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Study of biodiesel produced using a heterogeneous catalyst from *Balanites aegyptiaca* in southwestern Algeria

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Abstract:
Over the last few decades, a large number of researchers have focused on the diversification of energy sources, and particularly renewable energy sources. It has been revealed that the *Balanites Aegyptiaca* (desert date) plant exists in several places around the world, particularly in Asia and Africa. This tree is widely present in the south of Algeria, and more precisely in the Wilayas of Adrar, Bordj-Badji Mokhtar and Tamanrasset. For this reason, it was decided to study the possibility of using it in the production of biodiesel. In this work, biodiesel was produced from the oil of *Balanites Aegyptiaca* fruit. Indeed, this oil was extracted by the Soxhlet extraction method using bioethanol as a solvent. The yield obtained reached 35.45%. As for the production of biodiesel by the transesterification reaction, it consists of adding ethanol or methanol and a heterogeneous catalyst from the shell of the *Balanites Aegyptiaca* fruit to the extracted oil. The yield obtained in this case was equal to 92.2%. Then, the biodiesel produced was characterized in order to determine its physic- chemical parameters such as the density, viscosity, refractive index, pour point and flash point. The values obtained for the characteristics of the biodiesel produced were found to be very close to those recommended by the ASTM standards

1. Introduction

Industrial development necessitates increased energy demand, primarily met by hydrocarbons. However, their large-scale exploitation raises issues of high costs and impending depletion, with peak production anticipated soon [1,2]. Additionally, fossil fuel combustion contributes significantly to greenhouse gas emissions, leading to global warming and detrimental ecological impacts [3,4].

Biomass energy offers a sustainable alternative, with biofuels like biodiesel emerging as viable solutions to energy and environmental challenges [5]. Biodiesel, produced from natural oils and fats, is an attractive renewable fuel due to its economic and ecological benefits [6]. Traditional biodiesel production often relies on basic or heterogeneous catalysts [7,8,9].

Homogeneous catalysts possess several characteristics that make them effective for biodiesel production. However, their separation is often challenging, and in large-scale operations, managing and assembling these catalysts can be complicated [10].

Heterogeneous catalysts possess several advantageous qualities that make them suitable for biodiesel production. They can be easily separated from the products, are non-corrosive, and are environmentally friendly. Additionally, they can be regenerated for reuse [11].

Balanites aegyptiaca, a drought-resistant tree found in the Sahelian zone, produces oil-rich fruits and holds significant ecological value. Given the growing demand for fossil fuels, this research explores the potential of *Balanites aegyptiaca* kernels as an innovative, environmentally friendly biodiesel source, addressing gaps in existing studies and contributing to sustainable energy solutions in Algeria [12,13].

This study aims to use bioethanol as an organic solvent for oil extraction instead of hexane. The bioethanol is derived from *Balanites aegyptiaca* fruit, with the goal of reducing the cost of biodiesel production.

2. MATERIALS AND METHODS

- **Plant material**

The *Balanites aegyptiaca* (Figure 1) fruits were selected for the production of biodiesel because this plant is widely available in our country and the kernel of the fruit of this tree contains a high level of fat.

Figure 1. *Balanites aegyptiaca* tree

- **Sampling locations**

The fruit harvest was carried out in March 2021, in the region of Timiaouin, a municipality that is located south of Adrar (extreme south of Algeria), east of the great Sahara (Tanezrouft). Timiaouin is 150 km south-west of Bordj Badji Mokhtar, 1000 km south-east of Adrar, 465 km south-west of Tamanrasset and 1820 km south of the capital Algiers. The region of Timiaouin is an important producer of desert date palms (*Balanites aegyptiaca*). The present study was carried out at the bioconversion laboratory of the renewable energy research unit (URER/MS) in the city of Adrar.

- **Materials and experimental setup**

The materials used in this study are methanol (CH_3OH, 99.5%), absolute methanol, ethanol (96%), phenolphthalein (1%), potassium hydroxide (KOH,85%), bioethanol from *Balanites Aegyptiaca* fruit pulp, as well as kernel and fruit shell of *Balanites aegyptiaca*.

Figure 2 illustrates the main products obtained from *Balanites aegyptiaca* fruit in the field of bioenergy.

The bioethanol used was obtained by fermentation of the *Balanites aegyptiaca* fruits according to the method described by Boulal et al. (2019) [14] and also based on the study that was carried out by Hadri et al. (2022) [15].

To obtain the heterogeneous catalyst, we first clean and wash the grain hulls, then dry and grind them. They are then burned in an oven at a temperature of 800°C.

The oil is extracted from the kernels of *Balanites aegyptiaca* fruit using a Soxhlet apparatus with bioethanol as the organic solvent for extraction.

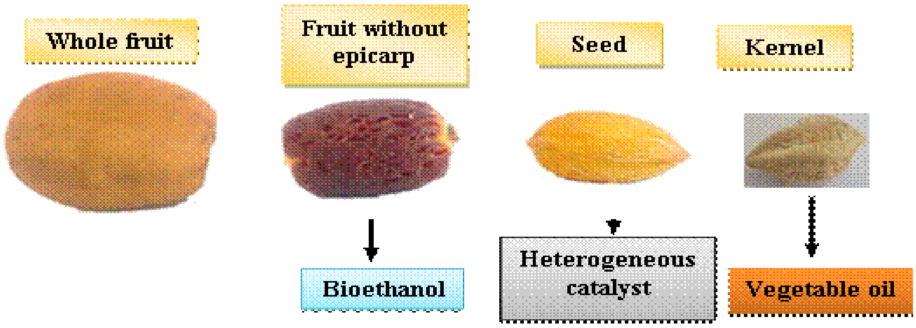

Figure 2. Main Extracts Obtained from Balanites aegyptiaca fruit

- **Soxhlet extraction**

The process of oil extraction involves several phases. The seeds of *Balanites aegyptiaca* are first collected and cleaned, and then dried in an oven at a temperature of 105°C. Oil extraction from the seeds of *Balanites aegyptiaca* is carried out using the Soxhlet extraction apparatus. The first step consists in pouring 300 ml of bioethanol into a round bottom flask. Next, 10 g of *Balanites aegyptiaca* kernels are crushed, roasted, and placed in a paper filter cartridge.

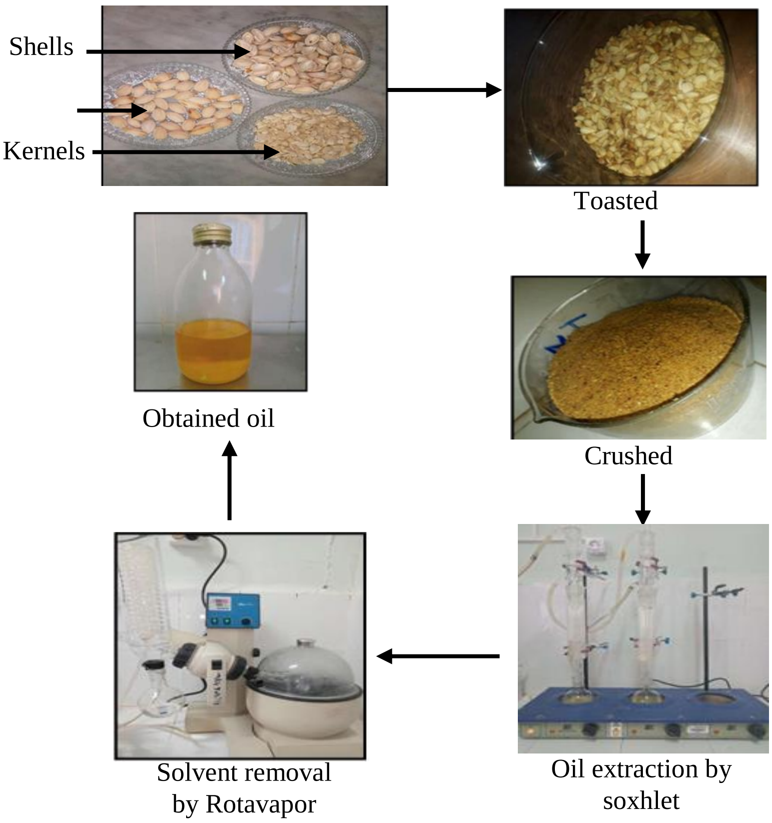

Figure 3. Diagram of oil extraction from *Balanites aegyptiaca* seeds.

The resulting product is then introduced into the center of the extractor. The extraction operation is repeated for 6 hours. Afterwards, the resulting mixture, which contains oil and bioethanol, is distilled using a rotary evaporator or rotavapor to obtain pure oil. The amount of extracted oil is then weighed to determine the oil content, and finally stored in a glass bottle for later use for biodiesel production. The different stages of oil extraction process are illustrated in Figure 3.

The acid value (AV) was determined based on the following equation 1:

$$AV(mgKOH/g\ of oil) = \frac{V \times N \times 56.1}{W} \qquad (1)$$

V:Volume (ml)

N : KOH normality

W : Oil Weight

- **Biodiesel production protocol**

a- Transesterification process of *Balanites Aegyptiaca* oil

The criteria for using the one-step or two-step transesterification process depend on the free fatty acid (FFA) content. If the percentage content of FFA is less than 3%, then the one-step transesterification process, with a basic catalyst, is possible. However, if the FFA content exceeds 3%, then the two-step transesterification process is preferred for the production of biodiesel [16].

In the present study, the oil acid index of *Balanites Aegyptiaca* Ia is equal to 1.15 mg KOH/g of oil, which corresponds to an FFA content equal to 0.57%. Therefore, a transesterification treatment step is sufficient to have biodiesel. The experiments were carried out at a reaction temperature equal to 60°C, with a molar ratio (methanol / oil) = (9/1), a concentration of heterogeneous catalyst from the shell of Balanites seeds equal to 7 % by weight. It should be noted that this catalyst was obtained by calcination at 800°C [17], with a reaction time fixed at 60 minutes [18]. Next, the oil, methanol, and catalyst were introduced into a batch reactor and heatedto the required temperature. The stirrer speed was maintained at 500 rpm. At the end of the reaction,the resulting mixture was centrifuged at a speed of 3000 rpm. After the centrifugation step, three phases were obtained: the upper phase representing the biodiesel, the middle phase representing the glycerol, and the lower phase the catalyst. It should be mentioned that the excess alcohol present in the biodiesel was evaporated by a rotary evaporator (rotavapor), at a mild temperature and under a moderate vacuum [19].

The process flow chart of biodiesel production is illustrated in Figure 4. The yield (in percent) of biodiesel can be calculated using Equation 1given below:

$$\text{Biodiesel yield (\%): } Y=100\times GMEP/GOUR \qquad (2)$$

GMEP = grams of methyl ester produced

GOUR = grams of oil used in reaction

b- Characterization of the raw material

The raw material, i.e. Balanites Aegyptiaca seeds, was characterized by calculating its water content according to Standard NF V 03-921, fat content according to Standard NF EN ISO 734-1 (2000), and ash content according to the French Association for Standardization (AFNOR V 03-922).

c- Characterization of Balanites Aegyptiaca oil

The physico-chemical properties of *Balanites aegyptiaca* oil to be determined are the acid index (AFNOR T60 204), density (AFNOR T 60 214), refractive index (AFNOR T 60 212), kinematic viscosity (ISO 3104, October 1994), and saponification index (AFNOR T 60 206, 1990).

d- Characterization of the biodiesel

This characterization consisted in determining the kinematic viscosity according to Standard ISO 3104 (October 1994), the density according to the French Association for Standardization (AFNOR T 60 214), the flash point according to Standard ASTM D93, the pour point according to Standard ASTM D97, the refractive index according to Standard AFNOR T 60 212, the cetane number according to ASTM D 976, and finally the sulfur content according to ASTM D4294.

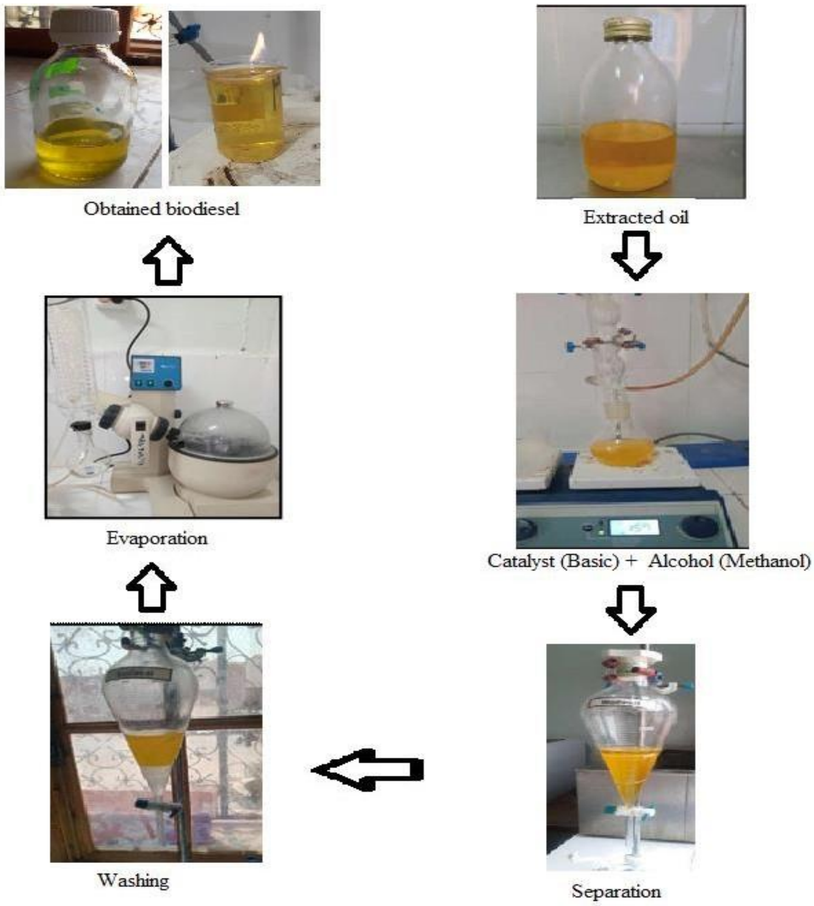

Figure 4. Biodiesel production flowchar

3. RESULTS AND DISCUSSION

- **Determination of some biochemical parameters of *Balanites aegyptiaca* seeds**

The biochemical composition of these seeds is shown in Table 1.

- **Plant material**

The *Balanites aegyptiaca* fruits were selected for the production of biodiesel because this plant is widely available in our country and because the kernel of the fruit of this tree contains a highlevel of fat.

TABLE 1. Biochemical composition of *Balanites aegyptiaca* seeds

Humidity (moisture)	6.3
Ash	7.23
Fat	48.52
Dry matter	92.932

Oil seeds have water contents varying between 3% and 9%, depending on the species and variety. The water content of *Balanites aegyptiaca* seeds was found equal to 6.3%. This value, which is relatively low, can still be lowered even further. Note that water is responsible for the alteration reactions of oil. Low water content ensures good storage of the seeds. Several studies have shown that when the water content decreases, a better oil yield is obtained [20].

- **Physico-chemical analysis of *Balanites aegyptiaca* oil**

The results obtained from the physico-chemical analyses are summarized in Table 2.

TABLE 2. Physico-chemical characteristics of *Balanites aegyptiaca* oil

Density at 40°C	0.916
Viscosity (mm^2/s) at 40°C	30.875
Refractive index at 20 °C	1.4688
Acid index (mgKOH/g)	1.15
Saponification index	308.55
Extraction yeild (%)	35.45

The refractive index R of oils varies with the degree of unsaturation of the molecules constituting oil. This degree of unsaturation is related to the number of unsaturated bonds in a molecule. The refractive index increases with the degree of unsaturation of fatty acids contained in fat [21].

The data reported in Table 3 indicate that *Balanites aegyptiaca* oil has a refractive index close to that of olive oil.

TABLE 3. Refractive indices of some oils

Oils	**Refractive index**
Balanites Aegyptiaca	**1.4688**
Argan	1.4711
Coloquint	1.474
Olive	1.466 - 1.468

The viscosity value found at 40°C is slightly lower than that of the oil extracted from the powder of date pits; it is approximately equal to 30.875 mm^2/s.

Furthermore, one should also know that the acid number characterizes the purity and stability of oil at room temperature. It defines the quality of the oil as well. It is worth emphasizing that the acidity of oil is a parameter that has a very high commercial importance. The acid index of the oil extracted from the *Balanites Aegyptiaca* fruit is close to that of castor oil; it is equal to 1.15 mgKOH/g of oil.

-Low acidity index (less than 4 mg KOH/g): High quality, stable oil, less prone to oxidation, suitable for cooking and cosmetic manufacturing.

- Moderate acidity index (4 - 10 mg KOH/g): Average quality, the oil may have undergone some decline but remains usable. Should be avoided in sensitive applications.

- High acidity index (more than 10 mg KOH/g): Low quality, unstable oil, prone to oxidation and degradation. Should be avoided, especially for cooking.

In conclusion, the acidity index of Balanites Aegyptiaca (1.15 mg KOH/g) indicates a high-quality and stable oil, making it an ideal choice for various uses and reflecting good stability compared to other oils like castor oil [22].

Moreover, it is important to recall that the saponification index corresponds to the mass of potassium hydroxide (KOH) that is necessary to neutralize the fatty acids in the oil. It was found that the saponification index of *Balanites Aegyptiaca* oil is higher than that of other oils, like for example soybean oil whose index varies between 184 and 195 KOH/g. This parameter can be employed in calculating the average molar mass of the fatty acid constituting the oil.

- **Characterization of the biodiesel produced**

Table 4 presents the physico-chemical properties of biodiesel obtained from Balanites oil. It is important to note that the properties of this biodiesel are in accordance with the standards set by the ASTM, except for the cetane number which is a little lower than the standard value.

In addition, the value of the higher heating value (HHV) or higher calorific value (HCV) of the synthesized methyl esters was measured and was found equal to 25.2 MJ/kg. This value is close to that of mastic oil which is 39 MJ/kg. In view of the expected energy performance in the case of our biodiesel, it can be said that the results obtained are highly interesting. However, it should be noted that the HHV of our biodiesel is lower than that of conventional Diesel whose value is 45.4 MJ/kg. This may probably be due to the excess oxygen in the biodiesel [23].

On the other hand, the pour point of our biodiesel was found equal to -2.8°C which is close to that of local commercial diesel whose value varies between -15°C and -7°C, depending on the climate. It should be emphasized that the pour point value is of great importance during the transport of biodiesel, especially in cold countries.

TABLE 4. Characteristics of biodiesel.

Parameters	**Value**	**Standard ASTM**
Density (25°C)	0.87	0.86 - 0.90
Refractive index	1.4482	-
Viscosity (mm^2/s)	3.925	1.9 - 6.0
Flash point (°C)	188.2	Min. 130
Pour point (°C)	-2.8	-
Calorific value (MJ/kg)	25.25	-

Cetane index	46.6	Min. 47
Sulfur content (% by weight)	0.039	Max. 0.05
Distillation temperature at 90% (°C)	346	Max. 360

The yield of biodiesel produced from Balanites oil is 92.2%. This value is higher than that obtained by Ijaz et al. (2016) [24] (90%), who used castor oil with a homogeneous catalyst. On the other hand, Saydut et al. (2016) [25] achieved a yield of 97.3% using the same catalyst with sunflower oil. Regarding the work done by Khelafi et al. (2018), the yield of biodiesel obtained from sunflower oil was 96.7% when using a heterogeneous catalyst derived from date seed. This finding shows that the biodiesel yield depends on the type of oil and catalyst nature. [26],[27].

According to Table 5, Therefore, the results obtained allow us to conclude that the characteristics of the biodiesel obtained in this work are close to those recommended by international standards.

- **Infrared absorption spectroscopy**

The Fourier transform infrared (FTIR) spectroscopic analysis is based on the absorption of light in the infrared spectral range which extends from 400 to 4000 cm^{-1}. Each absorption band is associated with a type of functional group, as shown in figure 5.

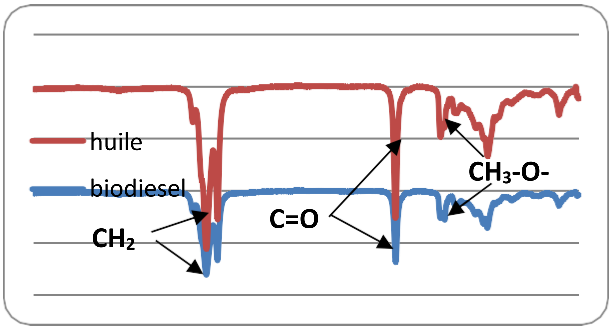

Figure 5: Spectre IR d'huile de Balanites et son biodiesel

The findings suggest that the oil surface presents numerous absorption peaks which correspond to various functional groups that are often responsible for the adsorbent-adsorbate bonds.

Figure 5 depicts the IR spectrum,which presents the absorption bands corresponding to functional groups which may present different modes of vibration.

• Mode of vibration of the functional group C–H

The bands observable in the 2830.94 cm^{-1} region are certainly due to the C–H bond stretching vibrations in the CH_2 groups.

• Mode of vibration of the functional group C=O

The most intense band of the spectra is observable between 1801 and 1629 cm^{-1}. This band is attributed to the stretching vibration of the C=O group, characteristic of esters.

• CH_3-O- functional groups

The observed absorption bands correspond to the CH3-O- vibrations that characterize the methyl esters. This corresponds to the region around 1454.89 cm^{-1}.

On the other hand, Figure 6 shows that the distillation of the diesel obtained is limited to the temperature of 303°C which corresponds to the distillation rate of 90%. It was revealed that beyond 330°C, the biodiesel breaks down and gives other products. It should be noted that the distillation curve is not linear, as it involves several stages, which can be explained by the fact that the biodiesel under study is a mixture of several compounds. This is confirmed by the IR spectrum in figure 5, which shows that biodiesel is a mixture of esters and differs from each other by the length of the carbon chain.

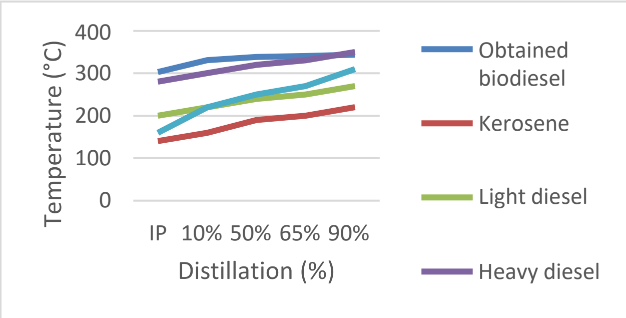

Figure 6. Quality control of biodiesel by distillation.

Beyond 331°C, the curve changes slope for a distillation rate of 10%.Then, it remains almost stable. Below 303°C, no fraction of the biodiesel is distilled, which means that the biodiesel does not contain any water. A simple comparison of the biodiesel obtained with other hydro-fuels allows saying that our biodiesel is similar to heavy diesel because its viscosity is higher than that of conventional diesel [28].

4. CONCLUSIONS

In this study, we demonstrated that it is possible to produce bioethanol from the fruit of Balanites by fermenting its pulp. This bioethanol can then be used as a solvent to extract oil from the nuts. Additionally, the shells of the Balanites fruit can be transformed into catalysts to promote the conversion of Balanites oil into biodiesel.

The extraction yeild of Balanites oil, using bioethanol as solvent, reached 35.45%.

It is worth highlighting that the biodiesel was produced by the transesterification reaction of Balanites oil. The resulting biodiesel yield was 92.2%. However, it should be noted that its viscosity is lower than that of the oil used. The characterization of the biodiesel produced showed that the density, viscosity, flash point, sulfur content, and distillation temperature are within the range set by the ASTM standards. However , the biodiesel produced has a low HHV which leads to the conclusion that this biodiesel should be blended with fossil diesel to improve its HHV and use it as a biofuel for diesel engines.

This study shows that the bioethanol extracted from the fruits of Balanites, as well as the oil extracted from its seeds, and the biocatalyst obtained from the shells of its seeds, can be considered a primary and economical source for producing biodiesel. The biodiesel produced is an alternative and environmentally friendly energy, with the possibility of reducing its cost by relying entirely on cheaper natural sources.

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