

Sustainable Biodiesel Production from Waste Cooking Oil Using Diatomite as a Catalyst

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Abstract

Biodiesel production from waste cooking oil via the transesterification process is one of the sustainable alternatives to fossil fuels. This study explores single use of diatomite as an alternative catalyst for biodiesel production that can significantly benefit the green energy industry. The experiment manipulated the methanol-to-oil ratio and catalyst dosage required by transesterification of fresh and waste cooking oil to maximize biodiesel yield. The use of diatomite in the transesterification of waste cooking oil produced a biodiesel yield of 90.49%, which was closely comparable to the 93.28% yield obtained from fresh cooking oil. The optimal condition was achieved with a 6:1 molar ratio of methanol to oil and 1.0 wt% diatomite. FTIR analysis of biodiesel sample from fresh and waste cooking oil produce new peaks at 1435 cm^{-1} attributed to methyl bending vibration that occurs in alkanes which reflects characteristic of the biodiesel composition. In addition, presence of methyl ester was confirmed at 1196 cm^{-1} which verified successful transesterification process. Diatomite's reusability was efficient, achieving up to 70% conversion after three cycles, showing its potential to reduce biodiesel production costs. This study demonstrated that diatomite was an effective catalyst for the transesterification process, contributing to the advancement of sustainable biodiesel energy.

1. Introduction

Biodiesel is a renewable fuel that can be produced from natural feedstock such as vegetable oils, animal fats, algae and waste cooking oil. It serves as a sustainable alternative to conventional petroleum-based diesel which provides significant environmental benefits especially on reducing greenhouse gas emissions and improving air quality. Biodiesel is produced via transesterification process which converts triglycerides, the primary components of oils and fats, into methyl or ethyl esters in the form of biodiesel and also generate glycerol, a valuable by-product as shown in figure 1 [1]. This process involves reaction of triglycerides with an alcohol typically methanol in the presence of a catalyst such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). Transesterification reactions occur in three steps where its start with breakdown of triglycerides into diglycerides

and monoglycerides which later convert the monoglycerides into esters, and finally separating the esters (biodiesel) from glycerol [2].

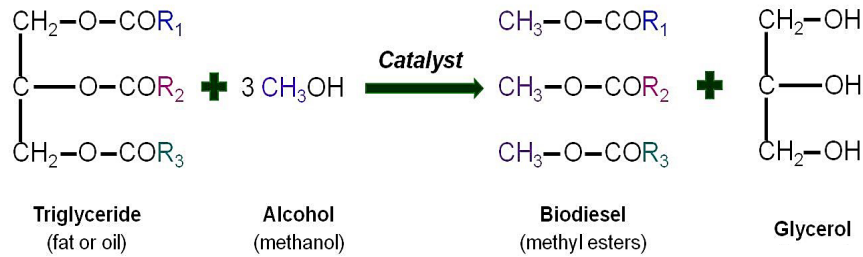


Fig. 1 Transesterification of triglyceride with methanol

Several crucial factors must be measured to maximize biodiesel yield in transesterification process such as type of catalyst, the alcohol-to-oil molar ratio, reaction temperature, reactant purity, and the free fatty acid (FFA) content [3]. These variables significantly influence the efficiency and outcome of the transesterification reaction. Catalysts play a vital role in enhancing the reaction's efficiency, with sodium hydroxide (NaOH) and potassium hydroxide (KOH) being the conventional catalyst used in biodiesel industry. Generally, catalysts used in transesterification are classified into two categories which are homogeneous and heterogeneous catalysts [4]. The difference exists in their downstream processing requirements, particularly in the purification of biodiesel. Homogeneous catalysts, typically easier to implement, require extensive purification steps, while heterogeneous catalysts offer advantages in recyclability and simplified separation processes [5]. The careful selection and optimization of these factors are essential for achieving high biodiesel yield and quality in industrial applications.

Most catalysts used in transesterification are non-recyclable, which presents challenges for sustainability and cost-effectiveness in biodiesel production. In this study, diatomite will be explored as an alternative catalyst that can be reused for the next cycle of transesterification process. Diatomite is a naturally occurring sedimentary rock mainly composed of the fossilized remains of diatoms, which are microscopic algae [6]. These diatoms have hard, silica-based cell walls which accumulate in aquatic environments over time. The material is light, porous, and has a high surface area, making it useful in various industrial applications. Diatomite is commonly used as a filtration aid, an abrasive, and an insulating material [7, 8, 9]. In the context of catalysis, diatomite's high porosity and surface area make it an excellent candidate for use as a support for catalysts or as a catalyst itself. Study by Modiba et al. [10] explore potential of heterogeneous catalyst by doping potassium hydroxide (KOH) onto diatomite on transesterification of waste vegetable oil into biodiesel. The result obtained shows that maximum biodiesel yields up to 90% could be attained when methanol to oil ratio used was 30%, and catalyst to oil ratio reached 5.0% within 4-hour reaction time. Another work utilizing waste cooking oil as feedstock for biodiesel production achieved maximum of 96.47% biodiesel yield when diatomite supported by CaO/MgO composite were used as catalyst at optimum condition of 6% catalyst and 15:1 methanol to oil ratio [11]. Considering the high methanol-to-oil ratio and the catalyst dosage requirements, the efficiency of diatomite as a single-use catalyst has not been fully realized, emphasizing the need to explore its potential to enhance cost efficiency.

In this work, the aim was to explore diatomite as a single-use catalyst for biodiesel production using waste cooking oil, aiming to improve sustainability and reduce dependence on conventional catalysts. The study also seeks to determine the optimal parameter for the transesterification process such as dosage of catalyst, temperature, and alcohol-to-oil ratio to maximize biodiesel yield. Additionally, the reusability of diatomite as a catalyst will be thoroughly investigated to assess its potential for repeated use and its impact on the overall process efficiency.

2. Materials and Methods

2.1 Material

Waste cooking oil served as biodiesel feedstock was collected from a local restaurant in a nearby area. The waste cooking oil was filtered several times to remove the unwanted moisture in the oil. Natural diatomite used in this study was obtained from China. Prior to use, diatomite was thoroughly rinsed, ground manually into powdered form and washed with deionized water. It was then dried in an oven at 60°C overnight before being used as a catalyst in the transesterification process. Methanol (99.8%), sodium hydroxide (NaOH) and potassium hydrogen phthalate (KHP) were supplied by a local chemical supplier.

2.2 Method

2.2.1 Experimental Set-up

Table 1 shows the operating conditions for the transesterification process while figure 2 describes the apparatus setup used in this research. A 250 ml three-neck bottom flask was connected to a condenser and a thermometer was placed in the flask to monitor the temperature of the reaction.

Table 1 Experimental condition for transesterification process

Parameter	Specifications
Temperature(°C)	60- 70
Catalyst Weight (%)	0.5- 1.0
Reaction Time	Not Fixed
Methanol to Oil Ratio	3: 1 - 12:1

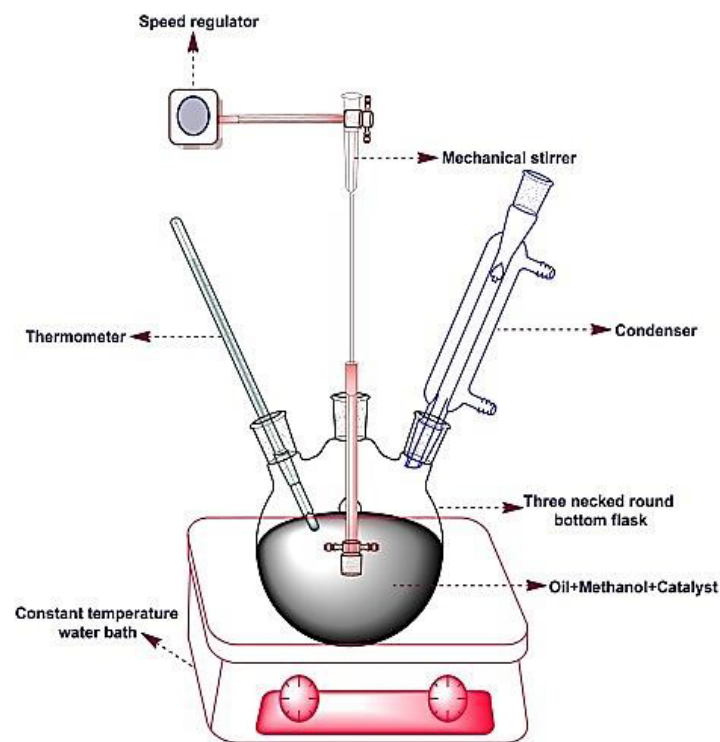


Fig. 2 Transesterification process set-up

2.2.2 Preparation of Sodium Hydroxide Solution

The titration method was conducted to determine the acid value and the percentage of free fatty acids (FFA) in the oil using sodium hydroxide (NaOH). This procedure was done to ascertain the dosage of catalyst required for the transesterification process [10, 11]. Four grams of NaOH were dissolved in a 1000 mL volumetric flask and the solution was then standardized to determine the molarity of the NaOH. To standardize the solution, four grams of potassium hydrogen phthalate (KHP) were added to the conical flask and mixed with 50 mL of water and a few drops of phenolphthalein as an indicator. The solution was titrated with the NaOH until the first appearance of a pink color was observed which demonstrated the completion of the neutralization process. The molarity of the NaOH solution was then calculated using Equation 1:

$$\text{Molarity of alkali} = \frac{(M \times 100)}{(V \times 204.2)} \quad (1)$$

where M is the mass of potassium hydrogen phthalate in grams and V is the Volume in milliliters of sodium hydroxide.

2.2.3 Transesterification Process

The waste cooking oil was transferred into a three-neck bottom flask. In a separate beaker, methanol and diatomite were prepared according to the specified dosages, which were based on the feedstock amount. The prepared solution was then added to the three-neck bottom flask. The stirrer speed was set to the desired control, and the flask was heated to a temperature range of 60°C to 70°C. Once the temperature reached 60°C, the reaction time was recorded, and the transesterification process commenced. At 10-minute intervals, titration was performed to monitor the conversion of free fatty acids (FFA) in the waste cooking oil. The percentage of FFA was calculated using Equation 2 [14].

$$\text{Percentage of free fatty acid, FFA (\%)} = \frac{\text{Volume of NaOH} \times \text{Molarity} \times 25.6}{\text{Weight of sample (g)}} \times 100\% \quad (2)$$

Once the reaction reached the desired conversion, the biodiesel mixture was allowed to settle. The settling process resulted in the separation of the biodiesel into three phases. The catalyst, diatomite, was separated from the biodiesel using a pipette and recovered for reuse in the next cycle. The biodiesel was then purified using a rotary evaporator (rotavapor) to remove residual methanol and water.

2.2.4 Biodiesel Purification Process

The purification process was done by using BUCHI Rotavapor®R-210. During the drying process, this machine was used to evaporate the water and methanol from the biodiesel sample. The evaporation process was repeated twice for complete removal of water and methanol. The process was carried out at 90°C and the pressure was set to be 100mbar.

2.3 Catalyst Reusability

Diatomite was separated from the previous transesterification reaction mixture via centrifugation. 50 ml reaction mixture was centrifuged at 3500 rpm for 10 minutes. The supernatant was decanted, and the diatomite precipitate was recovered and thoroughly washed with hexane for 20 minutes to remove residual oil and methanol [15]. Washed diatomite was dried overnight in an oven at temperature of 60°C. The catalytic performance of recovered diatomite was tested by conducting three consecutive transesterifications of fresh and waste cooking oil. The dried diatomite was subsequently reused in the next cycle, which was carried out for 1 hour under fixed conditions. For fresh cooking oil, 0.25 wt% diatomite and a 6:1 molar ratio of methanol was used, while conditions for waste cooking oil were fixed to 0.5 wt% diatomite with the same 6:1 molar ratio of methanol. The biodiesel yield was calculated after each cycle as in Equation 3 [16].

$$\text{Biodiesel yield, \%} = \frac{\text{Weight of biodiesel produced (g)}}{\text{Weight of oil (g)}} \times 100 \quad (3)$$

2.4 Analytical Methods

2.4.1 Fourier Transform Infra-Red (FTIR) Analysis

The functional groups and chemical bonding presence on the sample for biodiesel production was determined by using Nicolet 6700 FTIR spectrometer (Thermo Nicolet Corp., Madison, WI). The sample was characterized using Attenuated Total Reflection (ATR) method and the spectrum was recorded between the range of 650 – 4000 cm⁻¹ at the resolution of 4 cm⁻¹.

2.4.2 Scanning Electron Microscopy (SEM) Analysis

The surface morphology of diatomite before and after transesterification process were examined using SEM, model Hitachi Microscope TM3030Plus. The sample were observed at the accelerated voltage of 15-kV under various magnifications from 50 μm - 100μm.

3. Results and Discussion

3.1 Effect of Methanol Ratio on Biodiesel Yield

Several sets of experiments were conducted by varying the molar ratio of methanol to oil, ranging from 3:1 to 12:1. The catalyst concentration was selected based on the optimized results from the preliminary reaction.

Typically, the reaction of three moles of methanol per mole of triglyceride produces three moles of biodiesel and one mole of glycerol. However, the maximum biodiesel yield was achieved at higher molar ratios, specifically 6:1 and 12:1, for both fresh and waste cooking oil, as shown in figure 2.

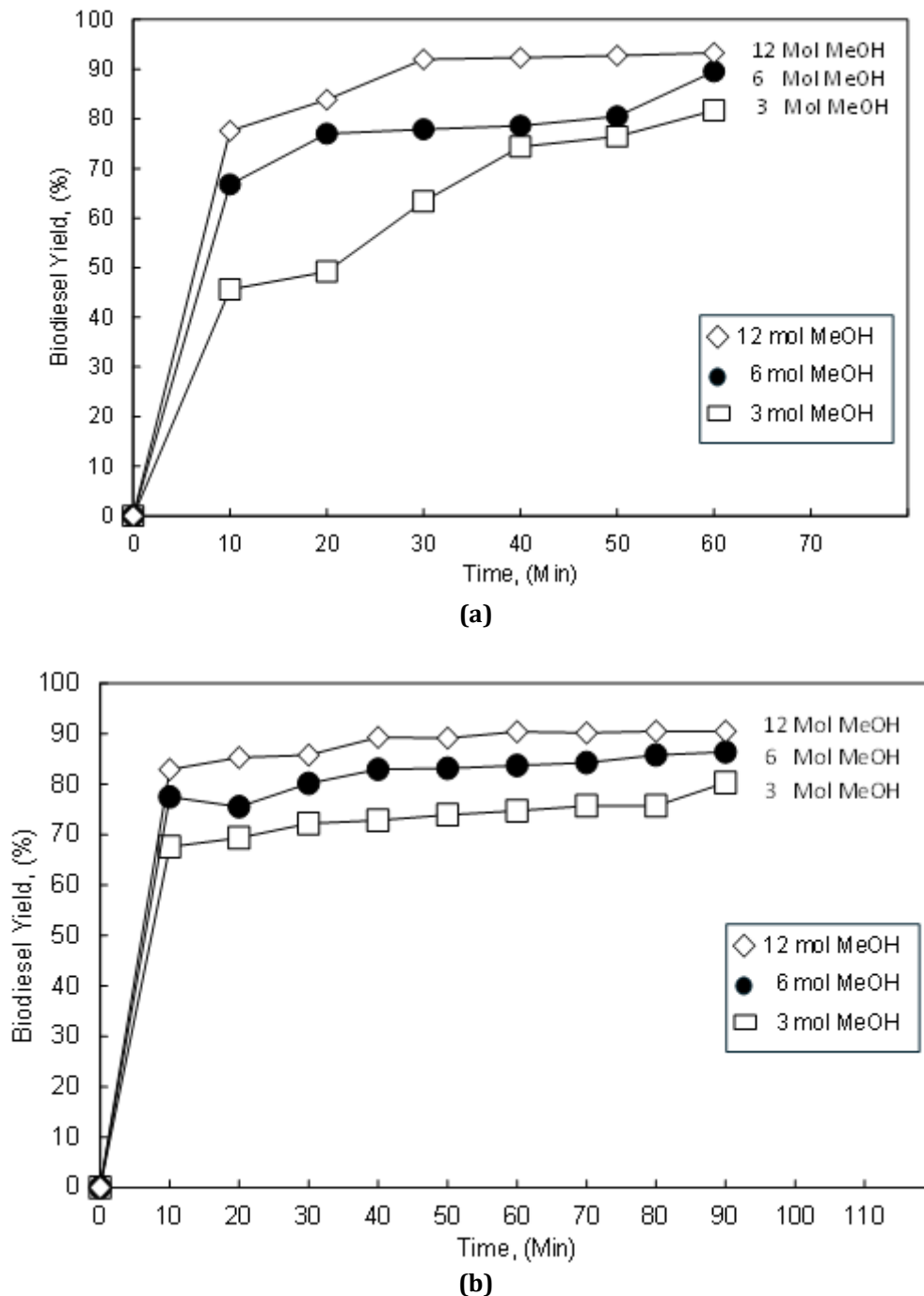


Fig. 3 Effect of methanol molar ratio on the transesterification of (a) Fresh cooking oil; and (b) Waste cooking oil

As shown in figure 3, higher biodiesel conversion and a more complete reaction can be achieved by using a molar ratio greater than the stoichiometric value for the transesterification process. The biodiesel yield for both fresh and waste cooking oils increased when the molar ratio was raised from 3:1 to 6:1, with fresh cooking oil yielding 81.67% to 89.56%, and waste cooking oil yielding 80.31% to 86.37%. At a 12:1 molar ratio, the biodiesel yield showed minimal variation compared to the 6:1 ratio, but it required a longer settling time. This is due to the large amount of methanol added, which complicates the separation of the biodiesel and water layers. The excess methanol enhances emulsification, owing to the presence of the polar hydroxyl group in methanol, making biodiesel recovery more challenging and increasing the cost of methanol recovery [13, 14]. Figure 3 illustrates the differences in biodiesel yield for both fresh and waste cooking oils in relation to the molar ratio.

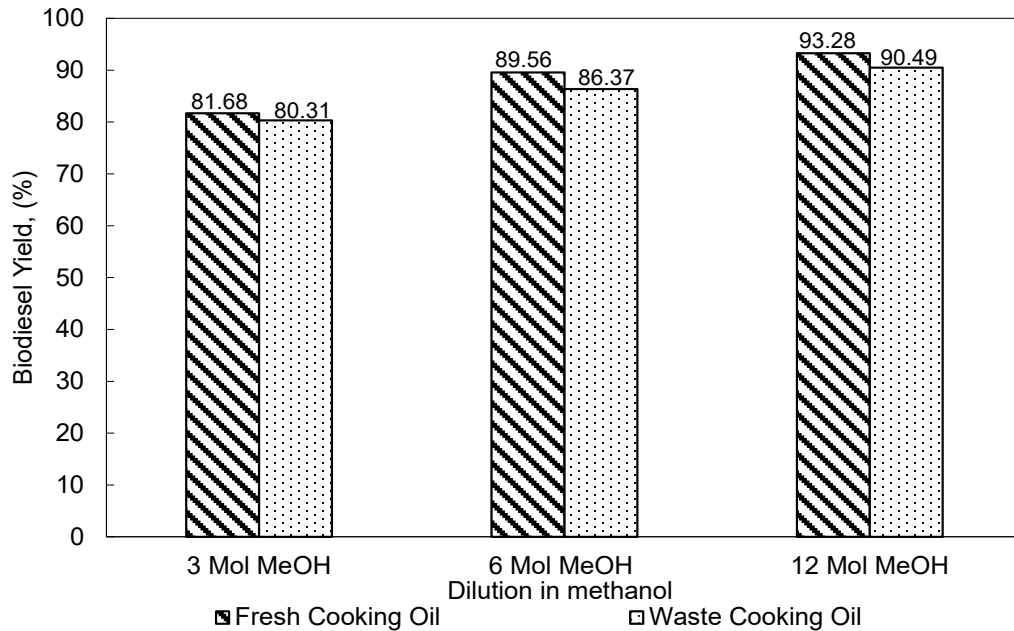


Fig. 3 Biodiesel yield with molar ratios of fresh cooking oil and waste cooking oil

Biodiesel yield from waste cooking oil is lower than from fresh cooking oil, due to higher viscosity and impurities. Waste cooking oil has higher viscosity, which requires more methanol to improve solubility and enhance the interaction between oil and methanol, optimizing the conversion process [19]. Figure 3 demonstrates that a molar ratio of 12:1 produced the highest biodiesel yield for both fresh cooking oil (93.28%) and waste cooking oil (90.49%). These results are consistent with findings by Razuki et al. [20] and Ghavami et al. [21], demonstrated that biodiesel yields from waste cooking oil can be increased by employing a high molar ratio of methanol with a reduced catalyst dosage. Additionally, Eswararao et al. [22] achieved a biodiesel yield of 97.64%, though their process required a reaction time of up to 3 hours and a catalyst dosage of 7 wt%. In contrast, for this study, the optimized methanol molar ratio was set at 6:1, as it is more cost-effective, and the yield can be further enhanced by increasing the catalyst dosage. The results indicate that the molar ratio of methanol to oil is a critical factor influencing biodiesel yield and purity, alongside other variables such as catalyst concentration and reaction time.

3.2 Effect of Catalyst Dosage on Biodiesel Yield

The effect of diatomite dosage on the transesterification process was assessed by varying its concentration from 0.25wt% to 1 wt%, based on the feedstock weight. A methanol-to-oil molar ratio of 6:1 was used for both waste cooking oil and fresh cooking oil. Figure 4 illustrates the change in diatomite color after the transesterification process.

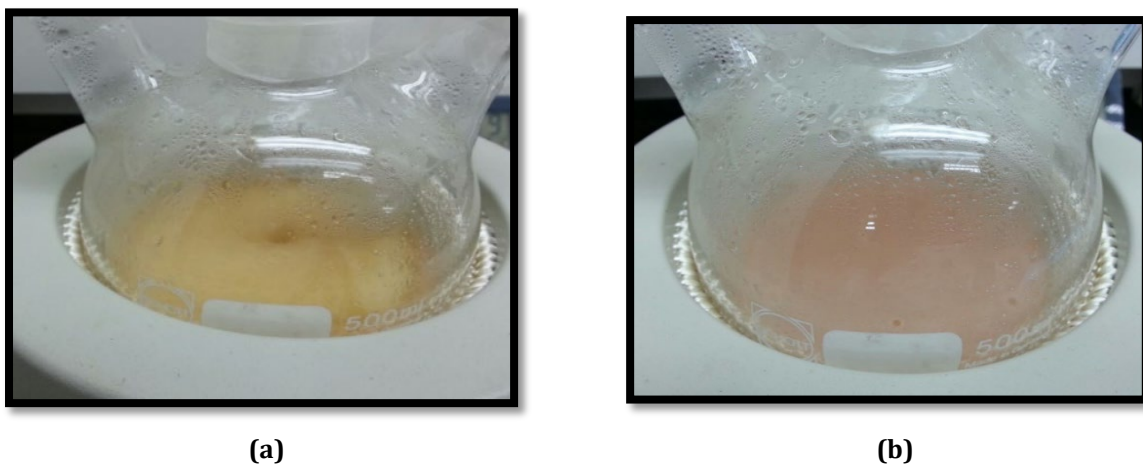


Fig. 4 Change in diatomite color for (a) Waste cooking oil; (b) Fresh cooking Oil

The diatomite undergoes a color change during the transesterification process due to its reaction with the methanol-oil mixture. For fresh cooking oil, the diatomite changes from white to pink, while for waste cooking oil, it turns light brown. The color changes to light brown in the case of waste cooking oil is attributed to the presence of impurities in the feedstock. Figure 5 illustrates the impact of catalyst dosage on the biodiesel conversion during the transesterification process.

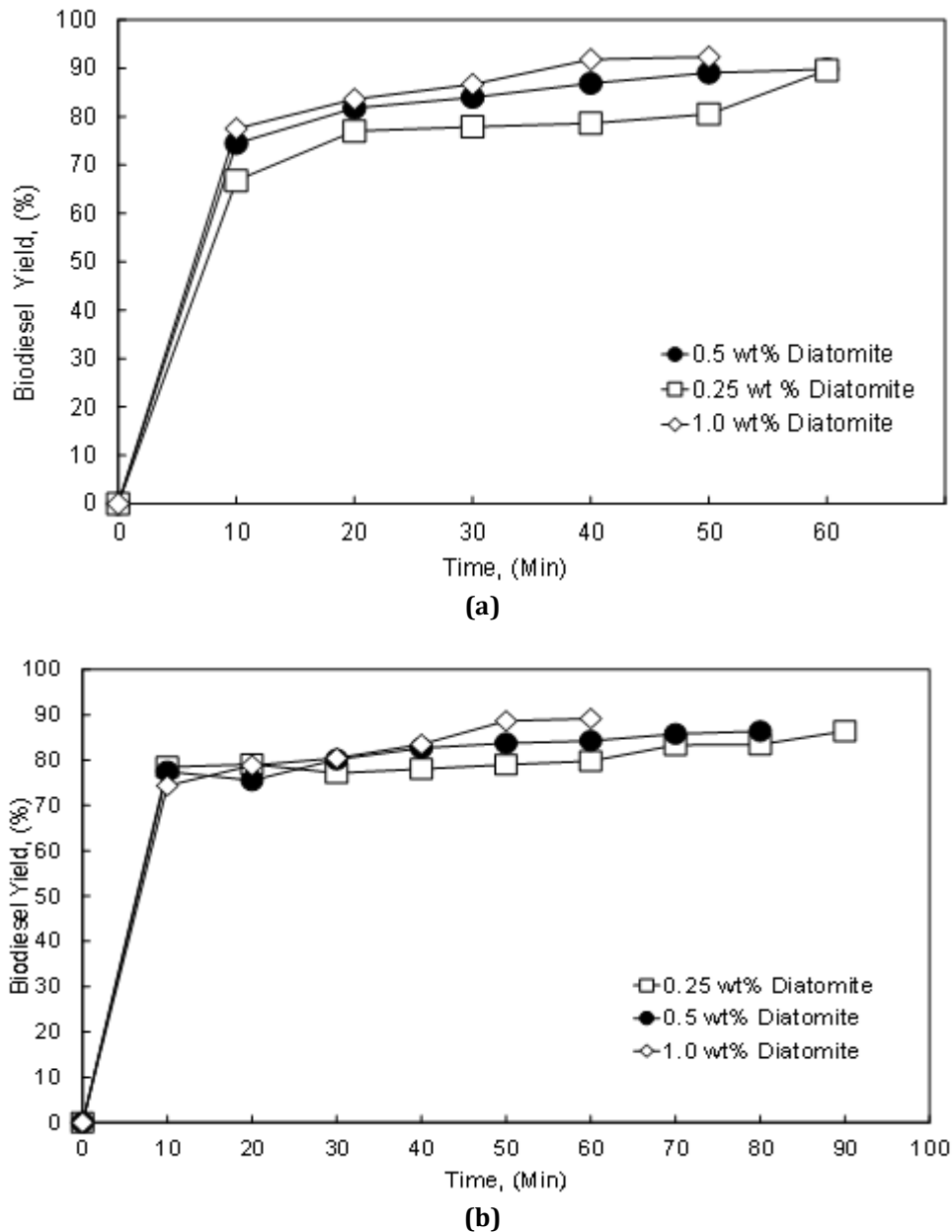


Fig. 5 Effect of catalyst dosage on the transesterification of (a) Fresh cooking oil; and (b) Waste cooking oil

The experimental results in figure 5 demonstrate a direct relationship between catalyst dosage and biodiesel yield for both fresh and waste cooking oil. The highest biodiesel conversion occurs when 1.0 wt% diatomite is used, as a higher catalyst dosage increases the number of active sites available for the reaction, leading to better biodiesel yield. Additionally, a higher catalyst dosage reduces the reaction time required to achieve optimal biodiesel yield. While the yield for both fresh and waste cooking oils showed minimal variation when the catalyst dosage increased from 0.25 wt% to 0.5 wt%, the reaction time for waste cooking oil to reach its maximum yield was shortened by 10 minutes. Overall, the catalyst not only increased the biodiesel yield but also improved the reaction's efficiency, reducing the time needed for the transesterification process. The overall biodiesel yield is shown in figure 6.

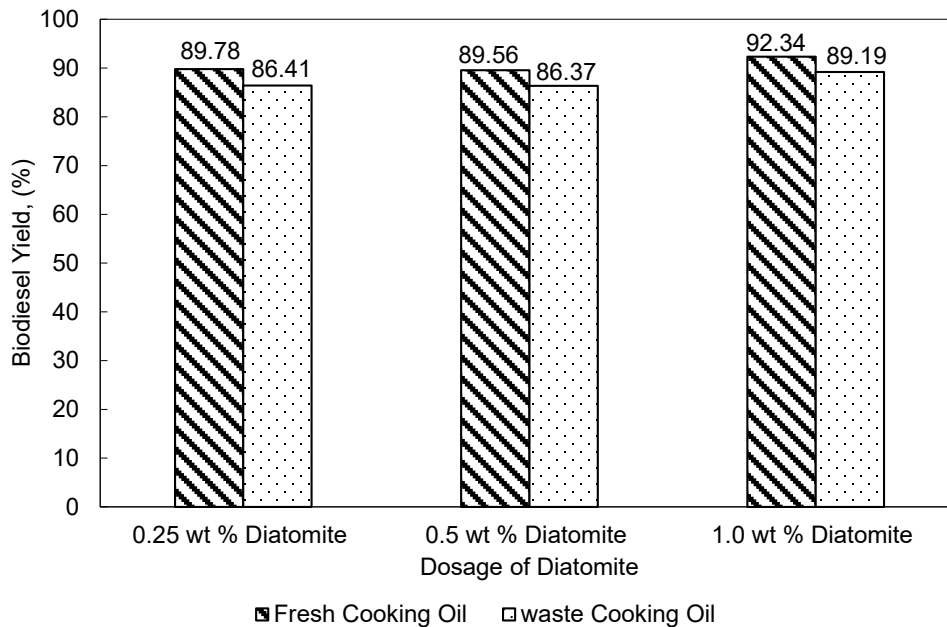


Fig. 6 Biodiesel yield with dosage of catalyst of fresh cooking oil and waste cooking oil

Using diatomite as a catalyst, biodiesel yield from waste cooking oil reached 86.37% with a 0.5 wt% catalyst dosage and a 6:1 molar ratio of methanol in just 80 minutes. In comparison, Lee et al. [23] achieved the same biodiesel yield with a 5 wt% calcium oxide (CaO) catalyst and a 12:1 molar ratio of methanol over 6 hours. Similarly, conventional catalysts such as potassium hydroxide (KOH) doped with alumina required a higher catalyst loading of up to 7%, a methanol-to-oil ratio of 12:1, and a longer reaction time of up to 2 hours to achieve a maximum biodiesel yield of 73.7% [24]. In this work, 0.5wt% of diatomite as catalyst capable to attain more biodiesel yield compared to conventional catalyst. However, it was reported that doping of diatomite with other catalyst capable to achieve higher biodiesel yield more than 90%. Study by Abukhadra [25] successfully attained 98.1% of biodiesel yield from waste cooking oil when 3.25% wt of catalyst derived from kaolinite and diatomite were used under experimental conditions of 40°C, 12:1 methanol to oil ratio with 90 min of transesterification reaction. Additionally, doping of diatomite with $\text{Fe}_2(\text{SO}_4)_3$ & $\text{Zr}(\text{SO}_4)_2$ as a catalyst produce biodiesel yield at 98.9% from waste cooking oil but this method require 10% loading of catalyst in the transesterification process [26]. High dosage of catalyst incurred additional costs due to its expense and the need for a complex pre-treatment process, leading to increased production costs. In this work, the highest biodiesel yields for fresh cooking oil and waste cooking oil using single use of diatomite were 92.34% and 89.19%, respectively, achieved in 50 and 60 minutes. Since the yield difference between 0.5 wt% and 1.0 wt% diatomite for fresh cooking oil was minimal, 0.25 wt% diatomite was chosen as the optimal catalyst dosage to reduce costs. For waste cooking oil, however, 1.0 wt% diatomite remained the most effective catalyst dosage.

3.3 Reusability of Catalyst

There are two main features that must be considered when selecting a catalyst which are stability and reusability. Therefore, to investigate the reusability of the catalyst, the catalyst was pre-treated and reused under optimum reaction condition. The optimum condition for fresh cooking oil was 0.25wt% diatomite, 6:1 molar ratio of methanol whereas for waste cooking oil was 0.5wt% diatomite, 6:1 molar ratio methanol and all the reactions were carried out for 1 hour. As depicted in figure 7, the biodiesel conversion decreased slightly with each reuse of the catalyst but remained above 75% after three cycles. The catalyst performed similarly for both fresh and waste cooking oil. The reduced activity may be due to pore blockage or the accumulation of product layers or intermediates, which limit its effectiveness. Figure 8 shows the biodiesel yield decreased after each reuse of the catalyst. In the second run, the yield dropped by 5.33% for fresh cooking oil and 4.93% for waste cooking oil. By the third run, the biodiesel yield decreased by approximately 8% for both oils. In contrast, Lee et al. [23] demonstrated that waste obtuse horn shell-derived CaO catalysts could be reused up to three times, yielding 70% biodiesel in the final two runs. However, diatomite showed better reusability, maintaining a yield of 85% in the second run and 5% higher yield in the third run compared to other studies. In industrial settings, catalysts such as potassium hydroxide or sodium hydroxide are non-reusable, leading to wastewater generation during the separation process [27]. Therefore, diatomite proves to be an effective catalyst with good reusability and a competitive economic advantage. Further research is recommended to enhance the stability and properties of diatomite.

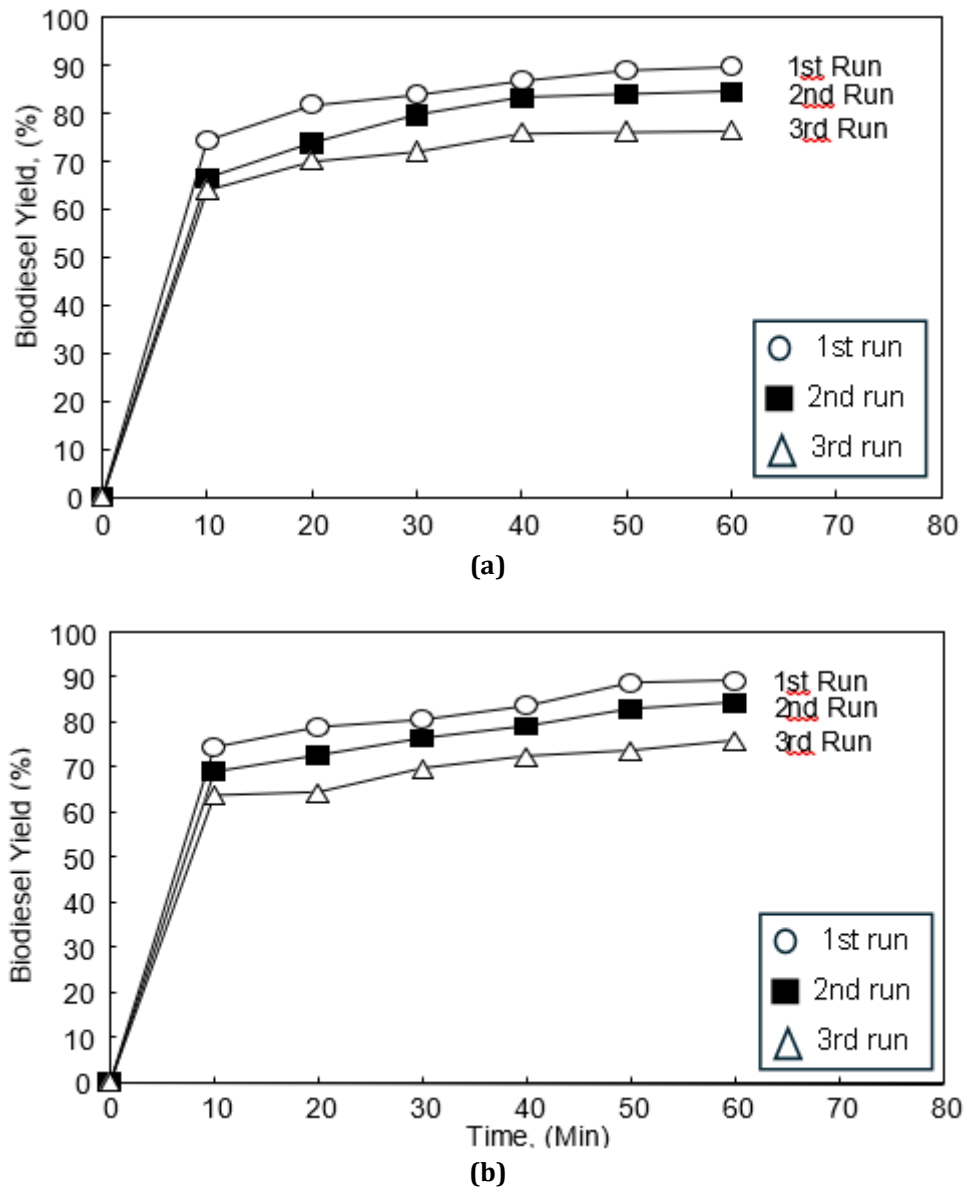


Fig. 7 Reusability of catalyst for (a) Fresh cooking oil; and (b) Waste cooking oil

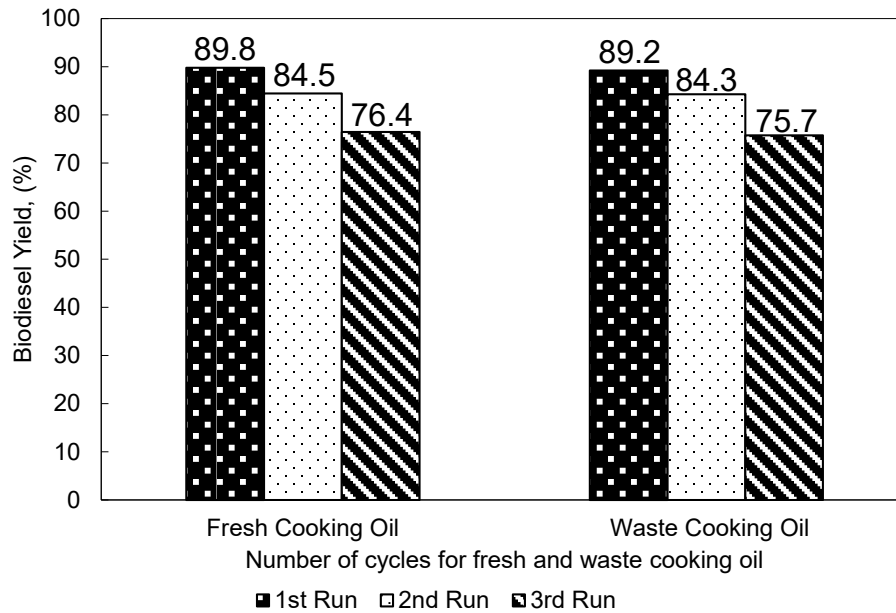
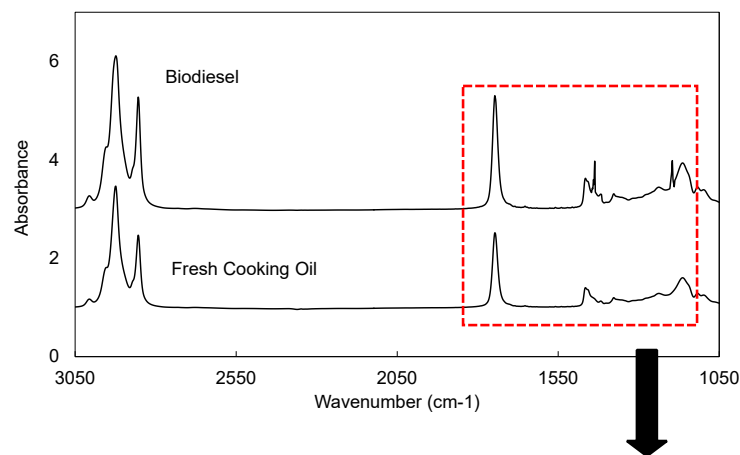


Fig. 8 Reusability of catalyst for fresh cooking oil and waste cooking oil

3.4 FTIR Analysis

The FTIR spectra of biodiesel produced from waste cooking oil and fresh cooking oil were compared with the spectra of their respective initial feedstocks, as illustrated in figures 9 and 10. This comparison highlights the differences in the spectra before and after the transesterification process. Based on the FTIR results, no significant peaks were observed in the range of 1700–2899 cm^{-1} for both feedstock and biodiesel samples. However, strong peaks between 2800–3000 cm^{-1} were observed in all samples, indicating stretching mode of CH_2 and CH_3 from lipids in the form of free fatty acid chain in the biodiesel samples [28]. In contrast, fresh and waste cooking oils display CH_2 and CH_3 stretching vibrations, representing lipids in the form of oils and fats, respectively [29, 30]. Furthermore, ester carbonyl ($\text{C}=\text{O}$) group of triglycerides were identified at 1740 cm^{-1} in all samples aligned with the findings from Sharma et al. [31]. A distinct characteristic peak at 1435 cm^{-1} , resulting from the transesterification of fresh and waste cooking oil was observed in the biodiesel spectra while absent in fresh and waste cooking oil spectra. This peak corresponds to the methyl bending vibration that occurs in alkanes which reflects characteristic of the biodiesel composition [28, 32]. Additionally, another peak that distinguishes biodiesel from fresh and waste cooking oil can be found at 1196 cm^{-1} correspond to $\text{C}-\text{O}$ (CH_3O) stretching in accordance with the observations of Sree et al. [33] who also highlighted these peaks as key indicators for biodiesel characterization. Since biodiesel is a mixture of long and short-chain methyl esters, appearance of $-\text{COC}$ stretching modes of the ester functional group in the biodiesel samples indicates presence of methyl ester which is the key component of fatty acid methyl esters (FAMES) [28, 30].



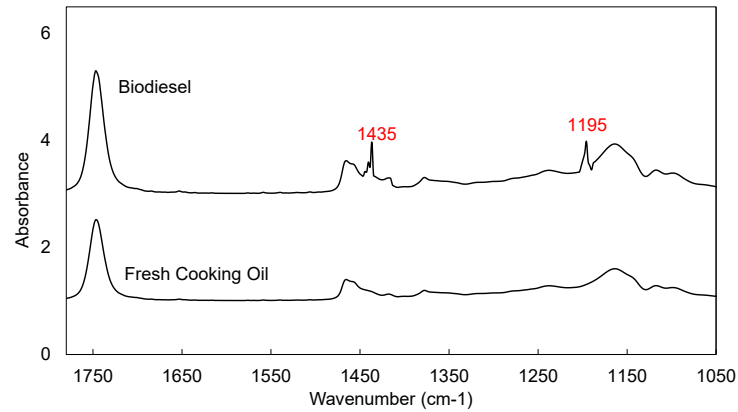


Fig. 9 FTIR spectra for fresh cooking oil and biodiesel

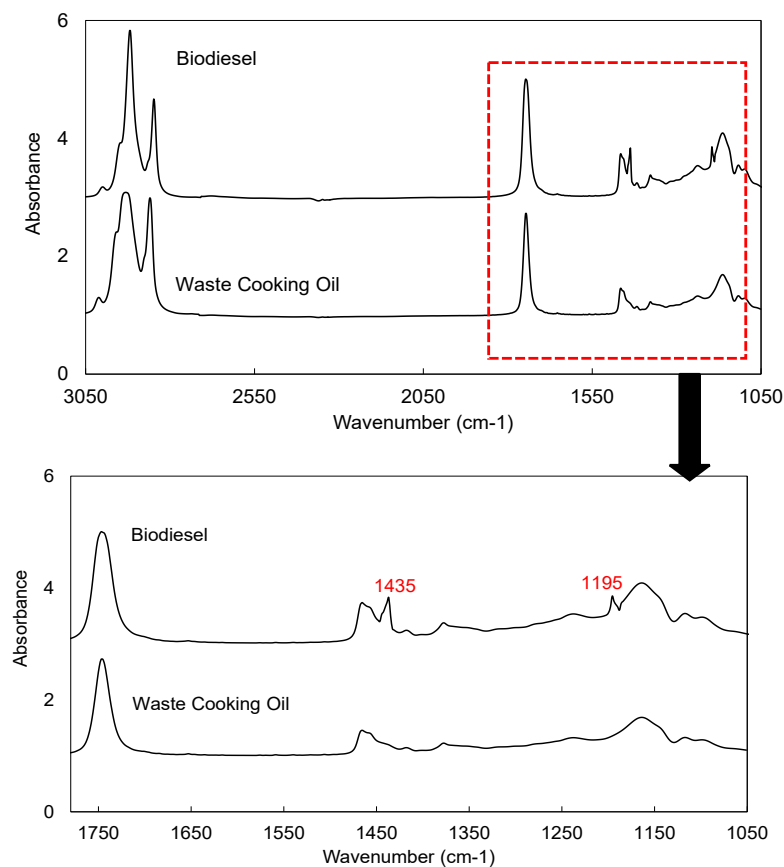


Fig. 10 FTIR spectra for waste cooking oil and biodiesel

3.5 SEM Analysis

SEM analysis was performed on diatomite samples before and after the transesterification process to observe changes in morphology and reactivity. Figures 11 show the SEM micrographs, highlighting noticeable differences in surface textures between the samples before and after the reaction.

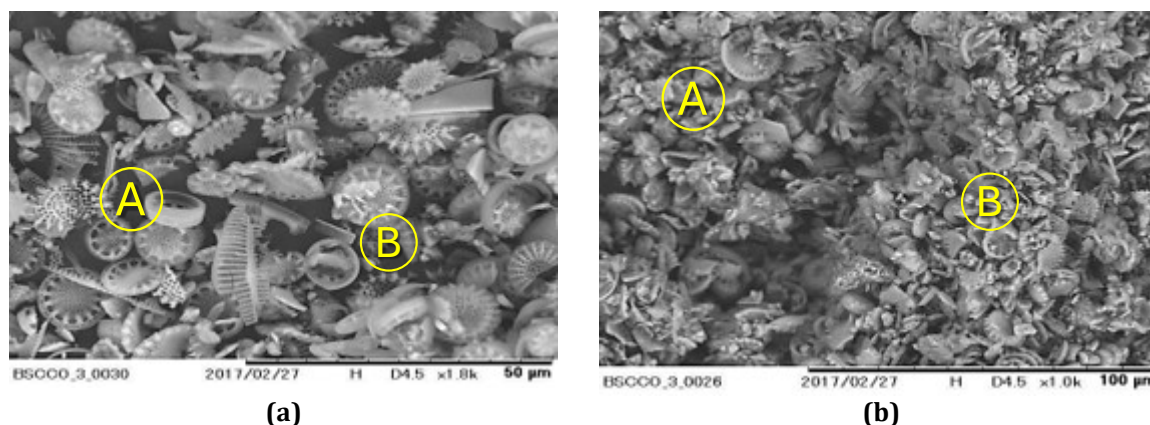


Fig. 11 Microscopy image of (a) Raw diatomite; and (b) Diatomite after transesterification reaction

Diatomite in figure 11(a) shows rigid honeycomb structure representing its unique properties as catalyst as pointed in the region A and B. These structures provide ample active sites for catalytic reactions and contribute to high surface area, porosity and stability of diatomite as catalyst [34, 35]. After the transesterification process, region A and B in figure 11(b) show appearance of small particles and fragments of broken diatom frustules as the result of catalytic applications of diatomite. Despite undergoing the reaction, the SEM images confirm that the diatomite still has few honeycombs structure indicating its potential for reuse as a catalyst in subsequent processes.

4. Conclusions

In conclusion, single use of diatomite as catalyst in transesterification of waste cooking oil proved to attain high biodiesel yields. SEM analysis confirmed its unique macroporous structure and high porosity, which contributed to simplifying the biodiesel purification process. Optimization of the methanol-to-oil ratio and catalyst dosage resulted in biodiesel conversion rates of 89.6% for waste cooking oil under optimal conditions and similar efficiency for fresh cooking oil, with shorter reaction times and lower catalyst usage. Additionally, diatomite demonstrated reusability, maintaining biodiesel yields of up to 75% after three cycles. Diatomite's low cost, renewability, and promising catalytic performance position it as a viable option for the biodiesel industry. Future research could focus on enhancing its stability and reusability to further reduce production costs and improve overall efficiency.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Pramila Tamunaidu, Yogeswary Vadivelu; **data collection:** Azlan Nur Rasyid Amin; **analysis and interpretation of results:** Nurhamieza Md Huzir, Yogeswary Vadivelu; **draft manuscript preparation:** Nurhamieza Md Huzir, Pramila Tamunaidu. All authors reviewed the results and approved the final version of the manuscript.

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