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OPTIMIZATION AND CHARACTERIZATION OF SYNTHESIZED BIODIESEL PRODUCED FROM REFINED PALM KERNEL OIL

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ABSTRACT

In this study, response surface methodology (RSM) was used to study the transesterification reaction of refined palm kernel oil for biodiesel production. The three main factors that drive the conversion of triglycerides into fatty acid methyl esters (FAME) were studied according to a full factorial design at two levels. These factors were catalyst (NaOH) concentration, temperature and reaction time. Analysis of variance (ANOVA) was used to determine the significance of the factors and their interactions which primarily affect the first of the two transesterification sets. The effect of reaction parameters (molar ratio, catalyst weight and reaction time) was studied using RSM while the reaction temperature was kept constant at 60^{o}C. Optimum methyl ester yields 93% was obtained at oil to methanol molar ratio of 1:3(0.33), a catalyst load of 2.3g and reaction time of 140 minutes. The optimum methyl ester was characterized for fuel properties and the results obtained ascertain the eligibility of palm kernel oil methyl ester for use in diesel engines since they were within the acceptable standards set by American Society of Testing and Material (ASTM D 6751). And also, the study showed a good agreement with the experimental results, demonstrating that this methodology may be useful for industrial process optimization.

Keywords: *Analysis of variance, Alkaline based catalyst, Biodiesel, Process optimization, Refined palm kernel oil. Response surface methodology (RSM).*

INTRODUCTION

The response surface methodology (RSM) can be defined as a collection of statistical and mathematical techniques that is used in the aim of developing an adequate functional relationship between a number of inputs (variables x 1, *x2, x3, xn)* and an output (response *y*). Generally, the relationship between the response and the variables is unknown however; it can be approximated using a low degree polynomial, (Carley, Kamneva and Reminga, 2004; Khuri and Mukhopadhyay, 2010; Dean, Voss and Draguljić, 2017).). If there is a continuous range of values for the variables as well as for the response, respond surface methodology is very useful for optimizing the response value. All RSM problems generally use either the first degree polynomial or the second degree polynomial or a combination of them both to establish a relationship between the response (y) and regressions (x1, x2, and x3….xn). An appropriate experimental design must be used to collect data to get an efficient approximation of the polynomial. There are three basic methods for collecting data

- A retrospective study based on historical data.
- An observational study.
- A designed experiment (Anderson-Cook, Borror and Montgomery, 2009).

The central composite design (CCD) is the most commonly used response surface methodology model; and it contains three points such as:

- A factorial design point of two level (2^k) made up of either combination of the $+\propto$ and$-\propto$.
- An axial point represented as 2k, set up axially at a distant point of $+\propto$ and $-\propto$ from the middle to produce a quadratic equation.
- The replicate also known as the centre point.

The expression for calculating the number of experiments by the central composite design is

$N= 2^k + 2k + No$

Where N, represent the total number of experiments, and No represents number of replicates occurred, and k represent the factors or parameters to be considered (Anderson-Cook, Borror and Montgomery, 2009). The use of a design software or Minitab can be required for the central composite design under RSM. The response surface methodology was applied in the pyrolysis of palm kernel shell optimization, based on central component design (CCD) and the result showed high level of efficiency dependent on the flow rate, reaction time, and catalyst (**La**kshmi, *et al*., 2020). This work investigated the effect of three different reaction parameters (ratio of ethanol to PKO, catalyst concentration and reaction time) on the yield of biodiesel produced from PKO oil using response surface methodology (RSM). Effect of combination of Methanol-NaOH was also investigated to compare the results as its affect the yield.

MATERIALS AND METHOD

Optimization of Biodiesel

Effects of operating parameters such as time of reaction, catalyst concentration as well as oil-methanol mass (or mole) ratio were investigated using response surface methodology (RSM). It was used to determine the optimum conditions for biodiesel production from refined palm kernel oil, three variables were studied at both high and low levels. The expected response is biodiesel (methyl ester) yield. The low level of methanol: oil mass ratio was 1:2 and the high level was 1:6. The low level of catalyst load chosen was 1.5g and the high level was 3.0g NaOH catalyst by weight. The reaction time chosen for the lower level was 60 minutes and 120 minutes for the higher.

Characterization of the Biodiesel Produced

Determination of specific gravity (relative density)

Specific gravity and density of biodiesel produced was determined in accordance with ASTM D1298 using Hydrometer Method. A clean dry empty 50ml density bottle was weighed and the mass recorded as M, it was then filled up with distilled water and subsequently with the samples. The mass of the bottle and water was taken and recorded as M_1 and that of bottle and biodiesel as M_2 respectively.

Determination of flash point

Flash point was determined in accordance with ASTM**D 93**. A sample of the biodiesel was heated in a close vessel and ignited. A pensky-martens cup tester was used. It measures the lowest temperature at which application of the test flame causes the vapour above the sample to ignite. The biodiesel was placed in a cup in such quantity as to just touch the prescribed mark on the interior of the cup. The cover was then fitted onto the position on the cup and Bunsen burner was used to supply heat to the apparatus at a rate of about 5^{o}C per minute. During heating, the oil was constantly stirred. As the oil approaches its flashing, the injector burner was lighted and injected into the oil container after every12 second intervals until a distinct flash was observed within the container. The temperature at which the flash occurred was then recorded. This procedure was repeated three times and the average taken.

Determination of cloud point

The cloud point of the biodiesel produced was determined in accordance with ASTM**D 2500**.

Table 1: Actual Design

Runs	Block	Reaction Time (mins)	Oil-methanol ratio (mol/mol)	Catalyst load (g)
1	Block 1	60	0.17	1.5
2	Block 1	60	0.50	1.5
3	Block 1	60	0.17	3.0
4	Block 1	60	0.50	3.0
5	Block 1	120	0.17	1.5
6	Block 1	120	0.50	1.5
7	Block 1	120	0.17	3.0
8	Block 1	120	0.50	3.0
9	Block 1	90	0.05	2.3
10	Block 1	90	0.61	2.3
11	Block 1	90	0.33	1.0
12	Block 1	90	0.33	3.5
13	Block 1	40	0.33	2.3
14	Block 1	140	0.33	2.3
15	Block 1	90	0.33	2.3
16	Block 1	90	0.33	2.3
17	Block 1	90	0.33	2.3
18	Block 1	90	0.33	2.3
19	Block 1	90	0.33	2.3
20	Block 1	90	0.33	2.3

Sample of the biodiesel was placed in a test jar to a mark and then placed inside a cooling bath. The temperature at the bottom of the test jar that is the temperature at which the biodiesel starts to form cloud was taken as the cloud point.

Determination of kinematic viscosity

The kinematic viscosity of the produced biodiesel was determined in accordance with ASTM **D 445**. A viscometer was inserted into a water bath with a set temperature and left for 30 minutes. The sample of the biodiesel was added to the viscometer and allowed to remain in the bath as long as it reaches the test thermometer. The sample was allowed to flow freely and the time required for the meniscus to pass from the first to the second timing mark was taken using a stop watch. This procedure was repeated three times and the average value taken. This value was then multiplied with the viscometer calibration to give the kinematic viscosity.

Determination of pour point

Pour point of the produced biodiesel was determined in accordance with ASTM **D 97**. Sample of the biodiesel was kept in the freezer to about50^0C then placed in a heating mantle to melt. The temperature at which the biodiesel starts to pour is taken as the pour point.

Determination of Ash Content

The sample was put on a metal plate and placed over an ignited burner until the entire organic matter was charred. It was transferred to a muffle

furnace and maintained at 550 ^{o}C for a few hours until grey ash was obtained, after which it was cooled in a desiccators. The ash residue was weighed and values recorded (Food Safety and Standards Authority of India, 2012)

Determination of Sulphur content

Sulphur content was determined with the aid of the sulfinert method. This method was developed specifically for deactivating metal surfaces that contact organo-suphur compounds. In this method, untreated stainless steel absorbs or reacts with hydrogen sulphide. A sulphinert layer is applied to stainless steel surface which in turn prevents these sulphur compounds from reacting with the metal surface in contact. This method is useful in obtaining precise and accurate sulphur levels in samples (Restek Technical Guide, 2002).

Determination of Saponification value

1ml of methyl ester was measured and poured into a conical flask. 25ml of KOH was added to it, a blank was also used. The sample was well covered and placed in a steam water bath for 45 minutes with periodical shaking at 50 ^{0}C. 1 ml of phenolphthalein was added to the mixture and titrated against 0.5M HCl to get the end point which was a colour change from pink to colourless. The Saponification value was calculated as:

$$Saponificationvalue = \frac{(b-a)x\ 28.05}{Weightofmethylestersample} \quad (1)$$

Determination of iodine

0.25ml of biodiesel sample was poured into a glass-stopper bottle of about 250 ml capacity. 10ml of carbon tetra chloride was then added to the sample to dissolve. Subsequently, 25ml of wij's solution was added and a stopper was inserted and allowed to stay in the dark for 30 minutes. 10ml of potassium iodide solution KI,10wt% and 100ml of water was introduced and the mixture was thoroughly mixed and titrated with 0.5M sodium thiosulphate solution (Na_2SO_3) using starch as indicator (titration = '***a***'ml) which gave a pink colour. A blank is carried out at the same time starting with 10ml of carbon tetrachloride (titration = '***b***'ml).Titrating back with chlorofoam gives a colourless mixture.

$$Iodinevalue = \frac{(b-a)x\ 1.269}{Weightofpalm\ kerneloilsample} \quad (2)$$

Determination of Cetane index

The cetane index was determined according to the experiment reported by Lapuerta *et al.;* 2004 using this corelation

Cetane Index (CN) = 46.3 + 5458/SV + 0.225/IV (3)

Where SV = saponification value

IV = Iodine value

RESULTS AND DISCUSSION

The details of the results obtained in the optimization and characterization of synthesized biodiesel are as discussed in this chapter.

Optimization conditions for biodiesel production

This work investigated the effect of three different reaction parameters (ratio of ethanol to PKO, catalyst concentration and reaction time) on the yield of biodiesel produced from PKO oil using response surface methodology RSM. Effect of combination of Methanol-NaOH was also investigated to compare the results as its affect the yield.

Molar ratio of palm kernel oil (PKO)-to-alcohol

The type of alcohol and molar ratio of alcohol to oil is an important variable in transesterification process.

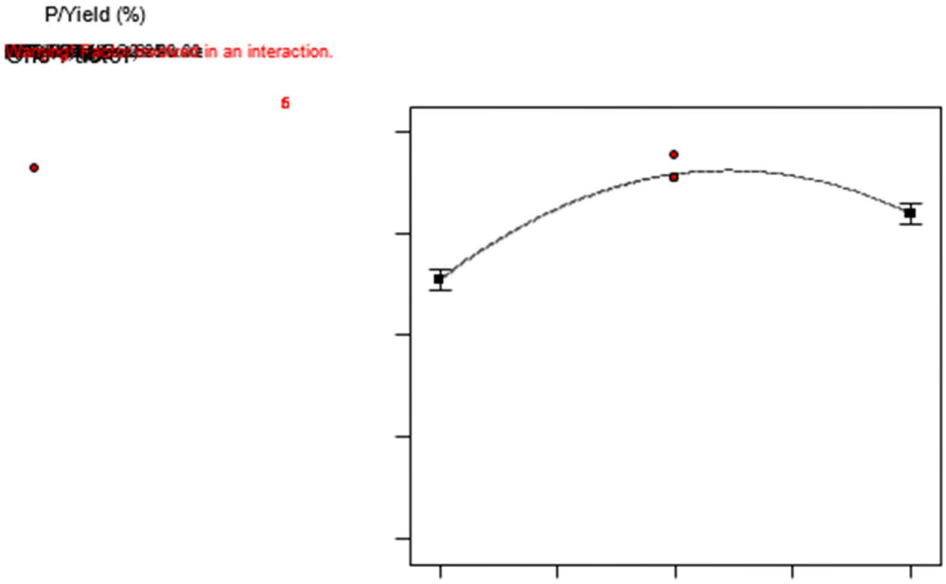

Figure I: Effect of oil to methanol ratio on methyl ester yield

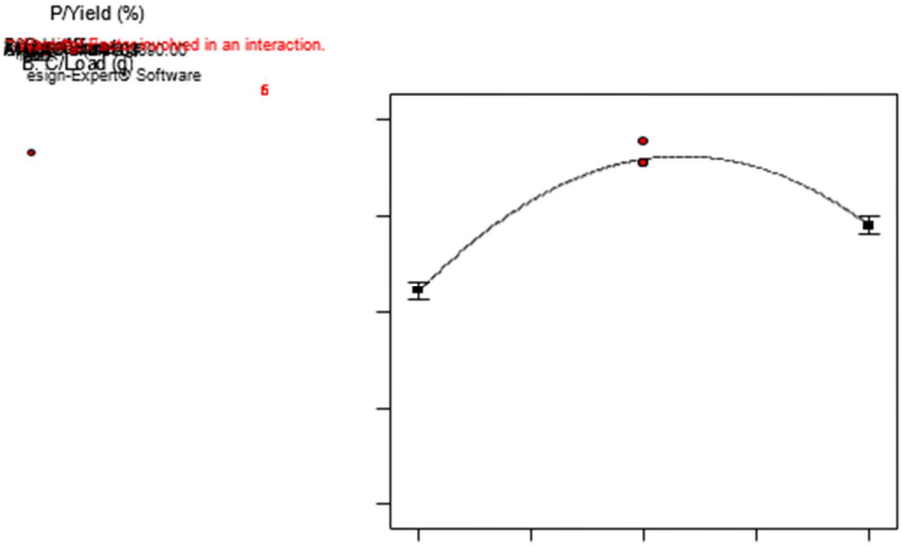

Figure II: Effect of oil to methanol ratio on methyl ester yield

As in most reaction the transesterification reaction proceeds until it reaches equilibrium. This reaction requires a 3:1alcohol/oil molar ratio,but this equilibrium can shift towards forming more products and therefore achieving higher ester conversion, which requires an excess of alcohol for complete conversion of fatty acid esters. Several studies available in the literature reported the importance of molar ratio of methanol:oil in the formation of biodiesel (Sujata, *et al.,* 2022). Molar ratio of methanol to oil is directly proportional to the biodiesel yield (Sujata, *et al.,* 2022)..

Quantity and type of catalyst

The type and quantity of catalyst used in this process is also important because it does not only speed up reaction but can also cause hydrolysis and saponification (Meher and Naik, 2006) which interfere with the separation of glycerol and

biodiesel purification. The basic catalysts are much more used than the acidic, because of their higher catalytic activity and better quality of biodiesel produced. The basic catalysts are more efficient. In this study, the effect of the concentration of the catalyst on the yield was investigated.

From the Fig. I, the maximum yield (93 wt %) was obtained at oil to methanol ratio 1:3(0.33) Higher mass ratio of reactant increases the contact between the methanol and oil molecules so the methyl ester concentration increases with increasing mass ratio of methanol to oil. But the production yield decreases with increased mass ratio of reactant (Lalita, Sukunya and Peesamai 2004).

From the Fig. II above the maximum yield (93 wt %) was obtained at oil to methanol ratio 1:3(0.33). Higher mass ratio of reactant increases the contact between the methanol and oil molecules so the methyl ester concentration increases with increasing mass ratio of methanol to oil. But the production yield decreases with increased mass ratio of reactant (Lalita *et al* 2004).

Table 2 Experimental Data of Biodiesel Yield at Different Conditions

Runs	Oil-methanol ratio (wt %)	Catalyst load (g)	Reaction time (mins)	Experimental yield (wt. %)	Predicted value
1	0.17	1.5	60	77	76.53
2	0.50	1.5	60	82	81.42
3	0.17	3.0	60	80	80.11
4	0.50	3.0	60	85	84.50
5	0.17	1.5	120	82	82.40
6	0.50	1.5	120	84	83.79
7	0.17	3.0	120	85	85.49
8	0.50	3.0	120	86	86.38
9	0.05	2.3	90	80	79.64
10	0.61	2.3	90	84	84.50
11	0.33	1.0	90	75	75.47
12	0.33	3.5	90	81	80.67
13	0.33	2.3	40	85	85.81
14	0.33	2.3	140	93	92.33
15	0.33	2.3	90	92	91.16
16	0.33	2.3	90	91	91.16
17	0.33	2.3	90	91	91.16
18	0.33	2.3	90	91	91.16
19	0.33	2.3	90	91	91.16
20	0.33	2.3	90	91	91.16

The FFA content has significant effects on the transesterification of glycerides with alcohol using a catalyst (Goodrum, 2002). A high FFA content leads to soap formation and makes the separation of the ester exceedingly difficult, and as a result, decreases the yield of the ester (Goodrum, 2002). The free fatty acid (FFA) of CPKO sample was 7.012% compared to 1.59 and 1.189% reported by ASTM and Kuwornoo and Ahiekpor (2010) respectively was extremely high while that of RPKO was 0.91% which is lower than ASTM specification but falls under values recommended for oils to be used for biodiesel production. Table 2 showed that at various molar ratio of oil to methanol and reaction time using a catalyst load of 2.3g gave a higher yield than 3.0g. However, using 3.0g of catalyst gave a high yield within a short time of 20 minutes. The FFA content has significant effects on the transesterification of glycerides with alcohol using a catalyst (Goodrum, 2002). A high FFA content leads to soap formation and makes the separation of the ester exceedingly difficult, and as a result, decreases the yield of the ester (Goodrum, 2002). The free fatty acid (FFA) of CPKO sample was 7.012% compared to 1.59 and 1.189% reported by ASTM and Kuwornoo and Ahiekpor (2010) respectively was extremely high while that of RPKO was 0.91% which is lower than ASTM specification but falls under values recommended for oils to be used for biodiesel production.

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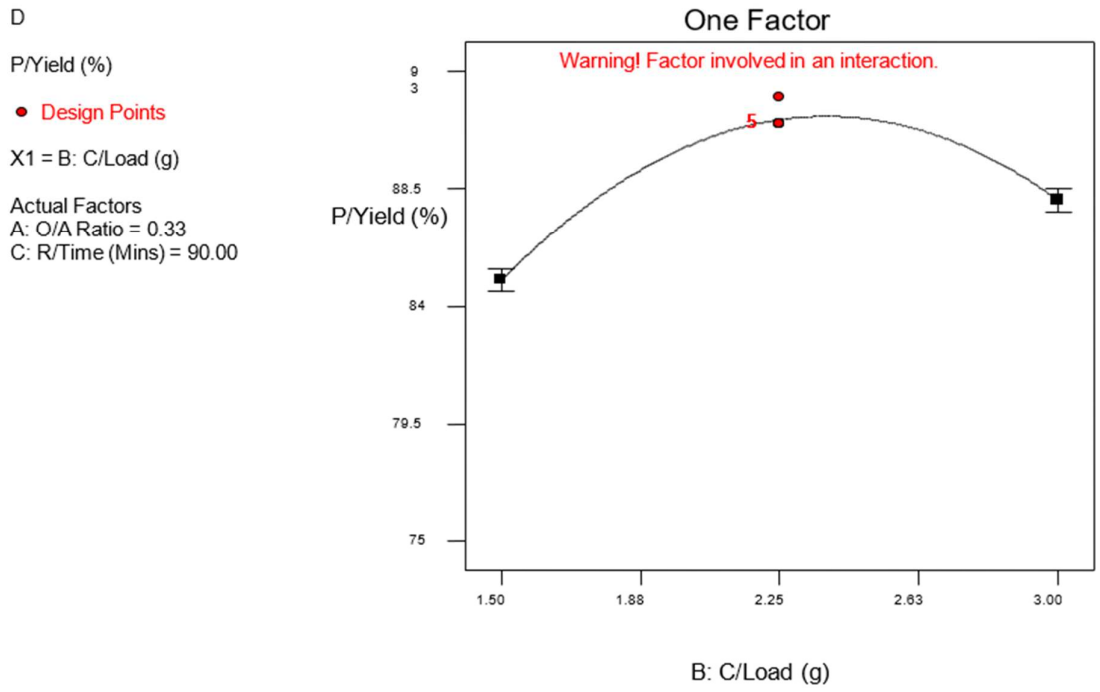

Figure III: Effect of catalyst on biodiesel yield

From the Fig. III above, the highest yield was obtained at catalyst load of 2.25g .It can be observed that the production yield decreases with increased sodium hydroxide concentration from 2.3 to 3.5 by oil weight, because of soap formation from the reaction of oil and excessive amount of catalyst used. The methyl ester concentration increases with increased catalyst concentration at lower methanol: oil mass ratio. However, catalyst concentration had no detectable effect on methyl ester concentration at higher oil methanol mass ratio.

Effect of Reaction time

An important variable is time of reaction. In general, the increase in time increases the conversion of triglycerides proportionally. The optimum point was obtained at the highest time of 140 minutes though the production yield is nearly independent of reaction time but the methyl ester concentration increases with increased reaction time. Due to the increasing of mixing and dispersion of methanol in oil phase with reaction time, which is in accord with the work of Freedman *et al.*

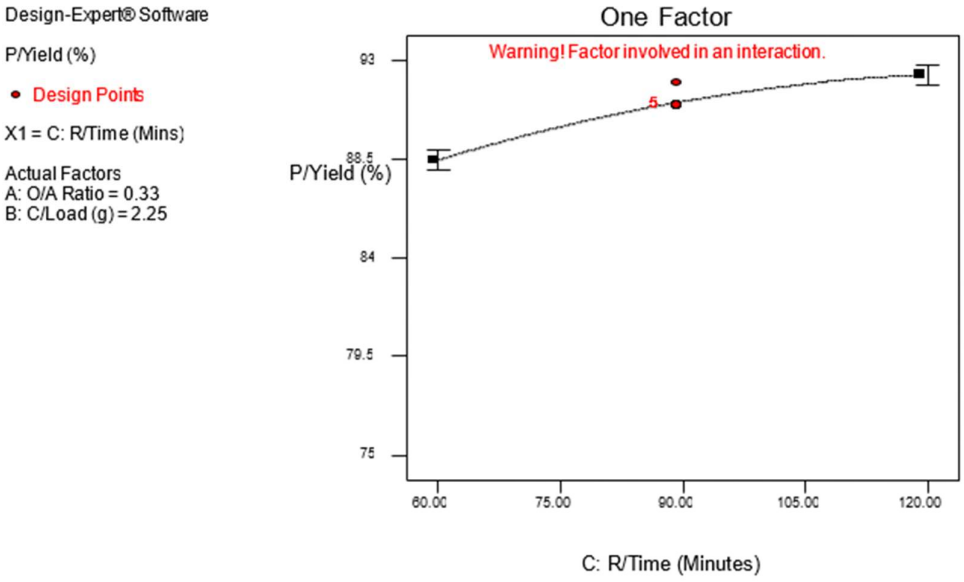

Figure IV: Effect of reaction time on methyl ester yield

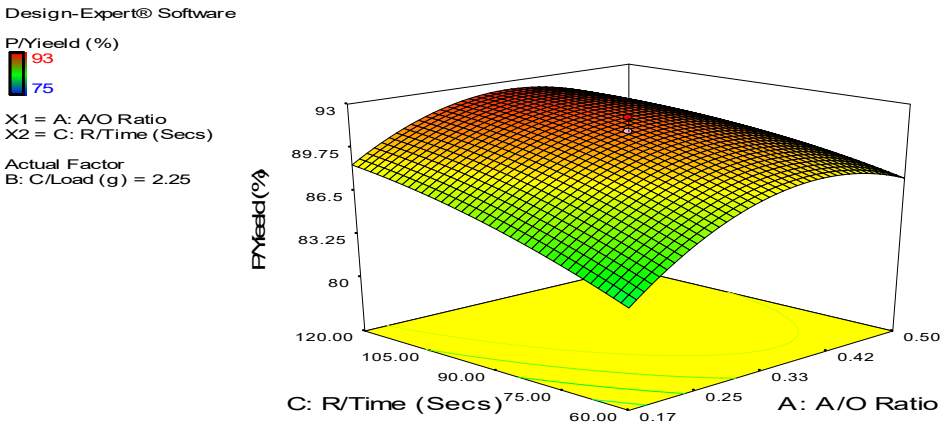

Figure VI: Interaction between reaction time and oil-methanol ratio

The optimum time gave the optimum yield this confirms the claim of David and Julius (2010); ester concentration increases with increased time. This is due to the increased mixing and dispersion of alcohol in oil phase with reaction time.

Effect of interaction between process variables on methyl ester yield

This section shows the effect of interaction between catalyst loads, oil to methanol ratio, and reaction time on biodiesel yield.

Interaction of oil: methanol ratio and catalyst load

The above fig. VI shows that increase in both time and catalyst will increase methyl ester yield but with longer period of time the yield begins to drop because all the catalyst have been used up and the equilibrium shifts to the left which favors the formation of more glycerol than methyl ester (Boonmee *et al.,*2010).

Statistical Analysis and Model

The Model F-value of 146.96 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.

Table 3 ANOVA for response surface quadratic model

Source	Sum of Square	Degree of freedom	Mean Square	F value	p-value prob>F	
Model	538.13	9	59.79	146.96	<0.0001	Significant
A-O/A Ratio	28.50	1	28.50	70.04	<0.0001	
B-C/Load(g)	32.57	1	32.57	80.05	<0.0001	
C-R/Time(minutes)	51.24	1	51.24	125.95	<0.0001	
AB	0.13	1	0.13	0.31	0.5916	
AC	6.13	1	6.13	15.05	0.0031	
BC	0.13	1	0.13	0.31	0.5916	
A^2	149.00	1	149.00	366.22	<0.0001	
B^2	308.90	1	308.90	759.19	<0.0001	
C^2	7.90	1	7.90	19.43	0.0013	
Residual	4.07	10	0.41			
Lack of fit	3.24	5	0.65	3.88	0.0814	
Pure error	0.83	5	0.17			
Cor total	542.20	19				

Values of "Prob > F" less than 0.0500 indicate model terms are significant.In this case A, B, C, AC, A2, B2, C2 are significant model terms.Values greater than 0.1000 indicate the model terms are not significant.If there are many insignificant model terms (not counting those required to support hierarchy),model reduction may improve the model.

Final equation in Terms of coded Factors

P/Yield (%) = 91.16 + 1.44A + 1.54B + 1.94C - 0.12AB - 0.87AC - 0.12BC - $3.22A^2$ - $4.63B^2$ - $0.74C^2$

Final equation in terms of actual factors

Biodiesel yield (%) = 9.45750 + 104.05888A + 39.93066 B + 0.28362C – 1.00100AB – 0.17518AC – 5.55556E-003BC – 115.98989A2 – 8.23060B2 – 8.22919E-004C2.

A=PKO/Methanol ratio, B= Catalyst load (g), C=Reaction time (minutes)

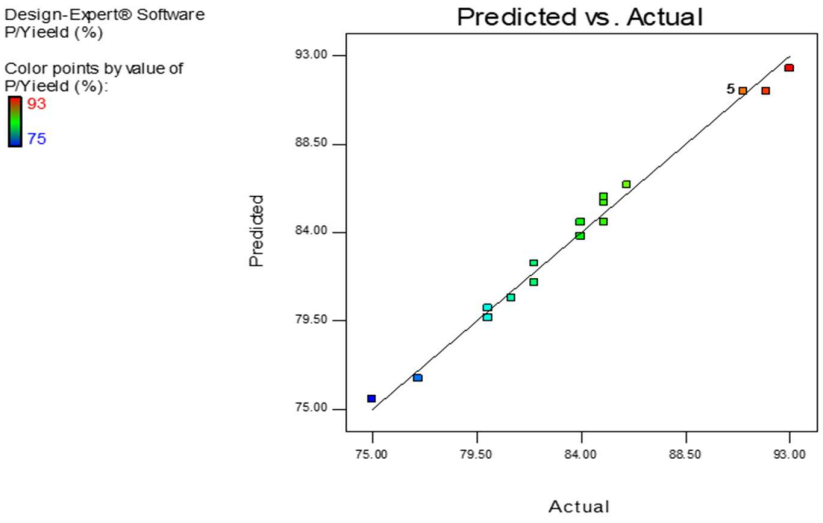

Figure VII: Predicted vs. actual

Table 4 Measured Properties of Biodiesel Produced

S/N	Properties	Unit	Experimental value	ASTM biodiesel standard (ASTM D 6751)	ASTM fossil diesel standard (ASTM D 975)	Alamu *et al*
1	Flash point	^{0}F	89	130min (100-170)	60-80	167
2	Cloud point	^{o}F	-9	-3 to 12	-15 to 5	6
3	Specific gravity	-	0.801	0.86-0.90	0.95max	0.853
6	Sulfur content	%wt	1.22	0.05 max	0.50max	-
7	Water content	%	0.02	-		
8	Kinematic Viscosity	mm^2/s	4.3	2.52-7.5	26max	4.839
10	Ash content	-	0.5	0.07max	-	-
11	Saponification value	mgKOH/g	205.33	-	-	-
12	Iodine value	cg I_2 /g	13.5	-	-	-
13	Refractive index	-	1.431	-	-	-
14	Pour point	^{0}C	6	-15 to 16	-35 to -15	2
15	p.H	-	5.2	-	-	-
16	Cetane number	-	72	47 min	-	44

Biodiesel Characteraction

Flash Point determination

The flash point of oil is the lowest temperature in which it produces vapor. It is also the lowest temperature of ignition. This is beneficial in the aspect of storage and usage, in other to avoid fire outbreak or destruction. The flash point was gotten to be 89^0 F which is lower than the specified standard value of 150-170^{o}F (ASTM D 6751), and 167^{o}F reported by Alamu *et al.* (2007). The value of the flash point is also used to classify materials into flammable and combustible for the purpose of safety and shipping regulations.

Cloud point

Formation of wax at certain temperature defines the cloud point of oil (Owen and Coley, 1990). The cloud point obtained in this work is -9^{o}F which falls within ASTM specification for fossil fuel but lower than that of biodiesel specification as shown in the Table 4.

Specific gravity

The specific gravity of RPKO biodiesel was gotten to be 0.801, a value that falls within 0.86-0.90 range specified by ASTM standard (D 6751). This value also compared well with 0.853 reported by Alamu *et al.* (2007).

Density.

The density of the biodiesel produced was found to be 801kg/m^3 which is lower than 860-900 kg/m^{3}specified by European standard for biodiesel (EN 14214).

Sulphur content

The sulphur content of the produced biodiesel was determined to be 1.22 which is very high compared to ASTM permissible limit of 0.50 (ASTM D975)

Viscosity

This is an important quality of fuel atomization and distribution.The higher the viscosity the higher the drag in the injection pump and generation of high pressure. In this work the viscosity obtained was 4.3. For biodiesel to be used in diesel engines, the kinematic viscosity must be between 1.9 and 6.0 mm^2/s at 40^{o}C (ASTM D 6751). The kinematic viscosity obtained for the optimum-yield biodiesel produced in this work was 4.3mm^2/s as shown in Table 4. Though this result exceed the standard range specified, the reduction in the kinetic viscosity of the parent crude palm kernel oil from 26.21mm^2/s to 6.56mm^2/s thus indicates that the flow capability of crude palm kernel oil has been increased to a significant extent by transesterification. This increase fuel flow ability will enhance ignition potential. More so, slight change in the value obtained could be as a result of unseen foreign contamination in the process of determination.

Water content

Water content in biodiesel will definitely promote biological growth in diesel engine which may cause blockage to fuel filter as a result of slime formation.0.02% of water was contained in the biodiesel which is very good compare to 0.05% maximum specified by ASTM standard (D6751).

Pour point

The pour point of methyl ester was gotten to be 6^0C. Pour point (PP) is the lowest temperature at which the fuel suffers from gel formation and attains semi-solid state and becomes deprive of fits flow ability, which makes it no longer pump able.

Cetane number or CN

Cetane number is an indicator of the combustion speed; it is also an important factor in determining the quality of diesel. The higher the cetane numbers the better the ignition properties of the diesel. The cetane number obtained for the biodiesel produced was found to be 72 a value above the minimum 47 stipulated by ASTM (D 6751).This is an indication of the presence of longer fatty acid chains and more saturated molecules and also an indication that it will be of higher potential for engine performance.

CONCLUSION

Biodiesel with very good yield and comparable physicochemical properties was successfully produced from refined palm kernel oil (PKO). The major characteristics include flash point (89^0F), cloud point (-9^0F), specific gravity(O.801), sulphur content (1.22), water content (0.02%), kinematic viscosity ($4.3mm^2s$), ash content (0.5), saponification value (205.33), pour point (6^0C) and cetane number(72) which were in agreement with available literature and ASTM standard. The study also shows the outcome of the optimization of biodiesel synthesis using refined palm kernel oil as the feedstock through the method of transesterification and the use of central component design (CCD) for response surface methodology (RSM). Three parameters were placed into consideration; namely catalyst load, reaction time, and mole ratio of the oil to alcohol. The optimization of refined palm kernel oil biodiesel varying the three process various gave a consideration of catalyst load of 2.23 and mole ratio of 0.33 mol/mol with a yield of 93%.

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