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Research Article

Biodiesel from the waste cooking oil is catalyzed by eggshell of purebred chicken with methanol as a solvent

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ABSTRACT

The synthesis of biodiesel from the waste cooking oil was carried out using the catalyst from eggshell of purebred chicken with methanol as a solvent. Synthesis of the biodiesel was prepared in two steps, there are esterification and transesterification. Esterification was conducted in a mol ratio of methanol and waste cooking oil was 9:1, with H_2SO_4 as a catalyst. Transesterification with mol ratio methanol and waste cooking oil was 12:1, with CaO from eggshell of purebred chicken as a catalyst. CaO catalyst was yielded by calcinations egg shell of purebred chicken on 1000 ^{0}C for two hours. The calcination product was characterized with X-RD determine of CaO. The result of biodiesel was characterized based on ^{1}H-NMR, FTIR, GC-MS, dan ASTM *(*American Standard Testing of Materials*)*. Yielded of biodiesel theoretically was 40.298% and experiment was 36.779%. The main component that resulted in there is methyl stearic (40.21%).

Keywords: biodiesel, esterification, transesterification, CaO

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INTRODUCTION

The phenomenon of increasing fossil fuel prices that occur in almost all countries in the world (BPPT, 2014), has made researchers try to find alternative fuels that can be widely used in society, such as biodiesel. Chemically, biodiesel is a methyl ester of long-chain fatty acids derived from renewable fats such as vegetable oils and animal fats. Biodiesel has the advantage of being biodegradable and non-toxic to the environment. Vegetable oil is a promising basic material for making biodiesel because it is available in large quantities in nature. However, the use of basic ingredients for making biodiesel is more directed at the use of non-edible oils (Sutapa et. al, 2013) and waste cooking oil known as waste cooking oil (Rahadianti et. al, 2018). Waste cooking oil contains fatty acids, so it is a good candidate as a basic material for making biodiesel (Suroso, 2016; Rahadianti et. al, 2018). The content of free fatty acids in waste cooking oil is higher than that of new cooking oil so saponification tends to occur easily (Maneerung et. al, 2016). One way to reduce the occurrence of saponification can be pre-treated, namely the esterification reaction with acid before the transesterification reaction is carried out.

Transesterification is one way to produce biodiesel, where alcohol reacts with oil with the help of a chemical catalyst to produce fatty acid alkyl esters and glycerol. The type of alcohol that is often used is methanol because the carbon chain is short so it reacts faster. There are three types of catalysts can be used, namely acid catalysts, base catalysts, and enzyme catalysts. However, judging from the reaction rate and the economical side, alkaline catalysts are preferred. Alkaline catalysts are divided into homogeneous and heterogeneous base catalysts. Judging from its nature the use of homogeneous base catalysts such as sodium hydroxide and potassium hydroxide can lead to the formation of soap and is also very difficult to separate from the material formed. In contrast to the use of heterogeneous base catalysts such as CaO and MgO which can be easily separated from the material formed (Maneerung et. al, 2016; Zabeti et. al, 2009). However, from an economic point of view, this heterogeneous base catalyst is quite expensive so if it is used to produce biodiesel, it will require quite a large production cost. Therefore, one way to produce biodiesel using heterogeneous base catalysts but with low production costs is to utilize purebred chicken egg shells.

In general, most of the eggshell waste is disposed of without pretreatment. The largest constituent composition of eggshells is calcium carbonate, so eggshells have the opportunity to become the basic material which will be converted to CaO and used as a catalyst in the transesterification reaction to produce biodiesel (Tehujujuluw et. al, 2014). These things are the background of research on the manufacture of biodiesel from waste cooking oil using a catalyst from eggshells of broiler chickens with methanol solvent.

METHODS

Material

Waste cooking oil, methanol (Merck), H2SO4 (Merck), egg shells, phenolphthalein indicator, anhydrous Na2SO4, distilled water.

Tools

Pyrex glassware, pyrex reflux kit set, mortar, 100 mess sieve, Mammert electric heater, oven, magnetic stirrer (Science Ware), analytical balance, thermometer, furnace 47900 (Merck), Buchii vacuum evaporator, ray diffractometer -X Shimadzu Goniometer XD-3A, Shimadzu IR spectrometer (FTIR- 8201 PC), 1H-NMR JNM PMX 50 NMR spectrometer, ASTM test kit (Petroleum Technology Lab, FT UGM).

Procedure

a. Catalyst preparation
Chicken egg shells are washed with water until clean. Furthermore, the catalyst base material is dried in an oven at 100oC for 24 hours. As much as 1 kg of catalyst base material is then crushed using a mortar until smooth and sieved to a size of 100 mesh. After that, 2 g of eggshell solids were characterized using an X-Ray Diffractometer (XRD).

b. Synthesis of CaO from eggshells
Sifted chicken eggshell solids were then weighed as much as 500 g. The conversion of CaO was carried out by calcination of solid egg shells at 1000oC for 2 hours. After that, 2 g of calcified CaO was characterized using an X-Ray Diffractometer (XRD).

c. Waste cooking oil preparation
As much as 2 L of waste cooking oil is heated at 120^{o}C to evaporate the water in the oil. After that, the oil was filtered using Whatman 40 filter paper to separate large solid impurities. The filtered oil was then analyzed by infrared spectroscopy (FTIR) and 1H-NMR.

Production of biodiesel

Esterification process

150 g of used oil that has been heated and cleaned is put into a three-neck flask. The mole ratio of oil to methanol is 1: 9, assuming the molecular weight of used oil is 860 g/mol. Then added H2SO4 1.25% of the total amount of oil and methanol then refluxed at 60 - 65 oC for 2 hours. Two layers will be produced, namely a mixture of methanol and methyl ester on the top and triglycerides on the bottom. Then the two layers are separated.

Transesterification process

The triglycerides that had been separated in the esterification process were then transesterified with methanol (with a mole ratio of 1:12) and added with an alkaline catalyst CaO from egg shells with a weight variation of 11% of the total oil and methanol. Then the mixture was refluxed again at a temperature of 60 - 65 oC for 5 hours. The reaction mixture is cooled and 2 layers are formed, namely methyl ester (biodiesel) on the top layer and glycerol on the bottom layer. The two layers were then separated from the CaO catalyst, and then evaporated to remove residual methanol. The methyl ester was then washed with distilled water in a separatory funnel to dissolve the remaining glycerol. The final step was the addition of 1.5 g of anhydrous Na2SO4 to bind the remaining water, then filtered using Whatman 40 filter paper. The methyl ester obtained was analyzed for its chemical composition by GC-MS, IR and 1H-NMR, then tested for its physical characteristics by ASTM standard method (density, viscosity, flash point, pour point, fog point, codranson carbon residue).

RESULTS AND DISCUSSION

Synthesis and characterization of CaO from broiler egg shells

Synthesis of the CaO catalyst begins with cleaning and drying in an oven at 100°C for 24 hours to remove impurities and water, ground in a mortar until smooth and sieving until the size does not exceed 100 mesh. This screening process aims to enlarge the solid surface. The eggshell solids were calcined at 1000°C for 2 hours to synthesize CaO. The high-temperature heating process can remove CO2 from CaCO3 so that CaO can be obtained. The conversion of CaO obtained from the calcination of 50 g of eggshell is 54.942%.

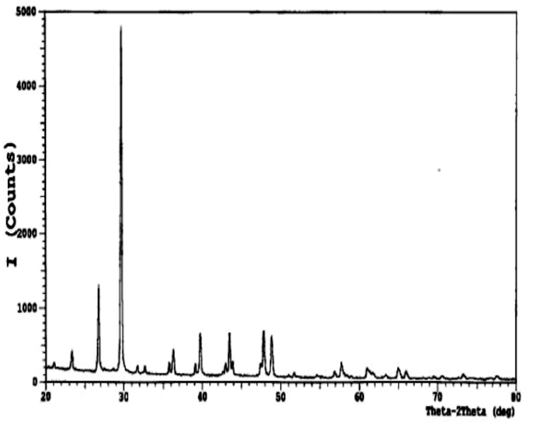

Figure 1. Diffractogram before calcination

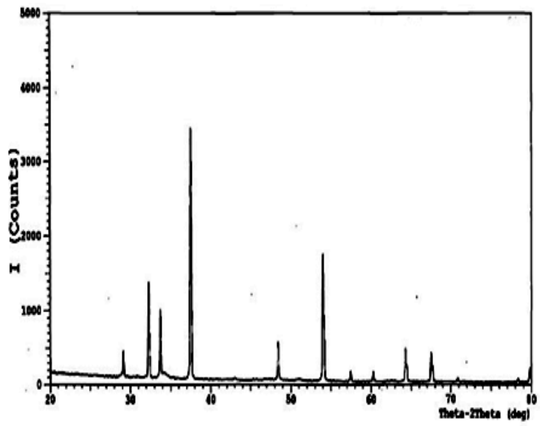

Figure 2. Diffractogram before calcination

XRD results on purebred chicken egg shells before calcining was shown by the diffractogram (Figure 1) which produced 2θ peaks with CaCO3 characteristics. Of the 16 visible peaks, there are 3 strongest peaks namely at 2ϴ = 29.6767 with an intensity of 3465, 2ϴ = 26.7654 with an intensity of 850, and at 2ϴ = 47, 8133 with an intensity of 507. Diffractogram of XRD results on purebred chicken eggshells that have been calcined at 1000 ºC (Figure 2) produces a change in the diffraction pattern. From the 13 visible peaks, there are 3 strongest peaks, namely at 2ϴ = 37.4728 with an intensity of 2897, 2ϴ = 53.9871 with an intensity of 1627, and at 2ϴ = 32.3139 with an intensity of 1061. XRD results of purebred chicken egg shells calcined showed almost the same diffraction pattern with pure CaO.

Biodiesel synthesis and characterization

The esterification reaction needs to be carried out first to reduce the free fatty acid content in waste cooking oil so that the biodiesel produced at the transesterification stage can be more optimal. The transesterification process was carried out using a CaO heterogeneous base catalyst obtained from calcined broiler egg shells. Experimentally, the conversion of methyl ester (biodiesel) was 36.779%. FT-IR testing was carried out to prove the presence of esters in the transesterification product. The presence of esters can be seen from the typical absorption in the C=O and C–O groups. Table 1. indicates a change in absorption at wave numbers 1165 cm-1 to 1172.72 cm-1 and 1180.46 cm-1. This indicates a change in one ester group to another ester form. So it can be proven that the transesterification process has been successfully carried out.

Table 1. Spektra FTIR biodiesel

jelantah oil	Biodiesel	explanation
3471.87	3464.15	-OH
2924.09 – 2854.65	2924.09 – 2854.65	$-C_{sp3}H-$
1743.65	1743.65	C=O (ester)
1458.18	1458.18	$-CH_2-$
1373.32	1365.60	$-CH_3$
1165	1172.72	C-O-C (ester)
725.23	725.23	$-C_{sp2}H-$

Then proceed with 1H-NMR spectroscopic analysis to determine the percentage of biodiesel conversion. The theoretical conversion of methyl ester (biodiesel) from waste cooking oil with CaO catalyst from egg shells and methanol solvent is 40.298%. Analysis of the physical properties of biodiesel was carried out using the ASTM (The American Society for Testing and Materials) method which was then compared with SNI standard diesel oil and biodiesel. The specific density of biodiesel from waste cooking oil with a CaO catalyst from purebred chicken eggshells and methanol solvent is 0.8780 (at 60/60 ^{o}F, kg/m^{3}) according to SNI biodiesel specifications. The kinematic viscosity value of biodiesel is also included in the SNI biodiesel standard, namely 5,000 (at 40 ^{o}C,

mm^2/s). The resulting flame test is 170.5^oC which is also in following the specifications of SNI biodiesel and has a higher value when compared to the limit of diesel oil. The resulting pour point is 12^o C which is also following the specifications of SNI biodiesel.

CONCLUSION

From the research results obtained, it can be concluded that:

1. Through the calcination process of purebred chicken eggshells at a temperature of 1000 ^oC, CaO of 54.942% is obtained. And the manufacture of biodiesel from waste cooking oil through esterification and transesterification processes using a CaO catalyst produces a conversion of 40.298%.
2. Characterization of biodiesel produced from waste cooking oil using a catalyst from purebred chicken egg shells has the qualifications as diesel fuel and approaches the characteristics of diesel oil according to ASTM.

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