**Modified CaO Catalyst from Golden Snail Shell (*Pomacea canaliculata*) for Transesterification Reaction of Used Cooking Oil**

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***Abstract****:* One of alkaline earth metal oxide is calcium oxide, CaO has more attention because it has high strength, low solubility in methanol, and can be synthesized from sources that are easy to obtain. One of the sources of CaO and showed its catalytic properties was the golden snail shell. In this study, synthesize ash golden snail shell modified with fly ash leached and non-leached as a source of silicate (SiO2) has been investigated. The CaO/fly ash catalyst was applied to the transesterification reaction of used cooking oil. There are three catalysts have synthesized, namely golden snail shell calcined (CK), 75% golden snail shell calcined modified by 25% fly ash (CKFA), and by 25% fly ash leached (CKFAL). Based on the results of characterization with spectrophotometer X-Ray Diffraction (XRD), golden snail shell calcined at 900 ˚C containing 93.94% Ca (OH)2. Modified CaO catalyst from golden snail shell and fly ash were active to convert used cooking oil become biodiesel.

**Keywords***: catalyst, fly ash, leaching, golden snail shell, transesterification reaction.*

1. **INTRODUCTION**

Biodiesel is a methyl ester as bioenergy or biofuels made from vegetable oils or animal oils by transesterification, esterification, or esterification-transesterification process (Knothe, 2000). Biodiesel has been selected as an interesting alternative fuel because it is renewable, biodegradable, non-toxic, and it has similarity of physical and chemical characteristics to conventional diesel fuel (Lotero, et.all., 2005; Sanjay, 2013). Besides, use of biodiesel as a fuel can decrease pollutants such as CO2, SO2, CO, dan HC gas (BSN, 2012; Huang, et.all., 2012; Endalew, et.all., 2012).

The abundant biodiesel feedstock is vegetable oils. The main contents of vegetable oils are triacylglycerol which has three ester or fatty acid chain (acyl functional group) that attached on glycerol functional group (Endalew, et.all., 2012; Issariyakul, et.all., 2012). One of the abundant and easily available vegetable oils is used cooking oil. The use of used cooking oil can help reduce the cost of biodiesel production. Used cooking oil is waste material derived from palm oil which has been used many times. Generally, free fatty acids in used cooking oil is quite high, so it can be caused saponification reactions (Moecke, et.all., 2016). Therefore, an esterification reaction is needed to reduce free fatty acids, using an acid catalyst (usually sulfuric acid, hydrochloric acid, and sulfonic acid) (Ullah, et.all., 2015). After the esterification reaction was carried out, then production biodiesel was carried out through a transesterification reaction using catalyst (Kolyaei, et.all., 2016).

A catalyst is needed to accelerate the conversion of triglycerides (oil) and alcohol into alkyl esters (biodiesel) and glycerol. The catalyst commonly used is a homogeneous base catalyst, for example NaOH, KOH, and other alkoxides. The use of homogeneous catalysts causes the saponification reaction that produce soap and water which can reduce biodiesel yield (Semwal, et.all., 2010).

 Therefore, many studies related to heterogeneous catalyst preparation for biodiesel production. Heterogeneous catalysts have several advantages, which are less toxic, non-corrosive, environmentally friendly, easily be separated and can be reused. Heterogeneous metal oxide catalysts, a type of catalyst often used in the biodiesel conversion process, metal oxides are easily available because they are abundant in nature (Semwal, et.all., 2010; Santos, et.all., 2015; Saadon, et all., 2014; Olutoye et.all., 2016; Bennett, et.all., 2014).

Heterogeneous catalysts that show good catalytic activity in transesterification reactions is calcium oxide (CaO). CaO catalyst from limestone has better performance than catalyst from Calcium Hydroxide and Calcium Carbonate to convert biodiesel from the palm oil. Biodiesel yield obtained by using CaO from limestone, Ca(OH)2, and CaCO3 are 89.98%, 85.15%, and 78.71% sequentially (Widayat, et.all., 2017). CaO catalyst can be prepared from natural material and waste material. The waste food material namely crab shell, egg shell, fish bone, and golden snail shell can be used as CaO sources.

Golden snail shell contains calcium carbonate, calcium phosphate, silicate, magnesium carbonate, phosphorus, potassium, sodium, iron, and other organic substances. Calcium carbonate converted through the calcination process to gain CaO (Shah, et.al., 2015; Nopriansyah, et.al., 2016; Pancawati, 2016; Puspitasari, et.all., 2018; Prastyo, et.all., 2011; Septiani, 2013).

 The use of golden snail shells as catalysts has been investigated by Ki et al. (Ki, et.all., 2017) for biodiesel transeterification reactions. The conclusion of the study states that golden snail shells have the potential as a catalyst for biodiesel production. Prastyo, et al. (2011) also conducted a study on the use of golden snail shells as a catalyst in the manufacture of palm oil-based biodiesel. The maximum biodiesel yield obtained was 94.43%.

The catalytic activities of CaO catalyst could be increased by adding support material. One of the supporting materials that can be used is fly ash. Fly ash is a by-product of coal combustion consisting of micro particulates. The high amount of SiO2 and Al2O3 in fly ash is very potential as a cheap catalyst support material, considering fly ash is a waste material (Jain, et.all., 2011; Chakraborty, et.all., 2010). According to Jain et al (2010) dispersion of fly ash on CaO catalyst increases catalytic activity due to increase the base strength (OH content) catalyst. Enggawati and Ediati (2013) modified the CaO catalyst from egg shell with fly ash to catalyze transesterification reaction of Nyamplung oil. Furthermore, Rodiah and Ediati (2015) synthesized CaO catalysts from modified dolomite with leaching and nonleaching fly ash. It was reported that CaO catalyst supported by fly ash was able to convert refined palm oil to biodiesel.

Based on the results from some of studies above, production of biodiesel in this study used golden snail shell (Pomacea canaliculata) as a source of modified CaO with fly ash from PT. Bukit Asam Persero (Tbk) Tanjung Enim, South Sumatra.

1. **RESEARCH METHODOLOGY**

**Tools**

The equipments used in this study such as beaker glass, porcelain dish, measuring glass, filter paper, dropping pipette, magnetic stirrer, mortar agate, analytical balance, oven, muffle furnace, glass funnel, triple neck flask, condenser. X-Ray Diffractometer is used to charcterize the catalysts.

**Materials**

 The materials used in this study are golden snail shell from Pelajau village Banyuasin South Sumatera, fly ash from PT. Bukit Asam Persero (Tbk) Tanjung Enim South Sumatera, used cooking oil from street vendors around the campus of UIN Raden Fatah Palembang, methanol pa from Merck, n-hexane pa from Merck, and aquades from Merck.

**Procedure**

**1. Preparation of Catalysts**

1. **Preparation of golden snail shell**

The golden snail shell is washed using clean water, and then crushed. After that the shell is dried for 24 hours at 110˚C, then calcined at 900˚C for 2 hours. The calcined golden snail shell is mashed with agate to get powder.

1. **Preparation of Fly Ash**

 Fly ash washed using hot water at glass beaker, then stirred for 30 minutes, this procedure is repeated 3 times. The deposited mixture was filtered, then dried at 100˚C for 24 hours. The powder obtained is called FA. The fly ash was leached with a 10% hydrochloric acid solution (25:1 (mL /g), then stirred using a magnetic stirrer at 80˚C for 1 hour. The precipitate is washed with distilled water for about 3 times then filtered. Then the precipitate is dried at 100˚C for 24 hours. The powder obtained is fly ash leaching (FAL).

1. **Preparation of golden snail shell catalyst/fly ash**

 Two beaker glass which contained of 75% (m/m) of calcined snail shells were added 200 mL aquades for each beaker glass and stirred until homogeneous. Then, the first beaker glass added by 25% (m/m) FA, and the other added by 25% FAL. The mixtures stirred at 70˚C, pH 12.1 for 4 hours then left for 24 hours. The precipitate obtained was oven-heated for 20 hours at 100˚C, then calcined at 800˚C for 2 hours.

**2. Characterization of Catalysts**

 The crystal structure of calcined golden snail shell was confirmed by Philips X-pert XRD Powder Diffractometer using CuKα radiation with an angle range of 2θ = 20-100˚. Furthermore, each golden snail shell/fly ash catalyst was characterized using XRD with CuKα radiation at an angle range of 2θ = 20-80˚ at a scanning speed of 1˚/min to identify the crystal structure of the catalyst.

**3. Transesterification Reaction**

 The transesterification reaction is carried out in a 250 mL three neck flask equipped with a condenser and magnetic stirrer. The reaction conditions used were 3% of the catalyst concentration by weight of oil, 1:30 of the ratio oil: methanol (m/m), 65 ˚C, 2 hours with the mixing speed of 1200 rpm. The biodiesel obtained is added n-hexane to dissolve the desired product. n-Hexane is separated from the product using rotary evaporator. Biodiesel obtained is analyzed by Gas Chromatography to determine biodiesel yield.

1. **RESULT AND DISCUSSION**

Golden snails included in the class of molluscs, have soft bodies protected by shells. Golden snail shells are composed of calcium phosphate, silicate, magnesium carbonate, iron, and other organic compounds, while the main content is calcium carbonate. Golden snail shells are hard textured so certain equipment is needed to destroy them. Golden snail shells are washed and dried to remove impurities and remnants of meat that are still attached to the shell before calcined at 900˚C for 2 hours to obtain golden snail shell powder called CK catalyst. The Calcination process serves to activate the catalyst because during calcination at high temperatures, the carbonate group decomposes to CaO (eqs. 1) which plays an important role in transesterification reactions (Aransiola, et.all., 2014; Shajaratun, et.all., 2014; Ilgen, 2011). Etuk et al (2011) reported that the main content in mash snail shell ash was CaO of 61.95%.

CaCO3(s) 🡪 CaO(s) + CO2(g) eqs. 1

Addition of supporting material is one way to increase the catalytic activity of CaO catalysts. Fly ash is used as a catalyst support material to increase the surface area of the catalyst. Fly ash consists of silica, alumina, iron oxide, lime, magnesium and alkali in various quantities with some activated carbon which is not burned and has a large surface area in the range of 40-115 m2/gm. In this study, fly ash is a source of silica (SiO2) which will be impregnated on the surface of the CK catalyst. The fly ash used in this study were fly ash washed with hot water (FA) and fly ash leached by HCl (FAL) to dissolve impurities that are not soluble in water. This study produced three types of catalysts with different compositions, see Table 1.

Then the catalysts are re-calcined to reactivate the CaO catalyst which has been changed to Ca(OH)2 after being dissolved in water in the preparation process. Reactions that occur according to the following equation 2. The presence of Ca(OH)2 was confirmed by an X-Ray Diffraction (XRD) spectrophotometer (Table 2).

**Tabel 1**. The composition of the catalyst synthesized

|  |  |  |
| --- | --- | --- |
| **No** | **Composition (% mass)** | **Name of catalyst** |
| **FA** | **FAL** | **CK** |
| 1 |  |  | 100 | CK |
| 2 | 25 |  | 75 | CFA |
| 3 |  | 25 | 75 | CFAL |

CaO(s) + H2O(l) 🡪 Ca(OH)2(s) eqs. 2

In this study, fly ash was used as a source of SiO2 which was dispersed on CaO catalyst from golden snail shell. The presence of SiO2 on the surface of the CaO catalyst affects the content of Ca(OH)2. This is caused by the reaction between Ca(OH)2 and SiO2, thereby reducing the levels of Ca(OH)2 on each catalyst. The catalyst phase was also confirmed by an XRD spectrophotometer. The diffractogram of calcined golden snail shell (CK) is shown in Figure 1. Based on the diffractogram the CK catalyst shows the peak that appears at 2θ = 28.64°; 34.13°; 47.26°; 50.99°; and 54.44° which is the peak of CaO. Then a new peak appears at 2θ = 17.95° and 47.26° which is the phase of Ca(OH)2. The peak that appears shows the occurrence of hydration during activation of the catalyst (Prastyo, et.all., 2011).

**Tabel 2**. The content of Ca(OH)2 on the prepared catalyst

|  |  |  |
| --- | --- | --- |
| **No** | **Katalis** | **Kandungan Ca(OH)2 (%)** |
| 1 | CK | 93,94 |
| 2 | CKFA | 67,22 |
| 3 | CKFAL | 68,06 |

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**Figure 1**. The diffractogram of CK catalyst

CaO/fly ash catalyst preparation has been carried out by Rodiah and Ediati (2015) that CaO catalysts from calcined dolomite have CaO peak characteristics at 2θ = 28.88°; 34.15°; 47.32°; 51.02°; and 54.54°. Prasetyo et al. (2011) synthesized the catalyst from golden snail shells and reported that calcined shells showed peak characteristics of 2θ = 32.1°; 37,3°; 53.9° for the CaO phase and 2θ = 17.9°; 28,6°; 34,1°; 46.9°; and 50.7° shows the phase of Ca(OH)2. The diffractogram of the CK catalyst showed no appearance of peak phase characteristics of CaCO3. This shows that CaCO3 was completely converted to CaO and Ca(OH)2 in the calcination process.

Figure 2 is a comparison of the diffraction patterns of the three catalysts, namely CK, CKFAL, and CKFA. There are several peak characteristics that emerge, among others CaO peaks originating from shells, and quartz peaks (SiO2), mulit (Al2O3.SiO2), and hematite (Fe2O3) originating from fly ash. Peak at 2θ = 28.78°; 34.06°; 47.33°; 50.85°; and 53.9° is the peak of CaO on CKFAL catalyst whereas in CKFA catalyst, the characteristics of CaO appear at 2θ = 28.71°; 34.13°; 47.26°; 50.79°; and 53.9°.

In addition, the new peak appears at 2θ = 29.45°; 32.84°; and 2θ = 29.39° is the peak of mulit (Al2O3.SiO2) from each CKFAL and CKFA catalyst respectively. Then peak at 2θ = 20.92°; 26.61°; 39,54°; and 2θ = 20.92°; 26.75°; 39.41°; is characteristic of quartz peaks (SiO2) from CKFAL and CKFA catalysts respectively. The highest peak that emerged was the peak of Ca2SiO2 indicating that the intensity of SiO2 on the CKFAL catalyst was higher due to the treatment of leaching, thus the silicates that interacted with CaO formed more dicalcium silicate. Dicalcium silicate formed from dicalcium silicate hydrate which is the reaction of conch shell powder (CaO) with fly ash (SiO2). The reactions are shown in the equations 2.

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**Figure 2**. The diffractogram of CK, CKFAL, and CKFA catalyst

Dicalcium silicate hydrate ((CaO)2(SiO2)(H2O)) changes to dicalcium silicate after calcination at 800° C for 2 hours. Water in dicalcium silicate hydrate evaporated and formed dicalcium silicate (Ca2SiO2) after calcination process. The presence of dicalcium silicate in the catalyst can increase the basicity of the catalyst because of the Si-O-Ca bond (Chakraborty, et.all., 2010).

2CaO + SiO2 + H2O 🡪 (CaO)2(SiO2)(H2O) eqs. 3

The catalysts are then used to catalyze the biodiesel production process from used cooking oil. Used cooking oil is waste cooking oil that has been used several times. Therefore, the content of free fatty acids (FFA) in cooking oil increases. Increased FFA is caused by a reaction between chemicals in oil (water, vitamins, and minerals) in the frying process. The FFA levels were determined by NaOH solution through the titration process. Used cooking oil in this study contained 0.63% FFA which was classified as low level, so that it can be directly used in the transesterification reaction without going through esterification. In this study, the transesterification reaction was catalyzed by three catalysts, namely CK, CKFA, and CKFAL. The mass of biodiesel from the reaction with CK, CKFA, and CKFAL catalysts was 20.43 grams, 18.62 grams and 19.89 grams, respectively (See Table 3).

**Table 3**. Biodiesel mass after evaporating

|  |  |  |  |
| --- | --- | --- | --- |
| **No.** | **Catalyst** | **Mass of used cooking oil (g)** | **Biodiesel mass (g)** |
| 1 | CK | 15,0111 | 20,4309 |
| 2 | CKFA | 15,0126 | 18,6241 |
| 3 | CKFAL | 15,0059 | 19,8990 |

From these results indicate that the CK catalyst shows better activity compared to other catalysts. Dispersion of fly ash on CaO catalyst affects catalyst activity. Catalysts dispersed with leaching fly ash have better catalytic activity than catalysts dispersed with non-leaching fly ash. This can be caused by the active side of the shell catalyst base being higher than that of the CFA catalyst and CFAL due to the dissolution of fly ash with acid. The acid leaching process causes dissolution of metal impurities which can cover the active side of the catalyst. The more active sides of the catalyst base, the higher the catalyst activity. Thus the conversion of biodiesel increases because the transesterification reaction depends on the number of active sides of the base (Ilgen, 2011).

On the other hands, CK catalysts are more active than CKFA and CKFAL catalysts. According to Rodiah and Ediati (2015), dispersion of fly ash on the surface of CaO can cover the active side of the catalyst, thereby causing a decrease in catalytic activity. Therefore, the main key to increasing the modified catalytic activity of CaO is to maintain the CaO side during the modification process (Ngamcharussrivichai, et.all., 2010). However, the difference between the mass of biodiesel generated from the use of CK catalysts (20.4309 grams) and CKFAL (19.8990 grams) is quite small. This shows that leaching fly ash can be used as a CaO catalyst support from a potential snail shell. In addition, the density of biodiesel (Table 4) with the use of CKFAL catalyst obtained are closer to the characteristics of biodiesel according to the biodiesel quality requirements (BSN, 2012), that the density of biodiesel allowed is between 850 - 890 Kg / m3 or 0, 85 - 0.89 g/cm3.

**Table 4.** Density of biodiesel observed

|  |  |  |
| --- | --- | --- |
| **No.** | **Catalysts** | **Density (g/cm3)** |
| 1. | CK | 0,8090 |
| 2. | CKFA | 0,9158 |
| 3. | CKFAL | 0,8295 |

1. **Conclusions**
2. In this study the CK, CKFA, and CKFAL catalysts were successfully prepared which were used to catalyze the transesterification reaction of used cooking oil into biodiesel.
3. Based on the mass of the biodiesel obtained, the CK catalyst produces a higher mass than the reaction catalyzed by the CKFA and CKFAL catalysts. But the mass of biodiesel obtained from catalysis with CKFAL and CK catalysts did not have a significant difference.
4. Based on the density characteristics of biodiesel, the reaction catalyzed by CKFAL catalyst has a density that approaches density in accordance with the quality standard which is 0.8295 g/cm3.

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