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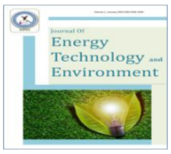

Optimized Biodiesel Production from used Cooking Oil Blend over a Bifunctional Catalyst Composite derived from Concrete Waste and Orange Peels.

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Abstract

In facilitating a shift from the traditional fossil fuel into biofuels, a heterogeneous bio-based catalyst was utilized to produce biodiesel. This bifunctional catalyst was produced using a combination of concrete waste and orange peels for the transesterification and esterification of a blend of waste oils (palm and vegetable oil) to produce biodiesel. The catalyst prepared possessed a large pore volume and rich surface area which made it adequate to synthesize biodiesel. The response surface model indicated a peak biodiesel yield of 82.59% at reaction conditions: 60 °C reaction temperature, 5 wt.% catalyst loading, 15:1 methanol to oil ratio, and at 60 mins reaction time. The existence of high active sites was indicated by the high biodiesel yield in the bifunctional catalyst. Optimization result as indicated from the analysis of variance (ANOVA) shows that the catalyst dose, the ratio of methanol to oil, reaction duration and temperature substantially influenced the production of biodiesel. The produced biodiesel had physical, compositional, and combustion characteristics that met established standards, making it an appropriate fuel suitable in compression ignition engines

1. Introduction

Energy is a vital resource, and the degree to which a nation has developed can be measured by its energy production data. Energy availability is critical for many industrial, transportation, and domestic functions [1]. Due to skyrocketing energy demand, global warming resulting from greenhouse gas emissions, rising price of crude oil, environmental pollution, and diminishing fossil fuel reserves, has resulted in increased search for renewable energy sources. The most common alternatives to fossil fuel are solar, wind, water and biofuels [2]. In order to eliminate the current dependence on fossil fuels, research initiatives are currently in progress to utilize more sustainable alternative fuels, such as biomass [3]. More also, the unstable pricing, environmental impact, and rising cost of fossil fuels has shifted attention to biofuels such as biodiesel [4].

According to existing literature, the standard procedure for producing biodiesel is transesterification [5]. In this process, a feedstock such as fat or oil undergoes a reaction with alcohol yielding alkyl esters (biodiesel), glycerol (by-product), and fatty acids with a catalyst present [6]. Nearly 95 percent of current biodiesel is synthesized from essential; oil, and it is widely used in the agriculture - based economy [7]. Specifically, used cooking oil (UCO) will be the focus of this research. UCO is produced in enormous quantities on an annual basis by different establishments, including hotels, restaurants, catering companies, and even private homes [8]. Managing fats and oils is considerably challenging because of the difficulties associated with waste disposal as well as the risk of polluting both land and water resources. Even though a small amount of UCO is repurposed into soap, the vast bulk is simply discarded into the surrounding environment. Therefore, exploring the possibilities for UCO reusability can potentially address the overall cost of producing biodiesel, thereby making a justification for this study.

In addition, biodiesel is traditionally made with homogenous strong base catalyst such as alkoxides and alkali metal hydroxides which are relatively expensive. More also, the corrosive nature and characteristics of these homogeneous basic or acidic catalysts make separation from the product mixture challenging. Therefore, to reach the requisite fuel quality, methods such as water washing and purification result in an increased production cost [9], [10]. In contrast, heterogeneous catalysts, seem more cost effective and practical for biodiesel production because of their environmentally benign nature and their relative ease of recovery [11]. Interestingly, the overall cost of biodiesel synthesis could be optimized by using waste materials to create heterogeneous catalysts [12]. Precursors for heterogeneous catalysts have included materials like snail shells [13], oyster shell [14], mussel [15] and waste chicken bones [16].

This study aims to promote research in a more cost-effective process and to optimize biodiesel production from a binary oil feedstock by producing a heterogeneous composite catalyst from waste concrete (calcium-containing material) and the peels of the orange fruit (biomass-based precursor). The catalyst's functionality was improved by doping it with tin (relatively cheap) and lithium (to enhance activity) [17], [18] to enhance its existing qualities. Modeling and improving the manufacturing process was accomplished by using Response Surface Methodology (RSM

2. Materials and Method

2.1. Materials

The used cooking oil (used palm oil (UPO) and used vegetable oil (UVO)) were obtained from domestic houses in Nigeria. The collected used oil contained various contaminants and was filtered using a muslin cloth before being heated to minimize the moisture content [6]. Concrete waste was gotten from a construction site in Benin City, Nigeria, for use as a precursor material for catalyst synthesis, while orange peels were procured in Benin City, Nigeria, from a fruit vendor. Other reagents used were purchased from BDH UK which were in analytical grade in the study were methanol, Tin chloride, lithium carbonate, ethanol, phenolphthalein, potassium hydroxide (KOH), hydrochloric acid, sodium hydroxide (NaOH), chloroform, benzene, Wij's solution and Deionized water (purchased by Local vendor).

2.2. Catalyst Preparation

Catalyst preparation was carried out using the method previously described [19]. The catalyst precursor was dried, ground and seized before been placed in a muffle furnace and heated at 700 °C for 4 h. Also, the concrete waste was ground, sieved to achieve particles smaller than 0.3 mm in diameter. After which the powder was calcined at 900 °C for 4 h in a muffle furnace. The Concrete waste-orange peels-Tin-Lithium (COSnLi) bi-functional heterogeneous catalyst was produced utilizing the wet impregnation approach adopted from literature [20]. 200 g of the calcined orange peels and concrete powder, tin chloride, and lithium carbonate (20 %, 65 %, 10 % and 5% respectively) were combined in deionized water (400 mL) and swirled vigorously for 4 h using a magnetic stirrer. Following a 6 h drying period at 110 °C, calcination was carried out on the resultant slurry for 4 h at 900 °C.

2.3. Production and characterization of biodiesel

A known quantity of waste optimal oil blend was weighed, and the mixture was placed in a 250ml three-necked glass reactor fitted with a reflux condenser and the set up was positioned on magnetic stirrer with inbuilt hotplate present. This optimal oil blend was heated according to the stated temperature in the design of experiment. A known quantity of methanol was agitated while being heated with the stirrer and allowed to mix for five minutes. The bifunctional catalyst was added to the reactor as the reaction proceeded as specified in the experimental design for the specified duration [21]. At the end of the reaction, the mixture was spun so as to separate the solids. As a result, the fluid was split into distinguished sections: the bottom layer containing glycerol (bye-product) and spent solid catalysts, and the top layer consisting of unconverted biodiesel. The recovered biodiesel was washed using distilled water so as to get rid of glycerol, residual methanol, and spent catalyst before drying to eliminate water molecules. Biodiesel yield = $\frac{\boldsymbol{mass\ of\ biodiesel\ produced}}{\boldsymbol{mass\ of\ waste\ oil\ blend\ \ used}} \times \mathbf{100}$ (1 was used to compute the amount of produced biodiesel [22].

$$\textbf{Biodiesel yield} = \frac{\boldsymbol{mass\ of\ biodiesel\ produced}}{\boldsymbol{mass\ of\ waste\ oil\ blend\ \ used}} \times \mathbf{100} \qquad \textbf{(1)}$$

Biodiesel's characteristics (flash point, cetane number, acid value, calorific value, density, moisture content, kinematic viscosity, and iodine value) were measured using known techniques [23].

3. Results and Discussion

3.1. Catalyst Characterization

X-ray diffraction (XRD) analysis

The result from the X-ray diffraction (XRD) analysis of the catalyst precursors and synthesized catalyst are shown in Fig. 1. The composite catalyst yielded significant peaks at specific angles: 22°, 24°, 26°, 28°, 34°, and 45° as displayed in Fig. 1. These peaks are characteristic of CaO, indicative of the predominant component derived from concrete waste (CaO). This finding aligns with previous study by Wang, Chen, Wen, Liao, & Chen, (2012) who first noticed that cement-made catalysts mostly consisted of CaO. Additionally, minor peaks at 47°, 52°, and 61° suggest the presence of K_2O, consistent with observations by [25] , who prepared solid catalysts containing K_2O. This oxide contributes to the catalyst's activity during transesterification, which is consistent with the results obtained from X-ray fluorescence (XRF) analyses, which further support the composition of the catalyst.

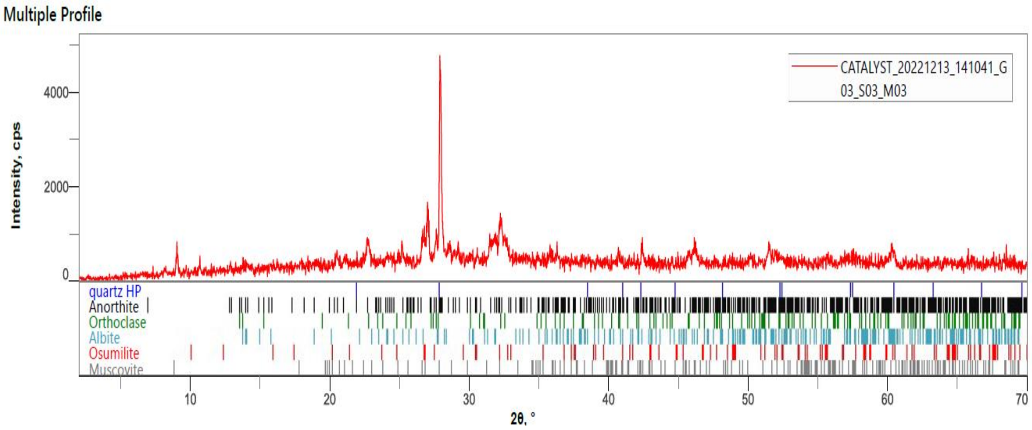

Fig. 1 XRD patterns of the composite catalyst

Scanning Electron Microscope (SEM)

The SEM micrographs in Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels.

illustrate the shape of the catalyst developed (Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels.

a) as well as the calcined precursor materials (Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels.

b and c). Due to their biological make-up, orange peels have a rough, irregular structure with rope-like appendages that are loosely packed like strands [19]. According to [19], the orange spongy nature is because of the sintering effect which occurs at high temperature during calcination. Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels.

b depicts the concrete waste with compact surfaces. The surface of the calcined catalyst appears to be aggregated with distinct pores and has very similar structure with the concrete waste but different heterogeneous distribution of the particle sizes. This can be linked with the catalyst having a higher proportion of concrete waste as precursor [26]. SEM images of orange ash in Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels.

c was a sign that the particles had broken down at high temperatures, resulting in a sponge-like, porous aggregation of complicated shape [19].

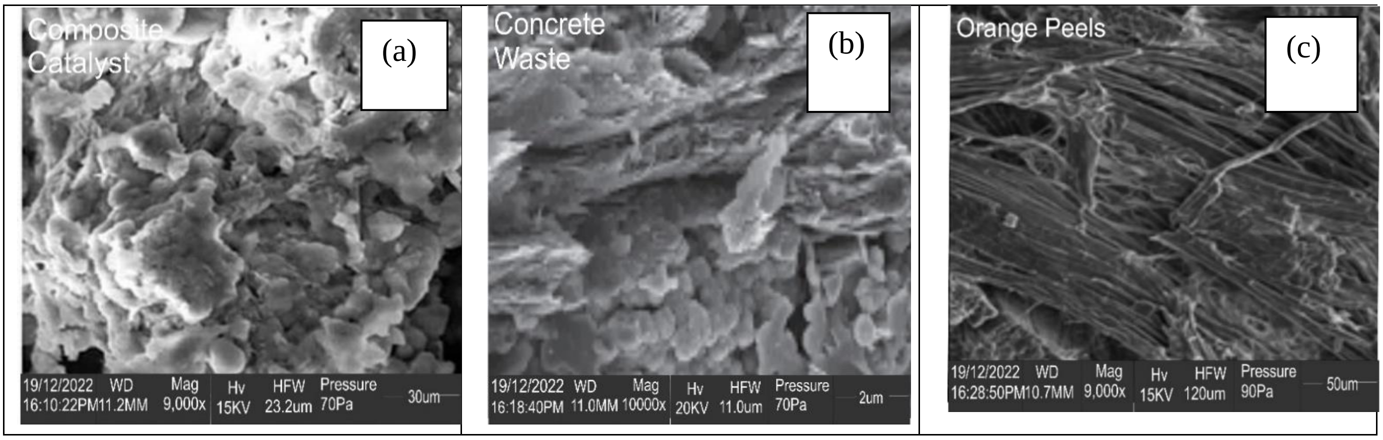

Fig. 2 SEM micrographs of the catalyst and precursor materials (a) Composite catalyst (b) Concrtet waste (c) Orange peels**.**

Fourier transform infra-red (FTIR)

The FTIR analysis result of the catalyst precursors and catalyst are shown in Fig. 3 (a-c). The main peaks representing the concrete waste occurred between 400 - 600 cm^{-1}, 1600 – 1800 cm^{-1}, and 2400 – 3600 cm^{-1} (Fig. 3b) while those for orange peels occurred at 400 – 600 cm^{-1}, 1600 - 1800 cm^{-1}, and 2800 – 3200 cm^{-1} (Fig. 3c). For the composite catalyst, the major peaks were recorded at 1800 – 2000 cm^{-1}, 2400 – 2800 cm^{-1}, and 2800 – 3200 cm^{-1} (Fig. 3a). The similar patterns observed in the spectra of the catalyst composite and the precursor materials (concrete waste) are as a result of the catalyst composite containing more concrete waste (65%). Peaks around 3200 cm^{-1}, 2800 cm^{-1}, 1690 cm^{-1}, and 600 cm^{-1} as seen in both concrete waste and the catalyst could be due to the presence of metal oxides such as CaO and SiO_2 [25].

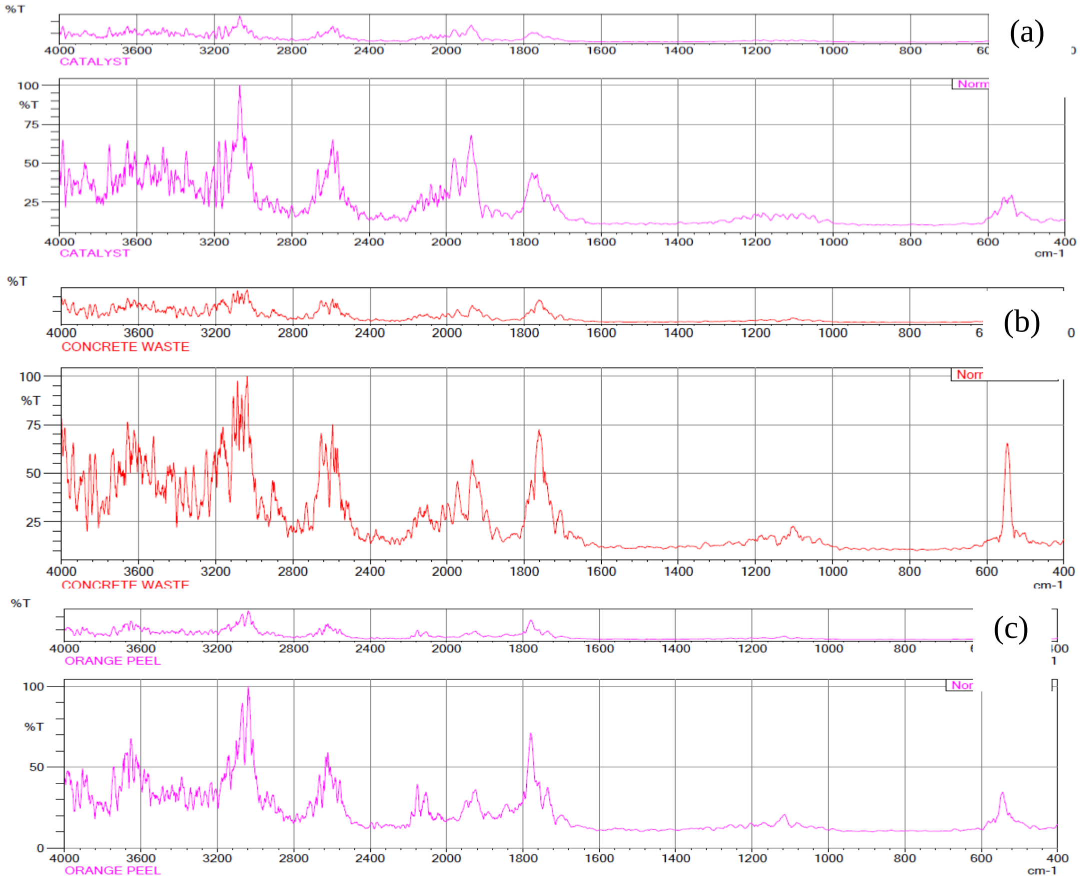

Fig. 3: FITR spectrum of the (a) composite catalyst, (b) concrete waste and (c) orange peels

Textural Properties of the catalyst composite

Table 1 presents the textural properties of the bifunctional catalyst. The textural characteristics reported in this study were superior to report given by Olatundun, Borokini, & Betiku, (2020) who used cocoa pod husk and plantain peel as precursor for catalyst (surface area = 18.86 m^2 g^{-1}, and pore volume = 0.043 cm^3 g^{-1}). Nevertheless, previous studies by Farooq and Ramli [16] who developed catalyst from chicken bones had similar textural properties (surface area = 95.4 m^2 g^{-1}, pore volume = 0.14 cm^3 g^{-1}). In addition, a higher pore volume meant there was space for reactant molecules to diffuse into the internal structure of the catalyst increasing their chances of interacting with active sites. Consequently, catalysts with higher pore volumes generally have better access to the active sites and exhibit enhanced catalytic performance.

Table 1: Textural Properties of Catalyst composite.

Surface Area(m^2g^{-1})	Pore Diameter (nm)	Pore Volume (cm^3 g^{-1})
97.5	2.8	0.65

3.2. Optimization of the Production of Biodiesel using RSM

Comparing the Box Behnken Design (BBD) to a Central Composite Design (CCD) with three or four reaction variables, the BBD is more practical for evaluating the transesterification process factors [28]. Using the BBD to optimize the transesterification process for biodiesel production, the results in **Error! Reference source not found.** shows that the methyl ester yields for the optimum oil blend ranged between 60.10- 82.59 %. A minimum optimum oil blend methyl ester yield of 60.1 % was produced at reaction conditions: 60 °C, 5 wt.% catalyst loading, 6:1 methanol to oil ratio, and at 60 mins reaction duration. Furthermore, a maximum yield of 82.59 % was obtained with reaction conditions: 60 °C temperature, 5 wt percent catalyst loading, 15:1 methanol to oil ratio, and at 60 mins reaction duration. More also, the goodness of fit for the RSM model represented in Table 2 showed predicted R^2 and adjusted R^2 values were in good agreement, indicative of a strong relationship between the actual yield and the model's prediction. A high R^2 value (0.9830) demonstrates that the optimal production of methyl ester from oil blends can be predicted by the fitted model with a reasonable amount of accuracy. The adjusted R^2 which also indicates the model's high significance is 0.9661. Furthermore, the predicted R^2 value of 0.9225 is in good agreement with the adjusted R^2. The coefficient of variation (1.33) is relatively low, indicating that the model is reproducible. A model's ability to suit a design may be quantified by calculating the sum of squares of the predicted residual errors (PRESS).

Table 2: Goodness of Fit for RSM Model.

Parameter	Value
R^2	0.983
Adjusted R^2	0.9661
Predicted R^2	0.9225
Std. Dev.	0.9869
Mean	74.31
C.V. %	1.33
Adeq Precision	31.5326

3.3. Effect of input variables on biodiesel yield

Fig. 4 explains the interaction between biodiesel's yield, reaction time and methanol to oil ratio. Results showed that increasing either the methanol-to-oil ratio or reaction timing resulted in a higher biodiesel output, indicating that both factors positively impacted biodiesel yield. Previous research has revealed that the amount of biodiesel produced is dependent on the reaction duration, and our study confirms that the reaction proceeds with time until equilibrium [29] [30]. Also, an increased biodiesel output was observed with corresponding increase in methanol to oil ratio. This demonstrates that using a concentration of methanol above the stoichiometric threshold speeds up triglyceride conversion to biodiesel.

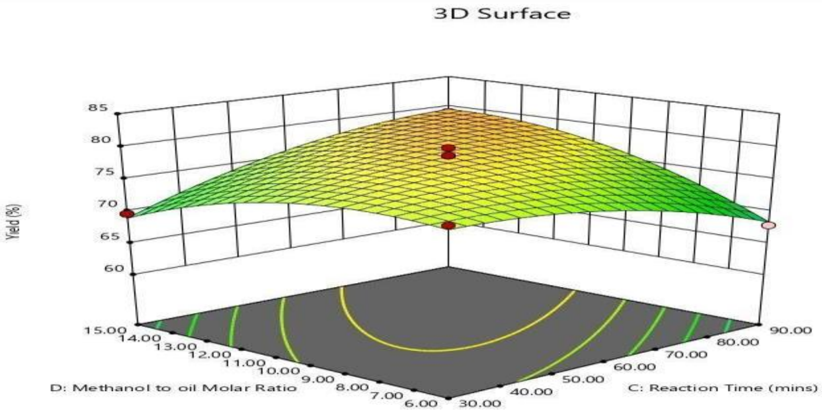

Fig. 4 Interactive effects between Methanol to Oil Ratio and Reaction Time.

With reference to the 3D plot in Fig. *5*, the biodiesel yield was observed to improve with increasing catalyst loading. The yield value reaches a maximum at a 5wt.% catalyst loading, and a 6:1 methanol to oil ratio respectively.

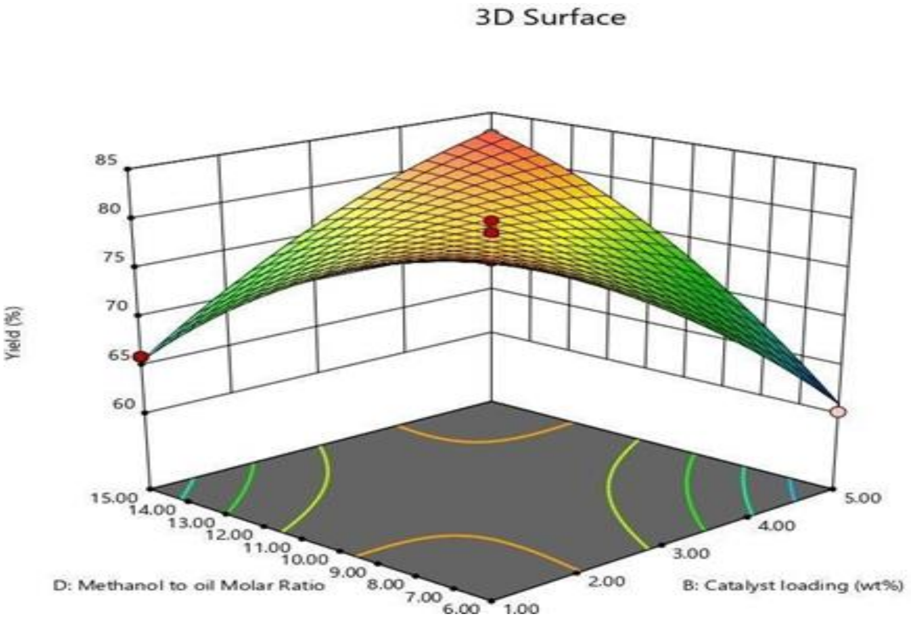

Fig. 5 Interaction between Methanol to Oil Ratio and Catalyst Loading.

Fig. 6 highlights the correlation between the yield of biodiesel, methanol: oil, and the temperature of reaction. The 3D plot indicates that a gradual increase in temperature results in a corresponding increase in reaction temperature. It is also evident from the process that the catalyst dispersion, mass transfer properties, and the interaction between the catalyst active site and methanol molecules are all influenced by higher reaction temperatures [31].

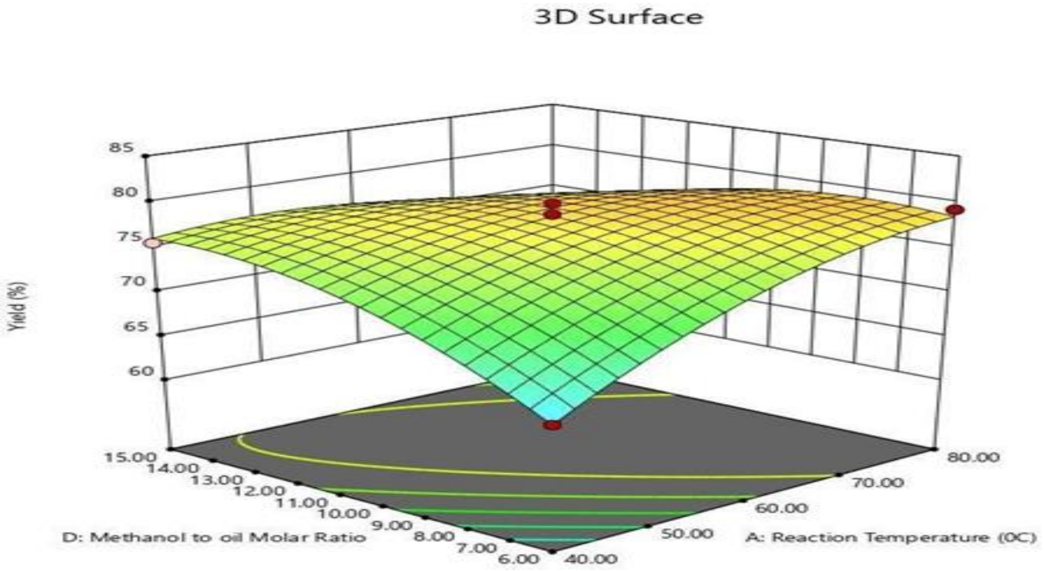

Fig. 6 Interactive effects between Reaction Temperature and Methanol to Oil Ratio

In this experimental study, the highest biodiesel yield (82.59%) required a temperature of 60 ℃. Similar to studies which reported utilizing the bones of chicken as catalyst at a reaction temperature of 57.5 ℃ and using soybeans, with highest biodiesel output of 93 % [32]. The relatively lower biodiesel yield observed in this study is likely associated with the high concentration of silicon oxide (SiO_2) (19.045%) present in the concrete waste compared to the calcium oxide (CaO). Consequently, the quality of the original concrete mix has a profound impact on the viability of concrete waste as a precursor for heterogeneous catalyst production.

3.4. Biodiesel Characterization

The biodiesel produced was characterized to determine its quality and comparison were made to standardized biodiesel properties. In this research work, the characterization results of the produced biodiesel are tabulated in Table 3. The biodiesel acid value obtained was 0.21 mg KOH g^{-1}. This showed that the biodiesel fuel had a low potential for corrosion [33]. A high cetane number of 55 and a caloric value of 39.17 MJ kg^{-1} was indicative of an excellent energy content and ignition properties of the produced fuel. A high flash point of 185 ℃ indicated high levels of safety during handling and transportation as the higher the flash points the less the risk of accidental ignition. The biodiesel viscosity obtained was 5.15 mm^2s^{-1}. This indicates that it is within a suitable range and it ensures that there is proper atomization during combustion. A high density of $876kgm^3$ showed an increased energy output per unit volume. Properly balanced flash point, viscosity and density lead to efficient combustion, reduced emissions and reliable engine operations. Furthermore, the result of the chemical composition analysis of the produced biodiesel using GC-MS is represented, The presence of 20 peaks in the total ion chromatogram. Each peak corresponds to a fatty acid methyl content of the biodiesel produced. The base peak of the mass spectra of the FAME was observed at 33.2 which contains 9,12-octadecadienal, cis-Vaccenic acid, and cis-13-Octadecenoic acid similar to established literature [34]. Interestingly, the biodiesel had a higher concentration of saturated fatty acid methyl esters. More saturated fatty acid methyl esters in biodiesel have been shown to improve thermal efficiency and reduce emission loads in prior studies.

Table 3: The Characteristics of Biodiesel

Property	**Value**	**ASTM D6751**	**EN 14214**

Physical State	Liquid	Not specified	Not specified
Color	Light brown	Not specified	Not specified
Moisture content (%)	0.02	≤0.03	≤ 0.02
Kinematic viscosity at 40 ℃ (mm^2 s^{-1})	5.15	1.9-6	3.5–5
Density (kg m^3)	876	860–900	850
Acid value (mg KOH g^{-1} oil)	0.21	≤0.5	≤0.5
Calorific value (MJ kg^{-1})	39.17	Not specified	≥35
Flash point (◦C)	185	≥130	≥101
Cetane number	55	≥47	≥51

4. Conclusion

The use of concrete scrap and orange peels as precursors in producing a heterogeneous bio-based catalyst for the transesterification of waste vegetable and palm oil mix into biodiesel was successfully achieved. it is worth noting that the produced bifunctional catalyst facilitated both esterification and transesterification processes simultaneously. the high yield of biodiesel obtained signifies that the catalyst possessed a large pore volume and excellent surface area. a maximum yield of 82.59% was obtained under the following conditions: 60°c reaction temperature, 5 wt.% catalyst loading, 15:1 methanol to oil ratio, and at 60 mins reaction time. the result indicates that reaction temperature and time, catalyst loading, and methanol-to-oil ratio had huge impacts on biodiesel production. In addition to its high physical and chemical qualities, biodiesel meets the requirements necessary for use ignition engines.

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