Transactions of NAMP 20, 2 (2024) 93-102

Transactions of
The Nigerian Association of
Mathematical Physics
Journal homepage: https://nampjournals.org.ng

CALCINED EGGSHELL – DOPED BIOCHAR AS CATALYST FOR BIODIESEL PRODUCTION FROM WASTE COOKING OIL: A PROCESS OPTIMIZATION STUDY

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ARTICLE INFO

Article history:
Received xxxxx
Revised xxxxx
Accepted xxxxx
Available online xxxxx

Keywords:
Biodiesel,
Calcined eggshell,
Optimization,
Biochar
Heterogeneous catalyst
Response surface methodology.

ABSTRACT

In spite of the numerous advantages associated with use of bio-based heterogeneous catalysts in production of biodiesel, leaching of active sites has remained one of its drawbacks that limits its commercialization. In this study, biochar produced from carbonization of rubber seed shells (RSS) was used as support for synthesis of a low-cost catalyst. The catalyst was synthesized by impregnating the biochar with calcined eggshells. The performance of the catalyst was assessed by conducting transesterification reactions using waste cooking oil (WCO) as feedstock. The transesterification process variables were studied and optimized using response surface methodology (RSM). Results obtained showed that the catalyst was effective in catalyzing the transesterification of WCO. The optimum reaction conditions were: methanol-to-oil molar ratio of 15.8:1, catalyst concentration of 4.0 wt.%, reaction temperature of 61.7 ^{o}C, and reaction time of 3.9 h, resulting in biodiesel yield of 93.1 %. Reusability assessment demonstrated catalyst stability to leaching, providing biodiesel yield of 80.1% after the fifth cycle.

1. Introduction

The consumption of energy has drastically increased since the dawn of the industrial era, which is a direct consequence of improvements in our lifestyle, technology, and transportation methods [1]. Fossil-based sources account for most of the global energy consumption. However, the finite nature of fossil fuel sources and the attendant environmental concerns made it necessary to search for sustainable and renewable alternative sources [2 – 4]. Biodiesel has been identified as a suitable alternative fuel for diesel engines due to its similar fuel properties to those of petro-diesel. Biodiesel is renewable and produces less harmful emissions than conventional petrol-diesel.

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https://doi.org/10.60787/tnamp.v20.384

Thus, its significant utilization would lead to a decrease in carbon dioxide, sulfur dioxide, unburned hydrocarbon, and particulate matter emissions [5, 6, 7].

Biodiesel is primarily fatty acids of alkyl esters produced from the transesterification of vegetable oil or animal fat, with a short-chain alcohol (methanol or ethanol) in the presence of a suitable catalyst. In the process, a by-product (glycerol) is formed alongside biodiesel. Commercialization of biodiesel production is limited by the high cost of the production process. About 80% of the cost of production comes from the cost of oil feedstock [8]. The use of traditional edible oil as feedstock in synthesis of biodiesel is currently being discouraged as a result of the competition it creates between food and fuel [9]. The affordability and cost-competitiveness of biodiesel in international energy market have necessitated search for alternative feedstocks that will reduce current high cost of the product [10]. Attention has shifted to use of cost-effective non-edible oils such as neem seed oil [11], rubber (*Hevea brasiliensis*) seed oil [1, 12, 13], mahua (*Madhuca Indica*) seed oil [14], date seed oil [15], Waste cooking oil [16, 17], etc. Each year, a significant amount of waste cooking oil is generated in Nigeria from the nation's top eateries, hotels, caterers, and homes, which degenerate into environmental issues, particularly in developing nations like Nigeria with poor waste management practices [10, 18]. Utilizing waste cooking oil feedstock for biodiesel production will not only reduce the cost of biodiesel production but also help mitigate the environmental consequences of its disposal.

This study aimed to synthesize a stable CaO-based catalyst from low-value sources (rubber seed shells and eggshells) for the production of biodiesel from waste cooking oil. In most parts of the world, especially Nigeria, agricultural and household wastes like rubber seeds and eggshells are widely available, and when they are disposed of indiscriminately, they pose a threat to the environment. There will be significant financial and environmental advantages to valorizing these wastes by using them to make biodiesel.

2.0 Materials and Method

2.1 Materials

The materials used in this study were waste cooking oil, rubber seed shells, and eggshells. The rubber seed shells were obtained from Rubber Research Institute of Nigeria (RRIN), Iyanomo, Edo state, Nigeria. The WCO and eggshells were obtained from various local food vendors in the Ugbowo neighborhood of Benin City, Nigeria.

2.2 Method

2.2.1 Pretreatment of Materials

The WCO was filtered using muslin fabric to remove any suspended food particles, and the purified oil was subsequently dried at 105 °C. The physicochemical properties of the oil were an acid value of 5.8 mgKOH/g, saponification value of 202 mgKOH/g, Peroxide value of 12.7 meq peroxide/kg, and viscosity of 20.3 mm^2/s [19].

The WCO, being acidic, was esterified with sulfuric acid in a three-necked batch reactor equipped with a reflux condenser and a magnetic stirrer for a period of 3h. The esterified oil was then separated, washed, and dried at 105 ^oC in the oven.

2.2.2 Catalyst Preparation

Eighty grams of rubber seed shells were subjected to carbonization at 600 °C for 4 hours in an inert muffle furnace with a nitrogen supply, yielding rubber seed shell biochar. Subsequently, twenty grams of this biochar were activated by heating with 100 ml of a saturated solution of potassium hydroxide for 4 hours to produce activated biochar. On the other hand, the pretreated

eggshells were calcined at 900°C to produce calcined eggshells. The sulfonated rubber seed shell biochar was then combined with the calcined eggshells in a 3:2 ratio and mixed with 150 ml of distilled water. This mixture was heated to dryness at 105°C, and the resulting residue was recalcined at 700°C to produce the catalyst known as calcined eggshell-impregnated rubber seed shells biochar (CERSS-BC) [20].

2.2.3 Catalyst Performance Evaluation

The effectiveness of CERSS-BC was tested by conducting a transesterification reaction on WCO. The experimental setup consisted of a three-neck glass reactor equipped with a reflux condenser and a thermometer. The catalyst and methanol were added to the reactor in the correct proportions and mixed thoroughly before adding 40 grams of the pretreated WCO. The reactor was sealed tightly to prevent methanol loss, and the contents were stirred continuously at a constant rate of 450 rpm. The values for the other reaction parameters were determined based on the experimental design (Table 1). At the end of the reaction period, the reactor's contents were centrifuged, and the solid catalyst was separated from the liquid through filtration. The filtrate was then transferred to a separating funnel and allowed to separate into two layers over 4 hours. The biodiesel and glycerol, which formed the upper and lower layers respectively, were collected into separate beakers. The biodiesel was washed with hot distilled water and dried at 105°C to ensure it was free from contaminants. The yield of biodiesel was then calculated using Equation 1.

$$\text{Biodiesel yield (\%)} = \frac{\text{Weight of biodiesel (g)}}{\text{Weight of WCO (g)}} \times 100\% \qquad 1$$

2.2.4 Design of Experiment

The influence of transesterification reaction parameters on biodiesel yield and the optimization of the transesterification process were studied using central composite design of response surface methodology. This was performed with the aid of Design Expert 7.0 software. The reaction variables studied were methanol-to-oil ratio (A), catalyst concentration (B), reaction temperature (C), and reaction time (D). The response was biodiesel yield. The ranges of the factors chosen are shown in Table 1.

Table 1: Ranges of the process factors studied

Variables	Symbols	Coded and Actual Levels				
		-α	-1	0	1	α
Methanol-to-oil ratio (mol/mol)	A	4:1	8:1	12:1	16:1	20:1
Catalyst concentration (wt%)	B	1	2	3	4	5
Reaction temperature (^{o}C)	C	50	55	60	65	70
Reaction time (hr)	D	1	2	3	4	5

3.0 Results and Discussion

A four-factor response surface methodology-based central composite design was used to model and evaluate the catalytic performance of CERSS-BC. Thirty experimental runs were conducted and the results show that biodiesel yields ranged from a maximum of 92.4% to a minimum of 15.3%, with an average of 62.0% under the different combinations of reaction conditions. The maximum biodiesel yield of 92.4% occurred at a temperature of 50 ^{o}C, methanol-oil molar ratio of 8, catalyst concentration of 2 wt%, and a reaction time of 8 h, while the minimum yield occurred

at temperature of 50 ^{o}C, methanol-oil molar ratio of 16, catalyst concentration of 2 wt%, and a reaction time of 4 h.

Multiple regression analysis performed on the experimental data revealed that a second-order quadratic polynomial model described the relationship between the process factors and the response in the transesterification of WCO using CERSS-BC as a catalyst. Equation 2 shows the quadratic model in terms of the actual values.

$$Y = -1406.03333 - 29.70938A - 49.8625B + 52.3825C + 86.44167D + 2.24688AB + 0.32500AC + 1.41250AD + 0.56500BC - 3.17500BD - 1.34250CD - 0.059896A^2 + 0.36667B^2 - 0.44633C^2 - 0.39583D^2 \quad (2)$$

Where Y is the biodiesel yield (%), A is the methanol-to-oil molar ratio (mol/mol), B is the catalyst concentration (wt.%), C is the temperature of reaction (^{o}C), and D is reaction time (h).

An ANOVA was performed to evaluate the fit and statistical significance of the second-order quadratic model, its terms, and their interactions. Results are shown in Tables 2 and 3. The model has an F value of 71.69 and a p-value of 0.0001, indicating it is statistically significant with only a 0.01% chance that this F value is due to noise. All model terms and interactions, except for A^2, B^2, and D^2, are significant with p-values less than 0.05. Terms with p-values greater than 0.1 are considered insignificant.

The model's lack of fit (2.35) is insignificant relative to pure error, indicating a good model. The model has an R^2 of 0.985, meaning it accounts for 98.5% of the observed variability in the transesterification process response. The adjusted R^2 is 0.972, and the predicted R^2 is 0.926, with a small difference of 0.046, showing strong correlation between actual and predicted biodiesel yield (Figure 1). The model's adequate precision of 34.93 indicates a sufficient signal, making it suitable for navigating the design space.

Table 2: ANOVA of the response surface quadratic model

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-Value**	**p-value Prob>F**	
Model	10500.38	14	750.03	71.69	< 0.0001	significant
A-Methanol-to-oil ratio	171.74	1	171.74	16.41	0.001	
B-Catalyst concentration	324.14	1	324.14	30.98	< 0.0001	
C-Reaction temperature	91.26	1	91.26	8.72	0.0099	
D-Reaction time	2873.28	1	2873.28	274.64	< 0.0001	
AB	1292.4	1	1292.4	123.53	< 0.0001	
AC	676	1	676	64.61	< 0.0001	
AD	510.76	1	510.76	48.82	< 0.0001	
BC	127.69	1	127.69	12.2	0.0033	
BD	161.29	1	161.29	15.42	0.0013	
CD	720.92	1	720.92	68.91	< 0.0001	
A^2	25.19	1	25.19	2.41	0.1416	
B^2	3.69	1	3.69	0.35	0.5616	
C^2	3415.09	1	3415.09	326.42	< 0.0001	
D^2	4.3	1	4.3	0.41	0.5312	
Residual	156.93	15	10.46			
Lack of Fit	129.38	10	12.94	2.35	0.1793	Not significant
Pure Error	27.55	5	5.51			

Cor Total	10657.32	29

Table 3: Fit and model statistics

Statistical parameter	Value
R^2	0.985
Adj R^2	0.972
Pred. R^2	0.926
Adeq. Prec.	34.93
Std. Dev.	3.235
Mean	61.95
C.V. %	5.221

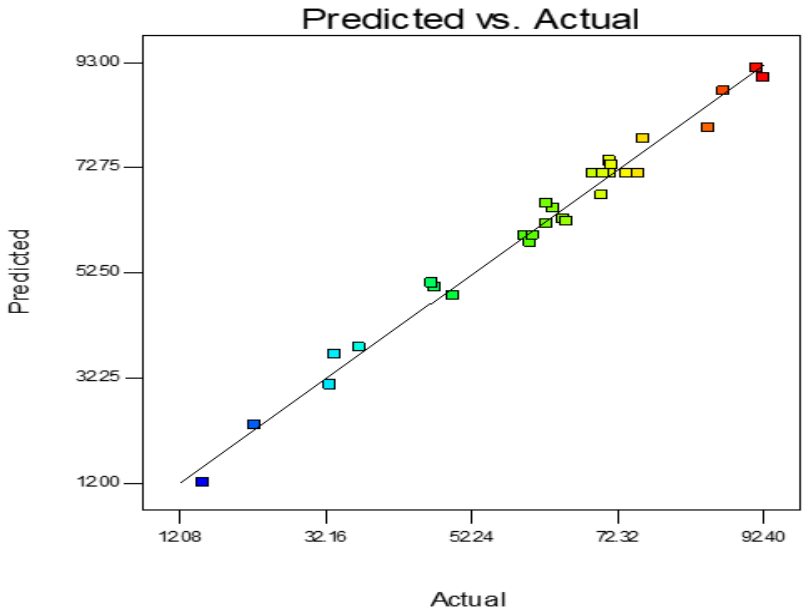

Figure 1: A plot of predicted against experimental biodiesel yield

3.1 The influence of process variables and their interactions on biodiesel yield

The response surface plot shown in Fig. 2(a) describes the effect of the interaction of methanol-to-oil molar ratio and catalyst concentration on biodiesel yield at a temperature of 60 ^{o}C and a reaction time of 3 h. At a lower catalyst concentration of 2 wt.%, biodiesel yield decreased from 72.6 to 55.3% with an increase in methanol-to-oil molar ratio from 8 to 16. This observation may be attributed to the availability of fewer catalyst active sites at a low catalyst concentration and the difficulty of separating the excess methanol from the produced biodiesel [21, 22]. However, at a higher catalyst concentration of 4 wt.%, increasing the methanol-to-oil molar ratio from 8 to 16 resulted in an increased biodiesel yield from 68.1% to 81.3%. On the other hand, no noticeable change in biodiesel yield was observed at a lower methanol-to-oil molar ratio of 8 when catalyst concentration was increased from 2 to 4 wt.%, but biodiesel yield increased with an increase in catalyst concentration at a higher methanol-to-oil molar ratio.

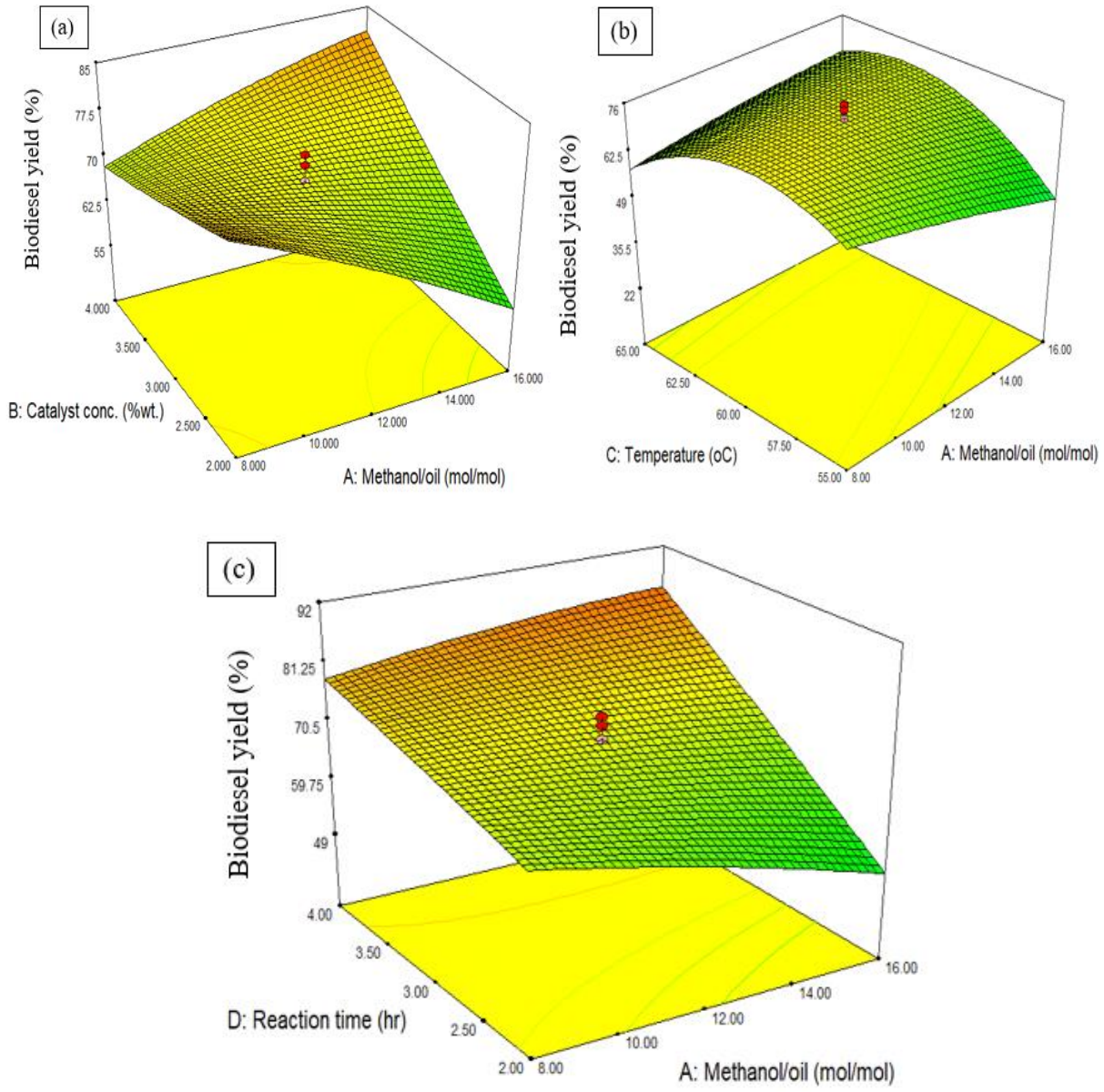

Figure 2: Response surface plot showing the influence of (a) methanol-to-oil molar ratio and catalyst concentration on biodiesel yield (b) methanol-to-oil molar ratio and reaction temperature on biodiesel yield (c) methanol-to-oil molar ratio and reaction time on biodiesel yield.

The response surface plot showing the influence and interaction of methanol-to-oil molar ratio and reaction temperature on biodiesel yield, at a fixed catalyst concentration of 3 wt.% and reaction time of 3 hrs, is shown in Fig. 2(b). It can be observed that irrespective of the value of the methanol-to-oil molar ratio used, increasing the reaction temperature from 55–63.75 ^{o}C resulted in an increased biodiesel yield. With a further increase in temperature from 63.75 ^{o}C to 65 ^{o}C, it was observed that the biodiesel yield began to drop. When the temperature is increased, the reactant molecules acquire more energy and the number of effective collisions among the reactant particles increases, leading to an increase in reaction rate and a higher biodiesel yield [23]. However, as the temperature is increased further, methanol acquires excessive energy, leading to vaporization of its molecules because of the low boiling point of methanol (64.5 ^{o}C). On the other hand, increasing the methanol-to-oil molar ratio at lower temperatures had a slightly negative impact on biodiesel

yield. Conversely, at higher temperatures, increasing the methanol-to-oil molar ratio increased the biodiesel yield.

The response surface plot showing the effect of the interaction of methanol-to-oil molar ratio and reaction time on biodiesel yield is presented in Figure 2(c). The plot shows that at any given methanol-to-oil molar ratio (within the range studied), biodiesel yield increased with an increase in reaction time from 2 to 4 hrs. Conversely, increasing the methanol-to-oil molar ratio at a shorter reaction duration resulted in a slight decrease in biodiesel yield, but at a longer reaction time, increasing the methanol-to-oil molar ratio increased biodiesel yield. A longer reaction time gives room for maximum contact of the reactants, thus enhancing the rate of reaction and consequently increasing biodiesel yield. Similar observations have also been reported in the literature [16] for biodiesel production from WCO over a bifunctional nano-catalyst.

3.2 Process optimization and model validation

The transesterification process model was optimized using a design expert numerical optimization tool. The biodiesel yield was maximized based on the ranges of values chosen for the process variables under study. The results show that a methanol-to-oil ratio of 15.79:1, a catalyst concentration of 3.95 wt.%, a reaction temperature of 61.65 ^{o}C, and a reaction time of 3.99 hrs were the optimum transesterification conditions. Under these optimum conditions, the maximum biodiesel yield was 93.11%.

To validate the developed model, transesterification reactions were conducted in triplicate under the optimum reaction conditions using CERSS-BC as the catalyst. Table 4 shows the results obtained. The optimum average experimental value of biodiesel yield was higher than the predicted optimum value of 93.1%, representing a percentage error of 0.43%. This result shows that the model can be used adequately and efficiently to predict the yield of biodiesel produced from WCO using CERSS-BC as a catalyst.

Table 4: Model validation of biodiesel production process using CERSS-BC as catalyst

Methanol/oil molar ratio (mol/mol)	Catalyst concentration (wt.%)	Reaction temperature (^{o}C)	Reaction time (hr)	Predicted biodiesel Yield (%)	Experimental biodiesel Yield (%)
15.79	3.95	61.65	3.99	93.1	92.5
15.79	3.95	61.65	3.99	93.1	91.9
15.79	3.95	61.65	3.99	93.1	93.8

3.3 Leaching and Reusability of the Catalyst

The result of the leachability experiment conducted on CERSS-BC suggested that the CERSS-BC leaches slightly into the reaction system. This was evident from the biodiesel yield of 12.1% obtained when the WCO was reacted with the filtrate from the methanol-catalyst mixture. The resistance of the CERSS-BC to leaching of active sites into the reaction system could be ascribed to the formation of a Ca-O-Si bond between the active sites and the activated carbon [24]. Al-Sakkari et al. [25] reported a 20% biodiesel yield from a leaching test conducted on cement kiln dust used as a catalyst in the transesterification of soybean oil. In comparison to Al-Sakkari et al. [25], the results of this study show that the presence of activated carbon as support improves the stability of the catalyst.

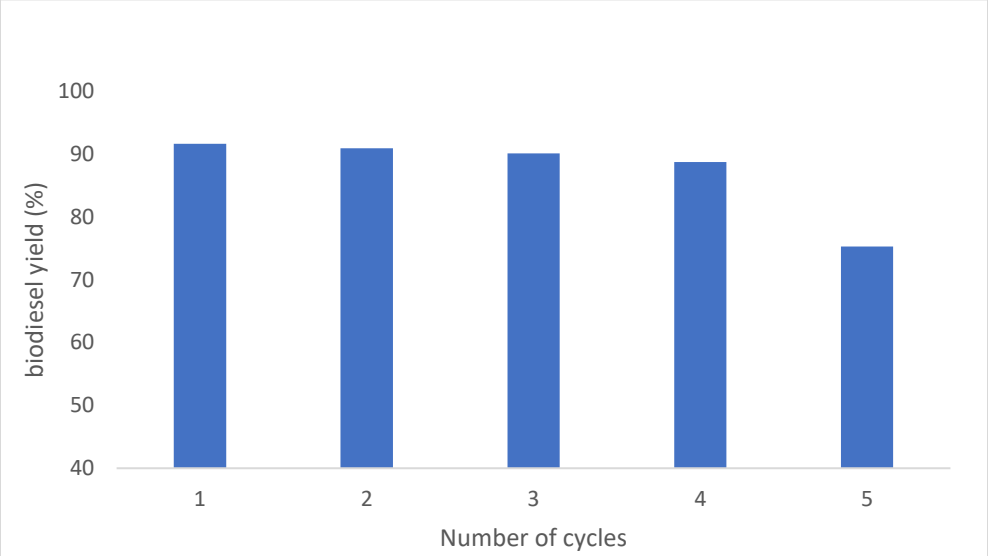

Figure 3: Reusability of CERSS-BC in biodiesel production

The results of the reusability study are presented in Figure 3. The results showed that biodiesel yield gradually dropped from 91.6% to 75.3% after the fifth cycle. This represents a drop of 17.5% in yield after five successive cycles. The decrease in biodiesel yield observed as the number of cycles increased may be attributed to the fouling of the active sites of the catalyst with glycerol. Hence, the porosity and number of active sites available for the subsequent reaction are reduced, leading to a decrease in the yield of biodiesel. These results implied that CERSS-BC as a catalyst for biodiesel production can be reused up to five times while still producing a reasonable yield of biodiesel.

3.4 Physicochemical properties of the produced biodiesel

The physicochemical properties of the WCO biodiesels produced are shown in Table 5. The fuel properties of the WCO biodiesels produced fall within the ranges of the recommended ASTM 6751 and EN14214 international standards. This implies that the WCO biodiesel can be used directly or blended with petro-diesel as a fuel for internal combustion engines.

Figure 5: Physico-chemical properties of the produced WCO biodiesel

Properties	Test method	Value	ASTM 6751 standards	EN14214 standard
Viscosity at 40 ^{o}C (mm^2/s)	AOAC	3.99	1.9 - 6.0	3.5 - 5.0
Density at 25 ^{o}C (kg/m^3)	AOAC	879.7	-	860 - 900
Iodine value (g I_2/100g fuel)	D5554-15	38.8	-	120 max.
Acid value mg KOH/g	AOAC	0.18	0.8 max	0.5 max
Pour point (^{o}C)	ASTM D97	-0.8	-15 - 10	
Flash point (^{o}C)	ASTM D93	152.2	100 – 170	120 min.
Freezing point (^{o}C)	-	-8	-	-
Cetane number	ASTM D4737	50.2	47 Min.	51 min.

4.0 Conclusion

This study has successfully utilized eggshell-impregnated biochar derived from rubber seed shells as catalyst for biodiesel production from waste cooking oil. An optimized biodiesel yield of 92.7% was obtained with methanol/oil molar ratio of 15.8:1, catalyst concentration of 3.9 wt.%,

temperature of 61.7 ^{o}C, and reaction time of 4.0 h. Leaching of catalyst was insignificant, and obtained catalyst demonstrated capacity to be reused up to five times without appreciable loss of catalytic activity. The values of the properties of produced biodiesel were found to be within the ranges recommended ASTM 6751 and EN14214 standards

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