ABUAD INTERNATIONAL JOURNAL OF NATURAL AND APPLIED SCIENCES
ISSN: 2955-1021
AIJNAS Volume 3, Issue 2, 2023 pp 16-24
https://doi.org/10.53982/aijnas.2023.0302.02-j

https://journals.abuad.edu.ng/index.php/aijnas

Hetero-Alkali Catalyst for Production of Biodiesel from Domesticated Waste: (Used Waste oil)

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Abstract
Biodiesel, a fuel derived from renewable sources, has garnered significant attention from energy researchers over the past two decades as a clean alternative to diesel fuel. This increased interest can be attributed to the alarming impact of climate change caused by the use of traditional diesel fuel. This paper focuses on showcasing the qualities of biodiesel produced from used waste oil and the positive impact on the alarming change in climate today. The observable characteristics of used waste oil for the synthesis of biodiesel in the presence of an ethanolic CaO-K_2O-SiO_2 base catalyst created from leftover palm kernel empty bunches was rigorously explored in this study. The catalyst obtained from Palm Kernel Bunch Stem (PKBS) was characterized using Scanning Electron Microscope (SEM), Fourier-Transform Infrared Spectroscopy (FTIR), Extended Range Spectroscopy - Flame Photometry (XRS-FP), Brunauer-Emmett-Teller analyzer (BET) isothermal adsorption and qualitative analysis. Reusability of catalyst and economic evaluation of the synthesized biodiesel were also evaluated. The quality of the Oil was identified through standard techniques by examining its physicochemical characteristics as well as other elements. According to the findings, the improved Used Waste Oil (UWO) characteristics met the specifications for oil required to produce biodiesel. The Used Waste Oil's physicochemical properties included the oil's physical condition as liquid/dark brownish at 28, acid value of 0.96 (mg KOH/g oil), FFA (% oleic acid), 0.48, iodine value of 152.00 (I2g/100g), and peroxide value of 5.1 milli-equivalent of peroxide/kg of oil, among others. The obtained catalyst demonstrated high basic strength with potassium oxide (61.63 wt/%) being the predominant component. At run 5 with 98.52 (%wt /wt), 65 minutes reaction time, 4.0 (%wt) catalyst amount, reaction temperature of 70 , and a 7:1 ethanol to oil ratio produced the highest biodiesel yield. The study concluded that, UWO can possibly be utilized as an economically benign feedstock for the production of biodiesel, and the resultant catalyst could potentially be employed in industries as bio-base.
***Keywords**: Palm kernel bunch Stem, Transesterification, Characterization, Used Waste Oil (UWO), Biodiesel, BET.*

INTRODUCTION

Some of the current world problems include the depletion of petroleum reserves, rising environmental pollutants, and increased demand for energy owing to the increase in population. Given a corresponding spike in world populations, energy demand will definitely grow shortly. As a result, countries worldwide are still interested in developing alternative fuel sources that can adequately meet the expanding need for energy [1].

Additionally, climate change, acid rain, and pollution are the most important globally environmental challenges caused owing to fossil fuels usage [2]. By 2030, a significant amount of carbon dioxide (emissions is projected. If the global temperature exceeds a 2 increase, the potential consequences could include the extinction of numerous species and a significant impact on human populations [3]. During the past 30 years, as more automobiles have been put on the road worldwide, the transportation sector has continuously expanded. Global transportation energy demand is predicted to increase at a 1.8% annual rate between 2005 and 2035 [4]. After industry, transportation is the second largest energy consumer worldwide, consuming 30% of the total energy supply, 80% of which is used for highways. This industry is expected to have the fastest growing energy utilization and currently consumes almost sixty percent of the world's oil. The majority of fossil fuels utilized in the transportation sector, roughly 97.6% come from oil, with natural gas making up a very small portion of this total. This industry is expected to be responsible for around three-fourths of the rise in oil demand between 2006 and 2030. There will probably be a significant increase in global usage of oil in transports as well as other sectors around 2005 and 2035 [4]. Due to limited fossil fuel supplies and the knowledge that increasing

greenhouse gas emissions are contributing to climate change, interest in boosting alternative energy sources [5] like biodiesel has surged.

As reserves of fossil sources of fuels are running out and people are becoming more aware that rising (CO_2) levels impact climate change, the passion for promoting renewable energy sources like biodiesel has increased [5]. Sustainable biofuel production is crucial for stopping climate change, protecting biodiversity, boosting local economies, especially in developing nations, and guaranteeing global energy security [6]. Renewable and clean fuels made from bioenergy feedstock have the potential to reduce global poverty, increase food security, and reduce greenhouse gas emissions.

Through biological, biochemical, or chemical processes which include anaerobic digestion, fermentation and, transesterification bioenergy like biodiesel, biogas, and bioethanol can be generated from biological material. In contrast, buried materials like artifacts residue or fossils of substances that decompose are what causes the production of coal, crude oil, and petroleum-based substances in the earth's crust. The fact that biodiesel is renewable and biodegradable makes it more environmentally friendly than conventional fuel. Due to the absence of sulfur and carbon neutrality of biofeed stocks, it also significantly reduces harmful emissions from engines like sulfur oxides (SOx) and carbon oxides (COx). Recently, biodiesel has gained a lot of attention, and many developing countries have changed their laws to encourage the use of renewable energy sources, particularly biodiesel produced from various source feed stocks in the agricultural, industrial, transportation, and other sectors [7]. Vegetable oils, animal fats, and oil waste are the three main categories under which the feedstocks used in the manufacturing of biodiesel can be categorized [8, 9,10]. Several oils, including non-edible ones like *Jatropha curcas Linn,* Shea butter, *Moringa oleifera*, kenaf, bitter melon, oil palm *(Elaeis guineensis),* kahalari melon, waste oils and pumpkin *(Cucurbita maxima)* seeds, have been employed recently in the production of biodiesel [11, 12, 13]. Price, consistency in the quality and chemical configuration of feedstock, regular availability of the feedstock, elasticity to expand supply, cost of transporting and preprocessing are important factors that must be considered when determining feedstock for biodiesel production.

Animal fat and vegetable oils cannot totally replace fossil diesel fuel at the current rate of production. Used Waste Oil (UWO) can be employed to produce biodiesel at a cheaper price and with a broader selection of feedstock alternatives. There are usually specific quantities of Used Waste Oil available everywhere. These are made locally when food are cooked or fried in oil, such as in hotels, restaurants, KFC, etc. Statistics on the quantity of accessible feedstock have never been available in Nigeria. However, the amount of Used Waste Oil that is thrown away annually in Nigeria can be assumed with some degree of knowledge. This translates to over 32 million metric tons of waste each year. Better disposal of Used Waste Oil offers a significant challenge since there are worries about disposal and possible degradation of both land and water resources. Some of the Used Waste Oil is employed to manufacture soap and as an oil-based supplement for animal feed. However, considerable quantities of Used Waste Oil are carelessly dumped in landfills and rivers, contaminating the environment.

MATERIALS AND METHODS

Used Waste Oil (UWO) and Palm Kernel Bunch Stem (PKBS)

The Used Waste Oil (UWO) was obtained from a cafeteria in Federal University Otuoke. At the same time, the empty palm bunch (EPB), free of edible seeds, was acquired from a palm kernel milling site located at Otuoke village Bayelsa, Nigeria. The UWO was preheated at a temperature 120 to allow liminality of the fluid for easy flow of the liquid. The heated oil was filtered to remove the adherent unwanted materials. The filtered oil (pure oil) was kept in a clean jar to be processed further.

Empty palm kernel bunches were manually separated from the stem, which then underwent a 32-day process of sun drying. A manually operated machine was employed to grind the dried stalk into powder. The obtained powder was sieved using 0.05 μm sieve mesh. The fine powder was then further calcined in a furnace for four (4) hours at 900. Thereafter, the calcined powder was sealed after cooling down to room temperature and placed in an airless, tightly closed container. Chemicals such as ether, ethanol, calcium chloride and hydrochloride used in this study, were of analytical standard and need no further refining.

Catalyst Characterization

BET Procedure

Prior to BET analysis, in order to remove any adsorbed loaded entities from the catalyst surfaces, the sample was preheated at 150^0C for 45mins under helium flow. The sample was exposed to a mixture of 5% CO_2 and helium, flowing at a rate of 25 ml/min, for duration of 40 minutes. The CO_2-temperature-programmed desorption (TPD) technique, using a BEL 132 Cat instrument from Japan, was employed to evaluate the alkaline properties of both the calcined powder and the mixed powder surfaces.

XRF Procedure for Catalysts Analysis

The elemental compositions of the samples were analyzed using a dispersive X-ray Fluorescence (XRF) spectrophotometer from AXS Bruker. The spectrophotometer employed an Rh source and a 2.2 kW power tube. The specific surface area was determined through N2 adsorption/desorption isotherm analysis using the Brunauer–Emmett–Teller (BET) method. This analysis was conducted on a volumetric adsorption analyzer at a temperature of 196°C. The surface area and pore size analysis were performed using the Belsorb III instrument from Japan.

FTIR Procedure for Catalysts Analysis

The Agilent Technologies Model Cary 122 630 FT-IR spectrometer, operating in the spectral range of 400 to 4000, functions based on a fundamental principle shared with NDIR analyzers. This principle revolves around the ability of various gases to absorb infrared (IR) radiation at frequencies specific to their molecular properties. However, unlike NDIR analyzers that focus on narrow frequency bands, FTIR spectroscopy employs a dispersive approach by conducting measurements across a wide spectrum of frequencies.This testing procedure involves aiming an x-ray beam at both calcined samples and mixed calcined samples, and then measuring the intensity of the scattered x-rays based on their outgoing direction. In a separate step, an FTIR spectrometer directs beams of infrared (IR) light at the sample and quantifies the amount of light absorbed by the sample and the frequencies at which this absorption occurs. The obtained spectra are then evaluated and matched against a reference database to identify the components present.

SEM procedure for catalysts analysis

Scanning electron microscopy (SEM) is employed to analyze the powder's morphology prior to consolidation. A sample stub for SEM is prepared by attaching two-sided carbon tape, onto which UHMWPE powder is sprinkled. A thin layer of light gold or platinum (100 Å) is deposited on the sample, and the SEM chamber is used to examine the samples. The flakes exhibit diameters ranging from 50 to 100. The surface morphology is revealed using field emission scanning electron microscopy (FE-SEM) with the QUANTA FEG 250 instrument.

Biodiesel Synthesis

UWO Biodiesel Production

The oil basis was kept at 300 ml, transferred to 1L reactor, and preheated at 60oC for about 2 hours on a hot plate magnetic stirrer. A predetermined amount of ethanol was mixed with catalyst before being added to the heated oil. It had three levels visible: the oil layer, the layer of whitish ethanol-oil-catalyst, and the layer of clear ethanol. The resulting mixtures were placed in a monitored setting to undergo chemical reactions until reaction time was reached. To separate and purify the product, it was moved to a dividing funnel. Glycerol was taken out of the funnel's bottom, and green diesel with catalyst was separated by filtration, rinsing with methanolic sodium hydroxide, and rinsing with distilled water. For biodiesel synthesis the wet green diesel was dried over calcium chloride, and the recovered catalyst was purified and reused. The quantity produced was determined in terms of % (w/w), as shown in equation below, and the end result was the methyl-ester referred to as biodiesel.

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

Analyzing Statistical Data

Results of biodiesel obtained along with variables constraint were analyzed with Microsoft Excel 8.0 to determine regression parameters and the level of significance of the variables.

Table 1: Experimental variables constraint

S/N	Reaction time(min)	Catalyst Conc. (%wt	Reaction temp.	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

RESULTS AND DISCUSSION

Physical Properties of UWO

The composition and content of oil were examined using physicochemical analysis to determine the quality of purified UWO purchased from a restaurant. Table 2 displays the final results. The produced UWO had a dark-brownish color, a specific gravity of 0.92, and a moisture content of 0.002% at ambient temperature. The oil's refraction index and color observation were consistent with earlier studies that had been published. According to Adepoju et al. [14], the moisture content was 0.045%, and the specific gravity was 0.91. The viscosity value observed in this study was 6.58 cP. This value is within the previously published range (15.15–15.9 cP) for discarded used oil [15].

Chemical Properties of UWO

The chemical properties of oil samples constitute one of many important considerations for evaluating their state and quality. Results regarding the chemical

characteristics of UWO are presented in Table 2. The low free fatty acid-FFA content of UWO discovered in this investigation suggests of the oil's strong resistance to hydrolysis. Adepoju and Olawale [14] found a high value of 8.52 mg KOH/g oil, whereas Kirubakaran and Arul [15] achieved 0.67 for the FFA of UWO. The oil's low acid value (A.V) of 0.96 mg KOH/g suggests that it is likely to have a prolonged shelf life and is suitable for consumption.

Table 2: Physicochemical and Other Characteristics of WUO

Parameters	Mean values
Physical properties	
Physical state at 28°C	Brownish yellow
Moisture content (%)	0.002
Specific gravity	0.92
Viscosity (cP) at 40°C	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 UWO exhibited a saponification value of 186.40 mg of KOH/g of oil, which indicates a high triglyceride content. This is within the acceptable range of 175-250 mg of KOH/g commonly seen in various seed oils, such as those derived from maize, mustard, raspberry, sunflower, safflower, and other seeds, as reported by Salimon and Yong [16]. Because of UWO's high iodine value (152.00 g of I2/100 g of oil), the oil is unsaturated. A low peroxide number refers to the amount of hydro peroxides in oil and denotes an excellent defence oxidation. . In this present study, low value of 5.1 milliequivalent (meq) of peroxide per kilogram of oil was discovered for UWOs in the oil. Since the UWO has a high iodine value and low peroxide value, it can be stored for a very long time without deteriorating.

Other Properties of UWO

Additional fuel characteristics, including cetane number, HHV, and API of the UWO, were identified (Table 2). The cetane rating gauges the fuel's ignition delay and combustion quality. The delay interval is shortened, and combustibility is increased with a higher cetane number. Low cetane gasoline smokes; therefore, it becomes harder to ignite. Industry standard for biodiesel requires minimum of 40 cetane numbers [17]. The UWO cetane number (41.38) found in this investigation demonstrated high fuel potential. An oil's higher heating value (HHV) is referred to as the amount of heat released when one mole of a compound entirely burns to CO_2 and H_2O at the initial temperature and pressure. The latent heat of the vaporization of water in the by-products of combustion was taken into account when calculating the HHV for the UWO in this investigation, which was 39.51 MJ/kg. The HHV found in this investigation fell within the previously reported range of values for vegetable oils (37.47–40.62 MJ/kg). The UWO was therefore a good option for usage as an industrial feedstock, as evidenced by the physicochemical properties of sample oil. The UWO API found in this present study was low at 22.30; however, transesterification of UWO might enhance its fuel characteristics.

Catalytic Characterization and Analysis

BET Analysis of Catalyst

Utilizing several analyzers, the catalyst's characterization was done. According to BET adsorption analysis, which was done via data reduction collection using the DA technique and employed nitrogen as an adsorbate with a mol. wt. of 28.013 for the catalyst mass of 0.12 g used for sampling, Fig. 1 shows the plots of various pore volumes acquired by the diameter of the sample. The most significant DA micro pore volume was found to be 0.318 cc/g, which relates to a 2.80 nm diameter of the pore at a best energy (E) of 0.807 kJ/mol. The sample started to develop a large surface area with an elevated level pore diameter as the plot approached its apex, which increased the reaction rate. Using the BJ adsorption technique, Fig. 2 illustrates the connection between surface area, pore diameter, and cumulative volume. The sample's surface area, pore volume, and pore diameter, which were all measured at 442.708 m^2/g, 0.217 cc/g, and 2.132 nm, respectively, reveal the sample's nature as a catalytic base for the generation of biodiesel.

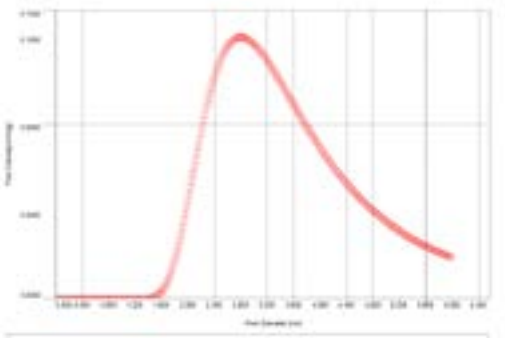

Fig. 1: Micro pore volume against pore diameter

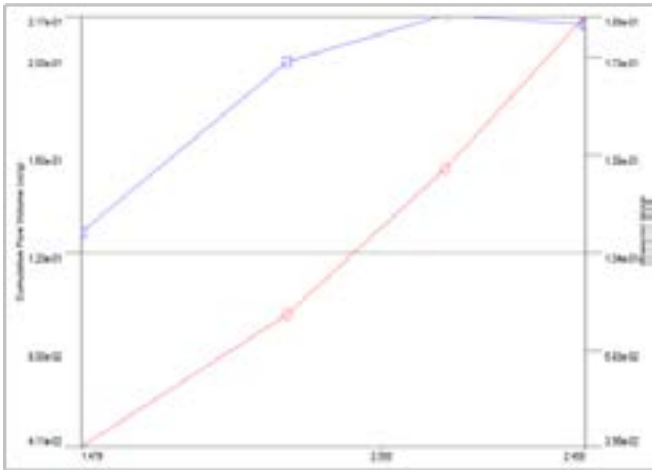

Fig. 2: Cumulative pore volume, surface area against pore diameter

XRS-FP Analysis

Based on the XRS-FP analysis of the catalyst, which showed the concentration method, the percentage mole, the intensity methodology used by Gaussian, and the components found in the catalyst using XRS-FP analysis, they are indicated in Table 3. The catalyst contains several different elemental compounds. Still main one is potassium oxide, which came about when the potassium carbonate in the stalk of the palm bunch completely decomposed during burning. The dominance of potassium oxide (61.63 wt. /%) on catalyst shows that it is a viable catalyst for manufacturing of biodiesel. Other chemical substances were present in trace amount and contributed to biodiesel synthesis. Nevertheless, the presence of SiO_2 (12.779 wt. / %) showed that powder catalyst possesses some acidic strength and also serves as a weak base.

Table 3: XRS-FP Analysis Report

Component	Concentration (wt. %)
SiO_2	12.779
Fe_2O_3	0.932
CaO	7.361
MgO	2.477
Al_2O_3	3.605
K_2O	61.628
Others	11.218
Total	100

SEM Analysis

The schematic representation of the structural view of the morphology revealed by the catalyst analysis by SEM is shown in Fig. 3a, 3b and 3c) at magnifications of 300x, 500x, and 1000x, respectively. The catalytic sample looked to have aggregated, porous-looking cracking structures with a shiny but partially separated characteristic that suggested they would be soluble in polar liquids.

The product may be regarded as glittering due to ZnO that was included in the synthesized catalyst. However, sample's appearance of paleness, brilliance, and opaqueness may be caused by the presence of SiO_2. The existence of K_2O and CaO, which denote the base catalyst nature for the creation of biodiesel, cause of the crystallized aggregates, rare white appearance, caustic alkaline, and brilliant glossy glaze at room temperature.

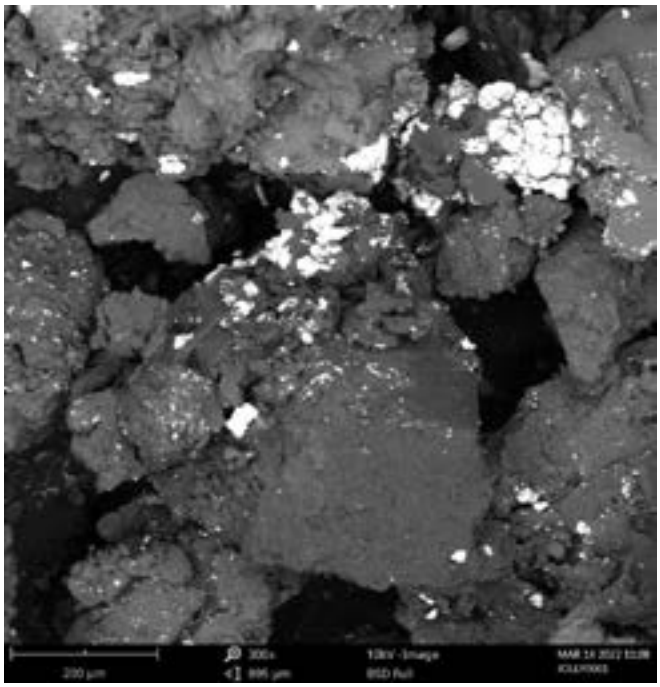

Fig. 3(a): SEM image at magnification of 300x

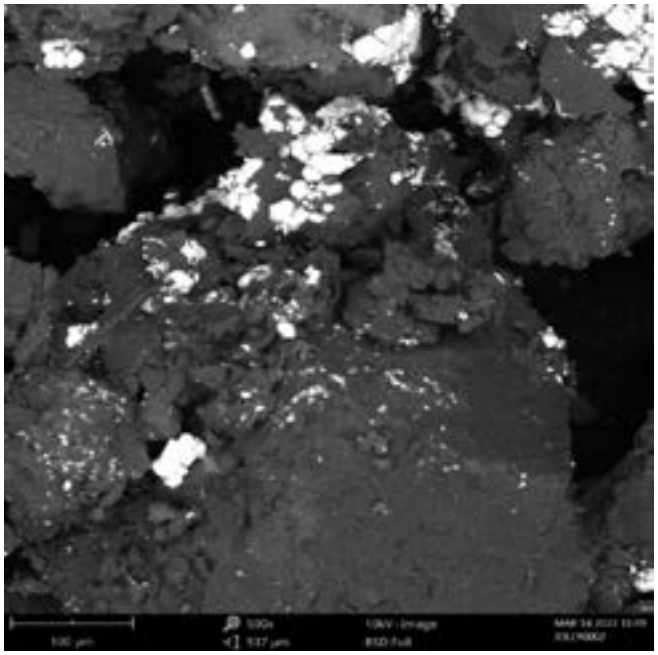

Fig. 3(b): SEM image at magnification of 500x

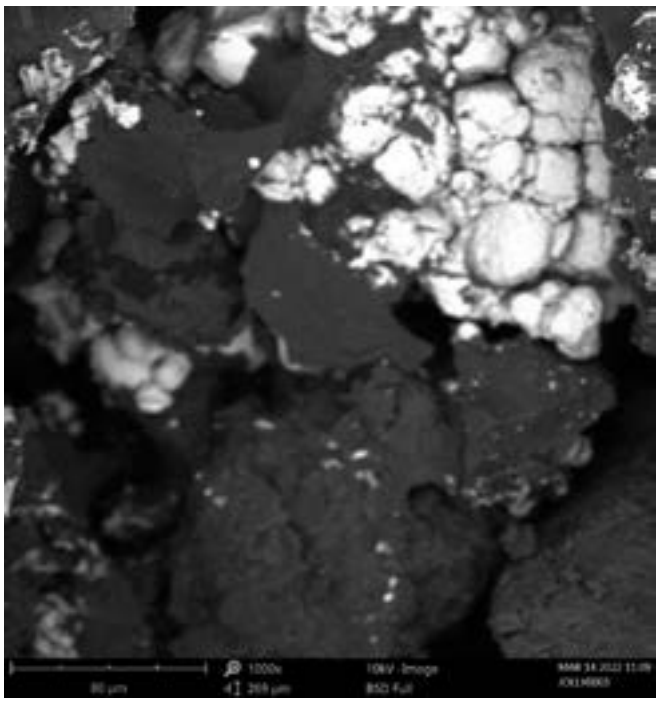

Fig. 3(c): SEM image at magnification of 1000x

FTIR Analysis

The frequency assignment approach was used for the interpretation of the spectra. Fig 4. Shows the FTIR spectrum and peaks indicated were assigned to various functional groups. The distinct components present at varying intensities and angular phases are used to classify infrared ray (IR) phases. The mid- infrared spectrum may generally be classify classified into four regions; the single bond region (2500-4000 cm^{-1}), the triple bond region (2000-2500 cm^{-1}), the double bond region (1500-2000 cm^{-1}), and the fingerprint region (600-1500 cm^{-1}) [18, 19]. The frequency bands between 3981.21, 3603.15 and 3518.28 cm^{-1} region corresponds to -OH stretching, which reveals phenols, while the peak bands 3888.62 cm^{-1} indicate the presence of C-H band stretch attributed to aldehyde. Also, the band observed at 732.97 and 879.57 cm^{-1} were for alkyl halides. The characteristics absorption band exhibited at 2654.14, 2407.24, 2260.65 and 1905 cm^{-1} were for functional group C=O, stretch which is attributed to the presence of aromatic ketones. The band observed at 3441.12, 3371.68, 3225.09 and 3294 cm^{-1} was for amine due to N-H stretch and 1697.41, 1629.97 and 1450.52 cm^{-1} was for primary amine due to N-H bend. Also observed bands at 1087.89 and 1041.60 cm^{-1} were for ethers due to C-O bend. The absorption at 3039.91 and 2931.90 cm^{-1} are ascribed to the presence of C-H bends, which indicates alkenes, and also peak at 1381.08 cm-1 is attributed to sulphonamide due to the S =O stretch.

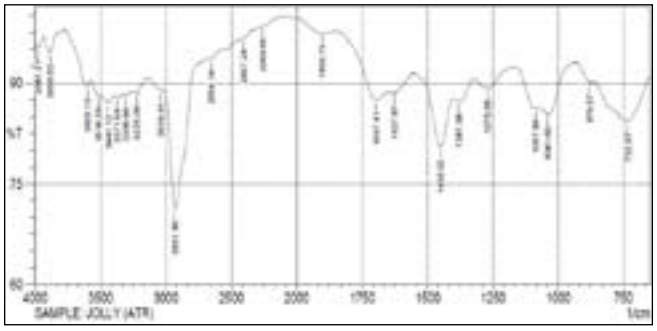

Fig. 4: FTIR Analysis of Catalyst

Transesterification of UWO to Biodiesel

The results of the biodiesel produced by UWO, together with a number of other components and biodiesel production, are shown in Table 5. Run 5 had the best biodiesel with a reaction time of 65 minutes, a catalyst loading of 4.0 (wt /%), a reaction temperature of 70 °C, and a molar ratio of 7:1 ethanol to oil. This yield was measured in weight per cent (%w/w).This shows that, the yield of biodiesel rises as the reaction conditions are enhanced. However, at temperatures exceeding 70, the yield decreases. This decline is attributed to catalytic nature of the bio-base, which leads to aggregation and clustering. Consequently, the burning temperature that produced high FAME conversion is the optimal burned temperature for catalytic conversion of powder to catalyst response for FAME conversion of 99% (wt.). In addition, biodiesel yield decreases because, at elevated ethanol boiling temperatures above the standard temperature, solvents evaporate during the reaction process.

Microsoft Excel Analysis (MEA)

The plot in Fig. 5 revealed that variables considered in various ranges were significant, with a coefficient of determination (R^2) of 0.8164. R^2 values greater than 0.800 are regarded significant since they demonstrate a high level of agreement between the experiment's findings and the factors considered [20]. Plotting model equations against yield showed the slope and intercept of various factors.

Table 5: Biodiesel Yield and Constraint Variables

S/N	React time (min)	Catalyst conc. (%wt.)	Reaction temperature	Ethanol-Oil molar ratio (vol./vol.)	Biodiesel yield(%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

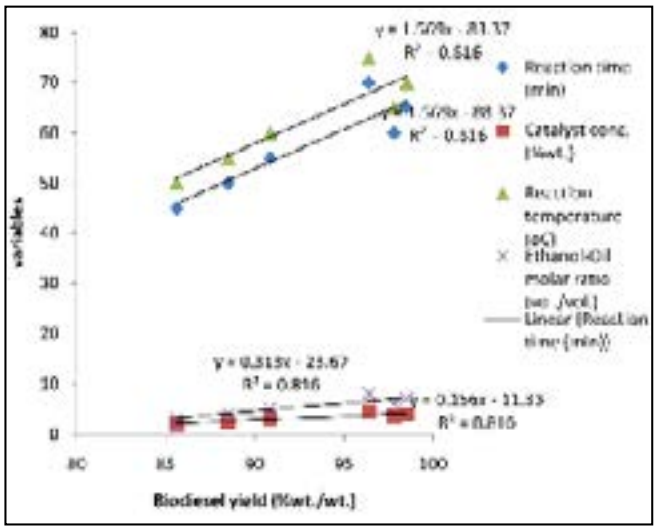

Fig 5: Excel plots of biodiesel yield against the variables

Physicochemical Properties of Biodiesel

Based on its content and composition, the physicochemical characteristics of the produced biodiesel were evaluated for acceptability using AOAC procedures [21]. Table 6 presents the results of the product's characteristics. Results presented in Table 6 demonstrate that the biodiesel produced meets the requirements of ASTM D6751 and EN 14214, two American and European biodiesel regulations. In this study, it has been demonstrated that the biodiesel produced has the potential to serve as a viable alternative to conventional diesel fuel commonly utilized in industrial and commercial applications.

Table 6: Biodiesel's Physicochemical Composition and Other Characteristics

Parameters	Biodiesel	ASTM D6751	EN 14214
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 (g I_2/100g oil)	116.00	-	120 max
Peroxide value (meq O_2/ kg oil)	2.48	-	-
Other properties			
Cetane number	50.14	47 min	51 min
Diesel index	55.75	50.40	-
API	38.98	36.95	-
Higher heating value (HHV)	36.15	-	-

CONCLUSION

The study successfully established that Used Waste Oil possesses properties similar to oil and can be utilized in the production of biodiesel. Additionally, the stem of charred palm kernel bunches showed a significant increase in potassium content (61.628% wt.), which can serve as a bio-base for biodiesel synthesis. The statistical analysis conducted using MS-Excel confirmed the significance of the selected variables, as indicated by the regression parameter R-square exceeding 0.8. The produced biodiesel exhibited characteristics comparable to those of conventional diesel, meeting the specified biodiesel requirements. Consequently, the study's findings suggest that biodiesel derived from leftover cooking oil can effectively replace regular gasoline.

FUNDING

There was no funding for this study.

DECLARATION OF COMPETING INTEREST

No conflicting interests are disclosed by the authors. .

ACKNOWLEDGMENT

The authors acknowledge two anonymous reviewers for their perceptive criticism and beneficial suggestions.

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