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Optimized Biodiesel Production from Pine Oil

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Abstract— *Biodiesel, quality is affected by several process and parameters such as temperature, catalyst, molar ratio among others. The present work was carried out to optimize the operational variables such as methanol/oil molar ratio (3:1-9:1), temperature (50ºC-80ºC), catalyst con-centration (0.7%-1%) and sodium methoxide (catalyst) with mixing intensity 600 rpm and reaction time 90 minute fixed constant throughout the reaction. Response surface methodology was used to find the codded factor (theoretical yield) and actual factor (experimental yield) and central composite design was used to design our graphs. The cetane index of B20 blends based on trans-esterification at the different temperature, molar ratio, and catalyst concentration, was also determined. The result obtained showed that the optimum set of transesterifications reaction conditions are: 7:1 molar ratio of methanol/oil, (70ºC) reaction temperature, 0.90 % catalyst concentration, and 600 rpm which produced the maximum yield 81.45 %, Also after blending with petrodiesel, the cetane index of B20 was obtained as 80.80 at 24.9 ºC. In conclusion, the methanol: oil ratio of 7:1, catalyst concentration of 0.9 % and temperature of 70 ºC gave the optimum yield of pine oil biodiesel. Optimization studies using RSM Software also agree with experimental data. Pine oil biodiesel was blend with petrodiesel to increase its stability and performance*.

Keywords— *Biodiesel: Pine oil: Transesterification: Response Surface Methodology (RSM)*.

I. INTRODUCTION

Biodiesel, as defined by ASTM, is a fuel made from monoalkyl esters of long-chain fatty acids sourced from vegetable oils or animal fats, often referred to as B100. It is created through a chemical process called transesterification, in which triglycerides react with alcohols in the presence of a catalyst, resulting in the formation of fatty acid alkyl esters. Glycerine, or glycerol, is a by-product of this process. When methanol is used as the alcohol, the biodiesel produced is known as fatty acid methyl esters.

Biodiesel have received considerable attention because of its renewable, eco-friendly attributes [12]. The properties of a biodiesel fuel that are determined by the structure of its component fatty esters include ignition quality, heat of combustion, cold flow, oxidative stability, viscosity and lubricity [26]. Because of having some advantages as higher cetane number, reduced emissions of particulates (NOx, SOx, CO) and hydrocarbons, reduced toxicity, improved safety and lower lifecycle CO_2 emissions at the same time low temperature property is the main disadvantage of biodiesel [15] Biodiesel improves the lubricity, which results in longer engine component life [2] [5] [3]. Bio-diesels are fuels obtained from the transesterification of either animal fat or used/fresh vegetable oil which is tantamount to the diesel synthesized from petroleum [10]. It is made by using a process called transesterification reaction, which is the process of converting vege-table oil or animal fats into a material that powers an engine that uses diesel. The popular process of producing biodiesel is by the employment of base-catalyzed transesterification reaction. It involves the use of a catalyst and an alcohol usually methanol or ethanol pro-ducing methyl-ester or ethyl-ester respectively. Furthermore, it has a reduced molecular weight, excellent viscosity and volatility.

The need for petroleum energy is on the high rise and according to Kolesnikov, 2011 who researched for British Petroleum Company un-der the ‘Evaluation of the Status of Global Energy’. He revealed the proven reserves to stand at 862 billion tons for coal, methane –187 trillion m3 and petroleum – 1,383 billion barrels. He reported that considering the rate at which these resources are exploited, coal may finish by the year 2129; methane may vanish in the next 60 years while petroleum may stop to find its way to the refineries in 40 years ahead. Today, various ways have been used for the analysis but none or little has shown the possibility as to the ease of readily getting it, the cost effectiveness, and the overall impact to human, plants and air. One of the possible alternatives is the transesterification of used vegetable oil and animal fat into biodiesel. The way petroleum products are known to emit some poisonous by-products to the atmosphere, fuels produced from animal fat and used vegetable oil has been recommended to be a good source of energy [16]. The aim of the study was to optimize the operational variables such as methanol/oil molar ratio (3:1-9:1), temperature (50ºC-80ºC), catalyst concentration (0.7 %-1%) and sodium methoxide (catalyst) with mixing intensity 600 rpm and reaction time 90 minute fixed constant throughout the reaction.

II. MATERIALS AND METHODS

A. Materials

Glass wares,Distilled water, Oven,Volumetric flask (250ml), Beakers (250ml, 500ml) Measuring cylinder (100ml), Seprating funnel, Soxhlet extraction unit, Heating mantle, Stirrer, Thermometre, Electronic weighing balance, Density bottle, pH meter, Hot plate Phenolphthalein indicator, Petri-dish and Detergent.

All reagents used were analytical grade without any further purification which includes, Methanol, ethanol, diehyl ether, Phenolpthalein powder, distilled water, sodium hydroxide pellets.

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B. Sample Collection

Crude sample of pine oil was purchased from Technology incubation Centre, Farm Centre Kano state.

C. Physicochemical Analyses of Pine Oil

2.80 g of oil was weighed in a 250 mL conical flask and dissolved with 50 mL of ethanol, 5drops of phenolphthalein indicator were added and the solution was titrated with 0.10eq/dm3solution, the endpoint was the pink colour. Using the equation 1 below the acid value was calculated [28].

D. Determination of Free Fatty Acid

The free fatty acid (FFA) was determined from the results of the acid value using the FFA equation [28].

$$FFA = \frac{\text{Acid value}}{2} --------(2)$$

E. Determination of Specific Gravity

$$Spcific\ gravity = \frac{c-a}{c-a} --------(3)$$

Where,
a=Weight of empty density bottle
b=Weight of density bottle + distilled water
c=Weight of density bottle + oil

F. Determination of Moisture Content

The pine oil sample (5g) was weighed in a crucible and placed in an oven at 110°C, and set for an hour, removed, and re-weighed until a constant weight was obtained [29].

G. Biodiesel Production Process

The insoluble impurities were first removed from the pine oil sample by filtration, after which the oil was heated at 110°C for 10 min to remove the moisture. The pre-treated pine oil sample was then subjected to base-catalyzed trans-esterification process using $NaOCH_3$ Sodium methoxide as catalyst to produce biodiesel. The process was carried out at fixed reaction time of 90 min and a constant mixing intensity using a reflux system on a hot plate equipped with magnetic stirrer. The reaction temperature, methanol/oil ratio and catalyst concentration were the target parameters used to optimize biodiesel production in the study. [4].

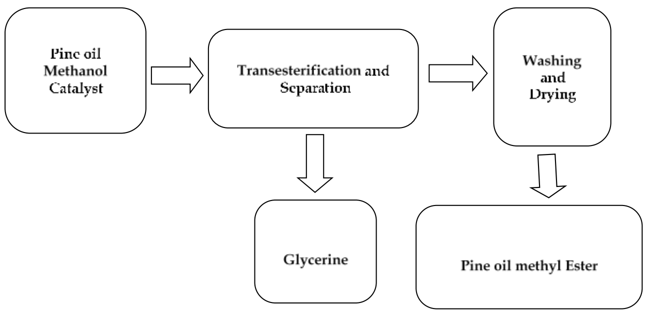

Figure 1: Block diagram indicating pine oil methyl esters production.

After the reaction is complete, the mixture was transferred into a separating funnel and allowed to settle for 24 hours to ensure the separation of two layers. The upper layer is the biodiesel, and the lower layer is the glycerol by-product. After the separation, the glycerol layer was carefully drained off while the biodiesel layer was washed with warm distilled water (around 50-60°C) several times to remove any remaining impurities or catalysts and then heated at 110°C to remove moisture and excess methanol [4].

H. Preparation Biodiesel Blend (B20)

Biodiesel blends of B20 were prepared by properly mixing 10ml of Pine oil methyl biodiesel with 40ml of petroleum diesel.

I. Determination of Cetane Number of Biodiesel

This is a measure of ignition quality of the diesel fuel; the higher this number the higher the diesel quality hence, the easier it is to start a standard (direct injection) diesel engine. The Cetane number of all the biodiesel blends was determined at National Research institute for Chemical Technology (NARICT), Zaria.

J. Parameter Analyses

Response surface methodology was used to find the codded factor (theoretical yield) and actual factor (experimental yield) and central composite design was used to design our graphs. The cetane index of B20 blends based on trans-esterification at the different temperature, molar ratio, and catalyst concentration, was also determined.

III. Results and Discussion

Physicochemical analysis of crude pine oil showed that pine oil served as good feedstock for biodiesel production, due to low FFA and most of the parameters values were within the range of ASTM (Table 1).

TABLE 1: Physicochemical Analysis of Pine Oil Compared to ASTM

Parameters	Values	ASTM
Specific gravity (g/cm^3)	0.89	0.87-0.90
Acid value(mgNaOH/g)	0.325	≤1
FFA(mgNaOH/g)	0.162	≤0.5
pH	5.46	5.3-6.07
Specific gravity of thermally heated Pine Oil(g/cm^3)	0.94	–

A. pH Values of Pine Oil, and Biodiesel (B100)

pH of crude oil is and biodiesel is very important. It is used as a guide in determining whether the oil is pure, and gives guide in deter-mining removal of excess contaminant. After the transesterification separation, the biodiesel (B100) had a pH of 8.52 while the crude pine oil had a pH of 5.46.

TABLE 2: pH Values of pine oil, and biodiesel (B100)

S/NO	SAMPLE	pH
1	Crude pine OIL	5.46
2	Pine oil B100	8.52

The free fatty acid is the percentage by weight of a specified fatty present in the oil or biodiesel for example percentage of palmitic acid. Acid value and FFA may be converted from one to another using conversation factor is calculated after knowing the acid value, the free fatty acid was determined after determining acid value of the sample. From

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Table 3, it was observed that the FFA obtained after transesterification reaction was low compared to the FFA of the oil before tranesterification reaction, which is also within the range of ASTM. FFA seriously affect the yield and quality of Biodiesel, because cetane number is inversely proportional to the degree of unsaturation associated with the fatty acid in the oil, and also during the tranesterification reaction high FFA in oil lead to the formation of more soap, which increased the viscosity of the oil, and decrease the yield of Biodiesel. Most of the values obtained are within the range of ASTM Values, except for density which is slightly above the range; this recommended that, it is better to blend Pine oil Methyl Esters with petrodiesel in other to reduce the viscosity of pine oil Methyl Esters, for better atomization, combustion and also to increase its stability. Increasing in viscosity complicates the recovery of the methyl esters (Clements and Hanna 1998; Zhang et al., 2003). In fact, this restricts the glycerin separation and it was suggested that saponification consumes the base catalyst and reduces product yields [1].

TABLE 3: Physicochemical Analysis of POME and Petrodiesel Compared to ASTM.

Parameters	Sample	Values	ASTM
Density(g/cm^3)	POME(B100)	0.90	0.88
Density(g/cm^3)	Petrodiesel	0.85	0.85
Acid value(mgNaOH/g)	B100	0.132	0.5
FFA(mgNaOH/g)	BI00	0.06	0.25
pH	B100	8.32	7-8

The reaction variables employed during optimization of pine oil methanolysis were methanol/oil molar ratio (3:1 to 9:1), catalyst ($NaOCH_3$) concentration (0.7% to 1%), and reaction temperature (50ºC to 80ºC) while the reaction time and stirring were constant at 90 minutes and 600 rpm respectively throughout the reaction.

From table 4 it was observed that as temperature increase from 50ºC-70ºC biodiesel yield increased from 57.58%-81.46%. While in-creasing temperature above 70ºC decreased the yield of biodiesel from 81.46-68.80%. In another study, Rashid et al. (2015) investigated similar effects of temperature during optimization of tranesterification for production of biodiesel using the spesia Oil, the yield directly increased with an increase in reaction temperature with optimum contents (98.1%) at 65ºC on the other hand, further in-crease in reaction temperature did not positively affect the ester yield; nevertheless a slight decrease was documented. catalyst con-centration also affect biodiesel yield as observed from below table increase in catalyst concentration from (0.7%-0.9% oil weight basis w/w) significantly increased the biodiesel yield from 77.9%-81.46%, Further increased in catalyst concentration above 0.9% de-creased the biodiesel yield from 81.46%-68.11%. It was also reported that, based on methanolysis of Thesphia Oil, catalyst concentration is also an important variable that affects the biodiesel yield. The increase in the catalyst concentration from 0.50 to 0.90% in-creased the methyl esters yield from 82.7% to as high as 98.1%, Further increase in concentration of the catalyst, i.e., >0.90% did not increase the esters yield. Rather, the yield was slightly decreased [11]. Addition of an excessive amount of alkaline catalyst leads to the formation of an emulsion through increasing viscosity and complicates the recovery of the methyl esters [7],[18]. Molar ratio also affect the yield of biodiesel there is slightly decreased of biodiesel yield from 3:1-5:1, after increasing methanol to oil ratio for about 7:1 a significant increase of biodiesel yield was observed from 75.12-81.46%. While increasing of methanol to oil ratio to about 9:1 decreased the biodiesel yield. In previous study [9], [13] who reported best esters yields utilizing the molar relation around 6:1 during the methanolysis of *Pongamia pinnata*, tobacco, and rapeseed oils respectively. Similar result also obtained from Rashid et al (2015). Methanol/oil ratio 7:1 offered the highest biodiesel yield (98.1%). When the methanol/oil molar ratio was increased further, i.e., 7:1 to 9:1, the methyl ester contents slightly decreased.

TABLE 4: Performance of Temperature, Catalyst and Molar Ratio on the Yield of POME

Temperature ºC (%Yield) 7:1, O.9wt %	Catalyst Wt % Concentration(%Yield) 7:1, 70ºc	Molar Ratio (%Yield) 70 ºC, O.9wt %
50(57.58)	0.7(77.9)	3:1(76.26)
60(76.77)	0.8(78.7)	5:1(75.12)
70(81.46)	0.9(81.46)	7:1(81.46)
80(68.80)	1.0(68.11)	9:1(61.31)

Blending biodiesel with petrodiesel may be advantageous for mitigating the poor cold flow properties of biodiesel from many lipid feed-stocks. On the other hand, blending at higher ratios may compromise cold flow properties [14].

B. Effect of Catalyst Concentration on Cetane Number

The Cetane number of biodiesel blend (B20) was determined using cetane number analyzer (AFIDA-SA6000-0, United Kingdom) [4]. The highest cetane number was obtained at catalyst concentration 0.9, molar ratio 7:1, and temperature 70 ºC, which is also the best yield among others catalyst concentration (Table 5).

TABLE 5: Catalyst concentration against Cetane number of B20 blend

Catalyst Concentration (wt%) 7:1, 70 ºC	(%Yield)	Cetane Number B20
0.7	77.9	79.30
0.8	78.7	76.10
0.9	81.45	80.80
1.0	68.11	79.90

C. Effect of Temperature on the cetane number of B20 blend

Temperature has effect on the cetane number of B20 blends, the highest cetane index was obtain at 70ºC, 0.90%w/w, catalyst concentration and 7:1 molar ratio, which is also the one that gave the highest yield (Table 6).

As observed from the result, molar ratio has effect on cetane number. Increase in methanol during transesterification of pine oil slightly decrease the cetane number of the biodiesel blends, the maximum cetane number was obtain at 3:1, 0.90 catalyst concentration and 70ºC (Table 7).

TABLE 6: Cetane number at various temperature during transesterification reaction

Temperature ^{O}C 7:1, 0.9wt %	(%Yield)	Cetane Number of B20
50	57.58	80.0
60	76.77	78.0
70	81.46	80.8
80	68.80	78.5

TABLE 7: Cetane Number of B20 Blend at Various Molar Ratio during Transesterification Reaction

Molar Ratio 70^{O}C, 0.9wt %	(%Yield)	Cetane Number of B20
3:1(76.26)	76.26	81.40
5:1(75.12)	75.12	81.30
7:1(81.46)	81.46	80.80
9:1(61.31)	61.31	79.90

D. Optimization Using Response Surface Methodology

It was observed that increased in the reaction temperature, catalyst concentration and molar ratio of methanol/oil increased the biodiesel yield (Table 8).

TABLE 8: Performance of temperature, catalyst, and molar ratio on yield of Biodiesel

Standard	Run	Catalyst (wt %)	Temperature (°C)	Molar Ratio (g/g)	% Yield
1	3	1.0	50.00	3.0	91
2	11	0.6	65.00	6.0	92
3	13	0.85	65.00	0.95	93
4	8	1.0	80.00	9.0	94
5	7	1.0	50.00	9.0	94
6	4	1.0	80.00	3.0	95
7	9	0.85	39.77	6.0	95
8	19	0.85	65.00	6.0	97
9	1	0.70	50.00	3.0	97
10	15	0.85	65.00	6.0	98
11	5	0.70	50.00	9.0	99
12	6	0.70	80.00	9.0	99
13	17	0.85	65.00	6.0	98
14	2	0.70	80.00	3.0	98
15	10	0.85	90.23	6.0	99
16	12	1.10	65.00	6.0	99
17	16	0.85	65.00	6.0	98
18	18	0.85	65.00	6.0	98
19	14	0.85	65.00	11.5	97
20	20	0.85	65.00	6	98

Model; Final Equation in Terms of Codded Factors

Biodiesel Yield =+97.83+0.86*A-0.53*B +0.8* C +0.38*A*B-0.62*A*C-0.12* B*C -0.26*A2 -0.79* B2 -0.97* C2

Final Equation in Terms of Actual Factors:

Biodiesel Yield= +63.62167+0.14927*temperature+47.03670*catalyst+2.71491 *methanol oil ratio+0.16667*temperature*catalyst-0.013889*temperature*methano oil ratio-1.1565E-003* temperature^2 -35.13617*catalyst2-0.10748*methanol oil ratio^2

E. Response Surface Plots

It was observed from Figure 2, 3, 4 and 5 that increase in catalyst concentration from 0.7 % to 0.77 % and the reaction temperature from 50°C to 57.5°C resulted in significant increase on Biodiesel yield. While further increasing of temperature above 57.5°C and catalyst concentration above 0.77 % had significant effect on Biodiesel yield [15]. In another study reported that: as reaction temper-ature increased from 45-65°C biodiesel yield increased, and also increased in catalyst concentration from 0.5-0.9%w/w also increased biodiesel yield.

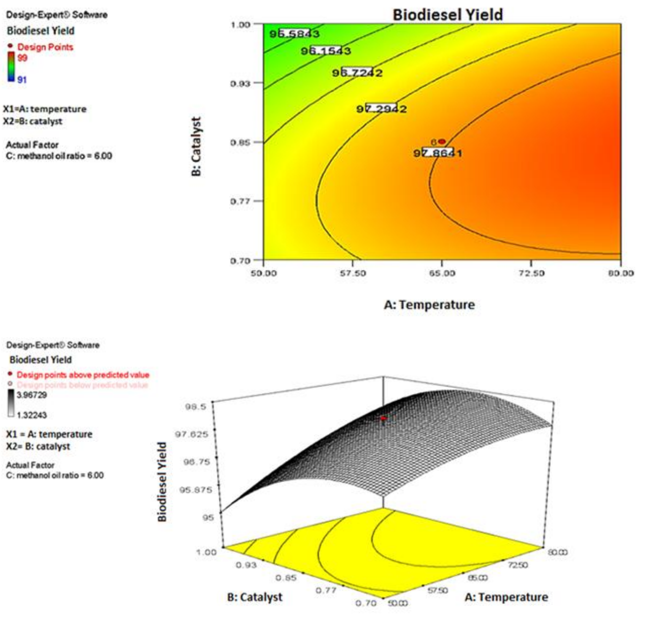

Figure 2 and 3: Effect of temperature and catalyst concentration on the yield of Biodiesel.

From Figure 3 it was observed that increase in reaction temperature from 50°C to 57.5°C and molar ratio of methanol/oil from 3 to 4.5 w/w resulted insignificant increase in Biodiesel yield from 99.72 % to 99.93 %, increase of molar ratio above 4.9 and temperature above 57.50°C had significant Effect on Biodiesel yield. [13]. Rashid et al. (2005) reported that there is significant increase in biodiesel yield from methanol/oil ratio 3:1-7:1.

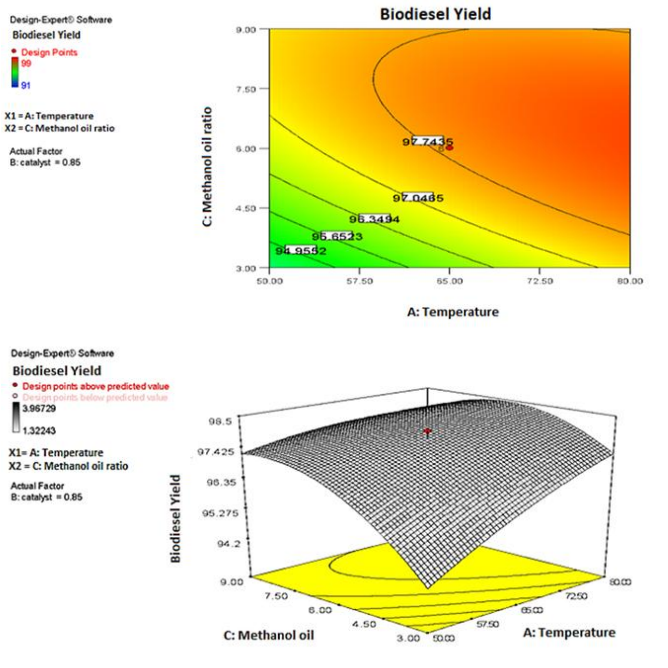

Figure 4 and 5: Effect of temperature and molar ratio on the yield of

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Biodiesel.

IV. Conclusion

Biodiesel has been successfully prepared from low free fatty acid pine oil via transesterification using a homogenous catalyst (sodium methoxide). The effect of methanol: oil ratio, catalyst concentration and temperature on the yield of biodiesel were evaluated. It can be concluded that 0.9 %, 7:1 and 70ºC gave the optimum yield of pine oil Biodiesel. Optimization studies using RSM Software also agree with experimental data. Pine oil Biodiesel properties were similar to that of petrodiesel. The pine oil biodiesel was blend with petrodiesel to increase its stability and performance.

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