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CaO/Natural Dolomite as a Heterogeneous Catalyst for Biodiesel Production

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Abstract. In the present study, the CaO/Natural Dolomite as a heterogeneous catalyst was applied to synthesize biodiesel from coconut oil. The physico-characteristics of CaO/Natural Dolomite catalyst were determined using X-ray diffraction (XRD), X-Ray Fluorescence, and porosity analysis (specific surface area, average pore size diameter and total pore volume). The performance of CaO/Natural Dolomite catalyst was examined in a batch reactor for transesterification reaction of coconut oil with methanol. From the experiments, the optimum process conditions were achieved at a 60°C of reaction temperature, a 5 wt.% of catalyst amount, and 6 : 1 of methanol to coconut oil mass ratio. The CaO/Natural Dolomite catalyst exhibits high catalytic activity and reliable to be applied in biodiesel synthesis as a heterogeneous base catalyst.

Introduction

In Indonesia, the need for energy has increased annually due to the growth in population and industrialization. Despite the exploration of the reservoir be intensified continuously, the diminishing of fossil fuels is occurred due to inequality between the need and supply of energy. In the past few decades, biodiesel has received much attention from many researchers as a renewable energy source to replace fossil fuels. Biodiesel has several benefits i.e. degraded, non-toxic, low hazardous exhaust emissions, and does not contain sulfur [1]. Biodiesel is typically synthesized by chemically reacting fatty acids or triglycerides with a short chain of alcohol to yield fatty acid methyl esters [2]. Various types of vegetable oil and animal fats can be employed as raw materials for biodiesel production [3]. The conventional biodiesel synthesis is conducted with the presence of homogeneous catalysts (acid or alkali). However, the use of homogeneous catalysts lead to many limitations, such as a difficult separation process, cannot be reusable, more corrosive and produced a large amount of wastewater [4]. The searching of heterogeneous catalysts has received much concern related to the problems posed by homogeneous catalysts. In previous literature, many researchers have reported the application of heterogeneous catalyst for biodiesel production .

Several types of heterogeneous catalysts based on natural resources have been developed and tested for biodiesel synthesis. This type of catalyst has several advantages, i.e. easy to obtain, available abundantly, have high porosity, does not require a complicated synthesis step, and inexpensive [5]. The natural mineral catalysts are very interesting to study due to the ability to be applied for biodiesel synthesis using the transesterification reaction pathway. The heterogeneous acid catalysts are considerably applied in biodiesel synthesis. Different heterogeneous catalysts such as, clays [6], zeolite [7], amberlyst [8], metal oxide [9-13], ion exchange resin [14], bio-char [15, 16], heteropolyacids [17] and polymer-based [18] have been examined to catalyze the various feedstocks for biodiesel synthesis. The main issue related to the biodiesel production is the competitiveness of its price compared to petroleum-based fuel price. Thus, the production cost should be reduced to gain the biodiesel price more reliable. One way to lower the cost is by utilizing low-cost feedstock. Various materials have been exploited as cheap feedstocks for

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biodiesel production such as animal fats (mutton [19], tallow [20], and lard [21]), non-edible oils [22-24], yellow grease [25], etc. The use of non-edible oil for biodiesel raw materials has limitations in connection with the oil production efficiency in every cultivation area. The raw material which has a good prospect as a feedstock in biodiesel synthesis is coconut oil.

The biodiesel synthesis from coconut oil over CaO/Natural Dolomite was investigated in this study. The CaO/Natural Dolomite were characterized using several technique including porosity, basicity, crystanility, and elemental analysis. Different operating conditions were varied to study their effect on catalytic activity performance. Based on the previous literature study, the reported research that related to the utilization of CaO/Natural Dolomite in biodiesel synthesis is still limited.

Experimental

Materials. Commercial coconut oil was purchased from local market in Sleman, Yogyakarta, Indonesia. The natural dolomite was gained from by the grocery farming supplier. Ca(NO3)2.8H2O was obtained Sigma Aldrich and methanol p.a. was acquired from Merck..

Preparation of CaO/Natural Dolomite Catalyst. The natural dolomite was screened to get uniform size into 200 mesh. Subsequently, the natural dolomite was calcined at 900°C in tubular furnace for 4 hours. The dolomite catalysts were synthesized by conventional impregnation process using metal salts of $Ca(NO_3)_2.8H_2O$. The 5% wt. of Calcium was doped into the calcined dolomite at 60°C for 8 hours while agitated vigorously. Then, the mixtures were dried in the oven at at 80°C for overnight to obtain the slurry. Finally, the slurry was calcined at 400°C for 4 hours to form CaO/Natural Dolomite.

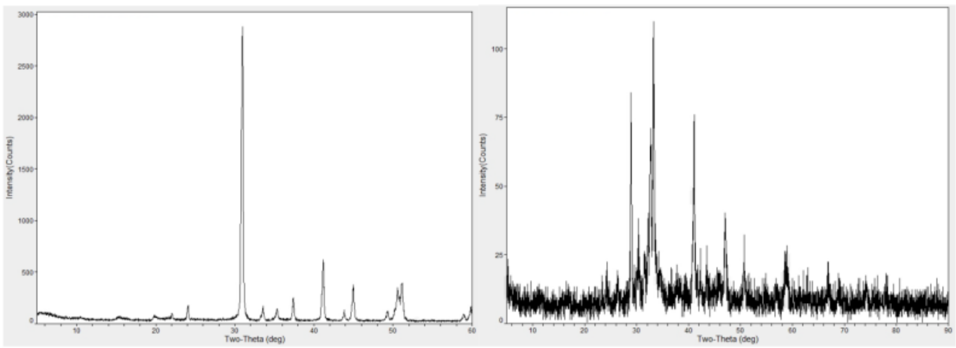
Characterization of Catalyst. The measurement of porosity was carried out using the N2 adsorption-desorption isotherm analysis. The XRD analysis was performed to identify the crystanility of CaO-dolomite catalyst. The element composition was determined by X-Ray Fluorescence.

Catalyst Performance. In this work, the coconut oil transesterification reaction was conducted on the batch reactor using a boiling flask placed on the top of a magnetic stirrer which completed with thermometer and condenser. The reaction procedures were applied using steps as follow: (i) methanol was mixed with coconut oil and added with CaO/Natural Dolomite catalyst; (ii) then the mixture was stirred vigorously while temperature was elevated; (iii) After the reaction was finished, the catalyst was separated from reaction product by centrifugation process; (iv) the remaining methanol was removed by distillation process; and (v) the biodiesel product was collected by using a separating funnel.The mass ratio of methanol /oil was studied from 2 to 6, while the weight of catalyst was varied from 1 up to 5 wt.%..

Results and Discussion

Characterization of Catalyst. Fig. 1 shows the XRD analysis patterns of the Natural Dolomite and CaO/Natural Dolomite catalyst. The crystalline structures of the natural dolomite and CaO/Natural Dolomite catalyst are indicated intensively in several peaks. The peaks at $2\theta = 32°$, 38°, 43°, 54°, 65°, 68° and 75° reveals the presence of CaO dan MgO. Furthermore, the existence of $MgCa(CO_3)_2$, $CaCO_3$, and $Ca(OH)_2$ are detected at $2\theta = 41°$, 51°, 19°, 29°, 35° and 47°. A high crystallinity of CaO–MgO structure was exhibited with the high intensities of the diffraction patterns both on the Natural Dolomite and CaO/Natural Dolomite catalyst. The determination of mineralogical composition of the Natural Dolomite and CaO/Natural Dolomite catalyst was carried ou using an X-Ray Fluorescence (XRF) spectrometer. Based on the XRF analysis, the Natural Dolomite was mainly composed 48.65 wt% of CaO, 13.41 wt% of MgO, and other substances. Meanwhile, on the CaO/Natural Dolomite catalyst contained 56.65 wt% of CaO, 10.24 wt% of MgO, and other compounds. It clearly showed that, the increasing of CaO content was indicated after the impregnation process. The surface area of the Natural Dolomite and CaO/Natural

Dolomite catalyst were tabulated in Table 1. The Natural Dolomite has a mesoporous structure with surface area of 19.32 m^2/g and average pore diameter of 15.6 nm, respectively. The pore volume shows at value of 0.0212 $cm^3.g^{-1}$. When more CaO was doped onto the Natural Dolomite, the surface area decreased to 12.73 m^2 and the average pore diameter enhanced to 16.54 nm. Meanwhile, a 0.0304 $cm^3.g^{-1}$ of pore volume was achieved.



(a) (b)

Fig. 1. The XRD Analysis Patterns of: (a) Natural Dolomite and; (b) CaO/Natural Dolomite Catalyst

Catalyst Performance

Effect of Mass Ratio. The transesterification reaction is limited by the equilibrium. To favour the forward reaction, the amount of methanol was performed excessively. The mass ratio of methanol to cooconut oil was varied in the range of 3:1 to 6:1 to investigate the effect of mass ratio methanol to coconut oil on biodiesel yield. The effect of mass ratio methanol to oil on the biodiesel yield was exhibited in Fig. 2. As shown in Fig. 2, when the mass ratio was increased from 2:1 to 6:1, the biodiesel yield was found to increase from 68% to 82%. The highest conversion was found at mass ratio of 6:1.

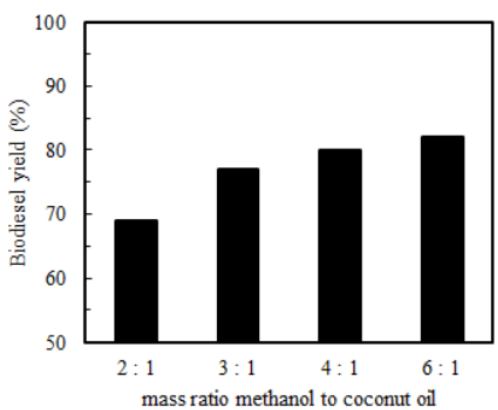


Fig. 2. Effect of Mass Ratio Methanol to Coconut Oil on Biodiesel Yield

Effect of Catalyst Amount. The study of catalyst effect to biodiesel yield is conducted by using the different catalysts amount i.e. 0.5; 1; 2.5; and 10 wt.% of coconut oil. Fig. 3 depicted the effect of catalyst amount on biodiesel yield. The biodiesel yield increased when the catalyst amount increased from 0.5 to 5 wt.% of coconut oil. The increasing of biodiesel yield might be due to the enhancement of the available total active sites number on catalyst surface.

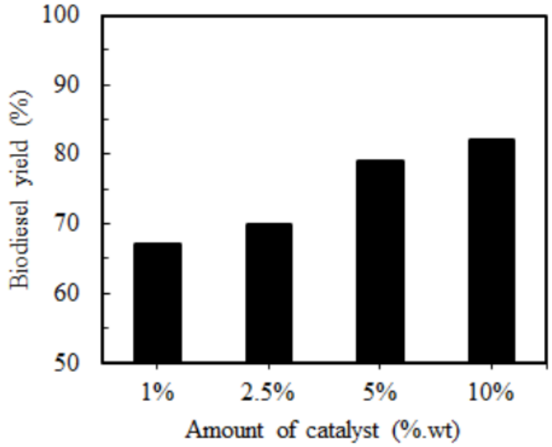


Fig. 3. Effect of Amount of Catalysts on Biodiesel Yield

Effect of Reaction Temperature. Different reaction temperatures (40, 50 and 60°C) were employed to study their effect on the biodiesel yield. Fig. 4 exhibits the influence of temperature on the transesterfication of coconut oil using CaO/Natural Dolomite catalyst. As depicted in Fig. 4, elevating of the temperature had favorably affected the biodiesel yield. An increase in biodiesel yield from 70 to 77% was obtained at an increasing temperature of 40 to 60°C. The maximum biodiesel yield is achieved at 60°C. An increase of biodiesel yield was due to the the increasing reaction rate at high temperature.

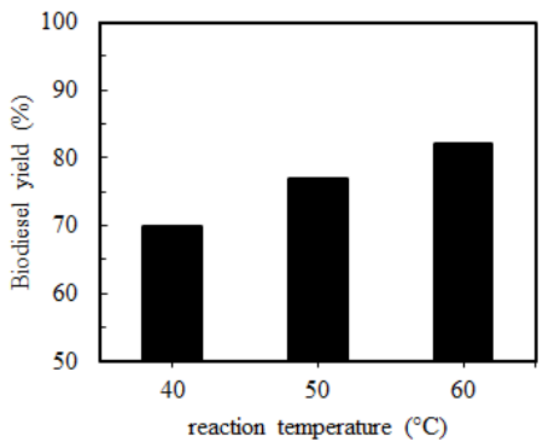


Fig. 4. Effect of Reaction Temperature on Biodiesel Yield

Conclusions

The CaO/Natural Dolomite catalyst exhibits high catalytic activity and reliable to be applied in biodiesel production as a heterogeneous base catalyst. The effects of the mass ratio of catalyst to oil (2:1–6:1), amount of catalysts (1–10 wt.%), and the reaction temperature (40–60°C) were studied to optimize the reaction conditions. From the experiments, the optimum process conditions were achieved at a 60°C of reaction temperature, a 5 wt.% of catalyst amount, and 6 : 1 of methanol to coconut oil mass ratio.

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