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Biodiesel Synthesis from Coconut Oil Using Calcined Scallop Shell Waste as the Heterogeneous Catalysts

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Abstract. Several efforts have been performed to make the biodiesel price more competitive with fossil fuels, such as using low price raw materials, including coconut oil and the use of heterogenous catalyst. In this research, the Calcined Scallop Shell was applied as a heterogeneous catalyst for synthesizing biodiesel from coconut oil. The catalyst was obtained from calcination of scallop shell waste. The catalytic activities of Calcined Scallop Shell catalyst during transesterification reaction was influenced by several reaction conditions including methanol to oil mole ratio, reaction temperature, and concentration of catalyst. The biodiesel yield of 91.7% was obtained at a methanol to coconut oil mass ratio of 12:1, 60 °C reaction temperature, and catalyst concentration of 10% wt. of oil.

Introduction

Efforts to find substitute renewable energy sources are continuing to repace fuels derived from fossil fuels. The limited reserves of fossil energy in the world and even Indonesia, triggering the world's attention on renewable energy needs is increasing. One of the renewable energy sources currently being studied is biodiesel. Biodiesel as a monoalkyl ester is an alternative as a substitute fuel from diesel because it has the same physical properties. Generally, monoalkyl esters from plant oils and animal fats are used to synthesize biodiesel. Vegetable oils and animal fats, as well as their derivatives, have the potential to replace diesel fuel.

Biodiesel is generally obtained from the synthesis process that mainly encountered in renewable resources which are transesterification of triglycerides or esterification of free fatty acids. To speed up the process and produce a high transformation of methyl esters, a base catalyst or an acid catalyst is added to the solution. Two types of conventional catalysts applied in biodiesel synthesis include homogeneous acids catalysts (including hydrochloric and sulfuric acids) and homogeneous alkaline catalysts (including potassium hydroxide, sodium hydroxide, and alkali metal hydroxides). The subsequent catalysts have a number of disadvantages, including being consumable, caustic, as well as being required to advanced dissociation for product separation, which causes major environmental issues. Hence, studies on heterogeneous catalysts are more interesting for researcher since they are more beneficial than homogeneous catalysts such as low energy consumption, reusable, ease of product separation, and less separation process.

NaOH or KOH are widely used as homogeneous catalyst in the manufacture of biofuels from edible oils. However, homogeneous base catalyst have significant drawbacks, such as an advanced separation process following the reaction phase, which generates additional effluent and cannot be reused. Several investigations on the efficiency of heterogeneous catalysts in biodiesel synthesis have been investigated for many years. As a result, numerous solid catalysts such as hydrotalcite [1], metal oxides [7, 8], zeolites [9, 10], alumina [11, 12], clays [13, 14], and biomass-based catalysts [15-19] have been investigated utilizing various raw materials. The goal of the research was to develop a heterogeneous catalyst with high catalytic activities for the biodiesel synthesis. The transesterifications of vegetable oils to produce biodiesel which involved solid catalysts had investigated by many researchers. The various type of solid catalysts such as zeolite, clay, hydrotalcite, ion exchange resin, metal oxides, biomass wastes, zirconia, heteropolyacids, activated carbon, silica, tantalum pentoxide, etc. had been studied.

Recent years, the use of catalysts from biomass waste has intensively studied for the biodiesel production. An alternative material that has not been fully explored is the solid waste of shellfish. The solid waste of shellfish is one of the potential candidates to be used as a base catalyst because it has a high calcium oxide (CaO) content. From economic viability, the use of shellfish shells has many advantages, such as widely available, the price is low, the high content of $CaCO_3$, and stable under acidic and basic conditions. Several researchers revealed that solid-based catalysts showed comparable performance to some homogeneous catalysts when applied to biodiesel production. In this paper, scallop shell waste (*Placuna placenta*) is utilized as a raw material that contains CaO for producing biodiesel from coconut oil. Indonesia with a large sea area has an abundant amount of shellfish production. Processing this seafood will produce a huge amount of shellfish waste. So far, scallop shells have only been used to make handicrafts. The content of CaO in scallop shells can be used in the biodiesel synthesis as a base catalyst. As a catalyst, the utilization of scallop shell waste material is effective to minimize the cost of biodiesel products and providing an alternative for recycling natural mineral resources. The use of scallop shell as a catalyst has many advantages i.e., stable under acidic and basic conditions, available abundantly, cheap, and environmentally friendly.

From the literature search results, there are not many reports on the use of scallop shell waste as a new source of solid catalysts for the biodiesel synthesis. In this study the transesterifications of coconut oil using calcined scallop shell as solid catalyst was performed. The catalyst activity was assessed focusing on methanol to oil mole ratio, reaction temperature, and concentration of catalyst. The reusability of catalyst was also studied in this work.

Methodology

Materials. Coconut oil was obtained from a local market in Sleman Province, Yogyakarta, Indonesia, the scallop shell waste was collected from a seafood restaurant in Bantul Province, Yogyakarta, Indonesia. Methanol p.a. was purchased from Merck.

Catalyst Preparation. To ensure consistent size, the scallop shell was screened into 200 mesh. The shells were then calcined for 4 hours at 900°C in a tube furnace.

Catalyst Performance. The transesterification reaction of coconut oil was conducted in a batch reactor comprising a condenser, thermometer, magnetic stirrer, and a boiling flask in this study. The reactions processes were carried out in the following order: (i) coconut oil was combined with methanol before adding scallop shell that has been calcined; (ii) when the temperature was arise, the solution was mixed vigorously; (iii), the reaction product was separated from the catalyst by centrifugation after the process was finished; (iv) perform distillation to remove remaining methanol; and (v) collect biodiesel product using a separatory funnel. The the catalyst weight was varied from 1 to 5 wt.%. while the mass ratio of oil to methanol was varied from 6 to 2.

Results and Discussion

Temperature Effect on Biodiesel Yield. The activity test of calcined scallop shell catalyst was examined in the transesterification reactions of coconut oil with methanol. The influence of reaction temperature on biodiesel yield is depicted in Figure 1. When utilizing the calcined scallop shell catalyst, the biodiesel yield was 70.4 % and increased to 86.3% using reaction temperature of 35 - 55°C in 2 hours and a 12:1 methanol to oil mole ratio, respectively. The highest biodiesel yield of 91.7% was obtained at a 65 °C of reaction temperature.

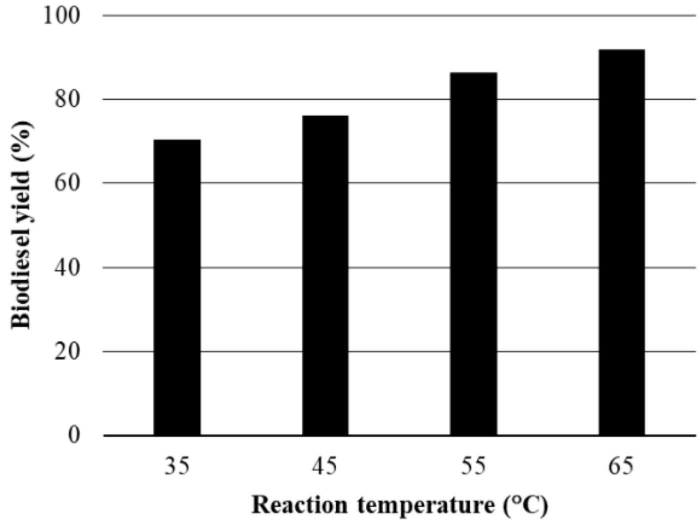

Fig. 1 Temperature Effect on Biodiesel Yield.

Methanol to Oil Mole Ratio Effect. The methanol to oil mole ratio is one of the most important elements that determines biodiesel yield. The mole ratio is the proportion of methanol mol numbers to methanol oil mol numbers. The mole ratio number was used extensively to direct the process to the biodiesel product synthesis. The catalytic actions of the calcined scallop shell catalyst utilized in the transesterification process, as shown in Figure 2, show biodiesel yields of 70.1%, 83.5%, 87.6%, and 91.7%, respectively, at a reaction time of 2 hours with methanol to oil mole ratio 3:1, 6:1, 9:1, and 12:1 at 60°C reaction temperature.

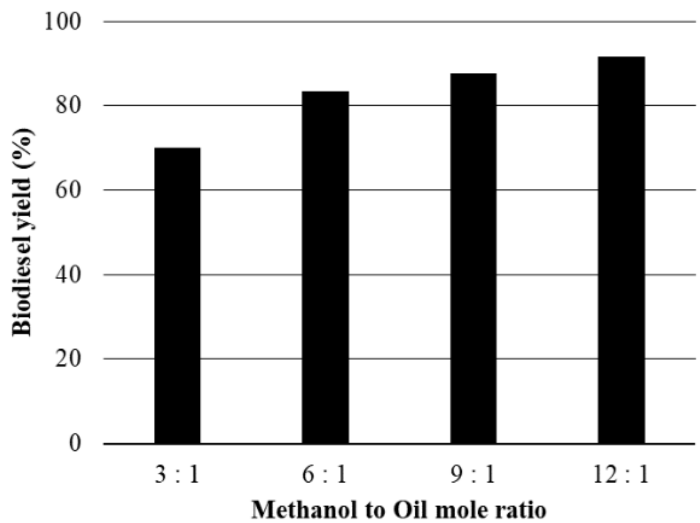

Fig. 2 Methanol to Oil Mole Ratio Effect on Biodiesel Yield.

Catalyst concentration effect. During the transesterification process, the catalyst concentration has an impact on the biodiesel synthesis. The amount of calcined scallop shell catalyst is changed between 1 and 10% by weight of oil. As seen in Figure 3, the increase in catalyst concentration increases the biodiesel yield. At 10% wt. of oil, the biodiesel yield achieves maximum. When the calcined scallop shell catalyst was used in the transesterification reaction, comparable results were achieved. When the amount of calcined scallop shell catalyst is loaded from 1 to 10% wt. of oil, the yield of biodiesel increase.

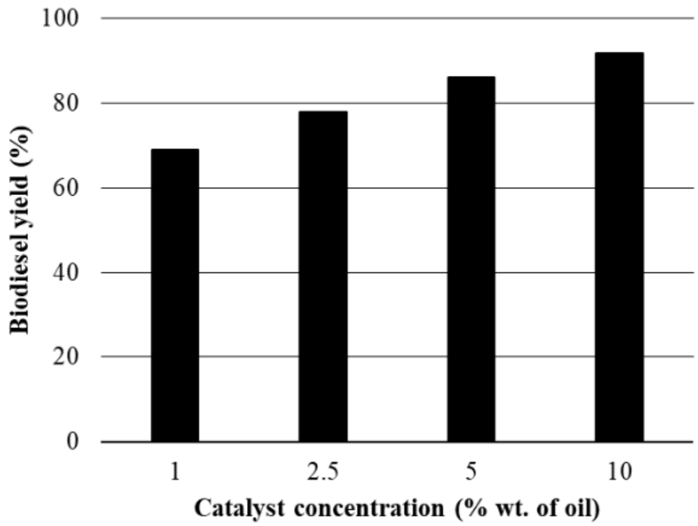

Fig. 3 Catalyst Concentration Effect on Biodiesel Yield.

Reusability. The Calcined Scallop Shell catalyst was employed for the transesterification process several times to assess its recyclability. The catalyst was then filtered before being utilized in a fresh reaction cycle with the required processing stages, including washing and drying. The findings for the catalyst utilized in three cycles are shown in Figure 4. The transesterification reaction was conducted under the following conditions: catalyst concentration of 10% wt. of oil, 60 °C reaction temperatures for 2 hours period, and methanol to oil mole ratio of 12:1. The activity was smaller in the 2nd and 3rd cycles compared to the 1st cycle, as seen in Figure 4. After the 3rd cycle, the catalytic activity dropped by up to 40%.

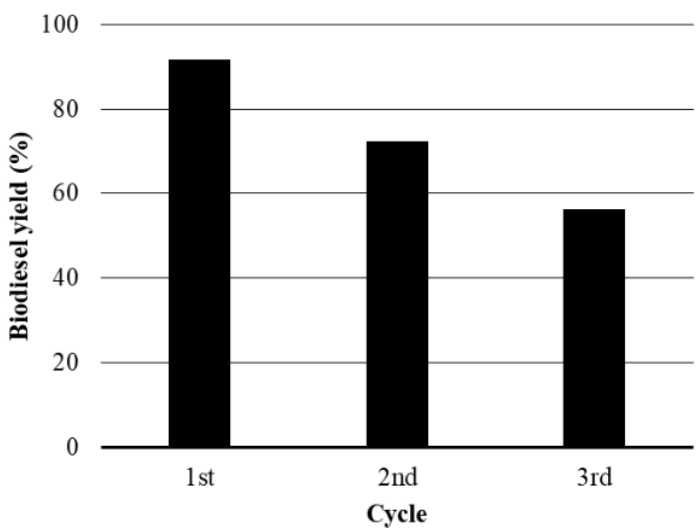

Fig. 4 The Reusability of Catalyst.

Biodiesel characterization. Figure 5 displays the FTIR spectrum for the biodiesel from coconut oil. The FTIR spectra was examined in the 600 cm^{-1} to 3500 cm^{-1} region. FTIR is an instrument that can supply information of compound that has functional group. As demonstrated in Figure 5, the existence of biodiesel is represented by CH_3 asymmetric bending about 1425–1500 cm^{-1} and O-CH_3 extending around 1100–1200 cm^{-1}. The C=O bending of the ester group in biodiesel is represented by a wavenumber approximately 1700 to 1800 cm^{-1}. The occurrence of an ester group about 2800–3000 cm^{-1} can be seen by looking at the CH peak. The =C–H stretching, C–H bending, and the C–H bond stretching are indicated by wave numbers of 675–1000 cm^{-1}, 1350–1480 cm^{-1}, 2800–3000 cm^{-1}. Likewise, the ester group is represented by the C=C and C-O bond extending from 1000–1300 cm^{-1} to 1651 cm^{-1}. The existence of an alcohol group and an aromatic group is shown by the peaks at 3000–3100 cm^{-1} and 3470 cm^{-1}.

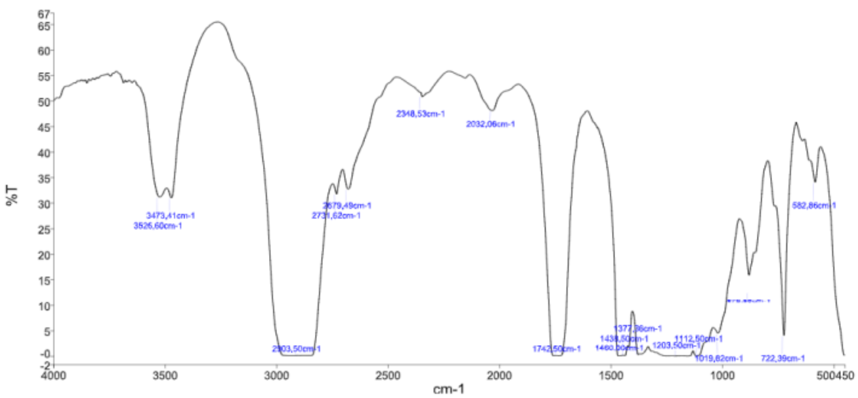

Fig. 5 FTIR spectra of biodiesel.

Conclusions

In this study, biodiesel synthesis from coconut oil was investigated using a Calcined Scallop Shell Waste as a solid catalyst. The biodiesel yield of 91.7% was obtained at a 65 °C reaction temperature, methanol to coconut oil mass ratio of 12:1, and catalyst concentration of 10% wt. of oil.

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