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Biodiesel Production from Castor Oil

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Abstract

Biodiesel is becoming one of the most attractive alternative biofuels for petroleum-based diesel fuels. The castor plant is one of the abundant non-edible oils found in many countries. This paper aims to study Libyan castor oil and its potential for diesel conversion. Experiments were carried out in the laboratories of the Specific Center for Training in the Oil Industries in Al-Zawiya. The oil was extracted using a Soxhlet extractor and n-hexane solvent at 60 °C. Transesterification reactions were conducted in a batch reactor (a three-neck flask was used, where the middle opening carries a reflux condensation unit) at 65 °C. The methanol-to-castor oil molar ratio was 6:1, with a catalyst concentration of 1 wt.% relative to the castor oil, and the reaction time was 30 min. Castor oil was analysed and found to have a fatty acid content of 0.7%. The productivity of biodiesel exhibited 80% yield. Standard test methods of analysis were conducted to determine the biodiesel's properties. Results indicated that the kinematic viscosity at 40 °C was 14.24 cSt, the density was 0.924 g/cm^3, the cetane number (CN) was 54 and the pour point was −15 °C, indicating that castor oil biodiesel is of high quality, with a high CN and a low pour point.

***Keywords**: Biodiesel; Castor oil; Alkaline transesterification; Renewable energy; and non-edible oil.*

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1. Introduction

Biodiesel is a promising alternative for replacing conventional diesel. Countries have produced about 36 billion litres of biodiesel globally [1]. Diesel is produced from various sources, such as food waste and animal waste from cows and chickens, which have a high potential to produce biogas due to their high organic content. Methane production may be achieved by adding bioaugmentation with bacteria, along with the adjustment of pH and using a semi-continuous system [2, 3].

Edible vegetable oils adversely affect the balance between market demands and food supply, accounting for roughly 70%–80% of the total cost of producing biodiesel [4]. As a result, finding less costly and non-edible sources has become the aim of the hunt for substitutes for these food oils. Castor seeds are one of these sources; they are regarded as one of the most promising choices for biodiesel because of their high oil content (roughly 46%–55%) [5, 6]. Castor oil is a plant suited to arid regions and dry climates. It is inedible and has no effect on food security. It is also a highly productive plant that grows quickly.

Castor oil contains high amounts of ricinoleic acid ($C_{18}H_{34}O_3$) at about 80%–90%, which is the basis for producing biofuels. It is also composed of

3% - 6% linoleic acid and 2% - 4% oleic acid, and the rest is saturated fatty acids. Castor oil produces diesel with a higher viscosity than other oils, making it an ideal choice for improving the viscosity of petroleum diesel and suitable for many industries such as plastics, cosmetics and paint. Miscibility, low iodine content and low freezing point make castor oil an appropriate raw material to produce biodiesel [6–8]. All these features of castor oil remarkably reduce the cost of biodiesel production.

Transesterification is the primary method used to lower the viscosity of vegetable oils and create alkyl esters resembling diesel. Hence, transesterified vegetable oils are commonly referred to as 'biodiesel'. The process involves an ester from one alcohol reacting with different alcohols to produce an ester from the latter alcohol and alcohol from the initial ester, such as methyl acetate and ethyl alcohol forming ethyl acetate and methyl alcohol [9, 10]. Several important conditions must be considered for this step: temperature, reaction time, catalysts, moisture, free fatty acid (FFA) content and the effect of the molar ratio between alcohol (methanol or ethanol) and oil (raw material) [8].

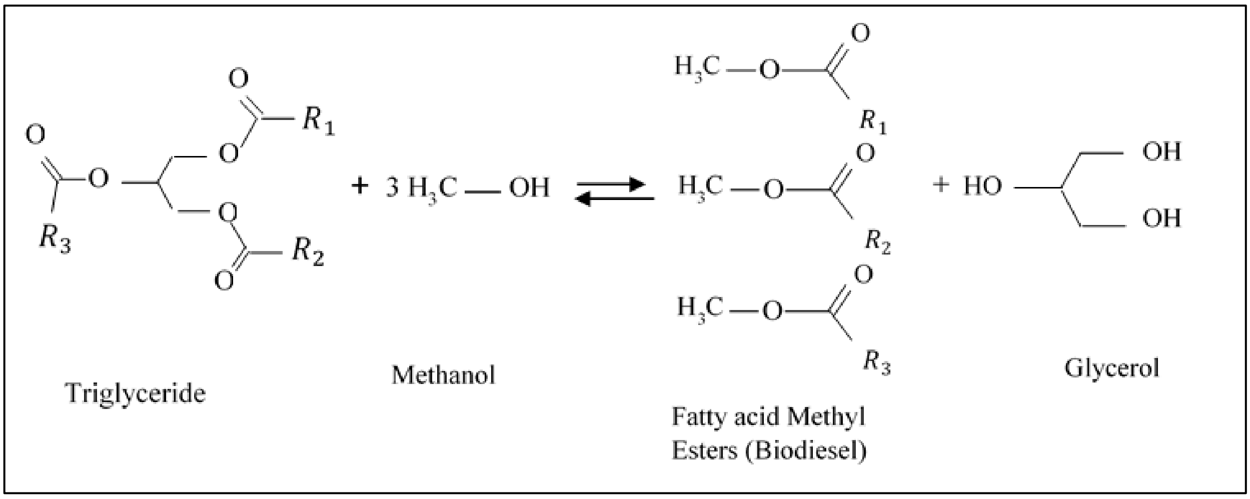

Fig. 1. Transesterification reaction of vegetable oil (triglyceride) and methanol to fatty acid methyl esters (biodiesel) and glycerol

High vital diesel fuel is very important to ensure enhanced engine performance with no operating problems. The evaluation of the quality of vital diesel fuel usually depends on physical features and general chemicals that resemble diesel and the chemical composition that affects the specific fuel properties. Motor viscosity and density represent the value of acid, pour points, flash points, iodine value, fatty acids and other properties [11, 12].

Biodiesel derived from Castor oil has benefits of being biodegradable, non-toxic, renewable and safe to handle. It also offers low greenhouse gas emissions, a high flash point and energy similar to that from fossil diesel [13]. Biodiesel is crucial for fighting climate change and enhancing air quality, which protects public health and ecological integrity [14]. Other advantages of castor beans in the esterification process are high miscibility of alcohol, low reaction temperature, low iodine content and low freezing point [6]. The present work investigated the extraction of castor oil from castor seeds and its ability to produce biodiesel.

2. Materials and Methods

The process of biodiesel production was divided into several levels. Fig. 2 shows the flowchart of these steps. Experiments were conducted in the laboratories of the Specific Center for Training in the Oil Industries in Al-Zawiya, Libya. The biodiesel production started with collecting castor beans and preparing them for the next steps. The preparation of castor beans included washing, drying, winnowing and grinding. Castor oil was extracted from the seeds via

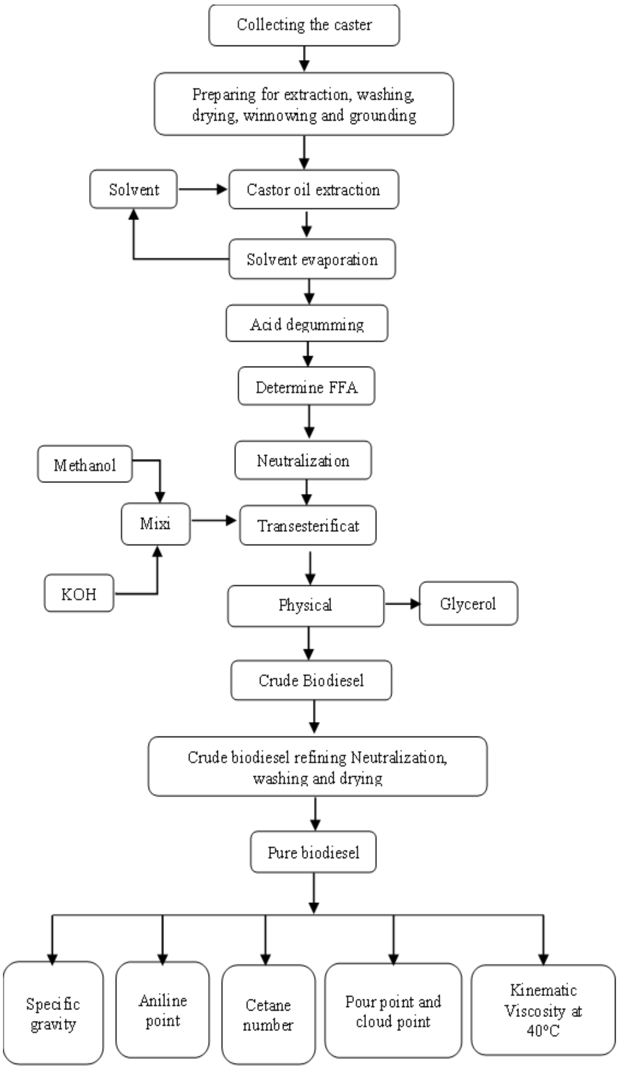

Fig. 2. Biodiesel production from castor oil by alkaline transesterification reaction

The evaporated solvent was reused in the extraction. After castor oil was obtained, it was refined to improve its quality. Degumming was the first refining step, followed by determination of the acid content. Afterwards, castor oil was prepared to produce biodiesel.

Transesterification reaction was carried out to produce biodiesel. The triglycerides of castor oil reacted with methanol in the presence of KOH as a catalyst. Then, the biodiesel was separated from glycerol (byproduct). The final step was purification of the biodiesel by neutralising KOH with acetic acid, washing the biodiesel to remove any contaminants (methanol, catalyst and soap), and drying the washing water. Then, the biodiesel properties were determined in accordance with ASTM tests.

2.1. Preparation of Castor Beans for Extraction

Castor beans were collected from farms in Surman City, Libya. These beans were prepared for oil extraction. The castor plant is shown in Figure (3). The castor beans had some undesired materials that should be separated by hand picking. The beans were washed carefully to remove any remaining dirt and then heated in the oven at 70 °C for 72 h to break down the cells containing the oil and dry any moisture present in the seeds. The shell was separated from the seeds by hand. Then, the beans were crushed to rupture the cell walls to release castor oil.

Fig. 3. Castor plant

2.2. Castor Oil Extraction

Castor oil was extracted using the Soxhlet apparatus, as shown in Fig. (4). A vacuum rotary evaporator was used to extract the solvent from the oil. Normal hexane (300 mL) was placed into a round-bottom flask. The thimble containing 10 g of the sample was placed in the extractor's centre. After the solvent was heated to 60 °C, the vapor rose through the vertical tube and condensed at the top. The liquid condensate trickled into the central thimble, which held the solid sample that was needed to be extracted. The extract was returned to the round-bottom flask after passing through the thimble's pores and filling the siphon tube. This process was continued for 30 min [15–17].

Fig. 4. Soxhlet extractor

A vacuum rotary evaporator was used to evaporate the hexane solvent from the castor oil. The sample was contained in the evaporation flask while the water bath temperature was 40 °C. After 30 min, the castor oil remained in the evaporation flask, and the solvent was received from the receiving flask. The evaporated n-hexane can be used for the next sample to be extracted. The amount of extracted castor oil was calculated using Eq. (1) as follows:

$$\%\ \text{castor oil yield} = \frac{\text{Weight of extracted oil} \times 100\%}{\text{Weight of total sample}} \quad \ldots (1)$$

The extracted castor oil was refined, and the FFA content was determined to neutralise to prevent the formation of the soap.

2.3. Castor Oil Acid Degumming

Crude castor oil (190 mL) was stirred and heated to 80 °C–90 °C by using a magnetic stirrer. Then, 4 mL of citric acid solution (30%) was slowly added to the crude oil. The mixture was stirred for 20 min, cooled to 25 °C, and washed with 10 mL of distilled water. The solution was transferred to a separating funnel to allow the gums to settle in the bottom of the funnel. The solution was settled for 24 h, and the gums were removed from the oil. Finally, the oil was transported to a centrifuge to remove the remaining gums and water [16].

2.4. Determination of Oil Acidity

The method of Kardash and Tur'yan was used to determine the acid value of the samples. This method is based on titration with ethanol, using phenolphthalein as an indicator [18]. The oil sample (1.0 g) was placed into a 250 mL-conical flask, followed by diethyl ether (25 mL), ethanol (25 mL) with 96% purity and three drops of phenolphthalein indicator. Then, the mixture was heated for 3 min by using a shaking water bath. Afterwards, the mixture was titrated against NaOH (0.025 M), with vigorous shaking until a pink colour appeared [19, 20]. The acid value (AV) of the oil is expressed in mg NaOH per gram of oil. The FFA percent (%FFA) was calculated from the acid value as follows:

$$\%\,FFA = \frac{AV}{1.99} \quad \ldots (2)$$

2.5. Neutralisation

FFA is removed from crude oils via neutralisation. This process was accomplished by adding 0.412 g of NaOH (0.1 M) to the degummed castor oil (158 g) and heating the mixture to 85% °C–95 °C, with mixing for about 45–60 min to break any emulsion that may form during neutralisation. The FFAs were converted into soap stock, which has a higher specific gravity (SG) than neutral oil and tends to precipitate at the bottom. The mixture then was transferred to a separating funnel. Afterwards, the mixture was placed into a separating funnel and left for 24 h to remove the soap that formed. Hot water was then added to the oil several times until the soap that was still present in the solution was removed.

Hot distilled boiling water (30%) was added to hydrate the neutralised oil. The mixture was stirred for 2 min and left in the separating funnel until two distinct phases were visible. The aqueous layer at the bottom was then eliminated. The process needed to be repeated to guarantee that the majority of gums and soaps were removed. The oil was dried in an oven set at 120 °C for 2 h to remove the water [21–23].

2.6. Determination of the SG of Castor Oil

For the measurement of the specific gravity, sp. gr, a clean and dry volumetric cylinder was used to determine the density of the oil with the volume of 50 mL and its weight (W0). The volumetric cylinder was filled with oil and reweighed to give W1. After the bottle washed and dried, the oil was substituted with water and weighed to yield W2 [24]. Then, the sp. gr was calculated as follows:

$$SG = \frac{W_1 - W_0}{W_2 - W_0} \quad \text{... (3)}$$

2.7. Biodiesel Production Using Transesterification

As illustrated in Figure (5), the reactor, a 250 mL three-neck flat bottom flask, was positioned with a reflux condensation unit in the middle opening (or neck). The thermometer was carried by one of the two side arms, whereas the other side arm served as the reactor's feed point.

The setup was mounted on the hot plate magnetic stirrer. The condenser was then connected to a tap for the inflow and outflow of cold water to prevent alcohol loss during the transesterification reactions.

Fig. 5. Transesterification process

Initially, 83 g of castor oil was poured into the reactor and heated to 65 °C. A solution of KOH in methanol (99.8% pure) was freshly prepared with the following conditions: KOH concentration of 1% of castor oil weight and methanol-to-castor oil molar ratio of 6:1. The heated oil was gradually mixed with the catalyst solution. Following the reaction, the mixture was left in the separating funnel at room temperature for 24 h to settle under gravity. Two layers developed during the separation process, with the glycerol phase at the bottom and the crude ester phase at the top. The tap at the bottom was opened to remove the glycerol phase from the biodiesel [25–27].

After the biodiesel was separated from the glycerine layer, it was purified with warm distilled water. Five additional washing steps were conducted with water only. The excess methanol and any remaining water were removed from the biodiesel by heating the biodiesel at 120 °C for 2 h [28]. The biodiesel yield (%) was determined using Eq. (4) as follows:

$$\text{Biodiesel yield (\%)} = \frac{\text{weight of produced biodiesel (g)}}{\text{weight of raw oil (g)}} \times 100\% \quad \text{... (4)}$$

2.8. Characterisation of Biodiesel

2.8.1. Kinematic Viscosity (μ)

ASTM D 445 is the standard method for determining the kinematic viscosity of diesel or biodiesel fuels. The viscosity of biodiesel was measured using a viscometer, as shown in Fig. (6). The kinematic viscosity was determined at 40 °C. The viscometer was placed in a water bath set at 40 °C.

After 10 mL of the sample was added to the viscometer, the sample and the viscometer were left to acclimate to the water bath for 0.5 h. The sample was kept in the water bath until it reached the equilibrium temperature of 40 °C. After the equilibrium temperature was maintained, the time required for the biodiesel to flow under gravity was measured using a calibrated glass capillary tube.

The kinematic viscosity was determined using Eq. (5) [29] as follows:

$$\mu = 0.0079578 \times t, \quad \text{... (5)}$$

where μ is the kinematic viscosity in mm^2/s, the constant 0.0079578 is the calibration constant of the tube and t is the time required for the biodiesel to flow under gravity through a calibrated glass in seconds.

Fig. 6. Kinematic viscometer

2.8.2. Pour Point

ASTM D97 was used to determine the pour point. The biodiesel was cooled at a predetermined rate, and its flow characteristics were checked at 3 °C intervals. The pour point was the lowest temperature at which the sample movement was detected [30]. The biodiesel Pour point was determined using ASTM D2500. A predetermined cooling rate was applied to the biodiesel and periodically checked. The Pour point was determined by measuring the temperature at which a cloud was first seen at the test jar's bottom [31].

2.8.3. Aniline Point

The aniline point is the lowest temperature at which equal volumes of aniline ($C_6H_5NH_2$) and oil form a single phase. It was determined using ASTM D611. A 10 mL sample of biodiesel was introduced into a test tube. An equal volume of aniline oil was added to the biodiesel. The mixture was combined and heated at a regulated rate to make the two phases miscible. The aniline point or mixed aniline point was then determined by cooling the mixture at a regulated rate and recording the temperature at which the two phases separated [32]. The aniline point test apparatus is depicted in Figure (7).

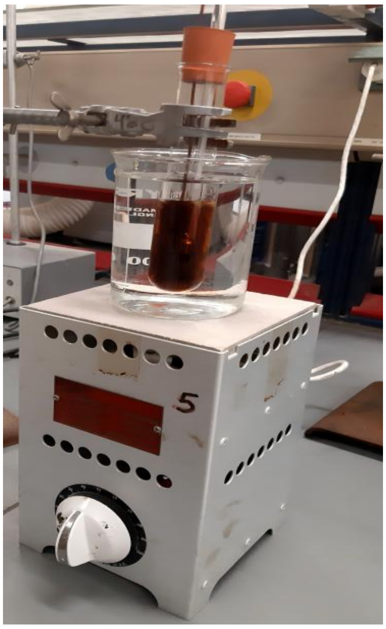

Fig. 7. Aniline point test

2.8.4. SG at 60 °F

A clean and dry volumetric cylinder was used to determine the density of biodiesel with a volume of 50 mL and weight W0. It was then filled with biodiesel, plugged and reweighed to yield W1. The oil was replaced with water after washing and drying the bottle and weighed to give W2 [33]. SG was expressed using Eq. (6) as follows:

$$SG_{\text{biodiesel}} = \frac{W_1 - W_0}{W_2 - W_0} \quad \text{... (6)}$$

2.8.5. API Gravity

The following equation was used to calculate the American Petroleum Institute (API) gravity on the basis of the biodiesel-SG value:

$$API = \frac{141.5}{\text{specific gravity}} - 131.5 \quad \text{... (7)}$$

2.8.6. Cetane Number (CN)

The CN index was determined in accordance with the method of Boerlage and Broeze (1994) [33] by using Eq. (8) as follows:

$$Cetane\ Index = \frac{Aniline\ point\ (℃) \times API\ Gravity}{10} \quad \text{... (8)}$$

3. Results and Discussions

The extraction results showed that every 1 g of castor beans after washing and drying in the oven yielded 0.51 g of oil, with a production rate of 51%. The raw castor oil produced contains some amounts of mucilage. Acid degumming was necessary to remove phosphatides and mucilage from the oil. The amount of lost oil was determined and obtained at 22% of the weight of the oil. The acid number of crude castor oil was 2.6 mg NaOH/g oil, indicating an FFA content of 1.3%. When the FFAs in the oil reacted with NaOH, soap was produced and then removed by washing with distilled water. Approximately 47% of the oil was lost during the neutralisation process. After the castor oil was neutralised, the acid number decreased to 1.4 mg NaOH/g oil, and the FFA content was 0.7%. After these preparatory processes were performed, the obtained castor oil can be used as a raw material for biodiesel production. The properties are shown in Table 1.

Table 1,
Castor oil properties

Property	Before refining	After refining
Density g/mL at 20 °C	---	0.952
Specific gravity	---	0.954
Acid value NaOH/g oil	2.6	1.4
FFA content	1.3%	0.7%

After the mixture was allowed to settle for 24 h, the transesterification process resulted in two layers. The lower layer, glycerol (a byproduct), was removed from the separating funnel, whereas the upper layer was biodiesel with a yellow colour. The produced biodiesel underwent purification, where the base catalyst (KOH) was neutralised with acetic acid and then washed five times with distilled water to eliminate the residual catalyst and excess methanol, as shown in Figure (8).

Fig. 8. Neutralisation and transesterification

3.1. Biodiesel Yield

This process achieved an 80% yield, indicating that the parameters used were effective and could be adjusted for optimal castor oil biodiesel production. However, some alcohol possibly remained unreacted, or some biodiesel was probably lost during the washing stage. This yield from castor oil is better than the results obtained by Dias, et al. (2013) [8], which ranged from 43.3% to 74.1%. Some studies, such as those by Elango, R.K. et al. (2019) and Meneghetti, S.M.P. (2006), reported yields exceeding 90% [8, 34]. The reason for the difference in productivity may be due to the nature of the oil and the purity of the raw materials. Many researchers have studied the potential yield of biodiesel from non-edible oils, including *Hevea brasiliensis*, *Ricinus communis*, *Gossypium*, and waste cooking oil, with yields of 45%, 48%, 96%, and 59.44%, respectively, when using methanol as the solvent and KOH as the catalyst [35].

3.2. Characterisation

The comparison results of castor oil biodiesel with petroleum diesel and various international standards are shown in Table 2. The produced biodiesel at 40 °C had an extremely high viscosity value (14.24 mm^2/s), exceeding the international standard limit (1.9–6 mm^2/s). This finding can be attributed to the presence of OH groups in ricinoleic acid tending to form hydrogen bonds with other hydrogen atoms present in other molecules [36]. The obtained biodiesel had a lower viscosity than that obtained by other studies, which were 15.5, 15.17, and 15.98 mm^2/s [27].

Table 2,
Properties of the produced castor biodiesel

Property	Unit	ASTM test method	Limits	Castor biodiesel
Viscosity at 40 °C	mm^2/s	D445	1.9–6	14.24
Density	kg/m^3	D1298	860–900	924
API gravity	---	---	---	21.639
Pour point	℃	D97	0	-15
Cloud point	℃	D2500	Report	No wax found
Aniline point	℃	D611	---	25
Cetane number	---	---	47 (min)	54

High viscosity is a major problem of biodiesel in diesel engines. The high value of viscosity of fuel causes depositions on the engine, which affects its function. Thus, the produced biodiesel cannot be directly used in diesel engines. It can be used by blending with petroleum diesel to minimise fuel viscosity.

The density of the produced biodiesel at 15 °C was 924 kg/m^3, and the Sg was 0.9248. This density value is above the international standard limit (860–900 kg/m^3). This high density is a consequence of the presence of the hydroxyl group of ricinoleic acid methyl ester [36]. It can be mixed with diesel without exceeding the standard accepted ratio for biodiesel density. The pour point of the produced biodiesel (−15 °C) was considerably far from the maximum allowed pour point of the European standard EN 14213 (0 °C max). This finding indicates that castor oil biodiesel could be used as a petroleum diesel additive for improving cold flow properties.

The cloud point was higher than the pour point. The produced biodiesel reached the pour point without the appearance of any wax crystal. Some researchers found the cloud points of their biodiesel to be −15 °C [20] and −14 °C [25]. Thus, castor oil biodiesel could be used in cold weather as a petroleum diesel additive.

The aniline point was determined to be 25 °C, and the CN was 54. This low aniline point may indicate the absence of aromatic compounds in the castor oil biodiesel. The CN of the biodiesel was above the lower limit of the standards (47 min). The high CN value represents its high ability towards ignition in engines after injection [36]. Other researchers obtained a varying CN at 43.7 min. This value is lower than that of the standard. Other researchers found that the CNs of castor biodiesel were 55.82 and 60.2 min [20, 37], respectively.

4. Conclusion

Castor bean plant was used in this study to produce biodiesel. Castor oil was extracted, and many steps were taken before biodiesel was created. The beans contained approximately 51% castor oil. A high yield of biodiesel was obtained from castor oil, more than 80%. The fuel properties of the biodiesel obtained from alkaline transesterification of castor oil were determined using standard methods and compared with the standard values of ASTM specifications. The produced biodiesel had a high CN (54 min) and a very low pour point (−15 °C), makes this biodiesel a good alternative in winter conditions. It also had a high viscosity (924 kg/m^3), which makes it unsuitable for use in diesel engines alone. Castor oil biodiesel could improve the environmental performance and flow characteristics of petroleum diesel.

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إنتاج الديزل الحيوي من زيت الخروع

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المستخلص

يعد وقود الديزل الحيوي أحد أكثر البدائل جاذبية لوقود الديزل القائم على البترول. تعتبر الزيوت النباتية من أكثر المواد الخام استخدامًا لإنتاج وقود الديزل الحيوي مثال ذلك زيت الخروع والذي يعتبر من الزيوت غير الصالحة للأكل والتي توجد بكثرة في العديد من دول العالم. تتضمن هذه الدراسة استخلاص زيت الخروع واستخدام الاسترة القلوية المتجانسة لتصنيع وقود الديزل الحيوي. وقد تم استخدام عامل مساعد بتركيز ١٪ وزنيًا من هيدروكسيد البوتاسيوم ونسبة الميثانول إلى الزيت المولية ٦:١ في تصنيع وقود الديزل الحيوي. تم تحليل زيت الخروع ووجد أنه يحتوي على نسبة ٠,٧٪ من الأحماض الدهنية. بلغت انتاجية وقود الديزل الحيوي ٨٠٪. اعتمدت في هذه الدراسة طرق الاختبار القياسية للتحليل لتحديد خصائص وقود الديزل الحيوي. أشارت النتائج إلى أن اللزوجة الحركية عند ٤٠ درجة مئوية كانت ١٤,٢٤ سنتستوك، والكثافة كانت ٠,٩٢٤ غم/سم٣، والرقم السيتاني ٥٤، ونقطة الصب -١٥ درجة مئوية. أظهرت النتائج أن وقود الديزل الحيوي من زيت الخروع عالي الجودة، مع رقم سيتاني مرتفع، ونقطة صب منخفضة للغاية.
