

Title of Paper: Production of Biodiesel From Rubber Seed Oil using K-Pumice Catalyst: Modeling and Kinetics

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ABSTRACT

The production of biodiesel from rubber seed oil using a synthesized composite catalyst (K-Pumice) was investigated. A two-step approach involving an acid catalyst treatment, followed by an alkali catalyzed transesterification, was used to convert the oil into fatty acid methyl ester (FAME). In the first step, extracted rubber seed oil was pretreated with methanol and sulphuric acid to reduce its acid value. Reaction time (90 to 180 minutes), temperature (40 to 60°C), catalyst amount (2 to 10 wt.% of oil) and methanol to oil ratio (6:1 to 10:1 vol/vol) were the transesterification process variables varied to study their effects on biodiesel yield. A fit of the developed quadratic model's predicted biodiesel yield to the actual biodiesel yield data, gave an R^2 -value of 0.9978 and an F-value of 475.5. The high R^2 -value and F-value are indicative of a well fitted and reliable model. All the variables had positive effects on biodiesel yield. However, beyond the optimum values of 150 minutes, 55°C, 8 wt.% of oil and 9:1 vol/vol for time, temperature, catalyst amount and methanol to oil ratio respectively, the biodiesel yield was observed to decrease. The optimum yield of biodiesel was found to be 75.97%. The second-order kinetic model suitably described the biodiesel production process, with a rate expression of $-r_A = 0.0221 C_A^2$ at 55°C, and an activation energy of 30.030KJmol⁻¹. The produced biodiesel had a specific gravity of 0.87; an acid value of 0.43(mgKOH); flash point of 150°C and kinematic viscosity of 5.0 (mm²/s) at 40°C. The values of these properties were within ASTM D6751 standards, and compared favourably with values reported by other studies.

KEYWORDS: Biodiesel production, Rubber seed oil, K-Pumice, Modeling, Optimization, Kinetics

1. INTRODUCTION

There is a steady increase in the global energy demand [1]. Environmental concerns and sustainability issues have led to biodiesel being identified as a viable alternative to the traditional fossil fuels [2]. Biodiesel has over the years gained significant attention as it is renewable, non-toxic, biodegradable, less pollutant emitting, and a more environmentally friendly fuel source than fossil diesel fuel currently available. It has been studied as one possible solution in the imminent depletion of fossil fuels and is widely regarded

2. MATERIALS AND METHODS

The rubber seeds were milled and dried to constant weight in an oven (DHG-9101 Laboratory dry oven) at 105°C to constant weight. The seed oil was then extracted using n-hexane. K-Pumice was prepared using the method described in [4]. For the acid catalyzed esterification of the rubber seed oil, H₂SO₄ (7% vol/vol) was mixed with methanol. The mixture was then

to be very sustainable and eco-friendly [3]. Biodiesel is produced by transesterification of oils or fats derived from plant matter or animal wastes. The feedstock used to produce biodiesel makes use of CO₂ in the atmosphere in its photosynthesis [4]. During combustion, biodiesel does not emit sulfur or aromatic based compounds and it has a lower hydrocarbon and particulate matter emission. In total, biodiesel has 41% lesser greenhouse gas emission when compared to diesel [5]. Feedstock accounts for 60 – 75% of the total biodiesel production costs; and biodiesel production from cheap low-cost materials will reduce its cost, thus making it more competitive than conventional fossil fuels [1]. The oils that can be used as feedstock for biodiesel production can be grouped as edible oils, non-edible oils and waste oils. Most of the biodiesel production feedstock used of recent is from edible oil plants such as palm oil, rapeseed, sunflower and soybean. These compete with food, cosmetic and pharmaceutical uses, leading to food-fuel crisis and high price of biodiesel generated thereof [1]. In order to minimize food security concerns and biodiesel production costs, current research focuses on the use of non-edible oils and waste oils [6]. Rubber tree (*Hevea brasiliensis*) is one of the few versatile bioenergy crops with non-edible oil that could be exploited as low-cost industrial oil for the production of biodiesel.

The basic reaction involved in biodiesel production has been reviewed in literature. This is the reaction between triglycerides and methanol in the presence of a catalyst (transesterification), yielding the desired Fatty Acid Methyl Ester (FAME) and a byproduct, glycerol [7]. Because of the high free fatty acid content of rubber seed oil, and the need to avoid soap formation, conversion to biodiesel follows a two-step process involving a free fatty acid reduction step (acid esterification step) and the biodiesel production step (alkaline transesterification step). The current work focuses on the modeling, optimization and kinetics of the conversion of rubber seed oil to biodiesel using K-Pumice as an alkaline catalyst.

reacted with the rubber seed oil for 1hour, maintained at a temperature of 60°C and a stirring rate of 500rpm. This reduced the acid value of the rubber seed oil to 1.72 mg KOH/g oil.

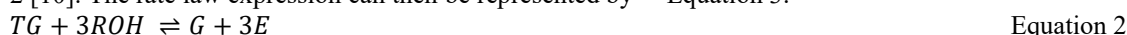
Factorial experimental design, using the central composite design (CCD), was applied in the transesterification of the esterified seed oil. Thirty(30) experimental runs in which the esterified oil was reacted with methanol in the presence of K-

Pumice catalyst, were carried-out in a 250ml three-necked round bottom reactor, equipped with a reflux condenser and a hot plate with magnetic stirrer. The factors varied were; reaction time (90 to 180 minutes); methanol to oil ratio (6:1 to 10:1); K-Pumice catalyst (2 to 10% by weight of oil) and temperature (40°C to 60°C), while the agitation speed was set at 500rpm. At the end of reaction time, the biodiesel (rubber seed oil methyl ester) was placed in a rotary evaporator to remove excess methanol, and washed three times with deionized water at 50°C to

remove entrained glycerol. The washed biodiesel was then dried over anhydrous sodium sulphate at 50°C. The biodiesel yield was measured by the method described in [8]. The specific gravity; acid value; flash point and kinematic viscosity of the biodiesel were measured by the Association of Official Analytical Chemists methods [9]. Response surface method (RSM) was then used to optimize the transesterification process variables. The percentage yield of biodiesel was computed from Equation 1 [8].

$$\text{Biodiesel yield (\%)} = \frac{\text{Weight of product obtained} \times \text{FAME (\%)}}{\text{Weight of rubber seed oil}} \times 100 \quad \text{Equation 1}$$

In the kinetic analysis of the process, the biodiesel production was assumed to follow a single step reaction, as shown in Equation 2 [10]. The rate law expression can then be represented by Equation 3.



$$-r_{\text{TG}} = \frac{-dC_{\text{TG}}}{dt} = K' C_{\text{TG}} C_{\text{ROH}}^3 \quad \text{Equation 3}$$

Where TG, ROH, G and E are triglyceride, methanol, glycerol and the fatty acid methyl ester (produced biodiesel). If the concentration of alcohol is considered to be in excess and the reverse reaction is ignored, then a pseudo first-order rate law can be written for the reaction as Equation 4 [10].

$$-r_{\text{TG}} = \frac{-dC_{\text{TG}}}{dt} = K_1 C_{\text{TG}} \quad \text{Equation 4}$$

Where $K_1 = K' C_{\text{ROH}}^3$

Also, due to the high methanol to oil ratio in the methanol phase, the rate law may be written in terms of TG only. If the reaction is assumed to be pseudo second-order, then the rate law can be expressed as Equation 5 [10].

$$-r_{\text{TG}} = \frac{-dC_{\text{TG}}}{dt} = K_2 C_{\text{TG}}^2 \quad \text{Equation 5}$$

The pseudo First-order and Second-order kinetic models were then tested on the experimental data., while the effect of temperature on the reaction rate constant was modeled by the Arrhenius equation in Equation 6.

$$K = A e^{-E_a/RT} \quad \text{Equation 6}$$

In terms of conversion of triglyceride to biodiesel, Equation 4 can be written as Equation 7, which was integrated to give Equation 8.

$$\frac{C_{\text{TGO}} dx_{\text{TG}}}{dt} = -k_1 C_{\text{TGO}} (1 - X_{\text{TG}}) \quad \text{Equation 7}$$

$$-\ln(1 - X_{\text{TG}}) = k_1 t \quad \text{Equation 8}$$

The rate constant k_1 was then determined from the plot of $-\ln(1 - X_{\text{TG}})$ against time.

For the pseudo second-order kinetic model, the rate law in Equation 5 was written in terms of conversion as Equation 9, which was integrated to give Equation 10. The rate constant K_2 was then determined from the plot of $\frac{1}{1 - X_{\text{TG}}}$ against time [10].

$$\frac{C_{\text{TGO}} dx_{\text{TG}}}{dt} = k_2 C_{\text{TGO}}^2 (1 - X_{\text{TG}})^2 \quad \text{Equation 9}$$

$$\frac{1}{1 - X_{\text{TG}}} = k_2 C_{\text{TGO}} t \quad \text{Equation 10}$$

The rate constants were evaluated at different temperatures, while the activation energy and pre-exponential factor were obtained from the Arrhenius plot of $\ln K$ against $1/T$.

3. RESULTS AND DISCUSSION

3.1 Quadratic Model and Statistical Analysis of Biodiesel Production Process

Results obtained from the K-Pumice-catalyzed transesterification of the esterified rubber seed oil based on central composite experimental design are presented in Table 1. The Design expert software version 11 that was used generated the quadratic mathematical model shown in Equation 11. Equation 11 is the regression model in terms of actual process variables, and can be used to make predictions of the response for the given values of each factor.

An excellent correlation exists between the predicted yields and the experimental yields as shown in Figure 1 and Table 1. The coefficient of determination (R^2) given as 0.9978 and the low

values of the residuals ($\leq \pm 0.42$) are indicative of a well-fitted and reliable model. Analysis of variance (ANOVA) is reported in Table 2, while the Fit Statistics is reported in Table 3. From the ANOVA, all the process variables had positive effects on biodiesel yield, with the catalyst amount being the most influencing followed by temperature, reaction time and methanol to oil ratio respectively. The model terms are said to be significant if the p-value is less than 0.05. A p-value less than 0.0001 indicate that the model is significant. Also, the high F-value (475.5) implies that the model is significant.

$$\text{Biodiesel yield (\%)} = -196.22292 + 0.473472 (\text{Time}) + 5.63333 (\text{Temperature}) + 5.83542 (\text{Catalyst amount}) + 19.66250 (\text{Methanol to oil ratio}) - 0.001750 (\text{Time})(\text{Temperature}) - 0.007708 (\text{Time})(\text{Catalyst amount}) +$$

$$0.018333 (\text{Time})(\text{Methanol to oil ratio}) + 0.012500 (\text{Temperature})(\text{Catalyst amount}) + 0.172500 (\text{Temperature})(\text{Methanol to oil ratio}) - 0.031250 (\text{Catalyst amount})(\text{Methanol to oil ratio}) - 0.001942 (\text{Time}^2) - 0.066917 (\text{Temperature}^2) - 0.361979 (\text{Catalyst amount}^2) - 1.88542 (\text{Methanol to oil ratio}^2). \quad \text{Equation 11}$$

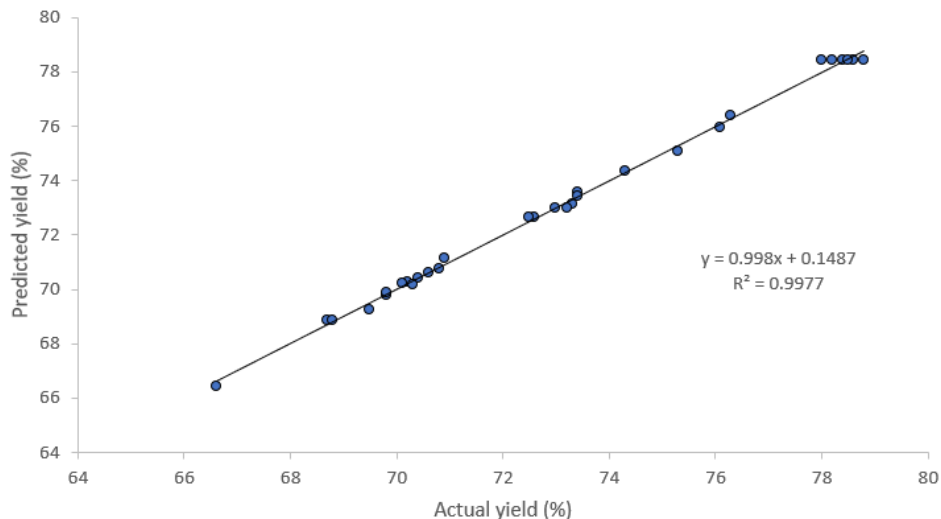


Figure 1: Correlation of predicted biodiesel yield and actual biodiesel yield

Table 1: The experimental design and the actual and model predicted biodiesel yields

Run	Time (minutes)	Temperature (°C)	Catalyst amount (wt.%)	Methanol to oil ratio (vol/vol)	Actual biodiesel yield (%)	Predicted biodiesel yield (%)	Residual
1	150	45	8	9:1	72.60	72.65	-0.05
2	150	55	8	7:1	73.00	73.00	0.00
3	90	55	8	7:1	74.30	74.34	-0.04
4	90	55	8	9:1	75.30	75.10	0.20
5	150	55	4	7:1	69.80	69.79	0.01
6	150	45	8	7:1	73.30	73.14	0.16
7	90	45	4	9:1	66.60	66.42	0.18
8	120	50	6	8:1	78.00	78.42	-0.42
9	120	60	6	8:1	73.40	73.59	-0.19
10	120	50	6	8:1	78.60	78.42	0.18
11	120	50	6	8:1	78.40	78.42	-0.02
12	90	55	4	9:1	70.20	70.29	-0.09
13	150	55	4	9:1	73.20	73.00	0.20
14	120	50	6	6:1	70.60	70.61	-0.01
15	180	50	6	8:1	72.50	72.64	-0.14
16	120	40	6	8:1	69.80	69.86	-0.06
17	120	50	6	8:1	78.50	78.42	0.08
18	90	45	8	9:1	70.80	70.74	0.06
19	120	50	2	8:1	68.70	68.86	-0.16
20	90	45	8	7:1	73.40	73.42	-0.02
21	150	55	8	9:1	76.10	75.97	0.13
22	90	45	4	7:1	68.80	68.85	-0.05
23	150	45	4	7:1	70.40	70.42	-0.02
24	120	50	6	8:1	78.20	78.42	-0.22
25	120	50	10	8:1	76.30	76.39	-0.09
26	60	50	6	8:1	70.10	70.21	-0.11
27	120	50	6	8:1	78.80	78.42	0.38
28	90	55	4	7:1	69.50	69.27	0.23
29	120	50	6	10:1	70.90	71.14	-0.24
30	150	45	4	9:1	70.30	70.19	0.11

Table 2: Analysis of variance for quadratic model

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value	
Model	359.11	14	25.65	475.5	<0.0001	Significant
A- Time	8.88	1	8.88	164.64	<0.0001	
B- Temperature	20.91	1	20.91	387.56	<0.0001	
C- Catalyst amount	85.13	1	85.13	1578.04	<0.0001	
D- Methanol to oil ratio	0.4267	1	0.4267	7.91	0.0131	
AB	1.10	1	1.10	20.44	0.0004	
AC	3.42	1	3.42	63.44	<0.0001	
AD	4.84	1	4.84	89.72	<0.0001	
BC	0.2500	1	0.2500	4.63	0.0480	
BD	11.90	1	11.90	220.64	<0.0001	
CD	0.0625	1	0.0625	1.16	0.2988	
A ²	83.80	1	83.80	1553.45	<0.0001	
B ²	76.76	1	76.76	1423.00	<0.0001	
C ²	57.50	1	57.50	1065.97	<0.0001	
D ²	97.50	1	97.50	1807.47	<0.0001	
Residual	0.8092	15	0.0539			Not significant
Lack of fit	0.4008	10	0.0401	0.4908	0.8417	
Pure error	0.4083	5	0.0817			
Correlated total	359.91	29				

Table 3: Fit statistics for quadratic model

Statistical parameter	Value
Standard deviation	0.2323
Mean of response	73.01
Coefficient of variance (%)	0.3181
R ²	0.9978
Adjusted R ²	0.9957
Predicted R ²	0.9920
Adequate precision	73.0419

In Table 3, the difference between Adjusted R² and Predicted R² (0.0037) is lower than the allowable difference of 0.2, indicating that the model can predict the response of the process within acceptable range. The adequate precision value of 73.0419 is also higher than the critical value of 4, again indicating that the model can navigate the design space for the optimization purpose [8].

3.2 Effect of Interacting Factors on Biodiesel Yield

The 3D plots of the interacting effects between process variables on the biodiesel yield are given in Figure 2 (a) to (f). The 3D-surface plot depicted in Figure 2 (a) shows the interaction between temperature and reaction time, with the catalyst amount and the methanol to oil ratio kept constant. Simultaneous increase in temperature and reaction time resulted in a corresponding increase in the biodiesel yield. This is because of the better mixing of rubber seed oil and methanol as temperature increased. However, at an optimum reaction time of 150 minutes, increase in the reaction temperature above 55°C led to a decrease in biodiesel yield. The optimum temperature for the biodiesel production process was found to be 55°C. Studies have shown that when temperature increases beyond the

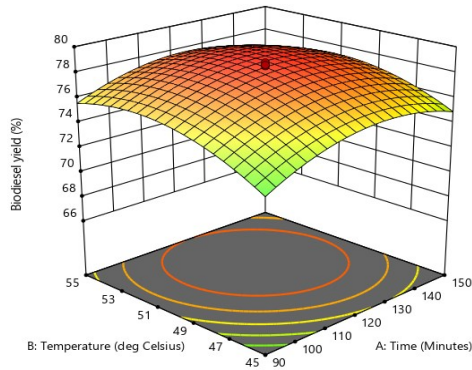
optimal temperature, the yield of biodiesel decreases due to acceleration of the saponification reaction of the triglyceride ([11]; [12]). An optimum temperature of 45 ± 5 for biodiesel production from Rubber seed oil was reported in [13], while in another study [14], a reaction temperature of 60°C was observed as the optimum.

Figure 2 (b) shows the interaction between catalyst amount and reaction time, with the reaction temperature and the methanol to oil ratio kept constant. There is an observed increase in the yield of biodiesel when the catalyst amount and reaction time are simultaneously increased. An optimum biodiesel yield of 75.97% was observed at a reaction time of 150 minutes. It could be observed from Figure 2 (c) to (f) that, simultaneously increasing the interacting variables resulted in increase in the biodiesel yield. However, increasing these variables beyond their optimal values resulted in a decrease in biodiesel yield [15]. In addition to affecting the yield of biodiesel, the catalyst amount also influences its colour. The higher the amount of catalyst, the darker the biodiesel produced. Therefore, the addition of a suitable amount of catalyst is important to the physical appearance of biodiesel [13]. Table 4 shows the

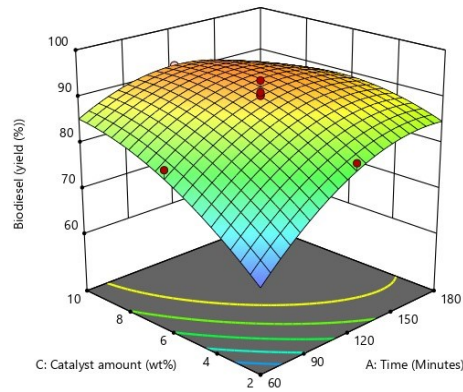
optimum value of each process variable for the biodiesel production process. The optimum temperature was found to be 55°C; while 150 minutes, 8 wt.% and a ratio of 9:1, were the optimum values for time, catalyst amount and methanol to oil ratio respectively. The optimum biodiesel yield was 75.97%.

Table 4: Optimized values

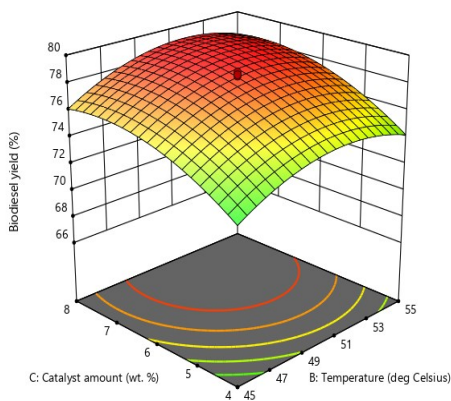
Parameter	Value
Time (minutes)	150
Temperature (degree Celsius)	55
Catalyst amount (wt. %)	8
Methanol to oil ratio (vol/vol)	9:1
Biodiesel yield (%)	75.97



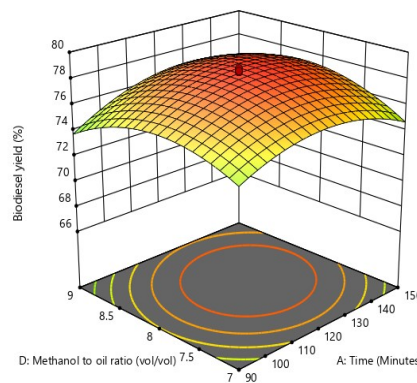
(a) Temperature and Time



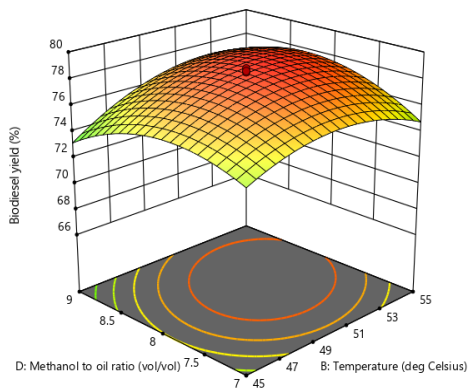
(b) Catalyst amount and Time



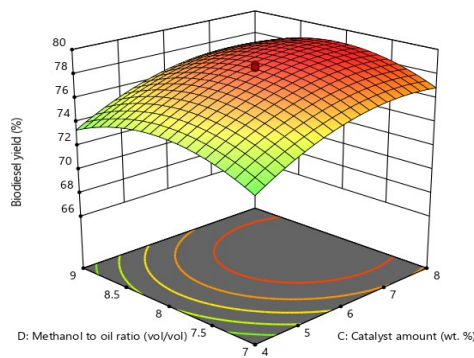
(c) Catalyst amount and Temperature



(d) Methanol to oil ratio and Time



(e) Methanol to oil ratio and Temperature



(f) Methanol to oil ratio and Catalyst amount

Figure 2: 3D plots for the interactive effects between process variables on biodiesel yield.

3.3 Kinetics

3.3.1 Kinetic Model

In Table 5, the R-square values for the rate law plots are presented for the pseudo first-order and the pseudo second-order kinetic models. The plots were obtained by varying the temperature and the reaction time, while keeping the catalyst amount and methanol to oil ratio at their optimum values of 8

wt.% and 9:1 vol./vol. respectively. From the R-square values, both kinetic models appear to fit the reaction data, but the pseudo second-order kinetic model which gave a higher R-square value for all the temperatures considered, described the reaction better. Hence, further kinetic evaluations were based on the pseudo second-order model.

Table 5. Values of R-square for rate law plots at different temperatures

Temperature (°C)	Pseudo first-order model	Pseudo second-order model
40	0.9669	0.9775
45	0.9637	0.9759
50	0.9596	0.9732
55	0.9525	0.9663
60	0.9410	0.9540

3.3.2 Rate Constants, Pre-exponential Factors, Activation Energy and Rate Law

Values of the rate constants k at temperatures ranging from 40 °C to 60 °C are shown in Table 6, the rate laws at these temperatures are presented in Table 7, while the Arrhenius plot from the biodiesel production process is given in Figure 3. It is observed from Table 3, that the rate constant increased with temperature up

to 55 °C, and then decreased thereafter. Indicating that the rate of formation of biodiesel increased with temperature up to an optimum of 55 °C, but reduced beyond this temperature. The Arrhenius plot of Figure 3 covers the temperatures range 40 °C and 55 °C. The activation energy E_a was computed to be 39.030 KJmol⁻¹, while the pre-exponential factor A , was calculated to be 3.93×10^4 .

Table 6: Values of rate constant, k at various temperatures

Temperature, °C (Kelvin)	Rate constant (k)	ln k	1/T (K ⁻¹)
40 (313)	0.0111	-4.5008	3.195×10^{-3}
45 (318)	0.0160	-4.1352	3.145×10^{-3}
50 (323)	0.0198	-3.9221	3.096×10^{-3}
55 (328)	0.0221	-3.8122	3.049×10^{-3}
60 (333)	0.0159	-4.1414	3.003×10^{-3}

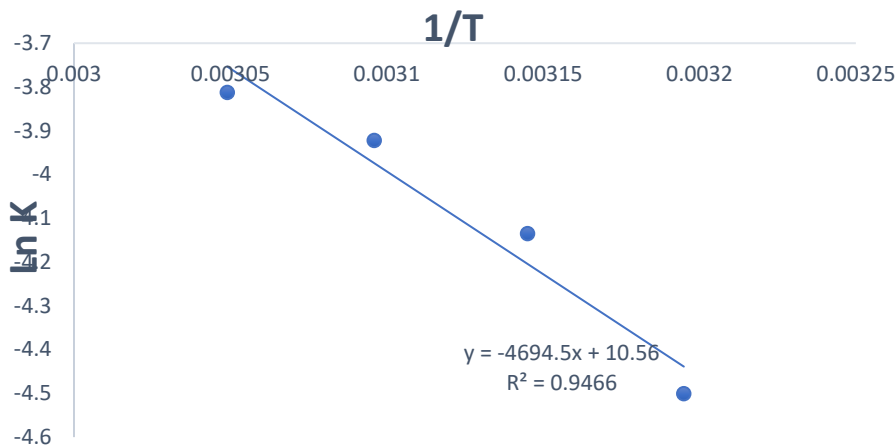


Figure 3: Arrhenius plot of ln k against 1/T for the biodiesel production process

Table 7: Rate law at various temperatures

Temperature, °C (Kelvin)	Rate law
40 (313)	$-r_A = 0.0111 C_A^2$
45 (318)	$-r_A = 0.0160 C_A^2$
50 (323)	$-r_A = 0.0198 C_A^2$
55 (328)	$-r_A = 0.0221 C_A^2$
60 (333)	$-r_A = 0.0159 C_A^2$

3.4 Characterization of Biodiesel

The specific gravity, acid value, kinematic viscosity and flashpoint of the biodiesel produced were characterized by following ASTM procedures. The values obtained were compared with literature values of biodiesel synthesized from rubber seed oil, as well as the ASTM D 6751 biodiesel standard. The results are presented in Table 8. The properties of the biodiesel obtained in the current study compared favourably with those from similar studies on biodiesel production from rubber seed oil, and were in agreement with the ASTM D 6751 biodiesel standard.

Table 8: Comparison of some properties of the produced biodiesel with those from other works

Properties	Specific gravity	Acid value (mgKOH/g oil)	Flash point (°C)	Kinematic viscosity at 40°C (mm ² /s)
ASTM Standard values	0.86 – 0.9	< 0.6	100 – 170	1.9 – 6
[13]	0.874	0.118	130	5.81
[16]	0.85	0.12	120	4.5
[17]	0.885	0.42	152	3.89
[18]	0.87	0.07	154.6	4.64
[8]	0.876	0.56	158	4.32
[15]	0.88	0.26	140	4.49
Present study	0.87	0.43	150	5.0

4. CONCLUSION

The production of biodiesel from a low-cost, non-edible feedstock, rubber seed oil, was investigated. The biodiesel yield was affected by reaction time, temperature, catalyst amount and methanol to oil ratio. Increase in each of these variables led to a corresponding increase in the yield of biodiesel. However, beyond the optimum values of 150 minutes, 55°C, 8 wt.% of oil and 9:1 vol/vol for reaction time, temperature, catalyst amount and methanol to oil ratio, there was an observed decrease in the yield of biodiesel. The optimum yield of biodiesel was found to be 75.97%. A quadratic model for the biodiesel production

process was derived. The model is suitable for the prediction of biodiesel yield. The kinetics of rubber seed oil transesterification was also investigated. The reaction was found to fit a pseudo second-order model, based on this, the rate constants at various temperatures, the activation energy and the pre-exponential factor were obtained. The determined physical properties of the produced biodiesel, compared favourably with those from other studies. K-Pumice catalyst has shown to be an effective catalyst for the synthesis of biodiesel from rubber seed oil.

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