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AUTHOR

Julfi Restu Amelia

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Production of biodiesel using heterogeneous catalyst (K-Impregnated bleaching earth) based on reactivated bleaching earth

Dewi A Iryani[1,4], Valerie Ixion[1,*], Ashilla Utari[1], Simparmin Br. Ginting[1], Ribut Sugiharto[2], Lathifa Indraningtyas[2,4], Julfi R. Amelia[3], Udin Hasanuddin[2,4]

[1]*Department of Chemical Engineering, Faculty of Engineering, University of Lampung, Indonesia*
[2]*Department of Agro-industrial Technology, Faculty of Agriculture, University of Lampung, Indonesia*
[3]*Study Program of Food Technology, Faculty of Food Technology and Health Sahid University, Indonesia*
[4]*Research and Development Center of Biomass and Bioenergy, University of Lampung, Indonesia*

Abstract:
Spent Bleaching Earth (SBE) is one of major solid waste generated by bleaching processes in the refinery oil industry. SBE contains oil, fat, pigments, and other impurities originated from crude oil. SBE contains about 25 to 40 % oil. Due to the large amount of oil and mineral composition in SBE, this waste is categorized as a hazardous waste which has a risk of explosion, flammable, reactive, corrosive and toxic based on Republic of Indonesia government regulation 101/2014. SBE is classified as category 2 of hazardous and toxic waste from a specific source with code number B413. In Indonesia, the volume of this waste is increasing every year proportionally with the growth of the refinery oil industry due to the consumption of cooking oil. In 2019, SBE produced from the vegetable oil refining process in Indonesia reached 779 thousand tons, of which 401 thousand tons were processed, while the rest (about 377 thousand tons) were stored or stockpiled. So that, an innovation is needed to anticipate the problem of this waste. In this paper, we describe the investigation about the preparing, reactivating, and synthesizing of SBE as heterogeneous catalysts for biodiesel production. In this study, SBE was extracted to release entrapped oil by using solvent. After oil extraction processes, the reactivated bleaching earth (RBE) was then reactivated by acid-activation processes (concentration of acids were H_2SO_4 15%). The RBE was then impregnated with KOH in varied concentrations (5%, 10%, 15%, 20%, and 25%) for 24 hours and at a constant temperature 60 ℃. After that, the K-impregnated bleaching earth is filtered using a sieve with 100 mesh size. The characterization of K-impregnated bleaching earth was conducted by XRF (x-ray fluorescence), FTIR (Fourier Transform Infra-Red) Spectrophotometer, and XRD (X-ray crystallography) analysis. The characterization result showed that highest values of K_2O for heterogeneous catalysts obtain in condition of reactivation by using the H_2SO_4 15% KOH 20% with the percentage of mineral composition K_2O are 64.509% and the percentage of crystallinity are and 11.96%. The performance as catalyst was then examined in biodiesel making process. The K-impregnated bleaching earth as heterogeneous catalysts successfully produce biodiesel with the highest yield is 82.24 %. It was obtained at a reaction time of 2 h, 9 % catalyst, methanol to oil ratio of 10:1, and the reaction temperature at 45 °C.

Keywords: *Spent Bleaching Earth; Re-activated Bleaching Earth; Heterogeneous Catalyst; Biodiesel; K-impregnated Bleaching Earth*

**Corresponding author. Tel.: +62-821-1344-6132*
E-mail: valerie.ixion1043@students.unila.ac.id

1. Introduction

Indonesian records as the biggest state for producing crude palm oil (CPO) for decades. Because of the huge quantities of CPO production, there would be more industries to process the oil for society's needs such as kitchen products (cooking oil, margarine), skincare products (soap), and could be the biodiesel for fuel. In the process of turning CPO into the new products through the refining process, it produces a solid waste named Spent Bleaching Earth (SBE). SBE still contains oil, fat, pigments, and other impurities originated from crude oil. SBE contains about 25-40% oil. Due to the quantities, the Republic of Indonesia Government states at Government Regulation No.101/2014 that SBE is categorized as a hazardous waste which has a risk of explosion, flammable, reactive, corrosive, and toxic. The waste of CPO bleaching processes such as spent bleaching earth (SBE) are categorized as hazardous and toxic waste. According to the type of this waste, in 2019 (especially Indonesia) it reached 779 thousand tons of SBE produced and only 401 thousand tons were processed. Hence, the rest of the waste is necessary to anticipate the problem of this waste such as processing the waste into catalyst heterogeneous for biodiesel production.

Biodiesel is produced efficiently using various types of homogeneous catalysts such as KOH, H_2SO_4 and NaOH. With the high level of catalytic activity of this homogeneous catalyst in the transesterification reaction, there are several drawbacks that hinder the application of homogeneous catalysts on an industrial scale. The homogeneous catalysts require high costs for the separation process from the product, can only be used once, cause corrosion of equipment, and produce toxic liquid waste, so the use of heterogeneous catalysts for biodiesel production is highly desirable. The heterogeneous solid catalysts has low production costs, does not cause corrosion in production equipment, is non-toxic, has high purity in biodiesel yields, is easy to separate and reuse (Abukhadra & Sayed., 2018; Costa & Lima., 2021).

Heterogeneous solid catalysts have been studied as a substitute for homogeneous catalysts, and have the advantage of being easy to reuse and also that heterogeneous catalysts are more environmentally friendly than homogeneous catalysts (Naser et al., 2021). Heterogeneous solid catalysts are usually used for biodiesel production which are categorized into several types, including alkaline solid catalysts, acid solid catalysts, mesoporous silica support catalysts and alkaline support catalysts (Soetaredjo et al., 2011; Naser et al., 2021).

SBE is a waste of crude palm oil (CPO) refining process composed of bleaching soil and crude oil. SBE can be extracted to obtain the oil and the remaining de-oiled bleaching earth (RBE). RBE is composed mainly of bentonite which has a high ion exchange power, so it can be impregnated with cations to be used as solid catalysts. Bentonite clay is a commodity mineral that can provide many benefits, both for entrepreneurs and for the state in increasing their foreign exchange. Potential deposits are quite widely spread in Indonesia and are generally found in the form of Ca-Bentonite. Bentonite plate can be used as an adsorbent because it has a layered structure, the ability to expand (swelling) and has exchangeable cations (Naqiatuddin et al., 2014). Bentonite is hydrophilic, so it is generally incompatible with most polymeric materials, and must therefore be chemically modified to make its surface more hydrophobic (Othman et al., 2006; Bukit et al., 2013).

Recently, the research using clay as catalyst for heterogeneous catalyst on biodiesel production has perfomed with a good result. The conducted by Abukhadra & Sayed, 2018 used kaolin because it has sufficient availability and lower costs than bentonite and montmorillonite. The main application of kaolinite in transesterification reactions is limited to being a precursor only in the synthesis of zeolite-A which involves high temperature calcination, and the product has a high cation exchange capacity which negatively affects the stability of the catalyst. Abukhadra & Sayed, 2018 synthesized a new catalyst from kaolinite (Kaol/K^+) trapped by K^+ with effective catalytic properties in the manufacture of biodiesel from vegetable oil through the transesterification process. The catalyst was synthesized with potassium hydroxide with different concentrations and then calcined at a temperature of 573.15K. The most alkaline content was at a concentration of 30% and the best % yield was 94.76% with an operating temperature of 343.15K, the ratio of oil: methanol was 1:14 for 180 minutes and the weight of the catalyst added was 15% of the weight of the oil. Based on previous research, in this study we conduct the preparation of RBE impregnation KOH as a base catalyst for biodiesel production by using crude palm oil as the feedstock in biodiesel production.

2. Materials and methods

2.1 Materials

Spent Bleaching Earth was used in this study obtained from PT. Wahana Citra Nabati, Jakarta, Indonesia. The SBE was extracted using the Soxhlet extraction method and n-hexane solution to separate the oil from the sample, called de-oiled bleaching earth (DBE), for 8 hours. The DBE was dried in an oven 105°C for 4 hours (until its weight was constant). After the sample dried, it was

sieved using a strainer 200 mesh to obtain the same particle size. The DBE sample was going to be activated using acid (H_2SO_4 15%) for 24 hours at room temperature with the ratio between DBE to acid solutions was 1:5. Then the mixture was gently separated to separate the solution from solid (reactivated bleaching earth; RBE) and washed with distillate water until the pH neutral. The RBE sample was dried in the oven for 10 hours to reduce water content at 105°C.

The RBE sample was washed in hydrogen peroxide solution (30%) at normal temperature for 30 minutes to remove organic impurities inside with the ratio between H_2O_2 solution to RBE was 2:1. Then the mixture was separated and the RBE was dried in the oven 110°C until the moisture content was 10%. Crude palm oil, (extracted from SBE) was used as raw material for production of the biodiesel with other additives in the form of methanol and an alkaline catalyst as a part to accelerate the process of formation of biodiesel and glycerol.

2.2 Solid catalyst preparation

Solid catalyst preparation was following the method of Soetaredjo, et al., (2011) with some modification. RBE was washed by using H_2O_2 solution and then it was prepared using the following procedures (2.1). The processes for production catalyst in this study using five variables of KOH concentrations were 5%, 10%, 15%, 20%, and 25% of KOH loadings and the ratio between KOH solution to RBE was 250mL KOH:100gr RBE. The impregnation process was the conducted by loading the sample of RBE in a three-neck round bottom flask (250mL) which was equipped with a magnetic stirrer, reflux condenser, thermometer, and hotplate. The impregnation of RBE with KOH was conducted at 60°C temperature for 24 hours and continuously stirred 400 rpm. After the impregnation process finished, the slurry was dried in the oven for 24 hours at 110°C, and calcined in the furnace at 500°C for 5 hours. The calcinated product is the solid catalyst. The solid catalyst was sieved using a strainer with 100 mesh size to obtain the uniformly of particle size.

2.3 Catalyst characterization

The sample of the solid catalyst was analyzed by Powder X-Ray Diffraction (XRD) patterns recorded on *PANalitycal MPD* at 40 kV and 30 mA using Cu Kα radiation of 0.001° of step size. The X-Ray Fluorescence (XRF) was recorded on *PANalytical Epsilon 3* to determine the elemental compositions of catalysts. And the qualitative analysis for the catalysts was conducted by *Fourier Transform InfraRed* method using Agilent Technologies Cary 630 with Happ Genzel function. The analysis of FTIR was carried out in a wavenumber range of 4000–650 cm^{-1}.

2.4 Esterification & transesterification of crude palm oil

The first step in the process of making biodiesel was to determine the FFA content in the oil. Previously, the oil samples were analyzed for their FFA content using the titrimetric analysis The permitted standard for FFA content is < 2.5%. If the oil contains more than 2.5% FFA, further treatment is needed to reduce the FFA content by carrying out an esterification process with an additional catalyst of 1% H_2SO_4 from the volume of oil used. The esterification process was carried out for 1 hour at a temperature of 60°C. Furthermore, the sample was transferred into a separatory funnel to separate the oil and methanol. The esterified oil was then rinsed using distilled water until the pH of the washing water was neutral. The last step, the sample was put the esterified oil in the oven until the weight of the oil was constant and will be used for the transesterification process.
Transesterification of the esterified palm oil was carried out using a three-neck round bottom flask equipped with a reflux condenser, temperature indicator, and magnetic stirrer and the reactor was placed in a hotplate. A known amount of heterogeneous (solid) catalyst (3%, 5%, 7% and 9%) was added to the mixture of the oil and methanol at (oil:methanol) 1:4, 1:6, 1:8, and 1:10 ratio. The mixture was then heated to the desired temperature (45°C, 50°C, 55°C and 60°C) with variables of time (60 min, 90 min, 120 min, and 150 min). Subsequently, then, the mixture was transferred to a

separatory funnel to separate the biodiesel product and the resulting glycerol. Then the sample was washed using distilled water by adding 100 ml of distilled water and left overnight so that there was a layer between the biodiesel and the washing water. Biodiesel was washed 3-4 times until the pH of washing water was neutral. Lastly, sample biodiesel put in the oven at 110°C for 7-8 hours until the biodiesel weight is constant.

3. Results and discussion

3.1 Catalyst heterogeneous characteristics

3.1.1 Catalyst heterogeneous results

All of solid catalyst samples were produced using the same condition variables processes (such as temperature, time, speed of stirring), the difference only on the KOH loading for each sample.

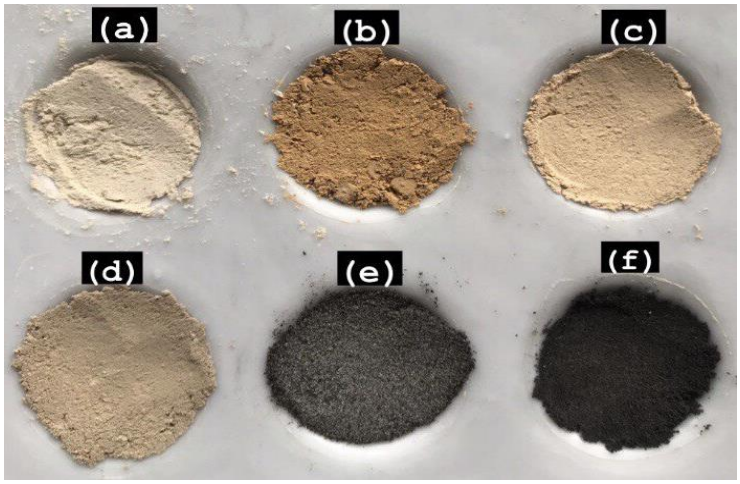

Fig. 1 Catalyst Heterogeneous with difference KOH loads samples: (a) RBE washed by H_2O_2 30% solution (b) KOH 5%, (c) KOH 10%, (d) KOH 15%, (e) KOH 20%, (f) KOH 25%

3.1.2 X-ray diffraction analysis

The results of XRD patterns for the solid catalyst (5%, 10%, 15%, 20%, and 25% KOH loads) and raw bentonite were given on Fig. **2**. As can be seen, when the loading of KOH was increased, the crystallinity of the catalyst was decreased (it showed the reversal from the journal of (Soetaredjo et al., 2011). This could be caused by the surface area pores of the particles which were still filled with silica from the bleaching earth. So, the large amount of K^+ cation or the less free vacancies surface area on the sample may cause the emergence of other compounds. The reflection observed from the XRD measurements around 2θ were = 81.13%, 78.44%, 57.27%, 11.96%, and 11.59% of crystallinity.

The more potassium compounds were loaded on the sample, the much freer vacancies were decreased which was the results directly with crystallinity values on each sample. The vacancies would be filled with K^+ and spread evenly when the amount of K^+ cation were under the saturation uptake. However, if the sample were loaded with too much K^+ cation, the cation would not be dispersed well, and it would cause only some part of the loaded K^+ could be decomposed. The excess of ion K^+ would cover the surface area of the catalysts and would cause the reduction of catalytic activity (Xie & Li, 2006).

3.1.3 Fourier transform infrared analysis

The continuous investigation about K-Impregnated Bleaching Earth Catalyst Performance samples was conducted by IR spectroscopy shown in Fig 3. The spectra showed the presence of different functional groups.

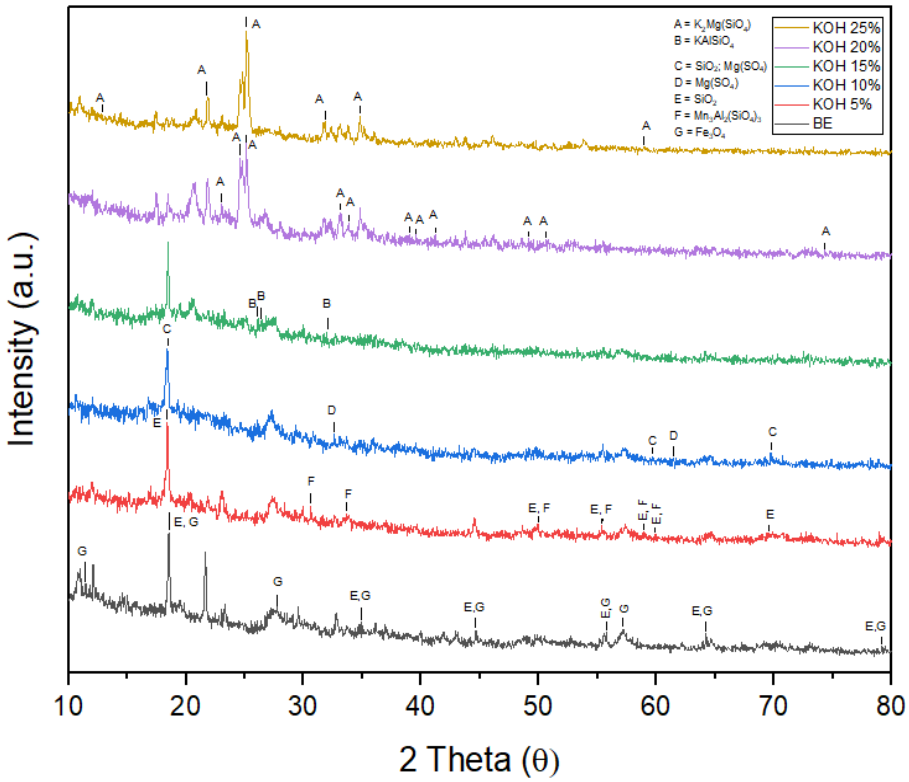

Fig. 2 XRD patterns for solid catalysts samples made of K^+ Impregnated RBE

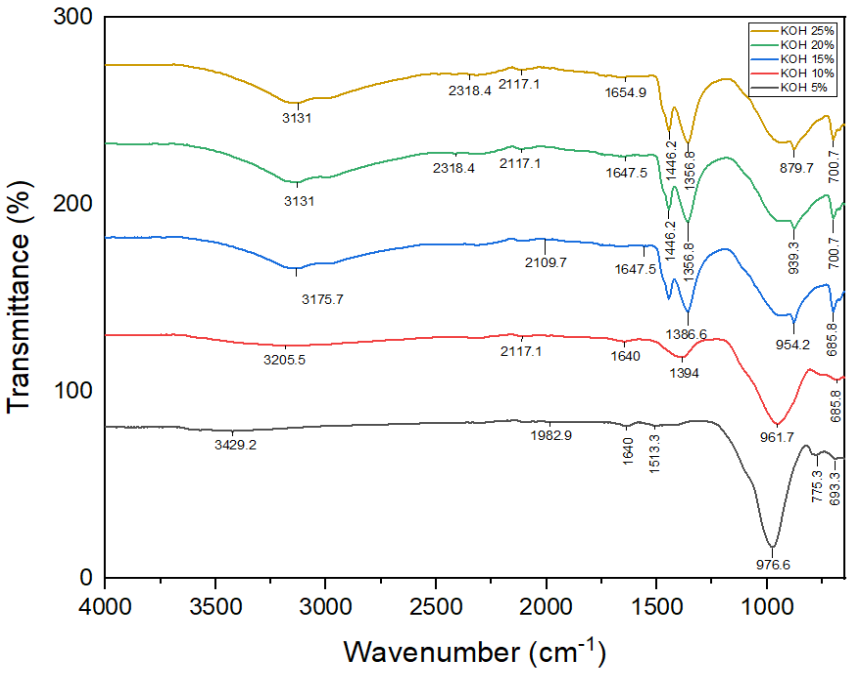

Fig. 3 Fourier Transform Infrared spectra of Solid Catalyst made of K^+ Impregnated RBE

From Fig 3. It showed that from the difference KOH loads would show the difference wavenumber for the same functional group. The addition of KOH obviously affected the structure of bentonite (as shown in Fig 3) with noticeable changes from the 15% KOH addition. The spectrum reveals the presence of functional groups such as Al-O-K (3429.2 cm-¹), H-O-H bending (16549.9 - 1640 cm-

^{1}), C=C and O-H bonds (1446.2 cm-^{1}) Si-O-Si stretching (976.6 - 961.7 cm-^{1}), OH bending bounded Al^{3+} and Fe^{3+} (954.2 - 939.3 cm-^{1}), Si-O stretching (775.3 cm-^{1}), and Si-O-Al (685.8 - 693.3 cm-^{1}) (Naser et al., 2021; Ali et al., 2016; Soetaredjo et al., 2011).

3.1.4 X-Ray fluorescence analysis

This analysis aims to determine the mineral content of catalyst heterogeneous (K-impregnated bleaching earth) with the help of x-rays. The figure and table below are showing the compared data of the mineral content between raw bentonite and catalyst heterogeneous with differences KOH loadings:

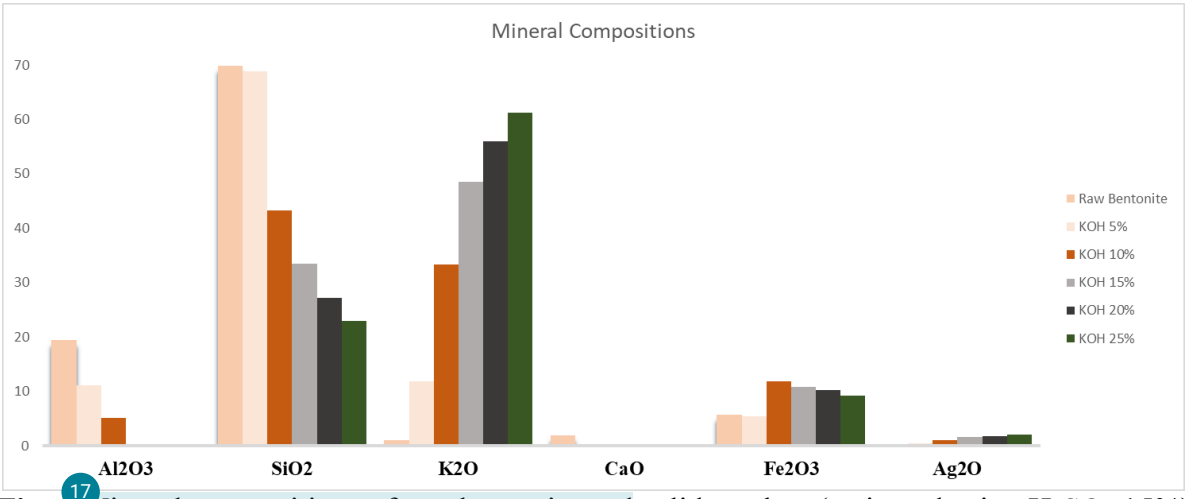

Fig. 4 Mineral compositions of raw bentonite and solid catalyst (activated using H_2SO_4 15%)

Table 1 Mineral compositions of raw bentonite and solid catalyst (activated using H_2SO_4 15%)

Compounds	Raw Bentonite (%)	Variables of KOH Concentration (%)				
		5%	10%	15%	20%	25%
SiO_2	69.920	42.533	58.758	32.068	18.427	30.592
K_2O	1.030	19.892	21.131	44.456	64.509	59.019
Al_2O_3	19.480	0.000	8.966	0.000	0.000	0.000
CaO	1.875	8.382	0.000	0.000	0.000	0.000
Fe_2O_3	5.661	14.764	6.724	15.118	10.360	6.531

The results show that solid catalysts which are activated by acids and impregnated by KOH have an increase of Potassium (K_2O) content due to the increase of KOH loading on the catalyst. However, the Potassium value decreased on the solid catalyst loading more than 25% of KOH. The sample which has the high potassium value is the solid catalyst with 20% KOH loads. In this study, the high value of K_2O at the sample can improve the catalytic performance for the biodiesel production.

3.2 The performance test of solid catalyst

To investigate the performance of solid catalysts (K-impregnated Bleaching Earth) we use catalyst with the highest of percentage of K content. The catalyst was used for transesterification processes

to produce biodiesel from crude palm oil. Experiment conditions for studying the performance of catalysts on the transesterification reaction were: amount of catalyst 9% based on the weight of palm oil, oil to methanol molar ratio 1:10, reaction time of 120 min and the reaction temperature at 60°C. Full results indicated that the amount of KOH loaded in RBE affected the conversion of palm oil into biodiesel (82.24%). The increase of the percentage of the solid catalyst added also increased the yield of biodiesel at a certain reaction time, temperature, and methanol ratio. The highest yield (82.24%) was obtained at 9% solid catalyst addition, 1:10 of the oil to methanol ratio, 45°C for 120 min. In this study, to determine the operation condition of biodiesel production it was used Taguchi method as in following table:

Table 2 The Effect of different process and variables on the yield of biodiesel (Using H_2SO_4 15% KOH 25%)

Amount Catalyst (%)	Oil:Methanol ratio	Temperature (°C)	Time (min)	%yield
3	1:4	45	60	71.96
3	1:6	50	90	67.67
3	1:8	55	120	68.74
3	1:10	60	150	69.98
5	1:4	50	120	68.22
5	1:6	45	150	75.25
5	1:8	60	60	68.32
5	1:10	55	90	67.95
7	1:4	55	150	63.53
7	1:6	60	120	73.34
7	1:8	45	90	72.80
7	1:10	50	60	79.90
9	1:4	60	90	63.26
9	1:6	55	60	66.04
9	1:8	50	150	77.66
9	1:10	45	120	82.24

3.2.1 Effects of the solid catalyst amount

To distinguish the effect of solid catalyst on the yield of biodiesel, the amount of catalyst was added in several ratio, such as between 3%, 5%, 7% and 9% by weight based on the amount of palm oil. The yield of biodiesel increased from 63,26% at 9% RBE/KOH catalyst to 82.24% at 9% catalyst. It can be seen from Table 1, in each comparison the catalyst has a different % yield. The more solid catalyst added, the more active base (K_2O) in the biodiesel production reaction system.

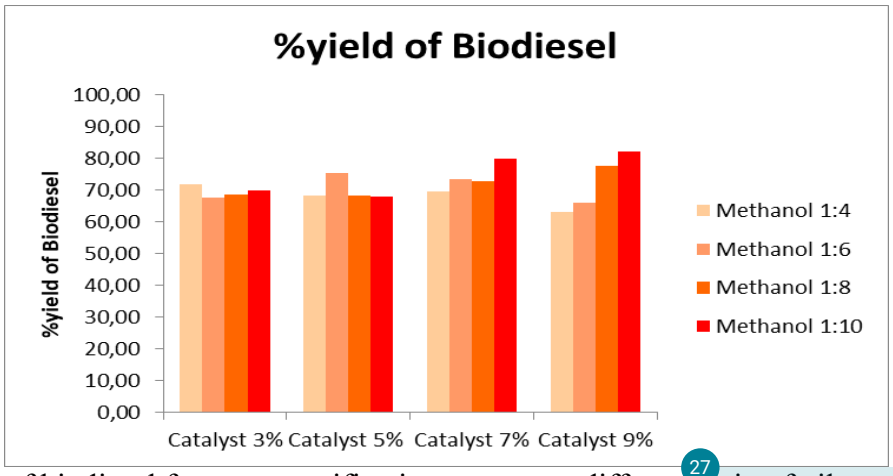

Fig. 5 Yield of biodiesel for transesterification process at different ratio of oil to methanol and percent of solid catalyst addition

3.2.2. Effects of reaction time

the studies conducted in this experiment had different variations in reaction times, from 60 min to 150 min. Table 2 shows that the highest biodiesel yield is produced from 120 minutes reaction time. The biodiesel yield decreases after 120 minutes reaction because the reaction equilibrium conversion has been reached. The transesterification reaction with methanol is a reversible reaction, so to change the reaction from the left to the right, it can be seen from the reaction equilibrium (Abukhadra et al., 2019).

3.2.3. Effects of temperature

The research on transesterification of CPO using a solid catalyst was carried out at temperatures ranging from 45 to 60°C. Based on the reaction temperature used, the transesterification reaction carried out at 45°C produced the highest yield, and the lowest yield was produced at a reaction temperature of 55°C. The complete results can be seen in Table 2. This is also influenced by the length of the transesterification reaction. One of the factors that affect the amount of biodiesel yield is temperature, reaction time and the amount of methanol added.

3.2.4. Effects of the ratio methanol

The effect of the ratio of oil to methanol as one of the factors in the oil transesterification process which in this study was carried out in the range of 1:4 to 1:10 with different weight factors of addition of catalyst, temperature, and time. The addition of methanol in the biodiesel formation reaction is one of the important things because in one mole of triglyceride will interact with three moles of methanol to produce three moles of FAME and one mole of glycerol (Abukhadra et al., 2019).

The surface area between the immiscible liquids does not affect as the ratio of methanol to oil increases, this can give high frequency values for solid catalysts which causes a considerable increase in the exposed surface area and catalytic activity (Abukhadra et al., 2019).

3.3. Biodiesel characteristics

3.3.1 Iodine number

Iodine number is the number of grams of iodine absorbed per 100 grams of oil, and it shows the degree of unsaturation of the oil. Unsaturated fatty acids in oils and fats are able to absorb a certain amount of iodine and form saturated compounds. The amount of iodine absorbed indicates the number of double bonds or unsaturated bonds in the oil (Suryani et al., 2015). From the results of the analysis of biodiesel samples obtained the value of the iodine number.

Table 3 Iodine number of biodiesel

No.	Sample	Iodine Number (%-mass (g-I_2/100g))	Iodine Number (SNI 7182:2015) (%-mass (g-I_2/100g))
1.	C: 3, M: 4, t: 60, T: 45	44.37	155 (max)
2.	C: 3, M: 6, t: 90, T: 50	39.67	
3.	C: 3, M: 8, t: 120, T: 55	27.31	
4.	C: 3, M: 10, t: 150, T: 60	28.47	
5.	C: 5, M: 4, t: 120, T: 50	28.61	
6.	C: 5, M: 6, t: 150, T: 45	31.22	
7.	C: 5, M: 8, t: 60, T: 60	30.33	
8.	C: 5, M: 10, t: 90, T: 55	32.09	
9.	C: 7, M: 4, t: 150, T: 55	30.10	
10.	C: 7, M: 6, t: 120, T: 60	32.54	
11.	C: 7, M: 8, t: 90, T: 45	31.70	
12.	C: 7, M: 10, t: 60, T: 50	34.60	
13.	C: 9, M: 4, t: 90, T: 60	36.34	
14.	C: 9, M: 6, t: 60, T: 55	34.49	
15.	C: 9, M: 8, t: 150, T: 50	32.72	
16.	C: 3, M: 4, t: 120, T: 45	31.87	

*Notes
C = Catalyst
M = Methanol
t = Time
T = Temperature

3.3.2 Cetane number

Cetane number is one of the most significant properties for determining the ignition quality of a fuel for use in diesel engines. The cetane number of methyl ester (biodiesel) is higher than that of vegetable oil and petro diesel. As an indicator of ignition quality, the cetane number is the main indicator of fuel quality in the realm of diesel engines. Conceptually, it is similar to the octane number used for gasoline (Gopinath et al., 2009). From the Indonesian national standard, the cetane number in biodiesel has a minimum of 51 and in the study conducted, the cetane number from each sample was higher than that of the standard (Table 4).

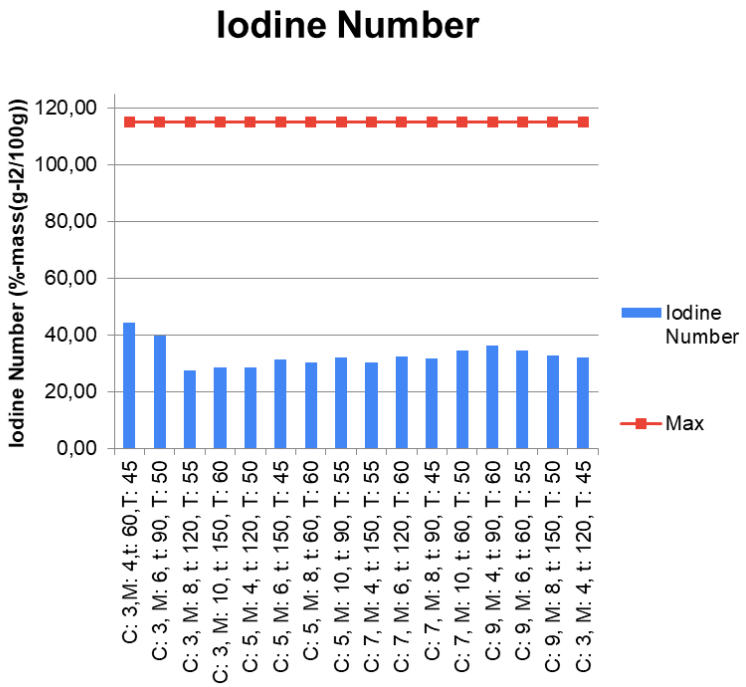

Fig 6. Iodine Number of Biodiesel

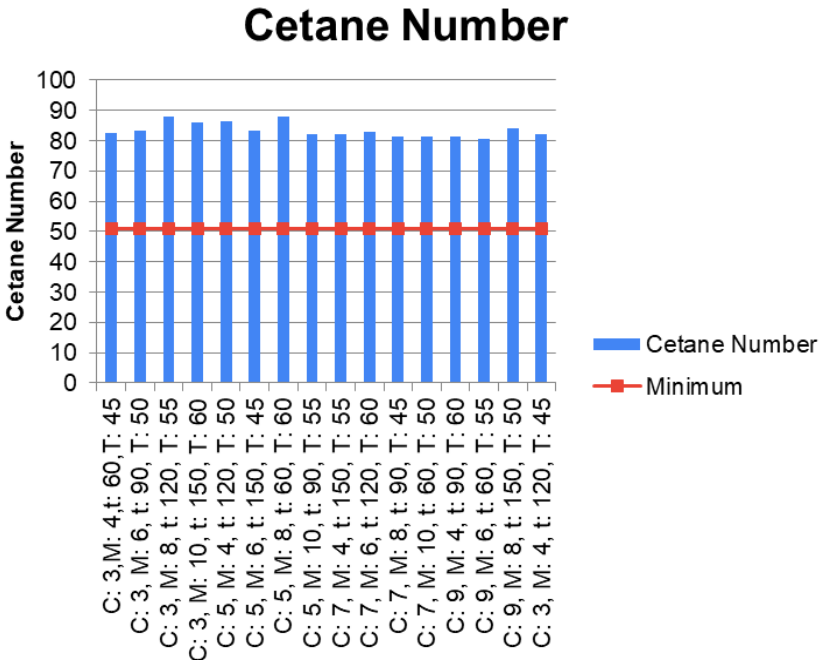

Fig. 7 Cetane number of biodiesel

Table 4 Cetane number of biodiesel

No.	Sample	Cetane Number	Cetane Number (SNI 7182:2015)
1.	C: 3, M: 4, t: 60, T: 45	82.42	
2.	C: 3, M: 6, t: 90, T: 50	83.21	

No.	Sample	Cetane Number	Cetane Number (SNI 7182:2015)
3.	C: 3, M: 8, t: 120, T: 55	87.86	51
4.	C: 3, M: 10, t: 150, T: 60	85.94	
5.	C: 5, M: 4, t: 120, T: 50	86.45	
6.	C: 5, M: 6, t: 150, T: 45	83.44	
7.	C: 5, M: 8, t: 60, T: 60	87.78	
8.	C: 5, M: 10, t: 90, T: 55	82.15	
9.	C: 7, M: 4, t: 150, T: 55	82.02	
10.	C: 7, M: 6, t: 120, T: 60	82.87	
11.	C: 7, M: 8, t: 90, T: 45	81.31	
12.	C: 7, M: 10, t: 60, T: 50	81.36	
13.	C: 9, M: 4, t: 90, T: 60	81.44	
14.	C: 9, M: 6, t: 60, T: 55	80.72	
15.	C: 9, M: 8, t: 150, T: 50	83.89	
16.	C: 3, M: 4, t: 120, T: 45	82.01	

Table 5 Comparison of biodiesel properties in this study with Indonesia National Standard (SNI 7182:2015)

No.	**Properties**	**Biodiesel Produced**	**SNI 7182:2015**
1.	%yield (%)	82.24	-
2.	Moisture Content (%-volume)	11.96	0,05
3	Density 28 C (kg/m^3)	0.00090426	850 - 890 (40 C)
4.	Cetane Number	82.01	51 (min)
5.	Iodine Number (%-mass (g-I_2/100g))	31.87	115 (max)

4. Conclusion

The impregnation of Potassium (from KOH) on Reactivated Bleaching Earth (RBE) with H_2SO_4 15% as the catalyst heterogeneous was worked. From the variation of KOH loads, it got the best result at KOH 25% loading. The heterogeneous catalyst went through the test performance and was used for supporting the manufacture of biodiesel production by esterification and transesterification from crude palm oil which has been absorbed once. The optimum condition to produce the highest

yield of biodiesel 82.24% was at: catalyst amount 9%wt, methanol to oil ratio 10:1, reaction time 120 min, and the reaction temperature at 45°C.

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