



# Optimisation and Kinetics of Rubber Seed Oil Biodiesel Production

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## ABSTRACT

Due to the increasing yearn to go environment friendly, keen interest has been placed on biofuels, this leads to further studies and research in biofuels. A search for suitable vegetable/plant oil that can work just as fossil fuels and be eco-friendly was made. Hence, the study of this work investigates the production of biodiesel from non-edible *Hevea brasiliensis* oil. The process was catalysed by two-step acid base transesterification. Reaction parameters, such as reaction temperature and time were studied. The yield conversion of biodiesel was found to be 50.2% under the optimum conditions of 1.0 wt % NaOH and 6:1 methanol: oil molar ratio at 60°C for a reaction time of 75 min. The kinetic study was carried out at various temperatures. The conversion of triglycerides into methyl esters obeys the pseudo first-order mechanism. The reaction-rate constants and activation energies were determined. The physical and chemical characteristics of *H. brasiliensis* and biodiesel were determined and compared with standard value.

**Keywords:** Biofuels, Non-edible, Methyl esters, Kinetic, Fossil fuel, First-order, Transesterification

## 1. INTRODUCTION

The growth in the global energy demands predominantly are petroleum based; the decrease in the petroleum reserve and increased interest in saving the environment from pollution have jeered up interest in the search of alternative sources for petroleum-based fuels [1]. Amidst the alternatives, available are the biofuels, and Nigeria has a great potential for this through varieties of sources. These sources include virgin or used vegetable oils, biogas, animal fat and tree-borne oilseed like *Jatropha*, *Neem*, *Hevea brasiliensis*, castor, oil palm citrus seed oil and sunflower oil and others have been considered by many researchers for the full benefits of biodiesel production to be realised [2–4].

Biodiesel is an alternative fuel for diesel engines that is produced by the chemical reaction of a vegetable oil or animal fat with an alcohol, such as methanol. The reaction requires the presence of a catalyst, usually strong base, such as sodium or potassium hydroxide, and produces new chemical compounds called methyl-esters. These esters are accepted as biodiesel. The entire process is known as Transesterification [5]. Biodiesel, due to its similarity in properties to that of the conventional diesel, can be blended at any ratio with the conventional one or used directly with no modification to the existing diesel engine. Among the various vegetable oil sources, non-edible oils are suitable for biodiesel production because of their abundance and relatively cheaper price. Among the non-edible oil sources, *H. brasiliensis* (rubber tree) is identified as potential biodiesel source and comparing with other sources, it has added advantage, in that it has rapid growth, higher seed productivity and is suitable for both tropical and subtropical part of the world [6].

### 1.2 Rubber Seed Oil (RSO) Derived from Rubber Tree (*Hevea brasiliensis*)

Seed cotyledon is the most reasonable source for vegetable oil for large-scale production of biodiesel. If the primary concerns (where the need to avoid feeding our food to cars and engines is the most notorious) are to be eradicated once and for all. Before now, the production of biodiesel was from any source where the most popular are our own source of food. Ethanol and biodiesel produced from food oil crops can have an economical

affect, such that the price of our food commodities begins to rise. Today, a new dimension has been taken. Domestication of seed bearing plants is being promoted. One of such plants is the one unpopular *Jatropha* plant. Large-scale investments sensitisation and extension work are being used to encourage people to take to its cultivation. But *Jatropha* seed is not found more massive when placed on a scale of comparison with the rubber seed [7,8]. This leads us to the advantages of rubber seed in the production of biodiesel.

Numerous advantages associated with the use of rubber seed to produce biodiesel exist. A few of these advantages are that rubber seed is a rich source of nutrients, but it is simply poisonous rubber seed contains cyanide in the presence of enzymes. This makes it a reserve for the production of non-edible products [9]. Rubber tree does not necessarily require cultivation of new plantation. Large plantations of rubber trees meant for production of rubber latex are rampant in Africa and Asia. It is more profitable to farmers because they can now make extra profit from harvesting rubber seeds [9]. It will re-invigorate the economy of local communities which have abandoned their plantation due to low demand for rubber latex. Rubber seed produces more oil and exploring existing plantation around the world will go a long way to providing large percent of the world energy consumption [10]. Due to these enormous and futuristic advantages of biodiesel oil, a study on kinetic and optimisation of biodiesel production from rubber seed oil (RSO) is of immense respondents. Studies of the kinetics of transesterification will provide parameters that can be used to predict the extent of the reaction at any time under particular conditions. Among several kinetic studies published on transesterification of simple esters, only a few were concerned with the transesterification of vegetable oil fatty esters [11]. Although methyl esters from RSO have been produced on a pilot scale in Malaysia and Nigeria, there are no published reports on its kinetics [12]. The kinetic study of non-edible vegetable oil with and without catalyst has not yet been presented [13].

Therefore, in this study, kinetics of transesterification of non-edible RSO to biodiesel fuel will be treated in batch reactor with methanol by using NaOH catalyst. Experiment on the effects of time and reaction temperature on methyl ester formation will be carried out.

Escalation in the price of petroleum-based product due to over dependence, pollution of environment as a result of combustion of fossil fuel and there are wastage of non-edible oil seeds that would have been put into use. No much work has been done regarding the development of biodiesel production technology in Nigeria due to lack of knowledge in its kinetics and optimisation.

## **2. METHODOLOGY**

### **2.1 Materials**

The materials used in the course of this research can be divided into two; materials used for the production of biodiesel and materials used for the characterisation and analysis of oil and biodiesel.

### **2.2 Rubber Seed Oil**

The RSO was collected from the rubber research institute of Nigeria, RRIN, Iyanomo, Edo state. Rubber seed kernel was already separated from their seed and was drying under the sunshine for 90 min to decrease their water content and to remove their film which cover the kernel surface, after which, the seeds are treated [15]. To extract the crude rubber seed oil (CRSO) from the rubber kernel, the seed oil is pre-pressed using a high pressure continuous screw press – expeller. The expeller typically consists of a barrel containing a stainless steel helical screw. The pitch of the screw flights gradually decreases towards the discharge end, to increase the pressure on the pulp as it is carried through the barrel. The oil is extracted passed the perforated disk at the exit. The physical properties of CRSO were performed.

Extracted oil is filtered and collected in a settling tank. Material removed from the oil, called foot, is fed back into the stream of fresh material. Material discharged from the press, called cake, contains 8 to 10% oil. The cake was crushed into coarse meal for further processing [15]. The oil which was mechanically extracted using the expeller fabricated by the institute is store in a cool, dry, dark, air tight container. The oil contains 6.0% of free fatty acid determined the research institute according to the (AOCS) American Oil Chemists Society official method. It also has 24.6% oleic acid.

The chemicals used for this experiment were obtained from a chemical store, supplied by Bureau of Drug and Narcotic (BDN) General Purpose reagent. These chemicals as shown in Table 1 are; sodium hydroxide, sulphuric acid, methanol, ethanol, phenolphthalein, potassium hydroxide, sodium thiosulphate, ethanoic acid, bromine acid, hanus iodide, hydrogen chloride, potassium iodide, chloroform and starch solution and petroleum ether.

The experiments were carried out in the chemical engineering department laboratory in Federal University of Technology (FUT), Minna.

### 3. METHODS OF ANALYSIS

The water bath with shakers of a maximum speed of 250 rpm and heating regulation was used for the transesterification process. The process was optimised and carried out at various time and temperature. The yield conversion percentage ( $X_i$ ) was determined by equation (1), where  $X_i$  was calculated dividing the volume of RSO used initially. The yield conversion is, then, multiplied by 100 to give the percentage.

$$X_i = \frac{V_{\text{bio}}}{V_{\text{RSO}}} \times 100 \quad (1)$$

The molar concentration was calculated using equation (2);  $M_i$  was determined by dividing the product of the density ( $d_i$ ) by the relative percentage  $X_i$  by the molecular weight of each component ( $Mw_i$ )

$$M_i \left( \frac{\text{mol}}{\text{l}} \right) = \left[ \frac{X_i \times d_i \times 1000}{Mw_i} \right] \quad (2)$$

#### 3.1 Characterisation

Acid value test saponification free fatty acid, iodine value burned carbon content, specific gravity, moisture content, and viscosity of oil were carried out using method as described by Salamah [15].

#### 3.2 Characterisations of the Oil and Biodiesel

The characterisation of the oil and biodiesel were carried out at room temperature and distilled water was used for all cleaning and uses.

#### 3.3 Transesterification

The system was maintained at atmospheric pressure and the experiments were carried out at a constant temperature and the reaction time varied. The agitation was kept constant at 400 rpm. The experiment was carried out with 0.5 wt % of sodium hydroxide, molar ratio methanol : oil 6:1. To examine the temperature dependency of the reaction rate constants, reactions at 45, 50, 55, 60 and 65°C were studied. The reaction time was varied for each temperature constant at 15, 30, 45, 60, 75, 90, 105 and 120 min.

### 4. BIODIESEL PRODUCTION

#### 4.1 Pretreatment

The esterification of RSO is performed to reduce high free fatty acid content. The free fatty acid in the oil is the fatty acid that is not bounded to the glyceride group and triglyceride (TG). It is necessary to reduce the free fatty acid to less than 2% to avoid soap formation during the process reaction [15].

500ml of oil was measured and heated to 120°C for 1 h to remove the moisture content. The oil was left to cool at room temperature. 14 ml of  $H_2SO_4$  was added to 160-ml methanol, the mixture was, then, added to the cooled oil. This was placed in a heating magnetic stirrer for 60 min at 60°C. The product was separated using a separating funnel. The glycerol, impurities and water were separated from the methyl ester.



## 4.2 Transesterification

Total 50ml of methyl ester was measured. Methoxide was prepared by adding 10ml of methanol to 0.5 wt % sodium hydroxide. The mixture was vigorously shaken until the sodium hydroxide was completely dissolved. The mixture was then added to the methyl ester. This was heated at a constant temperature at a particular reaction time at 400 rpm. At the end of the reaction time, the content was poured in a separating funnel and the biodiesel was separated from the glycerol.

## 5. RESULTS AND DISCUSSIONS

### 5.1 Rubber Seed Oil Analysis

RSO as a raw material in this experiment was supplied by the rubber research institute of Nigeria, Iyanomo, Edo state. The oil was extracted using a press screw, where the seed kernels having been separated from the seeds shell were dried and pressed. The analysis of RSO as shown in Table 1 was carried out to compare and confirm if the oil is suitable for the production of biodiesel. Various analyses of the oil were used, such as specific gravity, moisture content, density, acid value and others. The free fatty acid is of major importance because it shows if the reaction will produce soap or biodiesel. The RSO has a high fatty acid of 13.44 mg/Potassium hydroxide (KOH)/g, this has to be reduced by esterification. This reaction using sulphuric acid reduced the free fatty acid to 1.98 mg/KOH/g.

**Table 1: Rubber seed oil analysis**

Properties	Unit	Values	American Society for Testing and Materials
Viscosity @ 25°C	mm <sup>2</sup> /s	42	31.55
Density @ 25°C	g/ml	0.858	0.9485
Water content	% vol	0.17	0.24
Acid value	mg/KOH/g	13.44	12.63
FFA	%	6.72	6.315
Saponification	mg/KOH/g	83.17	85.57
Iodine value	Cgl/g	20.73	20.68
FFA after esterification	%	1.98	

### 5.2 Acid Value

The acid value of the RSO used for this work is 13.44 mg/KOH/g. The acid value recorded shows that the fatty acid is higher than other vegetable oil. This fatty acid recorded in this work is quite high because the RSO used in this research was not used immediately after its extraction. The oil was kept in storage for 5 months, resulting the increase in free fatty acid [15]. The longer the oil stayed in storage, the higher will be the fatty acid. A high fatty acid leads to saponification; hence, the use of H<sub>2</sub>SO<sub>4</sub> as catalyst in the esterification of the oil is advisable. This reduced the fatty acid to 1.98 mg/KOH/g.

### 5.3 Specific Gravity

The specific gravity of the oil is 0.823. This result was gotten using the gravity bottle. This analysis is important because it helps show if the product finally gotten is biodiesel.

### 5.4 Moisture Content

The moisture content of the oil is 0.17%. This is the moisture from the seeds. The moisture content in the oil needs to be checked because excess moisture in the oil can cause soap formation which can lead to low yield and difficulty in the separation of the biodiesel and the glycerol [14] Table 2 shows the analysis of biodiesel.

**Table 2: Analysis of biodiesel from rubber seed oil**

Properties	Unit	Value	ASTM Value
Flash point	°C	130	93
Water sediment	% vol	0.05	0.05
Kinematic viscosity	mm <sup>2</sup> /s	2.5	1.9–60
Sulphated ash	% mass	0.04	0.02
Sulphur	% mass	0.08	0.05
Cetane number		47	46
Cloud point	°C		
Acid value	mg/KOH/g	0.65	0.8
Free glycerol	% mass	0.02	0.02
Specific gravity		0.82	0.86
Density	kg/m <sup>3</sup>	820	860
Iodine value	Cgl/g	1,021	1,021
Total glycerine	% mass	0.27	0.25
Ester content	% vol	50.2	96.5%

### 5.5 Transesterification

The RSO having been esterified underwent a reaction process where the temperature and time were varied at molar mass oil: methanol 1:6 and the quantity of alkali catalyst used in all the runs was 0.5 wt %. Optimum time and reaction temperature for the production was observed to be 75 min at 60°C. From the experimental results in this work, concentration of the reaction was calculated and tabulated in Table 5.

### 5.6 Effect of Time

Various runs were carried out for the transesterification of the oil, in a batch process. The dependency of reaction time was studied at different time intervals ranging from 15 to 120 min. This runs ranging from 30 min to 120 min is shown in Table 3. In the course of the experiment, it was observed that the yield conversion as time varied was more visible compared with the yield as temperature was varied. At the initial time of 15 min, it was observed that the yield was small ranging from 17% at 45°C, 16.2% at 50°C, 17% at 55°C, 18% at 60°C to 17.4% at 65°C. From these runs, it is seen that at 15 min the optimum temperature is 60°C. The increase in reaction temperature in time increases the reaction rate and reduces the reaction time.

**Table 3: Yield of biodiesel for 45, 50, 55, 60 and 65°C at various times**

Time $X_A$	Temperature				
	45°C	50°C	55°C	60°C	65°C
0	0	0	0	0	0
15	0.17	0.162	0.17	0.18	0.174
30	0.22	0.23	0.24	0.26	0.234
45	0.34	0.32	0.352	0.378	0.34
60	0.456	0.39	0.404	0.49	0.382
75	0.444	0.42	0.41	0.502	0.39
90	0.43	0.41	0.4	0.49	0.386
105	0.43	0.386	0.39	0.486	0.378
120	0.424	0.384	0.384	0.47	0.38

Thus, if given longer contact time, most of the reactants will collide and react to give higher yield of biodiesel. As the time increased, it is observed that the yield increased rapidly but began to slow down at 90 min, then as the time increased, the yield became almost constant and slowly decreased. This could be because of the equilibrium conversion, showing that the reaction has gotten to its peak. Reaction time was found to significantly affect the yield of biodiesel [16].

### 5.7 Effect of Temperature

The temperature was varied to find the optimum temperature. As the temperature increased, the yield slightly increased at a constant time. At 45°C, the yield was 44.4% 75 min of reaction time, as the temperature increased at 60°C. The optimum yield 50.2% was noted after 75 min time reaction. This showed that the reaction temperature has a more significant effect on the conversion at higher reaction temperature than lower temperature. However, at higher reaction temperature, the possibility of collision among reactant particles became greater and easily obtains the necessary activation energy [17].

### 5.7 Kinetics

The kinetics reaction in Table 6 shows that, all the runs except at 45°C follows a first order kinetics as a function of the concentration of oil (TG and reaction temperature) which is similar to a work done by [18]. Due to the high methanol concentration (200% excess), methanol was not considered as a limiting reactant. This also shifts the equilibrium to the right (forward reaction); therefore the overall reaction was assumed to be a single-step shunt reaction, neglecting the intermediate reaction. Here, three ester groups were attacked by the methoxy ion at the same time. To find rate constant and the order of the reaction, the gradient of points in the curve were obtained and recorded. The  $\ln [C_A]$  and the  $\ln [-dC_A/dt]$  is gotten at interval points on the graph. The slope of these graphs gives the rate order whereas the intercept gives the rate constant.

**Table 4: The calculated value of the relationship of kinetic rate constant and rate order at 45°C**

Time	$C_A$	$dC_A/dt$	$\ln[dC_A/dt]$	$\ln C_A$
3.00	0.800	0.000778	-7.16	-0.223
15.00	0.710	0.000500	-7.60	-0.342
37.50	0.600	0.000417	-7.78	-0.511
47.50	0.505	0.000250	-5.99	-0.673

**Table 5: The calculated value of the relationship of kinetic rate constant and rate order at 50°C**

Time	$C_A$	$dC_A/dt$	$\ln[dC_A/dt]$	$\ln C_A$
7.5	0.790	0.000778	-7.16	-0.235
30.0	0.650	0.000556	-7.49	-0.431
46.5	0.565	0.000556	-7.49	-0.571
66.5	0.485	0.000267	-8.23	-0.724

**Table 6: The calculated value of the relationship of kinetic rate constant and rate order at 55°C**

Time	$C_A$	$dC_A/dt$	$\ln[dC_A/dt]$	$\ln C_A$
7.5	0.775	0.000333	-8.01	-0.255
17.5	0.675	0.000444	-7.72	-0.393
39.0	0.600	0.000714	-7.24	-0.511
60.0	0.510	0.000167	-8.69	-0.673

**Table 7: The calculated value of the relationship of kinetic rate constant and rate order at 60°C**

Time	$C_A$	$dC_A/dt$	$\text{Ln}[dC_A/dt]$	$\text{Ln } C_A$
6.0	0.8	0.00083	-7.09	-0.22
15.0	0.705	0.00056	-7.49	-0.35
34.5	0.580	0.00067	-7.31	-0.54
58.5	0.445	0.00033	-8.02	-0.81

**Table 8: The calculated value of the relationship of kinetic rate constant and rate order at 65°C**

Time	$C_A$	$dC_A/dt$	$\text{Ln}[dC_A/dt]$	$\text{Ln } C_A$
7.5	0.785	0.0033	-5.72	-0.24
15.0	0.709	0.0067	-5.01	-0.34
45.0	0.566	0.0042	-5.47	-0.57
60.0	0.530	0.0014	-6.55	-0.63

According to a biodiesel kinetics work done by [19], stated that, the rate order of the biodiesel reaction fits into a first order reaction model than the second order model. From Table 4, it is observed that the rate constant increased with increase in temperature. Rate constants are sensitive to temperature, which increases with rise in temperature, previously reported similar result in work on the biodiesel reaction using *Jatropha* [20]

### 5.8 Rate Order of Reaction

The rate order of reaction in this work shown in Table 6, has no regular pattern like some works previously done by [18]. At 45°C, the rate order is 2.2979. This indicates the reaction order at this temperature is second order. At 50°C, the rate order is 1.9854, this is a first order reaction. At 55°C, the order is 1.3422, order at this temperature is also first order. At 60, the order is also a first order with its slope as 1.3474. For 65, it is a first order with a value of 1.925. From this work, it shows that all the rate order is a first order reaction, except at 45° where it is a second order reaction these are shown in (Tables 6-8).

#### 5.8.1 Rate Constant

The rate constant is the exponential of the intercept of the curve. It is observed that the rate constant increased with increase in temperature. Rate constants are sensitive to temperature, which increases with rise in temperature [20] previously reported similar result in his work on the biodiesel reaction using *Jatropha*. At 45, the rate constant is 0.001233, at 50, its 0.001332, at 55, its 0.000674, at 60 it is 0.001084 and at 65 it is 0.00803. The results tabulated in Table 10 (Table 9).

**Table 9: Rate order and rate constant**

Temp.	Slope	Intercept	$K$
45	2.2979	-6.6982	0.001233
50	1.9854	-6.6208	0.001332
55	1.3422	-7.3025	0.000674
60	1.3474	-6.8274	0.001084
65	1.9250	-4.8346	0.008030

### 5.9 Arrhenius Equation

The Arrhenius equation shows effect of a change of temperature on the rate constant,  $k$ , dependent on the temperature of which the reaction takes place. Table 10 shows the inverse of temperature,  $1/T$ , and the  $\text{Ln } K$ .... These values are plotted to find its activation energy,  $E_a$  (in joules), the activation energy is derived from the

slope, the slope,  $E_a/R$ , where  $R$  is the universal gas constant. The intercept is  $A$  in the equation, representing a pre-exponential factor that has the same units as  $k$ . In this work,  $A$  is 1,7,769,605.51 and the activation energy is 63,185.2 J/mol/K.

**Table 10: Arrhenius equation**

$T(K)$	$K$	$\ln K$	$1/T$
318	0.001233	-6.6982	0.00314
323	0.001332	-6.6208	0.00310
328	0.000674	-7.3025	0.00305
333	0.001084	-6.8274	0.00300
338	0.00803	-4.8246	0.00296

## 6. CONCLUSION

The analysis of the RSO used in this research titled optimisation and kinetics of the production of biodiesel from RSO, shows that the oil is suitable for use as raw material for the transesterification and the production of biodiesel. Although the free fatty acid content is high, esterification using 5 mol of  $H_2SO_4$  with the RSO to methanol ratio 1:6 was used to reduce the free fatty acid, Free fatty acid (FFA), to 1.7% hence, reducing the tendency of soap formation. The time and temperature was varied to achieve the optimum time and temperature for the maximum yield of biodiesel production. The optimum time recorded in this work is 75 min at an optimum temperature of 60°C, giving a yield of 50.2%. This yield is the highest yield obtained in the course of the experiment. The kinetics reaction of the process for the production of biodiesel was established for the various temperatures. The rate of the reaction varied slightly. At 45°C, the rate order was a second order reaction. At 50°C, it showed a second order reaction, whereas at 55°C, the rate order was a pseudo first order reaction. From the results, it can be concluded that as the temperature increased the reaction appeared to be a pseudo first order reaction. Its rate constant, which is dependent on temperature, increased as the temperature increased. At 45, 50, 55, 60 and 65°C, the rate constants are 0.001233, 0.001332, 0.000674, 0.001084 and 0.00803, respectively.

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