Synthesis of Biodiesel from Kapok Seed Oil using CaO-Dolomite Catalyst and Its Comparison with Homogeneous Catalyst

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Synthesis of Biodiesel from Kapok Seed Oil using CaO/Dolomite Catalyst and Its Comparison with Homogeneous Catalyst

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Abstract. Biodiesel as renewable energy resources has been attracted more intense study because of the alternative effort to reduce dependency of fossil fuel. Commonly biodiesel production is produced using edible oils as feedstock. However, the problems related to the competition between foods versus energy supply have been concerned in the past few decades. The dependency on edible oils as biodiesel feedstock could be reducing by using non edible oils. Furthermore, using non edible oil as feedstock will make the biodiesel prices appropriate to compete with fossil fuel prices. In this research, synthesis of biodiesel from kapok seed oil using CaO/Dolomite catalyst as heterogeneous catalyst and its comparison with homogeneous catalyst was studied. The properties of CaO/Dolomite catalysts were determined in term of porosity analysis (adsorption and desorption of N_2), crystaline structure (X-Ray Diffraction), and analysis of element compositons (X-Ray Fluorescence). The biodiesel yield as high as was 92.9% achieved at following reaction conditions: 60°C of reaction temperature, 3:1 methanol to KSO mass ratio, and catalyst amount 10 wt. % of KSO.

Introduction

Today, total energy needs continue to rise in many areas due to dramatic population growth and intensified industrialization. Meanwhile, the supply of fossil fuel as a primary energy source cannot equal the rate of energy requirements. On the other hand, the discovery of new oil sources has continued to diminish in recent years. The use of alternative energy sources is urgently needed in order to overcome these problems. Biodiesel or monoalkyl ester is one of the potential renewable energy sources that can be obtained from fatty esterification of or transesterification of triglycerides. Many types of edible oil including rapeseed [1], corn [2], sunflower [3], soybean [4], palm oil [5], coconut [6], and rice bran oil [7] have been investigated as feedstocks for the biodiesel production. The concerning attraction to the replacement of the first generation biodiesel that applied edible oils has increased attention to the development of the raw materials from non-edible oils. Furthermore, the crucial variable that affects the economic viability of biodiesel is the raw materials price. The non-edible oil has an inexpensive price so that it can be minimize the biodiesel production cost and make the biodiesel more competitive compared to petroleum-based diesel. Ceibra petandra or kapok tree is a tropical tree which bears a lot of seed in the fruit. The seed contains oil in the range of 20-25 % of weight. Indonesia has an estimated area of 100,500 hectares of kapok plantation in 2015. Each hectare of kapok plantation can produce an approximate amount of 110 kg of seeds. Thus, kapok seeds have the potential to yield non-edible for more than 10 million liters annually. The Kapok Seed Oil (KSO) was obtained by extracted using conventional leaching methods with organic solvent or pressing. By considering the facts as mentioned, the development of KSO as a low-cost raw material for biodiesel production is attracting to investigate.

The production of biodiesel from edible oils commonly employs NaOH or KOH as homogeneous base catalysts. Nevertheless, the homogeneous base catalysts have some shortage, including an

advance separation process after reaction step, generate more wastewater, and cannot be reuse. The problems related to homogeneous catalysts can be solved with the heterogeneous catalysts application. In recent years, several studies have been conducting to investigate the performance of heterogeneous catalyst on biodiesel synthesis. As a result, various types of solid catalysts such as zeolites [7, 8], metals oxides [9, 10], clays [11, 12], alumina [13, 14], hydrotalcite [1], biomass-based catalyst [15-19] have been examined using different types of raw materials. The studies aimed to get a heterogeneous catalyst which has a good catalytic activity to biodiesel synthesis. Recently, the development of catalysts that synthesized from agricultural biomass waste has attracted attention due to the simple preparation methods, inexpensive cost, abundant, and can be anchored with active sites for the specific reaction.

In this research, synthesis of biodiesel from kapok seed oil using CaO/Dolomite catalyst as heterogeneous catalyst was studied for biodiesel synthesis using kapok seed oil as raw material. A series of the reaction parameters including the concentration of catalyst, temperature, and methanol to KSO mole ratios have been tested to examine the effect on biodiesel yield. The performance of CaO/Dolomite catalysts also had compared with the homogeneous NaOH catalyst.

Experimental

Synthesis of CaO/Dolomite catalyst

Firstly, Dolomite that collected from Gunung Kidul district, Yogyakarta province was calcined in a furnace at 800°C for 2 hours. Dolomite was then activated by mixing with H_2SO_4 0.5 M. Then, the Dolomite was filtrated and neutralized by dripping distilled water from the mixture. The neutralization process was completed when the wash water was already freed from sulfate ion. The catalyst was synthesized by impregnating CaO of $Ca(NO_3)_2.4H_2O$ (calcium nitrate tetrahydrate) on Dolomite. Following that, the solid was calcined at 500°C for 4 h and designated as CaO/Dolomite catalyst. The physico-characteristics of CaO/Dolomite catalysts were determined in term of porosity analysis (adsorption and desorption of N_2), crystaline structure (X-Ray Diffraction), and analysis of element compositons (X-Ray Fluorescence).

Transesterification using homogeneous NaOH catalyst

NaOH pellet was mixed with methanol into the concentrations of 1.0% wt. in a 500 ml boiling flask connected to a reflux condenser. A uniform methoxide solution was obtained by stirring the mixture vigorously. Then, KSO was poured into mixture corresponding to a 6:1 mole ratio of methanol to KSO. The temperature was raised to reach the desired value. The CaO/Dolomite catalyst was separated by filtration after the reaction was completed. In a separate funnel, the liquid phase was decanted until two layers were formed.. The biodiesel product was in top layer and the bottom layer was glycerol. Then, the remaining methanol was evaporated from biodiesel product.

Transesterification using CaO/Dolomite catalyst

The procedure of transesterification reaction using CaO/Dolomite catalyst was typically as follows: KSO, methanol and catalyst (5% wt. of KSO) were mixed in a 500 ml boiling flask connected to a reflux condenser. The temperature was elevated while stirred vigorously. After 2 hours, the reaction was stopped and the mixture was filtered to allow catalyst separation. The liquid phase was poured into the separating funnel and formed two phases: the biodiesel was in the upper layer and the formed glycerol was in the bottom layer.

Results and Discussion

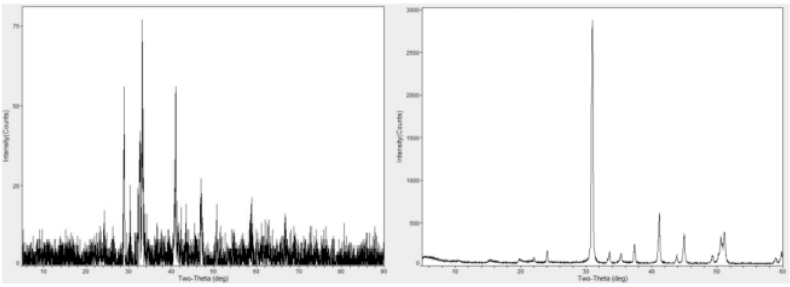
The physico-characteristics of CaO/Dolomite catalysts

Table 1 shows the physico-characteristics of Dolomite and CaO/Dolomite catalyst. The of measurement of N_2 adsorption and desorption resulted the BET surface area of 32.27 m_2/g for Dolomite and 15.64 m_2/g for CaO/Dolomite catalyst, respectively. A decrease in the BET surface area indicated the successful incorporation of CaO. Meanwhile, the pore diameter of the CaO/Dolomite catalyst shows an improvement in the diameter of the pore.

Table 1. The dolomite and dolomite catalyst characteristics

Samples	Specific surface area ($m^2.g^{-1}$)	average pore size diameter (nm)	total pore volume ($cm^3.g^{-1}$)
Dolomite	32.27	2.45	0.1367
Dolomite Catalyst	15.64	3.21	0.1234

XRD analysis was performed to analyze the crystaline structure of activated Dolomite and CaO/Dolomite catalysts. As illustrated in Figure 1, the activated Dolomite and CaO/Dolomite catalysts showed strong peak at 2θ= 32.2°, 34.2°, 37.4°, 53.8°, 64.2° and 67.4° indicating the existaence of CaO. To determine the chemical composition of activated Dolomite and CaO/Dolomite catalysts, the X-Ray Fluorescence (XRF) spectrometry was applied. The result showed that a 8.32% wt. of CaO/Dolomite catalyst was calcium oxide (CaO). It can be concluded that after the impregnation with $Ca(NO_3)_2.4H_2O$, the good incorporating of Calcium metal into Dolomite structure.



(a) (b)

Figure 1. XRD pattern of: (a) Dolomite; and (b) CaO/Dolomite catalyst.

Performance Test

Temperature effect

The performance test of homogeneous NaOH and CaO/Dolomite solid catalyst were studied in the transesterification reactions of KSO with methanol. Batch reactions were conducted at three different reaction temperatures under identical conditions. Figure 1 shows the results of temperature effect on the yield of biodiesel. It can be seen on Figure 1, the biodiesel yield increased from 44% at 40°C to 53% at 50°C and achieved 66% at 60°C in 2 h of reaction time when NaOH was used as the catalyst. When using the CaO/Dolomite catalyst, the similar trend was shown to that obtained with NaOH. At 40°C the biodiesel yield was 55% for whereas the yield increased to 66% and thereafter to 77% with molar ratio of 50 and 60°C in 2 h of reaction time and 6:1 of methanol to oil mole ratio, respectively. Meanwhile, as illustrated in Figure 1, with increasing the reaction temperature 40 to 50°C, the biodiesel yield was increased when the CaO/Dolomite catalyst was applied. At the reaction temperature of 60°C, the highest biodiesel yield of 88% was attained.

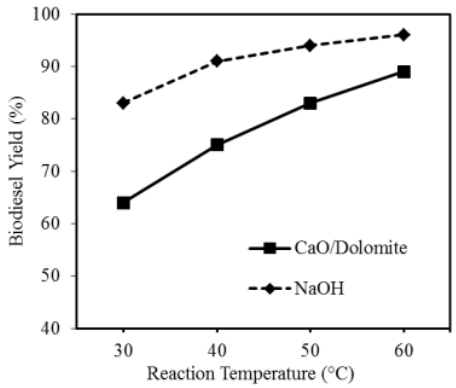


Figure 2. Temperature Effect on Biodiesel Yield.

Mole ratio of methanol to oil effect

The one of the crucial factors that influences yield of biodiesel is mole ratio of methanol to oil. Mole ratio is the ratio between the mol numbers of methanol to mol number of methanol oil. In order to divert the reaction to the biodiesel product formation, the mole ratio number was applied excessively. The catalytic activities of homogeneous NaOH catalyst used in the transesterification reactions, as exhibited in Figure 6, reveal the biodiesel yields of 44, 55, 66 and 77% which were obtained with mole ratio of methanol to oil 4:1, 6:1, 8:1 and 10:1, respectively at 60°C of reaction temperature and a reaction time of 2 h. For the CaO/Dolomite catalyst, as the mole ratio of methanol to oil increased from 4:1 to 10:1, biodiesel yield increased. The maximum biodiesel yield of 88% was obtained at a 10:1 of methanol to oil mole ratio.

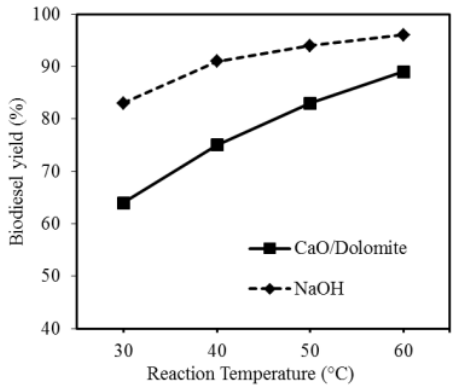


Figure 3. Mole Ratio of Methanol to Oil Effect on FFA Conversion.

Catalyst concentration effect

The catalyst concentration also effects on the yield of biodiesel during transesterification reaction. The catalyst concentration is varied in the range of 0.25–2% of sulphuric acid. As presented in Figure 4, the yield of biodiesel increases with the increasing of catalyst concentration. The biodiesel yield

attains the maximum at 2% of sulphuric acid. The simi results were obtained when using the CaO/Dolomite catalyst during transesterification reaction. The yield of biodiesel increases when the concentration of CaO/Dolomite catalyst was loading from 0.5 to 2 grams.

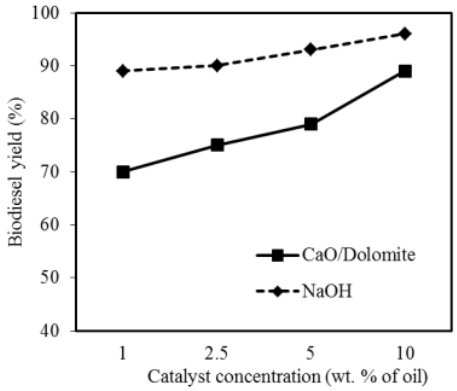


Figure 4. Catalyst Concentration Effect on FFA Conversion.

Conclusions

In this research, synthesis of biodiesel from kapok seed oil using CaO/Dolomite catalyst as heterogeneous catalyst and its comparison with homogeneous catalyst was studied. The biodiesel yield achieved as high as was 92.9% under the following conditions of reaction: 60 ° C reaction temperature, 3:1 methanol to KSO mass ratio, and 10 wt. % of KSO catalyst amount.

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