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Biodiesel Synthesis from the Waste Biomass

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ABSTRACT: *In this work, physicochemical properties of the waste used oil (WUO) were carried out for its aptness for biodiesel production. Burnt Animal Bone was used as a precursor for the biodiesel production. The produced biodiesel was characterized and the optimum biodiesel produced was determined via statistical analysis. This was with a view to add value to WUO and finding environmentally friendly alternative to fossil fuel. WUO was obtained from eatery in Yenegoa, Bayelsa State, Nigeria. The foreign materials and dirt in the oil was removed by filtration after preheating. The physiochemical and other parameters (cetane number, API, aniline point among others) properties of WUO were determined using standard methods. The BAB was burnt to ash in a furnace, sieved into fine powder, and then characterized using FTIR, SEM, XRF, BET adsorption, and qualitative analysis. Biodiesel production was done via base catalyst transesterification while statistical analysis was done using Microsoft Excel 8.0. In order to ascertain the quality of the biodiesel, the physicochemical properties were determined. Results showed that the refined WUO properties were in line with oil property requirement for biodiesel production. The physicochemical characteristics of the WUO showed physical state of the oil to be liquid/dark brownish at 28 ^oC, viscosity 6.58 cP at 28 ^oC, acid value, 0.96 (mg KOH/g oil), FFA (% oleic acid), 0.48, iodine value, 152.00 (g I_2/100g oil), peroxide value, 5.1 milli-equivalent of peroxide/kg of oil among others. The derived catalyst showed high basic strength with Calcium oxide (87.63 wt. %) as the dominant element in the catalyst. Optimum biodiesel yield was obtained at run 5 with 98.52 (%wt. /wt.) at reaction time of 30 min, catalyst amount of 2.0 (%wt.), reaction temperature of 100 ^oC, and ethanol-oil molar ratio of 4:1. The produced biodiesel properties conformed to the recommended standard ASTM D6751 and EN 14214. The study concluded that WUO could serve as feedstock for biodiesel production that is environmentally friendly and the derived catalyst could be used as a bio-base in Industries.*

KEYWORDS*:* biodiesel, palm kernel oil, waste used oil, biomass, characterization of biodiesel, transesterification, burnt animal bone, vegetable oil and animal fats.

INTRODUCTION

World's energy needs are majorly supplied through petrochemical sources. Coal and natural gases, with the exception of hydroelectricity and nuclear energy are finite and at current usage rates will be consumed shortly. The high energy demand in the industrialized world as well as in the domestic sector and pollution problems caused due to the widespread usage of fossil fuels make it increasingly necessary to develop the renewable energy source of limitless duration and smaller environmental impact than the traditional one. This has stimulated recent interest in alternative sources for petroleum- based fuels. One possible alternative to fossil fuel is the use of oils of plant origin like vegetable oils and tree borne oil seeds. This alternative diesel fuel can be termed as biodiesel.

The palm tree (*Elaeis guineensis*) is arguably the richest tree on earth when it comes to natural endowments. It is blessed with a lot of priceless resources, which among them is the **palm kernel oil.** Palm kernel oil is extracted from palm fruit. It is dark black in color and distinguishes itself with a unique taste and smell. Palm kernel is among the tropical fruits that is attracting much attention both within and outside the African continent.

Biodiesel is an alternative fuel similar to conventional or fossil diesel. Biodiesel is one of the potential alternative energy sources that can be derived from renewable and low-grade origin through different processes. Biodiesel can be produced from straight vegetable oil, animal oil/ fats, tallow and waste cooking oil. The process used to convert these oils to Biodiesel is called transesterification. One of the processes is alcoholysis or transesterification in the presence of a suitable catalyst. Most biodiesel produced at present is from waste vegetable oil, though oil straight from the agricultural industry represents the greatest potential source it is not being produced commercially simply because the raw oil is too expensive. After the cost of converting, it to biodiesel has been added on it is simply too expensive to compete with fossil diesel. Waste vegetable oil can often be sourced for free or sourced already treated for a small price. (The waste oil must be treated before conversion to biodiesel to remove impurities). The result is Biodiesel produced from waste vegetable oil and can compete with fossil diesel.

Experimental Procedure

MATERIALS AND METHODS

Biodiesel

Researchers are looking for a suitable substitute for petroleum-based fuels due to the high degree of pollution they cause in the ecosystem (land, air, and sea). Renewable energies (biomass, hydropower, sun, tidal waves, wind, and geothermal) have been considered as promising sources of energy. (Yingying *et al*., 2012; Thanh *et al*., 2012). However, biomass is regarded as the most cost-effective source of renewable energy.Rudolf Christian Karl Diesel's first engines were designed to run on vegetable peanut oils (Ma *et al.*, 1999; Ahmad *et al*., 2014), but they were later redesigned to accommodate the properties of petro-diesel when crude oil became abundant.

Production of Biodiesel from different triglyceride sources is an alternative fuel to petrol-diesel. The American Society for Testing and Materials (ASTM) defines biodiesel as mono-alkyl esters produced from various lipid feedstock including vegetable oils, animal fats, etc. literally, it has been accepted as a fuel and fuel additive worldwide and registered with the U.S. Environmental Protection Agency (EPA). Owing to the worries about petroleum availability and the current increase in petroleum price, the usage of biodiesel in conventional diesel engines has attracted much attention.

The most common method used in producing biodiesel is a transesterification process. In this process, lipid feedstocks are converted into biodiesel. One mole of triglyceride reacts with three moles of alcohol to produce three moles of mono-alkyl ester and one mole of glycerol. To improve the rate of reaction and biodiesel yield, a catalyst is usually added with excess alcohol, which shifts the equilibrium to the product side since the reaction is reversible. Traditionally, the transesterification process utilizes solvents including ethanol or methanol and homogeneous catalysts such as KOH, NaOH, and H_2SO_4. However, this method has several shortcomings such as extensive separation process, wastewater generation, and equipment Corrosion.

Biodiesel blend is the blend of petroleum diesel and biodiesel (methyl ester). A blend of 5% biodiesel and 95% regular diesel is called a B5 blend. Biodiesel has similar physical characteristic as diesel oil and in addition it is a renewable energy and safe for the environment. Biodiesel can be used easily because it can be mixed at any proportion with diesel oil, hence enabling us to apply it immediately for diesel engines that are available without much modification ; easy biodegradability; 10 times less poisonous compared to the ordinary diesel oil, the waste product is not black, less Sulphur and other aromatic contents, hence the combustion emission produced is safe for environmental and perform less accumulation of carbon dioxide gas in atmosphere thus lessen furthermore global heating effect.

Oil Palm

Elaeis guineensis is the palm family (Arecaceae), cultivated as a source of oil. The oil palm is grown in its native west and central Africa, as well as in Malaysia and Indonesia (Editors of Encyclopaedia Britannica 2021). Oil palm is mainly, perennial crop cultivated from its vegetable oil, which is composed of both palm oil and kernel oil. Palm oil is derived from the mesocarp, whereas kernel oil is derived from the endosperm or kernel. Both vegetable oils from oil palm are constituents of many foods, oleo chemicals, medicinal and health products, household products, and industrial products. About 16% of world palm oil is converted to biodiesel. Oil palm is originally the second largest source of edible oil, next only to soybean. It contributes approximately one-fifth of the world's production of oils and fats, and belongs to the genus *Elaeis*. There are two important species in the genus *Elaeis*,E. *guineensis* (African oil palm) and *E. oleifera* (American oil palm). African oil palm is commercially cultivated and a high-yielding species.

Feedstocks Used in Biodiesel Production

Vegetable oils, animal fats, and waste oils are the most common feedstocks used in biodiesel production (Nurudeen *et al*., 2021; Basumatary *et al*., 2020; Cholapandian *et al*., 2021). Several oils, including non-edible oils, such as Jatropha curcas Linn, Oil palm (Elaeis guineensis),

Moringa oleifera, Shea butter, Bitter melon, Kenaf, Kahalari Melon, palm kernel, animal fat, waste oils, and Pumpkin (Cucurbita maxima) seeds, have been used in the production of biodiesel in recent years (Adepoju *et al*., 2020; Adepoju *et al*., 2021; Anietie *et al*., 2022). Price, consistency of feedstock quality and chemical composition, regular accessibility of the feedstock, elasticity to expand supply, and cost of transport and pretreatment are all important factors to consider when selecting feedstock for biodiesel production.

The current generation of animal fat and vegetable oils is insufficient to totally replace fossil diesel fuel. Waste cooking oil can be used to reduce biodiesel production costs and broaden its feedstock base. Certain volumes of discarded cooking oil are available all over the world. These are produced locally anywhere food is cooked or fried in oil, such as hotels, restaurants, KFC, and so on. Nigeria lacks statistical information on the amount of feedstock available. Nonetheless, an accurate assumption can be established for the waste cooking oil discarded annually in Nigeria. This amounts to around 32 million metric tonnes of garbage per year.Better disposal of old waste oil poses a significant challenge due to issues related with dumping and potential pollution of water and land resources. Some of the wasted cooking oil is utilized to make soap and as an oil additive in fodder production. Nonetheless, large amounts of spent waste oil are illegally dumped into landfills and waterways, causing contamination. (Adepoju *et al*., 2022) used waste used oil to produce biodiesel, which helped to reduce greenhouse gas emissions while also reducing landfill and water pollution.

Transesterification of Oils.

Transesterification or alcoholysis occurs when different types of oils and triglycerides combine with alcohol, typically methanol or ethanol, to generate esters and glycerine. A catalyst is introduced to the reaction to make this feasible (Sahani *et al.*, 2018). The total process is often a series of three reversible reactions. The first process created diglycerides from triglycerides, the second produced monoglycerides from diglycerides, and the final step produced glycerine from monoglycerides. Esters are formed in all of these processes. The stoichiometric relationship between alcohol and oil is 3:1. An excess of alcohol, on the other hand, is usually more appropriate for improving the reaction to the intended result. This method has been frequently utilized to lower triglyceride viscosity. Transesterification is a reversible reaction that occurs mostly via mixing the reactants. The presence of a catalyst (a strong acid or basic) on the other hand, speeds up the conversion.

Variables Affecting Transesterification Reaction

Depending on the setting utilized, many factors influence the transesterification process. FFA and moisture content, catalyst and concentration type, molar ratio of oil/methanol ratio, temperature and reaction duration, and the influence of mixing are all factors to consider. This section discusses the consequences of various variables.

Physicochemical Properties of WUO

The evaluation of physicochemical properties of WUO such as: moisture content, relative density, viscosity, acid value, saponification value, peroxide value, specific gravity, refractive index, mean molecular mass, %FFA, higher heating value, cetane number and pH were determined by the methods of AOAC. Iodine value was obtained by Wij's method, while API,

Diesel index and Aniline point were obtained. While moisture content and peroxide value was determined by the method reported by Adepoju *et al.,* 2012).

Moisture Content (AOAC,1990)

5 g of oil sample was carefully weighed into a moisture dish of 5 cm diameter and 2 cm deep with tight-fit-slip-over cover. Then it was placed in the oven with temperature of 125^0C above boiling point of water at working pressure of 95 mmHg for 30mins interval and the weight of the sample was noted respectively until a constant weight was achieved when there was no additional loss of 0.05%, The moisture content was calculated using the equation below

$$\% \text{ Moisture content} = \frac{initial\ weight\ of\ sample - final\ weight}{initial\ weight\ of\ sample}$$

Acid Value (AOAC,1990)

The acid value was determined by dissolving 5g of oil sample in a hot mixture of 25 ml (95%v/v) diethyl ether and 25ml ethanol in a 250 ml flask. The hot solution was neutralized with 0.1 M potassium hydroxide solution using three drops of phenolphthalein as an indicator. The acid value would be calculated using Equation below

$$\text{Acid value} = \frac{V \times N \times 282}{W} \times 100$$

Mean Molecular Mass

This was determined by the equation cited by

$$Mean\ molecular\ mass = \frac{56}{saponification\ value} \times 1000$$

FFA – Free Fatty Acid (AOAC,1990)

The FFA was obtained by

$$\% \text{ FFA} = \frac{\boldsymbol{Acid\ \ value}}{\mathbf{2}}$$

Peroxide Valve (AOAC, 1990)

Oil sample of 2 g was weighed into a 250 ml Pyrex flask; 40ml of the solvent mixture (2:1 glacial acetic acid / chloroform) and 2 g KI powder were added. The mixture was boiled briskly in a water bath for 1 min at a temperature of 70^{o}C. To the flask containing 40 ml of 50% KI, boiled mixture was added; the resulting mixture was washed twice with 50 ml of distilled water into flask. The content of flask was then titrated with 0.004M sodium thiosulphate ($Na_2S_2O_3$) solution by using starch as indicator. The peroxide value was calculated as indicated below

$$\text{Peroxide Value (meq}0_2\text{/ kgoil)} = \frac{volume\ of\ Na_2\ S_2\ O_3}{weight\ of\ the\ oil\ sample\ in\ kg}$$

Saponification Value (AOAC, 1984)

Oil sample of 2 g was weighed into a 250 ml Pyrex flask with 25ml of 0.1M ethanolic potassium hydroxide which added. The content was constantly stirred and was allowed to boil gently for 60min, with a reflux condenser placed on the flask containing the mixture to achieve uniform temperature. Two drops of phenolphthalein indicator were added to the warmed soap solution and then titrated with 0.5 M HCI to the end point until the pink colour of the indicator just disappeared. The saponification value was calculated via

$$S.V\ (mgKOH\ /goil)\ = 28.05 \times \frac{(Vol.of\ HCl\ required\ by\ blank - Vol.of\ 0.5\ NHCl)}{weight\ to\ sample\ in\ g}$$

Iodine Value (Wij's method)

Oil sample of 0.26 g would be dissolved in 10ml of cyclohexane 20ml of Wij's solution was added, the stopper flask was allowed to stand for 30 min in the dark at room temperature and 20ml of 10% potassium iodide solution was added. The resulting mixture was then titrated with 0.1M $Na_2S_2O_3$ using starch as indicator iodine value was calculated using

$$Iodinevalue = \frac{(B-S)\times N\times 12.69}{Weight\ of\ the\ oil\ sample}$$

Where N = Concentration of sodium thiosulphate used

B= volume of sodium thiosulphate used for blank

S= volume of sodium thiosulphate used for determination.

Aniline Point

This was determined by the equation described below

$$Aniline\ point = \frac{\boldsymbol{diesel\ index\times 100}}{\boldsymbol{API}}$$

Specific Gravity

The specific gravity of the oil sample was measured using the specific gravity bottle. The bottle was full with water and the weight was noted. After a while, the bottle was also full with oil sample and the weight was recorded also.

The expression for specific gravity (Sp.gr) is given below

$$Sp.gr = \frac{\boldsymbol{w_1 - w_0}}{\boldsymbol{w_2 - w_1}} = \frac{\boldsymbol{weight\ of\ the\ oil}}{\boldsymbol{weight\ of\ an\ equal\ volume\ of\ water}}$$

P-anisdine (AOAC,1990)

This was determined by measuring the absorbance value at 350 nm of a mixture of solvent 20 ml isooctane and p-ansidine (1:1) with 1g of oil sample in UV- Spectrophotometer at room temperature. The p-anisidine value was obtained using

p- anisidine = $100\times$ $\boldsymbol{Absorbance\ value\ obtained}$

Cetane Number (ASTM D2015)

This was calculated using

$$Cetane\ no = 46.3 + \frac{\boldsymbol{5458}}{\boldsymbol{Sap.Value}} - \boldsymbol{0.225\ iodine\ value}$$

Higher Heating Value (HHV) – (ASTM D2015)

This was calculated using

HHV (MJ /kg) = 49.43 – [0.041(Sap. value) + 0.015(Iodine value 0]

Viscosity (AOAC,1990)

A viscometer glass tube which was held in vertical direction was used. The sample of the Waste used oil or biodiesel was drawn into a capillary tube via suction to a marked volume. The sample was then allowed to flow down into the lower bulb and the time taken to pass through the marks was noted. The kinematic viscosity was calculated using

Kinematic viscosity = time taken x viscometer factor

API

This was determined by the equation citied as described below

$$\text{API} = \frac{141.5}{specific\ gravity\ \ at\ 15^0c} - 131.5$$

Diesel Index

This was determined by the equation citied as described below

$$\text{Diesel index} = \frac{centane\ number - 10}{0.72}$$

Totox Number

The Totox number was calculated according to the equation below

Totox number = 2 x Peroxide value – p-Anisidine Value

Analysis of Burnt Animal Bone

The analysis of the BAB was carried out using the following equipment SEM to observe the various signals that contain information about the surface topography and structure of the BAB. The XRS-FP analyser is to derive the elemental peak intensities corresponding to each element, and to establish the elemental concentrations in the calcined catalyst powder. FTIR analyser is to identify the organic, polymeric, and in-organic compound present in BAB. BET analysis is to measure the surface area by gas sorption, and give multipoint data, as well as full isotherms for porosity determination (Adepoju *et al*., 2021).

BET Procedure for Catalysts Analysis

Prior to BET analysis, in order to remove any adsorbed loaded entities from the catalyst surfaces, the samples were first warmed at 150 **ºC** for 45 min under helium flow. Next, for 40 min, 5% CO2 was applied to the sample while helium was still flowing at a flow rate of 25 ml/min. By using CO2-temperature-programmed desorption (TPD) [manufactured in BEL132 Cat, Japan], the fundamental strength of surfaces made of calcined powder and blended powder was evaluated.

XRF Procedure for Catalysts Analysis

By using a dispersive X-ray fluorescence (XRF) spectrophotometer with a Rh source and tube with a power of 2.2 kW, the elemental compositions of the samples were examined. Using the Brunauer-Emmett-Teller (BET) method, the catalyst's specific surface area was determined through N2 adsorption/desorption isotherm analysis (Surface area & pore size analysis [Belsorb III, Japan]), which was completed at 196°C on a volumetric adsorption analyser.

FTIR Procedure for Catalysts Analysis

Similar to NDIR analyzers, FTIR spectrometers (Agilent Technologies Model Cary 122 630 FT-IR spectrometer) that operate in the 400–4000 cm-1 spectral range rely on the fact that many gases absorb IR radiation at species-specific frequencies. But since FTIR spectroscopy is a dispersing approach, measurements are made over a wide spectrum as opposed to a restricted range of frequencies. With this test method, calcined materials and calcined mixed samples are exposed to an x-ray beam, and the scattered intensity is measured as a function of

the outgoing direction. When an FTIR spectrometer shines IR beams at a sample and records how much of the beam and at what frequencies the sample absorbs infrared light, the beam is split, then scattered**.**

SEM Procedure for Catalysts Analysis

In order to assess the morphology of the powder prior to consolidation, scanning electron microscopy (SEM) is performed. The steps are simple to follow. An SEM sample stub is taped down using two-sided carbon tape, and the UHMWPE powder is dusted on top. The samples were coated with light gold, platinum (100), and then analyzed in a SEM chamber. The flakes have a diameter of 50 to 100 m. Through the use of field emission scanning electron microscopy, the surface morphology was revealed (FE-SEM, QUANTA FEG 250).

Biodiesel Synthesis

Experimental Design

Major parameters affecting the synthesis of biodiesel using the designed catalyst were taken into consideration, and the findings were statistically analyzed. Table 3.1 displayed the variable range used in this study. These were done twice, and the outcomes were noted, recorded, and examined using the statistical program Microsoft Excel 8.0 to determine the regression parameters.

WUO Biodiesel Production

The oil base was held at 300 ml, transferred to a 1 L reactor, and preheated at 60 **ºC** for 2 hours on a hot plate magnetic stirrer. A given amount of catalyst was dissolved in a known weight of ethanol and put to the warmed oil. Three layers were observed: the oil layer, the whitish ethanol-oil-catalyst layer, and the clear ethanol layer. The generated mixes were subjected to a chemical reaction at a specified temperature until the reaction time was reached. The product was transported to a separating funnel for separation and purification. Glycerol was removed from the bottom of the funnel, while green diesel with catalyst was separated by washing with methanolic-sodium hydroxide, filtering, and washing with distilled water. The recycled catalyst was purified and reused, while the wet green diesel was dried over calcium chloride and then filtered to produce biodiesel. The final product was the methyl ester known as biodiesel, and the yield was determined in terms of% (w/w) as specified below

$$\textit{Experimental yield \% (w/w)} = \frac{Weight\ of\ oil\ biodiesel\ produced}{weight\ of\ oil\ sample} \times 100$$

Statistical Data Analysis

The results of biodiesel obtained along with variables constraint was analysed with Microsoft Excel 8.0 to determine the regression parameter and the level of significant of the variables.

Table 1: Experimental variables constraint

SN	Reaction time (min)	Catalyst conc. (%wt.)	Reaction temperature (ºC)	Ethanol-Oil molar ratio (vol./vol.)
1	45	2.0	50	3
2	50	2.5	55	4
3	55	3.0	60	5
4	60	3.5	65	6
5	65	4.0	70	7
6	70	4.5	75	8
7	75	5.0	80	9

RESULT

Quality Characterization of Waste Used Oil (WUO)

Physical Properties of WUO

The content and compositions of the oil were subjected to physicochemical examination in order to assess the quality of the refined WUO purchased from the restaurant.

Table 2 displays the outcomes. The obtained WUO had a dark-brownish color and a specific gravity of 0.92 at ambient temperature. Its moisture content was 0.002%. The oil's color and refractive index were observed and corresponded with previously reported findings. According to Adepoju *et al*., (2014), the moisture content was 0.045% and the specific gravity was 0.91. The viscosity, which is a gauge of an oil's resistance to shearing, was 6.58 cP. This value is within the previously stated range (15.15 – 15.9 cP) for waste used oil (Cholapandian *et al*., 2021: Kirubakaran *et al.*, 2020).

Chemical Properties of WUO

Chemical qualities are one of the most crucial factors in figuring out the state and quality of oil samples right now. The findings regarding the WUO's chemical characteristics are presented in Table 2 The study's findings on WUO's low FFA level were suggestive of the oil's strong resistance to hydrolysis. A high value of 8.52 mg KOH/g oil was observed by (Adepoju *et al.*, 2014), compared to (Kirubakaran *et al.*, 2010) 0.67 for the FFA of WUO. It was demonstrated by the oil's low acid value of 0.96 mg KOH/g oil that it was not only ingestible but also had a potential for a lengthy shelf life

Table 2: Physicochemical and Other Characteristics of WUO.

Parameter	Mean values
Physical properties	
Physical state at 28℃	Brownish yellow
Moisture content (%)	0.002
Specific gravity	0.92
Viscoisty (cP) at 40℃	6.58
Chemical properties	
%FFA (as oleic acid)	0.46
Acid value (mg KOH/g oil)	0.96
Saponification value (mg KOH/g oil)	186.40

Iodine value (g I_2/100g oil)	152.00
Peroxide value (meq O_2/kg oil)	5.10
Other properties	
Cetane number	41.38
API	22.30
Higher heating value (HHV)	39.51

The WUO showed a high saponification value of 186.40 (mg of KOH/g of oil), indicating a high concentration of triglycerides. This falls within the range (175-250 mg of KOH/g of oil) commonly observed in other seed oils such as corn, mustard, raspberry, sunflower, and safflower (Yong and Salimon, 2006). The WUO has a significant amount of unsaturation because to its high iodine value (152.00 g of I2/100 g of oil). The hydroperoxide content of the oil is measured by the peroxide value, and a low number denotes a high level of oxidation resistance. In this investigation, 5.1 milli-equivalents of peroxide/kg of oil were the figure for WUO, which is a low amount. High iodine content combined with low levels of peroxide value suggests that the WUO could be stored for a long period without deterioration.

Catalytic Characterization and Analysis

BET Analysis of Catalyst

Utilizing several analyzers, the catalyst's characteristics were determined. Based on the results of the BET adsorption study carried out by data reduction acquisition using the DA method and nitrogen as adsorbate with mol. wt. 28.013 for catalyst mass of 0.12 g utilized for sampling, Figure 1 showed the plots of different pore volumes acquired via sample diameter. At a best energy (E) of 0.807 kJ/mol, the maximum DA micropore volume was discovered to be 0.318 (cc/g), which equates to 2.80 nm in diameter pores. It was noted that the sample started to develop a large surface area with a high pore diameter as the plot approached its apex, increasing the reaction rate. Relationship between pore diameter, surface area, and the cumulative volume utilizing the BJ adsorption method are shown in Figure 2. The sample's nature as a catalytic basis for the generation of biodiesel was demonstrated by the maximum surface area, pore volume, and pore diameter measurements, which were attained at 442.708 m2/g, 0.217 cc/g, and 2.132 nm, respectively.

XRS-FP Analysis

The concentration method, the percentage mole, and the components discovered in the catalyst using the XRS-FP analyser are as shown in Table 2 and are based on the XRS-FP analysis on the catalyst which demonstrated the intensity method carried out by Gaussian. The catalyst contains a number of different elemental compounds, but the main one is Calcium oxide, which came from the full disintegration of the Calcium carbonate present in the Waste Animal Bone during burning. Calcium oxide's prominent role in the catalyst (89.50 weight percent) demonstrates its appropriateness as a viable catalyst for the production of biodiesel. Meanwhile, other elemental compounds were also identified in small concentrations, which also aids in the formation of biodiesel. The presence of SiO2 (3.555weight percent) however, demonstrated that the powder catalyst has some acidic strength, while SiO2 also functions as a weak basic.

SEM Analysis

Figure 3, 4 and 5 show the sematic image of the structural outlook of the morphology displayed of the catalyst analysis via SEM at magnifications of 300x, 500x, and 1000x, respectively. Images revealed cracking structures of the catalytic sample with a shining but partially segregated trait, aggregated with a porous appearance indicating potential solubility in polar solvent. The presence of ZnO in the catalyst could explain the glittering appearance. However, the whiteness, brightness, and opacity of the sample may be attributed to the presence of SiO2. The presence of K2O and CaO, which indicate the base catalyst nature for the formation of biodiesel, could explain the beautiful glossy glaze, whitish pale appearance, caustic alkaline, and crystalline solids at room temperature.

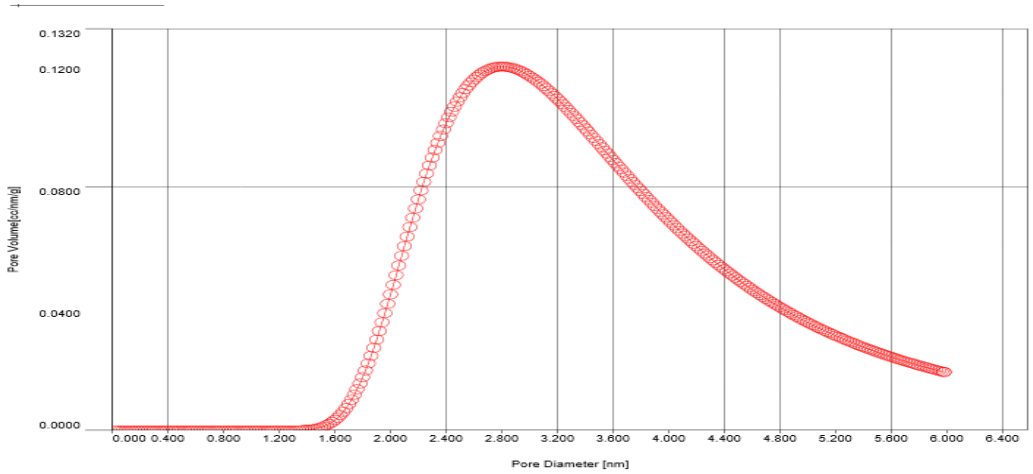


Figure 1: Micropore Volume Against Pore Diameter

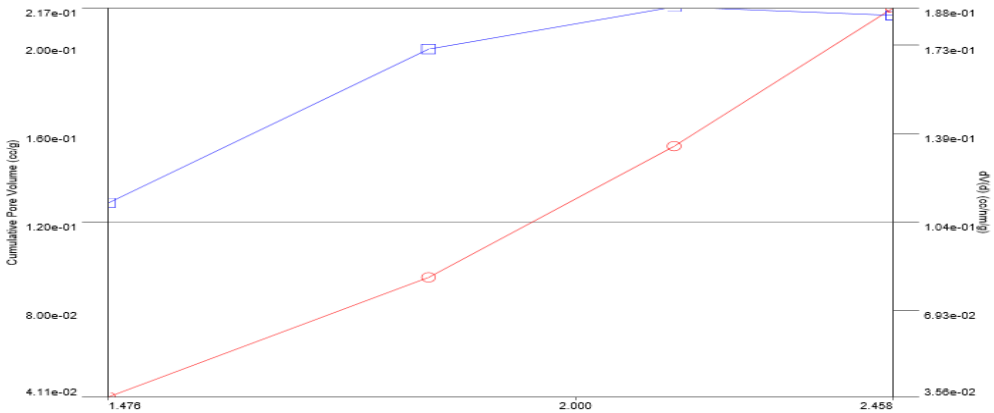


Figure 2: Cumulative Pore Volume, Surface Area Against Pore Diameter

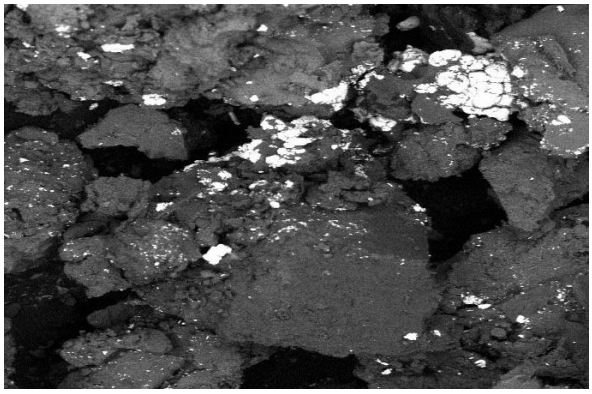


Figure 3: SEM Image at Magnification of 300x

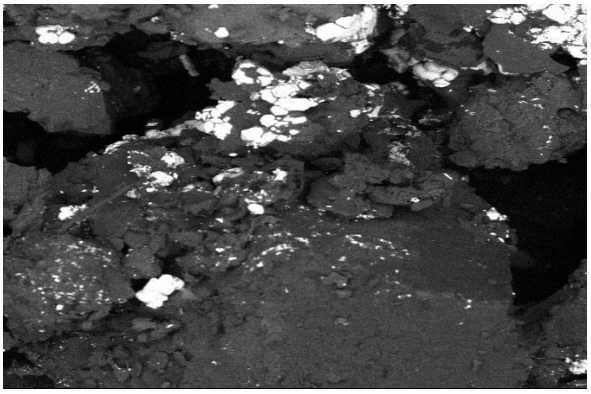


Figure 4: SEM Image at Magnification of 500x

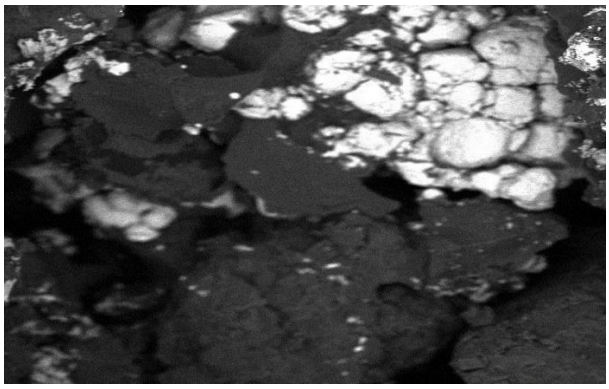


Figure 5: SEM Image at Magnification of 100x

Table 3: XRS-FP Analysis Report

Component	Concentration (wt.%)
SiO_2	3.55
Fe_2O_3	1.074
CaO	89.457
Al_2O_3	3.950
Cl	0.549
SnO_2	0.515
Others	0.899
Total	**100**

FTIR Analysis

Table 3 shows the results of the FTIR 8400S examination of the burnt catalyst, including the scanning runs, peak, intensity, correlation intensity, base height, base length, area, and correlation area. At various wavelengths and angular phases, several functional elements can be identified. Normally, the mid-IR spectrum is separated into four regions: the single bond area (2500-4000 cm-1), the triple bond region (2000-2500 cm-1), the double bond region (2000-2500 cm-1) and the triple bond region (2000-2500 cm-1) (1500-2000 cm-1), in addition to the fingerprint region (600-1500 cm-1).

The following functional groups can be found in the fingerprint region: I aliphatic organohalogen compounds such as C-F, C-Cl, C-I, and C-Br. (ii) the out-of-plane bend in Alcohol, OH (iii). Phenol, C-O stretch, (iv) primary, secondary, and tertiary alcohol, C-O stretch, (v) primary or secondary, OH in-plane bend, (vi) phenol or tertiary alcohol, OH bend, (vii) the peroxide, C-O-O-stretch, (viii) the Epoxy, oxirane rings, and Aromatic ethers, aryl-O stretch, (viii) the Alkyl-substituted ether, and Cyclic ethers with large rings, C-O stretch, (ix) the primary , secondary, and tertiary, both amine and aromatic CN stretch, (x) the carboxylate salt, the P-O-C, aromatic and aliphatic phosphates, the carbonate ion, sulfate, nitrate, calcium, silicate etc can be found.

The following functional group exists in the double bond region: I nitrogen-oxy compounds, open-chain imino -C = N-, and open-chain azo -N = N- (ii) carbonyl compounds such as ketones, carboxylic acid, aldehydes, ester, amide, acid halide, and aryl carbonate. (iii) The N-H bend of the primary and secondary amines, (v) the olefinic (alkene) such as Alkenyl C=C stretch, aryl substituted C=C, conjugated C=C (iv) aromatic ring (aryl) such as C=C-C aromatic ring stretch
The following functional groups can be discovered in the triple bond region (2000-2500 cm-1): I Acetylenic (alkyne) compounds such as C-C terminal and medial alkyne, (ii) the transition metal carbonyl, (iii) the ester carbonyl, (iv) the nitrogen multiple and cumulated double bond compound such as Thiocyanate (-SCN), Isocynate (-N=C=O asym. stretch), Cyanate (-OCN and C-OCN stratch), aromatic (CH3-O-).

There are functional groups such as I Alkyne C-H stretch, (ii) Olefinic (alkene) such as terminal (vinyl) C-H stretch, pendant (vinylidene) C-H stretch, medial, cis-or trans-C-H stretch, (iii) Saturated aliphatic (alkene/alkyl) such as methyl C-H asym./sym stretch, methylene C-H asym/ sym stretch, methyne C-H stretch, methoxy, methyl ether O-CH_3, C-H stretch, methylamino, N-CH_3, C-H stretch (iv) The Acetylenic (alkyne) such as alkyne C-H stretch (v) hydroxyl group, H-bonded OH stretch, typical polymeric OH stretch, Dimeric OH stretch, internally bonded and non-bonded hydroxyl group, OH stretch, primary, secondary, tertiary alcohol, OH stretch, phenols, OH stretch (vi) Methoxy ether and oxy compound, C-H stretch (CH3-O-) (vii) Aliphatic and aromatic primary amine NHH stretch, secondary aliphatic and aromatic amine >N-H stretch, heterocyclic amine >N-H stretch, and imino compounds =N-H stretch (vii) S-H thiols stretch, (viii) common inorganic ions like ammonium ion.

The wavelength peak discovered in this study is within the aforementioned ranges, hence the investigation of calcinated wood ash as a catalyst for biodiesel synthesis proved viable.
The plot of intensity versus the angular phase diagram was reflected in further quantitative investigation on the catalyst utilized based on phase data view. The plot displayed a zig-zag pattern with quartz as the main compound and carbon as the main element. Other compounds having figures of merit based on weight fraction include Graphite, Adamite, Gahnite, Zinccite, and Willemite (Figure 6). The presence of quartz in the catalyst represents the catalytic current, which aided in the rhythmic vibrating of the phases. The graphite in the catalyst is responsible for high tension lubrication and low viscosity product. The presence of adamite in the catalyst aided in the production of biodiesel color; adamite is typically yellow in color and is responsible for light yellowish biodiesel created in this work. Other substances also contribute in the generation of biodiesel

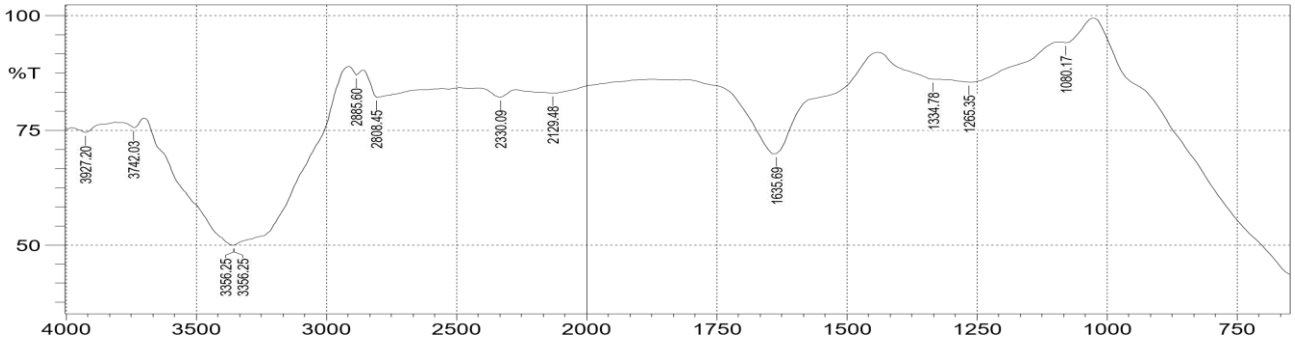
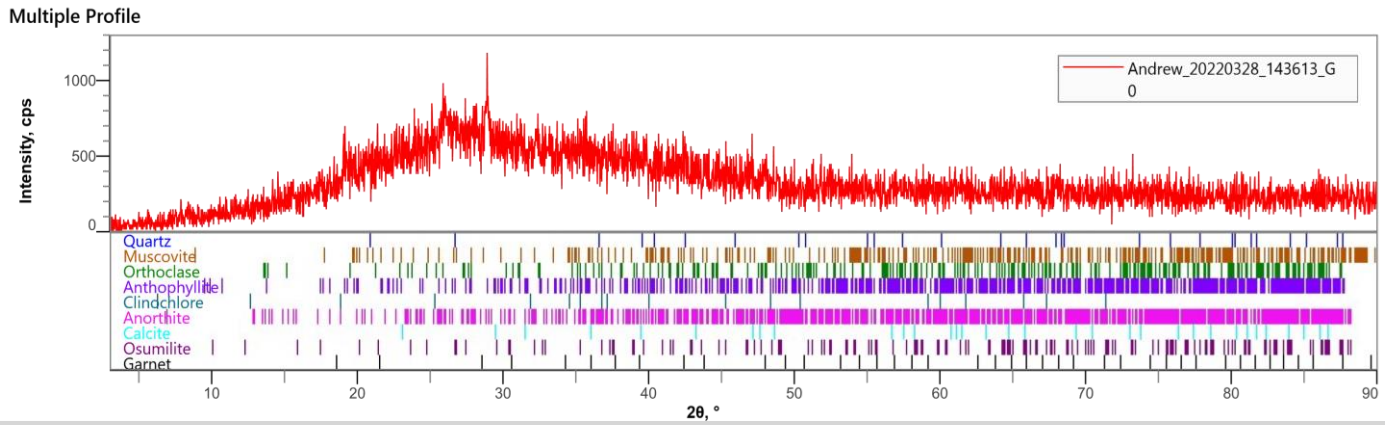


Figure 6: Weight Fraction



Dataset / Weight F…	Value, U…	Quartz	Muscovite	Orthoclase	Anthophyllite	Clinochlore	Anorthite	Calcite	Osumilite	Garnet
Loretta		42(3)	4.7(7)	7.3(10)	3.0(4)	3.8(11)	8(2)	19(2)	1.5(5)	10.3(12)

Figure 7: Quantitative Analysis Report

Plot of the results

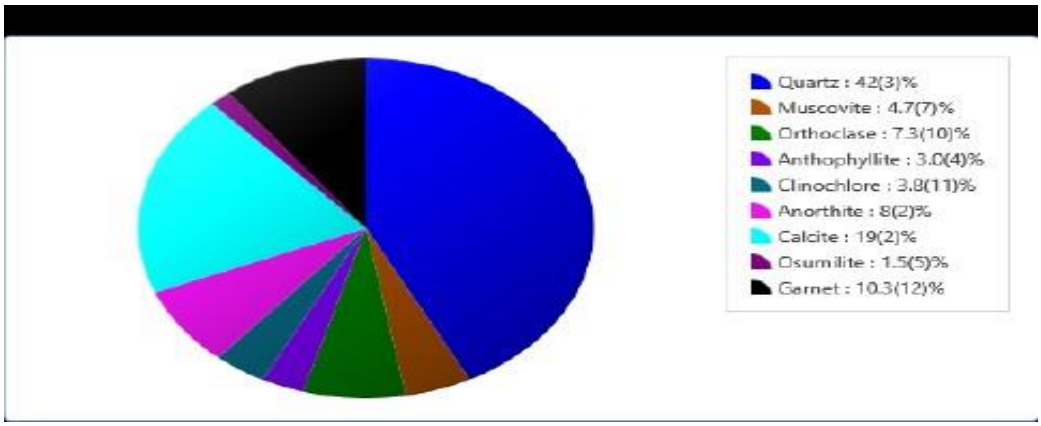


Figure 8: Pie chart plot of the results

Transesterification of WUO to Biodiesel

Table 4 displayed the findings of the biodiesel synthesized from WUO, which included the factor variables that varied in ranges as well as the biodiesel production. The maximum biodiesel production was produced at run 5 with 98.52 (%wt./wt.) at a reaction time of 65 minutes, a catalyst quantity of 4.0 (%wt.), a reaction temperature of 70**ºC**, and an ethanol-oil molar ratio of 7:1. This shown that the yield of biodiesel increases with increasing reaction conditions, but decreases at higher temperatures over 70**ºC** due to the catalytic character of the bio-base, which aggregates and clusters. As a result, the high FAME conversion burning temperature is the optimal burnt temperature for catalytic conversion of the powder to catalyst response for FAME conversion of 99%. (Wt.). Furthermore, ethanol boiling temperatures higher than the guideline temperature reduce biodiesel yield due to solvent loss during the reaction process.

Statistical Analysis

The acquired results were statistically analyzed using Microsoft Excel 8.0. The plot in Fig. 4.5 demonstrated that variables considered in various ranges were remarkably significant, with a high coefficient of determination R-square (R2) of 0.8164. Indicating a high degree of consistency between the experimental values and the factors taken into account. R2 values greater than 0.800 have been reported to be significant (Betiku *et al.,* 2011). The model equations indicating variable slope and intercept shown against yield.

Table 4 Biodiesel yield and Constraint Variables

SN yield	Reaction Time(min)	Catalyst conc.(%wt)	Reaction temperature (ºC)	Ethanol-Oil molar ratio (vol./vol.	Biodiesel (%wt/wt.)
1	45	2.0	50	3	85.60
2	50	2.5	55	4	88.50
3	55	3.0	60	5	90.86
4	60	3.5	65	6	97.84
5	**65**	**4.0**	**70**	**7**	**98.52**
6	70	4.5	75	8	96.40
7	75	5.0	80	9	92.70

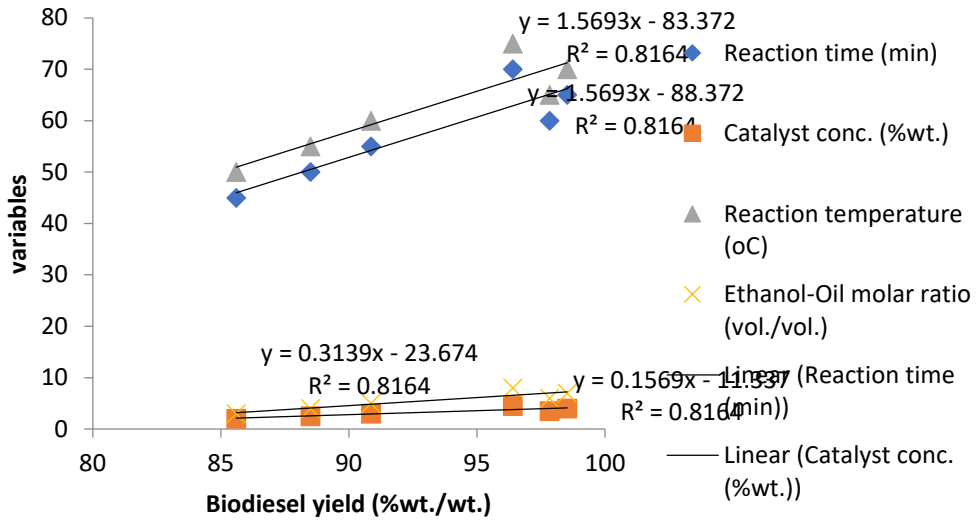


Figure 8: Excel Plots of Biodiesel Yield Against the Variables

Physicochemical Properties of Biodiesel

To assess the acceptability of the biodiesel produced in this study, the physicochemical parameters of the biodiesel were determined using AOAC, 1997 techniques. The parameters of the produced biodiesel were displayed in Table 4.4. These results were compared to the recommended biodiesel standard. According to the table, the quality of the produced biodiesel was in accordance with the standards established by American and European biodiesel standards (ASTM D6751 and EN 14214). This means that the biodiesel produced in this study can be used in place of conventional diesel in industries and for commercial purposes.

Table 5: Physicochemical and Other Characteristics of Biodiesel

Parameters	Biodiesel	ASTM D6751	EN14214
Physical properties			
Physical state at 28°C	Light brown	-	-
Moisture content (%)	0.001	0.05 max	0.02
Specific gravity	0.83	0.86-0.90	0.85
Viscosity (cP) at 40°C	2.56	1.9-6.0	3.5-5.0
Chemical properties			
%FFA (as oleic acid)	0.28	<0.40	0.25 max
Acid value (mg KOH/g oil)	0.58	<0.80	0.50 max
Saponification value (mg KOH/g Oil)	182.32	-	-
Iodine value (gI_2/100g oil)	116.00	-	120 max
Peroxide value (meq O_2/kg oil)	2.48	-	-
Other properties			
Cetane number	50.14	47	51min
Diesel index	55.75	50.40	-
API	38.98	36.95	-
Higher heating value (HHV)	36.15	-	-

CONCLUSION

From the result obtained in this study, the following conclusion can be drawn that Burnt animal bone was found to be rich in calcium content (89.457) and the Optimum biodiesel yield of 98.52 (%wt./wt.) was obtained at reaction time of 30 min, catalyst amount of 2.0 (5%wt.), reaction temperature of 100℃, and the ethanol-oil ratio is 4:1. A statistical analysis via Microsoft Excel proved that the constraint variable selected were remarkably significant with regression parameter R-square >0.8. The quality of the produced biodiesel was in line with the standard laid down by American and European standard for biodiesel (ASTM D6751 and EN 14214). The produce biodiesel in this study can replace conventional diesel that of economic standard and serve as IGR if other waste feedstock's can be harnessed.

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