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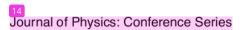
**Submission date:** 08-May-2023 03:04PM (UTC+0700)

**Submission ID:** 2087353133

File name: 11\_Mirzayanti\_2021\_J.\_Phys.\_\_Conf.\_Ser.\_1833\_012041.pdf (674.83K)

Word count: 3139

Character count: 16580



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To cite this article: Y W Mirzayanti et al 2021 J. Phys.: Conf. Ser. 1833 012041

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1833 (2021) 012041 doi:10.1088/1742-6596/1833/1/012041

#### Transesterification of jatropha curcas oil for biodiesel production Over NiO catalyst

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Abstract. The research paper deals with Jatropha curcas oil's transesterification process using the NiO catalyst as a heterogeneous catalyst to produce Biodiesel. The batch reactor in this process. Excess methanol is needed to shift the equilibrium towards the product and enlarge Biodiesel. The research study about the effect mole ratio of methanol: Jatropha curcas oil. The ratios are 12:1, 15:1, 18:1, and temperature of the reaction of 60, 65, 70°C. Experiment verification of the predicted yield and characterization of Biodiesel produced. The analysis biodiesel product by Chemical characterization using Gas Chromatography (G-C) analysis and ASTM 6751 standard used for the physicochemical properties. The maximum process conditions for the biodiesel yield by transesterification of Jatropha curcas oil over NiO catalyst 59.8% and the mole ratio of 15:1 methanol: Jatropha curcas oil. The 65°C reaction temperature used in this process enhances the transesterification reaction by observing 5 hours with a high yield of Biodiesel.

#### 1. Introduction

Biodiesel is a fuel that is processal from the esterification of fatty acids free or naturally available triglyceride transesterification is contained in renewable natural resources such as plant oils and fats. Biodiesel has attracted many researchers to study its synthesis with a wide variety of feedstock sources. Several types of oil that work converted into biodiesel, including soybean seeds, rapeseed, sunflower seeds, oil palm, nuts, camelina, canola, cotton, pumpkin, castor seeds, and fat [1]. Search for petroleum-based renewable fuel alternatives in concern with sustainable energy and clean environment develops different alternative technologies 12e fuel cell, electric power, hydrogen, and biomass-based fuels. Focus of renewable Fuel begins with the direct use of vegetable oils as an alternative to petroleum diesel in the diesel engine. Using vegetable oil as a fuel for diesel engines is an exsting technique from the engine development era [1]. Generally, plant oil or animal fat is an ester of saturated and unsaturated monocarboxylic acids with trihydric alcohol glycerides. These esters

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**1833** (2021) 012041

doi:10.1088/1742-6596/1833/1/012041

are called triglycerides, which can react with alcohol in the presence of a catalyst, a process known as transesterification.

In general, catalysts can be divided into two types, namely homogeneous catalysts and heterogeneous catalysts. Homogeneous catalysts are catalysts to have the same phase as the reactants, including transition metal compounds and acid-base catalysts. Homogeneous catalysts generally react with one or more reactants to form a chemical intermediate which then reacts to form the final product of the reaction, in a process that restores the catalyst [2][3]. The weakness of this homogeneous catalyst is the difficulty of separating from the product so that the equipment needed for the separation process is more and more costly production will be higher. The process carried out with this catalyst is not environmentally friendly because of the large amount of waste from the separation process and is corrosive. Soap formation through a saponification reaction occurs with a primary homogeneous catalyst. Heterogeneous catalysts are a more profitable alternative for methyl ester synthesis reactions because heterogeneous catalysts are in phase with the reactants and product compounds so that they are easy separated from products, can be reused, and non-toxic and environmentally friendly [4].

This study aimed to develop biodiesel production process. *Jatropha curcas* oil 11 s used through a transesterification process using a NiO catalyst as a heterogeneous catalyst, which was carried out in a batch reactor. The molar ratio between methanol - *Jatropha curcas* oil, reaction time, reaction temperature, fatty acid content, and water in the oil are factors that influence the transesterification reaction. In the 20 nsesterification process, methanol is more widely used because it is cheaper than other alcohols. Excess methanol is needed to shift the equilibrium towards the product, and enlarge biodiesel.

#### 2. Experimental methods

The feed 21 ck oil used in this experiment was extracted from *Jatropha curcas* oil. *Jatropha curcas* oil content unsaturated hydroxyl fatty acid, cis 9,12 hydroxy octadecanoic acid, which is commonly called risionoleic (ricinoleic acid), 89.5%. Methanol 99.8%, n-Hexane 98% from Merck, and KOH were bought from Sigma Aldrich and of analytical grade. This experiment used mole ratio methanol: *Jatropha oil* 12:1, 15:1, 18:1. Variation of the temperature of reaction used 60, 65, 70°C.

During the experiment, put methanol with a concentration mole ratio of 12:1 to *Jatropha curcas* oil into a three-neck flask, then add the 3% NiO catalyst between methanol with *Jatropha curcas* oil and stir until homogeneous. Next, add the *Jatropha curcas* oil slowly in a three-neck flask, which already contains methanol and catalyst. Then, refluxed for 5 hours 60°C accompanied by stirring and cooling. The reflux product is filtered, and the filtrate is put 60°C accompanied by stirring and cooling. The reflux product is filtered, and the filtrate is put 60°C accompanied by stirring and cooling. The reflux product is filtered, and the filtrate is put 60°C accompanied by stirring and allowed to stand for 30 minutes until 2 phases are formed. The methyl ester phase is washed with warm distilled water where the temperature is around 50°C until the washing solution is neutral, then let it stand then poured so that the glycerol separated with the methyl ester phase. The same treatment for variations in the mole ratio of methanol and castor oil 15:1, and 18:1, and temperature of the reaction. Pure methyl esters 7e analyzed for their characteristics. Chemical characterization using Gas Chromatography (GC). The physicochemical properties of the Biodiesel were characterized according to ASTM 6751 standard.

#### 3. Results and Discussion

This research was conducted in several steps, i.e., the transesterification process and product analysis. The catalyst used a 3% NiO catalyst be 32 een methanol with *Jatropha curcas* oil NiO. The methyl ester is produced from the reaction of *Jatropha curcas* oil with methanol, which calculates concentration using Gas Chromatography (GC) so that the desired yield can be obtained. The result of the reaction of maki 22 methyl ester forms three separate phases, where the top layer is the remaining unreacted methanol, the middle layer is methyl ester, and the bottom layer is the catalyst, as shown in

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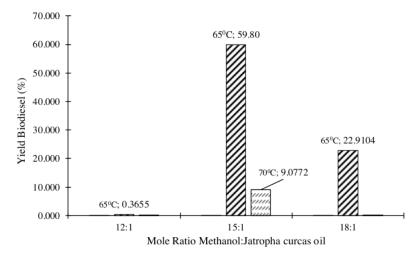
Figure 1. The methyl ester obtained from the transesterification reaction separated from the catalyst and the remaining reactants, then the methyl ester is GC tested and its characteristics.



Figure 1. Phase separation results from methyl ester synthesis reactions

3.1. The Effect of Comparison of Jatropha curcas Oil Moles ratio with Methanol on Heterogeneous Transeste 26 cation Process on Methyl Ester Yield based on Reaction Temperature.

Based on the effect of variations in the methanol: Jatropha curcas oil mole ratio in the heterogeneous transesterification process, the highest yield was 59.80%. Based on these results, it explains that the mole ratio of methanol to Jatropha curcas oil has a large effect on the formation of methyl ester yield by taking the basis of the number of moles of castor oil is 1 mol, so the following Figur 2 is obtained:



**Figure 2.** The relationship between methanol-Jatropha curcas oil molar ratio based on variations in reaction temperature

In this study, the use of 12:1 mole between methanol and castor oil resulted in a yiel 10 f 0.07%, incomplete conversion of methyl ester was due to inadequate methanol concentration to shift the equilibrium reaction to shift the formation of methyl ester products. The most significant yield is obtained using a 15:1 mole ratio of methanol to castor oil, which is 59.80%. The ratio of alcohol to

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fatty acids is 15:1, according to previous the report, which states that this ratio is the optimum molar ratio of methyl ester synthesis with heterogeneous acid catalysts [5]. From this result, it can be predicted that the magnitude of the mole ratio used in the heterogeneous transesterification reaction is caused by the collision. The collision of methanol against NiO is used and reacts with the triglycerides in the sample so that it requires a lot of methanol to react optimally. Meanwhile, the use of an 18:1 mole ratio between methanol and castor oil resulted in a yield of 9,077%, the conversion of methyl ester was not maximum and decreased due to the use of a large enough mole ratio so that the catalyst content decreased in line with the increase in methanol content. Besides that, alcoholis a particle that is sensitive to water [6]. In a previous study by [8] Kim et al. (2013) regarding the hydrotreatment of soybean oil to produce remwable diesel fuel using a Ni catalyst, it shows that the Ni catalyst can produce liquid products  $n-C_{17}/(n-C_{17} + n-C_{18}) = 0.93-0.96$ , which indicates that most of the triglycerides are deoxygenated by decarboxylation and/or decarbonylation. When Ni catalyst is used, both the conversion value and the diesel yield increase with the increase in reaction temperature with a maximum temperature of 400°C, mainly due to endothermic decarboxylation and decarbonylation reactions [9]. The high decarboxylation and decarbonylation values in using a Ni catalyst indicate that the C - C bond is cut. Catalysts containing transition metals as active materials such as Ni, Pd, and Pt show catalytic advity on cutting C - C bonds [9].

Meanwhile, the presence of water and free fatty acids inhibits the transesterification reaction, which will decrease the conversion of the methyl ester product. The maximum mole ratio obtained through this study is 15:1 with a conversion result 19 59.80%. The yield of 59.80% is far from what we expected, previous research by [8] used a nickel catalyst for transesterification of soybean oil with a maximum methyl ester conversion of 87% at 65°C. The magnitude of the effect of the reaction temperature on the heterogeneous transesterification process, according to the yield is 59.80%. From this percentage, it can be explained that temperature has an influence on the formation of methyl ester yield. At 60°C, the resulting yield is low, namely 0.3655%. This is because met and does not mix homogeneously with Jatropha curcas oil because, at this temperature, it is less than the boiling point of methanol so that the reaction does not run correctly. In this study, the most significant yield was obtained using a temperature 65°C reaction, which is 59.80%. At this temperature, it is a very suitable temperature for the reagents to mix homogeneously because methanol boils at 65°C. There is a deviation for the transesterification reaction at 70°C, obtained a methyl ester conversion of 22.91%. In this case, the temperature is too large, so that the methanol used evaporates.

#### 3.2. The physicochemical properties of the Biodiesel at 65°C

The comparison of density with Molar Methanol – Jatropha curcas oil (12:1, 15:1, 18:1) at 65°C for 5 hours. The quality standard of methyl ester according to SNI 04-7182-2006 stipulates that the density of methyl ester at 40°C is in the range 0.85 – 0.89 g.cm<sup>-3</sup>. The method ester density produced by heterogeneous transesterification in this experiment has an average 15 he of 0.8828 g.cm<sup>-3</sup>. The density of Biodiesel obtained through heterogeneous transesterification has a value of 0.8828 g.cm<sup>-3</sup> and has a viscosity of 6.431 mm<sup>2</sup>.s<sup>-1</sup> (cSt) according to comparison standard. Meanwhile, the acid number is 0.563 mgKOH.g<sup>-1</sup>, so it can be concluded that the quality of the Biodiesel obtained meets the standard limit. The quality standard of methyl ester, according to SNI 04-7182-2006 stipulates that the density of methyl ester at 40°C ranges from 0.85 to 0.89 g.cm<sup>-3</sup>.

In addition to other physical properties, namely viscosity. It can be explained that the optimum viscosity onditions in this experiment are using a 15:1 mole ratio with a value of 15.45 cSt. The lower the mole ratio of methanol: *Jatropha curcas* oil, the greater the resulting viscosity as in the mole ratio of 12:1 with a viscosity of 23.03 cSt, but the lower viscosity is also produced at a mole ratio of 19.87 due to the resulting yield. The comparison is also of little value. Kinematic viscosity is one of the main parameters in determining the quality of methyl ester because it has a parameter on the effectiveness of methyl estermining the quality of methyl ester because it has a parameter on the effectiveness of methyl estermining the direct use of vegetable oil has a viscosity far above the viscosity of diesel fuel. This is an obstacle to the direct use of vegetable oil as fuel. The kinematic viscosity of the methyl ester produced in this experiment averaged 6.437 cSt, which is much higher than the quality standard

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of methyl ester, according to SNI 14-7182-2006 which only ranged from 2.3 to 6.0 cSt at Table 1. The free fatty acid is a measure used to determine the properties of the resulting methyl ester, which is the higher the free fatty acid content, the resulting in the formation of ash during biodiesel combustion. The free fatty acid produced in this study is 0.563 mgKOH.g<sup>-1</sup>, and does not exceed the quality standard of SNI 14-7182-2006 methyl ester, which has a maximum limit of 0.8 mgKOH.g<sup>-1</sup>.

**Table 1.** Comparison of the Physical Properties of Methyl Ester with SNI 14-71822006 and ASTM D6751

Parameter	Units	Dogulta	SNI	ASTM
Parameter	Units	Results	04-7182-2006	D6751
Density	g.cm <sup>-3</sup>	0.8828	0.85 - 0.9	0.85 - 0.89
Viscosity	mm <sup>2</sup> .s <sup>-1</sup> (cSt)	6.431	2.3 - 6.0	1.9 - 6.0
Acid Number	mgKOH.g <sup>-1</sup>	0.563	Max. 0.8	Max. 0.8

#### 4. Conclusion

The results showed that the yield increased with the increase in the mole ratio of methanol: Jatrajia curcas oil, but the yield decreased when the mole ratio of methanol: castor oil was too large. The results showed that the yield increased along with an 25 rease in temperature, but the yield will decrease if the temperature is too large and exceeds the boiling point of methanol (65°C). The maximum condition for the most abundant biodiesel yield is obtained in the mole ratio of methanol to castor oil of 15:1 (mol/mol) at 6 °C for 5 hours was 59.80%. The results showed that the physical properties for density, viscosity, and acid number were in accordance with the quality standards of SNI 14-7182-2006 methyl ester.

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