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
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
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
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## Utilization of eggshell waste as low-cost solid base catalyst for biodiesel production from used cooking oil

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**Abstract.** A solid CaO-based catalyst of waste eggshell was developed for biodiesel production from used cooking oil. The waste eggshell powder was calcined in air at 900° for 4 h to convert calcium species in the eggshells into active CaO catalysts. The characterization of CaO catalyst was done by XRD and BET analysis. The CaO catalyst was then introduced for transesterification of used cooking oil (UCO) for testing of its catalytic activity. The experiment was conducted in batch type reactor that consists of three-neck glass equipped by reflux condenser and magnetic stirrer. Before transesterification process, the UCO was treated by coconut coir powder in order to reduce the free fatty acid content. The result showed that the catalyst was potentially use for transesterification of used cooking oil into biodiesel with relatively high yield of 75.92% was achieved at reaction temperature, reaction time, molar ratio UCO to methanol and catalyst amount of 65° C, 7 h, 1:15 and 6%, respectively.

### 1. Introduction

The energy consumption has increased every year in line with the increase in human population. This increase occurred in various sectors including transportation, power generation, industry and households. Until now, the world's energy needs are supplied by fossil fuels. By 2030, it is estimated that the increase in energy consumption reaches 53%, so that the oil reserves expected to be exhausted within the next 41-63 years [1]. In addition, the use of fossil fuels continuously, causing environmental impacts such as the increase of CO<sub>2</sub> emissions that result in accelerated of global warming, air pollution and other environmental issues. The rapid depletion of fossil fuels and the increasing energy needs has encouraged each country to make savings and conducting various studies to obtain renewable fuels. Biodiesel is a renewable fuel that can be used as a substitute for diesel fuel. Biodiesel is mono alkyl ester derived from many sources such as vegetable oils, animal fats or used cooking oil. The main factors affecting the cost of biodiesel production is the raw material and the type of catalyst used. In 2012, cooking oil demand in Indonesia is estimated to reach more than 4.7 million tons. After use, the cooking oil will undergo changes in chemical composition and contain carcinogenic compounds. Therefore, the used cooking oil is no longer worthy to be used for food processing. The abundant and availability of used cooking oil can be used as an alternative raw material for the manufacture of biodiesel [2]. Typically, biodiesel was produced through the transesterification process using homogeneous acid / alkaline catalyst [3], [4], [5] that usually called conventional process. This process is fast and efficient, for example more than 95% conversion is achieved at 60° C in 1 h of reaction time. However, it has many disadvantages including the formation of side products such as soaps [6], [7], [8], the complicated separation of the product with the catalyst [3]. In addition, the alkaline waste that formed in large enough quantities also requires a complicated separation process. This separation process requires considerable energy and the increasing of costs



production [7], [9]. Those problems can be mitigated by replacing homogeneous catalysis with solid catalyst. The use of solid acid catalysts in biodiesel production has been studied primarily from transition metal oxides such as zirconia and titanium oxide [9], [10]. The main obstacle of use of acid catalysts requires long reaction times and high reaction temperatures [9], [11]. Solid base catalyst such as calcium oxide [12], magnesium oxide [13], alkali alumina [14], hydrotalcite [15], zeolite [16] seems to be a promising strategy to substitute acid catalyst, but most of them use of synthetic chemicals that are relatively expensive.

This study developed a solid CaO-based catalyst of eggs shell for the production biodiesel from used cooking oil. The purpose of this study is to utilize CaO contained in the eggshell into a solid catalyst instead of homogeneous catalysts that will influence decreasing of biodiesel production costs, so the price of biodiesel can compete with diesel oil. Eggshell has a small porosity and high of CaCO<sub>3</sub> content that is indispensable in the industry of biodiesel [17]. Components of the eggshell include calcium carbonate (94%), magnesium carbonate (1%), calcium phosphate (1%), and other organic materials (4%). Based on the pore structure and content of CaCO<sub>3</sub>, eggshell provides opportunities for development as heterogeneous catalysts [18].

Utilization of this catalyst is expected to provide a solution to obtain a feasibility study of biodiesel production process that is effective and efficient and reduce environmental pollution. The study was conducted in the scale laboratory includes several stages, namely preparation of catalysts, catalyst characterization, and testing catalyst activity to determine the best operating conditions include amount of catalyst and reaction time.

## 2. Materials and Methods

### 2.1. Materials

Waste cooking oil was found from local street food vendors in Surabaya, East Java. The physical and chemical characteristics of UCO were analyzed using several methods based on the procedure described elsewhere [2]. The free fatty acid (FFA) content of 4.4946% was analyzed based on American Oil Chemist (AOCS) official Method (Ca 5a-71, 1993). Meanwhile, its density and water content of 0.9219 g/cm<sup>3</sup> and 0.78%, respectively were analyzed based on ISO 662 – 1089. FAME standards and internal standards of chromatographic grade were obtained from Sigma–Aldrich (Switzerland). Technical grade of methanol was purchased from local supplier Bratacho-Chem. Meanwhile eggshell was collected from the local bakeries in Surabaya, East Java, Indonesia.

### 2.2. Catalyst preparation and characterization

At first, the eggshells being crushed into small chips, then soaked and rinsed thoroughly several times with hot water to remove the impurities and other interference materials. The clean eggshell chips dried at 110°C for 24 h in the dry oven. The dried chips eggshells then powdered and sieved to the size of qualifying on 100 mesh and retained on 200 mesh. Calcination process was performed in the muffle furnace at 900°C for 4 h under static air. Surface area analysis of the catalyst was examined using Brunauer-Emmett-Teller (BET) methods. Sample was degassed at 105 °C prior to analysis and the adsorption of N<sub>2</sub> was measured at -196 °C. Meanwhile, X-ray diffraction (XRD) was measured by XPert MPD using Cu Ka radiation. The prepared catalyst from eggshell was assigned as ES-CaO, and then kept in a desiccator in the presence of silica gel in order to avoid water and CO<sub>2</sub> contacting with the catalyst.

### 2.3. Transesterification process

In this research, the collected UCO was pre-treated based on the procedure described elsewhere [2], [8]. First, the collected UCO was filtered to remove the food residues or the suspended particulate matters, and then it was heated at 100-105°C for 30 minutes to remove the excess of water content. Further, UCO was treated by adsorption process using coconut coir powdered (CCP) (wt.% to the UCO) to reduce the FFA content of the UCO. It has been stated in the last work [2] that the optimum amount of CCP is 7% (wt.% to the UCO), therefore it was used for treating UCO in this work. The treated UCO was analyzed to determine its final FFA content, and then it was ready subjected for transesterification process using ES-CaO catalyst.

Transesterification process was conducted in batch type reactor consists of three neck glass, equipped with reflux condenser and magnetic hot plate stirrer. The reaction was carried out at 65°C and molar ratio of UCO to methanol 1:15. Meanwhile, the amount of catalyst and reaction time were varied of 3-7% (wt.% o oil) with 1% of range and 2-7 h with range of 1 h, respectively. The amount of ES-CaO catalyst, methanol, and treated UCO were measured and introduced into the reactor, and allowed to keep a while until the reaction temperature of 65°C was reached. The reaction mixtures were stirred thoroughly at constant speed using magnetic stirrer. After desired duration time was reached, the reactor was cold down and the reaction mixtures were withdrawn at room temperature. The mixtures were filtered to separate the catalyst from the liquid phase. The liquid phase was introduced into a separation funnel where the glycerine was decanted, and then the excess methanol was recovered using a rotary evaporator. Biodiesel concentration was analysed by a gas chromatograph GC-14B (Shimadzu) equipped with a flame ionization detector and a capillary column HP-Innowax (30 m × id 0.25 mm, 0.25 μ m). Helium was used as the carrier gas throughout the whole experiment. Biodiesel analyses can be used to calculate the yield of biodiesel (%) defined as Equation 1 [19]

$$Y = \frac{w}{W} \times 100 \quad (1)$$

Where, w of ab means weight of actual biodiesel was calculated from weight of biodiesel produced (mg) in the experiment multiple by weight % of FAME in the sample. Meanwhile, W of oil is weight of oil (mg) used in the experiment.

### 3. Result and Discussion

#### 3.1. Characterization of ES-catalyst

The specific surface area prepared ES-CaO catalyst of 125.927 mg<sup>-1</sup> was obtained using BET methods. This result is much higher than that crab shells catalyst of 13 m<sup>2</sup> g<sup>-1</sup> that was stated by Boey et al. (2009) [20]. Meanwhile, mud clam shell and commercial CaO have the surface area of 68.57 and 62.91 m<sup>2</sup> g<sup>-1</sup>, respectively [21].

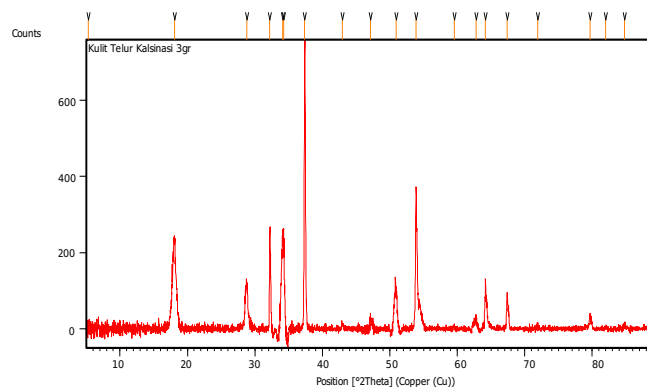


Figure 1. XRD patterns of eggshell (ES-CaO) catalyst calcined at 900°C

X-ray diffraction (X-RD) patterns of ES-CaO was depicted by figure 1. XRD results revealed that the composition of uncalcined eggshell mainly consisted of calcium–magnesium carbonate (CaCO<sub>3</sub>–MgCO<sub>3</sub>) with the absence of CaO peak (the figure was not shown). This result confirm with the composition of eggshell that mostly consisted by CaCO<sub>3</sub> (94%). However, for the calcined catalyst at 900°C (figure 1), CaCO<sub>3</sub> completely converted into CaO and release the CO<sub>2</sub> gas. The narrow and high intense peaks of the calcined catalyst showed the well crystalline structure of the catalyst that mainly consisted of the active ingredient CaO.

#### 3.2. Effect of reaction variable on yield of biodiesel

The yield of biodiesel was affected by many factors, such as the type triglyceride, type of catalyst used, reaction temperature, molar ratio of oil to methanol, catalyst amount or reaction time. Therefore, the

effect of reaction variables was studied in the presence of ES-CaO solid base catalyst. In this work we focused on the influence of the catalyst amount used and the reaction time on yield of biodiesel.

There are 2 keys parameter including free fatty acid and moisture content that are determining the viability of vegetable oils to be used in transesterification process [2], [22]. Water content will accelerate the hydrolysis reaction and simultaneously reduce the amount of ester formation [23]. To achieve 90% yield of biodiesel, then water content of triglyceride that used in the transesterification process not more than 0.5% [24]. Moreover, it was known that higher free fatty acid contents will lead to formation of soap and water. If free fatty acid content more than 3%, the homogeneous base catalyst is not suitable for the transesterification reaction [2], [24].

The UCO that was used in this work contained 4.4946% (wt. %) free fatty acid (FFA). In order to reduce the FFA content of the UCO, it was treated by adsorption process using 7% of coconut coir powder [2]. Coconut coir is one of the agricultural waste products often used as adsorbent in wastewater treatment [25]. Due to its availability and its abundant in the environment, make it to be a good source used in the adsorption process [2], [25]. The nature of coconut coir dust such as good structural stability, high water absorptivity and high porosity enhance its effectiveness as adsorptive or ion exchange capacities. Its composition includes lignin, cellulose, pentosan and ash were 36.15, 33.61, 29.27 and ash 0.61% (wt. % of dry basis), respectively [26]. After adsorption process, the FFA content of UCO was determined using AOCS method. The result showed its FFA content decrease into 0.2079%. Furthermore, the treated used cooking oil is ready for the transesterification process.

### 3.2.1. Effect of catalyst amount on yield of biodiesel

Investigating the influence of catalyst amount on yield of biodiesel the experiments were performed in varied catalyst amount of 3-7% with range of 1% (wt.% to UCO) under constant stirring speed using reaction temperature, reaction time and molar ratio of UCO to oil at 65 °C, 1 hr and 1:15, respectively. Figure 2 shows the influence of catalyst amount on yield of biodiesel. As depicted by figure 1, the amount of catalyst was significantly influence on yield of biodiesel. Yield of biodiesel increases with increasing the amount of catalyst. Without the presence of catalyst, no biodiesel was formed (data yield of biodiesel using 0 % catalyst amount was not shown in the figure).

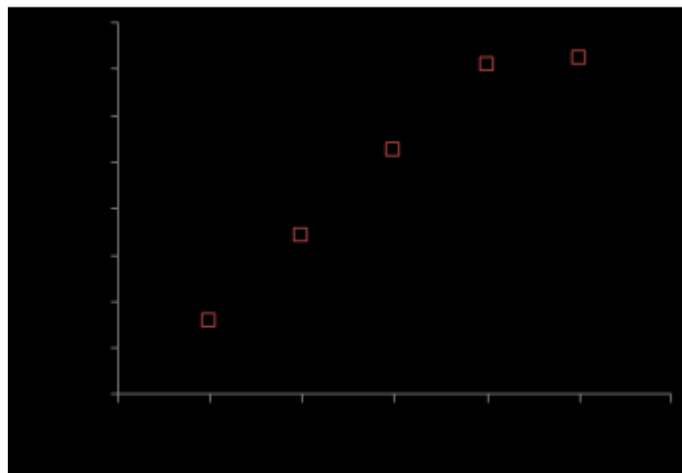


Figure 2. Effect of amount of ES-CaO catalyst on yield of biodiesel

In contrary, yields of biodiesel linearly increase with the increasing the amount of catalyst at 3 up to 6%. It is strongly evidence that the presence of catalyst affected the reaction rate [2]. In this work, the maximum yield of biodiesel of 5.62% was found at 7% catalyst amount of ES-CaO. However, at catalyst loading amount of 6-7% there is no increased significantly on yield of biodiesel. Therefore, for the economic point of view, 6% amount of catalyst was decided as the optimum condition, and it was selected for further studies.

### 3.2.2. Effect of reaction temperature on yield of biodiesel

Evaluating the influence of reaction time on yield of biodiesel on transesterification of UCO using ES-CaO catalyst, the experiments were conducted by varying time of reaction from 1 to 7 h with range of 1 h. Meanwhile, the others conditions were kept constant, at 65°C, molar ratio treated UCO to methanol of 1:15, and amount of catalyst of 6% (wt. % to UCO).

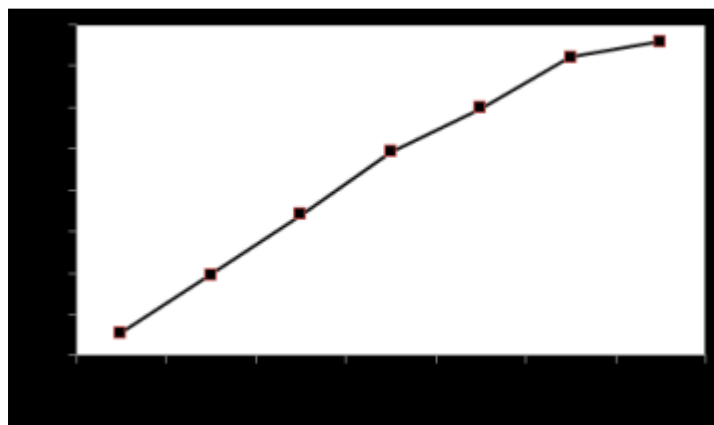


Figure 3. Effect of reaction time (h) on yield of biodiesel (%)

Figure 3 depicted that yield of biodiesel significantly increased with increasing of the time of reaction. In 1 h of reaction, yield of biodiesel was low, because it is a heterogeneous reaction and the fact that mass transfer is slow in the earlier of reaction. Huaping et al. (2006) reported the similar result [27]. After one up to 6 h, yield of biodiesel linearly increase from 5.54 up to 72.05%. At reaction time of 6 up to 7 h, biodiesel yields were gradually increased from 72.05 to 75.92 %. It seems that the equilibrium condition was achieved at 7 h with highest yield of 75.92%. It proved that the yield of biodiesel increased up to certain of reaction time. Refaat et al. (2008) and Asri et al. (2013) stated the similar statement [19], [28]. Therefore, the reaction time of 7 h was decided as the optimum reaction time on transesterification of used cooking oil into biodiesel using ES-CaO catalyst.

## 4. Conclusion

Calcium oxide (CaO) solid base Catalyst has been successfully prepared from eggshell waste by calcination process at high temperature (900°C). The eggshell waste (ES-CaO) catalyst has been introduced to transesterification process of used cooking oil with refluxed methanol. The high content of free fatty acid in used cooking oil reduced using 7% (wt.% to UCO) of coconut coir powder. The transesterification reaction was carried out under constant stirring speed at 65 °C and molar ratio of UCO to methanol 1:15. The result shown, that the amounts of catalyst and reaction time have significant effect on yield of biodiesel. The high enough biodiesel yield of 75.92% was obtained using 6% of catalyst amount within 7 h of reaction time. It proved that the eggshell waste catalyst seems to be very promising for producing biodiesel from used cooking oil. However, the activity of the eggshell catalyst should be improved to achieve the higher biodiesel yield (95%) in a shorter time of reaction. Therefore, the study of developing and engineering of CaO solid base catalyst derived from shell waste is still need to be encouraged to enhance the efficiency of biodiesel production from used cooking oil.

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