



Optimized Biodiesel Production from Waste Cooking Oil Using Nickel (II) Nitrate Doped Chicken Manure as Catalyst

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ABSTRACT

In this study, chicken manure was doped with nickel (ii) nitrate to produce catalyst for the synthesis of biodiesel from waste cooking oil. Characterization of the catalyst was achieved using Brunauer-Emmett and Teller (BET) analysis, fourier transform infrared spectroscopy (FTIR), scanning electron microscope and energy dispersive X-ray (SEM-EDX), and X-ray fluorescence (XRF). Box-Behnken design (BBD) was used in designing the experiments. The influence of various parameters including reaction temperature, time, methanol/oil ratio and catalyst loading were investigated. The catalysts characterization revealed the presence of high surface area ($314.443\text{m}^2/\text{g}$) and pore size (2.123nm) and also metallic oxides such as silicon, aluminum and magnesium. Optimum biodiesel yield of 99.4026% was achieved at a temperature of 69.81°C , reaction time of 60 minutes with a methanol/oil ratio of 6:1 and catalyst loading of 1wt.%. This study confirms that chicken manure doped with nickel (ii) nitrate could be used as a heterogeneous solid catalyst for the biodiesel synthesis from waste cooking oil.

Keywords: Biodiesel, Box-Behnken design, Chicken manure, Modelling, Waste cooking oil

INTRODUCTION

The energy demand is increasing steadily due to hastened modernization and large urban growth. Conventional energy sources are depleting daily due to their non-renewability nature [1]. As a result of the enormous dependence on crude oil as a key source of fuel for energy generation globally, prices of fossil fuels have risen dramatically in recent years. Furthermore, the use of these traditional energy supplies contributes to global warming, which must be addressed by using alternate energy sources. Environmental degradation is also a problem that can be alleviated by using alternative energy sources such as biodiesel [2].

Transesterification of triglycerides with alcohol using a suitable catalyst produces biodiesel [3]. Most existing biodiesel production methods employ vegetable oils as a feedstock, and it is generally catalyzed by homogeneous catalysts [4]. Using pure vegetable oil as fuel, conversion or adjustment of engine may be required. Vegetable oil can be blended also with fossil fuel, although incomplete combustion, which leads to carbon build-up in the cylinders, could be a serious issue. The chemical conversion of vegetable oils into biodiesel is preferable because of these factors [5].

Biodiesel has been widely considered as replacement for fossil fuels. Although, dependence of consumable oil as feedstock over a long period of time would have a negative influence on its supply for feeding and also would amplify some issues relating to the environment such as the major soil resource loss, deforestation, and the use of much of the accessible arable land. In addition, the price of sources of consumable oils has increased significantly in recent times, putting the biodiesel industry's economic viability in doubt [6].

Biodiesel can only become established and more accepted around the world if it is produced at a lower cost than conventional fuel [7]. And for this to be achieved, a cheap and functional catalyst must be used. Transesterification of oil employing homogeneous catalysts, distributed in alcohol is the conventional way of creating biodiesel [8]. Using such catalyst has a number of limitations, including the generation of soap, and difficulties recovering the catalyst, which demands a treatment process of wastewater and so elevates biodiesel production costs [9]. As a result, a new catalyst must be used. A catalyst that is cheap, eco-friendly, and also streamlines recovery process is required. Heterogeneous catalysts are being used as this new preferable option for low-cost, long-term commercial biodiesel production. Using solid heterogeneous catalysts in manufacturing biodiesel minimizes the limitations posed by using homogenous catalyst [10].

Furthermore, cost of consumable oil, which makes up a substantial portion of the overall biodiesel manufacturing cost, is high; hence a low-grade feedstock like waste cooking oil (WCO) would be preferred. Also, with increased human population, demand for chicken for meat and egg production has increased and thus, more poultries have been established to meet this up rise in demand. This in turn, has created an increase in amount of chicken manure. Using chicken manure as catalyst would help the environment and reduce production cost of biodiesel [11].

Production of biodiesel is dependent on multiple process factors such as feedstock of catalyst, catalyst loading, reaction time, temperature, methanol/oil ratio, agitation speed etc. RSM can be used in optimizing process variables affecting biodiesel yield and thereby creating a more cost-effective process. RSM is a tool that can be used for statistical modelling and designing of experiments with multivariable. It enables lowered number of experiments when compared with the conventional one-factor-at-a-time method for the same number of process factors. Empirical models for describing processes can also be developed using RSM [12].

MATERIALS AND METHODS

Materials

WCO used in this study was collected from a local restaurant within Benin City, Nigeria. The collected WCO was first filtered with muslin cloth to remove traces of food particles and then heated at 100°C to lower the content of moisture. The properties of the WCO used are shown in Table 1. Chicken manure used for catalyst production was obtained from the University of Benin model farm, Benin City, Nigeria. Nickel nitrate and other analytical grade reagents used were purchased from a vendor within Benin City, Nigeria.

Table 1: Physiochemical properties of WCO

Property (Unit)	Value
FFA (%)	4.63
Density (g/cm ³)	0.91
pH	5.17
Iodine value (g I ₂ /100g)	108.6
Saponification value (mg KOH/g)	215.99
Peroxide value (meq/kg)	17.3
Viscosity (m Pas @40°C)	39

Catalyst Preparation

Chicken manure collected was sun-dried for several days to remove moisture and then grinded. The powdered dry chicken manure was then heated to 300°C in a carbonization process for 4 hours to remove any remaining odor. The sample was then soaked with 0.1M KOH for 24 hours, dried in the sun for 1 hour and in an oven at 100°C for 1 hour, and calcined for 6 hours in a muffle furnace at 900°C. The wet impregnation approach utilized by Ali et al., (2018) was then used to create the doped chicken manure. To create a slurry solution, calcined chicken manure was added after nickel nitrate had been dissolved in distilled water. After that, it was swirled for around an hour to ensure thorough mixing. The resulting slurry solution was then put on a heated magnetic stirrer running at 80°C and swirled until all of the water had been completely evaporated from it. The resulting solid was dried for several hours in the sun and then for 1 hour at 100°C in an oven. The resulting doped chicken manure was then subjected to a 6-hour calcination process at 900°C. The resulting catalyst was kept in a desiccator, to allow cooling and then kept in an airtight container for further use.

Catalyst Characterization

The physicochemical properties of the catalyst were assessed using various characterization tests. The BET analysis determined the surface area and pore properties. FTIR spectroscopy analysis showed the bond structure and interaction. SEM-EDX analysis showed the surface structure and elemental composition. And XRF analysis determined the composition of oxides.

Biodiesel Production and Characterization

Transesterification was carried out following the procedure by Reda, 2014; 50g of WCO was poured into a 250-ml conical flask and placed on a hot plate magnetic stirrer, and heated to a specific temperature, a specific weight of the catalyst was dissolved in a specific volume of methanol and then transferred into the WCO in the conical flask. The specific values were in consonance with the experimental data generated using BBD. In conclusion of the reaction, the mixture in the conical flask was poured into a separating funnel and left overnight to allow separation of the glycerol and biodiesel. The biodiesel was washed thrice using hot distilled water to ensure removal of any impurities. The washed biodiesel was then dried in an oven. The yield of biodiesel produced was calculated using Equation (1).

$$\text{Biodiesel yield} = \frac{\text{mass of biodiesel produced}}{\text{mass of WCO used}} \times 100 \quad (1)$$

Experimental Design and RSM Modelling

RSM using BBD was used to design the experiment using Design Expert 13. The effect of four process variables (methanol/oil ratio, catalyst loading, reaction temperature and reaction time) on the response (biodiesel yield) was explored. Table 2 shows the coded and actual levels of the BBD variables.

Table 2: Coded and actual levels of BBD variables

Process variables	Units	Factor	Coded Low	Mean	Coded High
Methanol/oil ratio	Mol/mol	A	6	12	18
Catalyst loading	Wt. %	B	1	3	5
Reaction temperature	°C	C	40	55	70
Reaction time	Min.	D	60	90	120

The relationship of the process (independent) variables and the response is given by a second-order polynomial as shown in Equation (2).

$$Y_i = b_0 + \sum b_i X_i + \sum b_{ij} X_i X_j + \sum b_{ii} X_i^2 + e_i \quad (2)$$

Where,

Y_i denotes the predicted response,

b_0 is the value at intercept,

b_i is the coefficient of first order,

b_{ii} is the quadratic effect coefficient,

b_{ij} is the interacting effect coefficient,

X_i and X_j are the process variables that affect the response, and

e_i is the experimental random error.

This resulted in generation of 29 experimental runs which were performed in the Laboratory randomly, to avoid any systematic error in the outcomes.

RESULTS AND DISCUSSIONS

Catalyst Characterization

BET Analysis:

An important physical analysis of the doped chicken manure catalyst prepared was conducted by the application of BET surface analysis technique. The catalyst had surface area of 314.443 m²/g, pore diameter of 2.123 nm and pore volume of 0.174 cc/g. High surface area indicate the presence of adequate active sites for attachment and better reaction. Additionally, good pore characteristics allow for faster diffusion of reactants and products from the pores to the reaction sites and to the catalyst surface [15]. The nitrogen physisorption isotherm and pore distribution are shown in Figures 1 and 2 respectively.

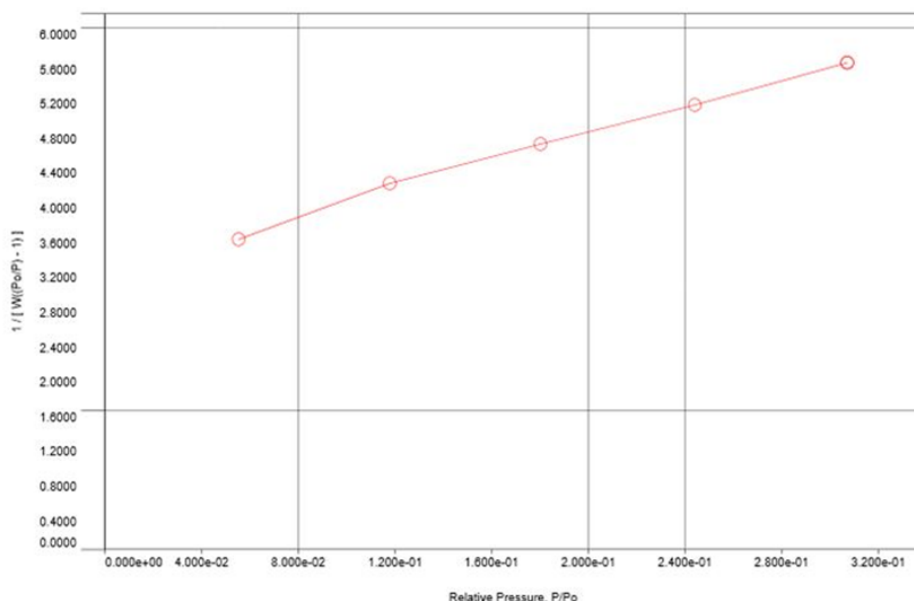


Figure 1: Nitrogen physisorption isotherm for catalyst

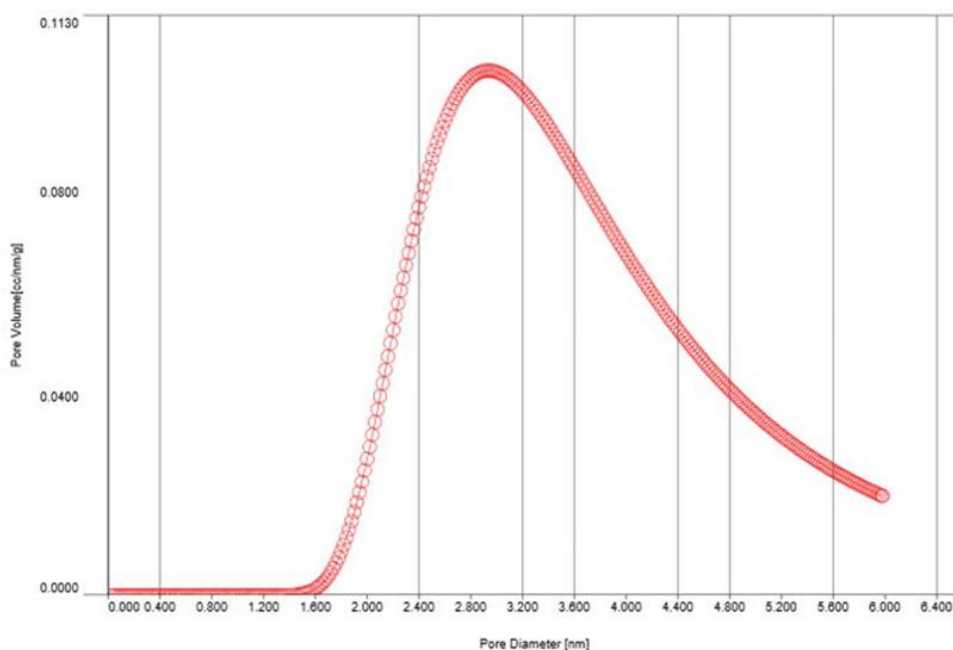


Figure 2: Pore size distribution for catalyst

FTIR Analysis:

The FTIR spectrum of the catalyst is shown in Figure 3. The catalyst spectrum shows distinguishing peaks at 1028.7cm^{-1} , 913.2cm^{-1} , and 797.7cm^{-1} . The absorption peak of the catalyst at wavenumber of 1028.7cm^{-1} can be attributed to the hydroxyl group stretching found. The presence of this peak can be as a result of moisture presence on the catalyst surface [16]. The O-H out of plane vibration overlapped with the molecule's fingerprint absorption, the presence of an aromatic phosphate group resulting from the P=O stretch, and the carbonyl groups from transitional metals can be responsible for the observed peaks. Peaks within the

range seen in the catalyst could be traced to the presence of metal oxides such as silicon, aluminum and magnesium [17]. These FTIR results correspond with the XRD results obtained.

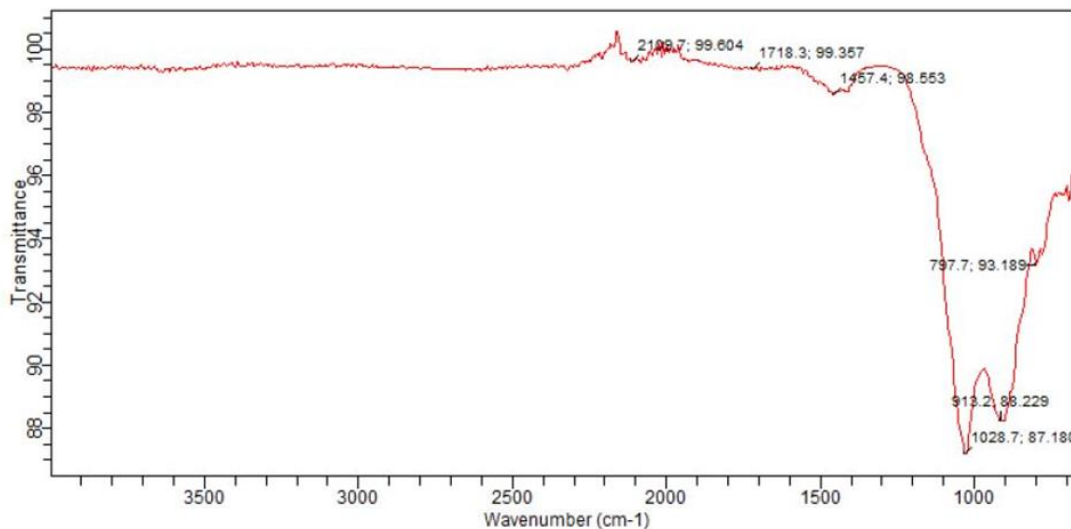
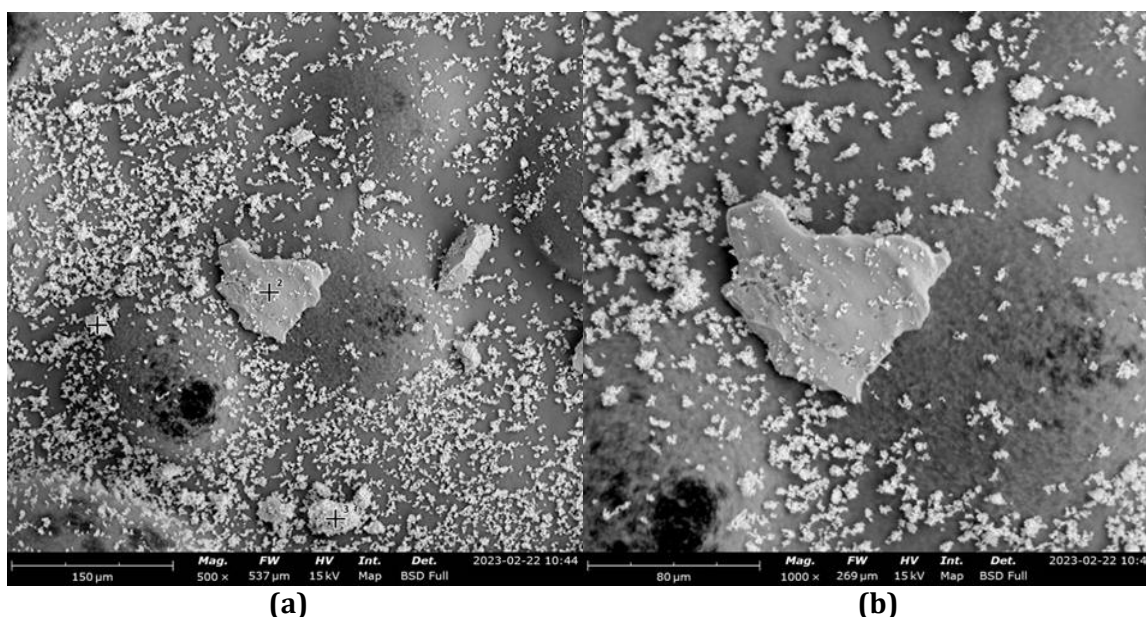


Figure 3: FTIR spectrum of catalyst

SEM-EDX Analysis:

Morphologies of catalyst in different magnifications are shown in Figure 4. SEM determined the surface morphology the catalyst. The image of the catalyst from SEM analysis shows particle agglomerations, although a lot smaller particles are seen around these agglomerations. Similar observations was seen in researches by Akhabue et al., (2020) and Maneerung et al., (2016). The EDX pattern showed on Figure 5 indicates that the catalyst had within it metals like potassium and americium. The actual weight concentration of these metals and the other elements present are shown in Table 3. The metals present in the catalyst are very reactive and this can explain the catalytic behavior of the catalysts.



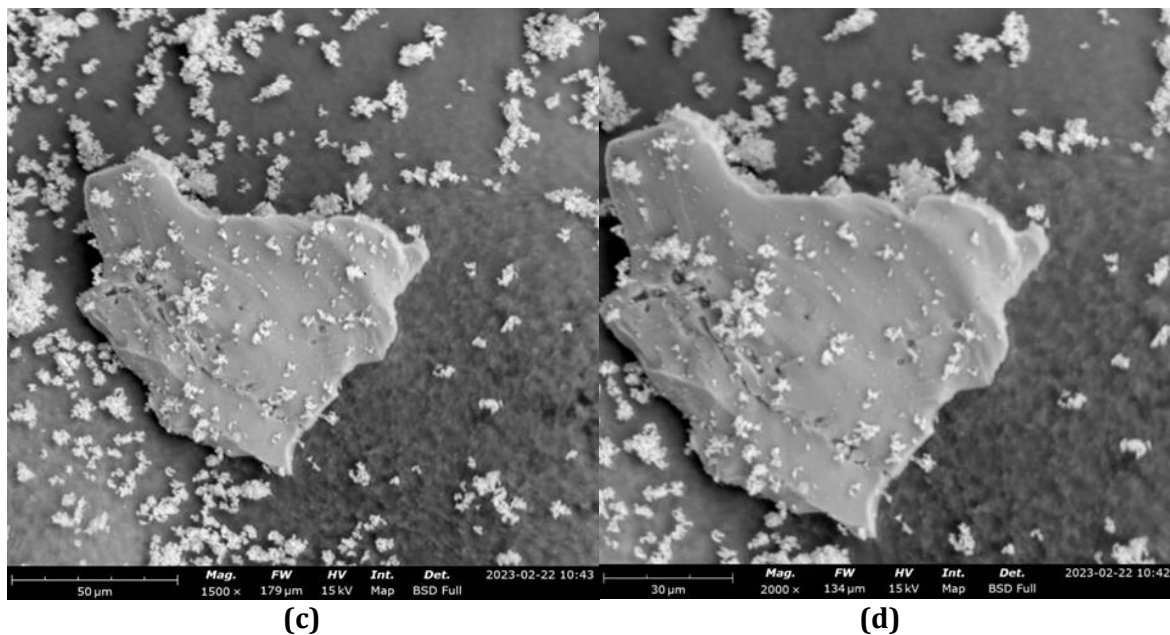


Figure 4: SEM images of catalyst (a) x500 (b) x1000 (c) x1500 (d) x2000

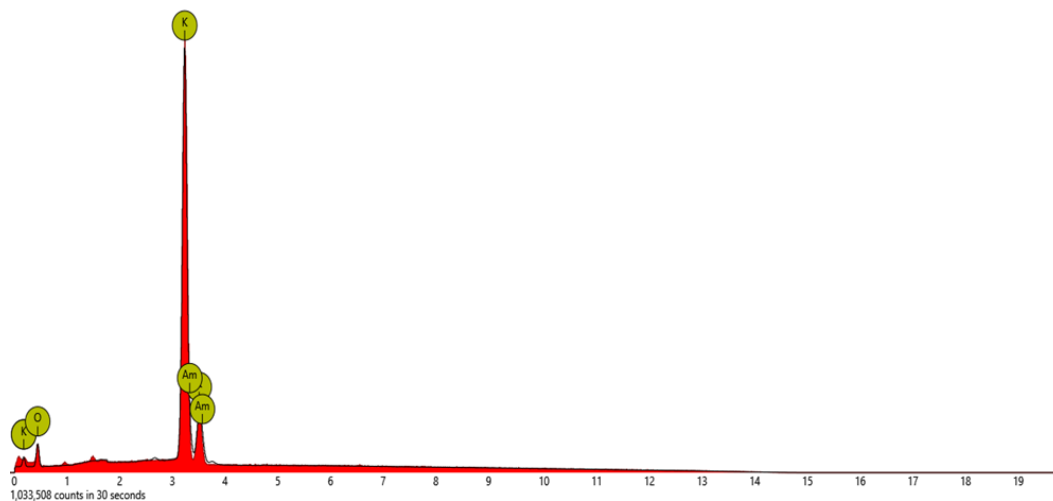


Figure 5: EDX pattern of catalyst

Table 3: Elemental composition of catalyst obtained from EDX

Element number	Element name	Element symbol	Weight Concentration (%)
8	Oxygen	O	19.53
7	Nitrogen	N	7.11
19	Potassium	K	62.41
95	Americium	Am	10.95

XRF Analysis:

The elemental composition of the catalyst determined by XRF is shown in Table 4. The data obtained shows that silicon, aluminum and magnesium as the major metal oxides component of the catalyst. These metal oxides present enhance transesterification due to its catalytic capabilities [20].

Table 4: XRF data for catalyst

Component	Concentration (%)
Silicon oxide (SiO ₂)	17.231
Vanadium pentoxide (V ₂ O ₅)	0.124
Chromium oxide (Cr ₂ O ₃)	0.097
Manganese oxide (MnO)	0.082
Iron (iii) oxide (Fe ₂ O ₃)	1.07
Cobalt tetroxide (Co ₃ O ₄)	0.045
Nickel oxide (NiO)	0.008
Copper oxide (CuO)	0.139
Niobium pentoxide (Nb ₂ O ₃)	0.083
Molybdenum trioxide (MoO ₃)	0.066
Tungsten oxide (WO ₃)	0.099
Calcium oxide (CaO)	1.264
Magnesium oxide (MgO)	22.163
Barium oxide (BaO)	0.122
Aluminum oxide (Al ₂ O ₃)	26.499
Tantalum pentoxide (Ta ₂ O ₅)	0.108
Titanium oxide (TiO ₂)	0.527
Zinc oxide (ZnO)	0.041
Chlorine (Cl)	30.134
Zirconium oxide (ZrO ₂)	0.097

Optimization by RSM

Four variables were optimized: methanol/oil ratio, catalyst loading, reaction temperature and reaction time. Table 5 shows the experimental design of the transesterification reaction of WCO to produce biodiesel with the experimental and predicted responses. Equation (3) represents the quadratic model (coded-basis).

$$\text{Yield}_{\text{CM}} = 37.323 - 3.128A - 14.832B + 2.938C + 0.027D + 0.27AB + 0.022AC + 0.002AD + 0.045BC + 0.007BD - 0.006CD + 0.004A^2 + 1.053B^2 - 0.027C^2 + 0.002D^2 \quad (3)$$

When an independent variable in a regression equation has a positive sign, it implies that increasing the variable will result in an increase in the response, whereas a negative sign would cause a decrease in the response[21]. From Equation (9), reaction temperature (C), reaction time (D), interaction factors (AB, AC, AD, BC and BD) and quadratic factor (A², B² and D²) have positive effects on the percentage yield of biodiesel while methanol/oil ratio (A), catalyst loading (B), interaction factor (CD) and quadratic factor (C²) had negative effects on the percentage biodiesel yield from the process. Analysis of variance (ANOVA) used to find the importance of the model is given in Table 6.

P-value and F-value are demonstrated the model significance and its consistency, respectively. According to Table 6, F-value is 19.53 and the p-value is <0.0001 means the designed model is significant.

Table 5: Transesterification experimental design with experimental yield and RSM predicted yield

Run	Methanol/oil ratio (mol/mol)	Catalyst loading (wt%)	Reaction temperature (°C)	Reaction time (min.)	Experimental yield (%)	RSM Predicted yield (%)
1	18.00	1.00	55.00	90.00	81.75	77.25
2	6.00	3.00	55.00	60.00	78.41	81.65
3	12.00	3.00	55.00	90.00	75.5	75.32
4	12.00	3.00	40.00	120.00	70.39	72.91
5	12.00	1.00	70.00	90.00	80.75	81.91
6	6.00	3.00	70.00	90.00	67.75	69.02
7	18.00	3.00	55.00	60.00	80.07	84.22
8	12.00	3.00	70.00	120.00	63.73	62.49
9	12.00	3.00	55.00	90.00	75.5	75.32
10	12.00	3.00	55.00	90.00	76.25	75.32
11	12.00	3.00	55.00	90.00	74.12	75.32
12	18.00	5.00	55.00	90.00	84.92	79.12
13	6.00	3.00	55.00	120.00	63.75	63.25
14	12.00	5.00	70.00	90.00	59.75	62.98
15	12.00	5.00	55.00	120.00	58	59.44
16	6.00	5.00	55.00	90.00	59.75	54.8
17	6.00	3.00	40.00	90.00	75.65	75.88
18	18.00	3.00	70.00	90.00	74.65	75.87
19	12.00	5.00	40.00	90.00	70.42	70.94
20	12.00	1.00	55.00	120.00	73.75	75.96
21	12.00	1.00	40.00	90.00	87	85.44
22	18.00	3.00	55.00	120.00	71.75	72.15
23	6.00	1.00	55.00	90.00	93.75	90.1
24	18.00	3.00	40.00	90.00	80.32	80.5
25	12.00	1.00	55.00	60.00	89.3	91.38
26	12.00	3.00	55.00	90.00	72.25	75.32
27	12.00	3.00	40.00	60.00	84.57	83.47
28	12.00	3.00	70.00	60.00	87.25	82.4
29	12.00	5.00	55.00	60.00	73.18	74.49

Table 6: ANOVA data for the RSM model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2115.37	10	211.54	19.53	<0.0001	significant
A-Methanol/oil ratio	98.61	1	98.61	9.11	0.0074	
B-Catalyst loading	838.01	1	838.01	77.38	<0.0001	
C-Reaction temperature	99.02	1	99.02	9.14	0.0073	
D-Reaction time	696.32	1	696.32	64.30	<0.0001	
AB	345.40	1	345.40	31.89	<0.0001	
AC	1.24	1	1.24	0.1148	0.7387	
AD	10.05	1	10.05	0.9279	0.3482	
BC	4.88	1	4.88	0.4510	0.5104	

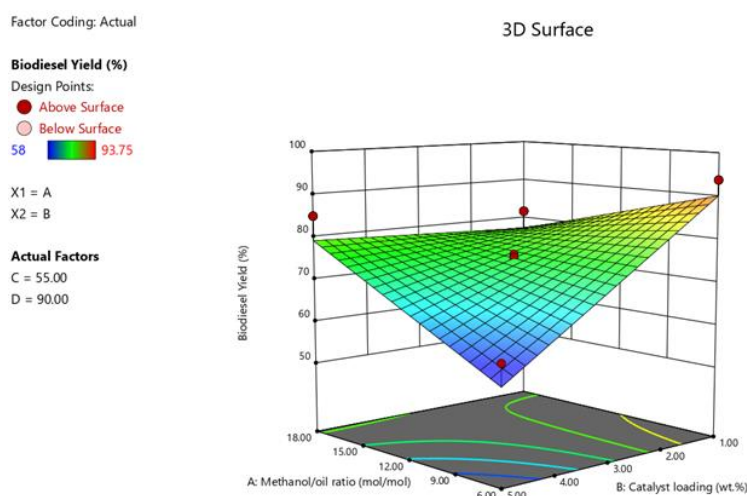
BD	0.0342	1	0.0342	0.0032	0.9558	
CD	21.81	1	21.81	2.01	0.1730	
Residual	194.93	18	10.83			
Lack of Fit	184.91	14	13.21	5.27	0.0601	not significant
Pure Error	10.02	4	2.50			
Cor Total	2310.30	28				

The quality of model fit is assessed by the coefficient of determination (R^2). The fit statistics data showed that the R^2 and adjusted R^2 values were 0.9156 and 0.8688 respectively. The difference of less than 0.2 indicates that the predicted R^2 and adjusted R^2 values corresponds. Therefore, this model could be used in the theoretical prediction of the biodiesel production process.

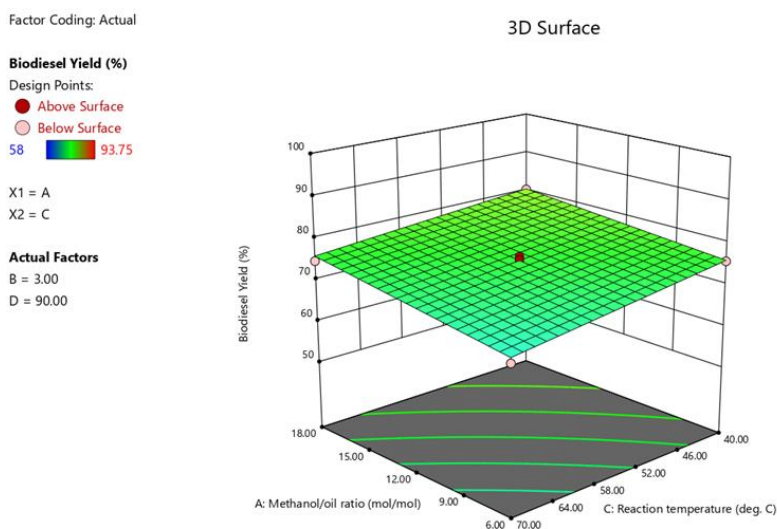
Effect of Input Variables on Biodiesel Yield:

The effects of process parameters on biodiesel production were analyzed by three- dimensional (3D) surface plots. Figure 6 shows the 3D surface plots representing two independent variables while keeping other variables constant. According to the Table 6, methanol/oil ratio, catalyst loading and reaction temperature are the most significant variables for biodiesel production using the catalyst, with methanol/oil ratio being the dominating factor, having the highest sums of squares. The 3D plots show the interactions between the variables and biodiesel conversion. The optimum yield of biodiesel of 99.4026% was achieved at a temperature of 69.81°C, reaction time of 60 minutes with a methanol/oil ratio of 6:1 and catalyst loading of 1wt. %.

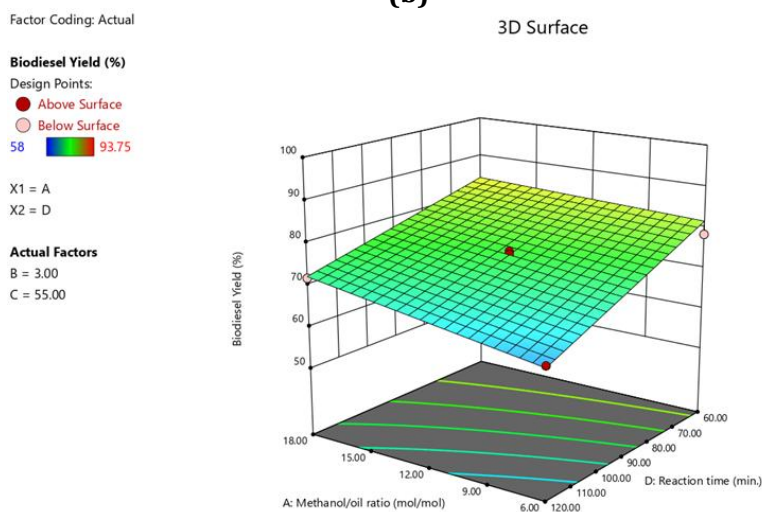
With increase in methanol /oil ratio, there was an increase in the biodiesel yield with maximum yield obtained at 6:1. For the catalyst loading, lowered catalyst loading resulted in an increased biodiesel yield with the maximum yield of 93.75 % at 1wt% as shown in Figure 6a. From Figure 6b, it can be seen that a lower temperature gave lesser biodiesel yield and maximum yield was obtained at 55°C. Increasing reaction time, gave an increase in the yield of biodiesel as seen in Figure 6c.



(a)



(b)



(c)

Figure 6: 3D plots showing the interaction between (a) Methanol/oil ratio and catalyst loading (b) Methanol/oil ratio and reaction temperature and (c) Methanol/oil ratio and reaction time

Biodiesel Characterization

Table 7 summarizes the various properties tested and their corresponding values obtained for the biodiesel produced using nickel (ii) nitrate doped chicken manure catalyst as compared to ASTM D6751 and EN14214 standards of biodiesel. Low FFA percentage contributes to improved oxidative stability and longer shelf life, while low acid value indicates minimal corrosiveness of the biodiesel which would cause no engine damage. High thermal stability indicates a fuel with a high flash point, which makes it safe for transportation. The fuel's calorific value indicates that it also contains a lot of energy. The presence of a high cetane number demonstrates that the fuel is of good quality and will burn cleanly. Low fuel viscosity results in good fuel flow, which makes fuel atomization during spraying simple.

Table 7: Physicochemical properties of biodiesel produced and standards of biodiesel

Properties (Unit)	Value	ASTM D6751	EN14214
Acid value (mg KOH/g)	0.18	0.8max	0.5max
Flash point (°C)	146.4	100 to 170	>120
Calorific value (MJ/kg)	37.42	-	>35
Cetane number	58.67	>47	>51
Viscosity (mm ² /s) at 40°C	5.22	1.9 to 6	3.5 to 5

CONCLUSIONS

Biodiesel was produced from WCO using nickel (ii) nitrate doped chicken manure as catalyst. The characterization results of the catalyst indicated that nickel (ii) nitrate doped chicken manure can be utilized as a solid heterogeneous catalyst for the biodiesel production. Furthermore, RSM was successfully utilized for the prediction and optimization of process parameters for production of biodiesel. The optimum yield of biodiesel of 99.4026% was achieved at a temperature of 69.81°C, reaction time of 60 minutes with a methanol/oil ratio of 6:1 and catalyst loading of 1wt.%. Biodiesel produced using the catalysts was within appropriate range when compared with ASTM and EN standards

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