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TRANSESTERIFICATION OF LOW-GRADE PALM COOKING OIL TO PRODUCE BIODIESEL USING SPENT BLEACHING CLAY CATALYSTS

(Transesterifikasi Minyak Masak Kelapa Sawit Gred Rendah Kepada Biodiesel Menggunakan Pemangkin Tanah Pemutih Terpakai)

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Abstract

In this study, spent bleaching clay doped with potassium hydroxide and sodium hydroxide (SBC/KOH and SBC/NaOH) were used as catalysts in the transesterification process of low-grade cooking oil to produce biodiesel. The physicochemical properties of the catalyst were studied using Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), X-ray Diffraction (XRD), Brunauer-Emmett-Teller (BET), and Field Emission Scanning Electron microscopy with Energy Dispersive X-ray Spectroscopy (FESEM-EDX). Several parameters including the types of catalyst, calcination temperature of the catalyst, catalyst dosage, ratio of oil to methanol, reaction time, and reaction temperature were used to determine the optimal conditions for the transesterification reaction. According to the experimental findings, the catalyst effectively converted free fatty acids (FFA) into fatty acid methyl esters (FAMEs). The maximum biodiesel yield of 47.66 % was obtained at optimum reaction conditions: catalyst amount 2 wt.% of SBE/KOH calcined at 500 °C (SBC/KOH 500), reaction time 2 h, reaction temperature 55 °C and oil to methanol molar ratio 1:20.

Keywords: clay, cooking oil, biodiesel, transesterification, catalyst

Abstrak

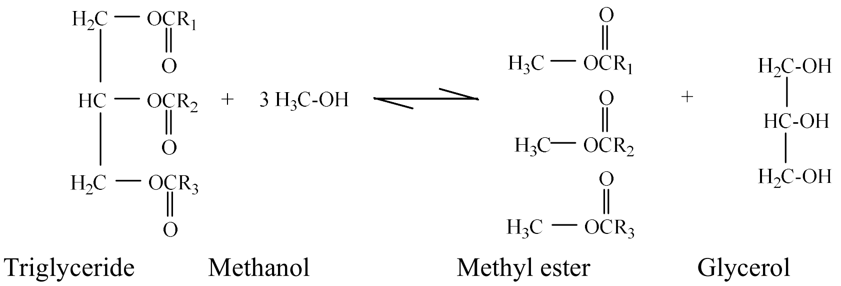
Dalam kajian ini, tanah liat peluntur bekas yang didop dengan kalium hidroksida dan natrium hidroksida (SBC/KOH dan SBC/NaOH) digunakan sebagai mangkin dalam proses transesterifikasi minyak masak gred rendah untuk menghasilkan biodiesel. Sifat fizikokimia mangkin dikaji menggunakan spektroskopi inframerah transformasi Fourier (FTIR), analisis termogravimetrik (TGA), pembelauan sinar-X (XRD), Brunauer-Emmett-Teller (BET), dan mikroskop elektron pengimbasan pancaran medan dengan serakan tenaga sinar-X (FESEM-EDX). Beberapa parameter termasuk jenis mangkin, suhu pengkalsinan mangkin, dos mangkin, nisbah minyak kepada metanol, masa tindak balas, dan suhu tindak balas telah digunakan untuk menentukan keadaan optimum bagi tindak balas transesterifikasi. Menurut penemuan eksperimen, mangkin secara berkesan menukar asid lemak bebas (FFA) kepada asid lemak metil ester (FAMEs). Hasil biodiesel maksimum sebanyak 47.66 % diperoleh pada keadaan tindak balas optimum: jumlah mangkin 2 wt.% SBE/KOH dikalsinkan pada 500 °C (SBC/KOH 500), masa tindak balas 2 jam, suhu tindak balas 55 °C dan minyak kepada metanol nisbah molar 1:20.

Kata kunci: tanah liat, minyak masak, biodiesel, transesterifikasi, mangkin

Introduction

The search for alternative fuels has gotten a lot of attention recently due to environmental concerns about the increasing uncertainty of global energy supply and production, the high price of petroleum products, and the usage of fossil fuels. Biodiesel has recently acquired appeal in both developed and developing countries' marketplaces. Biodiesel is made from vegetable oil or animal fat, which is derived from monoalkyl esters from long-chain fatty acids [1]. Biodiesel is becoming more popular as an alternative fuel due to its appealing chemical properties, such as biodegradability, non-toxicity, and carbon neutrality [2]. Biodiesel has a lot of the same qualities as petrodiesel, but it's free of harmful components like sulfur and aromatics. Diesel and biodiesel are both fuels used in internal combustion engines, but they have significant differences in terms of their origin, composition, production, and environmental impact .Biodiesel produces less NOx gases and emissions less than petrodiesel. Furthermore, biodiesel helps to reduce pollution and may reduce the effects of global warming. In comparison to petrodiesel, it is less toxic and easy to store [3]. However, there are a few disadvantages to using petrodiesel. Petrodiesel may contain a tremendous degree of pollutants and greenhouse gas (GHG) emissions, which could negatively affect individual well-being and the environment. Studies have shown that petrodiesel may emit dangerous contaminants. such as nitrogen oxide (NO), carbon monoxide (CO), sulfur dioxide (SO_2), particulate matter, and hydrocarbons [4].

Vegetable oils and animal fats must require severe transesterification to reduce oil viscosity and prevent serious damage to biodiesel. Transesterification of animal fats and vegetable oils with presence of alcohol and a catalyst produces biodiesel, a popular alternative fuel. Methanolysis, also known as transesterification, is a catalyzed reaction that produces biodiesel and glycerol. The reaction causes short-chain alcohol to replace the glycerol in the triglycerides. The process begins with three reversible processes that convert triglycerides to diglycerides, to monoglycerides, and to glycerol. From a single triglyceride molecule, each step produces an ester, giving three ester molecules [5]. The reaction is shown in scheme 1.



Scheme 1. Methanolysis reaction of triglyceride

In this study, heterogeneous base catalyst, potassium hydroxide (KOH), and sodium hydroxide (NaOH) were studied because it produces high biodiesel yield, high glycerol purity, low cost, and are environmentally friendly [6]. Furthermore, the heterogeneous catalyst has a larger surface area than the homogeneous catalyst. Low-grade cooking oil was selected as a feedstock due to its low viscosity and easy to obtain and spent bleaching clay (SBC) was used as a supporting catalyst.

SBC is a type of industrial waste produced by bleaching of crude palm oil in the vegetable oil refining sector [7].

SBC undergoes refining process in the palm oil industry before it is used for other purposes. SBC output is estimated to be between 1.6 and 2.0 million tonnes per year, based on oil production of 128.2 million metric tonnes in 2007 [8]. Moreover, SBC includes 20–40 % colored pigments, fat, and oil by weight, and it contains MgO, which could be used as a catalyst to speed up the production of biodiesel. Therefore, this study aims to produce biodiesel from low grade cooking oil with a highly efficient SBC nanocatalyst. Various analytical technologies were used to characterize catalysts and analyse products.

Materials and Methods

Chemicals and materials

SBC was collected from PGEO Edible Oils, Perai, Penang and low-grade cooking oil was purchased from the nearest shop. All the analytical grade chemicals (KOH, NaOH, methanol, n-hexane) were purchased from Merck.

Catalyst preparation

SBC was dried at 65 ℃ overnight in an oven before it was used for further experiments. SBC doped KOH and NaOH (SBC/KOH and SBC/NaOH) were synthesized via wet impregnation method. About 8g of calcined SBC, 8g KOH, and 120 mL deionized water were mixed and placed in a 250 mL beaker, heated at 70 ℃ for 3 hours with constant stirring. The obtained mixture was filter using filter paper and dried at 65 ℃ in an oven overnight. Next, it was ground and heated to 600 ℃ for 2 hours at a rate of 5 ℃/min in a furnace with a flow of nitrogen [9]. The same procedure was repeated with SBC/NaOH catalyst using 8g of NaOH instead of 8g of KOH during the impregnation process.

Transesterification process and identification of biodiesel products

About 0.2g of catalyst and 1:20 molar ratio of oil to methanol was added to 10g of oil. The catalyst was activated by dispersing methanol at 50 ℃ for 20 min at constant stirring. The reflux process was conducted at 55 ℃ with continuous stirring for 2 hours [10]. Once the transesterification process was completed, the product was cooled down and filtered with filter paper to separate the catalyst. The filtered liquids were placed in the separatory funnel and left overnight. After overnight, the liquids would be separated and formed into 2 layers. The lower layer was removed, and the upper layer was taken to separate the biodiesel from excess methanol and by-product. The extraction of biodiesel was conducted using Heidolph Hei-VAP rotavapor at 40 ℃ with 80 rpm. The biodiesel liquid was obtained, and by using Equation 1 below, the weight percent of FAME was calculated.

$$\text{Weight percent of FAME} = \frac{\text{Weight of transesterified liquid}}{\text{Weight of low-grade cooking oil used}} \times 100\ \% \tag{1}$$

Optimization parameters

This study was investigated with different parameters such as the effect on the type of catalyst (SBC/KOH and SBC/NaOH), calcination temperature (500 and 600 °C), catalyst dosage (1-4 wt.%), ratio of oil-to-methanol (1:12-1:20) and reaction time (1-3 hours) to predict the optimized condition for transesterification reaction. The range of each parameter was determined from previous studies [11, 12].

Characterization methods

The catalysts were characterized using several characterization methods such as Fourier-transform infrared spectrometer (FTIR), thermogravimetric analysis (TGA), X-ray diffraction (XRD), field-emission scanning electron microscopy with energy dispersive X-ray (FESEM-EDX) and Brunauer-Emmett-Teller (BET) to study the physical and chemical properties of the catalyst. The cooking oil and biodiesel were tested using Fourier-transform infrared spectrometer (FTIR) and gas chromatography with flame ion detector (GC-FID).

Results and Discussion

Characterization of catalysts

The functional groups on the surface of the catalysts were identified using FTIR spectrometer. Figure 1 shows the FTIR spectrum of pure SBC and SBC/KOH 500 catalysts. According to Cong et al. for pure SBC catalyst, there is a peak at 464 cm^{-1}

which indicates the bending vibration of Si-O-Si, while the peak at 1656 cm^{-1} refers to the stretching vibration of S=O [13]. The S=O bond was observed in SBC due to phyllosilicates group components presence in bleaching material of SBC [14]. Furthermore, the band at 3485 cm^{-1} for the SBC/KOH 500 corresponds to Si-O-H stretching vibration due to a water molecule absorbed on its surface [13]. In SBC/KOH 500 catalyst, S=O vibration can also be seen at around 1410 cm^{-1}.

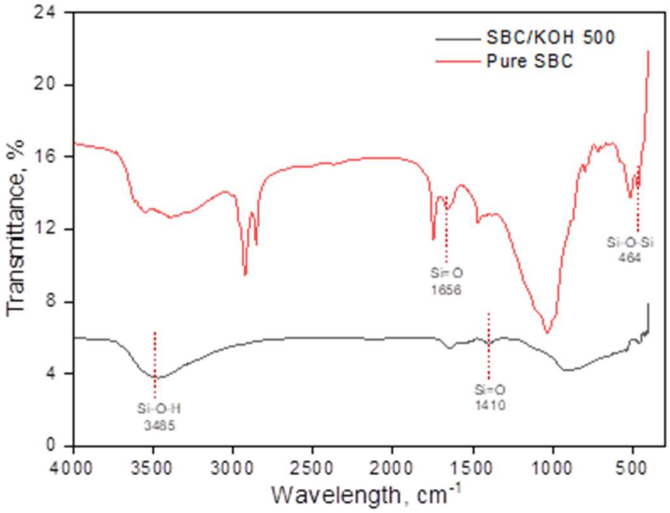


Figure 1. FTIR spectrum of pure SBC and SBC/KOH 500 catalysts

The TGA curve was obtained using thermogravimetric (TG) and derivative thermogravimetric (DTG) analysis. The weight loss curve for SBC was produced and examined using the TGA technique. The weight of SBC drops rapidly from 220 °C to 380 °C, which is due to volatile matter evaporation [13]. The second-phase weight loss indicates that the adsorbed oil on the bleaching clay contributes about 40% of the total weight. Figure 2 shows the DTG pattern in the black line suggests that peak weight loss is observed at 350 °C under an O_2 atmosphere.

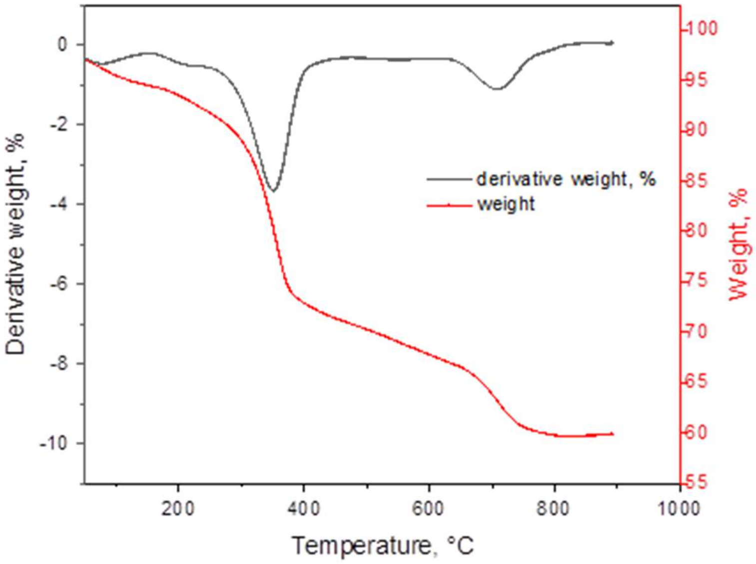


Figure 2. TGA profile of SBC as catalyst

The surface morphology and elemental compositions of the SBC/KOH 500 catalyst were identified using FESEM and EDX spectroscope. Figure 3 shows the micrograms of the SBC/KOH 500 catalyst in two different magnifications. Based on Figure 3, SBC/KOH 500 catalyst was found to have non-uniform irregular agglomerated flaky-like structures. This can be due to the calcination process, which affects the surface texture and increases the surface area for catalytic activity [11].

Next, EDX analysis was used to predict the elemental composition of the SBC/KOH 500 catalyst. The analysis result is shown in Table 1. The result proved that all elements such as K, Fe, Ca, Mg, Na, Al, and Si exist in the clay. Based on the BET analysis, SBC/KOH 500 catalyst has a higher surface area due to the higher composition of K in the catalyst (15.7 wt.%). The catalytic site for reaction increases with surface area, enhancing the productivity of the catalyst.

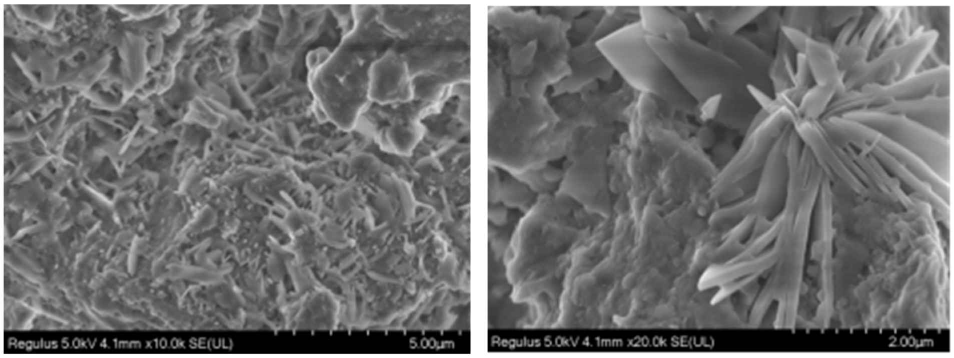


Figure 3. FESEM micrograms of SBC/KOH 500 catalyst: (i: small magnification (10k); ii: big magnification (20k))

Table 1. Elemental compositions of SBC/KOH 500 catalyst by EDX analysis

Elements	**O**	**Si**	**Al**	**Mg**	**Na**	**Fe**	**Ca**	**K**
Loading, wt.%	42.7	7.4	2.9	1.6	0.1	0.58	4.8	15.7

The XRD analysis of the SBC/KOH 500 catalyst is shown in Figure 4. The XRD pattern of SBC/KOH 500 represented the crystalline phases of quartz (SiO_2) and potassium peroxide (K_2O) [15]. The characteristic diffraction peaks for quartz (SiO_2) were found at 2θ= 20.87and 39.48° while the diffraction peaks for K_2O were observed at 2θ = 26.65, 29.46, and 36.07°. This result shows that the main component of the SBC/KOH 500 catalyst was K_2O with a small contribution from SiO_2.

The surface area, pore volume, and pore width of the catalysts were determined using BET analysis. The BET results for SBC, SBC/KOH, and SBC/NaOH catalysts were tabulated in Table 2. The surface area, pore volume, and pore width obtained for SBC/KOH catalyst were 22.62 m^2/g, 0.10 cm^3/g, and 21.05 nm, respectively. The surface area of SBC/NaOH is smaller than SBC/KOH. The higher surface area of the SBC/KOH catalyst is caused by the presence of more active sites [16]. The pore size distribution of both catalysts is represented in Figure 5.

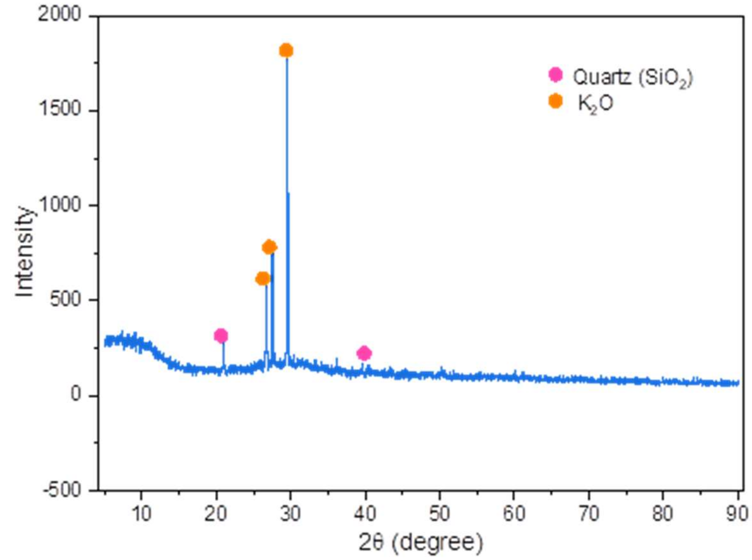


Figure 4. XRD diffractograms of SBC/KOH 500 catalyst

Table 2. Surface areas, pore volume, and pore width of the catalysts

Catalysts	BET Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Width (nm)
SBC	3.77	0.02	21.60
SBC/KOH	22.62	0.10	21.05
SBC/NaOH	8.88	0.05	26.27

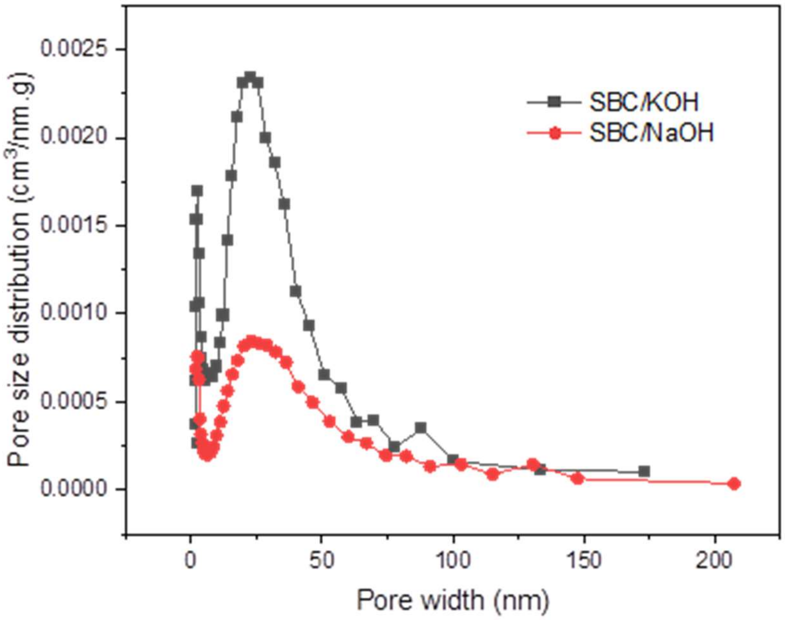


Figure 5. Pore size distribution plot of SBC/KOH and SBC/NaOH catalysts

Characterization of oil and biodiesel

Figure 6 shows the FTIR spectra of low-grade cooking oil and biodiesel produced from the SBC/KOH 500 catalyst. The stretching vibration at 1748 cm^{-1} in the FTIR spectrum indicates the presence of the C=O bond [17]. This group indicates that carbonyl functional groups are present in the oil and biodiesel. The characteristic peaks found in the region 2912 cm^{-1} and 2849 cm^{-1} indicate stretching vibrations of CH_3 and CH_2 while bending vibrations of CH_3 and CH_2 groups were found at the peak 1375 cm^{-1} and 1164 cm^{-1} respectively. The FTIR spectrum of synthesized biodiesel was found to be very similar to low-grade cooking oil due to the chemical nature similarities of the methyl esters and triglycerides. Therefore, further analysis by gas chromatography is required to support the results.

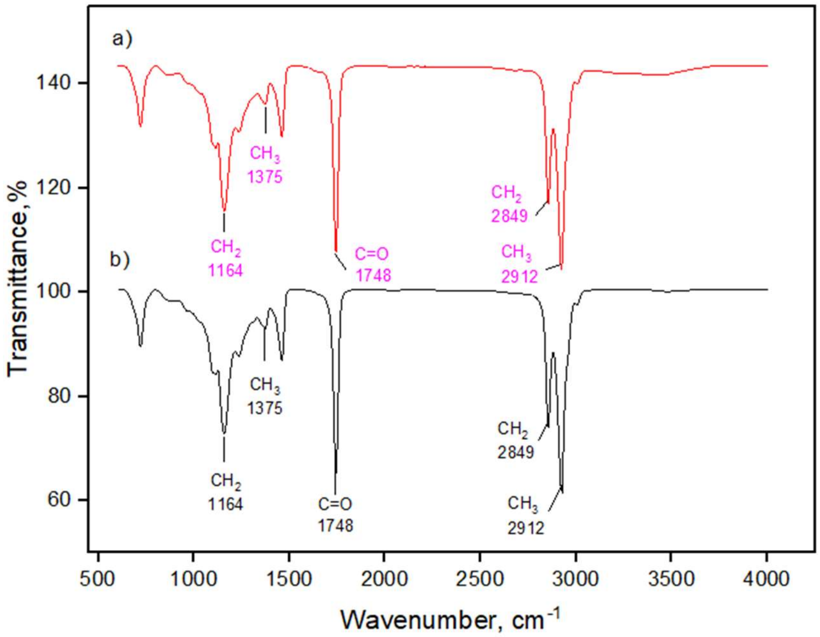


Figure 6. FT-IR spectra of low-grade cooking oil and biodiesel produced from SBC/KOH 500 catalyst

Figure 7 shows the fatty acid methyl ester (FAME) compound present in the biodiesel for SBC/KOH 500 catalyst. As described by Enguilo Gonzaga et al. low-grade cooking oil contains major fatty acids such as palmitic acid (16:0), stearic acid (18:0), oleic acid (18:1), linoleic acid (18:2), and linolenic acid (18:3) [18]. From Figure 7, the identified compounds in biodiesel were methyl myristate, methyl palmitate, methyl heptadecanoate, methyl stearate, methyl oleate and methyl linoleate. The details of the composition are tabulated in Table 3. This data was consistent with the research reported by Roschat et al. regarding the biodiesel production of Krabok seed oil through transesterification reaction using CaO as a catalyst [19]. In comparison, the biodiesel from Krabok seed oil demonstrated the major fatty acid methyl ester, namely methyl decanoate, methyl myristate, methyl palmitate, methyl heptadecanoate, methyl oleate, methyl palmitoleate, methyl stearate and methyl linoleate. The derived biodiesel using SBC/KOH 500 catalyst showed presence of similar fatty acids methyl ester. Therefore, this result shows good agreement with previous research.

Table 3. Fatty acid methyl ester (FAME) compounds present in the sample using gas chromatography with flame ion detector (GC-FID)

Sample	Tr, Retention Time (min)	ID (Peak)	FAME Compound
Biodiesel	4.376	C14:0 (8)	Methyl myristate
	5.726	C16:0 (12)	Methyl palmitate
	6.234	C17:1 (15)	Methyl heptadecanoate
	6.680	C18:0 (16)	Methyl stearate
	6.858	C18:1 (18)	Methyl oleate
	7.852	C18:3 (21)	Methyl linoleate

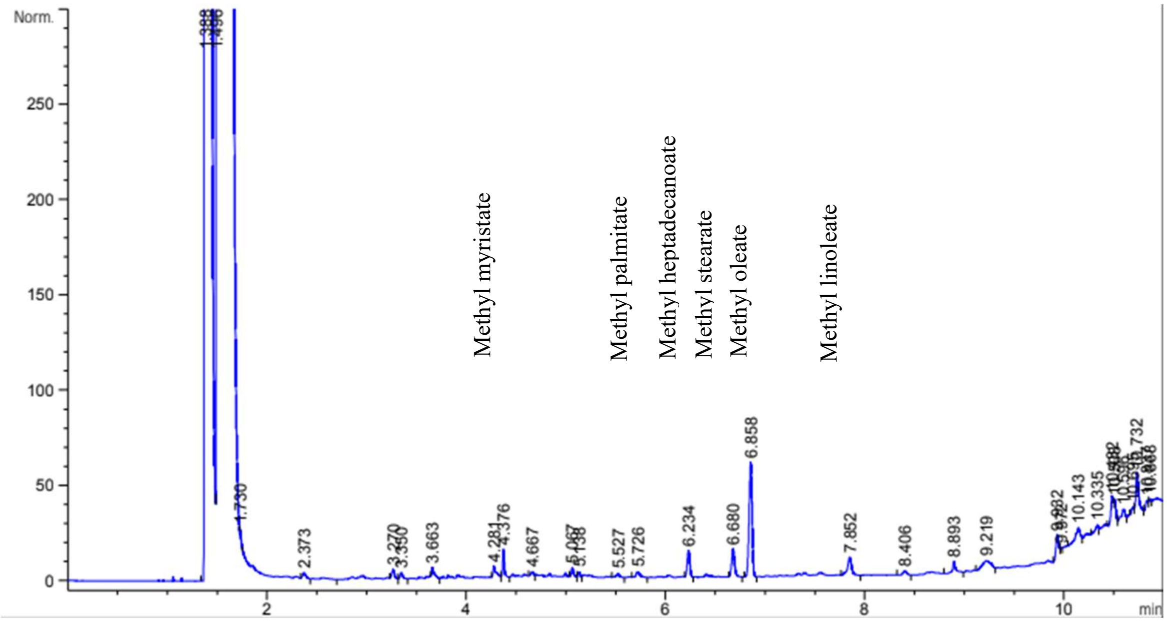


Figure 7. Characterization of biodiesel using gas chromatography using flame ion detector (GC-FID) for SBC/KOH 500 catalyst

Transesterification of low-grade cooking oil into biodiesel

Two different types of SBC catalysts were employed to study the effect of catalysts on the transesterification reaction. Figure 8 illustrates the biodiesel yield percentages obtained using SBC/KOH and SBC/NaOH. Based on the results, it is evident that the SBC/KOH catalyst outperforms the SBC/NaOH for biodiesel production, yielding a higher percentage of biodiesel (39.06%). These observations indicate that the SBC/KOH catalyst exhibits superior performance when compared to the SBC/NaOH catalyst. The high fundamental strength observed is attributed to the K^+ and OH^- pairs [18]. Besides, the higher yield can be attributed to the higher surface area of the catalyst, which provides a higher active site for the reaction. This result agrees with the findings obtained from the BET analysis. Therefore, the SBC/KOH catalyst was selected as the best catalyst for further experiments.

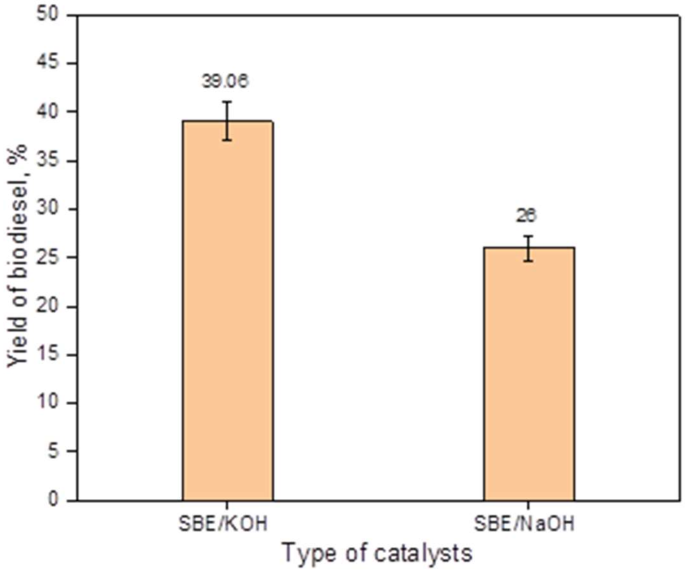


Figure 8. Effect of SBC/KOH and SBC/NaOH catalyst on biodiesel yield

The effect of calcination temperatures, specifically 500°C and 600°C, was studied using SBC/KOH. The selection of this calcination temperature was based on the findings of Cong et al., who reported it as the optimum calcination temperature for achieving high biodiesel production [13]. Hence, low calcination temperatures were chosen for energy conservation and cost reduction [20]. Figure 9 shows that the percentage biodiesel of SBC/KOH 500 and SBC/KOH 600 were found to be 47.66 and 39.06%, respectively. This finding was supported by the XRD pattern in Figure 4. The presence of SiO_2 and K_2O in XRD patterns has been found to increase the surface area of the catalyst [15]. Therefore, it can be concluded that the SBC/KOH catalyst 500 exhibits superior performance compared to the SBC/KOH 600, thereby making it the best catalyst for further experiments.

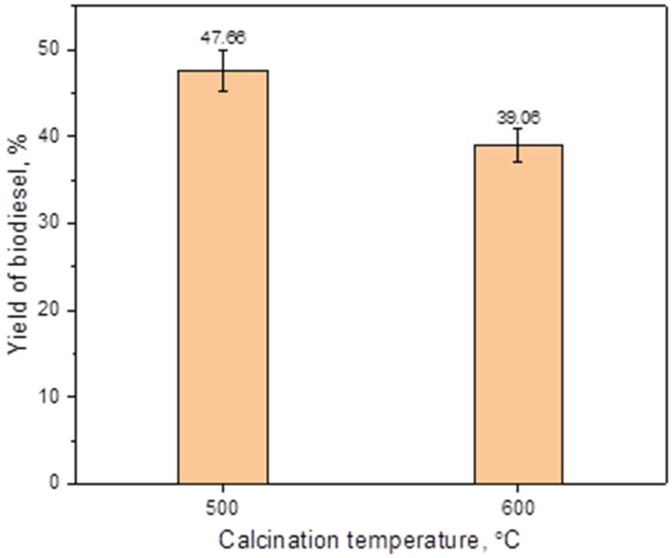


Figure 9. Effect on calcination temperature of SBC/KOH

Figure 10 displays the effect of varying the catalyst loading at 1, 2, and 4 wt.% of SBC/KOH 500. The highest biodiesel recorded was 47.66% when utilizing a catalyst consisting of 2 wt.% of SBC/KOH 500. This finding indicated that the interaction between the reactants and the solid catalyst was limited to a maximum catalyst loading of 2 wt.%, resulting in a discernible effect on the rate of the forward transesterification reaction. However, upon increasing the catalyst loading to 4 wt.%, a decrease in biodiesel production to 16.03% was observed. This is due to mixing inefficiencies resulting from the increased viscosity in the reaction vessels, which is a direct consequence of the increased catalyst concentration [21]. Since the maximum biodiesel yield was obtained when the catalyst loading was 2 wt.%, this value was chosen as the best catalyst loading for further optimization of the remaining reaction parameters.

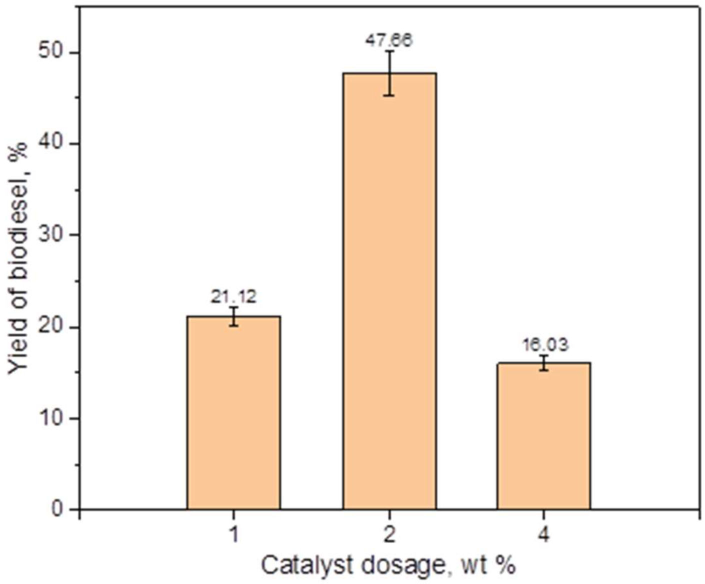


Figure 10. Effect of catalyst dosage on biodiesel yield

The effect of the ratio of oil to methanol from 1:12 to 1:20 was studied in this experiment. Based on the result in Figure 11, the percentage yield of biodiesel for the ratio of 1:12, 1:16, 1:18, and 1:20 were 30, 46.82, 47.14, and 47.66%, respectively. When the ratio of oil-to-methanol increased, the product yield also increased indicating that a large amount of methanol is required to reduce the viscosity of the reaction media and to enable the contact between the two phases [22]. Therefore, the 1:20 ratio of oil to methanol was chosen as the optimal value for the following studies.

The present study examined the effect of reaction time, with the corresponding result shown in Figure 12. The biodiesel production increased from 26.88 to 47.66% as the reaction time was increased from 1 to 2 hours. The likely reason for this observation can be attributed to the presence of the heterogeneous mass transfer system of the catalyst [18]. At 3 hours of reaction time, the biodiesel production decreased to 4.94% due to the backward reaction in the methyl ester, which produces more glycerol than methyl ester [19]. Hence, the reaction time of 2 hours was selected as the optimum time.

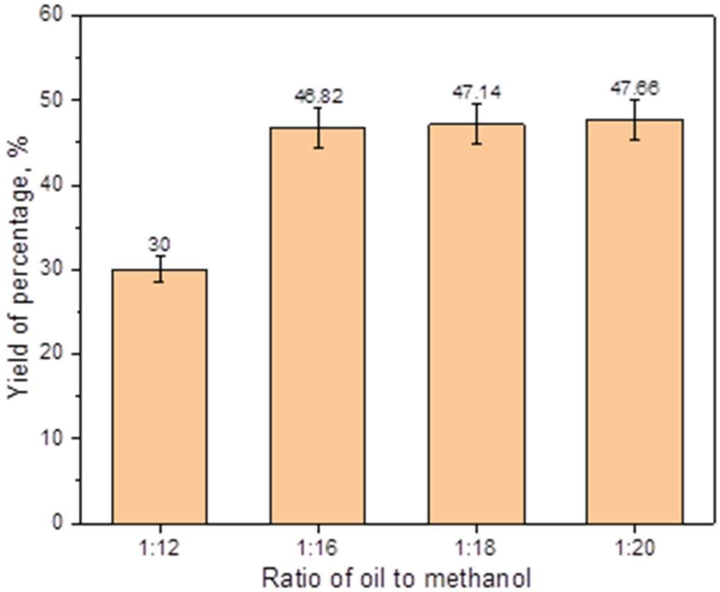


Figure 11. Effect of ratio oil to methanol in transesterification

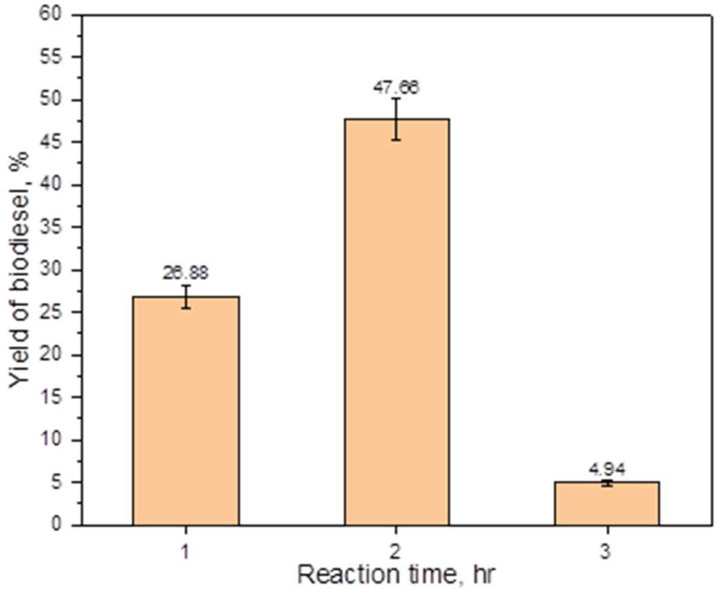


Figure 12. Effect of reaction time on the transesterification process

Conclusion

In this study, the catalytic activity of the SBC/KOH 500 catalyst for the transesterification of cooking oil to FAME was investigated. Based on the result, the SBC/KOH 500 showed the potential as a catalyst in the transesterification process and can be used as an alternative catalyst to produce biodiesel from low-grade cooking oil. The highest yield of biodiesel was 47.66% with transesterification reaction condition of 2 hours reaction time, 1:20 oil to methanol ratio, 2 wt.% catalyst loading, and temperature of 55 °C.

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