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The use of MgO/SiO_2 as catalyst for transesterification of rubber seed oil with different alcohols

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Abstract. In principle, biodiesel production relies on the transesterification reaction of fatty acids contained in vegetable oil or waste rich in fatty acids with short-chain alcohols with the help of a catalyst. The purpose of this study was to obtain information about the most suitable alcohol for the transesterification of rubber seed oil into biodiesel. In this study, the transesterification of rubber seed oil was carried out with three different types of short-chain alcohols, namely methanol, ethanol, and 2-propanol. Each alcohol was used with a ratio of 3:1 to oil and transesterification was carried out in the presence of MgO/SiO_2 catalyst with an amount of 10% (by weight of catalyst/volume oil) at 70 °C for 6 h. Transesterification products were analyzed using Gas chromatography-mass spectrometry (GC-MS) analysis to confirm the conversion of fatty acids in the oil into esters. The results showed that the reactivity of alcohols is in the order of methanol > ethanol > 2-propanol with the percentages of conversion of oil to products are 90.1, 73.3, and 63.2%, respectively. These results indicate that methanol is the most suitable alcohol for transesterification of rubber seed oil.

Keyword: MgO/SiO_2 catalyst, transesterification, rubber seed oil, biodiesel, alcohols

1. Introduction

Awareness of energy issues and environmental problems associated with the combustion of fossil fuels has prompted many researchers to consider using alternative energy sources that do not come from petroleum and its derivatives. Many renewable energy sources have attracted the attention of researchers to develop, one of which is biodiesel [1] because it can be produced through a simple chemical process, is more environmentally friendly, biodegradable, non-toxic [2], and has low viscosity [3].

In principle, biodiesel production involves transesterification reaction of the triglycerides with alcohol. Triglycerides are a blend of fatty acids, typically from biological sources, including vegetable oils, animal fats, or even waste oils. Superior foodstuffs in biodiesel are edible oils such as coconut, palm, maize, and soy. However, the move to non-edible oils as alternative raw materials is currently occurring. The advantages of using non-edible oils are the availability and avoid competition between food and energy needs [3,4]. Many plants are known to contain non-edible oil which have the potential as raw materials. Among them are *Jatropha curcas*, *Madhuca indica*, *Hevea brasiliensis*, *Terminalia catappal*, *Moringa olifera*, *Pongamia pinnata*, and *Calophyllum inophyllum* [1,4]. A major role in competitive biodiesel development is the selection of oil feeds and alcohol forms used in the transesterification reaction. A number of short-chain alcohols have been investigated for biodiesel processing, such as methanoli, ethanol, propanol, iso-propanol, n-butanol, tert-butanol, and cyclohexanol [5-7].

Chemically, the transesterification reaction of triglycerides with alcohol generally runs slowly because the two reactants are not reactive, therefore a catalyst is needed to trigger the reaction to produce biodiesel. Selection of the right catalyst will affect the transesterification process, the type and concentration of the catalyst correlate with the yield as reported [8-10]. The limitations of using homogeneous catalysts are the difficulty of separating the catalyst from the reaction mixture, the occurrence of saponification, corrosion, and impossibility to reuse catalyst [4,11], have resulted in high energy consumption and high cost of biodiesel production technology [12,13]. Therefore currently, research is being carried out in the direction of finding a better catalyst, one of which is the use of a heterogeneous catalyst. Many chemical solids are used as heterogeneous catalysts for biodiesel production, including metal oxides [14-16], zeolites [17-18], mixed metal oxides [14,19], and metal oxides with supports [8,10,20].

The suitability of the raw material, namely edible oil and the form of alcohol used, is another aspect that affects the performance of the catalytic transesterification reaction. The overall kinetics of the reaction depends on the rates of the conversion of triglycerides to diglycerides, monoglycerides and alcohol esters. Rubber-seed oil, since it is plentiful particularly in tropical countries, has an oil content of 40 – 50 percent and is a feedstock for biodiesel production. A further analysis is carried out to determine the suitability of the rubber seed oil reactor to the form of alcohol used in the transesterification reaction. The study investigated the efficacy of rubber seed oil transesterification products with MgO/SiO_2 catalyst in the effects of alcohol forms, namely methanol, ethanol, and propanol. A GC-MS analysis has been performed to classify compounds in the transesterification product.

2. Material and methods

2.1. Materials
The chemicals in this research, methanol, ethanol, and 2-propanol available from Merck, $Mg(NO_3)_2.6H_2O$, and NaOH from Aldrich, silica extracted from rice husk, and rubber seed oil. GCMS-QP2010 SE SHIMADZU type is used for description transesterification products.

2.2. Preparation of MgO/SiO_2 catalyst
Preparation of catalysts MgO/SiO_2 was conducted by the sol gel method. Rice husk silica was dissolved in 10% NaOH solution and then transferred into an Erlenmeyer flask placed in an ultrasound agitator. A specified volume (50 mL) of 1.0 N $Mg(NO_3)_2$ solution with MgO content of 20 wt%, put into the silica sol slowly and under ultrasound agitation, followed by adding 10% HNO_3 solution by drop wise until the sol was completely formed into a gel. The gel was aged for 3 days and finally, oven-dried at 110 °C for 8 h, followed by crushing into powder and then calcined at 800 °C for 6 h.

2.3. Process of transesterification
For all transesterification reactions, the respective ratio of alcohol (methanol, ethanol, and 2-propanol) to oil was adjusted to 3.1, and MgO/SiO_2 catalyst 10 wt.% respect to oil. The mixture was put in a two neck bottle fitted with a reflux condenser and a thermometer, with a reaction at 70 °C for a period of 6 hours. The sample was cooled to a room temperature. It was moved for 6 hours to a separate funnel to remove the catalyst and the mixture was separated into two layers. Excess alcohol was eliminated by evaporation and biodiesel volumes were quantitated in order to measure percent conversion. Transesterification products were interpreted by the GC-MS instrument to classify the types of fatty acid esters.

3. Results and discussion

3.1. Methanol

GC chromatogram of the transesterification product of rubber seed oil with methanol using MgO/SiO_2 catalyst was presented in Figure 1.

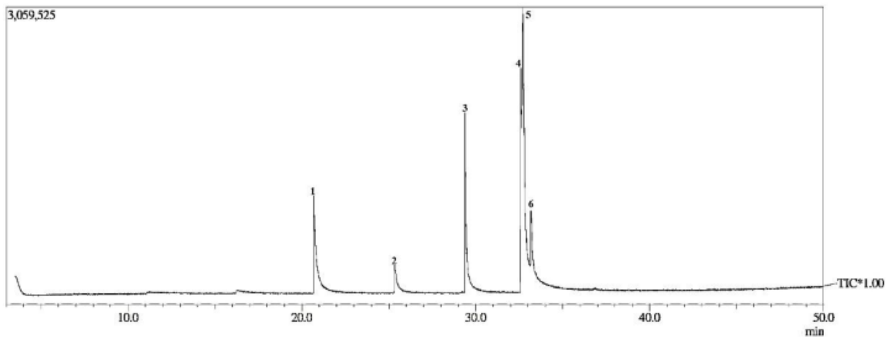

Figure 1. Chromatogram of rubber seed oil transesterification product with methanol.

Figure 1 explains that there are six types of compounds contained in the transesterification product of rubber seed oil with methanol which is characterized by the appearance of six peaks at different and significantly separated retention times. The types of compounds in the transesterification product were investigated with the support of the Library System WILEY 299 LIB and NIST 62 LIB database and the results are tabulated in Table 1.

Table 1. The components transesterification product of rubber seed oil and methanol.

Peak number	Retention time (min)	Molecular formula	Compound name	Relative percentage (%)
1	20.69	$C_{13}H_{26}O_2$	Methyl laurate	11.29
2	25.33	$C_{15}H_{30}O_2$	Methyl myristate	3.32
3	29.38	$C_{17}H_{34}O_2$	Methyl palmitate	15.66
4	32.60	$C_{19}H_{34}O_2$	Methyl linoleate	15.81
5	32.71	$C_{19}H_{36}O_2$	Methyl oleate	47.81
6	33.18	$C_{19}H_{38}O_2$	Methyl stearate	6.11

Table 1 data shows that the fatty acids contained in oil have reacted with methanol and produced fatty acid methyl esters (FAMEs) with the main component is methyl oleate, this ester corresponds to oleic acid as the main fatty acid in rubber seed oil [9,21].

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3.2. Ethanol

The chromatogram product of transesterification of rubber seed oil with ethanol is given in Figure 2.

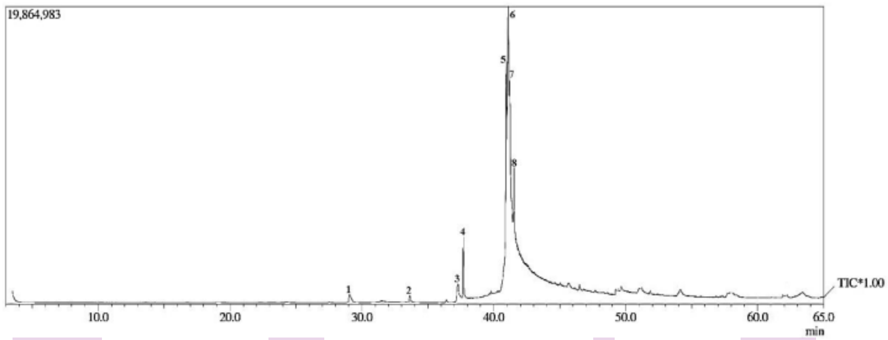

Figure 2. Chromatogram of the transesterification product of rubber seed oil with ethanol.

The chromatogram in Figure 2 views several separate peaks at different retention times. These peaks describe the different types of compound components and are found with the guidance of the Library System WILEY 299 LIB and NIST 62 LIB database as presented in Table 2.

Table 2. The components transesterification product of rubber seed oil and ethanol.

Peak number	Retention time (min)	Molecular formula	Compound name	Relative percentage (%)
1	28.97	$C_{14}H_{28}O_2$	Ethyl laurate	1.36
2	33.55	$C_{16}H_{32}O_2$	Ethyl myristate	0.71
3	37.10	$C_{16}H_{32}O_2$	Palmitic acid	2.94
4	37.52	$C_{18}H_{36}O_2$	Ethyl palmitate	4.30
5	40.55	$C_{20}H_{36}O_2$	Ethyl linoleate	21.77
6	41.00	$C_{20}H_{38}O_2$	Ethyl oleate	31.20
7	41.17	$C_{18}H_{32}O_2$	Linoleic acid	26.54
8	41.44	$C_{20}H_{40}O_2$	Ethyl stearate	11.18

Table 2 appearances that most of the rubber seed oil has been converted into a mixture of fatty acid ethyl esters (FAEEs), ethyl oleate as the main component. The presence of linoleic acid and palmitic acid components in a relatively large amount (29.48%) indicates that not all triglycerides have been changed into ethyl esters. This is possible because the ethanol reactivity is not sufficient to react completely with the fatty acids in the rubber seed oil during the reaction. Other researchers [5] have expressed that increasing the carbon chain decreases the reactivity of the alcohol.

3.3. 2-propanol

The third type of alcohol is 2-propanol, which is linear alcohol with a higher carbon chain length than methanol and ethanol. The transesterification product of rubber seed oil with 2-propanol using MgO/SiO_2 catalyst was analyzed by GC-MS and the chromatogram is presented in Figure 3.

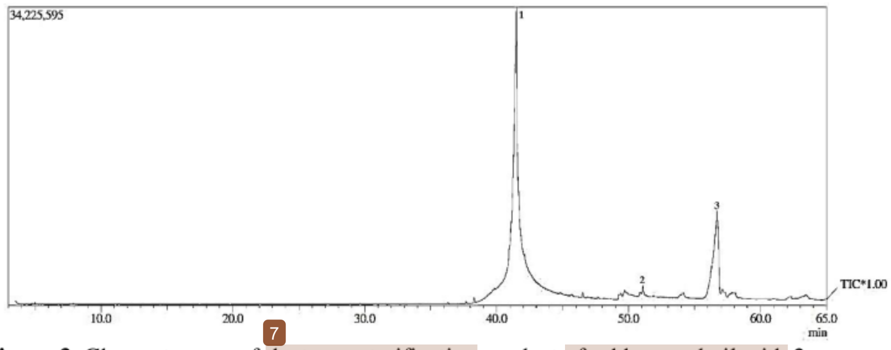

Figure 3. Chromatogram of the transesterification product of rubber seed oil with 2-propanol.

Figure 3 displays that there are three types of components contained in the transesterification product of rubber seed oil with 2-propanol and the results of their identification of the types of compounds are summarized in Table 3.

Table 3. The components transesterification product of rubber seed oil and methanol.

Peak number	Retention time (min)	Molecular formula	Compound name	Relative percentage (%)
1	41.51	$C_{18}H_{32}O_4$	Linoleic acid	72.55
2	51.10	$C_{27}H_{50}O_6$	Tricaprylate glycerol	0.74
3	56.70	$C_{39}H_{74}O_6$	Trilaurate Glycerol	26.71

The data in Table 3 arrays that 2-propanol cannot react completely with triglyceride contained in the oil as indicated by the absence of the formation of a suitable propyl ester in the transesterification product. The existence of linoleic acid in large amounts (72.55%) indicates that triglycerides cannot be converted to alkyl esters and into impurities in the product. Linoleic acid (C18: 2) was reported as one of the constituent components of fatty acids in rubber seed oil [22], this acid is a polyunsaturated fatty acid group, so it is easier to react than saturated or monosaturated fatty acids such as oleic acid (C18: 1). Another impurity in the product is glycerol indicated by the formation of white deposits in the mixture. Fatty acids and glycerol in the products of the transesterification reaction have been reported as impurities [23].

From the three research activities that have been described, it can be said that the transesterification reaction of rubber seed oil with short carbon chain alcohols can be carried out with the presence of MgO/SiO_2 catalyst characterized by a product in the form of an alkyl ester mixture according to the type of alcohol used. Performance of MgO/SiO_2 as a catalyst in the three types of rubber seed oil transesterification reactions with various types of alcohol have been achieved. The catalyst acts as a reaction induced self-separating agent [24] and during the reaction process, the catalyst mixes with the two reactants in the reactor. The catalyst facilitates the reaction between oil and alcohol so that the reaction rate increases with shorter reaction time [25]. Finally, MgO/SiO_2 as a solid heterogeneous catalyst can be separated from the reaction product by filtration. In general, the transesterification of rubber seed oil with different alcohols using a MgO/SiO_2 catalyst has been successfully carried out, which is indicated by the percent of conversion that can be achieved as shown in Figure 4. Based on the percent conversion value, it can be concluded that the order of alcohol reactivity used is methanol > ethanol > 2-propanol with values of 90.1, 73.3, and 63.2%, respectively. Methanol has the highest reactivity due to its shorter carbon chain, [26] also reported that the short-chain alcohols provide better conversions under the same reaction time and this is consistent with the researchers [5,7] who concluded that an increase in the length of the carbon chain causes a decrease in alcohol reactivity.

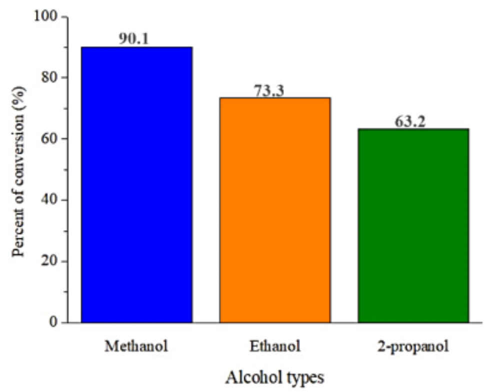

Figure 4. Effect of alcohol type on the percent conversion of rubber seed oil transesterification products using MgO/SiO_2 catalyst.

4. Conclusion

In this study, the type of alcohol that is reacted with rubber seed oil using a MgO/SiO_2 catalyst effects the conversion value of triglycerides to fatty acid esters. The shortest chain length in the alcohol, the higher its reactivity. The results showed that the order of reactivity was methanol, ethanol, and 2-propanol with conversion values of 90.1, 73.3, and 63.2%, respectively.

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