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Production of Biodiesel from Waste Cooking Oil Using KOH/Al_2O_3 as a Heterogeneous Catalyst

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Abstract: The catalytic performance of potassium hydroxide supported on alumina (KOH/Al_2O_3) for transesterification of waste cooking oil (WCO) was investigated. XRD, FTIR, and SEM techniques were used to characterize the catalyst after it had been synthesized using the wet impregnation method. The properties of the WCO and the produced biodiesel were evaluated. The main fatty acids of WCO were linoleic (45.61%), elaidic (33.86%), palmitic (10.32%), and stearic acid (4.80%), the acid value (2.29 mg KOH/g), FFA (1.15%), density at 25 °C (0.91 g/cm^3), viscosity at 40 °C (34.09 mm^2/s), water content (0.0017%), and flash point (206 °C). The XRD pattern of the catalyst showed diffraction peaks of $KAlO_2$ attributed to the orthorhombic crystal system having a 12.46% degree of crystallinity. The SEM micrographs confirmed the amorphous nature of the catalyst. The presence of K–O and Al–O bonds in the catalyst was confirmed by the FTIR. A biodiesel yield of 86.6% was achieved with the following variables: oil to methanol ratio of 1:4, catalyst loading of 1.5% (w/v%), reaction temperature of 55 °C, and a reaction time of 1 h. The results appeared to show a decreasing pattern of yield after 4 cycles of reaction from 86.60 to 51.50%. The fuel properties were density (0.8919 g/cm^3), viscosity (7.428 cSt), flash point (65 °C), acid number (0.54 mg KOH/g) and water content (0.0196%). Furthermore, the properties of the prepared biodiesel have been found to comply with the ASTM and EN standard specifications. This catalyst showed promising results for manufacturing biodiesel from low-cost feedstock.

Keywords: Biodiesel, waste cooking oil, catalytic performance, Heterogeneous catalyst, wet impregnation.

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1. INTRODUCTION

The majority of the world's energy demands are met by petroleum-based fuels, but they are finite and unsustainable. New alternative sources for petroleum-based fuel have been extensively researched as a result of depletion of petroleum reserves and rising environmental concerns (1). Biodiesel is a renewable energy source that can replace fossil-based diesel and can reduce the drawbacks of diesel emission (2).

The use of biodiesel as a fuel source has several advantages, in addition to being a renewable, sustainable, biodegradable source, generally non-toxic and environmental friendliness, as it reduces CO_2 emissions and hazardous compounds such as arithmetic, sulphur, particulate matter and NO_x, biodiesel shares a variety of similar physical and chemical properties with petroleum diesel (3-5). Biodiesel is superior to diesel fuel due to its higher oxygen composition, high cetane content, higher flash point, cleaner combustion, and appropriate lubrication characteristics. Therefore, typical compression-ignition engines may use pure biodiesel or biodiesel blends without the need for substantial modifications (6,7). Biodiesel is a clear liquid with a light- to dark-yellow color. It has a boiling point of more than 200 °C, a flash point of 100 to 170 °C, a cloud point of -3 to 12 °C, a pour point of -15 to 16 °C, a kinematic viscosity at 40 °C of 1.9–6.0 mm^2/s, a distillation range of 195–325 °C, a vapour pressure (mm Hg at 22 °C) less than 5, a freezing point (-3 °C), Cetane number of 48–60, lower calorific value of 37300–38500 kJ/kg, and upper calorific value of 39300–39600 kJ/kg; oxygen content of 10–11% (wt%); sulfur content of 0.05% (wt%) (5,7,8). The oxygen content of biodiesel enhances the engine's combustion process and contributes to reducing exhaust emissions. Diesel fuels with high cetane values have a variety of benefits, including improved cylinder combustion, easier engine running in cold weather, and longer engine life (9). Dilution, micro-emulsion, pyrolysis, and transesterification methods were proposed as solutions to lower the viscosity of vegetable and animal oils, reduce the molecular weight, improve the volatility of the fuel, and decrease emissions such as HC and CO. The most widely utilized among these processes is the transesterification method (7, 10).

Biodiesel is made from natural oils or animal fats and alcohol via a transesterification reaction, which converts triglycerides into esters known as fatty acid methyl esters (FAME) (11). Commercial production of biodiesel is possible from a wide range of renewable biomass feedstocks, including edible oils. (e.g. peanut, rapeseed/canola, soybean, sunflower, palm, corn, cottonseed, coconut, mustard, olive, and sesame oils) and non-edible oils (e.g. jatropha, castor, neem, jojoba, passion seed, and moringa oils), animal fats (e.g. pork lard, beef tallow, poultry fat, fish oil, chicken fat), and waste cooking oils (12, 13). Competition with food is brought on by the production of biodiesel from conventionally grown oilseed crops. Due to the increasing demand for edible oils for food and their current high cost for use as fuel, the use of non-edible vegetable oils and waste oils is essential in developing countries (12). WCO refers to cooking oils that have been used in food preparation but might no longer suitable to use in food production. WCO may come from domestic, commercial or industrial sources. A waste stream such as WCO requires proper management, and irresponsible disposal can result in problems (14).

Biodiesel is defined as fatty acid methyl esters (FAME) derived from the transesterification of triglycerides (vegetable oils or animal fats) with alcohol and a suitable catalyst (15). The main catalysts utilized can be classed as homogeneous or heterogeneous. Homogeneous catalysts (base/acid) react with the reaction mixture in the same liquid phase are commonly used for biodiesel production from different feedstocks under mild reaction conditions. However, these traditional catalysts cause several technical problems i.e. reaction corrosion, large amounts of waste water production, separation problems, thereby increase the cost of biodiesel production, whereas heterogeneous catalysts react with the reaction mixture in a separate phase, usually as a solid. Heterogeneous catalysts are non-corrosive, environmentally beneficial, and a green process. They may be recycled and reused multiple times, making biodiesel production more cost-effective; thereby resolve many of problems associated with homogeneous catalyzed biodiesel technology (16). The aim of this research was to investigate different transesterification process conditions; oil to methanol molar ratio, temperature, catalyst loading and reaction time on biodiesel production from WCO using potassium hydroxide supported on alumina as a heterogeneous catalyst. The reusability of the catalyst was also investigated over several consecutive runs and properties of the produced biodiesel were evaluated based on standards set of ASTM D6751 (17) and EN 14214 (18).

2. EXPERIMENTAL SECTION

2.1. Materials

Refined waste peanut oil was collected from Royal Broast fast food restaurant in Khartoum, Sudan. The oil was used once for frying. The solid particles and other impurities were filtered through normal sieves and then heated to 120 °C for 2 h to eliminate water traces. Then, the pre-treated WCO was stored in a clean glass air tight container. All chemicals used in this study were of analytical grade and used without any purification include: methanol (99%); ethanol (99%); potassium hydroxide (KOH) with purity 99%; aluminium oxide (Al_2O_3); were purchased from Merck Co. (Dannstadt, Germany)

2.2. Characterization of the WCO

The density, acid value, kinematic viscosity, flash point, water content and color the samples were assessed using the methods described by ASTM International (19-24). The fatty acids composition of the WCO sample was determined using (GC/MS-QP2010SE, Shimadzu, Japan) equipped with capillary column (Rtx-5MS- 30 m × 0.25 mm I.D × 0.25 µm) with the following analytical conditions: injector temperature was 300 °C; the injector was operated in the split mode; oven temperature was programmed from 60 °C to 300 °C at 10 °C/min; the carrier gas was helium at flow rate of 1.6 mL/min; volume of injection was 1 µL. The MS conditions were ion source temperature 200 °C, the interface temperature was 250 °C, the mass scan range was 40 – 500 m/z, and the total run time was 34 min. The spectra of the components were compared with the database of spectrum of known components stored in the GC-MS library (NIST).

2.3. Preparation of the Catalyst

Alkaline modified alumina was prepared via wet impregnation of aqueous solution of potassium hydroxide and alumina as a support. About 25 g of KOH was dissolved in 100 mL of distilled water. Then, the required amount of alumina powder was added to the solution (1:4 weight ration of Al_2O_3 to KOH). The slurry was vigorously stirred for 24 h, on magnetic stirrer at room temperature. The obtained precipitate was aged in mother liquid overnight (~24 h) at 40 °C. The solid product was separated by filtration using a vacuum filter system. It later dried in a 110 °C oven for 12 h. The dried solid obtained was calcined at 400 °C for 4 h in a muffle furnace (25).

2.4. Characterization of the Catalyst

The bulk structure, composition and crystalline structure of the synthesized catalyst were determine using X-ray diffractometer (XRD) (GNR, Italy), equipped with Cu Kα radiation (40 kV 40 mA) with a wavelength (λ) of 1.54 Å. The sample was scanned from 4 to 90° (2θ) and the phases were identified using X, Pert Highscore plus software by searching and matching the obtained patterns with the JCPDS (Joint committee of powder diffraction standards) database file. The morphology of the catalyst was studied by scanning electron microscopy (SEM), the micrographs were obtained by using a (TESCAN VEGA3) microscope; the elemental analysis was performed by energy dispersive spectroscopy (OXFORD EDS) detector. FTIR analysis of the catalyst was carried out using potassium bromide (KBr) powder method on a FTIR spectrophotometer (IRT racer-100, Shimadzu, Japan) with a resolution of 2 cm^{-1} at 500 – 4000 cm^{-1} range

2.5. Transesterification Reaction

The transesterification reaction of WCO was carried out with various catalyst loading, different methanol / oil molar ratios, various reaction temperatures and reaction time. Before starting the transesterification reaction, the oil was heated to 70 °C then cooled to the reaction temperature between 50 – 60 °C. The specified amount of oil, methanol and catalyst were carefully measured and poured in a 250 mL three-neck round-bottom flask equipped with a magnetic stirrer at 400 rpm and a water-cooled condenser. The reaction was allowed to proceed for a specified reaction time (1 – 2 h). Then reaction mixture was cooled to 25 °C. After cooling, the mixture was centrifuged at 3000 rpm for 10 min and the catalyst was filtered using a vacuum filter. The liquid was left in a separating funnel for 3 h for separation. The glycerol layer was discharged, and the excess methanol was washed from the biodiesel layer five times using warm distilled water (80 – 100 °C) in a separatory funnel (The amount of water was about 30% of fuel volume). Finally, the produced fatty acid methyl esters ($FAME_S$) were collected and the biodiesel yield was calculated using Equation 1.

$$Biodiesel\ yield\,(\%) = \frac{Weight\ of\ FAMEs}{Weight\ of\ oil} \times 100 \quad \text{(Eq.1)}$$

The catalyst was isolated from the reaction product in the first reaction, washed with methanol three times and dried at 60 °C in an oven for 2 h (25) and reused in the transesterification reaction in optimal conditions, the procedures were repeated three times.

2.6. Product Analysis

The resulting biodiesel from the transesterification reaction was analysed using appropriate ASTM standard methods. The following fuel properties were determined: density was determined using a 25 cm^3 pycnometer at 15 °C temperatures, according to Standard ASTM D-4052, viscosity, HK-1005A kinematic viscosity apparatus was used at a temperature of 40 °C according to Standard ASTM D-445, acid number was determined by dissolved the sample in a 50 cm^3 of ethanol and the acid presented in the sample was titrated with 0.1 N potassium hydroxide solution. Flash point was determined using a method ASTM-D93, closed cup, at which application of an ignition source causes the vapours of a specimen to ignite under specified conditions of test. Water content of the sample was determined by aquamax Karl Fischer coulometric titration, pre-titration is performed automatically (19-24). The characteristics of the produced biodiesel were compared with ASTM D6751 (17) biodiesel standard and EN14214 (18) fossil diesel standards to confirm their acceptability as a fuel in diesel engines.

3. RESULTS AND DISCUSSION

3.1. Characterization of the WCO

In this study, the WCO used as a feedstock was characterized based on its physicochemical properties. The properties were summarized in Table 1. Free fatty acid (FFA) was measured to determine the availability of WCO for using in transesterification directly. Previous studies have suggested feedstock FFA content of less than 2% for transesterification process (26), and if the acid value is very high, WCO requires a pre-treatment to decrease its fatty acid content before the conversion to biodiesel to prevent saponification reaction. The obtained results of showed an acid number of 2.29 mg KOH/g of oil and FFA of 1.15%, this implies that the WCO could be directly converted into biodiesel via transesterification process. Regarding the density, it was determined at 25 °C to be 0.911 g/cm^3, this value was in an agreement with those recorded by Mahesh et al. (1) and Yusuff et al. (27). The kinematic viscosity of WCO at 40°C was determined to be 34.09 mm^2/s, and this value was in the range from 39.07 to 28.8 mm^2/s, reported in previous studies (26,28,29). The flash point in this study was lower than that reported in the study done by Anisah et al. (26) as 247.7 °C. The value of the water content of the oil was much lower than the value determined in the previous study (30), the oil feedstock should be anhydrous (water content <0.3%). These data show that this feedstock is appropriate to obtain high ester yields.

Ten fatty acids were identified in WCO as shown in Table 2, the main fatty acids present in the WCO were linoleic acid (45.61%) followed by elaidic acid (33.86%), palmitic acid (10.32%) and stearic acid (4.80%). Approximately 45.61% of the fatty acids were found to be poly-unsaturated, 34.55% mono-unsaturated and 16.47% saturated fatty acids. The four main components were found in WCO: linoleic, elaidic, palmitic and stearic acids, were higher than that obtained in a previous study (31).

Table 1: Physicochemical properties of WCO.

Property	Experimental Values*
Acid value (mg KOH/g)	2.29
FFA (%)	1.15
Density at 25 °C (g/cm^3)	0.911
Viscosity at 40 °C (mm^2/s)	34.09
Flash point (°C)	206
Color	<4.0
Water count (%)	0.0017

* Values recorded as mean value.

Table 2: Chemical composition of WCO.

Fatty acid	IUPAC name	Formula	Structure	wt%
Linoleic	9,12-Octadecadienoic acid	$C_{18}H_{32}O_2$	C18:2	45.61
Elaidic	9-Octadecenoic acid	$C_{18}H_{34}O_2$	C18:1	33.86
Palmitic	Hexadecanoic acid	$C_{16}H_{32}O_2$	C16:0	10.32
Stearic	Methyl stearate	$C_{18}H_{36}O_2$	C18:0	4.80
Behenic	Docosanoic acid	$C_{22}H_{44}O_2$	C22:0	0.80
Palmitoleic	9-Hexadecenoic acid	$C_{16}H_{30}O_2$	C16:1	0.45
Myristic	Methyl tetradecanoate	$C_{14}H_{28}O_2$	C14:0	0.19
Lignocerc	Tetracosanoic acid	$C_{24}H_{48}O_2$	C24:0	0.19
Lauric	Dodecanoic acid	$C_{12}H_{24}O_2$	C12:0	0.06
Phthalic	1,2-Benzenedicarboxylic acid, bis(2-methylpropyl)	$C_{16}H_{22}O_4$	C16:0	0.05

* The obtained results in terms of fatty acid methyl esters from GC-MS library data system reviewed and the results listed out in the form of fatty acid chains.

3.2. Characterization of the Catalyst

The XRD analysis was conducted to determine the variation of the structure, amorphous content and degree crystallinity of the catalyst depicted in Figure 2. The XRD pattern of the catalyst show a diffraction peaks of $KAlO_2$ appears at 2θ of 17.06°, 22.89°, 32.86°, 33.71°, 34.11°, 37.24°, 37.66°, 39.52°, 42.67°, 68.16° and 89.83° which were attributed to orthorhombic type of $AlKO_2$ formed on the catalyst surface with lattice parameter (in Å) of 5.456, 10.9617 and 15.4893 for a, b and c respectively (Table 3). The $AlKO_2$ levels were observed to be consistent with the data obtained by the authors in (32), whereby some diffraction were similar and the other rapprochement to that found in this study. The similar peaks were 17° and 34°. Meanwhile, the characteristic peaks of alumina at angles of 2θ = 37° and 67.21° were almost unchanged on the XRD pattern, indicating that Al_2O_3 still retained its amorphous structure. A similar pattern was obtained by Santos et al. (32). As in a previous studies (33, 34) the formation of K_2O_2 is clearly observed at 2θ = 31°, 33º, 34º and 42° (JCPDS file (50-05241). In this study the catalyst was successfully converted to desired species (K_2O_2) for the reaction.

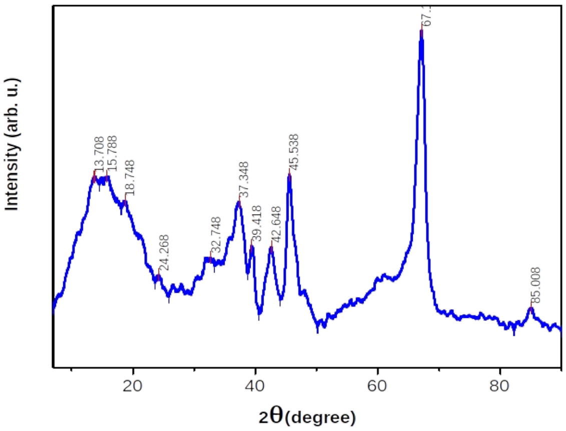

Figure 1: XRD pattern of the of KOH/Al_2O_3 catalyst.

Table 3: XRD matched peak analysis report.

Property	Amount
Formula sum	Al K O_2
Peaks matched	17
Crystal system	Orthorhombic
Unit cell (Å)	a = 5.4560, b =10.9617 and c = 15.4893
Calc. density (g/cm³)	2.813
Degree of crystallinity (wt%)	12.46
Amorphous content (wt %)	87.54

The particle morphology of the catalyst was captured via SEM, and the micrograph of KOH/Al_2O_3 presented in Figure 2 shows the particles of aluminium oxide impregnated by potassium hydroxide. The catalyst has irregular crystallinity and small sized crystals. Figure 2 clearly shows that there are no definite morphologies in the sample. It looks that the particles were agglomerated and form a cluster.

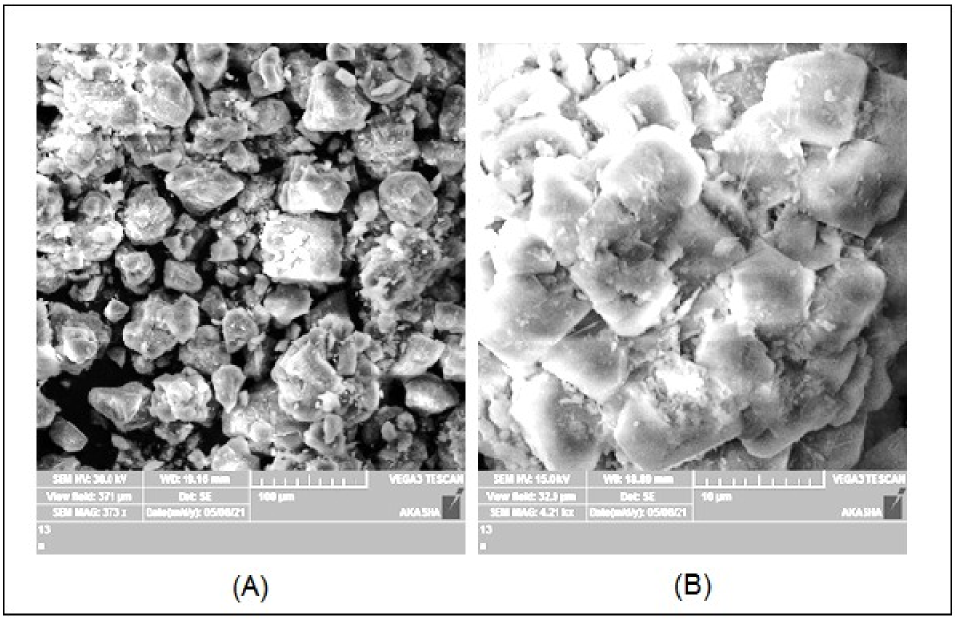

Figure 2: SEM image of KOH/Al_2O_3 catalyst.

The FTIR spectrum of the prepared KOH/Al_2O_3 in the range 400 – 4000 cm^{-1} was shown in Figure 3. The spectrum shows an intensive band at 3500 cm^{-1} belongs to the ν_s(Al)O-H stretching vibrations, three moderate bands observed at the frequencies of 1149, 1083 and 1026 cm^{-1} can be attributed to the K-O bond and the four weak bands at 1732, 871, 813 and 617 cm^{-1} are the combination bands. The absorption edge of the hydroxyl bands on the surface was found at 1732 cm^{-1}. The bands appearing in the region of 600 – 870 cm^{-1} include the tension of Al-O bonds, whereby aluminium exhibits vibrations extended in the region from 750 to 850 cm^{-1}. These absorption bands agree precisely with the ones previously reported in the literature (35) that introduced FTIR spectrum closed to that obtained in this study.

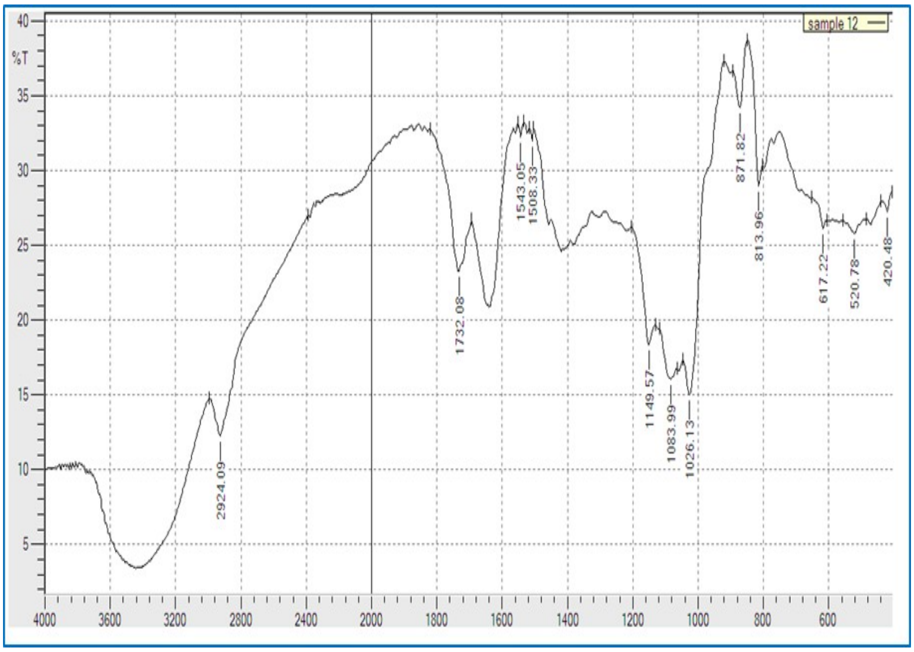

Figure 3: FTIR spectrum of KOH impregnated into Al_2O_3.

3.3. Transesterification Reaction

3.3.1. Effect of WCO/methanol molar ratio

The study of the influence of the WCO/methanol molar ratio (1:4; 1:5 and 1:6) was carried out at temperature of 50 °C for 1 h of reaction with 1% catalyst loading. Results in Figure 4 shows that the highest conversion was achieved with 1:4 WCO/methanol molar ratios, but afterwards shows a decline in conversion rate with the WCO/methanol molar ratio going from 1:5 to 1:6. An excess methanol increases the solubility of the by-product (glycerol) which then may initiate the reversible reaction to reduce the conversion. The optimum methanol/oil molar ratio was observed at 1:4. This result is in line with the work of Gimbun et al. (36); they produced biodiesel from rubber seed oil and found that the 1:4 ratio was the best ratio for generating good biodiesel quality.

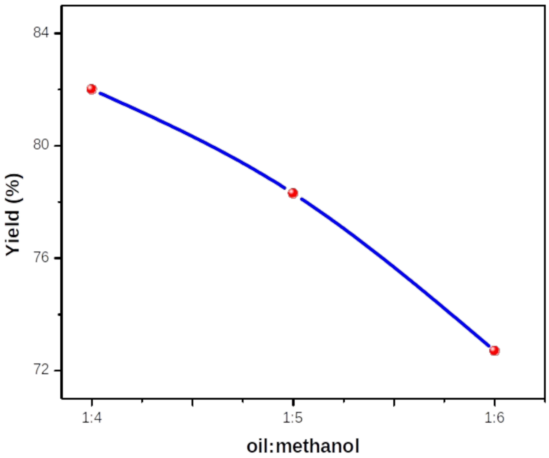

Figure 4: Effect of WCO /methanol molar ratio on biodiesel production.

3.3.2. Effect of the catalyst loading

The effect of KOH/Al_2O_3 catalyst loading (1 to 2 w/v %) on biodiesel yield was evaluated at oil to methanol ratio of 1:4, temperature of 55 °C with 1 h reaction time. It was shown in Figure 5 that the biodiesel yield increases from 84.5% to 86.6% as the catalyst loading increases from 1 to 1.5% and decreased to 81.1 % afterwards. This might be due to the formation of more triglycerides in the saponification side reaction as a result of the excessive addition of catalyst (37). It was found that the optimum catalyst loading in this study was 1.5% with biodiesel conversion of 86.6%.

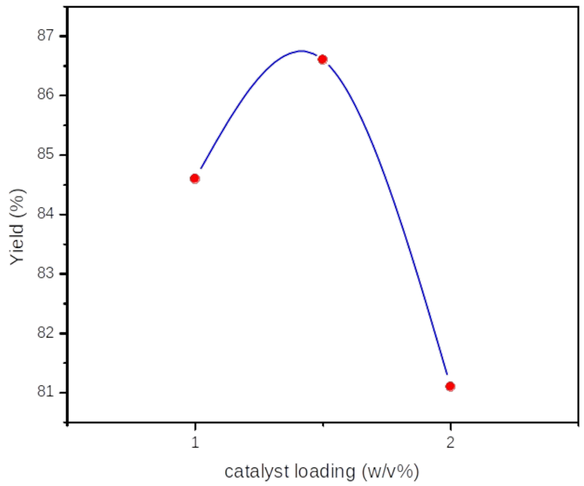

Figure 5: Effect of catalyst loading in biodiesel yield.

3.3.3. Effect of temperature

The effect of temperature on the transesterification of WCO was investigated by carrying out the experiments at a fixed WCO/methanol ratio of 1:4 and amount of catalyst of 1% (w/v %) with a reaction time of 1 h. The experimental temperature was in the range from 50 °C to 60 °C, as shown in Figure 6. It was observed that increasing the reaction temperature had a favourable influence on the degree of transesterification. With a rise in temperature, the conversion rose at a faster rate. At higher reaction temperatures, however, there was an opportunity of methanol loss and product darkness increased. High reaction temperatures also increased the assembly cost of biodiesel. The optimum temperature for this reaction was found to be 55 °C due to the lower boiling point (64.5 °C) at which methanol evaporates (38).

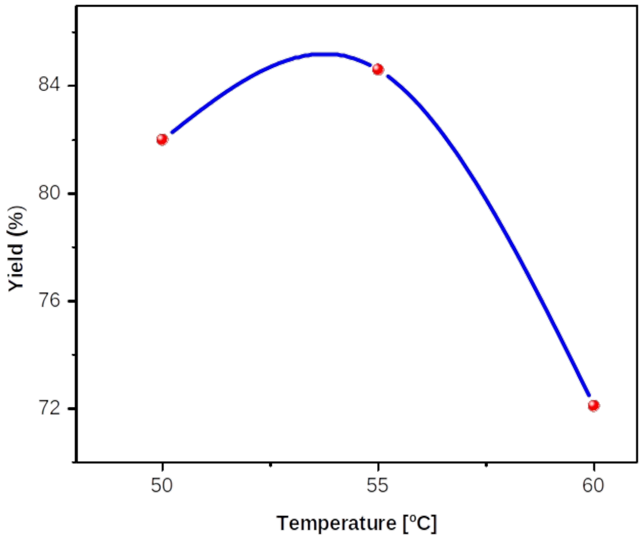

Figure 6: Effect of temperature in transesterification of WCO.

3.3.4. Effect of reaction time

The effect of reaction time was studied as it had a significant impact on the transesterification of waste cooking oil. The reaction time varied from 1 to 2 h in oil to methanol ratio of 1:4 with 1.5% catalyst loading at temperature of 55 °C. The transesterification reaction produces the maximum biodiesel yield of 86.6 % after 1 h of reaction. Almost all the oil has been transformed into methyl esters leading to a maximum conversion. However, the yield decreased after 2 hours of the reaction. This may be due to the longer reaction time led to a reversible process that led to an increase in glycerides (37).

3.3.5. Reusability test

Catalyst reusability is an important aspect in the development of biodiesel. The reusability of the catalyst was determined at the optimum conditions for four cycles as shown in Figure 7 it was noticed that after cycle 1 with yield of 86.6%, the cycle demonstrated a declining pattern of yield of biodiesel which was 68.7%, 60.6% and 51.5% for cycle 2, 3 and 4 respectively where the yield of biodiesel decreased due to a decrease in catalytic activity. The reduction in catalytic activity was caused by the loss of catalyst during the filtration process, activation, and morphology change of the catalyst used (2).

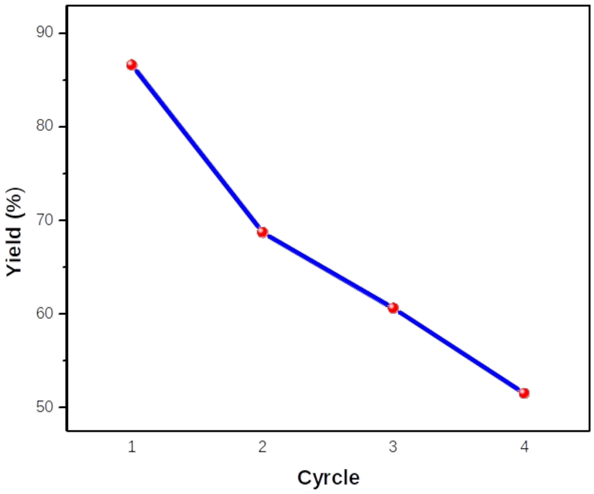

Figure 7: Reusability test for KOH/Al_2O_3 catalyst.

3.3.6. Characterization of produced biodiesel from WCO

By applying the aforementioned optimum conditions, the obtained biodiesel yield was ~ 86.6%. At molar ratio of 1:4, temperature 55 °C, catalyst loading 1.5% in 1 h. The produced biodiesel was evaluated on the basis of its fuel properties compared to ASTM 6751 (17) & EN 14214 (18) standards. Density, viscosity, acid number, flash point, and water content are the most important properties of biodiesel to measure the quality of the product.

Based on the data shown in Table 4 there isn't very much different from the ASTM 6751 (17) & EN 14214 (18) standards, except the viscosity still doesn't meet with the standards. This viscosity value is lower than that generated by Fathallah & Pinto, (39) and higher than Ashok et al. (40) and Ahmed et al. (41). However, because biodiesel is always combined with petroleum diesel in engine applications, the biodiesel in this study can be used as fuel (42).

Table 4: WCO biodiesel properties compared with ASTM & EN14214 Standards.

Property	WCO biodiesel	ASTM D6751	EN 14214
Density at 15 °C (g/mL)	0.8919	0.82-0.9	0.86-0.9
Viscosity at 40 °C (cSt)	7.428	1.9-6.0	3.5-5.0
Acid value (mg KOH/g)	0.54	0.50 max	0.50 max
Flash point (°C)	65	130 min	120 min
Color	<1.5	-	-
Water count (%)	0.0196	0.03 (v/v%)	500 (mg/kg)

4. CONCLUSION

The potential of potassium hydroxide supported on alumina (KOH/Al_2O_3) as heterogeneous catalyst for biodiesel production from peanut waste cooking oil (WCO) has been evaluated. The determined physicochemical properties of WCO make it an attractive alternative feedstock for biodiesel production. The optimal reaction conditions were: oil to methanol ratio 1:4 catalyst loading of 1.5%, reaction temperature of 55 °C, reaction time 1 h, with a yield of 86.6%. The fuel characteristics of the synthesized methyl ester from the WCO at the optimal process conditions were found to be in accordance with the ASTM 6751 & EN 14214 standard specifications. Reusability test shows that the synthesized catalyst is reusable up to 4 times. The catalyst employed gave a good performance and easily separates from the product mixture. Thus, WCO is a promising feedstock for biodiesel and (KOH/Al_2O_3) is a promising catalyst for methyl ester synthesis via heterogeneous catalytic transesterification under milder reaction conditions.

5. CONFLICT OF INTEREST

The authors declare no conflict of interest.

6. ACKNOWLEDGMENTS

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