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A simple method for extraction of Ricinus Communis L oil and its application for biodiesel production by ethylic route

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Abstract

Biodiesel is a clean fuel, and an alternative to the environmental complications caused by fossil fuels. However, the forms of its production are still the object of research in the literature because some issues related to process improvement and economically viable remain a challenge. Therefore, this research aims to produce biodiesel by the ethylic route from *Ricinus Communis L* oil extracted with a simple solid liquid extraction method without the use of the soxhled extractor. In this sense, *Ricinus Communis L* was extracted by n-hexane as solvent, obtaining biodiesel through ethyl transesterification via alkaline catalysis. The characterization of biodiesel in terms of – acid value (AV), combustibility and corrosivity to copper. The results indicated that the yield of the extracted oil was 14% in relation to the mass of seeds used, the value of the AV was 0.96mgNaOH / g, showing in this case that the oil can be used in the transesterification process to obtain biodiesel. The biodiesel yield obtained was 37% and the AV was 1.98mgNaOH / g, being considered above the acceptable value for its use in diesel engines, and should be neutralized to at least 0.8mgNaOH / g. Based on this work, biodiesel was obtained via ethyl esterification of *Ricinus Communis L* oil extracted from a simple and easy method.

Keywords: Biodiesel, oil, *Ricinus Communis L*, extraction, transesterification.

1. INTRODUCTION

Biodiesel is fuel from biomass, this is renewable, clean, biodegradable, non-toxic, sulfur free and other advantages. For these reasons, is considered the promisor fuel to overcome most of challenges caused by fuel from crude oil [1]. Besides that, in its structure possess oxygen atoms resulting in enhancement combustion process. As consequence, the combustion reaction is complete, avoiding the production of high quantities of carbon monoxide (CO), unburned hydrocarbons and particulate matters [2,3]. Generally, biodiesel is produced from transesterification reaction of vegetal oils and oils. In addition, in the biodiesel production Fur glycerin is also produced as byproduct [4]. Several crops such as, sunset, coconut, and others are used as a source of biodiesel. However, the oils from abovementioned crops, are expensive and compete with areas for food productions. Thus, non-edible source of oils are the paramount for biodiesel productions. There are several types of non-edible crops for oil production [5]. The *Ricinus communis* L, belongs to the family Euphorbiaceae, nowadays can be found in most of tropical countries [6]. The seeds of *Ricinus communis*, are generally used for biodiesel productions, because allows an easy production [7][8,9]. Soxhalet and solvent extraction are commonly used for *Ricinus Communis L* oils production. The yields in terms of oils is better than other source such as (soyben: 15-20% (p / p), sunset: 25-35% (p p / p), colza: 38-46% (p / p) e palm: 30-60% (p / p)[10]. Furthermore, the biodiesel produced from *Ricinus communis L* oil presents properties that are in agreement with the quality standard of biodiesel[11]. Many methods are applied for biodiesel production, such as hydrodynamic cavitation. The hydrodynamic cavitation is interesting methods, because allows the biodiesel with yields between 40 to 80 % in only 100 minutes [12]. In addition, for biodiesel production two routes are the cited in the literature, ethylic and methylic. Generally, the methylic route is most used for biodiesel production from *Ricinus communis L* oil. The use of methanol is more common than ethanol because of its low cost and high reactivity, but there is a growing interest in the use of ethanol because it is renewable, non-toxic and is safe [13,14]. Methanol is generally added in higher proportions than the stoichiometric ratio (3: 1), to change the equilibrium position of the reaction towards the product side. However, this fact strongly increases the final cost of the process, due to the fact that expenses with methanol are higher[15].

Given those background, the present work aims to produce biodiesel by ethylic route using *Ricinus communis L* oil extracted applying a simple method.

2. MATERIALS AND METHODS

2.1. Materials

Ricinus Communis L oil was produced applying a simple method. *Ricinus communis L* seeds were collected in Lichinga, Mozambique, were also used in this process, n-hexane (Supra Solvs), 95% diethyl ether (Rochelle Chemical); 99.9% ethanol and 37% HCl-RPE , Brande: CARLO ERBA. The tests for oil extraction and transesterification were carried out at the chemistry laboratory of the Rovuma University, and at the Agrarian Institute of Lichinga respectively

2.2. Methods

Ricinus Communis L oil was extracted with a simple solid liquid extraction method without the use of the soxhled extractor.

2.2.1. Collection and treatment of *Ricinus communis L* seeds

The collection of the *Ricinus communis L* was carried out in the city of Lichinga. The *Ricinus communis L* seeds were harvested without regard to their varieties, it was a random collection, as the *Ricinus communis L* variety issue was not being studied. The seeds collected, firstly were peeled and dried at 25 °C. The dried and peeled seeds were weighted and dried in oven (Ø 3871112- 48/ 228 V – 758 W) at 70°C during 30 minutes. Afterward, the seeds, were toasted at 60 °C during 30 minutes and weighted again. After drying, 600g of dry seeds were weighed and crushed by mortar and pistil, until an oily and pasty mass was obtained.

2.2.2. Solvent extraction of *Ricinus communis L* oil

The seeds treated in the previous step was mixed with hexane in the proportion 1:2 (600g of seeds to 300 mL of n-hexane). The mixture was heated at 68 °C for 10 minutes and was kept for 15 hours. After 15 hours the oil mass was filtered through a paper filter

coupled to a funnel, using an Erlenmeyer flask to collect the oil. The mixture containing oil and solvent was submitted to a vacuum evaporator under constant heating during 50 minutes at 68 °C. Then, the oil was left to cool naturally and was kept in the glass container Figure 6c.

2.3. Biodiesel production

In this experiment 74g of *Ricinus communis L* oil was used to biodiesel production. Firstly, the castor oil was heated at 65-70 °C. In parallel experiment, an ethanolic solution containing 45mL of ethanol and 1 g of NaOH was prepared, using magnetic stirrer, then was necessary to elevate the temperature until 50 °C during 48 minutes. After the dissolution, of NaOH, the solution was added to *Ricinus communis L*. The process was kept under constant stirring for 2 hours at 70 °C.

2.4. Purification of the product obtained from transesterification

The mixture of biodiesel and glycerol was removed from the funnel and heated to 80 °C until it returned to a liquid state, after it had solidified. Subsequently, 2 mL of HCl was added to neutralize the catalyst used in the transesterification. Then the product was added back to the separating funnel and the biodiesel was separated from the glycerol. The biodiesel was washed with warm water until the washing water reached neutral pH. Having then calculated the biodiesel yield by equation 1.

$$biodiesel\ yield = \frac{M_{biodiesel}}{M_{oil}} \times 100\%$$

Being

M_{oil} = Quantity of oil extracted in grams

$M_{biodiesel}$ = Quantity of biodiesel obtained in grams (1)

2.4.1. Evaluation of physicochemical properties of biodiesel

2.4.2. Acid value

2.2.4. Determination of acid value of *Ricinus communis L* oil

The acid value of *Ricinus communis L* oil was determined in triplicate. About 4.30 g of *Ricinus communis L* oil was placed in an Erlenmeyer flask, with 25 mL of ethyl ether / ethyl alcohol in a 2: 1 (v / v) ratio, 3 drops of phenolphthalein and titrated with 0.1 KOH solution mol.L⁻¹. The same procedure was used to determine the acid value of biodiesel. The results obtained were compared with the values standardized in the standards described in Table 1. The acid value was determined using Equation 2

$$AV = \frac{(V_{NaOH} - V_{white}) \times C_{base} \times MM_{base}}{M_{sample}} \quad (2)$$

Being

AV- Acid value (mgNaOH / g)

V_{NaOH} - Volume (mL) of the NaOH solution spent on the titration

V_{white} = volume of the titrant solution in mL for titration of the blank,

C = concentration of the titrant solution in mol L⁻¹

M_{sample} = mass of the sample in grams.

2.4.3. Combustibility test

Four cotton pads were embedded separately with 3mL of *Ricinus communis L* oil (OR), 3mL of castor biodiesel (BR), 3mL of petroleum diesel (DP) and 3mL of the mixture of 20% castor biodiesel with petroleum diesel (B20), respectively; The wads were placed in porcelain crucibles (one crucible for each wad) identified in OR, biodiesel of *Ricinus communis L* (BR), DP and B20. A matchstick was used as a heat source to see which one would be flammable and which characteristics of its flame;

2.4.4. Corrosion test

Two copper wires were placed in beaker cups and the beakers were identified in BR and DP. 2 mL of *Ricinus communis L* biodiesel was pipetted into the BR beaker until the copper wire was soaked. Afterward, 2 mL of petroleum diesel was pipetted into the DP beaker until the copper wire was embedded. The mixtures were left to stand for 48

hours. After 48 hours the wires were removed from the beakers and compared with the new wires (not used in the test-control).

3. RESULTS AND DISCUSSION

3.1. Solvent extraction of *Ricinus communis* L oil

Figure 1 shows the results regarding to seeds mass variation after two drying cycles. According to Figure 1, after first drying procedure the seeds lost 8% (mass.) compared to fresh seeds. While, after second drying procedure the mass loss was 9% in relation with fresh seeds. These results are yet expected because the fresh seeds present humidity and after drying loss a part of humidity. Figure 2 shows a pasty mass after seeds pulverization. The formation of this pasty mass is due to high content of oil and the reduction particle size allowing the interaction between particles. Furthermore, this step is particularly important because it allows the interaction between the grounded seeds with hexane. Figure 3 presents the macroscopically aspect of pulverized seeds in contact with hexane. In Figure 3-A, the phase separations are observable, suggesting that the solubilization had not started due, to high viscosity of the mixture. Then, after the heating 80 °C, the phase separations disappeared, forming a homogenous like mixture (Figure 3-B).

After a 35-minute (Fig. 3-C), a separation into unstable oily phases could already be seen; these phases were stable and very visible when the mixture was left to stand for another 15 hours. The phenomena observed in (Figure 3-B and 3-C), is consequence of heating process that resulted in decrease of viscosity, leading to enhance the dissolution of oil in hexane[16]. As a result, accelerated the extraction process. Subsequently, Figure 3-D, presents three phases after additional 15hours. The phase 1 (from bottom to top) consisted of husks, thicker grains and contained less oil, phase 2 consisted of finer grains and contained a greater amount of oil compared to phase 1, and, finally, phase 3 consisted of oil and some seed grains.

Fig. 3-D shows that, in addition to the action of the solvent in this process, the granulometry also influenced a lot, since in each phase, particles with different granulometry were arranged, and the phase with the highest granulometry (finer particles) is higher concentration of oils, which is why it is necessary to use the press to obtain finer grains in relation to those obtained in grinding, allowing a greater

homogenization of the material to be extracted with the solvent, in addition to reducing the density of the flakes.

Fig. 4-A shows the upper part of the mixture being decanted into a filter paper attached to the long-stem funnel in order to start the filtration of the crude oil. In Fig. 4-B, it is observed that all phases are being filtered under the same conditions. Fig. 4-C shows the filtered oil being collected into a single container, as the filtration was done separately due to the size of the materials used and due to the difference in appearance at each stage of the mixture, believing that this would facilitate the filtration of each phase. The crude oil can be seen in Fig. 4-D.

When the flask containing the oil was left open for the natural evaporation of the solvent, there was a reduction in viscosity, resulting from the reduction of the solvent by evaporation, but the process was very slow (it took a week to notice the difference in viscosity) therefore, the oil was subjected to evaporation under heating. During heating, intense bubbles were formed (Fig. 5-A). These bubbles were the result of the solvent being heated above its P.E, so the bubbles represented the boiling solvent. The bubbles reduced with the passage of time, until they disappeared completely (Fig. 5-B), and the solvent elimination process ended. After removing the solvent, 75mL of oil was obtained whose weight was 88.76g. In this case, the oil yield was 14% in relation to the mass of the seeds used (Fig.5-C).

By the method used to eliminate the solvent, it cannot be concluded that there was total elimination of the solvent in the oil, the most important being to ensure that there is a minimum of substances that can interfere in the transesterification process. Evaporation under continuous heating can be a good option when the solvent elimination process is to be efficient. However, care must be taken with the solvent used in the extraction process and the temperature to be used for heating.

3.1. Biodiesel production

3.2.1. *Ricinus communis* L oil transesterification

The preparation of sodium ethoxide took 48 minutes, as the dissolution of sodium hydroxide in ethyl alcohol was very slow and the transesterification was maintained for 2 hours. During the addition of sodium ethoxide to the oil under heating and continuous stirring (Fig.6-C), there was an immediate change in the color of the oil, which changed

from light yellow to orange (Fig. 6- C and D). In addition, it was observed that the oil became lighter. After 2 hours of reaction, under constant stirring and heating, the process was stopped. When the product was placed in the separating funnel, it dried immediately, with no phase separation (Fig. 6-D). this is due to the high value of free fatty acids that this oil presents, which in turn can make it difficult to separate phases after the transesterification reaction and can still cause problems with the formation of soap (saponification)[17]

3.2.2. Biodiesel purification

The biodiesel was purified eliminating the excess of NaOH by titration. After the addition of 35% HCl with the purpose of neutralizing the remaining catalyst and removes the salts of the product obtained in the transesterification process[10]. In the transesterification product, effervescence was registered which ended with the appearance of a white liquid which was separated by decantation. The final yield was 37% in relation to the OR mass used in transesterification. The final product of this process can be seen in Figure 7-D. Effervescence was the result of the reaction between hydrochloric acid (HCl) and sodium hydroxide (NaOH) used as a catalyst in transesterification. The production of *Ricinus communis L* biodiesel requires must be controlled, as it involves several parameters for the yield to be satisfactory[18,19], the yields shown in the Table 3 refer to the conventional methods, which involves a conventional reactor and the method of extracting oil from the seed of *Ricinus communis L* oil, but for the research in question no conventional extraction method was used and the reactor used was of low cost, however the biodiesel yield obtained was considered satisfactory under the conditions of study.

3.3. Physiochemical characteristics

3.3.1. Acid value

The acid value of biodiesel was 1.98 mgNaOH / g, which shows an increase (Table 1). According to the norms of the ANP legislation, the established specification is that the acid value is between 0.1 to 0.5 mg NaOHg-1[21]. However, it is observed that, having

the acid value of the oil as well as the biodiesel obtained, they presented values outside the established norm due to their high content of ricinoleic acid [22].

3.3.2. Combustibility test

Table 2 presents the phenomena observed in the combustibility test of *Ricinus communis L* biodiesel, comparing with the combustibility of *Ricinus communis L* oil, with petroleum diesel, as well as the mixture between DP and BR. The flame intensity of the OR (*Ricinus communis L* oil) was extremely low compared to that of the BR (*Ricinus communis L* biodiesel) Table 2. It is suggested that these differences were influenced by the cetane number of each substance, which is a parameter directly associated with fuel combustibility, so with the transesterification there was an increase in the cetane number of *Ricinus communis L* oil (converted into BR)[10]. Generally, the cetane number of biodiesel is much higher than that of mineral diesel, which justifies the fact that biodiesel has a more controlled ignition and burns better in the engine than mineral diesel oil itself [15]. On the other hand, the BR, DP and B20 flame were similar, with the advantage that the BR flame was controlled. A Table 2, also presented that OR and DP showed a reduced flame duration than BR and BR 20. Being that the flame of the BR had a much longer extension and the OR much smaller in relation to another sample (B20 and DP), with that it can be said that the BR is economical from the point of view of consumption in the engine, when compared with the DP.

Table 2, show the smoke release behavior of all samples. There was a greater release of smoke in the burning of DP (oil diesel) and the mixture of DP and BR, having been lower in the mixture compared to pure DP, whereas in the BR it was verified a lower release of smoke and when burning the OR the smoke release was not noticeable, such aspects can be seen in Fig. 8.

Figure 8-D, presents the feature after burning process of oil diesel and *Ricinus communis L* biodiesel, until the end of the process, the capsules that contained DP and B20 showed a greater amount of carbon black compared to the capsule in which the BR was burned. BR, the presence of carbon black was practically not verified at the end of the process, a fact that demonstrates the advantage of using BR in terms of the impacts of emissions when compared to DP. In this way, this observations are in agreement

with works that reported in their studies that fuels like, ethanol and biodiesel release less pollutants than crude oil derivatives [23].

3.3.3. Corrodibility

The corrosibility of *Ricinus communis L* biodiesel was perfumed using copper as a model metal (Figure 8-C). The BR presented a green color after 48hours of copper. However, the DP did not change the color, but the copper was corroded. The BR changed the color, due the oxidation reaction caused by copper [24]. The BR possess double bonds in the structure that can easily break, originating reactive radicals that can react with Cu^0 resulting in Cu^{2+} (green color). For these reasons, additives are necessary to inhibit the oxidative [25]. It can be seen from Fig. 9-C, that DP did not change in colour, but the wire that was dipped in the DP turned black (Fig.9-B), showing stability from the point of view of corrosion stability.

4. Conclusions

Solvent extraction is a simple process that avoids oil contamination, in addition to allowing solvent recovery. In this work, extraction with n-hexane proved to be efficient, since a yield of 14% was obtained in relation to the mass of seeds used in the process and the acid value (0.69 mg NaOH/g), considering within standards for the transesterification process (≥ 3 mg NaOH / g). Biodiesel was obtained through the ethyl route with a yield of 37% in relation to the *Ricinus communis L* oil mass, with an acid value of 1.98 mgNaOH / g, being outside the parameters of ANP 045, with the need to correct the acidity as that this oil has a high index of ricinoleic acid. The combustibility of *Ricinus communis L* biodiesel was better than petroleum diesel. However, by the corrosivity test, biodiesel proved to be very unstable, having suffered a degradation that resulted in the oxidation of the copper wire. The results of the acid value of *Ricinus communis L* biodiesel are in agreement with its effect on corrosivity under the same study conditions.

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Compliance with ethical standards

Conflict of interest: The authors declare that they have no conflict of interest.

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Table 1: Comparison between the Acid value of the biodiesel obtained and the pre-established value in different countries.

Table 2: Results obtained in the combustibility test of castor biodiesel

Table 3: Comparison of different yields of *Ricinus communis* oil and biodiesel

Figure 1: Seed weight at different drying stages

Figure 2: Product of crushing castor seeds

Figure 3: Castor oil extraction process. Aspects of the mixture before heating (a), after heating (b), 35 minutes after (c) and 15 hours after (d).

Figure 4: Process of filtering the mixture. Filtration (a, b), collection (c), castor oil collected (d).

Figure 5: Solvent elimination process in the oil. Beginning of the solvent elimination process (a), end of the process (b), final castor oil (c).

Figure 6: Ethical transesterification of castor oil. Heating the oil (a), dissolving NaOH in ethanol (b), adding sodium ethoxide to the preheated oil (c), mixing during the reaction (d); phase separation (e).

Figure 7: Purification of the final product. Washing process (a-c); Final product obtained (d).

Figure8: Combustibility test of castor biodiesel. Combustion starts (a), after 1 minute and 44 seconds (b); at 2 minutes (c); after the end of combustion (d).

Figure 9: Corrosion test of castor biodiesel. Copper wires before dipped in BR and DP (a); right after dipping (b); 48h later (c).

Figures



Figure 1

Seed weight at different drying stages



Figure 2

Product of crushing castor seeds

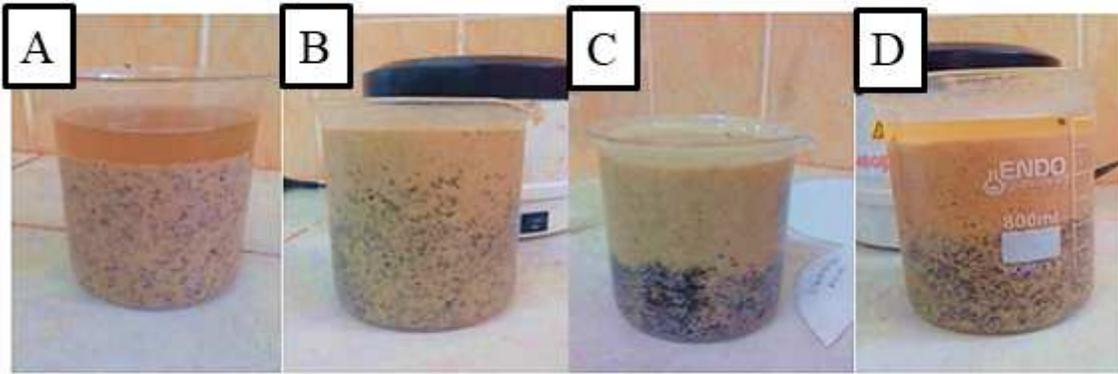


Figure 3

Castor oil extraction process. Aspects of the mixture before heating (a), after heating (b), 35 minutes after (c) and 15 hours after (d).

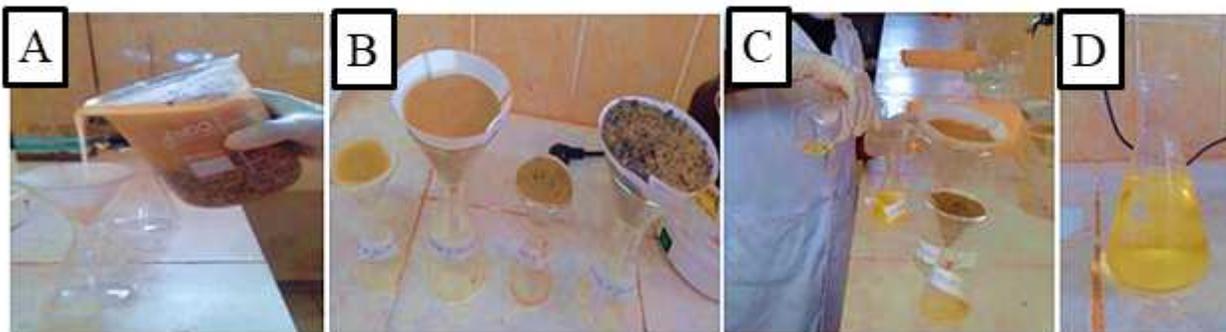


Figure 4

Process of filtering the mixture. Filtration (a, b), collection (c), castor oil collected (d).



Figure 5

Solvent elimination process in the oil. Beginning of the solvent elimination process (a), end of the process (b), final castor oil (c).

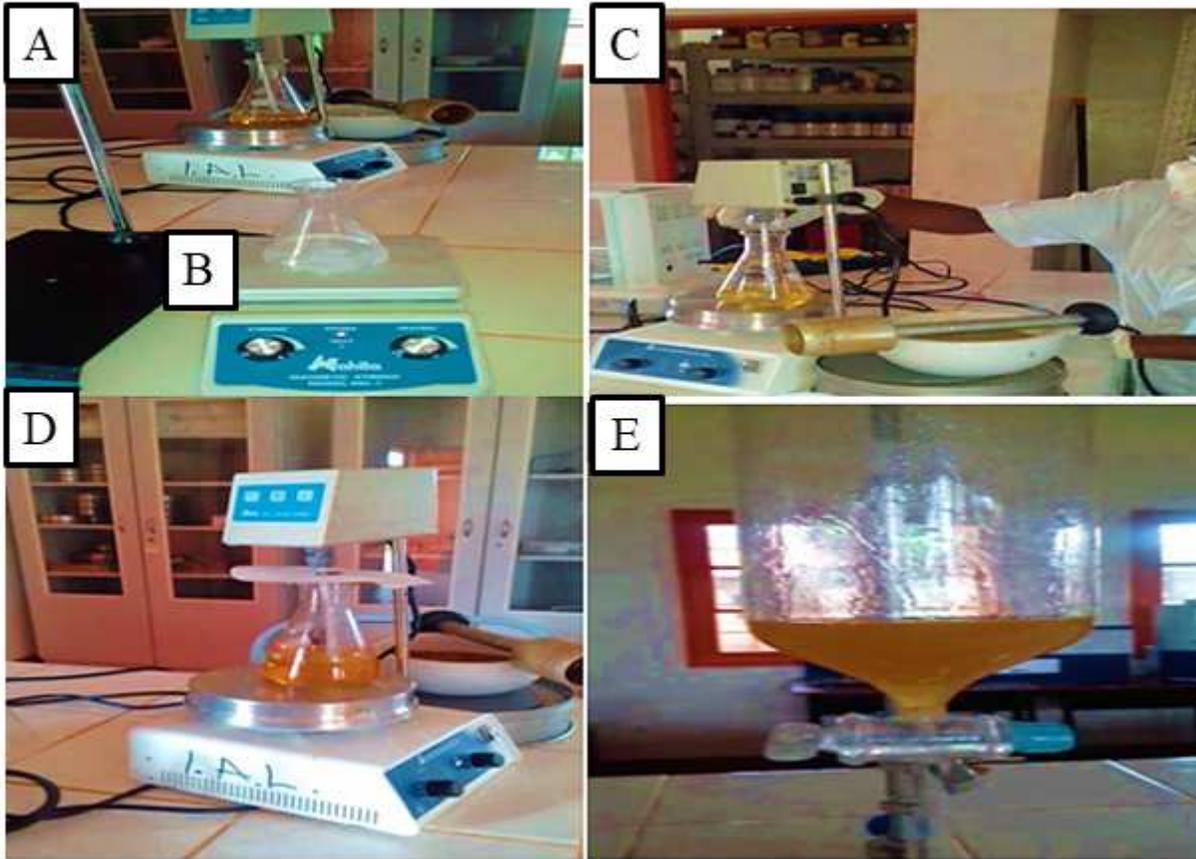


Figure 6

Ethical transesterification of castor oil. Heating the oil (a), dissolving NaOH in ethanol (b), adding sodium ethoxide to the preheated oil (c), mixing during the reaction (d); phase separation (e).

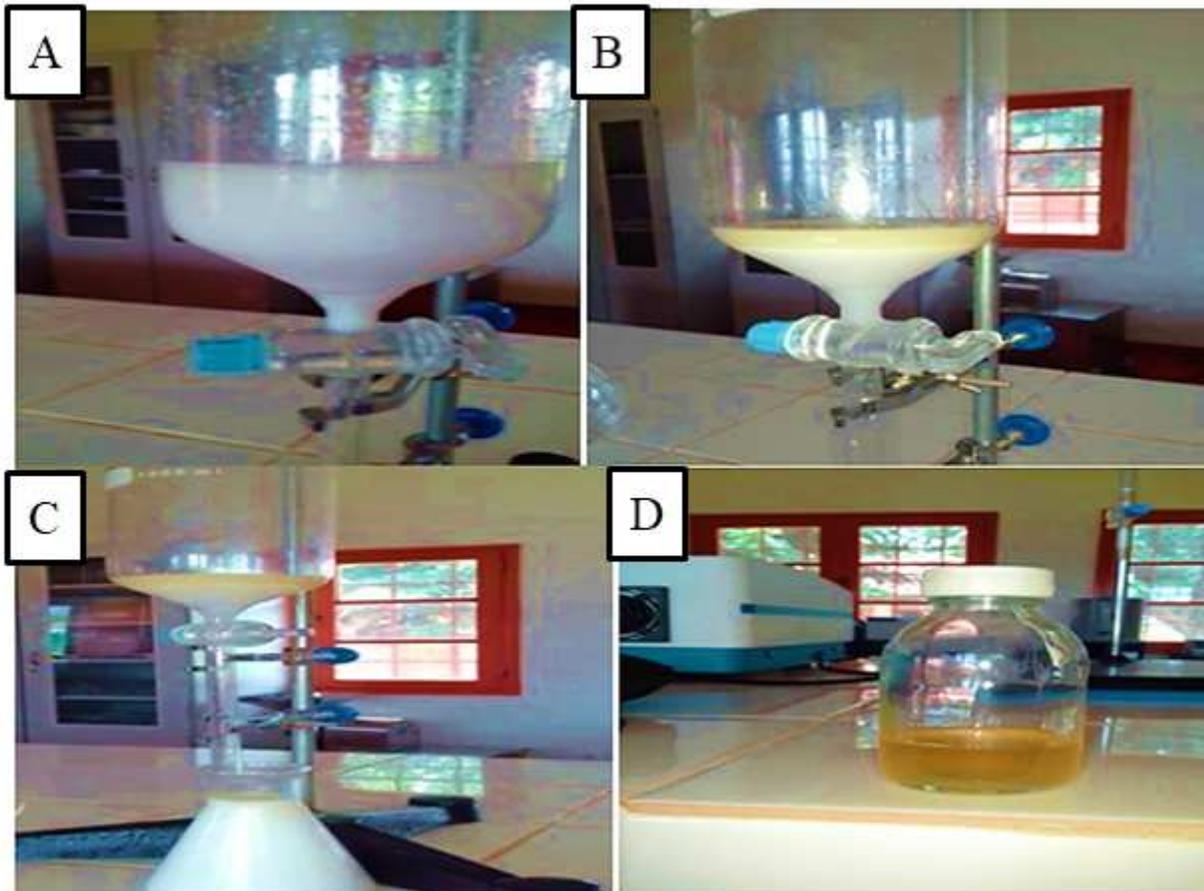


Figure 7

Purification of the final product. Washing process (a-c); Final product obtained (d).

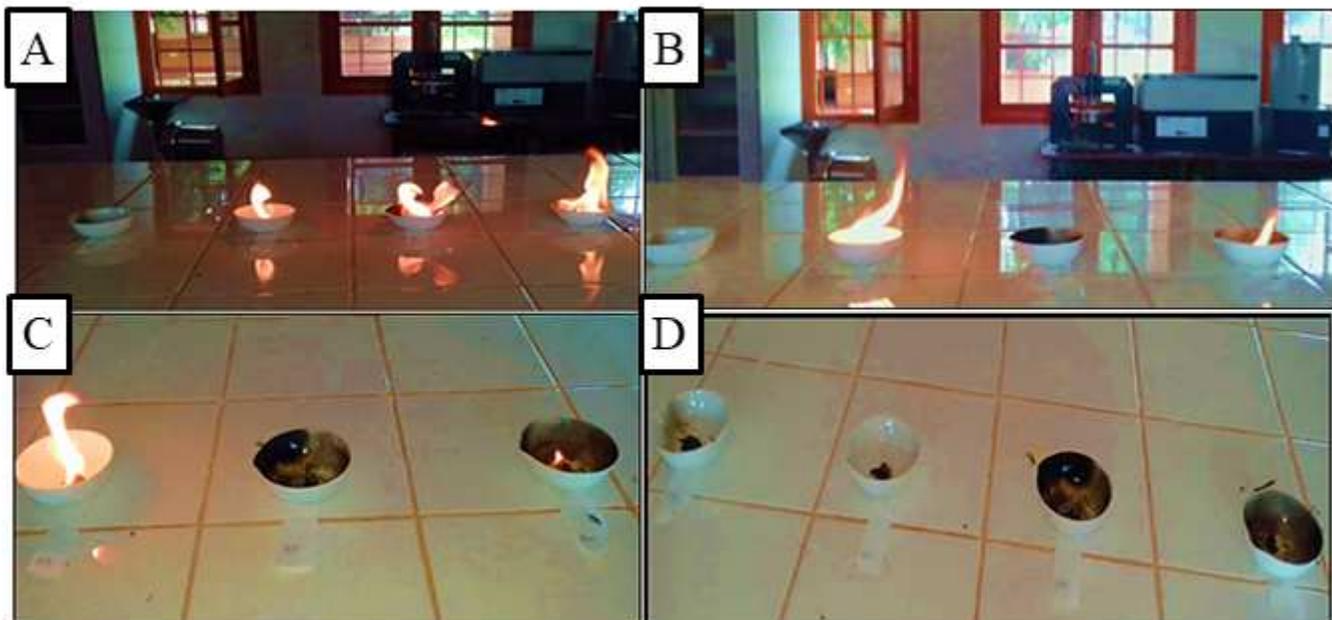


Figure 8

Combustibility test of castor biodiesel. Combustion starts (a), after 1 minute and 44 seconds (b); at 2 minutes (c); after the end of combustion (d).

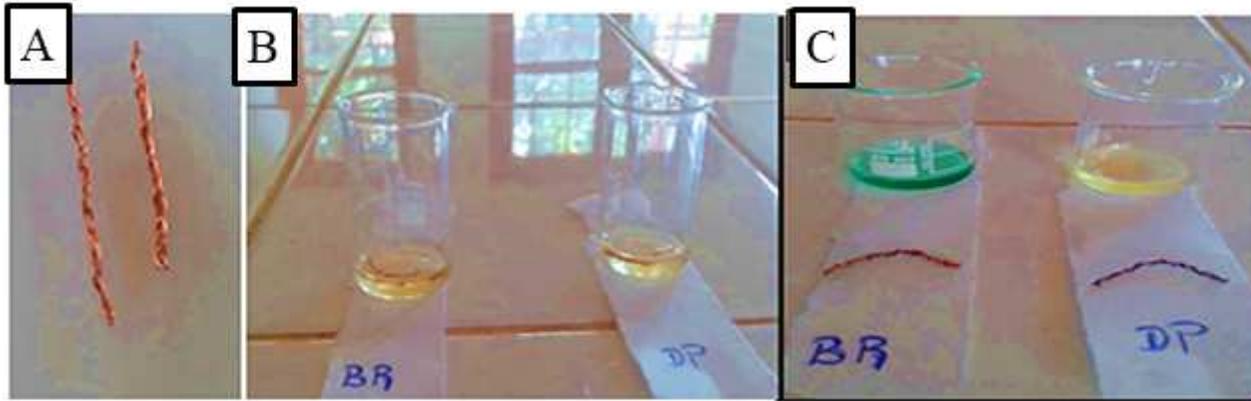


Figure 9

Corrosion test of castor biodiesel. Copper wires before dipped in BR and DP (a); right after dipping (b); 48h later (c).

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Table1.docx](#)
- [Table2.docx](#)
- [Table3.docx](#)