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Optimum Biodiesel Production from Shea Nut Oil and Determination of Reactor Pressure by Supercritical Transesterification

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Abstract
Oil reserves are decreasing, making the transition to sustainable petroleum substitutes all the more urgent. One potential alternative to fossil fuels is bio-based diesel, which is made from vegetable and waste vegetable oils. In order to find the optimal reactor pressure for determining optimised biodiesel production from shea nut oil utilising supercritical transesterification procedures, this study used response surface methodology (RSM). Research has shown that there are obstacles to producing biodiesel via transesterification processes, despite the fact that numerous catalysts have been developed for this purpose. Problems arise when using vegetable or waste oils that are high in free fatty acid and water content, when trying to convert them into soap, when purifying biodiesel and glycerine, when there is no way to recover or regenerate catalysts after a reaction, when there is harmful wastewater generated, and when acid catalysts must have a high alcohol-to-oil molar ratio and long reaction times to achieve full conversion. The problems listed above were addressed in this study. The process parameters that was carried out were a reaction duration (10-30 minutes), a molar ratio of methanol to oil (10:1-50:1), and a reaction temperature (100-320°C). The response surface methodology used for this experiment was the Box-Behnken Design. With a reaction period of 20 minutes, and a methanol-to-oil ratio of 30:1, the supercritical technique was able to obtain a higher yield of 99.28% biodiesel and a reactor pressure of 0.51 kg/cm². With slight changes in cloud and pour points, biodiesel that was produced using supercritical transesterification met the criteria set by ASTM D-6751 and EN 14214. According to the results of the gas chromatography/mass spectrometry (GC/MS) investigation, the shea nut biodiesel included 3.43% contaminants and 96.57% methyl esters. Since the problems caused by using an acidic or basic catalyst have been resolved, supercritical transesterification has become the preferred method.

Keyword*: Biodiesel, Response surface methodology, renewable substitute, molar ratio, Shea nut oil and reaction time.*

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I. INTRODUCTION

Human existence is predicated on the availability of energy. Energy is essential for many daily activities, including cooking, driving, and powering appliances [1]. There has been a lot of recent focus on environmental protection and renewable energy. For several ecological reasons, clean and renewable energy will form the backbone of the power grid. To address this, renewable energy sources like biodiesel are being produced to serve as a viable alternative to fossil fuels. Biodiesel is a clean-burning, environmentally friendly fuel that is made using a range of chemical processes, including micro-emulsion, thermal cracking, direct usage and blending, transesterification reaction, and [2], [3], [4] and [5].

In an effort to lessen biodiesel's negative effect on the environment, a number of catalysts have been created. Although the homogeneous acid-catalyzed approach can caused problems associated with wastewater discharge, it takes a long time for the reaction to complete and uses a high alcohol-to-oil molar ratio [6]. Enzymatic methods and ion-exchange resins are making waves in the biodiesel and glycerine purification industries due to their resistance to free fatty acids and water [7]. Nevertheless, ion-exchange resins have long reaction periods and enzymes are expensive. The production of water makes catalyst recovery difficult for homogeneous catalysts, such as NaOH and KOH, which are extensively utilised [8]. Though it may experience slower reaction rates owing

to mass transfer problems and deactivation with time, heterogeneous base catalysis is the best method for transesterifying triglycerides into biodiesel because it provides efficient catalyst recovery and has low environmental toxicity [9]. According to [10], there is another method, supercritical procedure that allows for fast transesterification without catalysts at high pressures and temperatures (45 to 65 MPa).

Supercritical transesterification is a relatively new approach that promises to offer benefits over the traditional transesterification method, including faster reaction durations, catalyst-free operation, and improved final product purity. Virgin and used edible oils from crops including soybean, rapeseed, and African palm are the most popular feedstocks [11].

The supercritical transesterification process was carried out in a batch reactor in this study. In order to optimise the process parameters for producing biodiesel from shea nut oil by supercritical transesterification, we looked at reaction temperature (100-320°C), molar ratio of methanol to oil (10:1-50:1), and reaction time (10-30 minutes). The transesterification operations were carried out using the Box-Behnken Design (BBD) response surface methodology. We documented the yield percentages, characterised the produced shea nut biodiesel, and looked at the GC-MS data from the supercritical transesterification.

II. MATERIALS AND METHODS

2.1 MAERIALS

Shea nut oil was sourced from a local market in Benin City, Nigeria, while oyster shells and plantain peels were obtained from a local eatery in Warri, Delta State. These materials were used to produce fatty acid methyl ester and were acquired fresh. Chemicals and reagents were sourced from local analytical chemical dealers and reputable global manufacturers, ensuring high quality.

2.2 METHODS

2.2.1 Biodiesel production and its characterization by supercritical transesterification.

The process parameters that carried out were a reaction duration (10-30 minutes), a molar ratio of methanol to oil (10:1-50:1), and a reaction temperature (100-320°C). The response surface methodology used for this experiment was the Box-Behnken Design. The total number of experimental runs was seventeen. Since a catalyst is not required for the supercritical transesterification process, 100 g of shea nut oil and an equal amount of methanol were added to the reactor. Placing the reactor on the magnetic stirrer that maintained a constant temperature was considered to initiate the reaction time in all tests. Glass wool was used to lag the reactor, and it was covered with foil. All through the reaction, the reactor was constantly agitated at a speed of 100 rpm. The resulting shea nut biodiesel was evaporated using a rotary evapourator. Results were obtained for the biodiesel yield (response one) ranging from Y_1% to Y_{17}% and for the reactor pressure (response two) ranging from P_1 to P_{17}. The reaction byproduct was transferred to a separating funnel and left to settle for approximately ten hours (10 hrs) in order to separate the biodiesel and glycerol layers [12].

$$\text{Biodiesel yield} = \frac{mass\ of\ biodiesel\ produced}{mass\ of\ shea\ nut\ oil} \times 100$$

Table 1: Coded and actual levels of BBD variables for supercritical transesterification

Name	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A: Temperature (°C)	100.00	320.00	-1 ↔ 100.00	+1 ↔ 320.00	210.00	77.78
B: Mole ratio	10.00	50.00	-1 ↔ 10.00	+1 ↔ 50.00	30.00	14.14
C: Time (minutes)	10.00	30.00	-1 ↔ 10.00	+1 ↔ 30.00	20.00	7.07

2.2.2 Crude Biodiesel Purification

Warm distilled water was used to wash the crude biodiesel in a separating funnel after maximal separation. The biodiesel was diluted with distilled water to avoid emulsions and allowed to percolate slowly because glycerol and methanol are highly soluble in water [13]. Once the water no longer had any colour to it, the operation was stopped. This meant that all contaminants had been removed.

III. RESULTS AND DISCUSSIONS

3.1 Properties of shea nut oil.

Table 2: Physiochemical properties of the Shea nut oil

S/N	Parameter	Literature Value Range	Experimental Result	Remarks
1.	Density (g/cm³)	0.90 – 0.93	0.9336	This was slightly over the typical range
2.	Viscosity	50-100	92	Reflects moderated thickness
3.	Specify gravity	0.91-0.92	0.9336	This was the same with the density, indicating purity

4.	Acid value (mg KOH/g)	3.5-6.0	26.367	It shows a High acid value, indicated a High FFA
5.	FFA mgKOH/G	1.75-3.0	13.184	High FFA content,
6.	Saponification value (mg KOH/g)	160-195	213.567	Higher than the typical range, this was because the oil has started ranciding
7.	Peroxide value (millieequivalents O_2/kg)	1.0-5.0	172	Extremely high, indicating significant oxidation
8.	Iodine value (g I_2/100g)	50-70	114.21	High unsaturation
9.	Flash point	225-245	230	Within range
10.	Pour point	12-20	15	Moderated pour point
11.	Cloud Point	15-22	17	Between the typical range
12.	Centipoise	40-90	80	Indicated a thick Consistency
13.	Water content	<7	5	
14.	FTIR	Matches typical O-H, C=O	O-H at 3526.01 cm^{-1} C-H at 418.65 cm^{-1}	Confirmed the presence of Fatty acids and triglycerides characteristic of Shea nut.

FTIR Analysis of shea nut oil

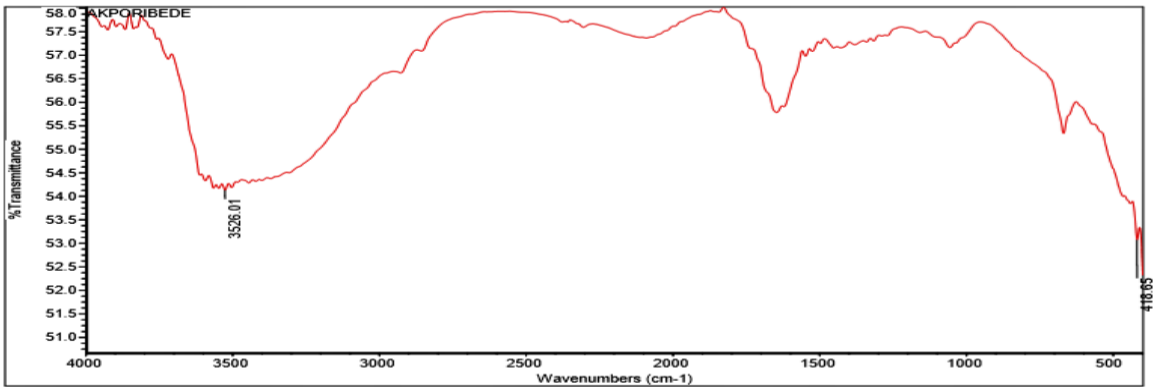


Figure 1: Shown the FTIR of the Shea nut oil

According to FTIR spectrum research, which revealed a peak at 3526.01 cm^{-3} for O-H stretching and a peak at 418.65 cm^{-3} for C-H bonding, shea nut oil does in fact contain functional groups, particularly fatty acids and triglycerides. This mixture lent credence to its hydrating claims made for use in cosmetics and lotions [14].

3.2 Process Modelling, optimization and biodiesel synthesis from shea nut oil via supercritical transesterification

The experimental design for the transesterification reaction of shea nut oil to produce biodiesel, including experimental results, RSM, and predicted responses, is detailed in Table 4. The quadratic model on an actual-basis is represented by Equation 2.

Biodiesel Yield = + 99.28 + 5.07 A − 5.16 B − 1.12 C − 14.38 AB − 2.75 AC − 8.54 BC − 25.87 A^2 − 3.73 B^2 − 17.70 C^2 (2)

To predict the response at various concentrations of each component, one can use the equation expressed in terms of coded factors. Level +1 represents the highest level of the component and level -1 represents the lowest level. This coding is done automatically. You can compare the coefficients of the elements to find out how important they are to the coded equation.

According to Equation 2, the % biodiesel production was positively affected by reaction temperature (A), and negatively by methanol: oil ratio (B), reaction duration (C), interaction factors (AB, AC, and BC), and quadratic factors (A^2, B^2, and C^2). At the 95% confidence level, model terms are deemed significant if their p-value is less than 0.05. That model term represents an input variable, and its values have a substantial impact on the biodiesel production yield. On the other hand, if the p-value of a model term is more than 0.05, it means that the input elements' values can be changed without significantly affecting the biodiesel production yield.

Factor coding provides a significant model with an F-value of 76.00 and a Type III partial sum of squares, which means that the result is not due to random fluctuation [15]. Important terms in the model with P-values less than 0.0500 include A, B, AB, BC, A^2, B^2, and C^2. Values greater than 0.1000 are deemed insignificant [16].

Biodiesel yield % respond, ANOVA for the RSM Quadratic model (supercritical method)

Table 3: Response 1: **Biodiesel Yield**

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	6090.53	9	676.73	76.00	< 0.0001	Significant
A-Temperature	205.64	1	205.64	23.09	0.0020	
B-Mole ratio	213.00	1	213.00	23.92	0.0018	
C-Time	10.08	1	10.08	1.13	0.3227	
AB	826.56	1	826.56	92.82	< 0.0001	
AC	30.14	1	30.14	3.38	0.1084	
BC	291.73	1	291.73	32.76	0.0007	
A^2	2818.47	1	2818.47	316.51	< 0.0001	
B^2	58.66	1	58.66	6.59	0.0372	
C^2	1319.49	1	1319.49	148.18	< 0.0001	
Residual	62.33	7	8.90			
Lack of Fit	62.33	3	20.78			
Pure Error	0.0000	4	0.0000			
Cor Total	6152.86	16				

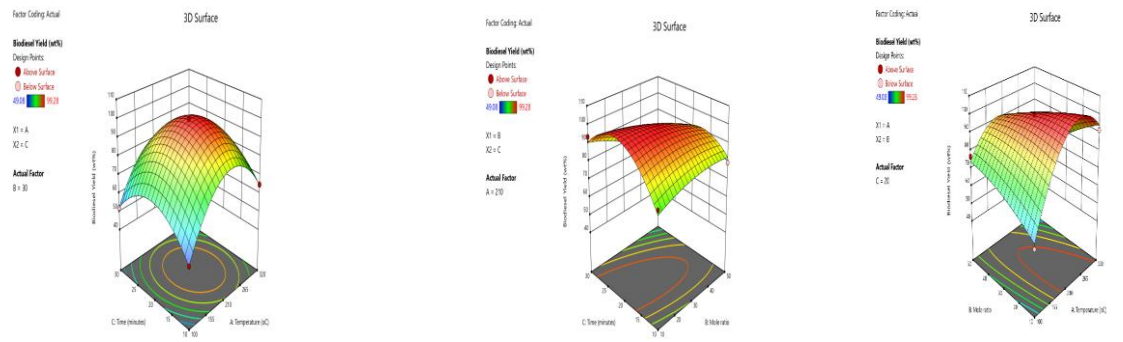
In the fit statistics data, we found an R^2 of 0.9899, an adjusted R^2 of 0.9768, and a projected R^2 of 0.8379. When the difference is less than 0.2, the anticipated and adjusted R^2 values match, so we can use this model to predict the biodiesel synthesis process. We also found a signal-to-noise ratio of 21.9635, which indicates a strong signal, and we can use this model to investigate the design space.

Table 4: Transesterification experimental design with RSM

Run	Temperature (°C)	Mole ratio	Time (minutes)	Biodiesel yield (wt %)	Reactor Pressure (kg/cm^2)
1	320	30	10	64.9	0.28
2	210	10	10	78.21	0.27
3	100	50	20	76.7	0.36
4	210	30	20	99.28	0.51
5	100	30	10	49.08	0.24
6	320	50	20	57.9	0.43
7	100	10	20	52.7	0.19
8	100	30	30	52	0.11
9	320	10	20	91.4	0.36
10	210	10	30	93.37	0.37
11	210	30	20	99.28	0.51
12	210	30	20	99.28	0.51
13	210	30	20	99.28	0.51
14	210	30	20	99.28	0.51
15	320	30	30	56.84	0.38
16	210	50	10	79.4	0.48
17	210	50	30	60.4	0.45

Effect of input variables on biodiesel yield

The effects of process parameters on biodiesel production were analyzed by three-dimensional (3D) surface plots.



(a) (b) (c)

Figure 2: 3D plots showing interaction between (a) Methanol/oil and reaction temperature (b) Reaction time and reaction temperature (c) Reaction time and Methanol/oil ratio.

Figure 2: shows the 3D surface plots representing two independent variables while keeping other variables constant. According to the Table 3, it was discovered that reaction temperature and methanol oil molar ratio

were significant, indicating changes in their values will significantly affect biodiesel yield. Reaction time was insignificant.

Figure 2 (a, b, and c) shows the effect of the process variables on biodiesel yield. With increase in methanol /oil ratio, there was an increase in the biodiesel yield also, with maximum yield obtained at 30:1. Increase in biodiesel yield with increase in reaction temperature. Although after maximum yield was obtained at a temperature of 210^0C, there was no significant increase or reduction in the yield of biodiesel produced. With increase in reaction time, an increase in biodiesel yield was observed. Maximum yield of biodiesel (99.28 %) was achieved at 20 minutes.

3.3 Process Modelling, Optimization, biodiesel synthesis and determination of reactor pressure during supercritical transesterification.

The experimental design of the transesterification reaction of the shea nut oil to produce biodiesel with the experimental, RSM and predicted responses are presented in Table 4. Equation 3 represents the quadratic model (actual-basis)

Reactor Pressure = +0.5100 + 0.0687 A + 0.0662 B + 0.0050 C − 0.0250 AB + 0.0575 AC − 0.0325 BC − 0.1575 A^2 − 0.0175 B^2 − 0.1000 C^2 (3)

Equation 2 shown that although interaction factors (AB and BC) and quadratic factors (A^2, B^2, and C^2) had negative impacts on the reactor pressure, reaction temperature (A), methanol:oil molar ratio, reaction duration, and interaction (AC) had favourable effects. Model terms with a p value of less than 0.05 are deemed significant at a 95% significance level. This implies that the reactor pressure will be significantly impacted by changes in the values of the input variable represented by that model term. On the other hand, model terms with p values higher than 0.05 are regarded as inconsequential, meaning that variations in the input factors' values won't have a major effect on the reactor pressure.

The quadratic models A^2 and C^2 from Table 5 were shown to be significant in predicting the reactor pressure, with p values less than 0.0001. Using factor coding, the analysis finds a significant model with an F-value of 89.87 and a Type III partial sum of squares, meaning that there is only a 0.01% chance that this result is due to random fluctuation. A, B, AB, AC, BC, A^2, and C^2 are notable model terms with P-values less than 0.0500. Values greater than 0.1000 are considered insignificant [15]. In situations when there are several unnecessary variables, model reduction may be beneficial. As summarised by [16], the use of P-values in statistics is controversial, highlighting discussions about the distinction between statistical and scientific significance, misconceptions about non-significant values, difficulties with point null hypotheses, and difficulties with multiple comparisons. This discussion aims to expand on their understanding with simplified answers.

Reactor pressure ANOVA for the Quadratic model

Response 2: Reactor Pressure (kg/cm^2)

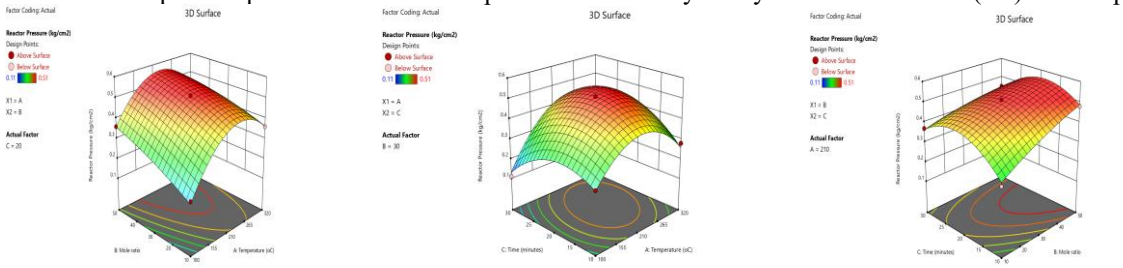
Table 5: ANOVA for the Quadratic Model

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	0.2513	9	0.0279	89.87	< 0.0001	significant
A-Temperature	0.0378	1	0.0378	121.70	< 0.0001	
B-Mole ratio	0.0351	1	0.0351	113.01	< 0.0001	
C-Time	0.0002	1	0.0002	0.6437	0.4488	
AB	0.0025	1	0.0025	8.05	0.0252	
AC	0.0132	1	0.0132	42.56	0.0003	
BC	0.0042	1	0.0042	13.60	0.0078	
A^2	0.1044	1	0.1044	336.15	< 0.0001	
B^2	0.0013	1	0.0013	4.15	0.0811	
C^2	0.0421	1	0.0421	135.51	< 0.0001	
Residual	0.0022	7	0.0003			
Lack of Fit	0.0022	3	0.0007			
Pure Error	0.0000	4	0.0000			
Cor Total	0.2535	16				

R^2 of 0.9914, adjusted R^2 of 0.9804, and anticipated R^2 of 0.8627 were displayed in the fit statistics data. The anticipated R^2 and adjusted R^2 values match if the difference is less than 0.2. As a result, the biodiesel synthesis process may be predicted using this model. The signal-to-noise ratio is quantified with sufficient precision. It is better to have a ratio larger than four. Your ratio of 28.0154 indicates a strong signal. You can investigate the design space with the aid of this model.

Effect of input variables on reactor pressure during supercritical transesterificatuion

The effects of process parameters on reactor pressure were analyzed by three-dimensional (3D) surface plots.



(a) (b) (c)

Figure 3: 3D plots showing interaction between (a) Methanol/oil and reaction temperature (b) Reaction time and reaction temperature (c) Reaction time and Methanol/oil ratio.

While all other variables were held constant, Figure 3 displays 3D surface plots representing two independent variables. Table 3 shows that reaction temperature and methanol oil molar ratio were significant, suggesting that changes in these values will have a significant impact on biodiesel yield. Reaction time was found to be insignificant.

In Figure 3 (a, b, and c), the influence of the process variables on the pressure within the reactor was shown. Reactor pressure during supercritical transesterification is affected by a number of variables, including temperature, reaction time, and the methanol to oil mole ratio. The reactor pressure was observed at 0.51 kg/cm², and 99.28% of the biodiesel was produced. Evaporation at critical limits is a potential side effect of increasing reactor pressure to a level where methanol boils. Excessive heat above 210°C may reduce pressure and yield due to the presence of unsaturated fatty acids in triglycerides. Through optimisation using the Box-Behnken design, the ideal parameters for synthesising fatty acid methyl ester (FAME) were determined to be 210°C, a 20-minute reaction duration, and a 30:1 methanol-to-oil ratio, and noted the reactor pressure at 0.51 kg/cm² and achieving a yield of 99.28%.

3.4 Performance of the RSM in supercritical transesterification method.

Based on their statistical values, the models predicted accuracy for transesterification was evaluated, and the results are shown in Table 6. At 0.9899, Supercritical achieved the high R^2 value. The experimental vs anticipated value charts in Figure 4, which are based on the models' R^2, support this conclusion. The values predicted by the RSM model, the actual values aligned closer to the reference line. Therefore, in the transesterification of shea nut oil, the predictions made by the RSM model were perfect.

Table 6: Fit Statistics

	Supercritical
R^2	0.9899
Adjusted R^2	0.9768
Predicted R^2	0.8379
Adeq Precision	21.9635
Std. Dev.	2.98
Mean	77.02
C.V. %	3.87

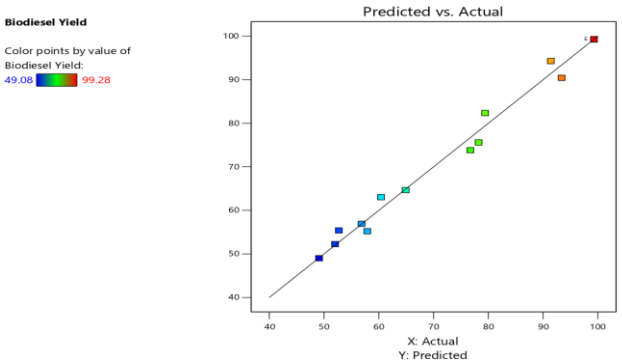


Figure 4: Predicted biodiesel vs actual biodiesel yield

Optimization of shea nut biodiesel yield

According to the numerical optimisation studied, optimum biodiesel yield by supercritical method of 99.28 % and reactor pressure of 0.51 kg/cm² was obtained with a methanol/oil ratio of 30:1, reaction temperature of 210°C, and reaction time of 20 minutes. Experimental runs were conducted in triplicate utilising the ideal parameters in order to verify these optimal circumstances. The supercritical method produced an average yield of 99.06%, showing a strong correlation with ideal yield.

3.5 Biodiesel Characterization

An examination of the physicochemical properties of the shea nut biodiesel produced under optimal circumstances yielded the results presented in Table 7. The biodiesel's properties are within the required range when compared to those of biodiesels required by ASTM D6751 and EN14214. Figure 5 shows the results of the supercritical method's sample analysis, and Table 8 shows the fatty acid profile of shea nut biodiesel that was created using the GC-MS test.

Table 7: Physiochemical properties of Methyl ester generated and Biodiesel Standards

Parameter/Unit	Supercritical Methyl Ester Values	ASTM Specifications (D6751)	EN 14214	N0.2 DIESEL
Specific gravity	0.890	0.88	1.9-6.00	0.851
Acid Value mgKOH/g	0.35	0.5 max	0.5max	0.5 max
Cloud point (^{0}C)	18	-3 to 12		-15 to 5
Kinematic viscosity @40^{o}C (mm^2/s)	6.0	1.9 – 6.0	3.5-5.0	1.9 to 4.1
Pour point (^{0}C)	15	-15 to 16		-35 to 15
Density (g/ml)	9.096	8.5 to 9.0	51minimum	0.834
Flash point (^{0}C)	170	110 to 170	>101	>55
FFA (mg KOH/g)	0.51	3 max		

Table 8: Fatty acid profile of biodiesel produced by supercritical transesterification

Pk#	RT	Concentration %	Compound IUPAC name	Compound Common name	Molecular weight (g/mol)	Molecular formular
1	4.821	2.03	Bemzoic acid, methyl ester	Methyl Benzoate	136.1479	$C_6H_5COOCH_3$
2	5.113	6.02	Octanoic acid, methyl ester	Methyl octanoate	158.241	$C_9H_{18}O_2$
3	7.567	3.86	Decanoic acid, methyl ester	Methyl decanoate	186.2912	$CH_3(CH_2)_8COOCH_3$
4	9.567	30.47	Dodecanoic acid, methyl ester	Methyl Laurate	214.3443	$C_{13}H_{26}O_2$
5	10.159	0.63	Dodecanoic acid	Lauric acid	200.32	$C_{12}H_{24}O_2$,
6	11.716	8.82	Methyl tetradecanoate	Myristate methyl ester	242.3975	$C_{15}H_{30}O_2$
7	13.215	0.64	1,2-Diphenyl Benzene	O-Terphenyl	230.3	$C_{18}H_{14}$ or $C_6H_5C_6H_4C_6H_5$
8	13.484	5.91	Pentadecanoic acid, 14-methyl-methyl ester	Methyl 14-methylpentadecanoate.	270.451	$C_{17}H_{34}O_2$
9	14.148	1.13	p-Dicyclohexy 1 benzene	DiDIcylohexlbenzene	242.3999	$C_{18}H_{26}$
10	14.537	1.03	p-Dicyclohexy benzene	DiDIcylohexlbenzene	242.3999	$C_{18}H_{26}$
11	14.903	19.76	9-Octadecenoic acid (Z)-, methyl ester	Methyl oleate	296.5	$C_{19}H_{32}O_2$
12	15.103	19.70	Stearic acid, methyl ester	Methyl stearate	298.504 g/mol	$C_{19}H_{38}O_2$

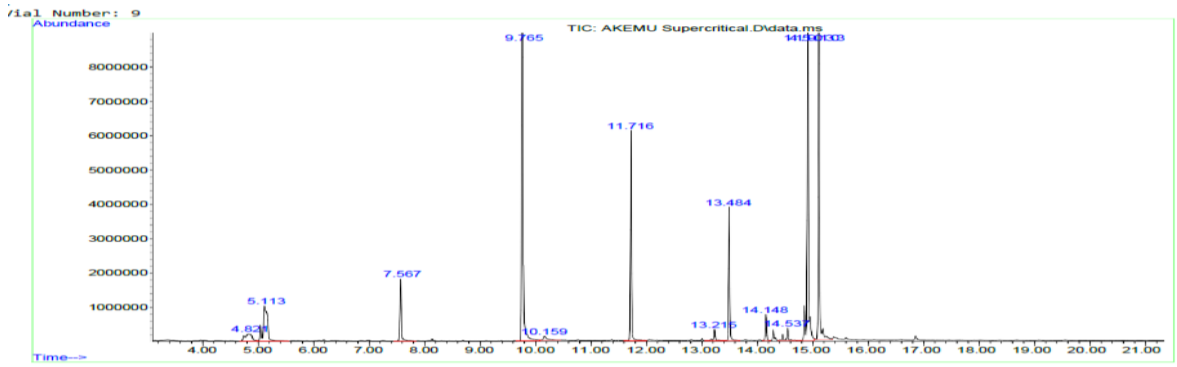


Figure 5: Chromatogram of biodiesel produced by supercritical transesterification

IV. CONCLUSION

The RSM showed strong predictive ability with an R^2 value of 0.9899 for the supercritical method. Additionally, RSM was effectively used to anticipate and optimise the process variables for the production of biodiesel. The optimised conditions for producing biodiesel using the supercritical method were a methanol/oil

ratio of 30:1, a reaction temperature of 210℃, and a reaction period of 20 minutes. The yield was 99.28% and the reactor pressure was 0.51 kg/cm2. The manufactured biodiesel met all requirements set out by ASTM D6751 and EN 14214. According to gas chromatography/mass spectrometry (GC/MS) research, the supercritical process produced methyl esters with an accuracy of 96.57% and an impurity level of 3.43%. Because of its shorter reaction time, ability to overcome problems caused by using acidic or basic catalysts, and potential usefulness as a replacement fuel for compression ignition engines, supercritical transesterification is preferred.

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