, 1162 cm^{-1}, and 1245 cm^{-1}. These bands are attributable to the stretching vibration of the C – O group of esters within an interval between 1000 cm^{-1} and 1300 cm^{-1}.
- *The (-CH_2-) n groups*. They characterize the long aliphatic chains of fatty acids, with a band at 718 cm^{-1}. The spectral analysis allowed concluding that date seed oil contains esters and aliphatic chains.

Acidity and acidity index

The results obtained showed the existence of a large number of acids (8.9776 mg KOH/g). Converting oil to biodiesel requires two consecutive processes, namely esterification and transesterification. The acidity (% oleic acid) of date seed oil (4.48% oleic acid) was found higher than the values found in three varieties of olive oil, namely Picual (0.15% oleic acid), Hojiblanca (0.20% oleic acid), according to a study carried out by Gutiérrez et al. (2002).

Characteristic of the biodiesel obtained

The biodiesels synthesized in the present study exhibited properties similar to those recommended by international standards (Standard ASTM D6751). Table 4 summarizes the results of the comparative study of synthesized biodiesel. It should be noted that viscosity remains very high.

Table 4. Physicochemical characteristics of biodiesel

Properties	**Biodiesel produced**	**ASTM D6751 Standard for biodiesel**
Viscosity at 40°C ($mm^2.s^{-1}$)	21.01	1.9 – 6
Calorific power (Heat value) (MJ/kg)	27.432	-
Flash point (°C)	170	≥ 93
Flow point (°C)	-6	-12 -7
Distillation (°C)	356	≤ 360
Density at 15°C (g/m^3)	0.8707	0.86 - 0.90
Refractive index	1.441	-

Biodiesel yield

Yield is a way to assess the extent to which the process of converting oil into biodiesel is successful. In this study, the oil conversion rate of date seeds (91%) was lower than the value (95%) found by Tarabet (2012) for the conversion of eucalyptus oil. However, it is much higher than the biodiesel yield from used vegetable oils (71%) found by Boulal et al. (2019). These results are close to those found in previous research studies.

Flash point

The flash point is the lowest temperature at which oil gives off enough vapor to ignite and begin to burn in the presence of a flame. If the heat source is removed, ignition stops, but not to support combustion. The flash point for biodiesel produced from date seed oil is 170°C. This value is in

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accordance with the standard value of biodiesel. It is higher than that of commercial diesel which is between 59°C and 96°C. It is therefore necessary to take safety measures during the storage, handling and handling of biodiesels synthesized from date seed oil.

Pour point

The pour point is the temperature at which the paraffin in fuel crystallizes to such an extent that the fuel freezes and no longer flows. The pour point of our biodiesel was found equal to -6°C. This value is close to the pour point of local commercial diesel which varies depending on the climate, between -15°C and -7°C. This result is of paramount importance because the pour point is very important when transporting biodiesel, especially in cold countries.

Refractive index

The value of the refractive index (1.441) was measured using a refractometer at a temperature of 40°C. This value is lower than that of date seed oils. This is certainly due to the change in the structure of oil and biodiesel.

Calorific power

The calorific value is defined as the energy released in the form of heat during the combustion reaction in the presence of oxygen. It is also the amount of energy released when one kilogram of fuel is burned.

The calorific value of biodiesel was found to be 27,432 MJ/kg; it is lower than that of conventional diesel whose value is 45.4 MJ/kg (Alloune et al., 2012). This is an encouraging result because a high calorific value has a positive influence on the combustibility of biodiesel.

Viscosity

Biodiesel obtained from date seed oil has a viscosity equal to 21.01 mm^2/s which is higher than the value recommended by ASTM D445 Standards (9 mm^2/s). In addition, the value of 11.13 mm^2/s found for biodiesel produced from olive-pomace oil is lower than that of diesel (41 mm^2/s) found by Chapuis et al. (2013). This finding is quite interesting because until now the problem of too high viscosities is still a serious concern to researchers throughout the world. Therefore, the results achieved by Boulal et al. (2019) are quite interesting.

It is worth noting that a high viscosity can cause practical problems when supplying engines with fuel (pressure at the injection pump, hose diameter too small) and for injection (too large droplets cause poor combustion and lead to unburned exhaust gases). On the other hand, a too low viscosity can cause leaks in the injector with a high pressure drop in the injection pump (Selaimia et al., 2018).

Density

The density values of the biodiesel produced are equal to 0.8777 g/cm^3 and 0.8707 g/cm^3 at the temperatures 20°C and 40°C, respectively. These values are almost identical to that recommended by the European Standards DIN EN 14214, between 0.86 g/cm^3 and 0.9 g/cm^3. In addition, Bettahar et al. (2016) found out that the density values of biodiesels are lower than those of oils.

4. CONCLUSIONS

The present study confirmed that the valorization of date seeds of low market value, from the Wilaya (Province) of Adrar with a view to their possible transformation into biodiesel, is possible. Also, it was found that the substrate obtained contains fat whose transformation into industrial products seems promising. The findings provided interesting insights about using this raw material for the production of oil from date seeds. Some biodiesel production methods were performed and the physicochemical properties of date seed oil were identified. Note that it is highly important to

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select the most appropriate raw materials for the production of biodiesel as this choice can reduce the total cost of production by two thirds.
Furthermore, methyl esters of vegetable oils (MEVO) obtained through the esterification and transesterification reactions (Ajala et al., 2017) exhibited a yield of 91%, under the operating conditions. The physical and chemical properties of the biodiesel from date seed oil were quite interesting, except for the density which was equal to 0.8777 g/cm^3. Also, the kinematic viscosity found in this work (41 mm^2/s) was lower than that found by Boulal et al. (2019) and Alloune et al. (2012). This finding turned out to be very interesting because the problem posed so far concerning the production of biodiesel is its very high viscosity value. Other analyses may provide additional information on the biodiesel produced. It would therefore be interesting to carry out other studies in order to determine additional physicochemical characteristics of biodiesel, on the one hand, and to think about applying this production process at an industrial scale.

5. ACKNOWLEDGEMENTS

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