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paper text:

12ORIGINAL RESEARCH Open Access Calcium oxide from Pomacea sp. shell as a catalyst for biodiesel production Yosephine Yulia Margaretha, Henry Sanaga Prastyo, Aning

Ayucitra* and Suryadi Ismadji*

31Abstract Heterogeneous catalysts are promising catalyst for the transesterification reaction of vegetable oils to produce biodiesel.

4In this study, the Pomacea sp. shell was used as the raw material for CaO catalyst. The calcination of Pomacea sp. shell was conducted at 900°C for 2 h. The raw material and the resulting CaO catalyst were characterized using X-ray diffraction, X-ray

fluorescence spectrometry, and Fourier transform infrared spectroscopy analysis. From the experimental results,

25it was found that the maximum yield of fatty acid methyl esters was 95.61% at

11the following reaction conditions: reaction temperature of 60 °C, a reaction period of 4 h, a ratio of methanol-oil at 7 :1, and amount of catalyst at 4% w/w. The

physical and chemical

7properties of biodiesel were determined based on ASTM standard, and the

values obtained were confirmed with the Indonesian National Standard (SNI-04-7182-2006) and (B100)-ASTM D6751-07b. Keywords: Biodiesel, Transesterification, Solid catalyst, CaO, Palm oil Background The extensive use of fossil fuel in human activities during the last several decades causes the depletion of fossil fuel source; therefore, the search of other alternative energies is crucially important [1,2]. One of the alternative fuels which have been developed over a century ago is bio-diesel. Biodiesel has been chosen as an alternative fuel because it is renewable,

20produces lower emission, and possesses high flash point, better lubrication, and high cetane number [3]. Studies about the

transesterification of vegetable oils or animal fats into biodiesel have been conducted by various researchers. At the time of reaction, the triglycerides will gradually be converted into diglycerides, monoglycerides, and glycerol. At each change of conversion, one mole of ester formed [4]. Different kinds of catalysts have been in use to produce biodiesel from a different kind of oil. The catalysts commonly used for biodiesel production are categorized into several types: homogeneous catalysts (sodium hydroxide, potassium hydroxide, sulfuric acid, etc.) [5], heterogeneous catalysts (cation-exchange resin, hydrotalcites, etc.), and enzymes (Chromobacterium viscosum, Candida rugosa, and Porcine pancreas) [6]. *

Correspondence: aayucitra@yahoo.com;

10suryadiismadji@yahoo.com Department of Chemical Engineering Widya Mandala Surabaya Catholic University, Kalijudan 37, Surabaya 60114, Indonesia

5Homogeneous catalyst has been used extensively for biodiesel production, but the use of this kind of catalyst began to decrease because it has several drawbacks: it cannot be recovered [7] or regenerated after the reaction and also it produced toxic wastewater.

To replace the homogenous catalyst, enzyme and heterogeneous catalysts have been developed and studied. The use of enzyme as a catalyst is less desirable because it is much more expensive than the homogeneous catalyst. Heterogeneous catalysts such as KOH/Al₂O₃ [8] and alumina/ silica [9] have

3a high level of alkalinity and reliable performance for biodiesel production. Nevertheless, these catalysts have some drawbacks

since

3they are easily dissolved in methanol, relatively difficult to synthesize, and susceptible to humidity.

For acid catalyst, higher molar ratio of methanol/oil should be used, and it requires longer reaction time [8,10,11]. Therefore, a new heterogeneous catalyst should be developed, which is reactive at

3 slow temperature and pressure, has short time reaction for various raw materials, easy to get,

has a high conversion, and low biodiesel production cost [12]. Golden snails (*Pomacea* sp.) are mostly found in muddy areas such as in a rice field. It is considered as pests; therefore, its presence in the rice field is not desired. However, the meat of *Pomacea* sp. has high protein content and can be utilized as animal feed or as © 2012 Margaretha

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an alternative food for human consumption. In Indonesia, this mollusk has been cultivated in small industrial scale. Its meat has been utilized as raw materials for various kinds of food, while the shell is discarded as waste. The utilization of *Pomacea* sp. shell as an alternative raw material in the production of solid catalyst for biodiesel

20 is expected to raise its economic value and

helps to overcome the waste problems of *Pomacea* sp. *Pomacea* sp.

16 shell can be utilized as a solid catalyst for biodiesel production because it contains CaCO₃ which can be converted into CaO during calcination.

CaO is a heterogeneous catalyst which is most widely studied because of its high alkalinity, low solubility, cheaper price than KOH/NaOH, and also the ease of handling [13,14]. This research was focused on developing

29 a new type of catalyst for biodiesel production

from *Pomacea* sp. shell. The capability of the catalyst derived from the snail shell was tested

26 on the transesterification of palm oil into bio-diesel. The process parameters for

biodiesel production were also studied. Methods Materials and catalyst preparation The *Pomacea* sp. shell was obtained from a small town called Pati, located in Central

13 Java, Indonesia. Prior to use, the shell was repeatedly washed using tap water to remove dirt

and other impurity material and then dried overnight at 100°C. The shell was then crushed and calcined at 900°C for 2 h in a tubular furnace to convert CaCO₃ into CaO. After the reaction is complete, nitrogen with a flow rate of 3 l/min was introduced to furnace to cool down the system and to prevent the air entering the system. Subsequently, the catalyst was removed from the furnace and kept in a desiccator to prevent contact with air. Methanol used in this study was purchased as analytical grade from MERCK, Germany. Palm oil was purchased from the local supermarket, and the composition of fatty acid in the oil was

analyzed using gas chromatography. The results are shown in Table 1. The gas chromatography analysis of fatty acid was performed using

13a Shimadzu GC-2014 equipped with FID and a DB-wax capillary column.

The operating condition for fatty acid determination as follows: helium was used as a gas carrier with a flow rate of 40 m/s. The

8 injector temperature was set at 200 °C, while the detector was operated at 300 °C. The initial oven temperature was 100 °C

and maintained in this isothermal condition for 2

22 min. Then, the oven temperature was increased to 220 °C at a heating rate of 10 °C/min and held for 33 min.

Other important parameters such as water content and acid value content in the oil are also given in Table 1. The acid value was determined according to ASTM Table 1 Composition of fatty acid in palm oil and other characteristics Composition

6 Fatty acid (%) Lauric acid (C12:0) 0. 81 Myristic acid (C14:0) 1. 22 Palmitic acid (C16:0) 42.42 Palmitoleic acid (C16:1) 0. 21 Stearic acid (C18:0) 4. 68 Oleic acid (C18:1) 40.78 Linoleic acid (C18:2) 9.45 Linolenic acid (C18:3) 0.

11 Arachidonic acid (C20:0) 0.32 Water content (%) 0.05 Acid number (mg KOH/g oil) 0.85 D664, and the water content was determined according to ASTM D6304-07. Transesterification reaction procedure The transesterification of palm oil into biodiesel using CaO derived from *Pomacea* sp. shell

24 was carried out in a 500 -ml three-neck round-bottom flask equipped with a reflux condenser, thermometer, and magnetic heating mantle. The

transesterification experiments were carried out at a temperature of 60°C and stirring speed at 700 rpm. The initial mass of the palm oil used in this study was 200 g. The amount of catalyst varied from 1% to 5% w/w, and oil to methanol ratios were 1:5 to 1:11.

25 Reaction time used in this study was 4 h. After the

reaction completed, the catalyst was removed from the mixture using vacuum filtration. The

8 excess methanol was then recovered from the biodiesel mixture using a rotary vacuum evaporator.

After the separation of methanol, the glycerol was removed from the biodiesel in a funnel separator. The biodiesel was obtained in the upper layer, while the glycerol as the byproduct was in the lower layer. For the comparison, biodiesel preparation using NaOH as a catalyst was also conducted under the same operating condition with those using CaO as catalyst. Catalyst characterization The raw material (*Pomacea* sp. shell) and the catalyst

29 were characterized using several methods such as X-ray diffraction,

X-ray fluorescense (XRF) spectrometry, Fou- rier transform infrared spectroscopy (FTIR) analysis, and nitrogen sorption analysis. XRD analysis was employed to investigate the crystalline structure of the raw material and catalyst. The diffraction spectra of both materials were obtained on a Philips X'Pert diffractometer (Almelo, The Netherlands) using CuK α as the radiation source. The measurement was conducted in 2 θ angle between 8° to 72°. XRF spectrometry was used to identify and determine the bulk composition of a catalyst, and for this pur- pose, Rigaku ZSX100e X-ray fluorescence (Tokyo, Japan) was employed. FTIR measurement was carried out under FTIR spectrophotometer (Shimadzu 8400S, Nakagyo-ku, Kyoto, Japan) using the KBr technique with a wave- number range of 4,000 to 700 cm $^{-1}$. Surface analysis of the catalyst was conducted by nitrogen sorption experi- ment at its boiling point (-196°C) using a Quadrasorb SI (Quantachrome Instruments, FL, USA). Degassing of catalyst before the nitrogen sorption measurement was conducted under vacuum condition for 24 h at a tem- perature of 150°C. The standard

17BET equation was employed to calculate the BET surface area of the catalyst.
The

BET calculation was performed

17in the relative pres- sure (p/p0) range of 0.06 to 0.3.

Biodiesel characterization Selected

28physical and chemical properties of biodiesel including viscosity,

cetane index, fatty acid composition, flash point, water and sediment content, and heat of combustion were determined. The values were then compared with the Indonesian National Standard (SNI- 04-7182-2006) and Biodiesel (B100)-ASTM D6751-07b. The cetane number was evaluated by ASTM D613 which is a standard method for cetane number of diesel fuel oil. The density was determined by ASTM D1298, while the kinematic viscosity using ASTM D445-10. Standard test method for water and sediment in middle distillate fuels using centrifuge (ASTM D2709) was employed to determine the water and sediment content in biodiesel. For

21flash point determination, ASTM D93 (standard test method for flash point by Pensky- Martens closed cup tester) was employed. The average values of

physical and chemical characteristics of

28biodiesel obtained in this study are summarized in Table

2. The

18fatty acid methyl ester (FAME) content in the bio- diesel was determined using gas chromatography (GC)

method. For this purpose, the GC (Shimadzu GC-2014) was

27equipped with a capillary column DB- wax (length 30 m × diameter 0.25 mm
× film thickness 0.1

μm) and a flame ionization detector operated at 300°C.

23Helium was used as the carrier gas with a linear velocity of 40 cm/s. The injector temperature was

250°C at splitless condition. The initial oven temperature was 50°C with an equilibration time of 3 min. After isothermal period, the oven temperature was increased to 250°C at a heating rate of 10°C/min and held for 8 min.

14Peaks of methyl esters were identified by comparison with the reference standard.

The

9biodiesel yield was calculated by the following expression: Yield = (weight of biodiesel × % FAME)/(weight of oil) × 100%. úME is the concentration of FAME obtained by GC analysis.

Results and discussion Characterization of catalyst The X-ray diffraction patterns of the raw material (Figure 1) and CaO catalyst (Figure 2) show the presence of CaCO₃ and CaO. The presence of CaCO₃ is indicated by 2θ at 26.2°, 33.1°, 37.8°, 45.8°, and 52.4°, while for CaO at 2θ, it is around 32.2°, 37.3°, and 53.8°. The presence of Ca(OH)₂ is also observed in Figure 2 as indicated by diffraction peaks at 18.0°, 28.6°, 34.1°, 47.0°, and 50.8°. Calcium oxide has strong basic property due to the presence of oxygen anion generated on its surface [15,16], and the exposure of this compound to ambient air results to the formation of Ca(OH)₂ due to the reaction of H₂O which is present in the air with its strong basic surface. The

26bulk composition of a catalyst was determined by XRF, and the

result is depicted in Figure 3. The composition of the catalyst produced by calcination of *Pomacea* sp. shell mainly consists of CaO (96.83%) with small amount of other metal as impurities as indicated in Figure 3. The FTIR spectra of both *Pomacea* sp. shell and catalyst are given in Figure 4. For both *Pomacea* sp. shell spectra, the characteristic peaks of C-O stretching and bending modes of CaCO₃ are observed at 2,513, 1,420, 867, and 3,117 cm⁻¹. During the calcination process, CaCO₃ decomposed into CaO and CO₂. This is in line with the FTIR spectra of the catalyst which indicate that the intensity of characteristic peaks belonging Table 2 Comparison of the properties of biodiesel produced from palm oil Property Biodiesel produced in this study SNI (B100)-ASTM D6751-07b

18Density at 15°C (g/mL) Kinematic viscosity at 40°C

(cSt) Flash point (°C) Cetane number Acid number (mg KOH/g) Water and sediment (vol.%) 0.87 ± 0.09 0.85 to 0.89 3.7 ± 0.2 2.3 to 6.0 163 ± 2.2 100 min 57 ± 2.2 51 min 0.40 ± 0.07 0.8 max 0.03 ± 0.006 0.05 max - 1.9 to 6.0 93 min 47 min 0.5 max 0.05 max With catalyst from *Pomacea* sp. shell with the Indonesian National Standard (SNI-04-7182-2006) and (B100)-ASTM D6751-07b. Figure 1 X-ray structure of *Pomaceae* sp. shell to CaCO₃ decreases. After the calcination process, a new peak appears at 3,620 cm⁻¹; this evidence indicates the formation of basic OH groups which attached to the calcium atoms [17]. The physical and chemical characteristics of CaO catalyst are summarized in Table 3. Transesterification of palm oil The transesterification of palm oil into biodiesel was conducted at different process parameters. Table 4 summarizes the yield of biodiesel obtained at different operating conditions (amount of catalyst 1% to 5% w/w, reaction time 4 h, and methanol to oil ratio 5:1 to 11:1). The maximum yield of biodiesel obtained in this study was 95.61 ± 2.43 at operation condition: catalyst amount of 4% w/w (catalyst to oil ratio) and methanol to oil ratio of 7:1. This result is comparable to that obtained by Chen et al. [18] in other heterogeneous catalyzed trans-esterification process and higher than the result obtained by Buasri et al. [19]. The maximum yield obtained from the process with heterogeneous catalyst was slightly lower than the process using NaOH as catalyst (97.24%). Even the heterogeneous catalyst produces lower yields of biodiesel, however, this process offers more advantages compared with homogeneous one, such as environmentally friendly, the catalyst is re-useable, etc.

15By increasing the amount of catalyst, the yield of biodiesel also increased,

as indicated in Table 4. In transesterification process, the CaO basic sites catalytically transform the methanol into nucleophile molecule which is much more reactive, and subsequently, this nucleophile attacks the carbonyl carbon structure of glyceride molecules [16,20]. As the amount of catalyst increases, the available catalytically basic sites also increase leading to the increase of biodiesel yield. Stoichiometry of a transesterification reaction shows that in order to produce biodiesel, 3 mol of methanol/1 mol of triglyceride was needed [21]. Since the transesterification reaction is a reversible reaction, the adding of excess methanol drives the reaction towards the formation of biodiesel. In general, the conversion of triglyceride into fatty acid methyl esters (biodiesel) increased when the

32methanol to oil ratio was increased from 5 :1 to 7 :1. However, further increased of methanol to oil ratio caused the yield

of biodiesel decreased, as indicated in Table 4. With the excessive amount of methanol, the formation of fatty acid methyl esters and glycerol becomes faster, and after the appreciable amount of glycerol was produced, the reaction between catalytically active sites of CaO with glycerol occurred and produced calcium glyceroxide [16,22]. This calcium glyceroxide is less active Figure 2 X-ray structure of the catalyst than CaO for transesterification of palm oil; therefore, less in the industrial scale production is its stability. The catalyst biodiesel was obtained. More detailed information about should be stable in a period time of operation without loss the reaction mechanism of the formation of calcium glyceroxide and its catalytic capability or activity. In order to test the stability and its effect on the transesterification reaction ability of CaO catalyst derived from Pomacea sp. shell, three may be found in the paper of Kouzu and Hidaka [16]. reaction cycles were carried out using the following reaction One of the important considerations in selecting a heterogeneous catalyst: the amount of catalyst was 4% w/w, methanol to oil ratio was 7 :1, and reaction time was 4 h.

15ratio was 7 :1, and reaction time was 4 h.

Furthermore, Figure 3 XRF spectra of the catalyst. 3620 3117,72 2513 1500-2000 867,91 1420 Figure 4 FTIR spectra of the raw material and CaO catalyst. three different procedures for spent catalyst treatment were employed. The first procedure is the cheapest and easiest procedure; the spent or used catalyst was directly used without any further treatment for subsequent transesterification process. The second procedure involved the washing of spent catalyst with methanol to remove biodiesel and unconverted palm oil from the surface of the catalyst. As the last procedure, re-calcination of spent catalyst was conducted to determine the stability of the catalyst. Figure 5 depicts the yield of biodiesel at different spent catalyst treatments under cycle reaction conditions. Figure 5 clearly shows that the treatment of spent catalyst has great influence on the yield of biodiesel. For untreated spent catalyst, the deactivation of the CaO catalyst already occurred in the second cycle, thus the yield of biodiesel significantly decreased from around 95% to 38% (Figure 5a). Further use of this spent catalyst made the conversion of palm oil into fatty acid methyl esters (biodiesel) did not occur. During the separation Table 3 Physical and chemical characteristics of the CaO catalyst

34Property Value BET surface area (m²/g) 17 Pore volume (cm³/g) 0.04 Mean pore size (nm)

3.2 Particle size (mesh) 80/100 CaO content (%) 96.83 SiO₂ (%) 0.60 FeO (%) 0.42 Others 2.15 of spent catalyst from the liquid mixture, the adsorption of H₂O from the ambient atmosphere by the basic sites of CaO catalyst could not be avoided which caused the deactivation of catalyst. The reaction between the basic sites of CaO and the ambient H₂O was fast, and Table 4 Effect of different process variables

15on the yield of biodiesel Amount of Reaction Methanol to Yield of catalyst

(%) time (h) oil ratio biodiesel (%) 1 4 5:1 7:1 70.86 ± 3.46 7:1 86.63 ± 4.72 9:1 78.98 ± 3.14 11:1 83.50 ± 2.87 2 4:5:1 81.91 ± 2.44 7:1 86.33 ± 3.48 9:1 83.51 ± 4.22 11:1 84.07 ± 2.73 3:4 5:1 86.93 ± 3.03 7:1 87.60 ± 1.98 9:1 91.26 ± 2.51 11:1 89.22 ± 4.02 4:4 5:1 91.91 ± 3.91 7:1 95.61 ± 2.43 9:1 90.16 ± 2.78 11:1 92.55 ± 3.82 5:4 5:1 93.48 ± 3.07 7:1 85.81 ± 4.32 9:1 85.28 ± 2.17 11:1 91.16 ± 3.44 (a) 120 (b) 100 100 90 yield of biodiesel, % (c) Yield of biodiesel, % 80 20 Yield of biodiesel, % 80 60 70 40 60 50 0 40 30 0,5 1,0 1,5 2,0 2,5 3,0 3,5 4,0 4,5 0,5 1,0 1,5 2,0 2,5 3,0 3,5 4,0 4,5 Reaction time, h Reaction time, h 100 Fresh catalyst 90 Second cycle Third cycle 80 70 60 50 40 0,5 1,0 1,5 2,0 2,5 3,0 3,5 4,0 4,5 Reaction time, h Figure 5 The yield of biodiesel at different spent catalyst treatments under cycle reaction conditions. (a) Untreated spent catalyst, (b) washing of spent catalyst with organic solvent, and (c) re-calcination of spent catalyst. according to Kouzu et al. [22], it took only 3 min to ap- preciably reduce the catalyst activity. The treatment of spent catalyst with methanol gave better yields of biodiesel than non-treated spent catalyst. Here, the conversion of palm oil into FAME for the second and third cycles was higher than the transester- ification process using non-treated spent catalyst, as indicated in Figure 5b. As mentioned in the previous paragraph, the contact between the basic site and ambient H₂O may cause the deactivation of catalyst due to the

33formation of hydroxide groups on the surface of the

cata- lyst, and during washing of this spent catalyst with methanol, the

33hydroxide groups on the surface of

catalyst were isolated; these isolated OH groups with the help of the basic site of CaO still could act as an active site for transesterification reaction [16,23]. Figure 5c shows the yield of biodiesel synthesized using re-calcined spent catalyst. After transesterification process, the surface of spent catalyst was covered by hydrate, carbonate, and other compounds such as unreacted oil, biodiesel, glycerol, etc.; therefore, the catalytic activity of this spent catalyst drastically reduced. In the re- calcination process, all of those impurities were perfectly removed from the surface of the catalyst. By removing those impurities, the available basic sites in CaO surface also significantly increase, and the catalytic activity is simi- lar to fresh catalyst. However, one of the drawbacks of using re-calcination method is the high energy requirement.

Biodiesel characterization

1Density is one of the most important characteristics of biodiesel. In a combustion system, pumps and injectors require a precise amount of fuel to provide proper

com- bustion [24], and

1fuel density is the main characteristic that determines the amount or mass of fuel injected into the combustion chamber.

The density of the biodiesel produced in this study is given in Table 2, and the value is in accordance with the Indonesian National Standard (SNI-04-7182-2006). Viscosity of biodiesel is another important characteristic of biodiesel; it affects the atomization of fuel upon injection into the combustion chamber. Fuel with high viscosity has the tendency to form deposit in the engine, but fuel with low viscosity may not provide sufficient lubrication in the combustion system. The Indonesian National Standard has set the standard viscosity for biodiesel which is in the range of 2.3 to 6.0 cSt, while the ASTM viscosity standard for

7biodiesel is 1.9 to 6. 0 cSt. The average viscosity

values of biodiesel produced from palm oil using CaO catalyst derived from *Pomacea* sp. shell is 3.7 ± 0.2 cSt. This average

7 viscosity is in the range of the standard value of viscosity of biodiesel from SNI and

ASTM. Flash point of biodiesel relates to the ignition of the fuel. In order to handle the fuel safely, moderate value of flash point is required. The biodiesel produced in this study possesses higher flash point value than SNI and ASTM standards (Table 2); therefore, this fuel is safe for handling and storage for a period of time. The combustion quality of diesel fuel during compression ignition is indicated by an index called as cetane number. This index has great influence on the performance of the engine especially during combustion and exhaust emission. The low value of cetane index creates the problem to the engine during start up and produces noise. Also, during the combustion, smoke exhaust gas will be produced [25]. The average cetane number of the biodiesel obtained in this study is higher than the standard value of SNI and ASTM (Table 2). The cetane number of biodiesel depends on the distribution of

30 fatty acids in the raw material (oil); the longer fatty acid carbon chains, the higher the cetane number.

10 In this study, palm oil was employed as raw material for

biodiesel production, where methyl palmitate and methyl oleate are the major compounds in palm oil. These fatty acids have cetane indexes of 59.3 and 85.9, respectively [25]. Therefore, the minimum standard of cetane index will be easily fulfilled if palm oil is used as the raw material for biodiesel production. Furthermore, the other physical characteristic of biodiesel is also affected by the composition and length of fatty acid as mentioned by Ramos et al. [26]. The control of acid number on biodiesel production is also important since this parameter indicates the quantity of free fatty acids and mineral acids present in the biodiesel. Biodiesel with high value of acidity will cause corrosion and engine deposits, especially in the fuel injectors. The increase of acid number during storage is a strong indication that the degradation of biodiesel takes place. Water contamination is also another important parameter in biodiesel quality. Several problems associated with the presence of excess water in biodiesel are low calorific value, corrosion, growth of microorganisms, oxidation, and degradation of biodiesel during long-term storage. The standard values of these parameters were fulfilled by the biodiesel produced in this study as indicated in Table 2. Conclusion A catalyst for biodiesel production was prepared from the *Pomacea* sp. shell. The transesterification reaction of palm oil into biodiesel using this heterogeneous catalyst was studied under various operating conditions, and the maximum yield of biodiesel (95.61 ± 2.43) was obtained on the condition as follows: catalyst 4% and methanol to oil ratio 7:1. The reusability and stability are very important criteria for large-scale production of biodiesel using CaO catalyst prepared from *Pomacea* sp. shell. The activity of spent catalyst could be restored by re-calcination process. The physical and chemical properties of biodiesel produced conform to the available standards. Competing interests The authors declare that they have no competing interest. Authors' contributions YYM and HSP collected the experimental data, AA drafted the manuscript, and SI developed the idea and corrected the manuscript. All

19 authors read and approved the final manuscript. Acknowledgments The authors would like to express their sincere gratitude to the editor (Professor Farivar Fazelpour) and anonymous reviewers for

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