



## Production of Biodiesel from Waste Vegetable Oil Using Chicken Eggshell Waste as Heterogeneous Catalyst

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### ABSTRACT

*In this study, the Production of Biodiesel from Waste Vegetable Oil using Chicken Eggshell Waste as Heterogeneous Catalyst was investigated. The eggshells were calcined for 5hr at 900°C. The catalyst derived from eggshells after calcination was characterized by Fourier transforms infrared (FTIR), scanning electron microscope (SEM) and X-ray fluorescence (XRF). The XRF results revealed that the catalyst contained 95.558% of CaO, the SEM image showed the catalyst morphology and the FTIR show peaks signifying the presence of certain functional groups such as the OH group. The optimum biodiesel yield obtained was 94.58% under the following optimum process conditions: methanol to oil molar ratio of 14:1, catalyst concentration of 5wt. %, reaction temperature of 60°C and reaction time of 100mins. Physiochemical properties of the biodiesel obtained were within the ASTM standards for biodiesel. Hence, heterogeneous catalyst obtained from eggshells wastes can be used for transesterification.*

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### INTRODUCTION

Biodiesel, a promising alternative fuel to petroleum-based diesel fuel used in compression ignition (CI) engines has become the focus of many researchers in recent times. Biodiesel is produced by reacting vegetable oils or animal fats with low molecular weight alcohol, preferably methanol and ethanol in the presence of a catalyst. This reaction is known as transesterification. The use of biodiesel fuel instead of petroleum-based fuel in CI engines has several advantages. These include its high flash point, non-toxic nature and above all, it is biodegradable and also renewable. Less emission of CO<sub>2</sub> and oxides of nitrogen and sulphur are also advantages of using biodiesel in CI engines (Monyem and Van Gerpen, 2001; Hessel *et al.*, 2007).

Biodiesel composed of fatty acid methyl esters (FAMEs) is commonly produced from vegetable oils and animal fats. However, all edible vegetable oils demand a high cost of production, which is the main hindrance towards

the commercialization of biodiesel since the feedstock cost contributes to the overall project cost. Low-cost vegetable oils, such as waste oil and non-edible oil, are considered feedstocks, but their sustainability is of concerns due to the limited quantity of production. Also, waste oils and fats (used cooking oils) are used as good feedstock, its costs approach half of the cost of virgin oil. Additionally, the use of waste vegetable oils (WVO) as a reactant for biodiesel plant helps in disposing of this waste and reduction of feedstock production cost. The FAMEs blend of biodiesel is produced through a catalytic transesterification reaction of a triglyceride feedstock and methanol. Catalysts used for transesterification are either homogeneous or heterogeneous. Homogeneous transesterification is the easiest process; however, it encompasses the intricacies of separation and purification of product because of the homogeneous nature of the reaction mixture (reactant, catalyst and product). It also is more likely to form soap which requires more washing and tends to form emulsions in the mixture. All

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these process complexities result in high production costs. Therefore, heterogeneous catalysts are the catalysts of the choice for biodiesel production nowadays because they overcome some of the drawbacks of the homogeneous catalyst process. Additionally, they exhibit high catalytic activity, selectivity, their lifetime is long and have a high tolerance to water and free fatty acids. CaO based catalyst is one of the most widely studied systems for biodiesel production because of its high activity, low production cost and availability. Moreover, calcium oxide is a high basic catalyst, noncorrosive and can be synthesized from waste materials consisting of  $\text{CaCO}_3$ , such as eggshells, mollusk and cockle.

In the present study, eggshell waste was used as a heterogeneous catalyst for the transesterification of waste vegetable oil (WVO).

## MATERIALS AND METHODS

The catalyst was derived from eggshell and characterized by scanning electron microscope (SEM), Fourier transforms infrared (FTIR) spectrophotometer and X-ray fluorescence (XRF). The effect of methanol to oil molar ratio, catalyst concentration, temperature and time on the yield of biodiesel was investigated.

### Materials

Waste vegetable oil and Chicken Eggshells were obtained as wastes from ASCL Staff Restaurant Ajaokuta, in Kogi State. The chemicals and reagents used were bought from Luco Scientific Company Limited, Lokoja. The physicochemical properties of the WVO are shown in table 1. The chemicals used were methanol (99.5%), ethanol (99.5%), sulphuric acid, potassium hydroxide pellets and benzene (99.5%). The chemicals were of analytical grade and supplied by Pyrex – IG Scientific Company, Benin City, Nigeria.

## METHODS

### Preparation of Catalyst from the Waste Eggshells

The waste eggshells were washed with tap water to remove impurities from their surfaces. The washed eggshells were thereafter

sun-dried for three days. The dried eggshells were crushed to fine powder particles using a wooden mortar and pestle. Calcination of the particles was carried out using a muffle furnace for 5 h and the temperature was set to  $900^\circ\text{C}$ . The hot calcined samples were removed from the muffle furnace before the temperature of the furnace could reach room temperature and immediately placed inside a desiccator to cool. The samples were removed from the desiccator and kept in an airtight container to prevent reaction with moisture.

### Characterization of the Catalyst obtained from the Eggshells

The chemical composition of the catalyst was analysed using an X-ray fluorescence spectrometer (PANalyticalMiniPal 4) with an in-built XRD. The surface morphology of the catalyst derived from eggshells was observed by a scanning electron microscope (SEM). The SEM images were observed using PhenomProx working at an accelerated voltage of 15KV and beam size of 4. Micrographs were observed at 50 and  $200\mu\text{m}$ . The functional groups present in the catalyst were analysed using a Fourier transform infrared (FTIR) spectrophotometer within the wavelength region of 650 to  $4000\text{ cm}^{-1}$ .

## BIODIESEL PREPARATION

### Pre-treatment of the Waste Vegetable Oil (Esterification of WVO)

The FFA content of the WVO as shown in Table 1 was 9.0%. Hence, esterification of the WVO was carried out to reduce the FFA content to less than 1%. 182g of waste vegetable oil was measured in a glass reactor with methanol to oil molar ratio of 7:1, catalyst ( $\text{H}_2\text{SO}_4$ ) concentration of 1 wt.%, reaction time was fixed for 2 h at a temperature of  $600^\circ\text{C}$  (Alamu, 2007 a,b,c,d). The reaction was carried out in a 1 litre round bottom flask that was placed on a constant temperature magnetic stirrer (Model No. HJ – 3D). At the end of the reaction, the product was allowed to separate in a 1-litre separating funnel overnight. The upper oil phase layer after separation was recovered by tapping off the lower layer. The esterified WVO was analyzed for FFA content. The esterification was repeated till

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the FFA content fell below 1%. Before transesterification, all batches of treated (esterified) oil (FFA < 1%) were mixed to ensure a constant quality of raw material throughout the experiment. The acid value of the mixed esterified oil was determined and noted.

### **Transesterification Reaction**

Transesterification reaction was carried out in a 250 ml conical flask using the temperature-controlled magnetic stirrer. In a typical experimental run, 50 g of the esterified oil was measured into the conical flask and pre-heated to the desired temperature using the magnetic stirrer. The effects of process variables of methanol/oil molar ratio (4:1 to 14:1), catalyst loading (1 to 7wt. %), reaction temperature (30 to 90°C), reaction time (20min. to 140min), and reusability of catalyst (1 to 4 times) on the conversion to FAME were studied. A known weight of calcined eggshell (catalyst) was dissolved in the methanol while stirring gently at room temperature to allow for complete dissolution with minimal methanol evaporation. The methoxide mixture (calcined eggshell + methanol) was thereafter added to the preheated oil and the stirrer and temperature control were turned on to start the reaction. After the reaction, the mixture was separated from the catalyst by decantation and filtration. The mixture was later transferred to a separating funnel and allowed to settle overnight, for ensuring result it can be left

for 2 days. After the 24hrs period, the reaction result was in two distinct liquid phases; the first was the Waste Vegetable Oil Methyl Ester (WVOME) or the biodiesel on the top and the second was the denser phase of glycerol. Methyl ester phase was applied to a separation process (for better results the methyl ester was pipette from the top) and showered by cold or warm deionized water several times until becomes clean to remove any traces of methanol, glycerol, catalyst and water in methyl ester. The washed biodiesel was thereafter placed in an oven and heated to 80°C to remove water that might still be present in the biodiesel. The drying lasted for about 1 hour but not so specific because it continued so long as traces of water droplets were noticed at the lower layer of the biodiesel. Finally, the dried biodiesel from WVO was weighted for yield calculations and the ester contents were analyzed for evaluation of product purity. The biodiesel yield was obtained using equation (1)

$$\text{Biodiesel yield (\%)} = \frac{\text{Mass of biodiesel (g)}}{50\text{g of treated WVO}} \times 100 \quad (1)$$

## **RESULTS AND DISCUSSION**

### **Characterization of WVO and Catalyst**

Waste vegetable oil (WVO) was characterized and the observed physicochemical properties are shown in Table 1.

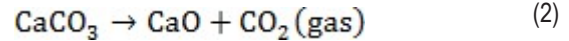
Table 1: Physicochemical properties of the waste vegetable oil

<b>Property (Unit)</b>	<b>Value</b>
Density at 25°C (kg/m <sup>3</sup> )	890
Viscosity at 25°C (mm <sup>2</sup> /s)	9.8
Iodine Value (I <sub>2</sub> /100g)	108.4
FFA Content (%)	9.0
Peroxide Value (meq kg-1)	16.4
Water content (wt%)	0.2
<b>Fatty acid Composition</b>	<b>(Mass%)</b>
Octanoic acid C8:0	1.618
n-Decanoic acid C10:0	7.407
Dodecanoic acid C12:0	50.454
Tetradecanoic acid C14:0	17.865
n-Hexadecanoic acid C16:0	9.878
Oleic acid C18:1	12.778

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The chemical composition of the catalyst derived from the eggshell is shown in table 2. The catalyst contained a very high composition of calcium oxide (95.558%) followed by SiO and Fe<sub>2</sub>O<sub>3</sub>, some elements were also detected in trace amounts. The high composition of CaO in the calcined eggshell is

a result of the fact that during calcination at high temperature, calcium carbonate present in the eggshell (Lacaet *al.*, 2017) is decomposed to form CaO and CO<sub>2</sub> according to the reaction equation shown in Eq.(2).

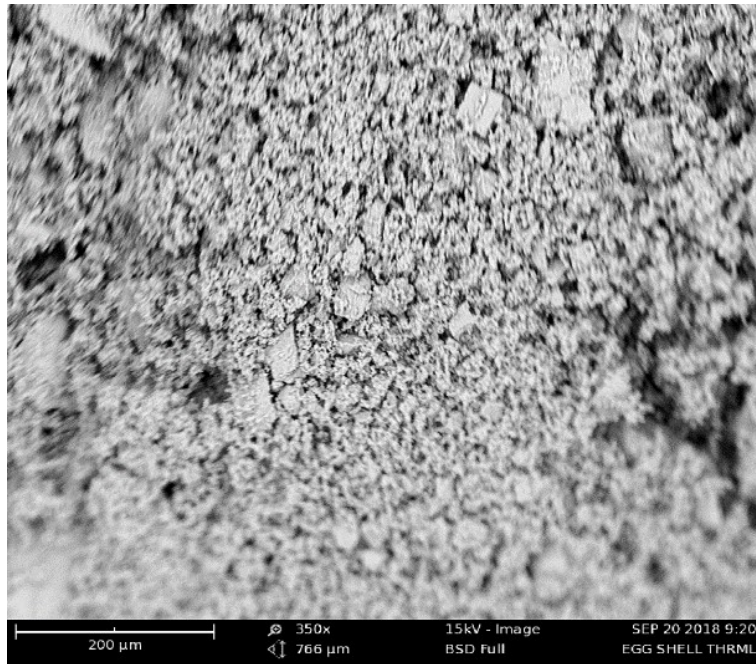


**Table 2:** Chemical composition of catalyst derived from eggshell

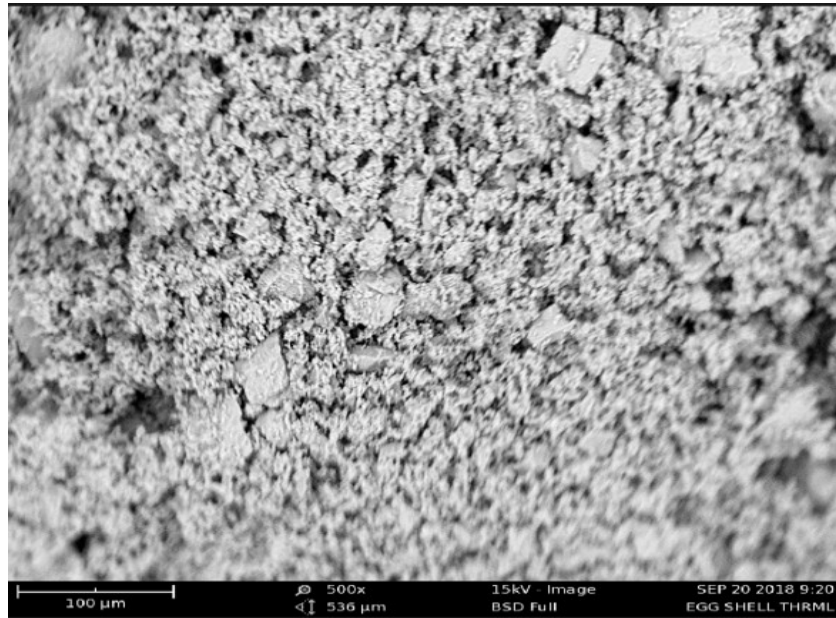
Major Oxides	Compositions (%)
CaO	95.558
Fe <sub>2</sub> O <sub>3</sub>	0.601
Cr <sub>2</sub> O <sub>3</sub>	0.202
Al <sub>2</sub> O <sub>3</sub>	0.365
SiO <sub>2</sub>	1.736
MgO	0.487
P <sub>2</sub> O <sub>5</sub>	0.296
Trace elements	0.756

The morphology of the catalyst derived from the eggshell was examined by observing the SEM images of the calcined

eggshells at different magnifications as shown in Figure 1.

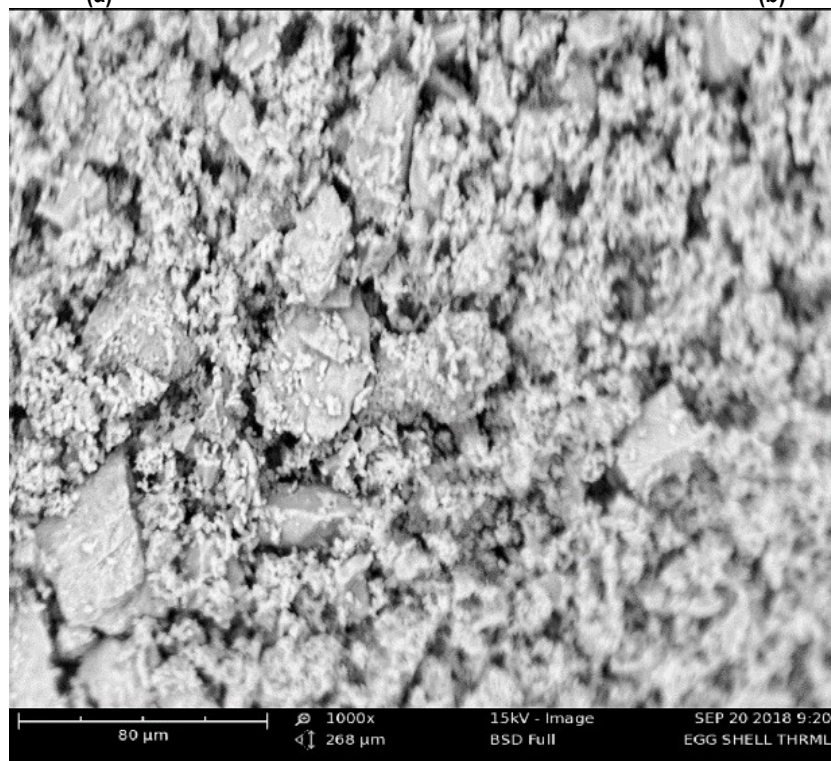


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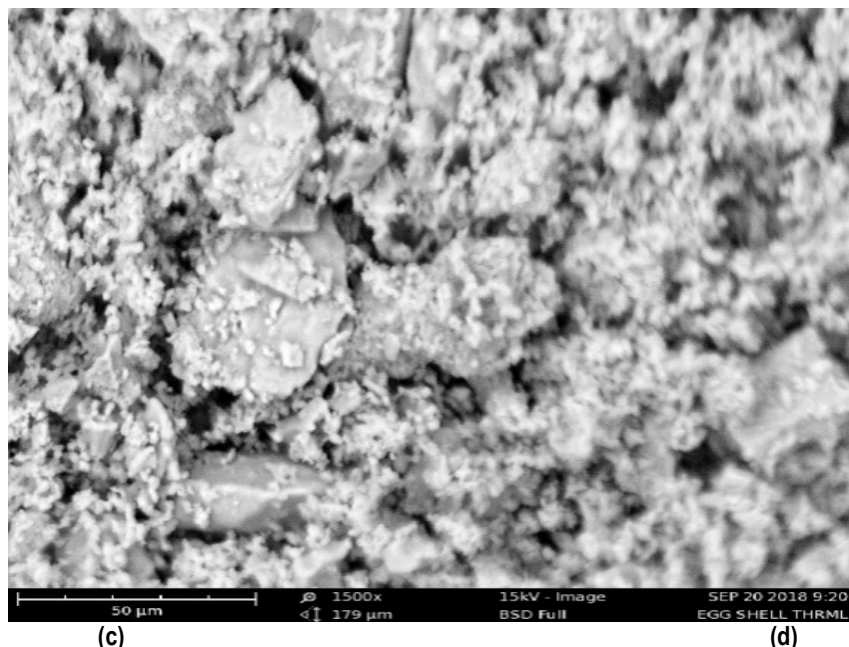


(a)

(b)



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**Figure 1:** SEM Image for calcined eggshell ash at 900°C (a) 200μm at 350 x (b) 100μm at 500 x (c) 80μm at 1000 x (d) 50μm at 1000x

The shape of the particles was almost regular as can be seen from the Figures. 4.1a, b, c and d similar observations have been reported by Yin *et al.* (2016) and Zeng *et al.* (2015). The FTIR spectrum of the catalyst derived from the calcined eggshells is shown in Figure 2. FTIR spectrum (4000-650 $\text{cm}^{-1}$ ) of the heterogeneous catalyst (calcined eggshell) (Fig 2) reveals major functional group absorption bands, notably for hydroxyl, and other groups. The spectrum showed distinct characteristic peaks at 3697.50, 3641.60, 2079.90, 1982.90, 1796.60, 1651.20, 1420.10, 872.20 and 711.90  $\text{cm}^{-1}$ . The broadband peaking at 1420.10 $\text{cm}^{-1}$  is assigned

to asymmetric stretching of OH bond while the peak at 3641.60 $\text{cm}^{-1}$  represents O-H stretching vibration for hydrogen-bonded OH groups. The large band area suggests a high concentration of OH groups from  $\text{Ca}(\text{OH})_2$  which is formed because CaO rapidly absorb moisture when exposed to the environment (Ho *et al.*, 2014). Other well-defined peaks are the 2079.90, 1982.90, 1796.60 and 1651.20  $\text{cm}^{-1}$  are attributed to the carbonyl functional group which occurs due to the re-carbonation of the CaO with  $\text{CO}_2$  in air. Also peaks 1095.80 and 711.90 $\text{cm}^{-1}$  are attributed to ester and C-O present respectively.

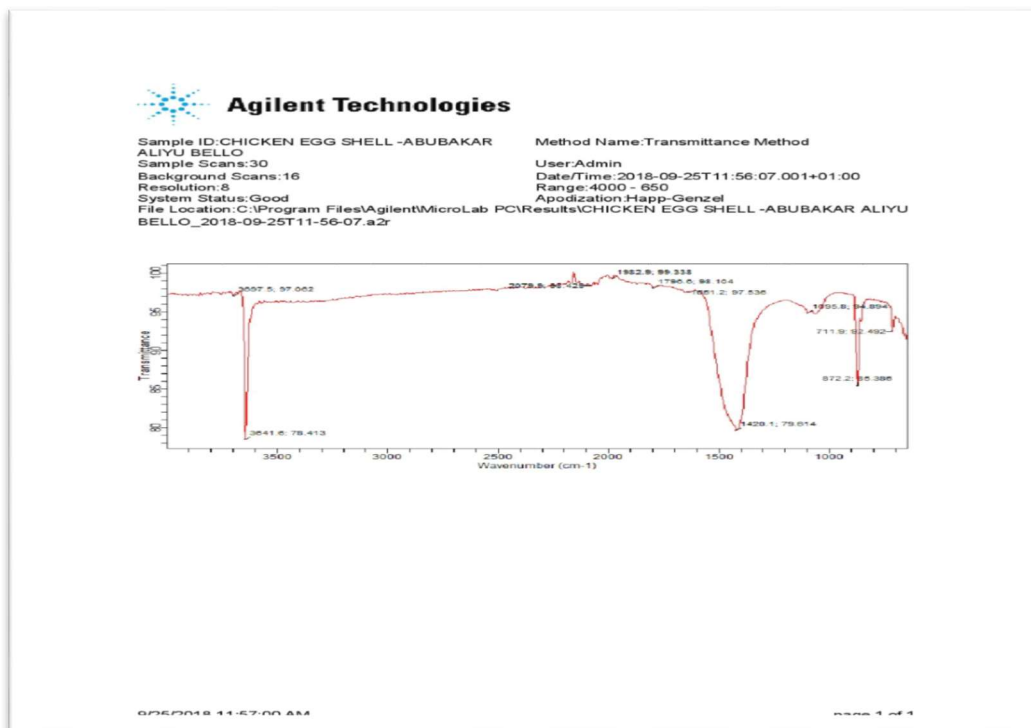


Figure 2. FTIR spectrum of the catalyst derived from calcined eggshell **Effect of Catalyst Concentration**

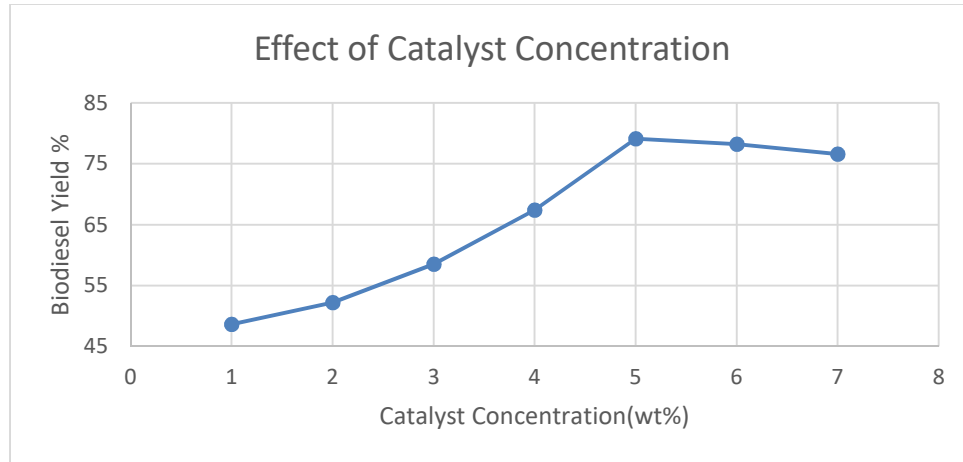
**Table 3:** Shows the Effect of Catalyst Concentration on Biodiesel Yield during the Experiment

OIL (g)	Catalyst Conc. (wt. %)	Reaction Temperature (°C)	Methanol to Oil Molar Ratio	Reaction Time (Minutes)	Yield (%)
50	1	60	6:1	60	48.6
50	2	60	6:1	60	52.2
50	3	60	6:1	60	58.5
50	4	60	6:1	60	67.4
50	5	60	6:1	60	79.1
50	6	60	6:1	60	78.2
50	7	60	6:1	60	76.6

The effect of Eggshell ash concentration was studied in the range of (1.0 – 7.0%w/v of oil), the reaction temperature, methanol to molar oil ratio and reaction time were kept constant at 60°C, 6:1 and 60mins, respectively. The results for different amounts of catalyst are shown in Table 3 above. Low yield (48.6-67.4%) was obtained with low catalyst concentration (1 – 4wt %), which was insufficient to catalyse the reaction to completion in the formation of methyl ester. This

observation corroborates with Lee *et al.* (2009) reported, that heterogeneous catalyst in biodiesel production should range from 3-10wt% for higher yield. Higher biodiesel yield (79.1%) was observed at an optimum point of 5.0 wt% catalyst concentration. Beyond this range (6 -7 wt %), a decrease in biodiesel yield was observed. This observation can be attributed to the availability of active sites on the catalyst for transesterification reaction (Birla *et al.*, 2012).

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**Figure 3:** Effect of catalyst concentration

From the regression analysis, R Square value of 0.947 was observed, indicating a very sound fit while the model equation for the yield of biodiesel was given as;

$$\text{Yield} = 43.42857143 + 5.592857143 \text{ Catalyst concentration} \quad (3)$$

**Effect of Reaction Temperature**

Table 4: Shows the Effect of Reaction Temperatures on Biodiesel Yield during the Experiment

Oil (g)	Catalyst (Wt %)	Temperature (°C)	Methanol to Oil Molar Ratio	Reaction Time (Minutes)	Biodiesel Yield (%)
50	5	30	6:1	60	36.39
50	5	40	6:1	60	45.48
50	5	50	6:1	60	55.67
50	5	60	6:1	60	84.59
50	5	70	6:1	60	76.82
50	5	80	6:1	60	57.52
50	5	90	6:1	60	46.57

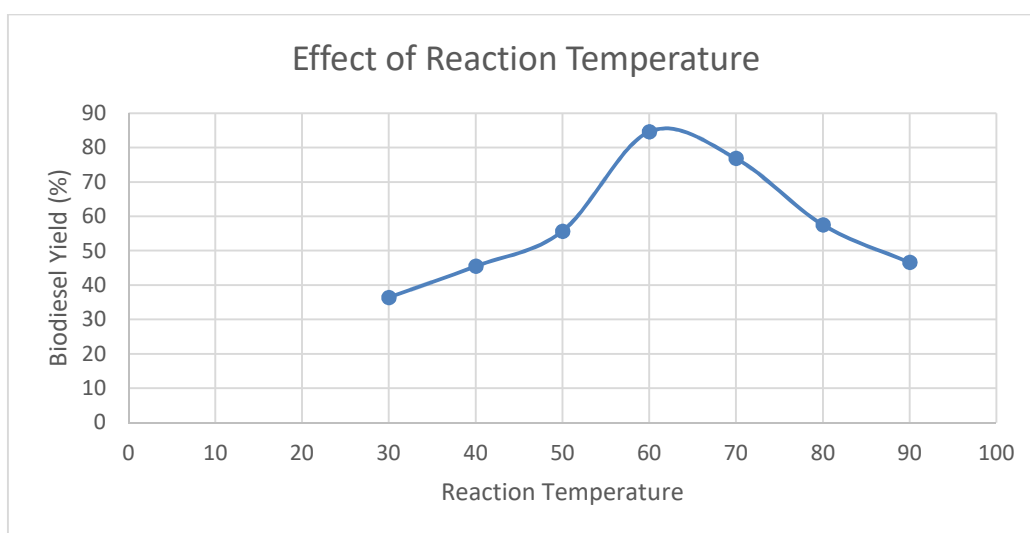
The temperature had an impact on the mass transfer of the reaction: the optimum temperature was found to be 60°C with a biodiesel yield of 84.59% under optimal conditions (catalyst loading 5.0 wt%, methanol to oil molar ratio 6:1 and reaction time of 60mins). An increase in the reaction temperature to 60°C was observed to bring about an increase in the reaction rate due to higher energy input and reduced mass transfer resistance (Birla *et al.*, 2012). Increasing the temperature beyond 60°C resulted in a decrease in the production because any reaction occurring beyond the methanol boiling point (65°C) resulted in its intense continuous vaporisation. Hence it remained in the gas phase in the reflux, causing a reduction of methanol in the reaction media (Birla *et al.*, 2012; Tariq *et al.*, 2012).

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**Table 5:** Shows the Effect of Methanol to oil molar ratio on Biodiesel Yield during the Experiment.

Oil (g)	Catalyst (wt. %)	Reaction Temperature (°C)	Methanol to Oil Molar Ratio	Reaction Time (Minutes)	Biodiesel Yield (%)
50	5	60	4:1	60	55.3
50	5	60	6:1	60	58.2
50	5	60	8:1	60	86.61
50	5	60	10:1	60	88.63
50	5	60	12:1	60	89.22
50	5	60	14:1	60	93.14



**Figure 4:** Effect of Reaction Temperature

From the regression analysis, R Square value of 0.828 was observed; this infers a very good fit. The model equation for biodiesel yield was given as;

$$\text{Yield} = 41.34071429 + 0.270607143 \text{reaction temperature} \quad (4)$$

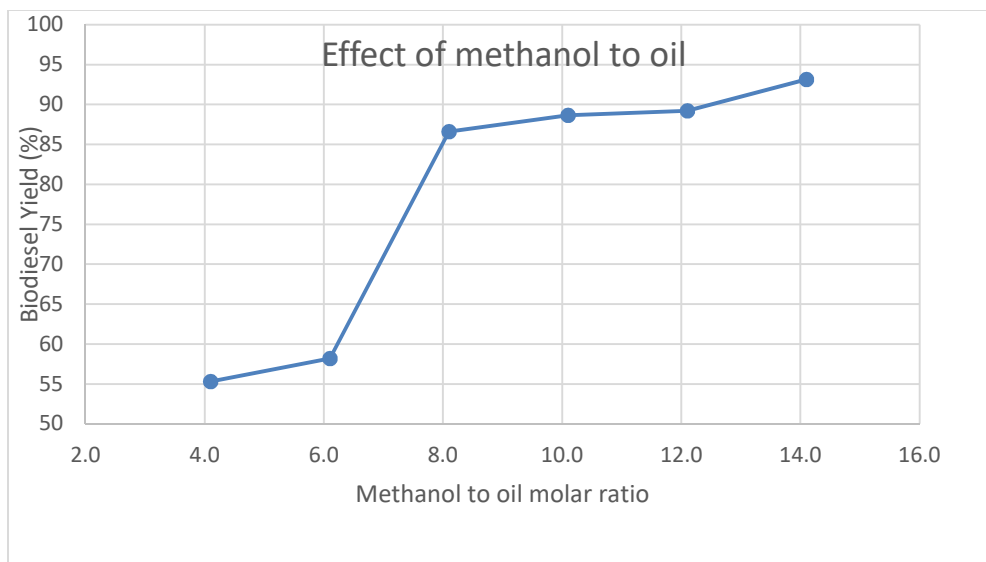
**Effect of Methanol to oil molar ratio**

The alcohol to oil molar ratio is one of the most important factors that can affect the yield of esters. The stoichiometry of the transesterification reaction requires a 3:1 molar ratio to yield 3 moles of ester and 1 mole of glycerol, but most researchers have found that excess alcohol was required to drive the reaction close to completion. In this research, methanol

was used as the preferred alcohol. The effect of methanol to oil ratio in the range of 4:1 to 14:1 was investigated, keeping Catalyst Concentration, Reaction Temperature and Reaction Time constant.

The optimum biodiesel yield was observed at Methanol to oil molar ratio of 14:1. It can be observed from Figure 5 that a decrease in the molar ratio below the optimum point (14:1) had a negative impact on the biodiesel yield. The yield decreased drastically to 55.3%, at methanol to oil molar ratio of 4:1. This is due to the dilution effect of insufficient methanol and a higher molar ratio will result in dilution effect and subsequent interference between the high molar ratio of methanol/oil and the catalyst (Tang *et al.*, 2013).

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**Figure 5:** Effect of methanol to oil ratio on the yield of biodiesel

A regression analysis revealed an R Square value of 0.937, indicating a profound fit while the model equation for the yield of biodiesel was given as;

$$\text{Yield} = 41.96638095 + 4.061142857 \text{methanol to molar oil ratio} \quad (5)$$

**Effect of Reaction Time**

**Table 6:** Shows the Effect of Reaction Time on Biodiesel Yield during the Experiment

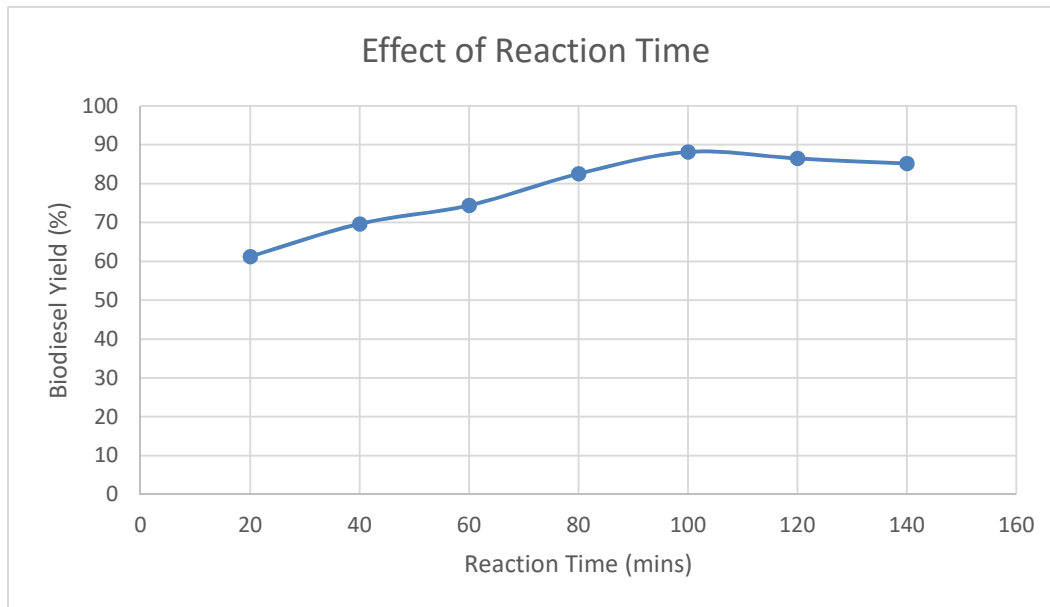
Oil (g)	Catalyst (wt. %)	Temperature (°C)	Methanol to Oil Molar Ratio	Reaction Time (Minutes)	Biodiesel Yield (%)
50	5	60	6:1	20	61.22
50	5	60	6:1	40	69.64
50	5	60	6:1	60	74.39
50	5	60	6:1	80	82.51
50	5	60	6:1	100	88.15
50	5	60	6:1	120	86.44
50	5	60	6:1	140	85.12

The conversion rate increases with reaction time. The reaction was very slow during the first minute due to the mixing and dispersion of methanol into vegetable oil. In this work, the effect of reaction time from 20 min to 140 min on the reaction yield was investigated, keeping catalyst concentration, reaction temperature and methanol to oil molar ratio constant.

Reaction time is one of the key parameters during the transesterification reaction for biodiesel production carried out in a glass

reactor using a calcined eggshell catalyst. Figure 6 shows an increase in the yield with time from 30 to 100 minutes with a catalyst amount of 5wt% relative to methanol to oil molar ratio of 6:1. The maximum yield of 88.15 % was obtained in 100 minutes. In the initial stages of the transesterification reaction, production of biodiesel was rapid, and the rate diminished and finally got to a steady state at a reaction time of 120minutes and beyond.

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**Figure 6:** Effect of reaction time on biodiesel

From the regression analysis, the R Square value of 0.970 was observed; this infers a very good fit. The model equation for biodiesel yield was given as;

$$\text{Yield} = 61.20142857 + 0.212607143 \text{ reaction time} \quad (6)$$

From the biodiesel produced, the yield obtained was 94.58% under optimum conditions. It was observed from the work of (Musa; 2015), the biodiesel produced has a yield of 98% using a solid catalyst in the form of powered sodium hydroxide and fibre on Straight Vegetable Oil. The process of esterification, before transesterification as in the work of (Abdulrasheed; 2015), reduce the free fatty acid (FFA) of the parent feedstock which in turn

improved the yield of biodiesel. In both cases observed, there is a reduction in the volume of glycerol produced. Improving the yield of biodiesel produced from any production system is very important as seen above. Other factors that might improve the yield are; energy and time used for the production.

#### Physicochemical Properties of WVO Biodiesel

The physicochemical properties of the biodiesel produced under optimal reaction conditions were Analyzed and compared with the ASTM D6751 biodiesel standard shown in table 7. It can be seen that the major fuel properties such as density and flash point are within the biodiesel standard

**Table 7:** Physico-chemical properties of WVO biodiesel.

Property	Unit	Value	Test Method
	Biodiesel Standard		ASTM D 6751
Viscosity at 25 °C	mm <sup>2</sup> /s	4.64	D445
Density	kg/m <sup>3</sup>	884	D4052
			1.9 – 6.0
			0.860 - 0.900

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Acid value	mg KOH/g	0.62	D664	< 0.80
Flashpoint	°C	162	D93	130
Cloud point	°C	8	D2500	min
Iodine value	gI <sub>2</sub> /100g	72.83	D664 11	-
Moisture content	(%)	0.02	-	<0.05

## CONCLUSION

Calcined hen eggshell contains 95.558% Calcium Oxide which was observed to have excellent activity in mediating the transesterification of waste vegetable oil containing 9.0% of FFA to biodiesel production. The transesterification reaction was observed to be optimal at a catalyst concentration of 5.0 wt. %, reaction temperature of 60 °C, the methanol-to-oil molar ratio of 14:1, and reaction time of 100 min, which gave a biodiesel yield of 94.58 %. The quality of the biodiesel produced under this optimum condition was found to be in the range of the ASTM standard for biodiesel.

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