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Microwave Assisted Trans-esterification of Cotton Seed Oil for Biodiesel Production

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Abstract

Biodiesel is a renewable, non-toxic and environmentally friendly fuel made from vegetable oils through a trans-esterification reaction with methanol. Nowadays, the manufacture of biodiesel usually takes a long time, which increases the manufacturing cost. The use of microwave can decrease the reaction time and the amount of catalyst. The purpose of this study was to study the utilization of microwave as a heat source in the trans-esterification of cotton seed oil with the addition of NaOH catalyst 0.25, 0.5, 0.75 and 1% (w/w) for 15 minutes. The trans-esterification of cotton seed oil with NaOH catalyst concentrations 0.5% (w/w), 5 minutes, molar ratio of 1: 12 with a microwave power of 400 watts achieved 99.11% conversion. The biodiesel obtained in this research has met the Indonesian National Standard for biodiesel (SNI-04-7182-2006).

Keywords: *biodiesel; cotton seed oil; microwave; transesterification*

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INTRODUCTION

Energy is one of the basic needs of human life for household, social and industrial activities. Along with the rapid growth of population and the challenges in economic, national and regional development, the need for energy fulfilment across the nation increases. The results of a study by the Ministry of Energy and Mineral Resources revealed that the Indonesia's annual energy consumption grows higher than 5% (Indonesian Energy Statistic, 2010). However, the current energy supply is still dominated by fossil fuels, whereas the crude oil reserves are continuously running out. Based on the aforementioned facts, the development of alternative fuels becomes urgent to overcome the possibility of future energy crisis. One of the alternatives is by producing fuels from vegetable and animal oils (Bhatti *et al.*, 2008). Generally, vegetable and animal oils contain triglycerides. Triglycerides containing low levels of free fatty acids can directly be processed into fuel by trans-esterification reaction using a base catalyst. On the other hand, if the triglycerides contain high level of free fatty acids and water contents, the trans-esterification can be catalyzed by acid catalysts (Maa and Hanna, 1999). The use of low concentration of catalyst in the trans-esterification reaction can be carried out by the assistance of microwave heating (Suryanto *et al.*, 2015; Yakub. Z, *et al.*, 2008). Biodiesel is a well-known alternative fuel comprising of methyl ester produced from vegetable oils, such as palm oil, cotton seed oil and other grain oils. Currently, there are two crop commodities developed to ensure the sustainability of the supply of biodiesel raw materials, namely Crude Palm Oil (CPO) and castor oil or Crude Jatropha Curcas Oil (CJCO). However, the use of CPO as a biodiesel raw material will certainly disturb its supply for export and uses in food and nutraceutical productions, such as cooking oil, margarine, food supplements, etc. A sustainable large supply of cotton seed oil has increased its potential as a raw material for biodiesel production (Norazahar *et al.*, 2012).

The development of processes for the production of alternative fuels, especially in regard to the type of energy sources used is highly essential. One of the promising energy source is the microwave that can accelerate the reaction rate (Gude *et al.*, 2013). It has been reported that heating with microwave can reduce the reaction time (Suryanto *et al.*, 2015). This study aims to study the variation of microwave power, catalyst concentration and reaction time on the yield of biodiesel.

MATERIALS AND METHOD

Materials

Cotton seed oil used in this research was obtained from chemical store PT. Sumber Rejeki Makassar. Methanol with 96% purity and sodium hydroxide (NaOH) with a purity of 99% were obtained from Intraco Chemical Makassar.

Method

The main apparatus used in this research was a microwave oven brand Electrolux type EMM2007X having a frequency of 2.45 GH_Z with the power ranged from 100-800 Watt. A two-necked flask reactor of Pyrex glass ware equipped with a magnetic stirrer and thermocouple. The experimental set-up can be seen in Figure 1.

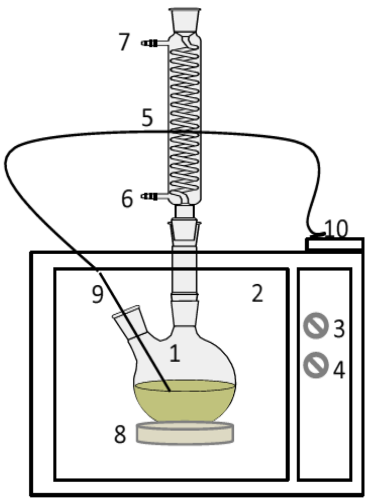

Figure 1. The experimental set-up of batch microwave trans-esterification process: 1. reactor, 2. microwave, 3. power control, 4. temperature control, 5. condenser, 6. inlet water, 7. outlet water, 8. magnetic stirrer, 9. thermocouple, 10. temperature indicator

A carefully measured 25 ml of cotton seed oil was introduced into a two-necked flask reactor. Then, a predetermined mixture of methanol and NaOH solution as the homogeneous catalyst was added to the reactor. The mixture was then heated and let to react under microwave irradiation generated at various power values (100, 264, 400, and 600 watts). The reaction was lasted for 0.5-30 minutes. The biodiesel obtained was separated from glycerol as by product and residual castor oil. Prior to analyses, the biodiesel was washed with distilled water many times to remove the catalyst to obtain alkali free biodiesel. The analyses include: moisture content, density, viscosity, flash point, pour point and phosphorus content. Calculation of biodiesel yield with equation

$$Yield = \frac{mass\ of\ product}{mass\ of\ feed} \times 100\%$$

RESULTS AND DISCUSSION

Characteristics of Cotton Seed Oil

Cotton seed oil was characterized for its physical and chemical properties. Cotton seed oil contains 3.182 free fatty acid. This condition suggested that cotton seed oil cannot be processed into biodiesel through one stage method. Therefore, the oil should be processed in two stages: esterification and followed by trans-esterification (Suryanto *et al.*, 2015). Cotton seeds oil have a viscosity of 27.6 mm/s^2 and a specific gravity of 0.914. The gas chromatography analysis revealed that cotton seed oil contains saturated and unsaturated fatty acids. This showed that the cotton seed oil is suitable to be used as a raw material for biodiesel production in regard to its thermo-stable fatty acid and its triglyceride structure is not easily damaged.

The Effect of Microwave Power on the biodiesel Yield

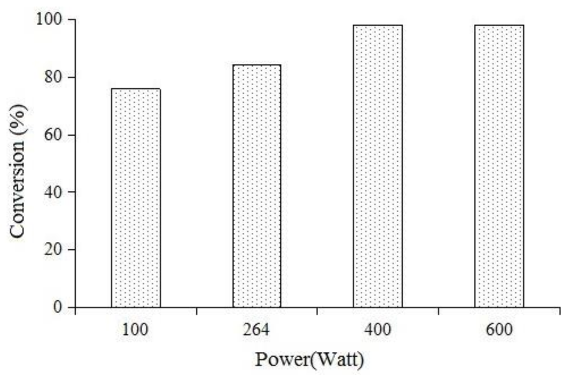

Figure 2. Effect of microwave power on the biodiesel yield of biodiesel (NaOH catalyst 0.5% (w/w) and 30 minutes)

Microwave power directly affects the amplitude of the wave, where a greater microwave power supplied to the microwave generator will generate a higher electric field generated (Manco *et al.*, 2011). The rotational speed of the polar molecule has a linear relation to the microwave amplitude. Figure 2 shows the effect of microwave power on the biodiesel yield for trans-esterification of castor oil using 0.5% of NaOH catalyst concentration for 30 minutes.

Figure 2 shows that the biodiesel yield increased as the applied microwave power increased. This result is in good agreement with previous reports, where the increasing microwave power in the microwave was found to be directly proportional to the yield of biodiesel (Demirbas, 2010; Azcan and Danisman, 2007; Marchetti *et al.*, 2007; Evangelista *et al.*, 2012**;** Basumatary, 2013).

Effect of NaOH Concentration on Biodiesel Yield

The use of catalysts in the trans-esterification greatly affects the chemical rate constant and reaction rate because it can directly decrease the activation energy (Levenspiel 1999). Figure 3 shows the effect of NaOH concentrations (0.25; 0.5; 0.75; and 1 (% w/w)) on the yield of biodiesel when the trans-esterification occurs for 5 minutes using 400 watts of power. Figure 3 shows that the biodiesel yield increased significantly as the catalyst concentration increased from 0.25 to 0.5% (w/w). However, when the catalyst concentration was further increased the biodiesel yield decreased. This decrease was likely due to excessive catalyst usage, which lead to the formation emulsion as a result of saponification of castor oil by NaOH solution. Under this situation, a number of methyl esters that have been formed as well as other methyl esters which may be trapped in the emulsion (Azcan and Danisman, 2007; Atadashi *et al.*, 2013; Basumatary, 2013, Suryanto *et al.*, 2015). In accordance with Arrhenius law, the decrease of activation energy causes the rise of the reaction speed constant.

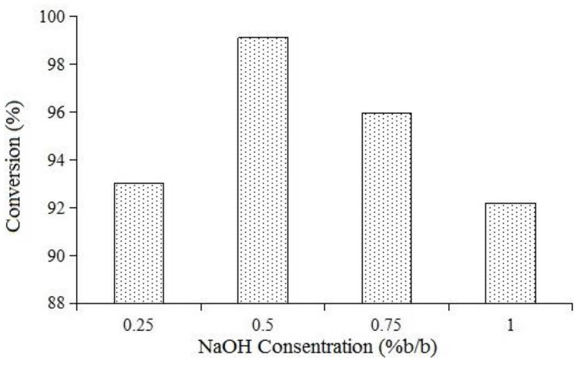

Figure 3. Effect of NaOH catalyst concentration and biodiesel yield (400 watts and 5 minutes)

Effect of Reaction Time on Biodiesel Yield

Conventional biodiesel production may achieve highest yield in 1 hour when 1% homogeneous catalyst was used. Yacob *et al.* (2009) reported a trans-esterification reaction using homogeneous catalyst under microwave irradiation. The highest yield (99%) was achieved at 7 minutes reaction time. Figure 4 shows the effect of reaction time on the yield of biodiesel when 0.5 % (w/w) NaOH was used as catalyst using 400 watts of microwave power.

Figure 4 shows that the yield of biodiesel achieved 65.4% when the reaction occurred in just 1 minute. Prolong the reaction time increases the biodiesel yield to some extent and reaches a constant value at about 99% after 5 minutes. No further increase in biodiesel yield was observed as the reaction time was longer than 5 minutes. Therefore, a reaction time of 5 minutes was selected as the most economical reaction time to produce high yield of biodiesel. It is proven that the use of microwave energy can shorten the reaction time. The microwave effects on the trans-esterification reactions could occur in two ways: 1) increase the reaction rate constant, and 2) increase methanol - microwave interaction due to high dipole moment value (Chen *et al.*, 2012).

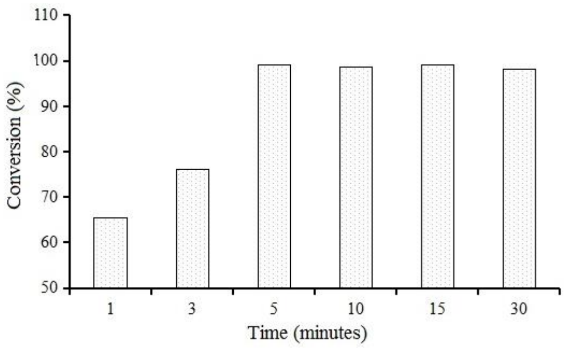

Figure 4. Effect of reaction time on the yield of the at NaOH 0.5% (w/w) concentration at 400 watt

Biodiesel Product Characteristics

Table 1 shows the characteristics of biodiesel obtained in this research. It is clear that the biodiesel has met the quality parameters set by the SNI 7128: 2012.

Table 1. Biodiesel characteristic

Parameter	Product	SNI 7128:2012	Test Method
Density at 40º C (g/cm^3)	0.887	0.850-0.890	ASTM D-1298
Kinematic viscosity at 40˚C (cSt)	2.89	2.3-6.0	ASTM D-7279
Flash point (ºC)	110	≥100	ASTM D-93
Pour point (ºC)	5	≤18	ASTM D-97
Water in% volume (mg/kg)	286	≤500	Karlfisher Colorimetric
Phosphorus (mg/kg)	0.97	≤10	X-Ray

CONCLUSION

The use of microwave energy on the trans-esterification of cotton seed oil using methanol decreased both the amount of catalyst and reaction time. The highest biodiesel yield obtained from microwave assisted cotton seed oil trans-esterification process using 400 watt power, 0.5% w/w NaOH catalyst for 5 minutes was 99%. The characteristics of the biodiesel have met the quality standards for biodiesel according to SNI 7128: 2012.

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