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OPTIMIZATION OF BIODIESEL PRODUCED FROM CHICKEN FATS USING RESPONSE SURFACE METHODOLOGY

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Abstract: Heterogeneous transesterification of chicken fat oil (CFO) to biodiesel over Zn/MgO catalyst and the optimization of the process have been investigated, response surface methodology (RSM) was employed to study the relationships of methanol to oil ratio, catalyst loading, and reaction time on methyl ester yield. The experiments were designed using Central Composite by applying 2^4 full factorial designs with two central points. Transesterification of CFO produced 92% maximum methyl ester yield at the optimum methanol to oil ratio = 12:1, catalyst loading = 4% w/w and reaction time = 90mins. The chicken fat methyl ester (CFME) properties like specific gravity, kinematic viscosity, flash point, fire point, cloud point, cetane number, sulphur content, and diesel index were determined and found to be 0.8528kg/l, 5.139cSt, 267k, 265k, 428k, 42 , 0.032%wt, and 45. However, interaction between methanol oil ratio and catalyst gave the largest effect on the chicken fat oil methy ester yield. The result indicated that RSM can be used to find the relationships among process variable response in efficient manner using minimum number of experiment.
Keywords: Catalyst; Trans-esterification; Biodiesel; Heterogeneous; Methyl ester

1 INTRODUCTION

Biodiesel has emerged as a promising renewable energy source due to its biodegradability, non-toxicity, and compatibility with existing diesel engines, amid growing environmental concerns and the need to reduce reliance on fossil fuels. The use of non-edible seed oil as raw materials to produce biodiesel has been recommended by many researchers to avoid the issues of hunger threats and high production costs associated with using edible seeds [1]. The choice of a suitable catalyst is another serious threat to the cost of biodiesel production today. The most commonly used catalysts in transesterification process are homogeneous catalysts like NaOH, KOH, $NaOCH_3$, $KOCH_3$, H_2SO4, HCl, and H_3PO4. The continuous application of these catalysts has a significant associated problem. The overall production cost increases due to catalyst expensive, energy consumption, severe corrosion problem, soap formation, generation of large waste water, and difficulty in catalyst recovery, despite achieving high yield and requiring shorter reaction time [2]. Researchers recently explored the feasibility and viability of non-edible feedstock and heterogeneous catalysts as catalyst options [3]. Producing biodiesel from waste animal fats, like chicken fat, is an appealing sustainable approach that reduces competition for edible feedstocks such as vegetable oils [4]. Chicken fat is a low-cost by-product with high lipid content, suitable for biodiesel production. Animal fats with high free fatty acid content often require pretreatment and process optimization to maximize biodiesel yield and quality.
Response Surface Methodology (RSM) is a statistical method frequently used to improve complicated processes, such as biodiesel production, by examining the impacts and interactions of many process factors. RSM enables researchers to improve important parameters—including temperature, catalyst concentration, reaction duration, and methanol-to-oil ratio—by constructing a prediction model for response variables, such as biodiesel yield and quality [5]. In biodiesel production from chicken fat, controlling these factors is critical for obtaining high conversion rates, lowering production costs, and ensuring the finished product fulfills biodiesel criteria [6]. This study attempts to improve the biodiesel manufacturing process from chicken fat using RSM to discover the best combination of reaction parameters. The outcomes of this study are likely to give significant insights into sustainable biodiesel synthesis from waste animal fats, highlighting RSM's efficacy in refining the process to boost output and efficiency [7].

2 MATERIAL AND METHOD

2.1 Materials

Chicken fat was purchased from Kabuga and roasted in a laboratory. The oil was heated to 500C and collected. Glassware was soaked in dilute nitric acid for 24 hours and rinsed in distilled water before experiments. Chemicals, reagents, and solvents used were analytical and general purpose grade. Methanol and NaOH were used as raw materials, and zn/MgO was used as a heterogeneous catalyst.

2.2 Pretreatment of the Chicken Fat

The free fatty acid (FFA) of chicken fat was found to be 4.16%, which needs to be reduced to ≤ 3% before transesterification. The chicken fat was dissolved using a magnetic stirrer at 60°C for 30 minutes, then filtered to remove suspended substances. 50g of the fat was weighed and poured into a 500ml conical flask. 20% of hydrochloric acid was added to the alcohol, mixed with the alcohol, and poured into a water bath at 60°C for 1 hour and 20 minutes. After the pretreatment, the mixture was poured into a separating funnel and left to settle overnight. The FFA of the pretreated fat was analyzed again, finding it to be 0.477%, which is very good for transesterification.

2.3 Physico-chemical Analysis of Chicken Fat Oil

The following are the physiochemical properties of the chicken fat oil that were determined Acid Value, Free fatty acid, pH of biodiesel, saponification value using AOCS methods and specific gravity was determined using ASTM D1298.

2.4 Synthesis of Biodiesel Using Zn/MgO Catalyst (Transesterification)

Zhu et al. [8]used a method to produce chicken oil-based biodiesel. The process involved pouring 40g of oil into a 500ml flask, adding 0.28g of Zn/MgO catalyst, and adding 20ml methanol. The mixture was refluxed under specific conditions, such as 1% catalyst loading, 60°C reaction temperature, 90 minutes reaction time, 9:1 methanol to oil molar ratio, and 600rpm agitation velocity. The mixture was then allowed to stand overnight for settling. The process was repeated with fixed amounts of fat oil sample, methanol molar to fat oil ratio, catalyst concentration, reaction time, and temperature. The oil yield was calculated using the following equation.

$$\% \text{ yield} = \frac{\text{weight of extractring oil}}{\text{weight of oil before extraction}} \text{ x } 100 \qquad (1)$$

The study analyzed yields based on temperature and reaction time. The highest yield was then assigned to the next temperature. In the second experiment, constant conditions were maintained, including temperature, methanol molar to fat ratio, catalyst concentration, and reaction time. The highest yield was then assigned to the next reaction time. The reaction time, catalyst amount, and temperature were also kept constant. The methanol molar to fat oil ratio was varied, and the highest yield was maintained at a constant temperature, reaction time, and catalyst concentration.

2.5 Influence of Reaction Parameters

2.5.1 Influence of methanol to oil molar ratio, catalyst loading, and reaction time

The study investigated the impact of varying methanol to oil molar ratio on biodiesel production rate. The ratio varied from 3:1 to 18:1 at a constant catalyst loading, 65°C temperature, 7h reaction time, and 600 rpm agitation velocity. After completion, the mixture was transferred to a separating funnel and allowed to stand for 24 hours. In order to determine the amount of catalyst on biodiesel production, various amount of catalyst ranging from 1% to 6% at constant reaction temperature of 65oC, 7 h reaction time and 600 rpm agitation velocity including the optimum methanol to oil molar ratio obtain above. After completion of the reaction, the reaction mixture was transferred into a separating funnel and allows standing for 24 h. This was done for all the catalyst. Impact of reaction time on biodiesel yield was performed from 2 to 7 h for heterogeneous catalyst while other parameters were maintained constant. The data obtained was used for kinetic study.

2.5.2 Preparation of biodiesel blends

Biodiesel blends such as B20 and B50 were prepared by thoroughly mixing petroleum diesel and pure biodiesel in a ratio of 80:20 and 50:50 in a beaker, respectively.

2.5.3 Determination of fuel properties of the biodiesel and its blends

The American Society of Testing and Materials [9] method was utilized to assess the fuel properties of biodiesel produced at Kaduna Refining and Petrochemical Company (KRPC), including kinematic viscosity, specific gravity, cloud point, flash point, fire point, and sulfur content, pour point using Encinar etal., [10]methodology (See Table 1).

Table 1 Experimental Range and Level Coded of Independent Variables

Factors	Range and levels		
	-1	0	+1

Time(min)	60	120	150
Catalyst	0.3	0.4	0.5
Methanol:oil	3:1	6:1	9:1

3 RESULTS AND DISCUSSION

3.1 Result and Discussion of the Physiochemical Parameters of Chicken Fat Oils (CFO)

Table 2 Physiochemical parameters of chicken fat oils (CFO)

S/N	Properties	Units	CFO	Standard
1	Moisture content	% wt	0.56	-
2	Acid value	mgKOH/g	0.897	<20.00
3	FFA	%	0.449	-
4	Saponification value	mgKOH/g	140.25	140-205
5	Ester value	mgKOH/g	139.35	-
6	Molar weight	g/mol	1,207	-
8	Specific gravity	-	0.877	0.87-0.90
9	Viscosity	MpaS	45.18	-
10	Conductivity	µsimen	0.0	-
11	Boiling point	^{0}(C)	145	-

Specific gravity is a crucial property of fuel, influencing cetane number, heating values, and fuel storage and transportation. Chicken fat oil has a specific gravity within the standard range of 0.87-0.9, slightly higher than petrodiesel. The petro-diesel used in this study had a specific gravity lower than chicken fat oil's 0.877. The acid value, which measures the free fatty acid (FFA) content in the biodiesel, was 0.897, indicating good quality. The saponification values of the oil were between 175-205mg KOH/g oil, with a high value of 140.25mg KOH/g oil, indicating that chicken fat oil is typically triglycerides and can be used in soap and shampoo production. Overall, the chicken fat oil biodiesel produced in this study demonstrates good quality and potential for soap and shampoo production (See Figure 1-2 and table 2).

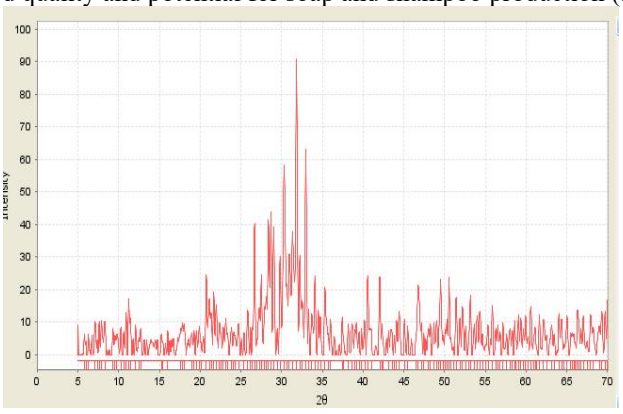

Figure 1 XRD Pattern of Zn

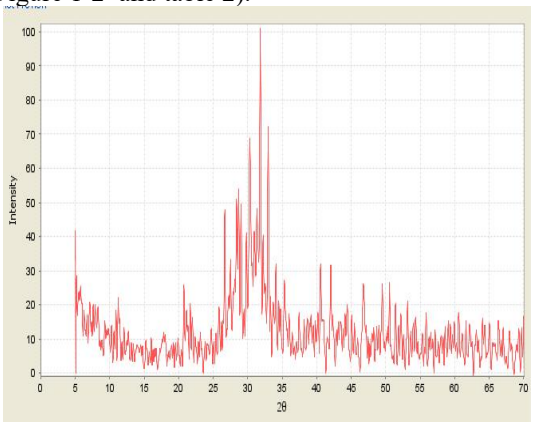

Figure 2 XRD Pattern of Zn/MgO

From the XRD results, the XRD diffractogram of Zn shows intensity of the peaks at 2 θ angle of 42.90, 53.760, 62.20, 73.70, 78.60, 83.70, 86.10 (JCPDS file 01-072-0447). Zinc shows additional small intensity peaks at 2 θ angles of 23.080, 26.70, 28.760, 40.70, 82.280, 90.10, 91.30, 109.760 which is as a result of the distribution of the Zn on the catalyst surface and is in good agreement with the JCPDS file No. 77 – 2176.

3.2 Characterization of Biodiesel

Table 3 Fuel Properties of Biodiesel Produced and its Blends

Fuel properties	Units	Chicken fat oil	B100	B50	ASTM(1998)
Kinematic viscosity @40^{0}C	CSt	0.51	1.5	1.58	1.9-6.0
Specific gravity	(kg/l)	0.8528	0.8432	0.810	0.820
Flash point	^{0}C	156	106	80	100-170
Fire point	^{0}C	110	94	89	-
Cloud point	^{0}C	5	2	-2	-6 to 12
Sulphur content	%	0.03	0.02	0.02	0.5
Cetane number	^{0}C	42	50.70	49.50	48-65
Diesel index	-	45	48	54	

B100= 100% pure biodiesel
B50= 50% biodiesel

The fuel properties of oil, biodiesel (B100) and its blends and B50 with that of petro-diesel were determined and the result was shown in Table 3. From table, it can be seen that the properties of biodiesel and its blends appeared to be much closer to those of petro-diesel. However, the properties of chicken fat oil were found to be much higher values than those of petro-diesel. For instance, kinematic viscosity which is 11.91 at 40oC, thus making it restriction on direct use as a fuel for diesel engines. After transesterification process, the viscosity of B100 was found to be less than that of chicken fat oil and a bit nearer to that of petro-diesel. Further decrease in viscosity was observed from 3.66 to 2.91 as the amount of petro-diesel was increased in the blend. Similarly, specific gravity of chicken fat oil is higher than that of petro-diesel, B100 and B50 . The flash point of B100 and chicken fat oil was found to be higher than that of the blends and petro-diesel which make them safe for handling. The cloud point of the oil and B100 was also higher than petro-diesel indicating the presence of waxes, which start to freeze at temperature less than 273 K [11] but the case of higher cloud point of B100 can be overcome by blending with petro-diesel. The sulfur content gives clear evidence that biodiesel reduces emission of greenhouse gases (GHG) as shows in the table 4. The sulfur content of B100 and chicken fat oil has much lower values than petro-diesel. The cetane number for B100 and B50 were determine to be 51.7, 49.5 and 48.6 which have found to complied with ASTM standard.

Table 4 Physical Chemical Parameters of Biodiesel

S/n	Properties	TEST METHOD	SPEC (for PD)	Values
1.	Colour			Golden and dark brown
2.	Specific gravity	ASTM D 1298	0.820min	0.8528
3.	Total Sulphur (%Wt)	ASTM D 4294	0.5max	0.030
4.	Viscosity @40^{0}C (cSt)	ASTM D 445	1.6 – 5.5max	1.539
5.	Flash point (^{0}C)	ASTM D 93	150min	156
6.	Cetane number			42
7.	Diesel Index		45min	45
8.	Fire Point (^{0}C)			140

The study analyzed the fuel properties of oil, biodiesel (B100), and its blends B20 and B50 compared to petro-diesel. The results showed that biodiesel and its blends were closer to petro-diesel, but chicken fat oil had higher values. The kinematic viscosity of chicken fat oil was 11.91 at 40 oC, making it restricted for direct use in diesel engines. After trans-esterification, B100 viscosity decreased to less than chicken fat oil and closer to petro-diesel. The specific gravity of chicken fat oil was higher than that of petro-diesel, B100, B50, and B20.
The flash point and cloud point of B100 and chicken fat oil are higher than blends and petro-diesel, making them safe for handling. However, the higher cloud point can be overcome by blending with petro-diesel. The sulfur content of B100 and chicken fat oil is lower than petro-diesel, indicating that biodiesel reduces greenhouse gas emissions. The cetane numbers for B100, B50, and B20 comply with ASTM standards, with values of 51.7, 49.5, and 48.6 respectively (See Table 5).

Table 5 Composition of Fatty Acid Methyl Ester (FAME) of the Synthesized Biodiesel

Peak No.	Retention time (min)	Peak (%)	Area	Identified compounds

1	13.908	0.94	Tridecanoic acid methyl ester
2	13.975	0.98	Dodecanoic acid methyl ester
3	16.317	1.21	Myristic acid methyl ester
4	18.550	14.22	15-methyl hexadecanoic methyl ester
5	20.442	62.22	9,12-octadecadienoic acid methyl ester
6	20.567	6.37	Pentadecanoic acid methyl ester
7	20.792	3.97	Linolelaidic acid methyl ester
8	21.783	1.18	Linolenic acid methyl ester
9	21.958	2.41	10-octadecanoic acid methyl ester
10	22.167	2.39	Heptacosanoic acid methyl ester
11	22.983	0.26	Eicosanoic acid methyl ester
12	23.792	2.30	Docosanoic acid methyl ester
13	24.592	0.39	Tricosanoic acid methyl ester
14	25.525	1.17	Tetracosanoic acid methyl ester

The GC-MS analysis of the prepared biodiesel revealed various fatty acid methyl esters (FAME) compositions, which were identified by comparing the profiles from the NIST107.LIB GC library. Istadi et al. [1]reported different FAME compositions in soybean oil-based biodiesel, including Hexadecanoic acid methyl ester, Hexadecadienoic acid methyl ester, octadecanoic acid methyl ester, 9, 12-octadecadienic acid methyl ester. Galadima & Oki also found various FAME content in biodiesel production from algae[12], which aligns with the results of this study.

3.3 Fourier Transform Infra-red Spectroscopy of the Biodiesel

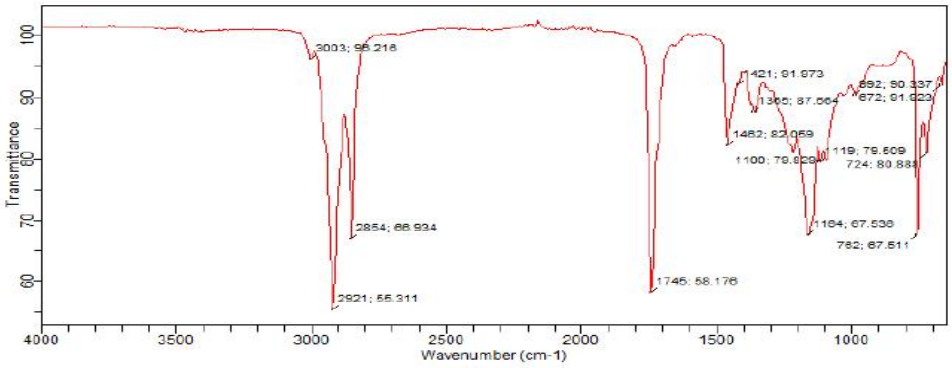

Figure 3 FT-IR Spectra for Biodiesel

3.4 Optimization of Reaction Parameters

The study aimed to optimize the reaction conditions for biodiesel production from chicken fat oil using a synthesized solid base catalyst composite (Zn/MgO). The initial experiment involved a 6:1 methanol to oil molar ratio, 3% w/w catalyst loading, and a 4 h reaction time (See Figure 3 and Table 6).

Table 6 Response Surface Result Yields

No of runs	Yield (g)	%Yield	Actual value	Predicted value
1	15.4	77	91.00	90.03
2	18.2	91	75.00	80.13
3	17.4	87	80.00	83.73

4	15.4	77	92.00	89.83
5	16.0	80	92.00	94.23
6	15.0	75	87.00	83.33
7	15.4	77	88.00	82.93
8	15.4	77	87.00	88.03
9	15.0	75	89.00	89.09
10	15.4	77	87.00	86.69
11	15.2	76	76.00	73.29
12	17.8	89	70.00	72.49
13	15.4	77	85.00	79.29
14	17.0	85	75.00	80.49
15	14.0	70	77.00	77.07
16	17.4	87	77.00	77.07
17	17.6	88	77.00	77.07
18	17.4	87	77.00	77.07
19	18.4	92	77.00	77.07
20	18.4	92	77.00	77.07

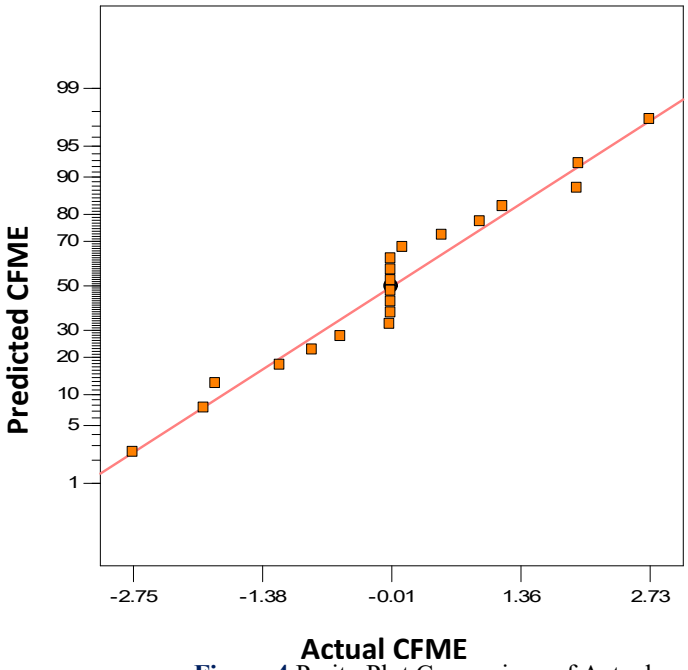

Figure 4 Parity Plot Comparison of Actual and Predicted Response

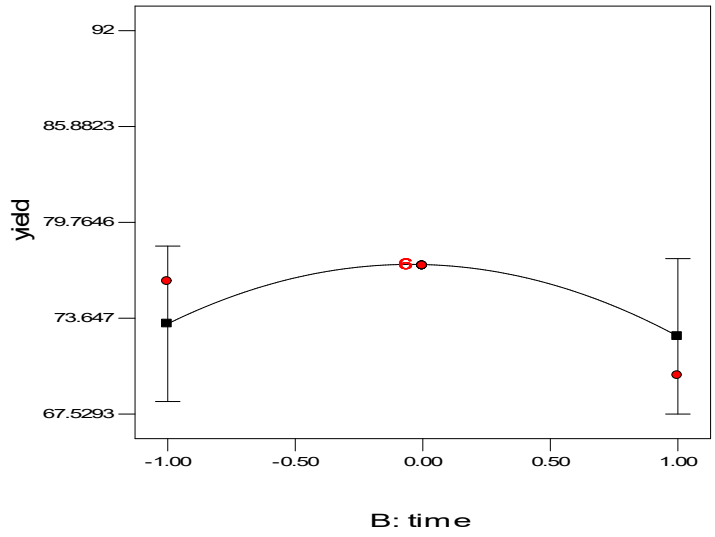

Figure 5 One Factor plot of Yield against Time

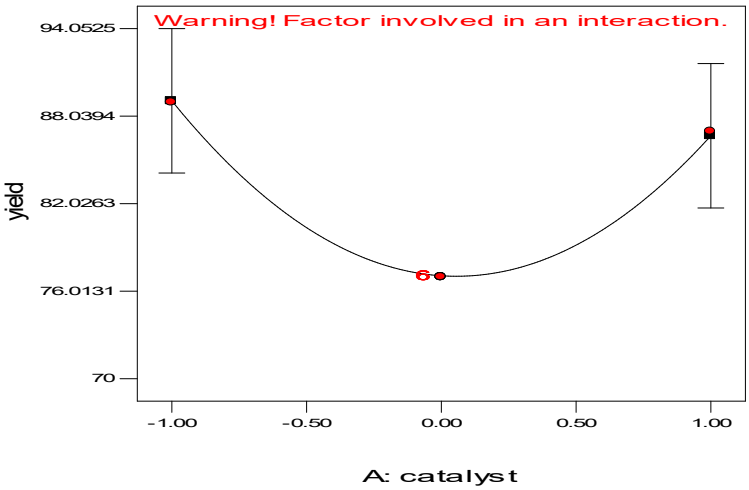

Figure 6 One Factor Plot of yield against Catalyst

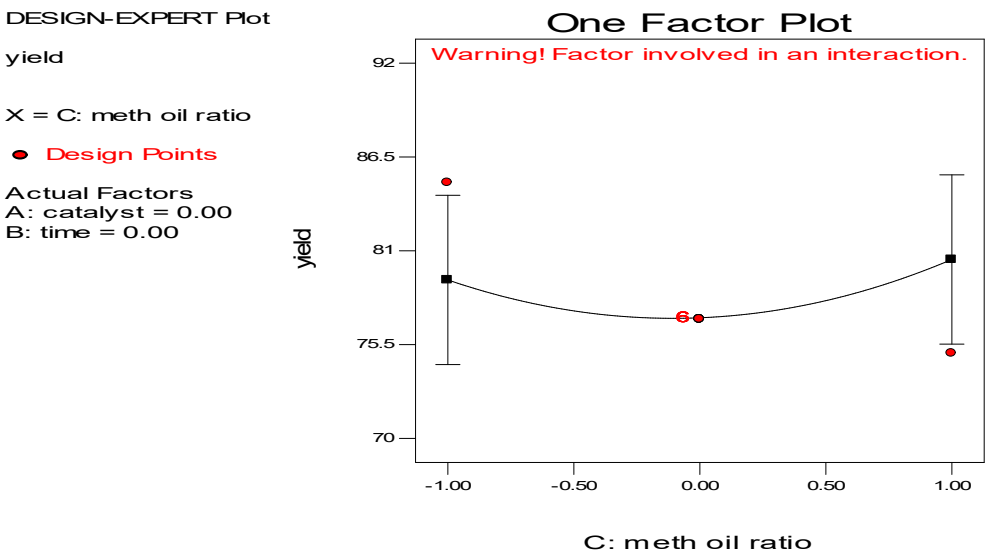

Figure 7 One Factor Plot yield against methanol oil ratio

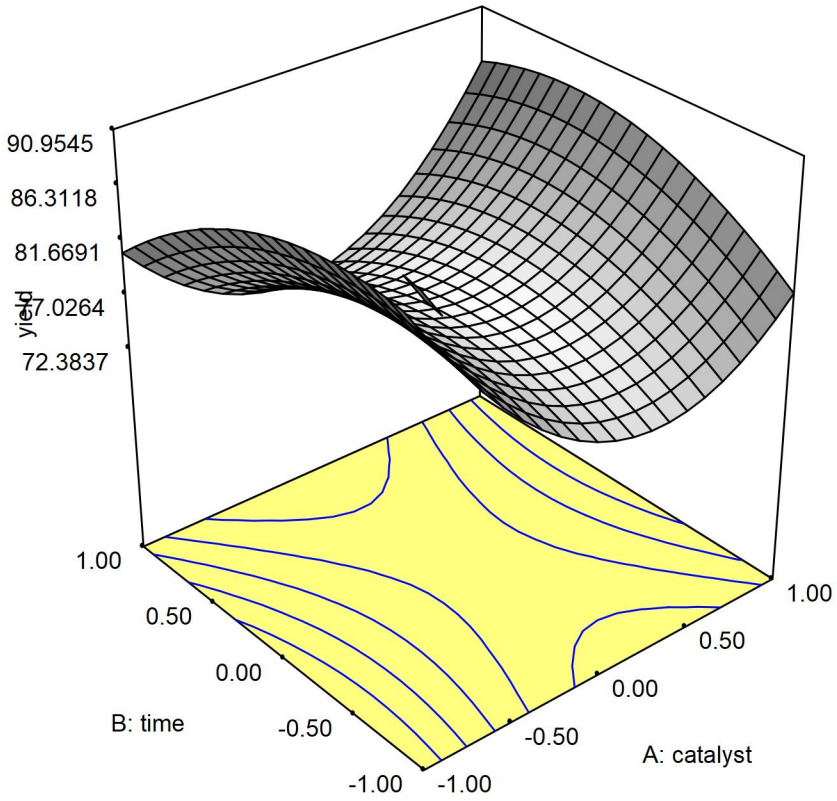

Figure 8 3-D Design Expert Plot of yield against Time & Catalyst

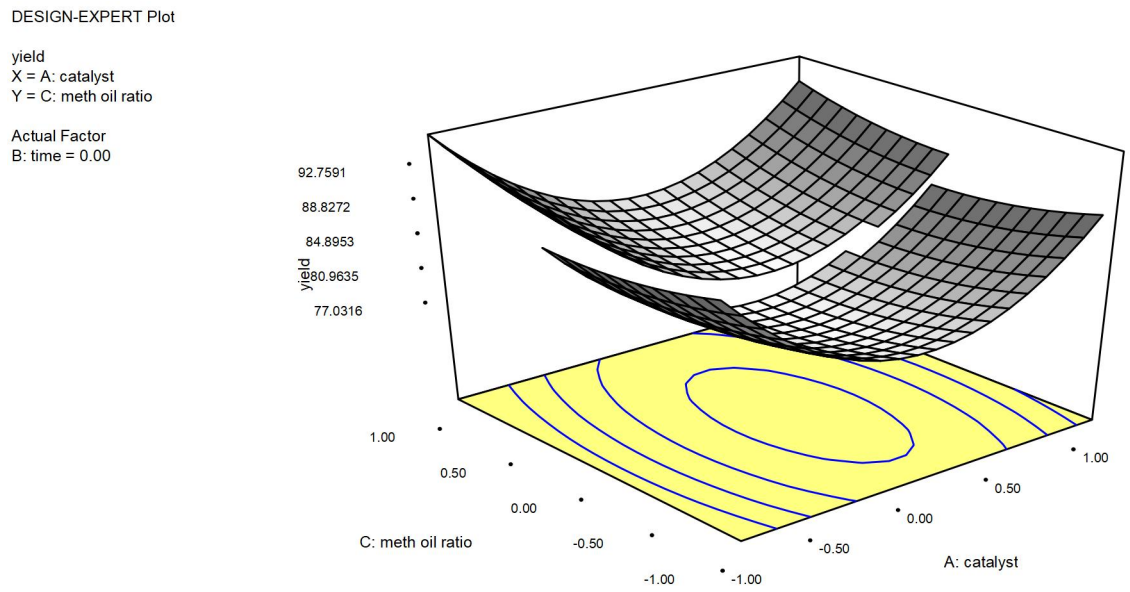

Figure 9 Plot of Yield Against Methanol- Oil Ratio & Catalyst

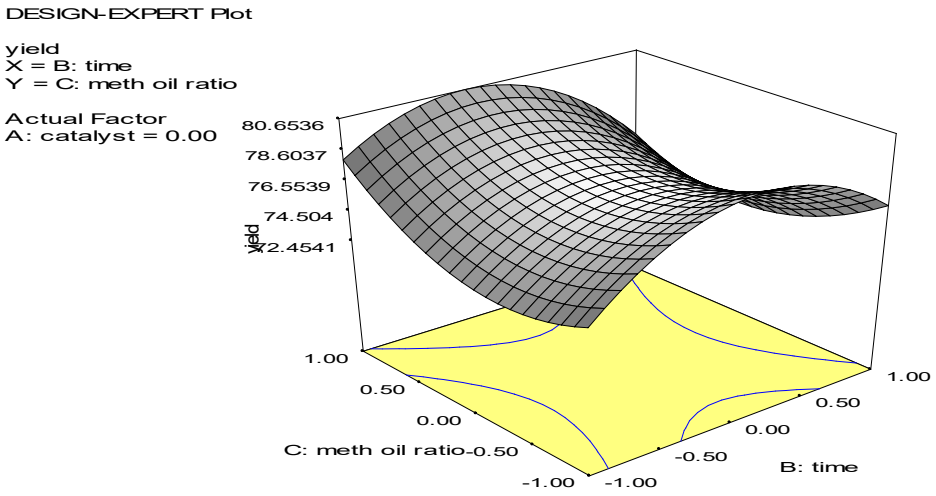

Figure 10 Response Surface Plot of Yield Against Methanol's Oil Ratio, Catalyst Loading & Time

3.4.1 The effect of catalyst concentration on biodiesel yield

The study examined the impact of catalyst loading on biodiesel yield, varying from 1%w⁄w to 6%w⁄w. Results showed that biodiesel yield increased with MgO loading from 1%w⁄w to 4%w⁄w, reaching a maximum yield of 72.85%. However, with further MgO loading, yield dropped from 72.51% to 71.74% due to soap formation, increasing reactant viscosity. This suggests the limitation of mass transfer resistance in heterogeneous systems. Therefore, a 4%w/w catalyst loading was chosen for optimization testing of other parameters (See Figure 4-10).

3.4.2 The effect of reaction time

The study examined the time it took biodiesel to convert, ranging from 30 to 90 minutes. Results showed an increase in reaction time, with the conversion reaching 92% at 90 minutes. Further time increase may increase conversion. A suitable reaction period for synthesizing methyl ester was found to be 90 minutes at 65°C, with a 9:1 methanol alcohol to fat ratio and 1.2%w/w catalyst quantity, which aligns with Strong et al.[13] findings.

3.4.3 The effect of methanol to oil molar ratio on biodiesel

Transesterification is a stoichiometric reaction involving methenol and oil to produce biodiesel and glycerol. However, methanolysis of triglyceride requires excess methanol to facilitate biodiesel formation and increase reaction rate. A study was conducted to examine the impact of methanol to oil molar ratio on biodiesel yield. The results showed a continuous increase in biodiesel yield with increased methanol from 3:1 to 12:1, leading to high yield. Excess methanol promotes methoxy species on the catalyst surface, causing an equilibrium shift in the direction of biodiesel. However, the yield remained unchanged above a 12:1 stoichiometric ratio due to the dissolution of glycerol in the methanol, hindering the interaction between reactants and catalyst, and affecting the separation of biodiesel and glycerol. The optimal stoichiometric ratio was found to be 12:1.

3.4.4 Response surface methodology using statistical

The study used ANOVA and full quadratic models to analyze responses. The predicted methyl esther yield was compared to the observed yield, and the parity plot showed that the model accounted for 90.00% of the variability in the data, with a coefficient of determination (R2) value of 0.9000.

3.4.5 Interactive effect of variables on CFME yield

The empirical model shows the interaction between methanol-oil ratio and catalyst loading on methyl yield at 90 minutes and constant 600C. Overloading methanol would inactivate the catalyst and reverse the transesterification and esterification reactions. The elliptical contour plots indicate significant interaction between methanol-oil ratio and catalyst loading. The non-circular contour plots at 3wt% Zn/MgO loading and constant 600C reveal significant interaction effects between methanol-oil ratio and reaction time. The CFME yield increases to the maximum and then decreases after overloading methanol at longer reaction time. The model provides insight into the reactions in the experimental range.

3.4.6 Optimization of methylester yield

The study optimized the simultaneous esterification and transesterification of CFO for the highest methylester yield. The response surface analysis showed a predicted methylester yield of 92.0% at a 12:1 methanol to oil molar ratio, 4.0 wt% catalyst loading, and 90 minutes reaction time at 600C. Additional experiments validated the optimization results, with observed and predicted yields of 89.83% and 92.0%, respectively, with a 2.4% error. Confidence intervals were determined using the standard deviation from the ANOVA table.

4 SUMMARY

This research focuses on synthesizing a Zn/MgO catalyst for biodiesel production using chicken fat oil. The oil's fuel properties were found to be higher than diesel engine oil, indicating its limitations. To ensure successful production, the oil was pretreated with an organic base to reduce acidity, free fatty acid, saponification, and pH. The biodiesel and its blends met ASTM standards and allowed for 2:8 blending with petro-diesel. The catalyst's optimized reaction parameters for transesterification were found to be 4%w/w catalyst loading, 65oC reaction temperature, 12:1 methanol to oil molar ratio, 7 h reaction time, and 600rpm agitation velocity. The homogeneous base catalyst yielded 97.8% biodiesel, while the basic heterogeneous catalyst yielded 72.5%. However, the heterogeneous base solid catalyst also provided high biodiesel yield and recycling ability. The catalyst's potential for large-scale production is evident due to its active basic site density and non-spontaneous kinetics and thermodynamics.

5 CONCLUSION

This research explores the production of chicken fat oil methyl ester through transesterification using two catalysts. The study examines the impact of reaction parameters on biodiesel yield. The optimal reaction condition yielded 72.5% biodiesel, confirming chicken fat oil's feasibility as a feedstock for biodiesel production and potential blend with petroleum diesel.

6 RECOMMENDATIONS

This research work has extensively studied the existing literatures on biodiesel production from triglyceride using heterogeneous materials as catalyst. Some issues still need to be well addressed in the future as outlined below.
Techniques such as Brunner-Emmett-Teller (BET), Temperature programmed desorption of CO2 (TPD-CO2), Transmission electron microscope (TEM), Thermo-gravimetric analysis and differential thermal analysis (TGA-DTA), X-ray Photoelectron Spectroscopy (XPS), Diffuse Reflectance UV-Visible Spectroscopy (DRUV-Vis) should be carried out to further elucidate structure and properties of the catalyst.

COMPETING INTERESTS

The authors have no relevant financial or non-financial interests to disclose.

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