

**Research/Technical Note**

Assessment of Low Temperature Refining Process of Castor Seed Oil for Biodiesel Production

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Abstract: In recent years research on biodiesel production from non-edible seed oil is receiving more attention to prevent food vs biofuels problems such as starvation in developing countries. Castor seed oil is potential non-edible biodiesel feedstock, but it has high viscosity and high acid value which are among the undesired factors in biodiesel production. The castor seed oil was extracted using soxhlet extraction method, refined using standard analytical methods and its physicochemical properties was determined using ASTM methods. The crude extracts of castor seeds oil which was characterized as high viscous oil, was refined to improve its quality, and physicochemical properties of refined and crude extracts castor seed oil was studied. The crude extracts oil (41.29±1.54%) produce was refined to reduce its high viscosity (233.0±2.00 mm²/s) and acid value (4.488±0.40 mgKOH/g). The viscosity and acid value of the oil significant reduced to 159.0±3.00 mm²/s and 2.805±0.20 mgKOH/g after the refining process. The others physicochemical properties such as flash point, saponification value, iodine value, moisture contents, specific gravity, refractive index, kinematic viscosity, cloud point, pour point and free fatty acids qualities were improved after refining. The study show that refined castor seed oil were more suitable in biodiesel production than crude castor seed oil.

Keywords: Castor Seed Oil, Soxhlet Extraction, Refining, Physicochemical Properties

1. Introduction

Castor oil has natural antibacterial and antifungal properties and is high in vitamin E, minerals, proteins and Omega 6 and 9 beneficial fatty acids. Its unusually have high ricinoleic acid ratio, which makes it beneficial to skin and hair. In fact, castor oil has traditionally been used topically for acne and other skin conditions, as well as hair loss, rashes and more [1]. Now, castor oil is massively used in biodiesel and bio-lubricants productions because of its unique physicochemical properties. The general problem of vegetable oils that make it difficult to be used as fuel are their high viscosity which affects their fluidity in engine and high acid value which may cause corrosion in the engine. So, there is need for pretreatments in order to reduce the viscosity and acid value [2].

Castor seed, *Ricinus communis L.*, is a member of the tropical spurge family, *Euphorbiaceae*, and can nowadays be found naturalized and cultivated in all temperate countries of the world. It belongs to a monotypigenus, *Ricinus*, and sub tribe, *Ricininae*. The evolution of castor and its relation to other species are currently being studied.

Castor is amongst the plants with the highest oil yield potential because of its high yield of seeds and the high oil content of its seeds [3]. It may be possible to obtain a maximum of 2000 kg_{oil}·hr⁻¹. Whereas rapeseed produces about 1000 kg_{oil}·hr⁻¹ and soybean only produces about 500 kg_{oil}·ha⁻¹ [4]. Therefore, castor oil is a promising source to produce biodiesel, decreasing the cultivated land.



Figure 1. Castor seeds.

About chemical and physical characteristics, castor oil composition is 80-90 % ricinoleic acid, 3-6 % linoleic acid, 2-4 % oleic acid and 1-5 % saturated fatty acids [1]. Ricinoleic acid is the main fatty acid from castor oil, this fatty acid possesses 18 carbons with three highly reactive functional groups: the carbonyl group in 1st carbon, the double linking or unsaturation in 9th carbon and the hydroxyl group in 12th carbon. This feature causes castor oil properties are different from other vegetable oils [5]. The high content of ricinoleic acid, with a hydroxyl group, is the reason for castor oil has especially high viscosity and density. Castor oil is also characterized by its high stability, high hygroscopicity and its solubility in alcohol, which affects the transesterification reaction [6].

2. Material and Methods

2.1. Preproduction Process

The Castor seed was procured from Yandodo, Kano State. The seed was sundried to reduce the moisture content and it was then grinded to reduce the particle size in preparation for extraction. The Castor seed was de-shelled. The de-shelled seed which was white in color was oven dried at 90°C for 45 minutes. The dried seeds were grounded using motor and pestle and weighed.

2.2. Oil Extraction Process

Extraction of castor oil was carried out by soxhlet extraction method. 100 cm³ of n-hexane was placed in round bottom glass flask, 50.0 g of the sample was placed in thimble and settled in the center of the extractor and Soxhlet apparatus was heated at 108°C. The solvent boiled and vaporized through the vertical tube into the condenser at the top. The condensate liquid started dropping into the thimble containing the sample and the extracted oil seeped through thimble into the flask via the siphon. After each hour the sample was dried at 70°C and weighed until the residue weight became constant. At this stage the amount of extracted oil was determined at the

end of each extraction cycle. The oil-solvent mixture was oven heated at 108°C for solvent recovery [7]; [8]. The yield of the oil was determined using equation 1:

$$\% \text{ extracted oil yield} = \frac{\text{weight of oil extracted (g)}}{\text{weight of the sample (g)}} \times 100\% \quad (1)$$

2.3. Pretreatment of Extracted Oil

2.3.1. Degumming

Exactly 100 cm³ of crude castor oil was weighed into 250 cm³ beaker placed on hotplate magnetic stirrer with agitation at 600 rpm and heated to 85°C, 100 cm³ of distilled water was added and allowed under the same condition for 30 minutes. The mixture was transferred into separating funnel and allowed to settle for 15 minutes, the oil which was at upper layer was separated by draining the lower layer. The oil was then taken back into a beaker and reheated to 85°C at 600 rpm, 60 cm³ of 0.1M of phosphoric acid was added and allowed for 30 minutes. 80 cm³ of distilled water was added and allowed for another 10 minutes before transferred into 500 cm³ separating funnel and allowed it to settle for 15 minutes. The degummed oil at the upper layer was separated by draining the lower layer. 100 cm³ of distilled water was added into separating funnel containing only degummed oil and was shaken for two minutes and allowed to settle. The water at lower layer was drained by opening the tab on the separating funnel. The washing was repeated for 3 times to ensure purity of the degummed oil [2].

2.3.2. Neutralization

The 100 cm³ of degummed castor oil was heated to 85°C in 500 cm³ beaker on a hotplate magnetic stirrer with agitation of 600 rev/min. 25 cm³ of 0.5 M of NaOH was added and allowed for 30 minutes, 100 cm³ of distilled water was added and then transferred into 500 cm³ separating funnel. The neutralized oil at the upper layer was separated from the lower. The neutralized oil was washed three times with 100 cm³ of distilled water. The refined oil was then reheated at 105°C for 1 hour to remove moisture [7].

2.4. Determination of Physicochemical Properties of Crude and Refine Castor Oil

2.4.1. Tests for Cloud Point and Pour Points

The oil sample (5 cm³) was measured in 5 cm³ test tube and placed in a freezer. The sample was taken out of the freezer every 1 minute to check any visible changes and the temperature was measured using digital mex-tech thermometer [9].

Observation for Cloud Point: At a particular temperature, crystals began to form in the sample; reading was taken [10].

Observation for Pour Point: At a certain temperature, gel and crystals were dissolved from sample (it's frozen) and then the temperature reading was taken [10].

2.4.2. Test for Flash Point

The flash point was determined according to ASTM D93 method. The sample was placed in the test cup to the prescribed mark in the interior of the cup. The cup was

mounted on to its position on the tester. Bunsen burner was used to supply heat to the apparatus at rate of 1°C per minute with constant stirring. A small test flame was directed into the cup intermittently. The flash point was taken as the temperature when the test flame caused the vapour above the sample to ignite [10].

2.4.3. Determination of Moisture Content of the Seeds

The grinded sample (40.0 g) of seeds was weighed, oven dried at 80°C for 7 hrs, and the weight was recorded every 2 hrs, until a constant weight is obtained. After each 2 hrs, samples were removed from the oven, placed in desiccator for 30 minutes, to cool, then removed and re-weighed. The percentage moisture in these seeds was calculated using equation 3 [11]:

$$\text{Moisture content} = \frac{w_1 - w_2}{w_2} \times 100\% \quad (2)$$

W_1 = Original weight of the sample before drying

W_2 = Weight of the sample after drying

2.4.4. Determination of Specific Gravity

The empty beaker of 50 cm³ capacity was weighed (W_0), filled with oil, and then reweighed (W_1). The oil was substituted with water after washing and drying the beaker and weighed (W_2) [11].

The expression for specific gravity is:

$$\text{Specific gravity} = \frac{\text{Mass of substance}}{\text{Mass of equal volume of water}} \quad (3)$$

$$S.G. = \frac{w_1 - w_0}{w_2 - w_0} \quad (4)$$

2.4.5. Determination of Refractive Index

Few drops of oil samples were transferred to glass slide of the refractometer. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross, In this case the pointer on the scale pointed to the refractive index and values were recorded [11].

2.4.6. Determination of Kinematic Viscosity

The dynamic viscosity was determined using Cannon Viscometer model 2020. The spindle (spindle number 63 and 04 were used) through calibrated spring of viscometer was rotated in 50 cm³ of the sample in 50 cm³ beaker. The viscous drag of the fluid against the spindle was measured by the spring deflection. The spring deflection was measured with a rotator transducer which provides a torque signal. The viscosity in centipoises was displayed on the screen of the viscometer. To obtain kinematic viscosity in centistokes (1 centistoke = 1 mm²/s), the dynamic viscosity was divided with density [12].

2.4.7. Determination of Acid Value

Equal volume (10 cm³) of each of diethyl ether and ethanol was mixed in a 250 cm³ beaker, then was added to 2.0 g of oil contained in a 250 cm³ conical flask and few drops of phenolphthalein were added. The mixture was titrated with 0.1M KOH to the end point with consistent shaking, a dark pink color was observed and the volume of 0.1M KOH was

recorded.

Acid value, Free Fatty Acid (FFA) and FFA conversion (%) were calculated equation 5 [11]:

$$\text{Acid value} \left(\frac{\text{mgKOH}}{\text{g sample}} \right) = \frac{\text{volume KOH}(\text{cm}^3) \times N \text{ KOH}(\text{mmol}/\text{cm}^3) \times 56.1 (\text{mg}/\text{mmol})}{\text{sample weight} (\text{g})} \quad (5)$$

$$\text{Free Fatty Acid} \left(\frac{\text{mgKOH}}{\text{g sample}} \right) = 0.5 \times \text{Acid value} \quad (6)$$

$$\text{FFA conversion} (\%) = \frac{A_i - A_t}{A_t} \times 100 \quad (7)$$

Where A_i = the initial acid value; A_t = the acid value at a certain reaction time.

2.4.8. Determination of Saponification Value

The oil sample (2.0 g) was weighed into a conical flask and 25 cm³ of 0.1N ethanolic potassium hydroxide (KOH) was added. The mixture was constantly stirred and allowed to boil gently for 15 minutes. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator were added to the warm solution and titrated with 0.5M HCl to the end point until pink color of the indicator disappeared. The same procedure was used for other samples and blank. Saponification value was calculated [11]; [13]:

$$\text{Saponification value} \left(\frac{\text{mgKOH}}{\text{g sample}} \right) = 56.1 \times \frac{N(V_0 - V_1)}{W} \quad (8)$$

V_0 = volume of the solution used for blank test

V_1 = volume of the solution of the sample used for determination

N = Actual normality of the HCl used

W = Mass of the sample (g)

2.4.9. Determination of Iodine Value

Sample of 2.0 g of oil was weighed into a conical flask and 25 cm³ of carbon tetra chloride (CCl₄) was added to dissolve the oil. Then 25 cm³ of Dam's reagent was added to the mixture using a safety pipette in fume chamber. Stopper was inserted and the content of the flask was vigorously swirled. The flask was placed in the dark for 2.5 hours. Then, 20 cm³ of 10% aqueous potassium iodide (KI) and 125 cm³ of water were added using a measuring cylinder. The solution was titrated with 0.1M sodium thiosulphate (Na₂S₂O₃) solutions until the yellow color almost disappeared. Few drops of 1% starch solution indicator was added and titration continued by adding sodium thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples. The iodine value (I. V) was determined by using equation 9 [11];[13].

$$\text{Iodine value} = \frac{126.9 \times M \times (V_0 - V_1)}{W} \quad (9)$$

M = Concentration of sodium thiosulphate used

V_0 = Volume of sodium thiosulphate used for blank

V_1 = Volume of sodium thiosulphate used for determination

W = Mass of the sample (g)

3. Results

The results obtained might vary depending on the method and solvent used for oil extraction, nature and type of sample,

environmental factors, method of transesterification etc [11]. The results obtained from these analyses are interpreted as follow:

Table 1. Physicochemical properties of crude and refined castor oil.

Properties	Unit	Crude castor oil	Refined castor oil
Extracted oil yield	%	41.29±1.54	28.09±0.50
Moisture content	%	2.11±0.01	–
Specific gravity	–	0.9555±0.01	0.9245±0.00
Refractive index	–	1.470±0.00	1.443±0.00
Cloud point	°C	3.17±0.31	4.00±1.20
Kinematic viscosity @ 40°C	mm ² /s	233.0±2.00	159.0±3.00
Pour point	°C	-24.0±2.00	-13.1±2.00
Saponification value	mg _{KOH} /g _{sample}	182.525±1.19	169.910±0.20
Iodine value	g _{I₂} /100g	81.533±1.35	78.216±1.80
Acid value	mg _{KOH} /g _{sample}	4.488±0.40	2.805±0.20
Free fatty acid	mg _{KOH} /g _{sample}	2.244±0.20	1.403±0.10
Flash point	°C	225.0±1.00	190±1.00

Values are Arithmetic Mean±Standard Deviation of three replicate determinations

4. Discussion

4.1. Refining of Crude Castor Oil

Refining is a process used to improve the quality of vegetable oil that will gives it characteristic to be use directly as fuel or to undergo upgrade process i.e. transesterification before been use as fuel [7]. There are three steps in refining process i.e. degumming, neutralization and bleaching. The degumming: the purpose of degumming is two folds; On the one hand, it is necessary to remove almost completely phosphatides or gums from the oil in order to produce fully refined oil, and, on the other hand, gums may be valuable by-products [2], neutralization: which is employ to reduce free fatty acid and soap formation and bleaching: to remove colour, odour, impurities and residual soap [7]. In this experiment, it was determined that after every degumming of 100 g of crude castor oil, 32g of gum were removed which was corroborate by saadia, [2] who obtained 30 g of gum after degumming 100 g of soya beans oil using 34 g of phosphoric acid in 58 cm³ of water.

4.2. Percentage Yield of Oil and Biodiesel

Castor oil obtained from castor beans contain about 38 -60 % of oil which can be used in production of paints, varnishes, lubricants, grease, hydraulic fluids, soaps, pharmaceuticals, cosmetics and so on [7]. The percentage yield of oil extracted from castor seeds oil in this experiment was 41.29±1.54% and similar result (43.18%) for castor seeds was obtained by Gregory *et al.*, [14], which is high than the value (37%) for castor seeds reported by Saribiyik *et al.*, [15] and lower than (48%) that obtained by Nakarmi and Joshi, [7]. The differences may be due to plant growing environment and method of extraction employed. After refining process, the yield reduced by 13.21% to 28.09±0.50%. Reductions may be due to removal of hydralable phospholipids, non hydralable phospholipids, metal salts and impurities during degumming and saponifying some free fatty acids and washing them away

during neutralization [7].

4.3. Acid Value

Acid value (AV) is a common parameter in the specification of fats and oils. It's defined as the weight of KOH in mg needed to neutralize the organic acids present in 1g of fat/ oil and it is measure of the free fatty acids (FFA) presents in the fats or oil. The high acid value implies that the oil has high susceptibility to decomposition [16]. Excess or higher acid and free fatty acid values of oil greater than 5% is not suitable for base catalyzed transesterification reaction [17]. The acid value of crude castor oil obtained is 4.488±0.40 mgKOH/g which is lower than 15.57 mgKOH/g obtained by Omohu and Omale [16] and high than the maximum value specified by ASTM of 2 mgKOH/g. After neutralization during refining of the crude castor oil, the acid value drop to 2.805±0.20 mgKOH/g. Nakarmi and Joshi [7] performed similar refining process on the castor oil, where their acid value of the crude castor oil drop from 2.629 to 1.566 mgKOH/g.

4.4. Saponification Value

This is the measure of the total free and combined acid in the oil expressed as the number of milligrams of potassium hydroxide required for the complete saponification on 1 g of the oil [16]. Saponification value is related to the average molecular weight of the sample. The molecular weight is inversely proportional to the saponification value. Saponification values increased with the yield of methyl ester [18]. The saponification of crude castor oil was decreases from 182.525±1.19 to 169.910±0.20 mgKOH/g after refining. A comparative result was obtained by Nakarmi and Joshi, [7]; where after their refining of castor oil, the saponification value decreases from 79.159 to 76.258 mgKOH/g. Decreases in the saponification value may be due to the facts that certain fatty acids had been neutralized during refining process. Higher saponification value would lead to the formation of more soap during transesterification with basic catalyst, which would

hinder the biodiesel formation and affect the quality of biodiesel produce [19]. Hence, refined castor with lower saponification value is more preferable for biodiesel production.

4.5. Iodine Value

Iodine value, also called Iodine number, in analytical chemistry, measure of the degree of unsaturation of oil/ fat ; the amount of iodine, in grams, that is taken up by 100 grams of the oil/ fat. Saturated oils/ fats take up no iodine; therefore their iodine value is zero; but unsaturated oils/ fats take up iodine [11]. The more iodine is attached, the higher is the iodine value, and the more reactive, less stable, softer, and more susceptible to oxidation and rancidification is the oil/ fat⁷. Drying oils used in the paint and varnish industry have relatively high iodine values (about 190). Semidrying oils, such as soybean oil, have intermediate iodine values (about 130). Nondrying oils, such as olive oil, used for soap making and in food products, have relatively low iodine values (about 80) [16].

The iodine value of crude and refined castor oil as presented in Table 3.1 are 81.533 ± 1.35 and 78.216 ± 1.80 gI₂/100g which could be categorized as nondrying oil and can be used in soap making. The low iodine values of both crude and refined castor oils indicate their lower degree of unsaturated compound. The refined castor oil has lower iodine value this may be due to hydrolysis that might took place during refining process. The iodine value of crude castor oil is similar with that (80.5 gI₂/100g) obtained by Sanchez *et al.*, [20] and lower than (84.19 gI₂/100g) and (93.5 gI₂/100g) obtained by Encinar *et al.*, [18] and Omohu and Omale [7].

4.6. Kinematic Viscosity

Kinematic viscosity is the most important property of oil because it affects the fluidity, lubricity and atomization of the fuel [21]. Fuels with low viscosity may not provide sufficient lubrication resulting in wear and high viscosity causes poor combustion and increases exhaust emission [22]. The kinematic viscosity of the crude castor oil as presented in Table Table 3.1 is 233 ± 2.00 mm²/s at 40°C, similar result of 224 mm²/s at 40°C was obtained by Rengasamy *et al.*, [22] and high results of 258.01 mm²/s at 40°C was observed by Encinar *et al.*, [23]. After refining, the viscosity dropped to 159.0 ± 3.00 mm²/s at 40°C. Reduction in the viscosity may be related to the removal heavy materials i.e. hydralable phospholipids, non hydralable phospholipids, metal salts etc. and impurities during refining process [7].

4.7. Specific Gravity

The specific gravity is a key fuel property, which affects the mass of fuel injected into the combustion chamber. This property directly affects the engine performance characteristic because the fuel injection pump meter works on by volume not by mass [22]. The specific gravity of crude castor oil was found to be 0.9555 ± 0.01 (Table 3.1) and significantly decreases to 0.9245 ± 0.00 after refining. Comparable result 0.961 of crude castor oil was obtained by

Encinar *et al.*, [23].

4.8. Flash Point

The flash point is the minimum temperature at which fuel gives momentary flash on ignition under specified test conditions. It is an important parameter for storage, handling and safety of the fuel. The flash point of crude castor oil was about 225.0 ± 1.00 °C which is lower than 286 °C that observed by Rengasamy *et al.*, [22] and almost similar with 229 °C ICSC (International Chemical Safety Cards) 1452 standard. After refining, the flash point decreases to 190 ± 1.00 °C which indicate increased in volatility of the oil. This indicates that both crude and refined castor oil are safe in handling and storage.

4.9. Cloud Point and Pour Point

The cloud point is the temperature at which dissolved solids are no longer completely soluble, precipitating as a second phase giving the fluid a cloudy appearance (Rengasamy *et al.*, [22]. While, the pour point is carried out to determines the freezing point of the sample. The cloud and pour point of crude castor oil obtained are 3.17 ± 0.31 and -24.0 ± 2.00 °C. Rengasamy *et al.*, [22] observed the cloud point of crude castor oil as 14 °C, While the Forero, [24] observed the pour point as -23 °C. The cloud point and pour point changed after refining process to 4.00 ± 1.20 and -13.1 ± 2.00 °C.

4.10. Refractive Index

This is the measure of the extent to which radiation is refracted on passing through the interface between two media. It indicates the clarity of the oil. The refractive index analysis show significant difference between the value 1.470 ± 0.00 of the crude and 1.443 ± 0.00 of the refined castor oil, this may be attributed to the fact that some impurities and other components were removed during refining Akpan *et al.*, [11]. Also, Akpan *et al.*, [11] observed differences between the value obtained for crude castor oil 1.4686 and that of the refined oil 1.467 .

5. Conclusion

The low temperature refining pretreatments was achieved at 85 °C which improved the physicochemical properties and efficiency of castor oil that can be used in biodiesel production and produces by products i.e. hydralable phospholipids e.g. phosphotidal choline, phosphotidal ethanalamine etc and non hydralable phospholipids which may be used as raw materials for some industries.

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