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Production of Biodiesel from *Jatropha Curcas* Seed Oil using Base Catalysed Transesterification

Fahad Ahmed¹, Saidat Olanipekun Giwa^{2*}, Maryam Ibrahim³ and Abdulwahab Giwa⁴

^{1,2,3}Chemical Engineering Department, Faculty of Engineering and Engineering Technology, Abubakar Tafawa Balewa University, Tafawa Balewa Way, Bauchi, Nigeria

⁴Chemical and Petroleum Engineering Department, College of Engineering, Afe Babalola University, KM. 8.5, Afe Babalola Way, Ado-Ekiti, Ekiti State, Nigeria

Abstract : This work has been carried out to produce biodiesel and investigate how the production from *Jatropha curcas* seed oil catalysed by potassium hydroxide is affected by some factors. The factors considered were catalyst load, methanol to oil ratio and reaction time. Before the production of the biodiesel was accomplished, *Jatropha curcas* oil, which was analysed to ascertain its suitability for the production, was extracted using solvent extraction method with n-hexane as the solvent. The results obtained showed that a methanol to oil molar ratio of 4 with KOH concentration of 2.5% w/w and 75 min reaction time gave the optimum yield of biodiesel. Biodiesel yield was found to reduce with increasing KOH concentration and higher methanol to oil ratio. That was because at higher methanol to oil ratio, the excess methanol was reacting with the KOH to form soap instead of speeding up the production of biodiesel. Also, the important properties of the biodiesel like the flash point, acid value, kinematic viscosity, iodine value, density and saponification value were found to be 156 °C, 4.77 mgKOH/g, 2.02 cm/s, 48.85 meq/g, 0.874 g/cm³ and 115.83 mgKOH/g, respectively. These values obtained were compared with specifications of ASTM D6751, and it was established that the *Jatropha curcas* seed oil methyl ester could be used as an alternative to/or blended with petrodiesel.

Keywords: Biodiesel, *Jatropha curcas*, transesterification, solvent extraction.

1.0 Introduction

Increased population size, increase energy demand, depletion of fossil fuel reserves couple with the environmental problems associated with fossil fuel usage have led to the search for alternative fuels which can be obtained from renewable source [1]. Fatty acid methyl esters (FAME) collectively known as biodiesel obtained from renewable vegetable oils such as *Jatropha curcas* seed oil is an alternative fuel for diesel engines [2].

Nigeria, being a tropical country, has a wide variety of domestic plant that produces oil-bearing seeds of sufficient volume potential and has one of the most extensive floras in Africa [3]. Oil-seed processing expands the use of crops and also brings values to waste products [4]. Vegetable oils derived from plant seeds have been playing vital roles in providing comfort to human lives in various aspects. Outside the realm of food manufacture, vegetable oils feature in variety of industrial uses ranging from the manufacture of soap to production of paints, varnishes, lubricants and plastics. The concern for fast depletion of petroleum oil and its

environmental impact has shifted interest to alternative sources of fuels, particularly biofuels, which are renewable and environmentally friendly ([5],[4]).

In the last few decades, there have been growing concerns over vegetable oils as source of material in preference to petroleum or mineral oil. The choice of biodiesel over diesel fuels includes its portability, availability, renewability, inherent lubricity, lower sulphur and aromatic contents ([6],[4]).

Biodiesel is a non-polluting, locally available, accessible, sustainable and reliable fuel obtainable from renewable sources such as vegetable oil or animal fats by transesterification [7]. It has an energy content of about 12% less than petroleum-based diesel on a mass basis [8].

Biodiesel is a biodegradable, renewable fuel that can be produced from a range of organic feedstock including fresh or waste vegetable oils, algae, oilseed plants and animal fats. Biodiesel has lower emission compared to petro diesel when burnt (either blended or pure). It does not contribute to the rise in carbon dioxide level in the atmosphere and it minimizes the intensity of greenhouse effect [9]. It can be directly used to replace petroleum diesel without modifying diesel engines since their properties, e.g., specific gravity, cetane number, viscosity, cloud point, and flash point, are similar ([10], [11], [12], [13]). As mentioned before, biodiesel is a renewable energy source [14,35-48] that has superior properties than that of petro-diesel fuel ([15], [16]) such as nontoxicity [17]. The research involving the production of fatty acid methyl esters are being embarked on nowadays because it is very important for today's world to identify an alternative to fossil fuel to meet the future demands for energy ([18], [19], [20]), based on the fact that diesel fossil fuel reserves dwindling and at a time will run out [21], especially for use in internal combustion engines, which reduce the peak flame temperature and thereby reduction in various emissions [22], as it (biodiesel) is an alternative fuel that can be prepared from renewable biological sources such as vegetable oils both (edible and nonedible oil) and animal fats ([23], [24]). The production of this biodiesel can be achieved through the use of a non-edible oil known as *Jatropha curcas* seed oil.

Jatropha curcas seeds contain 27-40% oil [25] that can be processed to produce a high-quality biodiesel fuel that is usable in a standard diesel engine, especially if the oil of the seeds is well extracted.

Many oil extraction methods are available base on accuracy, cost effectiveness, kind and nature of feedstock and efficiency. The methods include mechanical pressing, solvent extraction and supercritical fluid extraction. The mechanical pressing (expulsion) process involves the use of pieces of equipment like screw press or piston, extruder, expander and mortar. In this process, oil is expelled from a dry feedstock. The main drawbacks of this process are particle size and nature of feedstock. Also, the moisture content of the seeds has a role to play in the efficiency of the process. Possible innovation in the future could help to overcome the inefficiency of mechanical pressing. Nevertheless, mechanical pressing could still be applied to both small scale and large scale oil production for production of biodiesel. In solvent extraction, oil can be extracted chemically using solvent such as n-hexane, chloroform, and benzene. N-hexane is the most commonly used solvent that mixed with the feedstock paste and then, later, distilled in order to obtain pure oil [26]. This method is not expensive, and it is efficient. The oil, after extracted from the seeds, can be passed through a chemical process to yield biodiesel. The chemical process of converting oil to biodiesel is referred to as transesterification.

Transesterification is a process that replaces an alcohol functional group from an ester with another one by reacting vegetable oils with alcohol in the presence of a catalyst. This process converts oil into biodiesel. Suitable alcohol that are widely used for this purpose include methanol, ethanol and propanol, but, most frequently, the use of methanol or ethanol is most common [5]. The factors affecting the process (transesterification) include the molar ratio of alcohol to oil, the concentration of catalyst, the reaction time, the reaction temperature, the free fatty acid and the water content in oils or fats.

Biodiesel production is one of the current areas of research in academics because of the serious search for alternative fuel. Based on that, researchers have reported many pieces of work investigating biodiesel production from different seed oils and kernels via acid and based catalyzed transesterification. One carried out by [27] focused on the effect of temperature and mixing rate variation on biodiesel production from *Jatropha* using sodium hydroxide catalyst and methanol. In the work, high temperature and high mixing intensity were found to increase the rate constants of the process, and the reactions were also found to be governed by a second order rate equation. Although the mixing intensity was affected by time, it was observed to be unnecessary after 30 minutes. Also, [28] carried out another work carried out using the same catalyst (sodium hydroxide) and

discovered that the best combination of the parameters for production of biodiesel from *Jatropha curcas* were 6:1 molar ratio of methanol to oil, 0.92% NaOH catalyst, 60°C reaction temperature and 60 min of reaction time. The characteristics of the biodiesel produced like density, viscosity, flash point, cloud point and pour point were found to be very comparable to those of diesel. Another research carried out by [29] on kinetics of biodiesel production from *Jatropha curcas* seed oil by varying molar ratio of oil to methanol, reaction temperature and catalyst revealed that the optimum yield of the methyl ester could be achieved with the oil to methanol molar ratio of 1:16 and KOH catalyst concentration of 1.5%. Research work performed by [30] revealed that using KOH as catalyst, 70% conversion of Karanja oil to biodiesel could be achieved with optimum parameters of molar ratio of 6:1 at atmospheric pressure and reaction time of 120 to 150 min. A study carried out by [31] to investigate the potentials of *Jatropha* and castor oil for biodiesel production showed that that *Jatropha* gave higher biodiesel yield than castor oil. It can be noticed that most of the reported pieces of work focused more on the use of sodium hydroxide as the catalyst for the transesterification process.

From the literature review carried out, it has been discovered that few pieces of research work have been reported concerning investigating the effects of potassium hydroxide (catalyst) load, methanol to oil ratio and reaction time on the yield of biodiesel. In order to bridge this gap, this research has been carried out to produce biodiesel from *Jatropha curcas* seed oil using potassium hydroxide catalysed transesterification process. In order to achieve this aim, extraction of the oil from *Jatropha curcas* seed using solvent extraction method, characterization and evaluating the physicochemical properties of the oil, production of the biodiesel by transesterification, and characterization and evaluating the physicochemical properties of the methyl ester produced.

2.0 Methodology

2.1 Oil Extraction

In preparation for oil extraction from the *Jatropha* seeds, they (the seeds) were sun dried, shelled and weighed. After then, they were sun dried again, ground and the weight of the ground seeds was taken. Solvent extraction method, using n-hexane as the solvent, was employed in extracting the oil from the ground seed meal. The choice of n-hexane as the extraction solvent was owing to the fact that it is non-poisonous and volatile with high affinity for oil. Besides, it can be easily recovered. At the end of oil extraction, the extract was filtered and the solvent was recovered using a rotary evaporator. The oil was further evaporated in an oven at 105 °C to eliminate residual solvent and moisture content. The percentage yield was then calculated using the relationship given in Equation (1).

$$\%Yield = \frac{Weight\ of\ oil}{Weight\ of\ sample} \times 100\% \quad (1)$$

2.2 Determination of the Characteristics of the Oil

The oil extracted from the *Jatropha* seeds was characterized and its physicochemical parameters were determined to be sure that it would be suitable for biodiesel production.

2.3.1 Saponification value determination

To determine the saponification value of the extracted oil, the British standard method was used. Based on the method, 2.0g of the oil was placed in a 250ml conical flask and 25ml of 0.5 Nethanolic potassium hydroxide solution was added. A reflux condenser was attached and the flask content refluxed for 30 min on a water bath with continuous swirling until it simmered. The excess potassium hydroxide was titrated against 0.5N hydrochloric acid using phenolphthalein indicator while still hot. A blank determination was carried out under the same conditions. The saponification value was then calculated using Equation (2).

$$Saponification\ Value = \frac{(B_1 - R_1) \times 28.05}{Weight\ of\ oil\ sample} \quad (2)$$

where B_1 and R_1 are the volumes hydrochloric acid used for blank and sample titrations respectively.

2.3.2 Iodine value determination

Hanus method was used for the determination of the iodine value of the oil. This was carried out by placing 0.1 g of the oil in a 250ml conical flask, adding 10ml of anhydrous chloroform followed by the addition of 30ml of Hanus solution. The content was mixed and placed in a drawer for exactly 30 min. Thereafter, potassium iodide solution (10ml of 15% weight volume) was added to the flask in order to wash down any iodide at the stopper that was used for the flask. The entire content was titrated against 0.14 M $\text{Na}_2\text{S}_2\text{O}_3$ until the solution turned light yellow. After that, 2 ml of 1% starch indicator was added and the titration continued until the blue colour formed finally disappeared. A blank determination was carried out under the same condition, and the titre values were recorded. Using Equation (3), the iodine value was estimated.

$$\text{Iodine Value} = \frac{(B_2 - R_2) \times \text{Normality of } \text{Na}_2\text{S}_2\text{O}_3 \times 12.69}{\text{Weight of oil sample}} \quad (3)$$

In Equation (3), B_2 and R_2 are the volumes of $\text{Na}_2\text{S}_2\text{O}_3$ used for blank and sample titrations respectively.

2.3.3 Free fatty acid (FFA) determination

4.0g of the oil was placed in a 250ml conical flask and warmed. 25 ml of methanol was added with thorough stirring, followed by two drops of phenolphthalein indicator and a drop of 0.14N of sodium hydroxide solution. The content was titrated until a permanent light pink colour, which persisted for one minute, was obtained. The end point was thus recorded. The FFA value was calculated using Equation (4).

$$\text{FFA} = \frac{\text{Titre value} \times N \times 28.2}{\text{Weight of oil sample}} \quad (4)$$

where N is the normality of NaOH

2.3.4 Acid value determination

The acid value of the oil extracted from the *Jatropha* seeds was determined using the expression given in Equation (5).

$$\text{Acid Value} = \text{FFA} \times 1.99 \quad (5)$$

2.3.5 Peroxide value

The peroxide value of the oil was determined by first placing 1.0g of it in a 250 ml conical flask and adding 30ml glacial acetic acid/chloroform (3:2 v/v). The mixture was shaken until it became homogenous after which 1 ml of saturated potassium iodide solution and 0.5ml starch indicator solution were added. The content was titrated against 0.1N sodium thiosulphate until the dark blue colour given disappeared. Blank determination was also carried out, and the peroxide value was calculated from Equation (6).

$$\text{Peroxide Value} = \frac{(B_2 - R_2) \times \text{Normality of } \text{Na}_2\text{S}_2\text{O}_3}{\text{Weight of oil sample}} \quad (6)$$

In Equation (6), B_2 and R_2 are the volumes of $\text{Na}_2\text{S}_2\text{O}_3$ used for blank and sample titrations, respectively.

2.4 Biodiesel Production

Transesterification process, which is the process of converting extracted oil into biodiesel, was carried out in this work by reacting the extracted *Jatropha* oil with methanol in the presence of potassium hydroxide (which has been reported to give high yield and conversion of the transesterification reaction with minimal side reaction) as a catalyst to produce ester and glycerol. At the end of the reaction, glycerol and biodiesel formed two layers. After settling, the glycerol was at the bottom while the biodiesel was at the top of the container used. The layers were later separated from each other by draining the glycerol from the bottom of the flask containing the mixture. The initial triglyceride content of the oil was determined by titrimetric analysis. Keeping reaction

temperature and stirring rate constant at 65 °C and 460 rpm respectively, three different biodiesel production conditions were varied. Firstly, transesterification was done keeping the molar ratio of oil to methanol at 4:1 and 2.5% catalyst concentration and varying reaction time. Secondly, keeping the reaction time at 75 minutes, 4:1 molar ratio and varying the catalyst load, another set of biodiesel was produced. Thirdly, the methanol to oil molar ratio was varied while keeping constant the reaction time at 75 min and the catalyst concentration at 2.5%. Thereafter, another biodiesel production was carried out using the obtained optimum parameters from the investigations carried out before, and the yield of that was determined by titrimetric analysis and appropriate calculations.

2.5 Characterization of the Biodiesel

The biodiesel produced after the transesterification process was carried out was characterized to obtain its physicochemical parameters as outlined below.

2.5.1 Flash point

150ml conical flask was filled to a certain level with the biodiesel and heated at slow constant rate on a hot plate. The flash point was taken as the lowest temperature when the application of the test flame caused the vapour above the biodiesel sample to ignite.

2.5.2 Pour point

A cylindrical test tube was filled with the produced biodiesel to a certain level and attached to a wooden clamp bearing a thermometer. The sample was then allowed to cool below 0 °C in an ice bath. Thereafter, it was removed and tilted on the clamp and the set up observed at intervals. The lowest temperature at which the product was observed to flow was recorded as its pour point.

2.5.3 Cloud point

A cylindrical tube was filled with the biodiesel to a certain level and clamped with a wooden clamp bearing thermometer. The sample was allowed to cool below 0°C in an ice bath until its colour appearance turned white/cloudy. The temperature at which this cloudy appearance colour occurred was recorded as the cloud point of the biodiesel.

2.5.4 Viscosity

The produced biodiesel sample was poured into a long necked funnel with a finger placed at its bottom. A timer was set and started as the finger was released. When the sample ran off completely, the timer was stopped. The analysis was repeated four times and the average time was taken. The viscosity was thereby calculated using the readings taken.

2.5.5 Density

A conical flask was weighed and filled to a volume of 15ml with the biodiesel. The weight of the glass and the biodiesel was measured also. The weight of the biodiesel was determined, and the density was calculated as mass per unit volume.

3.0 Results and Discussion

The results obtained from the calculation of the yield of oil extracted revealed 54% of oil could be obtained from the *Jatropha* seeds used. The value was found to be good for a material like the *Jatropha* seeds.

Also, given in Table 1 are the properties of the extracted oil obtained from the analysis carried out. According to the results, the values obtained from the analysis of the oil especially free fatty acid, density and kinematic viscosity of the oil were found to compare well with the standard (ASTM), which was an indication that the extracted oil was good and suitable for biodiesel production.

Table 1. The physicochemical properties of the extracted oil

Properties	Oil
Acid value (mgKOH/g)	4.77
Saponification value (mgKOH/g)	115.83
Iodine value (mEq/g)	119.1
Free fatty acid (mgKOH/g)	2.3
Density (g/cm ³)	0.874
Kinematic viscosity (cm ³ /s)	4.57

From the properties for which the oil was analysed, the most important ones are free fatty acid (FFA) and acid value because they are the ones that are normally considered before and after biodiesel production [32]. Besides, they are those properties that are usually used to justify the suitability of an oil type for biodiesel production. In this case, as mentioned before, from Table 1, the acid value and %FFA of the extracted were found to meet the required specifications.

Also observed from Table 1 was that the saponification, peroxide and iodine values of the oil were found to be very good for an oil type that will not form soap during the process of biodiesel production. In addition, those values were discovered to compare very well with the values obtained in the in the work of [33].

After ascertaining that the extracted *Jatropha* oil was suitable for biodiesel production, it was employed in a transesterification reaction for the process and the results of the transesterification processes carried out are as outlined below.

3.1 Transesterification Process Using Literature Values

Using the transesterification conditions obtained from the work of [29_18], that is, methanol to oil molar ratio of 8:1, 1% sodium hydroxide concentration, 60 min reaction time and 65°C reaction temperature, even after two days of carrying out the experiment, no production of biodiesel was observed (see Figure 1). The reason for this was found to be as a result of the difference in seed used for the oil and type of catalyst used. It, therefore, means that some particular experimental conditions may not work at some instances especially if the reaction environments are different. That is why it is always good to obtain the working conditions for a particular reaction based on the materials and the environment being considered. As such, in this work, the parameters required for the biodiesel production was investigated by varying the parameters involved in the process.



Figure 1. The ‘product’ obtained from first transesterification process using the conditions of given in the work of [33]

3.2 Transesterification Process Carried Out by Varying Catalyst Load

Shown in Figure 2 are the results obtained when the catalyst load was varied while the other parameters were fixed. From the results given in Figure 2, it was discovered that the highest yield of biodiesel was obtained from the process when the catalyst load was 0.325g. At that point, it was found that the transesterification reaction activation energy was best reduced by the catalyst, and that caused speeding up of the reaction to give high yield of the product (biodiesel).

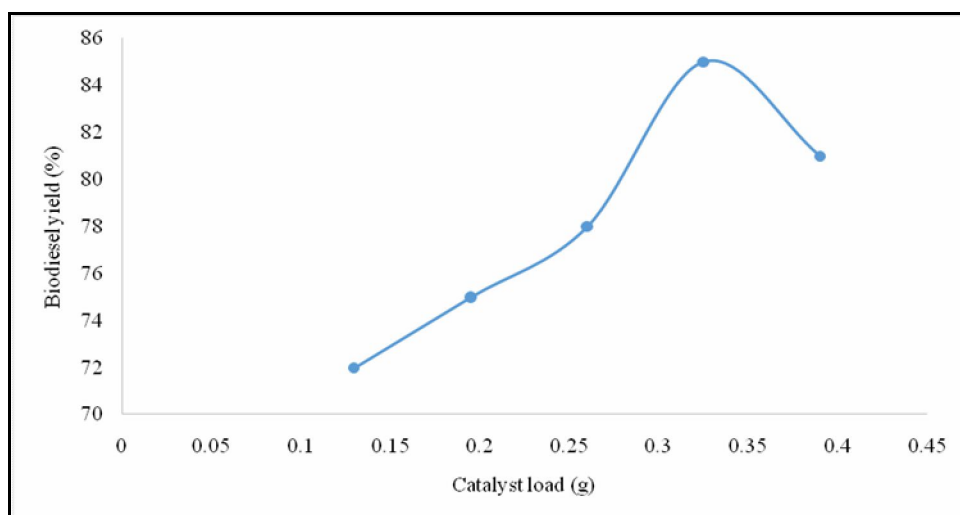


Figure 2. Biodiesel yield versus catalyst load ($T = 65^{\circ}\text{C}$, $\text{MOR} = 4$, $t = 75 \text{ min}$, $V_{\text{oil}} = 15 \text{ mL}$)

3.3 Transesterification Process Carried Out by Varying Methanol to Oil Ratio

Figure 3 shows the variation of biodiesel yield as the methanol to oil ratio was varied. Based on the information obtained from the table, the yield of biodiesel obtained from the process was found to be very low when the methanol to oil ratio used were 3 and 10, even though the global minimum of the yield was given with the ratio of 10.

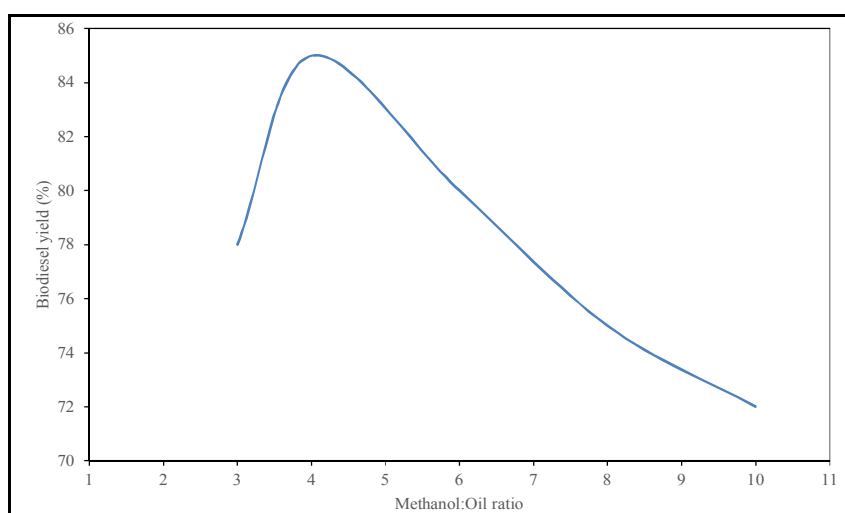


Figure 3. Graph of biodiesel yield against molar ratio (Catalystload = 0.325 g, $T = 65^{\circ}\text{C}$, $t = 75 \text{ min}$, $V_{\text{oil}} = 15 \text{ mL}$)

Also discovered from the figure was that the highest yield of biodiesel was given with the ratio of methanol to oil being 4. The results obtained have indicated that the molar ratio beyond 4 would not be very favourable to the process. In other words, the ratio of methanol to oil should not be made too high for proper conversion because the stoichiometric methanol to oil ratio is actually 3.

3.4 Transesterification Process Carried Out by Varying Reaction Time

In order to obtain the results given in Figure 4, the reaction time of the process was varied from 30 to 90 min while the other parameters were kept constant (molar ratio of methanol to oil was fixed at 4, the reaction temperature was 65°C while the catalyst load was 0.325g). It was discovered from the table that the yield of biodiesel obtained at low reaction time, for example, 30 min, was low due to the fact that the reaction could not get to completion within that time. All in all, the maximum yield of the biodiesel was found to occur when the reaction time of the process was 75 min. However, operating the process beyond 75 min resulted in a decrease in the yield. The reduction in the yield observed when the reaction time was very high (that is, greater than 75 min) was due to the fact the backward reaction of the process was being favoured then. As such, it is very good to maintain the optimum reaction of the process so as to get high yield of the desired product.

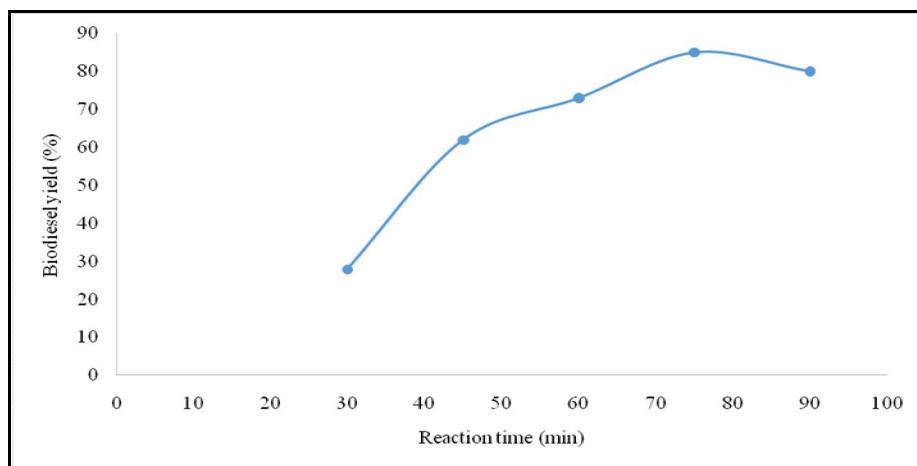


Figure 4. Variation of biodiesel yield with reaction time (Catalyst load = 0.325 g, MOR = 4, T = 65 °C, V_{oil} = 15 mL)

3.5 Transesterification Process Carried Out Using the Optimum Conditions

Using the obtained optimum values, which were reaction time of 75 min, catalyst load of 0.325 g, methanol to oil molar ratio of 4 obtained from the experiments carried out by varying those parameters affecting the yield of biodiesel produced from the extracted Jatropha oil with a reaction temperature of 65 °C, another experiment was carried out to produce biodiesel using those optimum values of the factors, and it was discovered that those values used could give 86% yield of biodiesel. The biodiesel produced using the optimum conditions was then analysed to obtain its physicochemical properties, and the results of the analysis were as given in Table 2.

Table 2. The physicochemical properties of the produced biodiesel

Properties	Biodiesel
Acid value (mgKOH/g)	0.413
Saponification value (mgKOH/g)	109.23
Iodine value (mEq/g)	105.3
Free fatty acid (mgKOH/g)	0.2065
Density (g/cm ³)	0.870
Kinematic viscosity (cm ³ /s)	4.2
Flash point (°C)	156
Pour point (°C)	<1
Cloud point (°C)	-9

Looking at the results given in Table 2, it could be observed that conversion of the Jatropha oil into biodiesel has been occurred because the analysis of the extracted Jatropha oil gave its free fatty acid to be 2.3

mgKOH/g while the produced biodiesel has been characterized to have 0.2065 mgKOH/g of free fatty acid. That was found to be an indication that high conversion of the triglycerides of the oil has been achieved.

Furthermore, the comparison of the other properties of the produced biodiesel with the literature values obtained from the work of [34] revealed that the produced liquid was, actually, biodiesel because the density, the cloud point and the flash point of the produced biodiesel, which were obtained to be 0.870 g/cm³, -9 °C and 156 °C were found to compare very well with the literature values that are 0.85-0.88 g/cm³, -3 to 12 °C and 130 to 170 °C, respectively.

4.0 Conclusion

The results obtained from this research work has clearly demonstrated that *Jatropha curcas* seed oil can be used as a feed to produce high quality biodiesel because it has been applied for that. Also, after characterizing, the properties of the produced biodiesel obtained from the *Jatropha* seed oil were found to fall within the literature standards. Furthermore, in addition to a reaction temperature of 65 °C, the variations of the parameters of operation of the transesterification process considered in this work has also shown that the optimum parameters at which high yield of biodiesel could be achieved were catalyst load of 0.325 g, reaction time of 75 min and methanol to oil ratio of 4.

Nomenclature

MOR	Methanol to oil ratio
t	Reaction time (min)
T	Temperature (°C)
V _{oil}	Volume of oil used for transesterification (mL)

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