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# Biodiesel Production from Waste Cooking Oil by Alkaline Transesterification Process

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## Abstract

The depletion of fossil fuels, coupled with frequent instability in their prices and environmental concerns related to the drawbacks of full dependence on conventional fossil fuels. considerably, biodiesel is found to be an ideal source of fuel that can efficiently replace conventional fuel diesel. Among various raw materials that can be exploited to produce biodiesel, waste cooking oils are highly recommended as an economically attractive option for biodiesel production. However, the high content of free fatty acids (FFA) in waste cooking oils presents the main barrier to the transesterification process. Therefore, in the current study, two types of waste cooking oils (WCO) collected from homes and restaurants were tested and the percentage of FFA in them was determined through a titration process. The results showed that the percentage of FFA in used cooking oils collected from homes was (1.1932%), in contrast with (2.0096%) in WCO observed from the restaurant samples. Thus, WCO collected from homes was candidate to produce biodiesel and it was characterized using FTIR. Transesterification process was carried out in the presence of an alkaline catalyst (KOH), and the yield of biodiesel was reached to 85.4% in the presence of a 1% catalyst at 65 °C degrees and methanol to oil ratio of 6:1 for 90 min. The resulting biodiesel was characterized using FTIR and GC-MS, its properties such as density, kinematic viscosity, cloud point, and pour point were diagnosed as good biodiesel properties based on ASTM standards. The results showed that the alkali-catalyzed transesterification process is an effective way to produce biodiesel from WCO as a source of energy in addition to reducing environmental pollution.

*Keywords: Biodiesel; Waste cooking oil; Transesterification; Alkaline catalyst; FAME*

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## INTRODUCTION

The world has faced significant challenges in recent decades due to the dual crises of petroleum consumption. The issues associated with conventional energy, widely acknowledged as the main contributor to global warming, and the looming threat of energy depletion driven by excessive fossil fuel consumption stemming from population growth. This has dramatically influenced various sectors, including environmental, health, and economic concerns [1, 2]. Thus, to fulfill the growing demands for clean energy, there is a worldwide challenge to explore alternative renewable, eco-friendly, biodegradable, low-cost sources of energy to meet the growing demands for energy and consequently mitigate the impacts of greenhouse emissions triggered mainly by reliance on fossil fuels [3, 4] Among several types of renewable energy are wind power, geothermal energy, solar energy, ocean energy and hydropower.

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Biomass derived energy, known as biofuel, has drawn more attention because of its features including the flexibility of feedstock, and inexpensiveness in synthesis. Biofuel refers to liquid fuels, encompassing either bioethanol or biodiesel [5]. Generally, biodiesel is a class of biofuel that is a renewable, economically viable, and environmentally friendly alternative energy source [6]. To address the global environmental crisis due to energy production, the production of biodiesel has become essential because it can enhance the quality of emissions from exhaust gases and is biodegradable. Moreover, biodiesel plays a crucial role in mitigating carbon dioxide emissions into the air [7]. This reduction occurs because the carbon dioxide released during biodiesel combustion is part of the natural carbon cycle, having been originally absorbed by the plants used to produce the feedstock. Consequently, the net increase in atmospheric carbon dioxide is significantly lower, aiding in the mitigation of climate change [8]. On the other hand, biodiesel is suitable for use in diesel engines either in its pure form or when mixed with petroleum diesel in certain proportions. This blend can also serve heating purposes [9]. Biodiesel can be produced by the transesterification of vegetable or animal fats, cooking oils, and non-cooking oils with short-chain alcohols such as methanol or ethanol in the presence of catalysts for biodiesel production and glycerin [5]. Also, it can be produced from algal oils through the process of transesterification or direct transesterification [10].

The advantage of using waste cooking oil to produce biodiesel is its low cost, as the price of waste cooking oil (WCO) is 2-3 times cheaper than clean vegetable oil. Thus, the total manufacturing cost of biodiesel can be significantly reduced [11]. In addition, it is reported that millions of tons of waste cooking oil are collected annually and used in various ways around the world [12]. The production of biodiesel from waste cooking oils contributes efficiently to prevent environmental pollution due to the environmental problems triggered essentially if WCO is disposed of in the environment, such as water and soil pollution, harm to human health, and disturbances in the ecosystem [13]. In the cooking process, a series of chemical and physical reactions occur, and the oil undergoes thermal oxidation, polymerization, and hydrolysis [14]. Hydrolysis occurs after the dissociation of triglyceride molecules, leading to the formation of Free Fatty Acids (FFA) [15]. The amounts of FFAs produced during fat oxidation in the cooking process can be assessed by titration using potassium or sodium hydroxide with a phenol naphthalene reagent [16]. Considerably, the high content of free fatty acids in the WCO affects dramatically on the production of biodiesel, and consequently leads to the formation of soap and [17].

The potential of biodiesel as an energy source alternative to petroleum diesel has been studied by numerous researchers. Biodiesel seems like a very good option because of its low toxicity and great biodegradability, among other factors. Additionally, it produces fewer emissions during combustion than petroleum-based diesel. The current study is aimed to produce biodiesel by using two types of waste cooking oils collected from homes and restaurants were tested and the percentage of FFA in them was determined through a titration process.

## MATERIALS AND METHODS

### *Sample collection*

Two types of used sunflower cooking oil samples were collected from homes and some restaurants in Basra province, Iraq. Then, the preliminary treatments were carried out to produce biodiesel. The biodiesel production process can be seen in **Figure 1**.

### *Cooking oil pretreatment*

Preliminary treatment of the used cooking oils was conducted by filtering with filter papers to remove impurities. Subsequently, an analysis of the used cooking oil was performed through a titration process to determine the free fatty acids (FFA) level. If the FFA percentage is less than 2%, the transesterification process can be carried out directly, while if the FFA percentage is more than 2%, the esterification process must first begin to reduce the FFA percentage [18]

### *Filtration process*

The oil was filtered using Whitman Millipore filter papers to remove impurities and stored in clean, sealed plastic bottles until use.

### *Titration process*

The titration process was performed based on Elsolh [19] for both types of samples. The oil was titrated with potassium hydroxide (KOH 0.1N) to determine the percentage of free fatty acids (FFA) present in the oil. Ten grams of filtered oil were placed in a 250 ml flask, followed by the addition of 50 ml of a solvent mixture comprised of ethanol and diethyl ether (1:1). The oil was stirred until it was completely dissolved. Next, 15 ml of ethanolic KOH solution was placed in a burette, and four drops of phenolphthalein indicator were added to the oil and solvent mixture. The mixture was titrated with continuous stirring until a color changed occurred. It was then allowed to stand for 30 seconds to confirm the new color, and the volume of KOH used in the titration was recorded. The following equation was applied to calculate the concentration of free fatty acids in the oil samples:

$$\%\ \text{free fatty acid} = \frac{V_{KOH} \times M_{KOH} \times MW}{10 \times E}$$

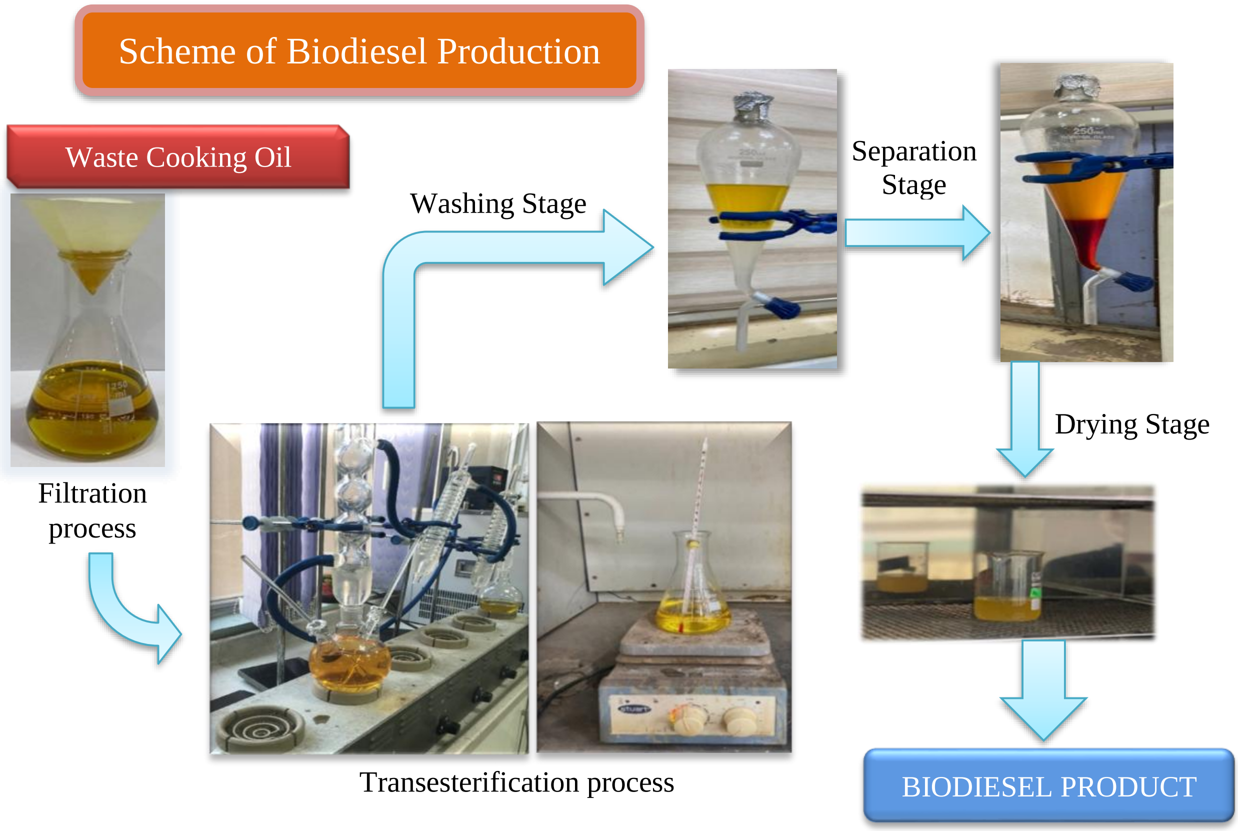

**Figure 1.** Scheme of Biodiesel Production

Where: $V_{KOH}$ was Volume of KOH (ml), $M_{KOH}$ molarity of KOH [0.1 mol/L], MW was molecular weight (314 g/mol), and E was initial weight of the oil in grams.

### *Transesterification process*

Following the preliminary treatment, the WCO was heated to 65 °C to eliminate any water residues. Subsequently, the transesterification process was carried out by mixing the catalyst KOH with the oil at a ratio of 1% by oil weight in a three-necked flask. Then, methanol was added at a methanol-to-oil ratio of 6:1. The temperature was maintained at 65 °C with continuous stirring for 90 minutes. After the specified duration, the mixture was transferred to a separation funnel and left overnight for phase separation. The resultant layers included an upper layer of biodiesel and a lower layer of glycerin. The biodiesel layer was then isolated for purification, which involved washing with hot distilled water at 70 °C in a 1:1 volume ratio of biodiesel fuel to wash water. The washing process was repeated several times. Finally, the biodiesel fuel was heated to 110 °C for 10 minutes in an electric oven to remove any remaining moisture

### *Separation stage*

The mixture was placed in a separation funnel for 24 h, during which two distinct layers formed: the upper layer consisting of biodiesel and the lower layer of glycerin. The layers were then separated, and the weight of each was measured.

### *Washing stage*

Biodiesel was washed with warm distilled water heated to 50 °C. The purpose of the washing process was to remove residual alcohol and glycerin present in the biodiesel. This washing procedure was repeated several times with gentle and slow agitation.

### *Drying stage*

Biodiesel obtained from the washing process can contain some amount of water, therefore, it was dried in an oven at a temperature of 110 °C for an hour and a half. The concentration of the produced biodiesel was calculated as a percentage according to the following formula [20]:

$$\text{Biodiesel yield \%} = \frac{\text{mass of the biodiesel produced (g)}}{\text{mass of the WCO (g)}} \text{ X } 100$$

### FTIR analysis of WCO and biodiesel produced

WCO and biodiesel produced were analysis by FTIR spectroscopy techniques (Bruker, Alpha II /Germany). One single spectrum was averaged over 24 scans at 4 $cm^{-1}$ resolutions in the wavelength range from 400 to 4,000 $cm^{-1}$.

### GC-MS analysis of biodiesel produced

Chemical composition analysis of biodiesel produced was done with GC-MS analysis, it was carried out by using an Agilent Technologies , 7890B GC system coupled to an Agilent Technologies 5977A MSD with EI Signal detector, using HP-5ms 5% phenyl, 95% methyl siloxane ( 30m*250um*0.25) , the oven temperature was set at 40 °C hold for 4 min then raised to 10 °C/min to 300 °C for 20 min , Helium carrier gas flow rate was 1 ml/min and purge flow of 3 ml/min .The injection mode was Split with injection temperature 290 °C and the injection sample volume was 1 micro letter. The mass spectrometer used Ion Source temperature 230 °C, the mass range 44-650 m/z, Data was run through the NIST 2014, data base as an additional tool to confirm identity of compounds.

### Physicochemical properties determination of WCO and biodiesel produced

WCO and biodiesel properties such as density, kinematic viscosity, cloud point, and pour point were determined and compared with the standard properties of diesel according to ASTM. The density was measured at 15 °C as specified in ASTM D1298 using a hydrometer by immersing it directly in the sample. The Kinematic viscosity of the WCO and biodiesel were measured with a viscometer by timing how long it takes for a specified volume of liquid to pass through a capillary tube under set conditions as follows in ASTM D445. The cloud point was determined as in the ASTM D2500 by pouring the sample into a test tube and then gradually cooling it by placing it in a cooling bath until cloudiness and crystallization were observed. The temperature at which the cloud point occurred was then recorded. Finally, the pour point was measured as in (ASTM D97).

## RESULTS AND DISCUSSION

### Biodiesel produced and the GC-MS analysis

Our results showed variation in the content of FFA between two WCO samples, where household cooking oil recorded 1.1932%, which is less than 2% in comparison with 2.0096% for restaurant WCO. Interestingly, the reason for differences in the content of FFA between the two samples is highly associated with the duration of WCO usage between the two samples, where the oil that was collected from homes, is considerably less used than oil used in fast food restaurants, which typically have a higher FFA content due to more frequent usage. Interestingly, the yield of free fatty acids has an interfering relation with the production of biodiesel in which high fatty acids contribute to a low reduction in biodiesel production because it triggers saponification. Moreover, high free acid content efficiently enhances the viscosity of the biodiesel production [21]. To overcome this problem, transesterification is generally considered a crucial step that is exploited efficiently to convert the high content of free fatty acids in WCO into fatty acid methyl esters. KOH was used in our study as a catalyst for transesterification. The reason we used KOH rather than any other alkali catalyst is that it dissolves faster than other alkaline catalysts like NaOH [22]. Biodiesel production efficiency reached 85.436%, which is considered high, indicating a successful transesterification process. This demonstrates that waste vegetable oil is an optimal source of biodiesel production [23]. The results of the GC-MS analysis of biodiesel produced from WCO revealed ten types of fatty acid methyl esters. The highest percentage was attributed to 9,12-Octadecadienoic acid, methyl ester, which accounted for 52.16 %, and the total percentage of all fatty acid methyl esters was 83.88 % (**Table 1** and **Figure 2**).

### FTIR analysis of WCO and biodiesel produced

FTIR analysis of WCO and biodiesel produced shown in the **Figures 3** and **4** and **Tables 2** and **3**). The results showed that the FTIR spectra of the WCO and biodiesel samples are very similar because the reaction simply consists of removing the glycerol and replacing a methyl group in the hydrocarbon chain [24], and this confirms that the FAME have some parts of the structures that are the same as the oils, i.e.: long chains composed of $CH_2$ groups, double C = C bonds inside the chain, C = O groups as well as C-O bonds in the ester groups [25]. The only significant difference can be seen in the ranges between 1000 and 1500 $cm^{-1}$. In particular, the bands located at 1160 and 1097 $cm^{-1}$ in the oil sample which correspond to the stretching vibration of the (C–O) group attached to ($CH_2$) shift to 1168 $cm^{-1}$ in the biodiesel sample. However, new bands at 1195 and 1437 $cm^{-1}$ were observed in the biodiesel sample associated with the bending and oscillatory vibrations of the ($CH_3$-O) group which are not present in the WCO spectrum. That indicating deformation vibrations of the $CH_3$ in the ester group

(O-$CH_3$) which indicates the conversion of WCO into biodiesel [26], In addition, there is another difference: the band around 1375 cm$^{-1}$ in the WCO spectrum is shifted towards lower wave number in the biodiesel spectrum.

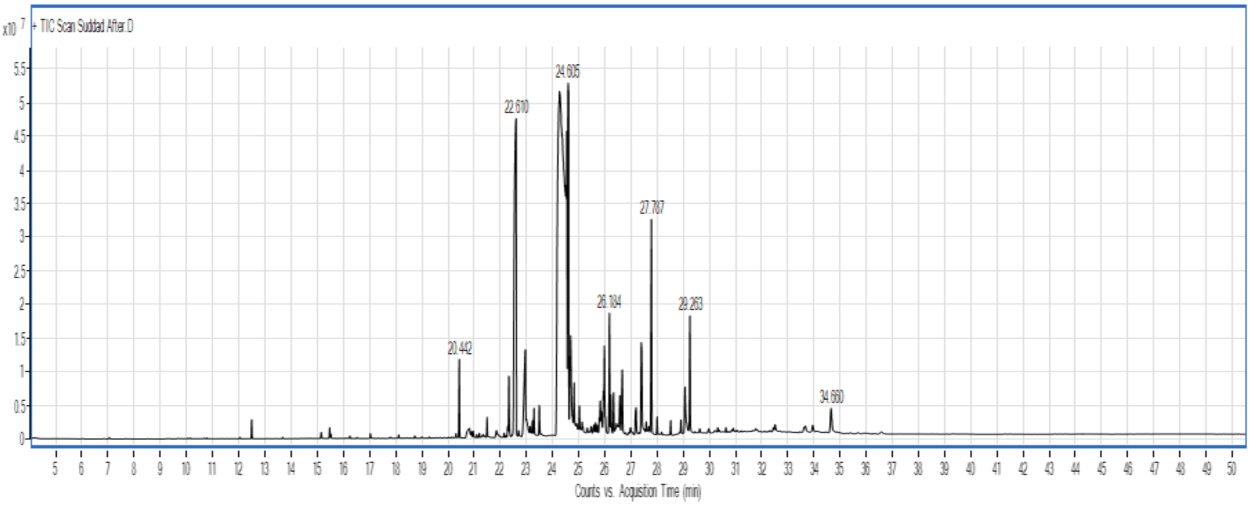

**Figure 2.** The absorption spectrum of biodiesel produced from the WCO using GC-MS technology

**Table 1.** GCMS analysis of biodiesel produced from WCO by alkaline transesterification method

| No. | Name of fatty acid methyl ester | Chemical formula | Molar mass (g/mol) | FAME % |
|---|---|---|---|---|
| 1 | 9,12-Octadecadienoic acid, methyl ester | $C_{19}H_{34}O_2$ | 249.47 | 52.16 |
| 2 | Hexadecanoic acid, methyl ester | $C_{17}H_{34}O_2$ | 270.45 | 15.66 |
| 3 | Docosanoic acid, methyl ester | $C_{23}H_{46}O_2$ | 354.61 | 4.25 |
| 4 | 11-Eicosenoic acid, methyl ester | $C_{21}H_{40}O_2$ | 324.5 | 3.75 |
| 5 | Eicosanoic acid, methyl ester | $C_{21}H_{42}O_2$ | 326.55 | 2.61 |
| 6 | Tetracosanoic acid, methyl ester | $C_{25}H_{50}O_2$ | 382.7 | 2.10 |
| 7 | 9-Hexadecenoic acid, methyl ester | $C_{17}H_{32}O_2$ | 268.43 | 1.34 |
| 8 | Myristic acid , methyl ester | $C_{15}H_{30}O_2$ | 242.40 | 1.26 |
| 9 | cis-10-Heptadecenoic acid, methyl ester | $C_{17}H_{32}O_2$ | 268.4 | 0.75 |
| 10 | Heptadecanoic acid, methyl ester | $C_{17}H_{34}O_2$ | 270.5 | 0.65 |
| | | | | 83.88 |

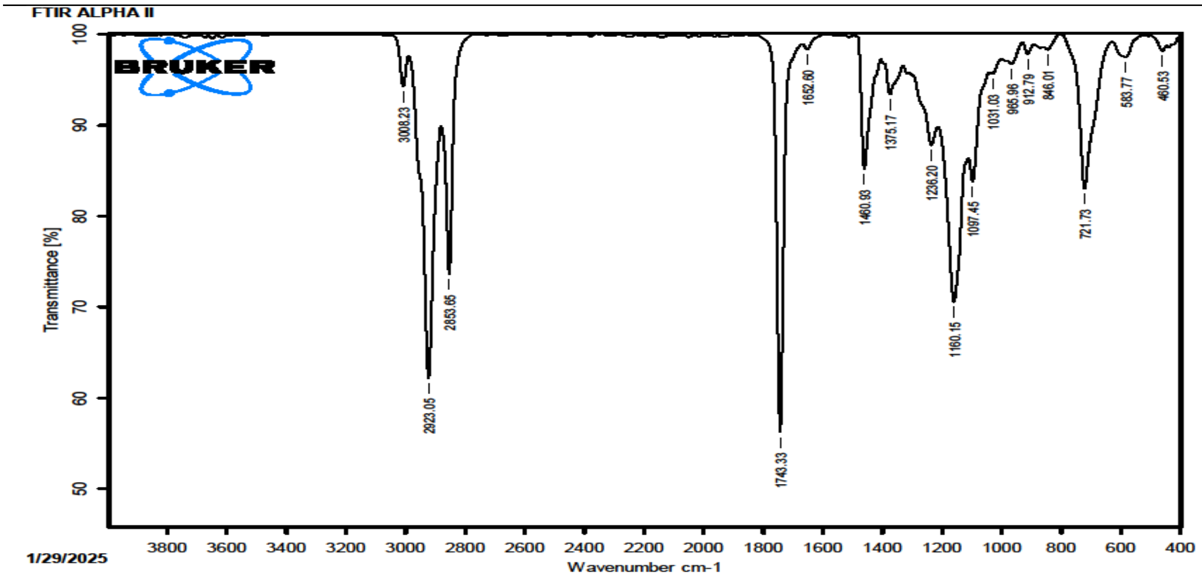

**Figure 3.** FTIR spectrum of WCO sample

**Table 2.** FTIR analysis of WCO

| Sample | Frequency cm$^{-1}$ | Bond | Functional group |
|---|---|---|---|
| WCO | 3008 | C-H stretch | Alkanes |
| | 2923 | $CH_2$ asymmetric stretching | Alkanes |
| | 2853 | $CH_2$ asymmetric stretching | Alkanes |
| | 1743 | C=O stretch | Esters |
| | 1652 | -C=C- stretch | Alkenes |
| | 1460 | C-H bend | Alkanes |
| | 1375 | $O-CH_2$ stretch | glycerol group (mono, di and triglycerides) |
| | 1236 | C-O stretch | Carboxylic acids,ester,ethers |
| | 1160 | C-O stretch | Carboxylic acids,ester,ethers |
| | 1097 | $O-CH_2$ stretch | glycerol group (mono, di and triglycerides |
| | 1031 | C-O stretch | Carboxylic acids,ester,ethers |
| | 846 | C-H bending | Alkanes |
| | 721 | C-H Rocking | Alkanes |

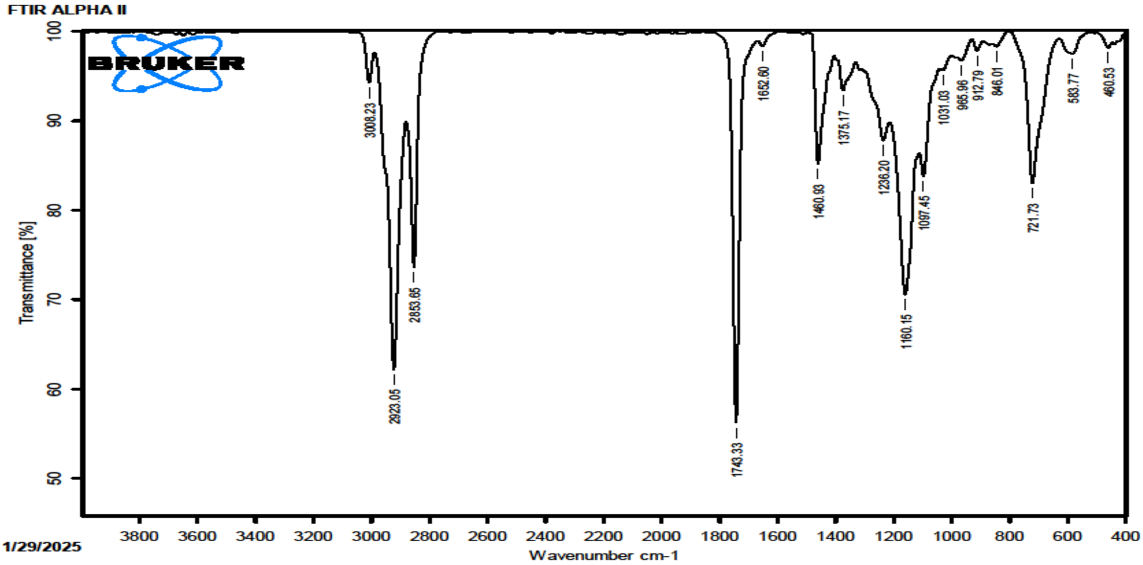

**Figure 4.** FTIR spectrum of biodiesel produced

**Table 3.** FTIR analysis of biodiesel produced

| Sample | Frequency cm$^{-1}$ | Bond | Functional group |
|---|---|---|---|
| **biodiesel produced** | 3008 | C-H stretch | Alkanes |
| | 2923 | $CH_2$ asymmetric stretching | Alkanes |
| | 2853 | $CH_2$ asymmetric stretching | Alkanes |
| | 1741 | C=O Stretch | Esters |
| | 1652 | -C=C- stretch | Alkenes |
| | 1460 | C-H bend | Alkanes |
| | 1437 | $CH_3-O$ stretch | Esters |
| | 1360 | C-H bending | Aliphatic |
| | 1243 | C-N stretch | Amide |
| | 1195 | C-O stretch | Esters |
| | 1168 | C-O Stretch | Esters |
| | 1016 | C-O Stretch | Esters |
| | 848 | C-H bending | Alkanes |
| | 722 | C-H Rocking | Alkanes |

### *Physicochemical properties determination of WCO and biodiesel produced*

The results of the physicochemical tests conducted on the produced biodiesel compare them with standard properties as shown in **Table 4**. These tests were performed according to the American Society for Testing and Materials (ASTM) standards. The tests measured density, Kinematic viscosity, and both the cloud and pour points. As observed in **Table 4**, These results showed clear differences between the properties of WCO and the biodiesel produced from it, as it was noted that WCO has a higher density 0.912 g/ $cm^3$ compared to the lower density of biodiesel at 0.844 $g/cm^3$ and this difference can be attributed to the chemical changes that occur during transesterification, resulting in the formation of lighter methyl esters, Likewise, the viscosity of WCO at 40 °C was 35.36 $mm^2/s$ significantly lower when compared to the viscosity of biodiesel at 4.951 $mm^2/s$ , this decrease in viscosity is the result of the decomposition of triglycerides into simpler, less viscous esters during biodiesel production [27]. In addition, the biodiesel produced showed a lower cloud point ( -6 °C) and pour point (-13 °C) compared to WCO (Cloud point: 1°C, pour point: -3 °C), indicating improvement performance at low temperatures. The transesterification process contributes to these changes, making biodiesel more suitable for cold weather conditions [28]

**Table 4.** Proximate analysis results of fine coal and palm shell

| Properties | WCO | Biodiesel | Standard | ASTM Method |
|---|---|---|---|---|
| Density (g\cm$^3$) @ 15 °C | 0.912 | 0.844 | 0.86_0.9 | D1298 |
| Kinematic Viscosity (mm$^2$\s) @ 40°C | 35.36 | 4.951 | 1.9_6.5 | D445 |
| Cloud Point (˚C) | 1 | -6 | -3 to +12 | D2500 |
| Pour Point (˚C) | -3 | -13 | -15 to +10 | D97 |

Different types of biodiesel feedstock offer various advantages and disadvantages. Edible vegetable oil is biodegradable, has low sulphur content, high oil content, and is relatively inexpensive. However, it also has high viscosity, competes with food crops, and may contribute to deforestation and habitat destruction. Animal fats are low-cost, help reduce waste, and face minimal competition with food crops, but they solidify at room temperature. Waste cooking oil can reduce pollution and waste while also competing minimally with food crops. On the downside, its availability can vary regionally, and it requires a well-developed collection infrastructure [29]

## CONCLUSION

Waste cooking oil (WCO) collected from homes is observed as an optimum raw material for the production of biodiesel through alkali transesterification. The properties of the biodiesel produced in this study highly meet ASTM standards. Moreover, the alkali transesterification process showed efficient conversion of FFA into FAME, and consequently our study concluded that biodiesel produced from WCO is highly recommended as reliable, cost-effective alternative to diesel fuel and a cleaner home energy source for cooking, which is considered a renewable energy and potentially an environmental tool for recycling waste vegetable oil after cooking.

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