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Transesterification of Waste Cooking Oil using Empty Fruit Bunches Ash-supported NaOH Heterogeneous Catalyst

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The rapid growth of the human population has led to an increase in waste production from various sectors especially palm oil industry, posing significant environmental and economic challenges. Waste cooking oil (WCO) and empty fruit bunches ash (EFBA) are among the problematic waste products, and their disposal, along with the adverse environmental impacts of fossil fuels, has driven the demand for sustainable energy sources. Heterogeneous catalysts have emerged as a preferred option for biodiesel production due to the limitations of homogeneous catalysts in the transesterification process. This study explores the effectiveness of a NaOH/EFBA heterogeneous catalyst in the transesterification of WCO and analysis of its physicochemical properties. NaOH was doped onto EFBA using Incipient Wetness Impregnation (IWI) method and calcined at 600°C. The catalyst was characterized using TGA-DTG, FTIR, SEM-EDX, and XRD techniques, and the biodiesel was analysed using GC-MS. Optimal transesterification conditions were found to be at 7 wt% catalyst loading, 30 minutes reaction time, and an 18:1 methanol to oil ratio, yielding 71.25% biodiesel. The biodiesel was confirmed by the detection of hexadecanoic acid methyl esters, heptadecanoic acid methyl esters, 9- octadecanoic acid methyl esters, and 9,12-octadecanoic acid methyl esters in the GC-MS analysis. The results demonstrate that the potential catalyst efficiently converted triglycerides in waste cooking oil into fatty acid methyl esters (FAME).

Keywords: Ash; Biodiesel; catalyst; heterogeneous; transesterification

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The global concern over the dwindling of fossil fuel reserves and environmental problems, particularly linked to the emissions of pollutants like sulphates and carbon dioxide causing global warming, has sparked a focus on the energy crisis. This concern has led to a search for an alternative fuel. Biodiesel, among options like hydrogen, solar, and wind power, stands out as a viable alternative. It can be produced from renewable sources such as vegetable oils, animal fats, and various types of algae through a process called transesterification [1]. The world mostly depends on non-renewable resources like coal, natural gas, and petroleum, but these supplies are limited and will ultimately run out. Biodiesel, a renewable alternative fuel derived from sources such as waste cooking oil (WCO), vegetable oil, jatropha oil, and animal fats, is gaining popularity despite the continued reliance on diesel engines in various industries [2]. Waste Cooking Oil (WCO) accounts for 20–32% of edible oil usage worldwide, with an annual production of 15 million tonnes. Over 60% of the one million tonnes produced annually in Europe are disposed of incorrectly, endangering human health and the environment. To lessen these effects, immediate action is required for appropriate disposal and recycling [3].

Transesterification, which involves converting vegetable or animal fats into a mixture of mono-alkyl esters of long chain fatty acids, is the usual method used to create biodiesel. For this reaction, several techniques are available, such as batch processing, supercritical procedures, ultrasonic and microwave techniques [4]. The chemical process known as transesterification produces biodiesel and glycerol as a by-product when triglycerides and alcohol react in the presence of a catalyst. The molar ratio of alcohol to oil, reaction time, type and quantity of catalyst, reaction temperature, and feedstock composition all affect the transesterification reaction. The most important of these variables in the dynamics of biodiesel generation is the alcohol-to-oil molar ratio [5]. The usual method for turning its oil into biodiesel is transesterification with KOH or NaOH as catalysts; however, catalyst recovery is difficult and frequently results in undesirable by-products like soap and emulsion [6]. According to Husin et al., in 2018,

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the synthesis of biodiesel is often carried out commercially using a homogenous base catalyst, like NaOH or KOH [7]. Nevertheless, this method has disadvantages, such as corroding equipment and managing the waste of sulphuric acid (H_2SO_4) produced during the neutralisation procedure. Homogeneous catalysts are frequently used in the transesterification of waste cooking oil (WCO), even though they are corrosive and difficult to extract from the final product. Previous reported research reveals that heterogeneous catalysts have additional advantages, such as making it easier for separating biodiesel and glycerol products through separation of phases [8].

Due to Malaysia's intense emphasis on agriculture, a large amount of biomass waste is produced, especially from oil palm, which is an essential crop in the nation's agricultural landscape. Malaysia produces more than 90 million tonnes of renewable biomass a year from its oil palm sector, making it one of the world's top producers of palm oil. The palm tree's trunk, fronds, shells, palm press fibre, and most importantly empty fruit bunches (EFB) are all included in this [9]. Potassium oxide from palm bunch ash can be utilised as an inexpensive solid catalyst in the manufacturing of biodiesel. With the goal of providing a sustainable substitute for conventional catalysts, the research focuses on converting empty fruit bunch ash into an active and affordable solid catalyst that will speed up the transesterification of vegetable oils into biodiesel [7].

MATERIALS AND METHODS

Waste cooking oil (WCO), the feedstock, was obtained from household waste, and empty fruit bunch ash (EFBA) from FTJ Bro Power Sdn. Bhd. Hydrochloric acid (HCl, purity > 32%), sodium hydroxide (NaOH, 99%), potassium heptadecanoate (purity > 99%), and phenolphthalein (99%) were purchased at analytical grade straight away and used without additional purification.

Sample Pre-treatment and Preliminary Testing

The obtained WCO was filtered to remove solid particles and other contaminants. Following filtering, the WCO was heated to 120°C and stirred for two hours to eliminate any remaining water and contaminants. The WCO that has been pre-treated was kept in a clean container. Three preliminary testing was conducted on the feedstock of WCO which are moisture test, acid value test and saponification value test. The water content of the WCO sample can be determined using a moisture test following the standard procedure outlined in AOCS Tb 1a-64. The acid value test was conducted in accordance with the AOCS Cd 3a-63 standard. The saponification value test is essential for assessing the quality and characteristics of an oil sample, as well as determining the average molecular weight of all fatty acids present. The standard procedure for this test follows ASTM D5558.

Catalyst Preparation and Characterization

To dope EFBA with NaOH, 20 g of EFBA was mixed with 44.56 mL of 2 M NaOH and heated to 90°C until dry. After the mixture dried completely, it was kept in an oven at 110°C for 12 hours. The treated EFBA was then ground into a smooth powder, resulting in the final product known as Na/EFBA. For the calcined sample, the same procedure was followed, but it was further processed with calcination, which involved burning it in a furnace at 600°C, resulting in the final product known as Na/EFBA600. The synthesized catalyst was characterized using TGA-DTG, FTIR, SEM-EDX, and XRD techniques to obtain an information of its physicochemical properties.

Transesterification Reaction

A series of transesterification reactions were conducted using four different methanol to oil molar ratios: 9:1, 12:1, 15:1, and 18:1. These reactions were also performed at various reaction times: 30, 45, 60, 75, and 90 minutes, all at the reflux temperature of methanol (65°C). The catalyst loadings applied during the reactions were 5, 7, 10, 13, and 15 wt%. For each reaction, 10 g of WCO were mixed with methanol and catalyst in a round bottom flask fitted with a reflux condenser and stirred. After each cycle, the top methyl ester phase and the glycerol phase were separated by centrifuging the reaction mixture at 4000 rpm for about 15 minutes. This process results in the formation of two layers: biodiesel (top layer) and glycerol (bottom layer). The top biodiesel layer is then transferred into a separating funnel and flushed with boiling water to remove impurities, excess methanol, glycerol, and polar compounds. This flushing process continues until the biodiesel changes from cloudy to clear. Once flushing is complete, the biodiesel is transferred into a beaker and weighed on an electronic scale to determine the mass of biodiesel produced. The beaker containing biodiesel is then treated with a small amount of anhydrous Na_2SO_4 to remove any traces of water for about 15 minutes. Finally, the biodiesel was weighed, and its mass was recorded for yield calculation. The final biodiesel product was analysed using GC-MS to confirm its composition and quality.

Biodiesel Analysis

Gas Chromatography-Mass Spectrometry (GC-MS) analysis (Agilent model) is a typical method used to assess the content of fatty acid methyl ester (FAME) in biodiesel samples. The sample preparation involved dissolving 0.125 g of WCO biodiesel in 10 mL of GC-grade n-hexane (purity > 99%) and adding 2.5 mL of a 10 mg/mL internal standard solution (methyl heptadecanoate). GC oven was programmed in three stages: initial temperature of 40°C, held for 1 minute, a gradual increase to 80°C, held for 2 minutes and a further increase to 250°C, held for 2 minutes, with a temperature ramp rate of 5°C/min, respectively. The injector temperature was set to 80°C, and the interface temperature was maintained at 260°C. To fill the GC intake port, 1 µL of sample will be introduced. The peak area of methyl heptadecanoate, the internal standard, and the peak area of FAME in biodiesel can be recognised and compared from the generated chromatogram. Calculating the proportion of impure yield from the transesterification reaction can be done with the following formula:

Percentage of biodiesel conversion,

$$A = \frac{Weight\ of\ biodiesel\ collected\ (g)}{10\ g\ of\ WCO} \times 100$$

The ester content cannot be computed until the impure biodiesel % has been established due to the existence of unreacted oil, glycerol, and other contaminants. The following formula can be used to calculate ester content when utilising Agllent method EN14103:

$$\text{Ester content (\%), } c = \frac{TA-AEI}{AEI} \times \frac{CEI \times VEI}{m} \times 100$$

c: Ester content of purity of FAME in biodiesel

m: Mass of the sample

TA: Total area

AEI: area of internal standard

CEI: Concentration of methyl heptadecanoate solution (mg/mL)

VEI: Volume of methyl heptadecanoate solution (mL)

$$\text{Mass of pure biodiesel, } Y = \frac{c}{100} \times \text{weight of biodiesel collected}$$

$$\text{Pure biodiesel (\%), } B = \frac{Y\ (g)}{10\ g\ of\ WCO} \times 100$$

RESULTS AND DISCUSSION

Based on the preliminary tests conducted, the moisture test revealed that the WCO sample contained 0.34% moisture content. Subsequently, additional analyses were performed, including free fatty acid (FFA) and saponification tests, which yielded values of 0.2213 wt% and 80.12 mg KOH/g, respectively. The molecular weight of the WCO, calculated from the saponification value, was 2100.94 g mol^{-1}. The moisture presence has been eliminated during heating process of WCO sample to prevent water from limiting the catalyst's performance in transesterification process. Then, the WCO sample was ready to be reacted with NaOH/EFBA catalyst.

Thermogravimetry Analysis – Derivative Thermogravimetry (TGA – DTG) Analysis

The TGA analysis of the NaOH/EFBA catalyst sample was displayed in Figure 1 where the thermal behaviour of catalyst was obtained. Initially, there is a significant weight loss at 58.04°C, where the sample loses 13.76% of its mass (2.14 mg). This phase likely indicates the loss of water or low weight volatiles [10]. A second phase of weight loss occurs at 562.77°C, with a 3.42% reduction (0.53 mg), probably due to the breakdown of organic compounds or intermediates [11]. The third phase is at 878.49°C, where the sample loses 11.42% of its mass (1.78 mg), indicating the decomposition of more stable components or the complete combustion of the catalyst's main parts. At 1000.24°C, a substantial residue of 71.17% (11.08 mg) remains, suggesting the presence of substantial amount of non-volatile or thermally stable materials in the NaOH/EFBA catalyst. Overall, the TGA graph shows the thermal stability and decomposition stages of the NaOH/EFBA catalyst, revealing the presence of thermally stable substances that result in a high residue at high temperatures. A higher calcination temperature may cause the fine crystals to sinter and the clusters to agglomerate, which can reduce the catalyst's activity [12].

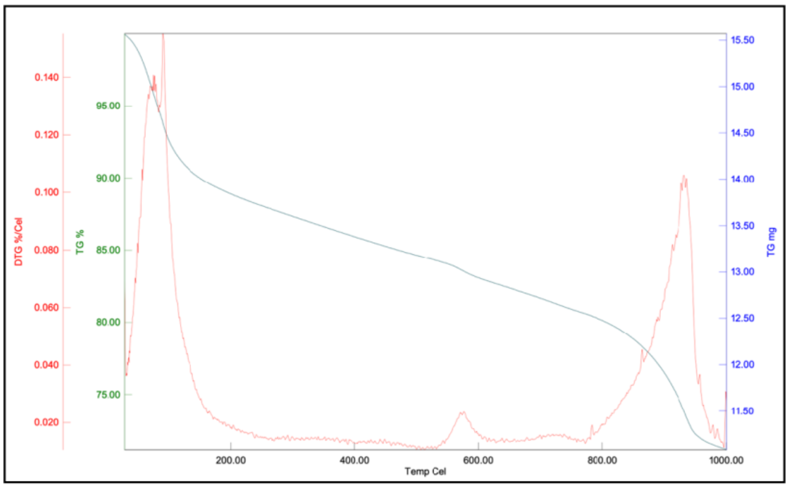

Figure 1. TGA – DTG Spectrum of NaOH/EFBA.

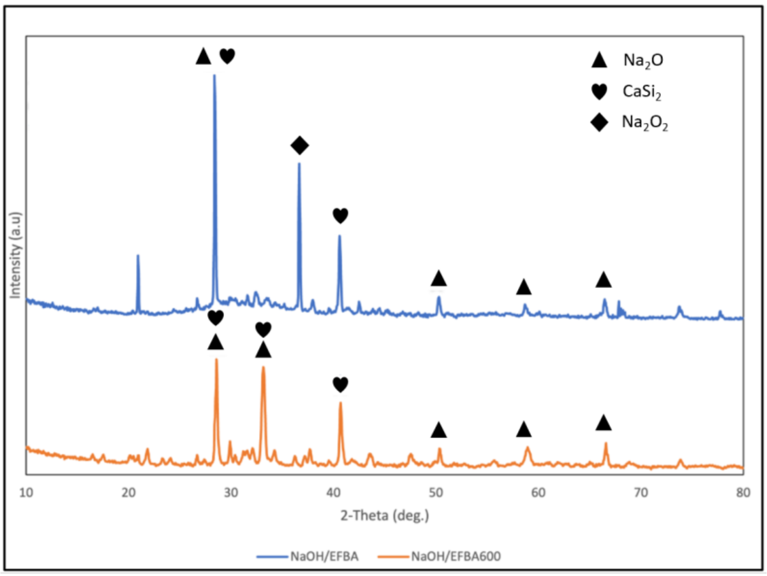

Figure 2. XRD spectrum analysis for NaOH/EFBA and NaOH/EFBA 600.

X – Ray Diffraction Spectroscopy (XRD) Analysis

The NaOH/EFBA catalyst is shown in Figure 2 in two separate forms: non-calcined and calcined. The XRD pattern of the NaOH/EFBA sample shows multiple unique peaks at 2θ of 20.9279°, 28.4102°, and 36.6346°. The strongest peak, which is located at 28.4102°, shows a highly crystalline phase that is probably connected to a particular catalyst chemical which show the presence of calcium disilicide ($CaSi_2$) compound with reference number JCPDS File No. 00-019-0251. A variety of phases is suggested by the formation of many peaks at different strengths. This is typical of a non-calcined sample where different components are present in their original or partially reacted forms. This observation indicates that the precursor compound of the solid catalyst remains undecomposed on the catalyst surface if the calcination temperature is below 400°C [12]. The reference number 98-006-0435 indicates that disodium oxide (Na_2O) is included in the recognized phase. Calcination typically induces phase transformations, leading to the formation of more stable crystalline phases and the removal of volatile components. The appearance of new peaks and the shift in peak positions compared to the non-calcined sample indicate the formation of new crystalline phases or the enhancement of existing ones. The identified phase remains disodium oxide (Na_2O), with a reference code JCPDS File No. 98- 018-0570, but the intensity and sharpness of the peaks suggest improved crystallinity. The results showed that increasing the calcination temperature to 600°C increased the degree of crystallinity of the potential catalyst [7].

Scanning Electron Microscopy with Energy Dispersive X – Ray (SEM – EDX) Analysis

The SEM-EDX analysis of the NaOH/EFBA catalyst reveals a detailed picture of its surface morphology and elemental composition. This analysis revealed the presence of various elements, including C, O, Si, K, Na, Mg, Al, Cu, S, and Cl, in both samples. The analysis for NaOH/EFBA, in Figure 3a shows the image of an aggregate small particles sample with 2000x magnification and indicates a significant presence of oxygen (56.40 wt%) and carbon (18.22 wt%), suggesting that the catalyst surface contains substantial amounts of oxygen and carbon element. The catalyst is also rich in sodium (4.28 wt%) and potassium (11.08 wt%) metal, which are essential alkali metals likely contributing to improving the catalyst's activity. For NaOH/EFBA 600 sample reveals its detailed elemental composition. Carbon and oxygen dominate the sample, comprising 21.98% and 65.52% of the weight, respectively.

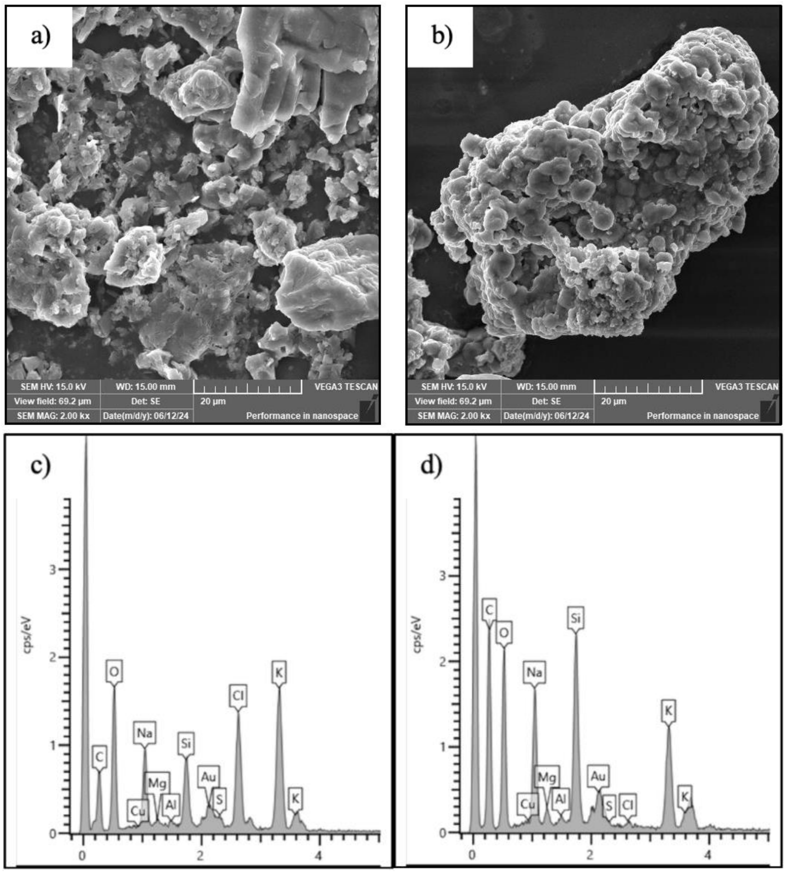

Figure 3. SEM images a) NaOH/EFBA and b) NaOH/EFBA600 and EDX spectrums c) NaOH/EFBA and d) NaOH/EFBA600

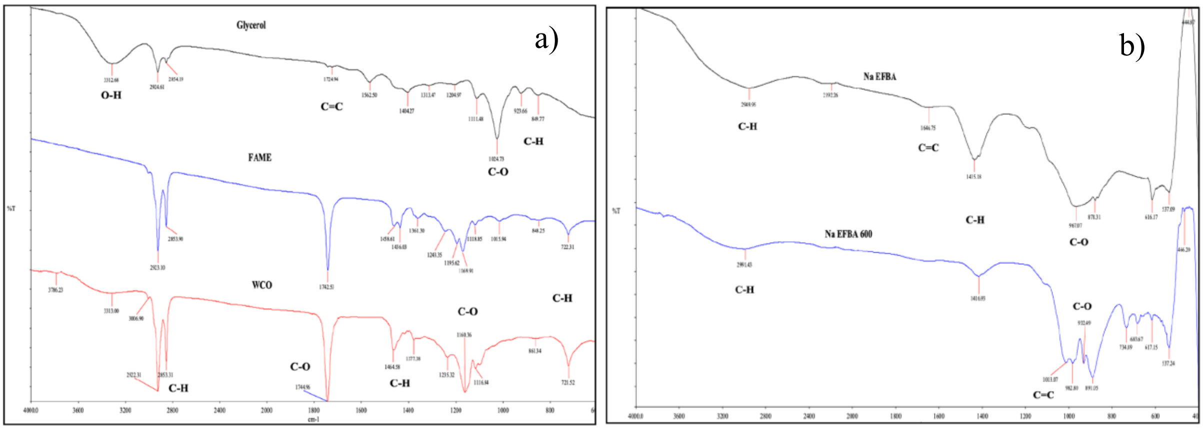

Figure 4. FTIR spectrum of a) Glycerol, FAME, and WCO, b) NaOH/EFBA and NaOH/EFBA600.

Fourier Transformed Infra-Red Analysis (FTIR)

The carbonyl group in the biodiesel sample was nearly identical to WCO even though the FFA fully converted to FAME [13]. There were three different oil samples which are glycerol, FAME and WCO used for this analysis as shown in Figure 4a. The presence of C-H compound stretching vibration of carbon-carbon single bond at the absorption peaks which are 2923.10 cm^{-1} and 2922.31 cm^{-1}. Also, C=O group of triglycerides was observed at the peak 1742.53 cm^{-1} and 1744.96 cm^{-1}. In addition, the peaks range at 1000 cm-1 to 1300 cm^{-1} indicated the presence C-O stretching vibrations of ester group. The stretching vibration of the C-O group attached to -CH_2 shifts from the C-O group attached to -CH_2 stretching vibration [8]. For glycerol, an additional peak at 3312.68 cm^{-1} was assigned to O-H stretching. Figure

4b show the FTIR spectra of NaOH/EFBA and NaOH/EFBA600. A peak at 2949.95 cm^{-1} and 2991.43 cm^{-1} indicates C-H stretching vibrations, which are typical for alkanes. Peaks at 1435.18 cm^{-1} and 1416.93 cm^{-1} correspond to C=C stretching vibrations, indicating the presence of alkenes. NaOH/EFBA 600 Peaks at 1013.07 cm^{-1} could indicate C=C bending vibrations. Peaks at 967.07 cm^{-1} and 932.49 cm^{-1} indicate C-O stretching, which is common in ethers or esters.

Transesterification Reaction-Effect of Methanol to Oil Ratio

In the transesterification process, the methanol to oil ratio is crucial in determining the yield and quality of biodiesel. Various ratios were conducted which are 9:1, 12:1, 15:1 and 18:1 molar ratio to identify the optimal conditions as presented in Figure 5. At a ratio of 9:1, the limited methanol amount leads to an incomplete reaction with triglycerides, resulting in a lower biodiesel yield of 24.78% and unreacted triglycerides. Increasing the ratio to 12:1 and 15:1 further improves the conditions where the biodiesel yield obtained were 24.90% and 35.73%, promoting a more complete conversion of triglycerides into FAMEs and resulting in a higher biodiesel yield. However, the optimal methanol to oil ratio was found to be 18:1 with the biodiesel yield of 55.52%. At this ratio, the transesterification reaction proceeds to near completion, significantly increasing the yield of biodiesel. The excess methanol ensures that almost all triglycerides are converted into FAMEs, maximizing the efficiency and output of the biodiesel production process [14].

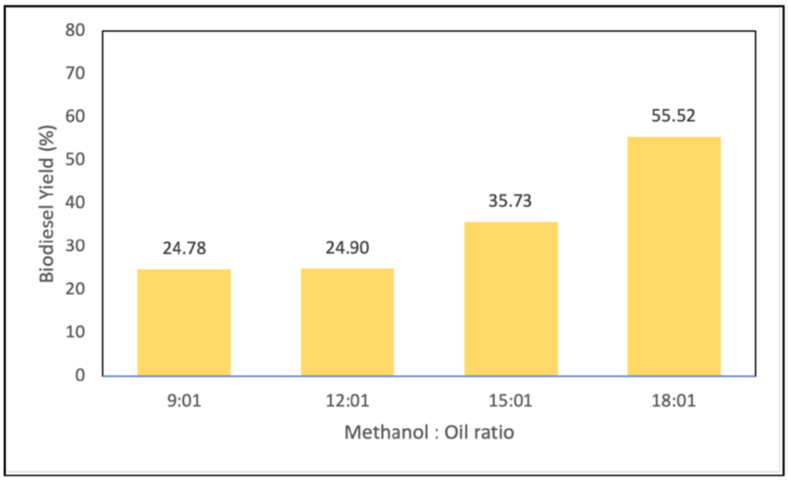

Figure 5. Methanol to oil ratio used in the transesterification reaction with 7 wt% of catalyst loading, 45 min reaction time and 65°C reaction temperature.

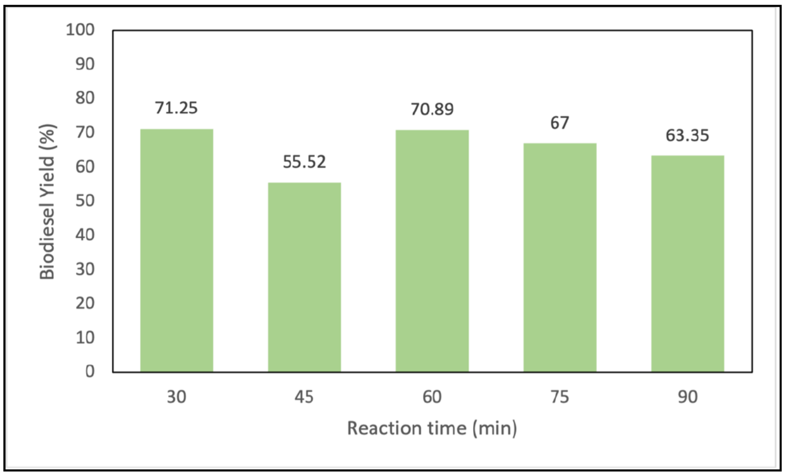

Figure 6. Variation of reaction time used in the transesterification reaction with 7 wt% of catalyst loading, 18:1 methanol to oil ratio and 65°C reaction temperature.

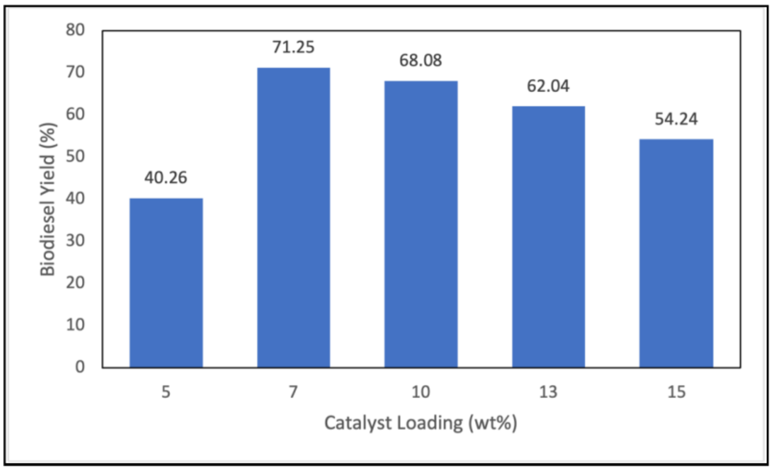

Figure 7. Catalyst loading wt% used in the transesterification reaction with 18:1 methanol to oil ratio, 30 min reaction time and 65°C reaction temperature.

Transesterification Reaction-Effect of Reaction Time

For the reaction time parameter, various reaction times were studied which are 30, 45, 60, 75, and 90 minutes to obtain a high percentage of biodiesel yield as revealed in Figure 6. Based on the data, the highest biodiesel yields were obtained at reaction times of 30 and 60 minutes. Although both durations produced nearly similar yields, the optimal reaction time yielding the highest percentage was 30 minutes. This behaviour can be attributed to the higher initial availability of unoccupied active sites on the catalyst surface. However, as the reaction time increases, these active sites become occupied, leading to a reduction in available surface area [15].

Transesterification Reaction-Effect of Catalyst Loading

Figure 7 indicated that the highest biodiesel yield was achieved by using catalyst loading of 7 wt%. Starting from 5 wt%, the percentage yield increased as the catalyst loading was raised to 7 wt%. However, further increasing the catalyst loading beyond 7 wt% to 10, 13, and 15 wt% resulted in a decrease in biodiesel yield. This suggests that a certain amount of catalyst is essential to drive the transesterification reaction efficiently, while excessive catalyst can lead to side reactions, catalyst agglomeration, or other issues that negatively impact the overall yield. Catalyst agglomeration will affect the active surface area of the catalyst, which subsequently decreases the reactivity [7]. Therefore, 7 wt% was identified as the optimal catalyst loading for maximizing biodiesel production.

Gas Chromatography–Mass Spectroscopy (GC-MS)

The biodiesel sample was subjected to further analysis using Gas Chromatography-Mass Spectrometry (GC-MS) to precisely determine the amount of pure Fatty Acid Methyl Esters (FAMEs) present. In this analysis, methyl heptadecanoate (C17) served as the internal standard for quantifying the ester content by comparing the peak areas of the FAME species in the sample to that of the standard. The biodiesel sample, produced using a NaOH/EFBA catalyst, was selected for GC-MS qualitative analysis under optimized conditions: a catalyst loading of 7 wt%, a methanol to oil ratio of 18:1, a reaction temperature of 65°C, and a reaction time of 30 minutes. From the GCMS data obtained, nine peaks were observed for the compound analysed in the biodiesel but only four peaks were matched to the methyl ester group as shown in Table 1. Methyl heptadecanoate was excluded as it served as the internal standard and was confirmed by the MS library to be present in the sample.

Table 1. Methyl ester peaks and compound name obtained in biodiesel using GC-MS analysis.

Sample Peak	Compound Name
21.403	Hexadecanoic Acid Methyl Ester
22.341	**Heptadecanoic Acid Methyl Ester**
23.274	9-Octadecanoic Acid Methyl Ester
23.589	9,12-Octadecanoic Acid Methyl Ester

CONCLUSION

In conclusion, this study has successfully determined the effectiveness of the NaOH/EFBA heterogeneous catalyst in the transesterification of waste cooking oil, identifying the optimal conditions for biodiesel production. The optimal parameters were determined to be a catalyst loading of 7 wt%, a reaction time of 30 minutes, and a methanol to oil ratio of 18:1, which resulted in a biodiesel yield of 71.25%. The biodiesel product was analysed using GC-MS, identifying four peaks corresponding to hexadecanoic acid, heptadecanoic acid, 9-octadecanoic acid and 9,12-octadecanoic acid methyl ester. The overall results from the catalyst characterization demonstrated that the NaOH/EFBA catalyst has a strong potential for further application in biodiesel production.

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The authors declare that they have no conflict of interest.

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