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Optimum Biodiesel Production from Ternary Non-Edible Oils Using Doped Snail Shell Catalyst: RSM and ANN as Tools for Modelling

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Abstract: Biodiesel is more environmentally friendly than conventional fuel, and it has become crucial as alternate source of fuel in the world we live in today. It can be produced by the transesterification of oil with alcohol in the presence of a catalyst. This study optimized the biodiesel production from ternary non-edible oils using catalyst developed from snail shell doped with nickel nitrate and cobalt chloride. The developed catalyst was characterized using Brunauer Emmet Teller (BET), Fourier Transform Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), and X-ray fluorescent (XFR) analysis. Biodiesel was produced from the ternary oil blend of waste vegetable oil (WVO), jatropha oil (JO) and neem seed oil (NSO) blended in 80:10:10 ratio and characterized by FFA of 4.7%. Production was carried out using Box-Behken design (BBD) of four input variables. Response surface methodology (RSM) and artificial neural network (ANN) used in modelling the process and compared to determine their performance. The doped catalyst was characterized with high surface area of 221.02 m^2/g and high CaO content of 58.7%, with other oxides such as SO_3, SiO_2, and Al_2O_3 present in significant quantities. RSM and ANN were efficient in the modelling of the process with high R^2 value of 0.9488 and 0.9827 respectively. Numerical optimization gave optimum biodiesel yield of 98.14 with a methanol/oil ratio of 16:1, catalyst loading of 1.83 wt%, reaction temperature of 70°C, and reaction time of 69 minutes. The properties of biodiesel produced were in conformity with ASTM D6751 and EN 14214 standards.

Keywords: Biodiesel, Snail shell, Modelling, Ternary oils

INTRODUCTION

Energy is the most basic essential for human survival. Energy sources are required for the production of food, transportation, and generation of electricity [1]. In recent times, great emphasis on the use of environmentally friendly energy sources as well as the preservation of nature has been made. For a variety of environmental factors, clean and renewable energy will form the bedrock of energy supply, and this is where the production of biodiesel being a form of renewable energy, as a viable alternative to fossil fuels comes in. Biodiesel is an environmentally friendly, clean-burning fuel that is produced using various chemical processes including, direct use and blending, transesterification reaction, thermal cracking and micro-emulsion.

Biodiesel production using transesterification method of any oil occurs in the presence of alcohol using a catalyst. Conventional biodiesel production process use edible oils as feedstocks and homogeneous catalyst [2, 3]. The cost of biodiesel production is majorly affected by feedstocks cost [4]. The cost of biodiesel feedstocks is up to 88% of the total biodiesel production cost [5]. As a result, the use of non-edible oils or waste cooking oils can effectively lower the cost of biodiesel production cost [6]. Also, with homogeneous catalyst, numerous limitations has been associated to its use including soap generation; inability to recover catalyst used, treatment of wastewater generated during purification etc. [7]. Considering these limitations, heterogeneous catalysts are being used as a replacement. Heterogeneous catalysts are eco-friendly and recoverable [8].

Biodiesel production is dependent on various process variables including methanol/oil ratio, catalyst loading, reaction time, reaction temperature, and agitation speed. In order to create a cost effective production process, optimization of these process variables are required. Response surface methodology (RSM) is a statistical tool used for designing experiments with multiple variables, modeling, and optimization of processes leading to acceptable results from a reduced number of experiments and cost [9]. The tool analyzes all possible individual and interactive effects of the independent process variables on the response variable(s) and then develops a model to describe the process. Artificial neural network (ANN) on the other hand, is a collection of biologically inspired (simplified model of a human brain) mathematical techniques designed for machine learning, regression, and statistical analysis of often complicated data [10]. ANN is made up of a number of training and recall algorithms that are linked to the neural structure, which includes neurons as processing elements and synaptic weights connected to the connections between neurons. The most favored network topology for processing speed, scalability, fault tolerance, and performance is feedforward and feedback neural network designs [11]. RSM and ANN has been reported as effective tools for modelling and optimization esterification and transesterification processes [10, 12, 13].

MATERIALS AND METHODS

Materials

WVO was collected from local food vendors within Benin City, Nigeria. The WVO was filtered and then heated at 100ºC to lower the content of moisture. JO and NSO were obtained from a vendor within Benin City, Nigeria. The oils were mixed in an optimal ratio of 80:10:10 for WVO, JO and NSO respectively. Snail shells for catalyst production was obtained from a GT foods in Benin City, Nigeria. Nickel nitrate, cobalt chloride and other analytical grade reagents used were purchased from a vendor within Benin City, Nigeria.

Catalyst Preparation

The snail shells obtained were broken and washed severally using water to remove impurities. The washed snail shells was then dried to ensure removal of moisture. The dried snail shell was grinded and then sieved to ensure size uniformity. The sieved snail shell was calcined in a muffle furnace at 800ºC for 4 hours [14].Wet impregnation method was used to create the doped catalyst made up of calcined snail shell mixed with nickel nitrate and cobalt chloride. It was mixed together in the proportion of 10 %, 15%, and 75% respectively. The mixture resulted in a slurry solution which was put on a heated magnetic stirrer at 100ºC with constant stirring until the water was evaporated from it. The resultant was then dried for several hours at 100ºC in an oven. The resulting mixture was subjected to a 3 hour calcination process at 700ºC in a muffle furnace [15]. The developed doped catalyst was then taken out and stored in an air tight container.

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 analysis showed the surface structure. XRD showed the crystalline phase. And XRF analysis determined the composition of oxides.

Experimental Design and RSM Modelling

Box Behken Design (BBD) was used to design the experiment using Design Expert 13. The effect of four input variables (methanol/oil ratio, catalyst loading, reaction temperature and reaction time) on the response (biodiesel yield) was explored. The BBD resulted in 29 experimental runs. Table 1 shows the coded and actual levels of the BBD variables.

Table 1: Coded and actual levels of BBD variables

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

Biodiesel Production and Characterization

One-step transesterification method described by Obahiagbon et al., [7] was used. Based on experimental design, mixed oils was measured in a specific amount into a conical flask, then heated on the magnetic stirrer to a specific temperature. The specific amount of prepared catalyst was measured into a beaker and measured methanol was added. The measured methanol and catalyst were mixed and the resulting solution poured carefully into the heating oil. Simultaneously the reaction time was set as stipulated. The mixture was allowed to react until reaction time had elapsed. This was repeated for the 29 experimental runs as generated by BBD. The mixture at the completion of each reaction was separated using a centrifuge. The yield of biodiesel produced was calculated using Equation (1).

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

ANN Model Development

The modeling of the one-step transesterification process of the mixed oil using the doped catalyst developed by ANN was conducted using a feedforward, multilayer architecture, and a back-propagation, learning algorithm that was based on the Levenberg-Marquardt method. The hyperbolic tangent sigmoid (tansig) and pure linear (purelin) activation transfer function was respectively implemented for the hidden and output layers. The experimental data were split into training (60%), validation (25%), and testing (15%) datasets to appraise the ability of the model to predict hidden data not used for training as well as its generalization capability [16,17].

Comparative Performance of RSM and ANN

The performance of RSM and ANN was evaluated using standard statistical indicators such as coefficient of determination (R^2), mean square error (MSE), and root mean square error (RMSE), as shown in Equations (2) – (4). The results obtained were used in comparing their performance to determine superiority.

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(x_{a,i}-x_{p,i})^2}{\sum_{i=1}^{n}(x_{p,i}-x_{a,ave})^2} \quad (2)$$

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(x_{p,i} - x_{a,i})^2 \quad (3)$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(x_{p,i} - x_{a,i})^2} \quad (4)$$

Where

- n is the number of experimental runs,
- $X_{p,i}$ is the estimated values,
- $X_{a,i}$ is the experimental values,
- $X_{a,ave}$ is the average experimental values.

RESULTS AND DISCUSSIONS

Properties of the Mixed Oil

The individual oils were characterized by FFA of 3.3, 11.48 and 8.13 %; and density of 0.87, 0.94 and 0.91 g/cm^3 for WVO, JO and NSO respectively before blending was carried out. The ternary oils blended in 80:10:10 ratio for WVO, JO and NSO respectively were analyzed and the result of the analysis are presented in Table 2.

Table 2: Physicochemical properties of the ternary mixed oil

Properties	Values
Acid Value (mg KOH/g)	9.40
FFA (%)	4.70
Saponification Value (mg KOH/g)	224.23
Peroxide Value (meq/kg)	16.7
Viscosity at 32.4ºC (MPa.S)	19.00
Density (g/cm^3)	0.88
Iodine value (mg KOH/g of oil)	90.10

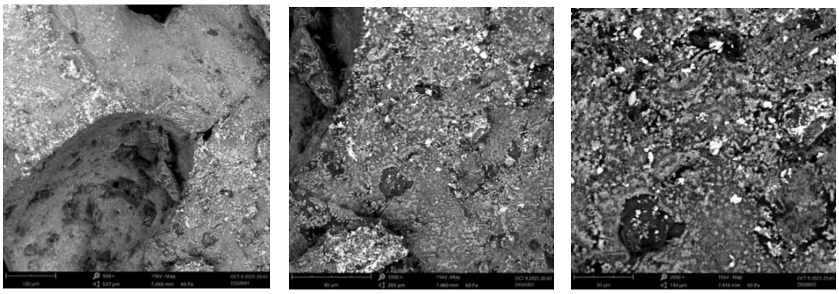
Catalyst Characterization

BET Analysis

BET analysis was carried out on the doped catalyst and the results obtained showed a surface area of 221.02 m^2/g; pore diameter of 2.82 nm, and pore volume of 0.134 cc/g. The high surface area and pore volume observed may be attributed to the effect of calcination which removed the moisture content that filled up the pores of the various catalyst resulting to an increase in their surface area and pore volume. A large surface area will allow easy diffusion of the reactants into the active sites of the catalyst, resulting in high activity of the catalyst [18].

SEM Analysis

The SEM images shown in Figure 1 depicts the surface morphology of the doped catalyst in different magnifications. The micrographs clearly indicates that the catalyst surface was characterized by irregular rough pores, with high porosity which are necessary as attachment sites for the reaction process. These irregularities can be caused by the heterogeneous nature of the catalyst precursor, which vary in composition and surface properties. Particle agglomerations are also observed on the surface, although lots of smaller particles are seen around these agglomerations. The porous nature indicates a high surface area which is necessary for effective catalyzed reactions. Kaewdaeng et al., [19] discovered through SEM analysis that catalyst developed from snail shell were rough with crack and high porosity.



(a) (b) (c)

Figure 1: SEM images of catalyst (a) x500 (b) x1000 (c) x2000

FTIR Analysis

The functional groups on the surface of the catalyst produced was elucidated via its FTIR spectra.

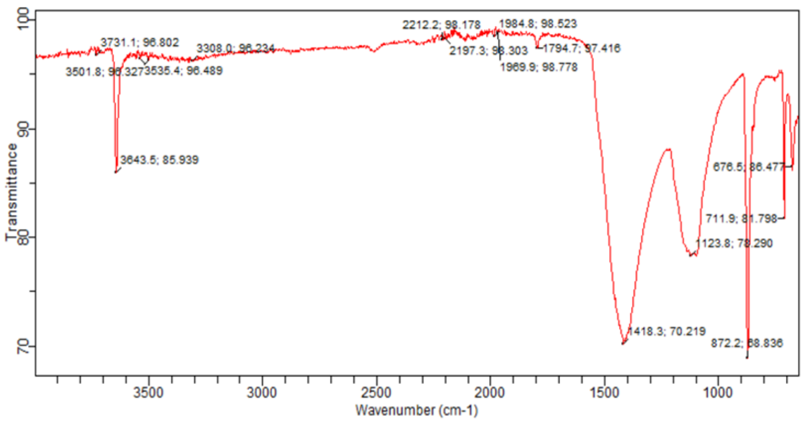


Figure 2: FTIR spectrum of catalyst

Figure 2 shows the FTIR spectra. A major peak at 872.2 cm^{-1}, and a set of prominent peaks at 676.5 cm^{-1}, 711.9 cm^{-1}, 1123.8 cm^{-1} and 3643.5 cm^{-1}. The peak at 676.5 cm^{-1} and 711.9 cm^{-1} corresponds to the presence of substituted benzene group and the peaks at 872.2 cm^{-1} and 1123.8 cm^{-1} corresponds to the presence aromatic ring due to C-H in plane bend. The peaks at 1794.7 cm^{-1}, 1984.8 cm^{-1}, 1969.9 cm^{-1} corresponds to the alkene and acid (acyl) halide group due to C-H stretch. The peaks at 3308.0 cm^{-1}, 3535.4 cm-1, 3501.8 cm-1, 3731.1 cm-1, and 3643.5 cm-1 corresponds to hydroxyl, phenols and primary alcohol due to the O-H stretch.

XRF Analysis

The composition of the catalyst obtained via XRF analysis is presented in Table 3. The result shows that the major constituents was calcium oxide (CaO) in a concentration of 58.7%. CaO is highly employed in biodiesel production process due to its low toxicity and high reactivity. Other oxides such as cobalt oxide (CoO), nickel oxide (NiO), sulfur trioxide (SO_3), silicon oxide (SiO_2) and aluminum oxide (Al_2O_3) were found present. The presence of CoO and NiO observed shows the incorporation of nickel nitrate and cobalt chloride in the catalyst in order to enhance the reactivity of the catalyst. The presence of these basic and acidic oxides in the various catalysts shows the ability of the catalyst to carry out a one-step transesterification of triglycerides effectively. The acidic properties gives the catalyst the ability to facilitate the esterification step of the biodiesel production process, while the basic properties facilitates the naturalization of high FFA content in the feedstock and also catalyze the transesterification step of the biodiesel production process.

Table 3: XRF data for catalyst

Component	Concentration (%)
SiO_2	17.671
Fe_2O_3	0.176
CoO	2.171
CaO	58.733
TiO_2	0.499
Al_2O_3	11.546
NiO	1.765
K_2O	0.398
Cl	0.627
SnO_2	0.310
SO_3	5.159

XRD Analysis

The XRD results of the catalysts is shown in Figure 3. The diffraction pattern obtained from the analysis of the catalyst's crystallinity provided information regarding its structural characteristics. Figure 3 shows that the catalyst had sharp peaks at diffraction angle of

23.84°, 27.18°, 30.06°, 36.70°, and 40.20°. The high sprectra shows the crystallinity of the materials. High peaks were observed for 2ϴ between 20° and 60°. The XRD analysis showed the presence dolomite, aragonite, zincite and quartz. Aragonite present is a polymorph of calcium carbonate indicating a correspondence with the XRF results. Similar results were reported by Kedir et al., [20], and Das et al., [21].

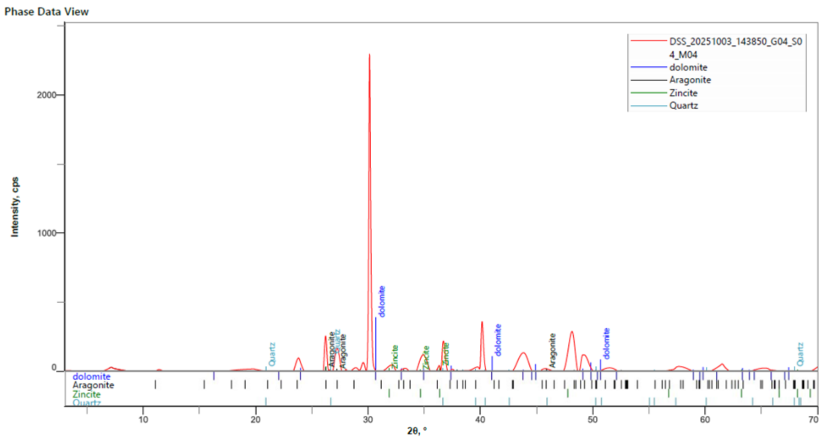


Figure 3: XRD result for the catalyst

Process Modelling and Optimization

RSM Modelling

The experimental design of the one-step transesterification reaction of the ternary mixed oils to produce biodiesel with the experimental, RSM and ANN predicted responses are presented in Table 5. Equation 2 represents the quadratic model (actual-basis).

$$Y_{DSS} = -178.32 - 3.21A + 66.97B + 0.94C + 3.21D - 0.83AB + 0.08AC - 0AD - 0.45BC - 0.10BD - 0.02CD + 0.07A^2 - 3.52B^2 + 0C^2 - 0.01D^2 \quad (2)$$

From Equation 2, catalyst loading (B), reaction temperature (C), and reaction time (D), interaction factors (AB, AD, BC, BD and CD) and quadratic factor (A^2 and C^2) had positive effects on the percentage yield of biodiesel while methanol: oil ratio (A), interaction factors (AB, AD, BC, BD and CD) and quadratic factor (B^2 and D^2) had negative effects on the percentage biodiesel yield. Using a significance level of 95%, model terms with a p value less than 0.05 are considered to be significant. This means that changes in the values of the input variable represented by that model term will have a significant effect on the yield of the biodiesel production. Conversely, model terms with p values greater than 0.05 are considered to be insignificant, indicating that changes in the values of the input factors will not significantly impact the yield of biodiesel production.

From Table 4, the quadratic model itself, was characterized with p value less than 0.0001 indicating its significance in modelling the yield of biodiesel production. The lack of fit was insignificant with p value of 0.0870. An insignificant lack of fit is desirable.

Table 4: ANOVA data for the RSM model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	959.31	14	68.52	17.09	< 0.0001	significant
A-Methanol/oil	5.33	1	5.33	1.33	0.2681	
B-Catalyst Loading	385.33	1	385.33	96.10	< 0.0001	
C-Reaction Temperature	5.33	1	5.33	1.33	0.2681	
D-Reaction Time	48.00	1	48.00	11.97	0.0038	
AB	100.00	1	100.00	24.94	0.0002	
AC	100.00	1	100.00	24.94	0.0002	
AD	0.0000	1	0.0000	0.0000	1.0000	
BC	81.00	1	81.00	20.20	0.0005	
BD	9.00	1	9.00	2.24	0.1563	
CD	25.00	1	25.00	6.24	0.0256	
A^2	40.00	1	40.00	9.98	0.0070	
B^2	80.22	1	80.22	20.01	0.0005	
C^2	6.27	1	6.27	1.56	0.2315	
D^2	41.08	1	41.08	10.25	0.0064	
Residual	56.13	14	4.01			
Lack of Fit	51.33	10	5.13	4.28	0.0870	not significant
Pure Error	4.80	4	1.20			
Cor Total	1015.45	28				

The fit statistics data showed R^2 of 0.9447, adjusted R^2 of 0.8894 and predicted R^2 of 0.7014. 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 prediction of the biodiesel production process.

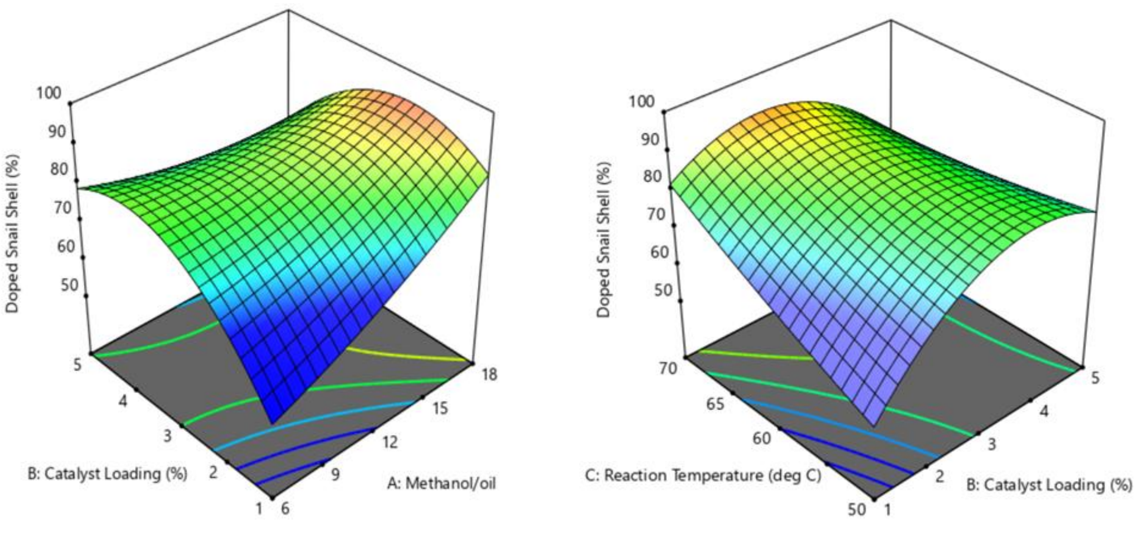
Table 5: Transesterification experimental design with RSM and ANN response

Run	A (-)	B (Wt%)	C (°C)	D (Mins)	Experimental	RSM	ANN
1	18	5	60	75	70.00	70.17	70.38
2	12	3	70	60	82.00	80.83	80.59
3	12	3	50	60	74.00	74.50	74.89
4	12	1	70	75	88.00	89.50	88.74
5	6	5	60	75	78.00	78.83	78.14
6	12	3	50	90	84.00	83.50	83.35
7	12	5	60	60	70.00	69.00	70.29
8	12	3	60	75	82.00	81.20	80.97

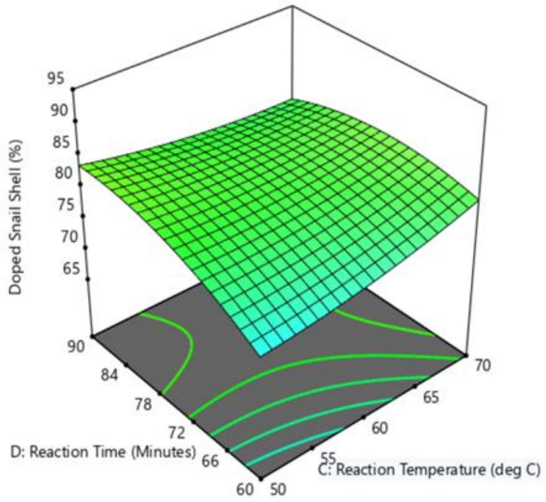
9	12	5	70	75	68.00	69.17	68.99
10	6	1	60	75	82.00	80.17	80.83
11	6	3	70	75	78.00	79.67	78.69
12	6	3	50	75	86.00	88.33	84.96
13	18	3	60	60	78.00	79.83	78.88
14	18	3	50	75	80.00	79.67	79.25
15	12	1	60	90	82.00	84.33	82.07
16	12	1	60	60	76.00	77.33	76.79
17	6	3	60	90	84.00	82.50	83.08
18	12	3	60	75	80.00	81.20	80.97
19	18	3	70	75	92.00	91.00	90.99
20	12	5	50	75	78.00	76.83	76.58
21	12	3	70	90	82.00	79.83	82.09
22	12	1	50	75	80.00	79.17	80.16
23	12	3	60	75	82.00	81.20	80.97
24	12	5	60	90	70.00	70.00	70.32
25	12	3	60	75	82.00	81.20	80.97
26	18	3	60	75	94.00	92.50	92.12
27	6	3	60	60	80.00	78.50	80.59
28	18	1	60	90	82.00	83.83	82.19
29	12	3	60	75	80.00	81.20	80.97

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)

<u>Figure 4</u>: 3D plots showing the interaction between (a) Methanol/oil ratio and catalyst loading (b) Catalyst loading and reaction temperature and (c) Reaction temperature and reaction time

Figure 4 shows the 3D surface plots representing two independent variables while keeping other variables constant. According to the Table 4, it was discovered that catalyst loading and reaction time were significant, indicating changes in their values will significantly affect biodiesel yield. Methanol/oil ratio and reaction were insignificant.

Figure 4 (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 18:1. Lowered catalyst loading resulted in an increased biodiesel yield with the maximum yield being obtained at 3wt%. Increase in biodiesel yield with increase in reaction temperature. Although after maximum yield was obtained at a temperature of 60^oC, 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 was achieved at 75 minutes.

ANN Modelling

ANN modelling of the one-step transesterification process consisted of an input layer of four neurons (methanol/oil ratio, catalyst loading, reaction temperature and reaction time), a hidden layer of 10 neurons, and an output layer of one neuron (biodiesel yield) as shown in Figure 5. The responses predicted from the ANN model are as presented in Table 5. R values of 0.9974, 0.9961, 0.9993 and 0.9973 was obtained from the regression plot for the training, validation, testing, and whole data sets indicating that there is a good correlation between the network output and the actual results as shown in Figure 6.

Also, the high R values confirmed good generalization and predictive capacity of the ANN model since it was able to predict the outputs of validation and testing data that were not part of the training set used to develop the model [16]. High R^2 value of 0.9827 was obtained suggesting 98.27% of the variations in both the experimental and predicted values can be explained, indicating a good fit of the model. A model is considered acceptable if $R^2 \geq 0.8$ [10].

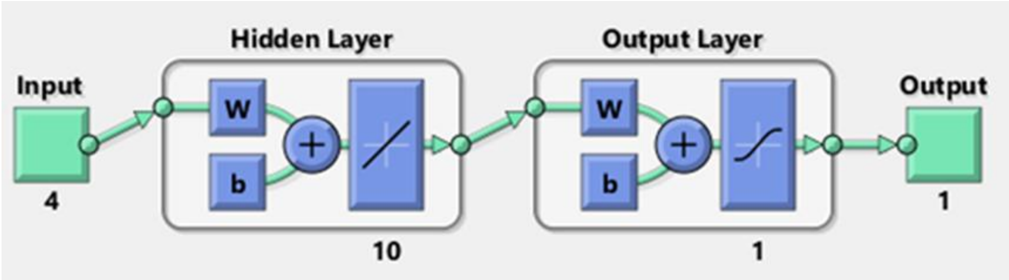


Figure 5: ANN model architecture

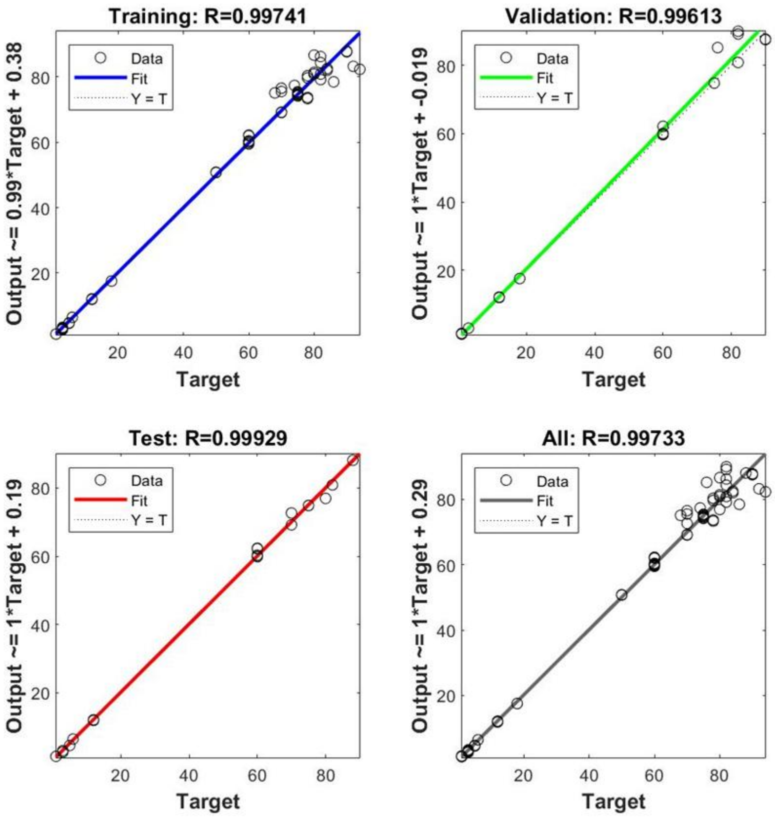


Figure 6: Regression plot of training, validation and testing

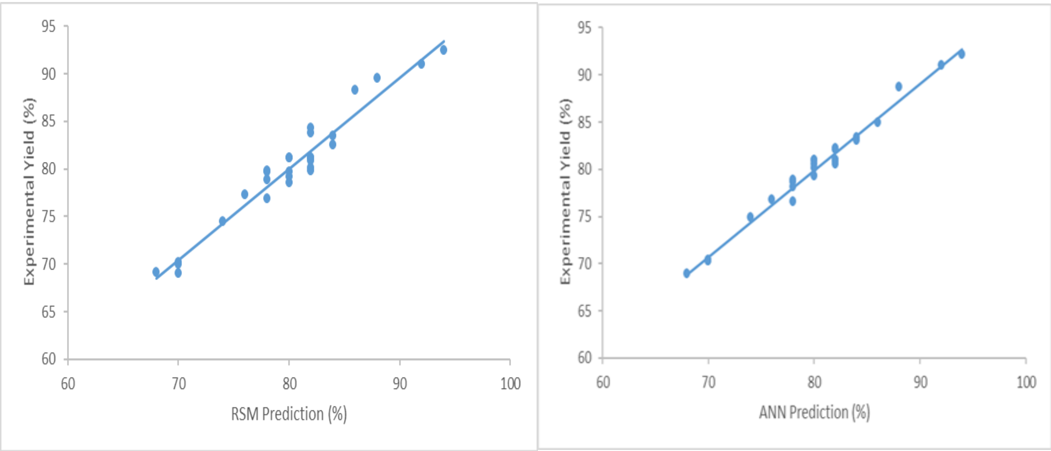
Comparison of Performance of the RSM and ANN Models

The performance of the models in terms of predictive accuracy for the one-step transesterification was compared based on their statistical values and the results obtained are presented in Table 6. ANN had the highest R^2 value of 0.9827, and the lowest MSE and RMSE values of 0.7891, and 0.8883 respectively.

This observation is collaborated by the plots of the experimental against predicted values shown in Figure 7 based on the R^2 of the models. The values predicted by the ANN model aligned closer to the reference line than the values predicted by the RSM model. Hence, predictions by the ANN model were superior to the RSM model in one-step transesterification of mixed oil.

Table 6: Comparison of RSM and ANN performance

Parameter	RSM	ANN
R^2	0.9488	0.9827
MSE	1.7963	0.7891
RMSE	1.3403	0.8883



(a) (b)

Figure 7: Experimental vs predicted yield using (a) RSM (b) ANN

Optimization of Biodiesel Yield

From the numerical optimization studies carried out, optimum biodiesel yield of 98.14% was achieved with methanol/oil ratio of 16:1, catalyst loading of 1.83 wt%, reaction temperature of 70℃. To validate these optimal conditions obtained, experimental runs were carried out in triplicate using the optimal parameters. An average yield of 98.81 % was obtained, indicating a good correspondence with optimal yield.

Biodiesel Characterization

The physicochemical properties of the biodiesel produced at optimum conditions were analyzed and the results are presented in Table 7. The properties of the biodiesel are well within the required standard as compared to ASTM D6751and EN14214 standards of biodiesel. Fatty acid profile of the biodiesel produced as obtained by GC-MS test is presented in Table 8 and the sample results is shown in Figure 8.

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

Properties (units)	Biodiesel produced	ASTM D6751	EN14214
FFA (%)	0.22	-	-
Flash point (°C)	149.8	>130	>120
Cloud point (°C)	-3.7	-	-

Pour point (ºC)	-8.5	<0	<0
Calorific value (MJ/kg)	42.62	35	-
Cetane number	70.56	>47	>51
Viscosity at 31ºC (MPa.S)	4.65	1.9 to 6	3.5 to 5
Density (kg/m^3)	885	-	-

Table 8: Fatty acid profile of biodiesel produced

Fatty acid	Nature	Chemical formula	Retention time (min)	Area (%)
Lauric acid	Saturated	$C_{12}H_{24}O_2$	19.761	11.52
Palmitic acid	Saturated	$C_{16}H_{32}O_2$	13.979	5.45
Myristic acid	Saturated	$C_{15}H_{30}O_2$	11.824	1.14
Stearic acid	Saturated	$C_{18}H_{36}O_2$	15.689	68.59
Behenic acid	Saturated	$C_{22}H_{44}O_2$	19.387	0.24
Lignoceric acid	Saturated	$C_{24}H_{48}O2$	20.929	0.16
Oleic acid	Unsaturated	$C_{18}H_{34}O_2$	20.720	4.78
Decanoic	Saturated	C11H22O2	6.745	6.86
Others				1.26

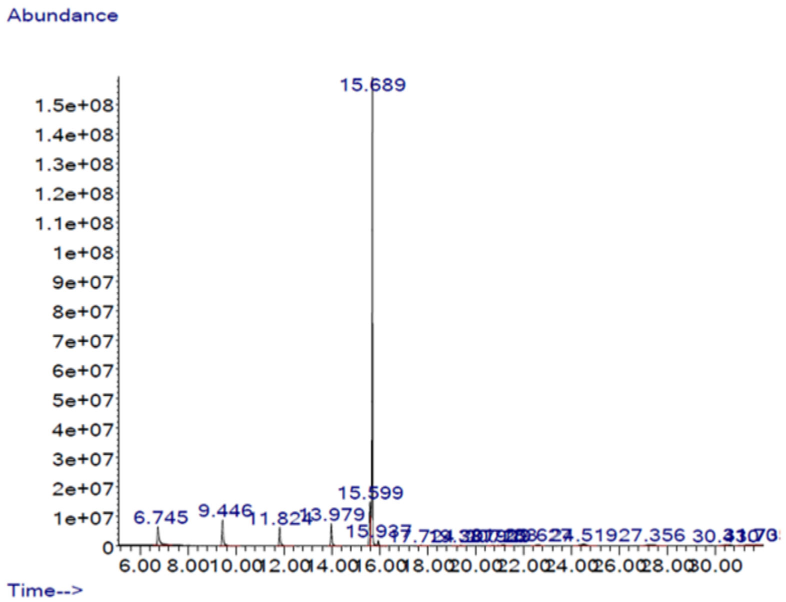


Figure 8: Chromatogram of biodiesel produced

CONCLUSION

Biodiesel was produced from ternary mixed oil using doped snail shell as catalyst. The characterization results of the catalyst indicated that doped snail shell can be utilized as a solid heterogeneous catalyst for the biodiesel production. Furthermore, RSM and ANN displayed high predictive capacity with high R^2 values of 0.9488 and 0.9827 respectively.

The prediction by ANN model was superior with higher R^2 value and lower MSE and RMSE values. Optimum biodiesel yield of 98.14% was obtained with a methanol/oil ratio of 16:1, catalyst loading of 1.83 wt%, reaction temperature of 70°C, and reaction time of 69 minutes. The properties of biodiesel produced were in conformity with ASTM D6751 and EN 14214 standards.

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