

## BIODIESEL FROM USED COOKING OIL VIA CALCIUM OXIDE LOADED ACTIVATED CARBON CATALYZED TRANSESTRIFICATION REACTION

<sup>1</sup>Abdulmumin Malam Usman

Tel: 08060345532

Email: hwardamu01@gmail.com

<sup>2</sup>HADIZA JIBRIL ABDULLAHI

07032965854

hadizaabdullahijibril@yahoo.com

<sup>3</sup>SHUAIBU O. SALIHU

08036008819

<sup>1,2,3</sup> Department of Chemistry

School of Secondary Education (Sciences)

Federal College of Education (Technical) Bichi

<sup>4</sup>BASHIR ABDULLAHI SA'ID

Department of pure and Industrial chemistry

Bayero University, Kano

### Abstract

*In this study, biodiesel was produced from the used cooking oil using a two-step catalyzed transesterification reaction. A calcined Calcium oxide activated carbon (CaO/AC) impregnation method serve as the composite catalyst. The CaO/AC and corresponding fatty acid methyl ester (FAME) were characterized by FTIR and XRD and for identification of functional groups, crystal structure of catalyst and basicity nature of the catalyst. Esterification and transesterification processes were optimized using response surface methodology (RSM) based on central composite design (CCD). Under optimal conditions of methanol to oil ratio for 20:1 at reaction temperature of 333K, catalyst loading of 0.3 mg and reaction time of 40 min. The optimal conversion of 92.7% was recorded for the transesterification of used cooking oil and CaO/AC as catalyst. The Physico-chemical analyses carried out on the produced biodiesel gave strong indication that it can serve as an alternative feedstock, having recorded a comparable flash point of 102 °C to American Society of Testing Materials and European Committee for Standardization standards. The acid value of the used cooking oil was (4.09), specific gravity was (0.9711 g/cm<sup>3</sup>) kinematic viscosity was found to be (9.5mm/s) and the <sup>PH</sup> was also found to be (8.52). These values indicates that the used cooking oil can be very useful of high yield with zero effect on the environment.*

Key Words: Cooking oil, Biodiesel, Transesterification, calcium oxide, Activated carbon.

### Introduction

Presently, our energy sectors rely on petroleum resources and thus there is urgent need to consider the current status of world's oil reserves and increasing environmental effects of these fuels (Dawodu *et al.*, 2014). Since fossil fuels increase emissions of greenhouse gas and bring about global warming the use of alternative resources such as biofuels are of paramount importance (Ozbay *et al.*, 2008). Biodiesel production interest is increasingly growing worldwide for it is the only renewable energy fuel up-to-date which could substitute fossil fuels in compression ignition engines (Tabatabaei, Ibrahim & Gaya, 2015).

Biodiesel (BD) is a mono alkyl esters comprising of long chain fatty acids that can be derived from vegetable oil, animal fats, and microbial oil (Sankaran & Show, 2016). The properties of BD that make it attractive substituted to fossil fuel include direct used in current diesel engines without any modifications, can blended with petroleum diesel in any proportion or directly used as B100, high cetane number of oxygen than petroleum diesel that is to say clean combustion and reduced emission, low or almost zero sulphur content and therefore no CO<sub>2</sub> emission and higher flash points making it safe for transport and handling (Sankaran & Show, 2016).

The BD production through homogeneous catalysis is too cost because of the soap formation, non reusability of the catalyst, handling and separation problems. Consequently, heterogeneous catalysis was applied at a commercial level for its vital application, such as easy separation from the reaction mixture, selectivity and appreciable and appreciable catalytic activity in reuse (Reddy *et al.*, 2015). Nevertheless, heterogeneous analysis brings up some problems, such the mass transfer diffusional resistance which decreases the reaction rate to a great extent compared to homogeneous catalysis. To tackle this problem, catalyst support is required that offers higher surface area and large number of the active sites in the methanolysis of oils or fats to form their corresponding fatty acid methyl ester. Activated carbon

supported metal oxides has been used in many chemical processes. This is owing to its chemical inertness, high temperature stability, tunable texture and surface properties (Tsonchiva, 2018).

There are many processes to produce BD from vegetable oils or animal fats such as direct use or blending, pyrolysis, micro-emulsion and transesterification. Among these, the most common process is transesterification of triglyceride by the use of alcohol such as methanol fatty acids methyl ester (FAME). Transesterification can be carried out by either a catalyst or non catalytic process, base catalysts, acid catalysts and enzymes have been commonly used in the BD production at all scales. CaO – based catalyst have mostly been used in the form of neat, supported, loaded and mixture. This is due to the oxygen anion present on the CaO surface (Marinkovic *et al.*, 2018).

### **Description of biodiesel**

Biodiesel is a light to dark yellow liquid, it is practically immiscible with water, it had high boiling point and low vapor pressure. Typical methyl ester (biodiesel) has a flash point of 150°C (300°F), making rather none – flammable. Biodiesel has a density of about 0.88kg/m<sup>3</sup> and when not contaminated with starting material can be regarded as non – toxic (Adelodun *et al.*, 2016).

Biodiesel and petroleum diesel vastly differ in their chemical composition. These differences give biodiesel different physical and chemical properties. The composition and properties of biodiesel depend on the feed stock used on the manufacturing process (Morrison & Boyd, 2008).

### **Product quality of biodiesel**

Biodiesel is a better fuel than petro diesel and has been known to break down deposit of residue in the fuel lines of vehicle that has previously been run on petrol fuel filter may clog with particular test if a guide transition of pure biodiesel is made, as biodiesel cleans the engine in the process. It is therefore, recommended to change the filter within (600 – 800 miles after first switching to a biodiesel blend (Kessler, 1985).

Prior to use as a commercial fuel the produced biodiesel must be analyzed using sophisticated equipment to ensure it meet any required specifications. Biodiesel's commercial fuel quality is measured by the American standard for testing materials (ASTM) standard. The standard ensures that biodiesel is pure and the following important factors in the fuel production process are satisfied.

- a) Reaction completion
- b) Removal of alcohol
- c) Removal of glycerine
- d) Absence of free fatty acids
- e) Low sulphur content (Kessler, 1985).

As such, biodiesel can be defined as the mono alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, for use in compression ignition engine (Pahl, 2005).

### **Statement of the Problem**

A national energy and renewable energy master plan drafted by energy commission of Nigeria encouraged biodiesel production, which covers all aspects of plantation, procurement of seed, extraction of oil, and other means. This is as a result of the huge environmental damage due to the excessive use of fossil fuel. As at 2018, diesel forms about 14% of the energy consumed in the form of hydrocarbon fuels, and its daily demand was found to be increasing in millions of litres (Alang, 2018). Biodiesel production can completely replace the current use of fossil fuels. The production of biodiesel will also boost the rural economy and positive impact to more than million lives in the area and generate thousands of jobs for both rural and urban areas (Morrison & Boyd, 2008).

Various catalysts were used for the methanolysis of Waste Cooking Oil including Acid catalyst which is costly, time consuming and corrosive. Untreated CaO showed slower reaction rate (Hassan & Nageswara, 2018).

### **Objectives of the study**

The objective of this study is to assess biodiesel from used cooking oil via calcium oxide loaded activated carbon catalyzed transesterification reaction.

### **Materials and Method**

All chemical were of analytical grade obtained from chemistry department, Bayero University Kano. All glass waves and apparatus used in this research were in good condition and washed thoroughly with deionized water and dried in an oven prior to usage.

### Apparatus

Conical flask, beaker, Burette, Measuring cylinder, retort stand, magnetic hot plate, thermometer and digital scale. All apparatus used in this research study were obtained in BUK departmental store and washed thoroughly with deionized water and dried in an oven prior to usage.

### Catalyst Preparation

The cleaned activated carbon was immersed in calcium oxide solutions of different calcium oxide loaded activated carbon was prepared by wet impregnation method. Concentrations (25 to 100 %). The impregnated samples were stirred for 6h at 600 rpm. After impregnation the mixture was filtered and oven – dried at 105 °C until a constant weight was obtained. The dried catalyst was calcined at 450°C for two hours. The resulting whitish black substance was labeled as the Calcium oxide loaded activated carbon.

### Esterification process

The waste cooking was heated to 60°C while stirred mechanically at 800 rpm under atmospheric conditions to homogenize the oil. The reaction was carried out at 250 ml three – necked round bottomed flask attached with reflux condenser and thermometer and place in water bath with temperature controller, as stannous chloride dihydrate,  $[(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) \cdot (0.1\text{g} - 0.5\text{g})]$  in 24.05g ethanol was heated to 60°C and added to the reaction flask containing pre-heated oil and stirred continuously for 1hour (Kombe *et al.*, 2015). The resulting substance is the ester of the waste cooking oil.

### Esterification Experimental Design

A three-level-five-factors CCD was employed in this optimization study, requiring 20 experiments. Reaction temperature (K), catalyst loading and reaction time (T) were the independent variables selected to be optimized for the pretreatment of WCO. The coded and uncoded (actual) levels of the independent variables are given in table 1a and 1b. Two replications were carried out for all design point (factorial and axial) except the center points (0,0) and the experiments were carried out in randomized order (Tiwari *et al.*, 2007).

Table 1a: Levels and Codes of Experimental Variables for Pretreatment of WCO.

Experimental Variables	Notation	Coded levels				
		Very low (-α)	Low (-1)	Central (0)	High (+1)	very High (α)
Reaction Temperature T(k)	A	308	318	323	328	333
Catalyst mass (mg)	B	0.1	0.2	0.3	0.4	0.5
Reaction time (min)	C	20	30	40	50	60

Table 1b. Experimental design for esterification from dx6 software (6.0.6)

Run	Reaction temperature (K)	Catalyst loading (mg)	Reaction Time (min)
1	0 (323)	0 (0.3)	-α (20)
2	0(323)	0 (0.3)	α (60)
3	0 (323)	0 (0.3)	0 (40)
4	-1 (318)	1 (0.4)	-1 (30)
5	0 (323)	0 (0.3)	0 (40)
6	0 (323)	-α (0.1)	0 (40)
7	0 (323)	0 (0.3)	0 (40)
8	-1 (318)	-1 (0.2)	1 (50)

Run	Reaction temperature (K)	Catalyst loading (mg)	Reaction Time (min)
9	- $\alpha$ (308)	0 (0.3)	0 (40)
10	0 (323)	0 (0.3)	0 (40)
11	0 (323)	0 (0.3)	0 (40)
12	1 (328)	-1 (0.2)	1 (50)
13	1 (328)	1 (0.4)	-1 (30)
14	1 (328)	1 (0.4)	1 (50)
15	-1 (318)	-1 (0.2)	-1 (30)
16	0 (323)	0 (0.3)	0 (40)
17	-1 (318)	1 (0.4)	1 (50)
18	$\alpha$ (333)	0 (0.3)	0 (40)
19	0 (323)	$\alpha$ (0.5)	0 (40)
20	1 (328)	-1 (0.2)	-1 (30)

### Transesterification process

A precise quantity of the pretreated oil was measured and poured into a conical flask. The oil was pre heated to 50 °c. A specific amount of calcium oxide loaded activated carbon catalyst was weighted and dissolved in the required amount of methanol. The CaO/AC catalyst solution was then added to the pre-heated oil in a 250 ml three-necked round bottom flask and placed on a hot plate magnetic stirrer at a particular temperature. The reaction was kept overnight for proper settling of the biodiesel, followed by separation, washing and heating to evaporate the remaining water molecules in it. The percentage of the biodiesel yield was determined by the equation below: (Amini *et al.*, 2017).

$$\% \text{ yield} = \frac{\text{weight of sample yield}}{\text{weight of all}} \times 100 \dots \dots \dots (1)$$

### Experimental Design

To find the optimum conditions of the transesterification process, the experiments were designed by RSM and a five-level, three-variable central composite design (CCD) was employed. The independent variables considered are the reaction temperature (*A*), mass of catalyst (*B*) and reaction time (*C*). As the CCD is rotatable, there are five level of these variables and five codes (-1.682, -1, 0, +1, +1.682). These coded levels are displayed in Table 1. Other variables such as agitation speed, oxygen pressure and delivery volume were kept constant. The plan contained 8 factor points, 6 axial points and 6 center points. The total number of performed runs were 20 experiments as informed by formula  $N(\text{experiments}) = 2^n + 2n + 6$ , where *n* is the number of variables.

**Table 2a: Levels and Codes of Experimental Variable.**

Experimental Variables	Notation	Coded levels				
		Very low (- $\alpha$ )	Low (-1)	Central (0)	High (+1)	very High ( $\alpha$ )
Reaction Temperature T(k)	A	308	318	323	328	333
Catalyst mass (mg)	B	0.1	0.2	0.3	0.4	0.5
CH <sub>3</sub> OH : Oil (v/v)	C	10:1	11:1	12:1	13:1	14:1

**Table 2b. Experimental design for transesterification from dx6 software (6.0.6)**

Runs	Reaction Temperature T(K)	Catalyst loading C (mg)	CH <sub>3</sub> OH: oil M(v/v)
1	-1(325)	-1(0.18)	-1(10:1)
2	1(333)	-1(0.18)	-1(10:1)
3	-1(325)	1(0.26)	-1(10:1)

4	-1(325)	1(0.26)	-1(10:1)
5	1(333)	-1(0.18)	1(12.5:1)
6	-1(325)	-1(0.18)	1(12.5:1)
7	1(333)	1(0.26)	1(12.5:1)
8	-1(325)	1(0.26)	1(12.5:1)
9	1(333)	0(0.22)	0(11:1)
10	0(329)	-1(0.18)	0(11:1)
11	0(329)	-1(0.18)	0(11:1)
12	0(329)	1(0.26)	0(11:1)
13	0(329)	0(0.22)	-1(10:1)
14	0(329)	0(0.22)	1(12.5:1)
15	0(329)	0(0.22)	0(11:1)
16	0(329)	0(0.22)	0(11:1)
17	0(329)	0(0.22)	0(11:1)
18	0(329)	0(0.22)	0(11:1)
19	0(329)	0(0.22)	0(11:1)
20	0(329)	0(0.22)	0(11:1)

## Result and Discussion

### Results

**Table 3a: Biodiesel Yields for methyl esters of CaO/AC catalyst**

Runs	% Yield CaO / AC	Predicted values
1	78.90	76.62
2	80.72	80.66
3	83.36	79.39
4	82.27	82.95
5	80.80	78.57
6	84.60	87.03
7	86.63	85.05
8	92.70	93.24
9	72.83	82.59
10	92.20	88.25
11	81.32	83.25
12	83.40	87.64
13	80.95	86.38
14	91.65	92.39
15	90.02	88.73
16	90.41	88.73
17	91.01	88.73
18	90.70	88.73
19	91.70	88.73
20	90.90	88.73

**Table 3b: Result of Validation for CaO and CaO/AC and SnCl<sub>2</sub>.2H<sub>2</sub>O catalyst**

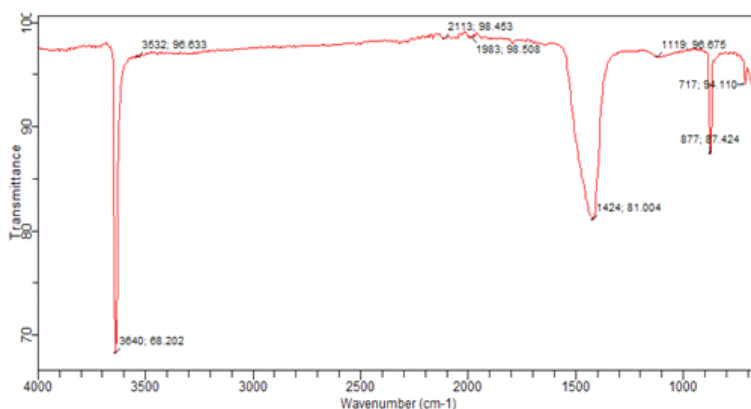
Experiment	Factor A: reaction Temperature (K)	Factor B: catalyst loading (mg)	Factor C: methanol to oil ratio	FAME conversion % actual Yield	FAME conversion % predicted Yield	Standard deviation
CaO	333	0.18	12.5:1	79.00	69.19	
1 <sup>st</sup>				79.02	-	
2 <sup>nd</sup>				78.93	-	

3 <sup>rd</sup>				79.23	-	
						0.1540
CaO/A C	333	0.26	12.5:1	92.70	93.24	
1 <sup>st</sup>				91.43		
2 <sup>nd</sup>				91.73		
3 <sup>rd</sup>				92.09		
						0.8846
	T(K)	C(mg)	t (min)	Experimenta l FFA	Predicted FFA	
SnCl <sub>2</sub> .2 H <sub>2</sub> O	333	0.30	40	0.895	1.09	
1 <sup>st</sup>				1.01		
2 <sup>nd</sup>				0.93		
3 <sup>rd</sup>				0.881		
						0.661

**Table 4a: Physical and chemical properties of WCO oil Biodiesel Compared to ASTM D6761**

S/N	Properties	Units	WCO	Biodiesel	ASTM D6761 STANDARD
1.	Moisture content	% wt	0.03	0.03	0.05 max
2.	Acid value	mgKOH/g	5.16	0.53	-
3.	FFA	mgKOH/g	2.58	0.265	-
4.	Saponification value	mgKOH/g	197.20	173.4	-
5.	Ester value	mgKOH/g	192.04	172.87	-
6.	Molecular weight	g/mol	876.40	-	-
7.	Kinematic viscosity	mm <sup>2</sup> /s	49.67	-	1.9 – 6.0
8.	Odour	-	Agreeable	Agreeable	-
9.	Colour	-	Yellow	Brown	-
10.	Boiling point	(°C)	125	-	-
11.	Specific gravity	-	0.94	0.8374	0.88

### Characterization



**Fig. 1a. FT – IR for CaO/AC**

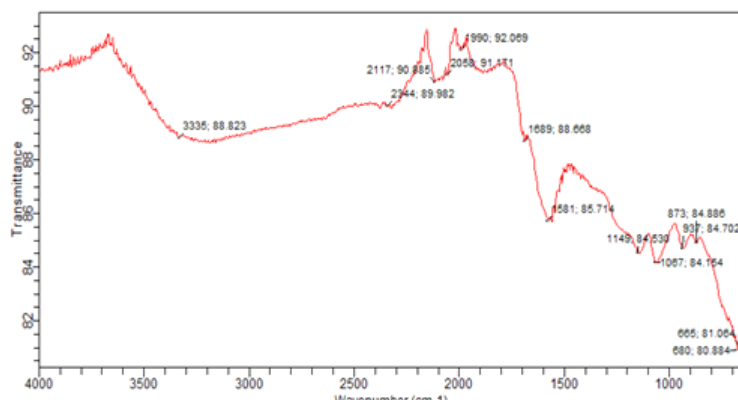


Fig. 1b. FT – IR for activated carbon

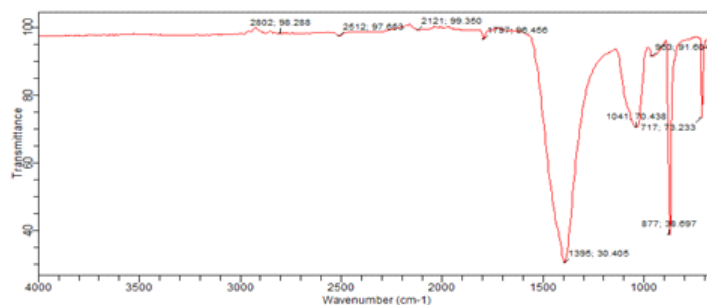


Fig. 1c. FT – IR for CaO

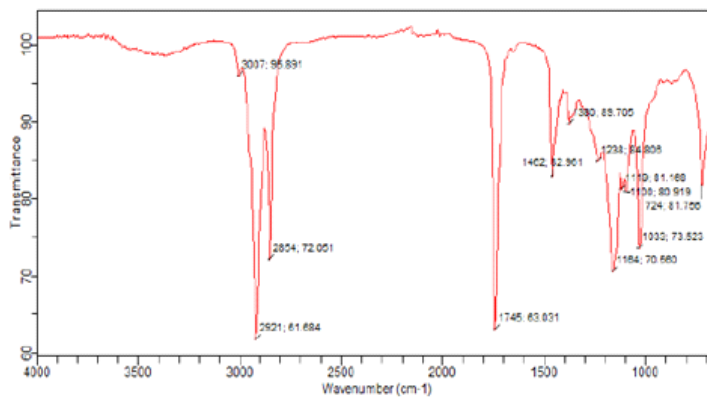


Fig. 1d. FT – IR for CaO/AC

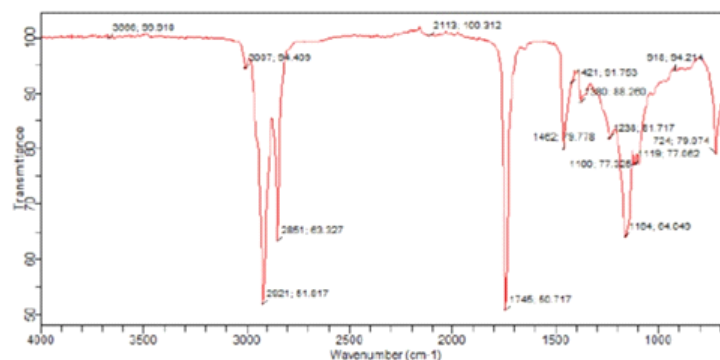


Fig. 1e. FT – IR for WCO

Table 3a. IR absorption data for WCO and biodiesel

S/N	WCO (cm <sup>-1</sup> )	CaO/AC biodiesel (cm <sup>-1</sup> )	Functional groups
1	2851 – 2921	2854 – 2921	C-H <sub>s</sub>
2	1745	1745	C=O in esters
3	1462	1462	C-H and O-H <sub>B</sub>
4	1119	1110	C- O in esters
5	-	-	C=C
6	724	724	C-O <sub>s</sub> , C-C <sub>B</sub>

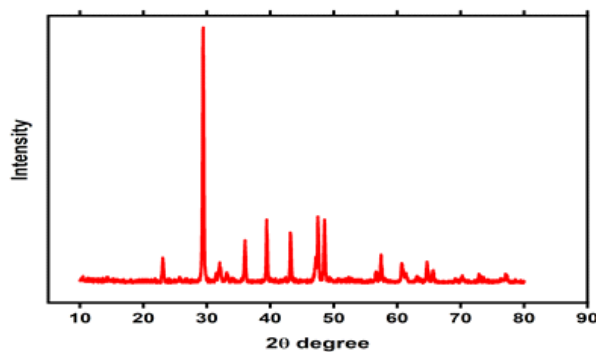


Fig. 2. XRD graph for CaO/AC

## Discussion

### Physicochemical properties of WCO and Biodiesel

The result of the physico-chemical characteristic of waste cooking oil and biodiesel, along with the standard ASTM D6751-02, are presented in table 1a. The major characteristic (moisture content, kinematic viscosity, acid value, free fatty acid and specific gravity) are in good agreement with the standard. From the table 1. the major properties of WCO oil were discussed as follows:

### Acid value and free fatty acid



This is the number of milligrams of KOH required to react with all the free fatty acids in one gram of oil. The higher the acid number, the higher the free fatty acid concentration in the biodiesel sample, leading to corrosion of engine components. This is usually a symptom of water in the fuel due to poor production or oxidative degradation. A significant level of free fatty acid in the fuel often results to soap formation with the attendant propensity to inhibit the transesterification reaction. The acid values determined for waste cooking oil and biodiesel were respectively 5.16 and 0.53 mg KOH/g. the acid value of WCO biodiesel corresponds to a free fatty acid level of 0.265 mgKOH/g FFA revealed that WCO Biodiesel falls in the range of internationally accepted standards. This is agreement with (Ojolo *et al.*, 2012).

#### **Saponification Value**

The saponification value obtained for WCO was 197.20. This high value show that the WCO biodiesel consist of mostly short chain fatty acid methyl esters. The magnitude of the saponification number varies inversely with the average chain length of the constituent fatty acids in the oily sample. The high saponification value for the precursor oil implies that the triglycerides of WCO have higher molecular weight of 876.40 fatty acids (saturated and unsaturated) and can be better suited for the manufacture of soaps, detergent and shampoo. The value of biodiesel was 172.87mg KOH/g which showed low tendency of soap formation. This is agreement with work (Ojolo *et al.*, 2012).

#### **Ester value of WCO oil and Biodiesel**

The closer the ester value of a biodiesel sample is to the saponification value, the better the quality because this implies that the free fatty acid content of the biodiesel is low. This is a desirable condition of biodiesel it is not detrimental to the operation of the ignition compression engine. The ester value for WCO oil was 192.04 mg KOH/g while that of biodiesel obtained in this study was 172.87 mg KOH/g. thus is agreement with (Ojolo *et al.*, 2012).

The fuel properties of this biodiesel are summarized in table 1b. Waste cooking oil biodiesel had comparable fuel properties with those of diesel and conforming to the latest standards for biodiesel. The basic properties were discussed as follows:

#### **Specific gravity**

The specific gravity of waste cooking oil was 0.94 kg/m<sup>3</sup> reduced to 0.8374 kg/L after transesterification. Hence the specific gravity of vegetable oil can be brought into acceptable ranges suitable for fuel purposes by transesterification. The specific gravity of WCO biodiesel was however lower than 0.880 kg/l for petro-diesel in agreement with work (Shote, Betiku & Asere, 2018). Blending of biodiesel with petro-diesel could equilibrate some of these properties. Such blends would be homogeneous given that their densities are quite close (Onanuga & Coker, 2013).

#### **Flash Point of WCO biodiesel**

The flash point of biodiesel refers to the temperature at which the fuel can ignite when exposed to a Heat source. The flash point is of relevance for safe handling, storage and transportation of fuel. The flash point of CaO/AC and CaO biodiesel were respectively 40 and 134°C. Transesterification greatly enhanced the volatility of the final product. The flash point of WCO biodiesel is however lower than of petroleum diesel (15°C) which was analyzed. This is agreement with (Alang, 2018). Hence blending is necessary to obtain a hybrid product that as more desirable characteristics.

#### **Kinematic Viscosity of WCO biodiesel**

The value of viscosity obtained for WCO oil and biodiesel were 49.67 mm<sup>2</sup>/s and 0.3226 mm<sup>2</sup>/s of 40°C respectively agreed with agreement with work (Shote *et al.*, 2018). The transesterification reaction drastically modifies the viscosity of vegetable oils and consequently improves their fuel properties as they become less liable to clog injector and relate tubing in the automobile engine (Onanuga & Coker, 2013).

#### **Diesel Index**

A high diesel index is an indication of the high ignition quality of the fuel. The diesel index of CaO biodiesel was 47 to 39 for both CaO/AC and conventional CaO catalyst which is closer to 44 for petroleum diesel standard in agreement with (Alang *et al.*, 2018).

#### **Sulphur content**

The sulphur content of vegetable oil originates from the soil on which the crops grow and confers lubricity to fuel. Significant amount of sulphur in fuels have adverse environmental effects. From the exhaust system, sulphur

compounds are pollutants which cause acid rain, destroying the vegetation and engineering structures. The values obtained for CaO/AC biodiesel was 0.022 respectively (Alang *et al.*, 2018).

### Characterization of Catalyst

#### FTIR

This experiment examined the chemical composition of calcined CaO/AC using Fourier transform infrared spectroscopy (FTIR) analysis. Figure 3D & 3e shows absorption bands of pure CaO and CaO/AC. FTIR spectral of convectional CaO catalyst were shown in Fig. 3d with bands centered at 717, 877, 963, 1041 and 1395. FTIR spectral of calcined CaO/AC are shown in fig. 4d show the absorption bands of pure CaO and CaO/AC. FTIR spectral of convectional CaO catalyst were shown in fig. 3d with bands centered at 2899, 2121, 2002, 1637, 1637, 1465, 1383, 985, 866, 739 and 693 $\text{cm}^{-1}$  as summarized in table 7. The intensity of these bands was due to thermal decomposition of  $\text{CaCO}_3$  and formation of CaO and  $\text{Ca(OH)}_2$ .

#### XRD

The XRD diffractogram from fig. 5 also confirmed the existence of CaO loads activated carbon via impregnation process. The diffraction peak at  $2\theta$  ranges between  $10^\circ$  and  $90^\circ$ . The FULPROF software (version 2017) was used for data handling. FULPROF software allowed estimating the average size of the crystallites. The average crystallite sizes of particles were estimated by the Scherer's formula as shown in equation (4.10) below:

$$D = 0.89\lambda / \beta \cos\theta \quad 4.10$$

Where D is the crystallite size,  $\lambda$  is the X-ray wavelength,  $\beta$  is the broadening of the diffraction peak and  $\theta$  is the diffraction angle for maximum peak. These value is 43.2 nm for the extracted biodiesel.

Basic strength of CaO/AC was determined by TPD –  $\text{CO}_2$  as presented in fig. 6. The CaO/AC sample shows maximum  $\text{CO}_2$  desorption peaks at  $797^\circ\text{C}$ . This is due to the high loading of CaO on to the activated carbon. The catalyst sample corresponds to moderate basic site strength. Hence, the increase in CaO loading on activated carbon decreases the basic strength. The work is in total agreement with the work by (Mansir & Taufiq, 2017).

### Characterization of Waste Cooking Oil Biodiesel

FT-IR spectra of waste cooking oil and produced from this oil was compared with each other due to presence of triglyceride and ester as illustrated in fig. 3a, 3b and 3c. However very small differences were observed where the peaks appeared at 2921, 2854, 2087, 1745, 1462, 1153, 1115, and 724  $\text{cm}^{-1}$  in the biodiesel respectively. So the disappearance of the peaks from the spectrum of the kernel oil at 2087, 2121, 1787, 2121, 1745 and 1741  $\text{cm}^{-1}$  and formation of new peaks at 1741 and 873  $\text{cm}^{-1}$  proposed that the biodiesel synthesized from waste cooking oil contains low water contents

The waste cooking oil indicates a peak at 2921  $\text{cm}^{-1}$  conforming the anti symmetric and symmetric and stretching vibration of carbonyl groups that is present in the triglycerides for CaO/AC biodiesel and 1741 $\text{cm}^{-1}$  for biodiesel. The peak at 1400-1200  $\text{cm}^{-1}$  region confirmed the bending vibration of C-O present in the esters and peak t 741  $\text{cm}^{-1}$  confirmed the stretching vibration of C-O stretching vibrations. Presence of broad peak in region 3100-3500  $\text{cm}^{-1}$  indicate high water contents of which are saturated and other not; some are aliphatic while a few are ringed structures (Aladetuyi *et al.*, 2015). The IR spectrum above confirms the stoichiometric relationship of triglycerides reacting with calcium oxide to give methyl esters of fatty acids. The peaks and related information confirm the formation and presence of esters in biodiesel, given that there is a total absence of aromatics.

### Conclusions

In this work a study of the calcinations of calcium oxide loaded activated carbon, determination of physicochemical properties of WCO, optimization of esterification and transesterification was carried out by response surface methodology (RSM). The parameters for esterification reaction such as: reaction temperature, catalyst loading and reaction time, and for transesterification reaction such as: reaction temperature, catalyst loading and methanol to oil ratio were studied. The analysis of variance (ANOVA) indicated that a satisfactory result was obtained. Furthermore, increasing both methanol and reaction temperature led to higher percentage yield by maintaining the catalyst loading as constant. The statistical developed for predicting yield shows a good agreement between experimental and calculated values demonstrating the importance of regression analysis as a tool for the optimization purposes. The experimental results suggested the optimum conditions as follows: reaction temperature at 333K, catalyst loading at 0.26 mg and methanol to oil ratio as 12.5:1 v/v; and that of pretreated oil was reaction temperature at 333K, catalyst

loading 0.3 mg and reaction time as 40 min. this optimized conditions were validated with the actual WCO pretreatment and biodiesel yield in 92.7% for CaO/AC.

### Recommendation

From the findings in this research, the following recommendations was made:

- i. Nigeria is blessed with a lot of waste cooking oil (sources of biodiesel) due to large number of population, industries in the country must realized and to profitably learn from countries with an already developed biodiesel industries.
- ii. The government and other private sectors should use this opportunity to eradicate poverty by encouraging the biodiesel venture.
- iii. All the stakeholders should be involved in every step for the development of a successful biodiesel industry.

### References

- Adelodun, U., Marinkovic, J., Thandavan, T.M.K., Abdulganiyu, S.M., Wong, C.S. & Nor, R.M. (2016). Evaluation of Williamson- Hall Strain and Stress distribution in ZnO Nanowires Prepared using Aliphatic Alcohol. *Journal of nondestruct Eval*, 34 (4), 1-9.
- Aladetuyi, A., Olatunji, G.A., Ogunniyi, D.S. & Odetoye, T.E. (2014). Production and characterization of biodiesel using waste cooking oil, fresh and recovered from spent bleaching earth. *Biofuel Research Journal*, 134-138.
- Alang, K. (2018). Ultrasonic method of bio-diesel production from kernel. *Journal of applied and natural science*, 5 (1), 1-4.
- Amini, Z., Hwai, C.O., Mark, D.H., Fitranto, K.B., Hoora, M.B. & Zul, I. (2017). Biodiesel production by lipase-catalyzed transesterification of ocimum basilicum I. (sweet basil) seed oil. *Journal of Elsevier*, 132(1), 82-90.
- Dawodu, Y., Alamu, O.J., Waheed, M.A. & Jekayifa, S.O. (2014). Effect of ethanol-waste cooking oil ratio on alkali-catalyzed biodiesel yield. *Journal of fuel*, 87 (1), 1529-1533.
- Hassan, G. & Nageswara, F. (2018). Biodiesel Production From jatropha oil (Jatropha curcas) with high free fatty acids: An optimize process. *Journal of Biomass and Bioenergy*, 31 (1), 569-57.
- Kessler, T.M. (1985). Influence of fatty acid methy esters on fuel properties of biodiesel produced from the seeds oil of curcubita pepo. *Nigerian Journal of Basic And Applied Science*, 19 (1), 81-86.
- Kombe, J., Shote, A.S., Betiku E. & Asere A.A. (2015). Characteristics of CO and NOx emission from combustion of transmethylated waste cooking oil oil-based biodiesel blends in a compression ignition engine. *Journal of King Saud University- Engineering Science*, 1-6.
- Mansir, M. & Taufiq, J. (2017). Production of bio-diesel from waste cooking oil and groundnut. *Journal of Advance Materials Research*, 367(1), 501-506.
- Marinkovic, J., Thandavan, T.M.K., Abdulganiyu, S.M., Wong, C.S. & Nor, R.M. (2018). Evaluation of Williamson-Hall Strain and Stress distribution in ZnO Nanowires Prepared using Aliphatic Alcohol. *Journal of nondestruct Eval*, 34 (4), 1-9.
- Morrison, M. & Boyd, K. (2008). Evaluation of biodiesel production process by the determining of the total glycerol content in biodiesel. *Journal of Energy Procedia*, 138 (1), 544-551.
- Ojolo, S.J., Adelaja, A.O. & Sobamowo, G.M. (2012). Production of bio-diesel from waste cooking oil and groundnut, *journal of advance materials research*, 367, 501-506.
- Ojoloet, H., Ayodeji, A. A., Fredrick, H.K., Mathew, O.A., & Ifeoma, U.N. (2012). Optimization of Experimental conditions for biodiesel production. *International Journal of Engineering & Technology*, 12(6), 1-4.
- Onanuga, O.K. & Coker, J.O. (2013). Ultrasonic method of bio-diesel production from kernel. *Journal of applied and natural science*, 5 (1), 1-4.
- Ozbay, G.O., Ayodeji, A. A., Fredrick, H.K., Mathew, O.A., & Ifeoma, U.N. (2008). Optimization of Experimental conditions for biodiesel production. *International Journal of Engineering & Technology*, 12(6), 1-4.
- Pahl, D. (2005). Synthesis of Eosin Y-Sensitized Ag-tio2 Nano-Hybrid for optimized photocatalytic degradation of Aqueous Caffeine. *Journal of Chem. Sco.*, 64(1) 1-11.
- Reddy, H., Peiter, A.S., Lins, P.V.S., Meili, I., Soletti, J.I., Carvalho, S.H.V., Pimentel, W.R.O., & Merchetti, S.M.P., (2015). Stirring and mixing in ethylic biodiesel production. *Journal of King Saudi University- Science*, 44 (1), 55-57.
- Sankaran, O. & Show, E. (2016). Optimization of biodiesel Production from refined cotton seed oil and its characterization. *Egyptian journal of petroleum*, 26 (1), 103-110.

- Shote A.S., Betiku E. & Asere A.A. (2018). Characteristics of CO and NO<sub>x</sub> emission from combustion of transmethyatedwaste cooking oil oil-based biodiesel blends in a compression ignition engine. *Journal of King Saud University- Engineering Science*, 1-6.
- Tabatabaei, I., Ibrahim, M.M. & Gaya, U.I. (2015). Synthesis of Eosin Y-Sensitized Ag-tio<sub>2</sub> Nano-Hybrid for optimized photocatalytic degradation of Aqueous Caffeine. *Journal of Chem. Sco.*, 64(1) 1-11.
- Tiwari, A.K., Kumar, A. & Raheman, H. (2007). Biodiesel Production From jatropha oil (*Jatropha curcas*) with high free fatty acids: An optimize process. *Journal of Biomass and Bioenergy*, 31 (1), 569-57.
- Tiwari, H.L., Sankaran, O. & Show, E. (2007). Optimization of biodiesel Production from refined cotton seed oil and its characterization. *Egyptian journal of petroleum*, 26 (1), 103-110.
- Tsonchiva, M. (2018). Influence of fatty acid methy esters on fuel properties of biodiesel produced from the seeds oil of curcubita pepo. *Nigerian Journal of Basic And Applied Science*, 19 (1), 81-86.