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Characteristics and catalytic activity of zeolite-a synthesized from rice husk silica and aluminium metal by sol-gel method

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Abstract. In this research, sol-gel process was applied to synthesize zeolite-A from rice husk silica and aluminum metal. For preparation of the zeolite, the specified amount of rice husk silica and aluminum metal was separately dissolved in NaOH solution, and both solutions were transfered in to a laboratory mixture for complete mixing and production of gel. The gel was converted into solid zeolite precursor by oven drying of the gel for 24 hours. The precursor was transformed into zeolite by subjecting to different calcination temperatures, and then characterized using different techniques. The zeolites were also tested as catalyst for transesterification of *Ricinius communis* oil. Development of structure of the zeolite was confirmed using FTIR and XRD techniques analyses, while SEM analysis revealed the characteristic of the samples as porous and multiphasic materials. The zeolites were found to exhibit good catalytic activity as revealed by the results of transesterification of *Ricinius communis* oil. Appreciable catalytic activities were also demonstrated by the reaction yields achieved which are in the range of 68 to 82%.

1. Introduction

Zeolite represents very important group of applied materials, due to their unique properties, capable of playing a variety of important roles such as adsorbents, ion exchangers, and catalysts. At present, one of catalytic reaction extensively investigated is transesterification of glycerides using low molecular weight alcohols, to convert the glycerides into their corresponding simpler alkyl esters, known as biodiesel. In the context of progressive increase of energy demand, biomass derived energy sources or biofuels, including biodiesel, continue to gain global interest [1-2].

In the manufacture of biodiesel, the catalyst is a major requirement, since glycerides and alcohol have very low reactivity toward each other, and therefore, without the aid of catalyst practically no reaction takes place. In the current practices for biodiesel manufacture, homogeneous catalysts remain as the most widely used [3-5], however heterogeneous catalysts continue to gain interest [6-8]. Heterogeneous catalysts offer a number of advantages over homogeneous catalysts, such as simple catalyst recovery, non-corrosive, as well as the possibility to reuse the catalyst [9]. With these various advantages they offered, the use of heterogeneous catalysts for transesterification of different vegetable oils continue to progress. Apart from this progressive development, it should be

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acknowledged that detailed reaction mechanism of transesterification involving heterogeneous catalyst has not been fully understood and remain a scientific challenge.

Considering the advantages, they offered, various heterogeneous catalysts have been developed and tested, such as Al-loaded CaO [10] and several synthetic zeolites. The promising potential of synthetic zeolites to support different utilizations is clearly illustrated by continuous development of various synthetic zeolites [11-13]. For production of biodiesel, this type of catalyst has been used to treat different raw materials, such as waste cooking oil [14], microalga oil [15], palm oil [12], triglycerides [16], butter [17]. Utilization of modified catalyst, in which zeolite pellet was coated with γ-alumina, for transesterification of waste cooking oil has also been reported [18].

In the use of zeolites as catalyst for biodiesel production, it is generally acknowledged that the main feature which determines the catalytic activity of zeolite is the chemical composition. In this respect, the general findings have shown that different zeolites most likely will lead to different performances when they are applied to the same raw material. The possibility varied performances implies that selection of the most suitable catalyst for specific raw material should be taken into account. For this reason, in this study zeolite-A was tested for transesterification of *Ricinus communis* oil, with the main purpose to assess whether this type of zeolite possess the viable potential for further development.

Besides development of catalysts, the search for raw materials is another basic aspect of biodiesel industry, with the main focus on exploring production of biodiesel from waste oil [14, 18] and non-edible vegetable oils as alternative raw materials to coconut and palm oil which has been used as traditional raw material. The main advantage that can be derived from this type of raw materials is to avoid the competition between food and energy source. There are many species of plant that produce vegetable oil with the amount that is worth taking into account and have been converted into biodiesel, such as *Jatropha curcas* [9, 19], rubber or *Hevea brasiliensis* [7, 20], and Castor or *Ricinus communis* L. [21-22]. Generally, such crops can grow in the region of tropical and subtropical climates [23] with significantly lower cultivation cost than that required for edible vegetable oil producing plants. In addition non-edible oil producing plants do not require intensive care to grow well and deliver high oil yields [24]. *Ricinus communis* oil is a promising alternative raw material for production of biodiesel considering its abundant availability and its existence as non-edible vegetable oil. In addition, the chemical composition of the oil is much simpler compared to the others, in which ricinoleic acid exists as the main component with the contribution between 83.85 to 87.62%, with other components such as palmitic, stearic, oleic, linolic, linolenic, arachidic, and arachidonic acids, in small percentages [21]. To some extent, this simple composition is advantageous since the composition and, therefore, the characteristics of the biodiesel derived from the oil are also simpler than those of biodiesels derived from other raw materials.

Acknowledging the important roles of catalyst and raw material in biodiesel industry, in this present investigation, sol-gel method was applied to prepare zeolite-A from aluminum metal and silica extracted from rice husk. The use of these raw materials is based on the solubility of rice husk silica and aluminum metal in NaOH solution. For sol-gel process, separately prepared rice husk silica solution and aluminum metal solution were mixed to produce the precursor of zeolite-A in the form of gel. The gel was dried and subsequently calcined at varied temperatures to transform the precursor into zeolite. The characteristics of zeolites were then evaluated in term of acidity using pyridine absorption method, functionality using FTIR, structure using XRD, and microstructure using SEM. The catalytic activity of the zeolites was tested in transesterification of *Ricinius communis* oil with methanol. The reaction product was analysed using GC-MS technique to examine whether the product composed of methyl esters which confirm that the zeolite-A synthesized functioned as catalyst.

2. Materials and methods

2.1. Materials

Sodium hydroxide and nitric acid from Aldrich, methanol from Merck, Al metal in the form of rods with diameter of 1 cm was from CV. Aluminum Jaya Perkasa Jakarta. A rice milling company in Bandar Lampung kindly provided rice husk sample.

The instruments used in this study are Fourier Transform Infrared Spectrophotometer (GX, Perkin-Elmer), Bruker D8 Advance for XRD instrument, FEI type Inspect S50 SEM equipped with EDAX AMETEK, and GCMS-QP2010 SE SHIMADZU.

2.2. Preparation of zeolite-A

The synthesis of zeolite-A was carried out adopting the method reported in previous study [12]. In brief, the preparation was conducted by first preparing a solution of 40 grams of NaOH pellets in 500 mL of distilled water. An aliquote of 250 mL of this solution was used to dissolve 27 grams of Al metal and the other 250 mL was utilized to dissolve 60 grams of rice husk silica [20]. To produce the zeolite precursor, both solution were transferred into laboratory mixer for complete mixing of the raw materials. The gel obtained was placed in the oven at 90°C for 24 hour drying process to produce dry solid. Powder sample prepared by grinding the solid was then calcined at varying temperatures of 600, 700, 800, and 900°C for 6 hours. The four samples were then characterized and tested as catalyst.

2.3. Catalytic activity trial

To evaluate their catalytic activity, the four samples of zeolite-A were then used as catalyst for transesterification reaction of *Ricinius communis* oil with methanol. The experiment was conducted adopting the procedure used in previous studies [7, 12]. To run the experiment, the reaction mixture was prepared by transferring 25 mL of oil, 50 mL of methanol, and 2.5 g of catalyst into 500 mL round bottom flask placed in heating mantle apparatus. The flask was connected with a reflux condenser for water cooling, and the reaction was run for six hours at fixed temperature 70°C. After the reaction was completed, the mixture was allowed to cool and then the catalyst was recovered by filterring the mixture. The filtrate was left in the funnel to allow the separation of the biodiesel layer and residual methanol and unreacted oil. The biodiesel was collected to determine the reaction yield, and then analyzed using GC-MS equipped with MS Library System NIST62, Wiley 7 database for identification of biodiesel components.

2.4. Acidity determination

In this study, the acidity of the zeolites was determined using gravimetric method, based on the chemisorption of pyridine by the sample.

2.5. FTIR analysis

For functionality analysis using FTIR, the zeolite sample was scanned from 4000 – 400 cm^{-1}. Another purpose of this analysis was to evaluate the effect of calcination temperatures on the functionality of the samples.

2.6. XRD analysis

The X-ray difractogram of the sample was produced using Cu Kα radiation operated at 40 kV energy and 40 mA current, with angle (2θ) in the range of 5–80° and the step of 2°/min.

2.7 SEM analysis

SEM analysis for microstructure evaluation was conducted on SEM instrument of FEI type Inspect S50 equipped with EDAX AMETEK.

3. Results and discussion

3.1. Acidity of zeolites

The acidity data of zeolites subjected to calcination at varied temperatures are shown in Table 1.

Table 1. Acidity of zeolit-A calcined at varied temperatures.

Calcination temperature (°C)	Acidity (mmol/g)
600	0.0451
700	0.0528
800	0.0829
900	0.0100

Experimental data in Table 1 reveal that the acidity of zeolites was quite significantly influenced by temperatures of calcination. It can be observed that increased temperature up to 800°C led to increased acidity, followed by sharp decrease for the sample calcined at 900°C. This decreased acidity is most probably associated with increased crystalline phase in the sample as demonstrated by the XRD results, thus reducing the capacity of the sample to absorb pyridine.

3.2. Functionality of zeolites

As can be observed in Figure 1, the FTIR spectra of the samples are basically similar regardless the calcination temperatures applied. The formation of bonds between O, Si, and Al is suggested by the appearance of distinctive peaks associated with the bonds between the three main elements of the samples.

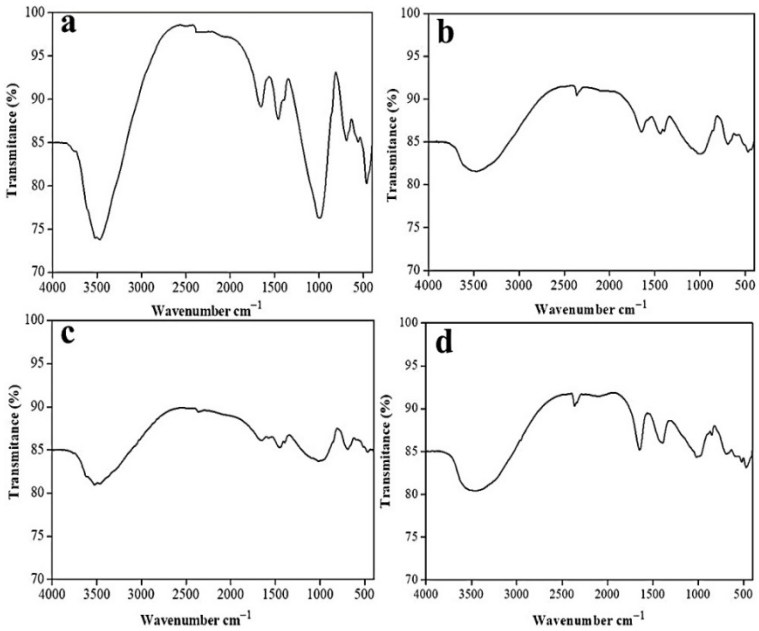

Figure 1. The FTIR spectra of zeolite-A samples calcined at 600 (a), 700 (b), 800 (c), and 900°C (d).

The Si-O-Si symmetric stretching is characterized by absorption bands in the area of about 1013 cm^{-1} and 465 cm^{-1} [25-27]. The peak at 3450 cm^{-1} is an indication of the presence of -OH group attached to the silica framework in the form of Si-OH, and absorbed water, which is confirmed by the peak at 1656 cm^{-1}. The formation of bond between Si and Al is characterized by the presence of peak located at around 691 cm^{-1}, assigned to stretching of Si-O-Al bond [28]. Absorption peak originated

from pyridine base is located at 1659 cm^{-1} which is related to the bending of N-H. The formation of a Lewis acid site, indicated by the peak in the position of approximately 1450 cm^{-1}, was observed in the samples calcined at 600°C (Figure 1a) and 800°C (Figure 1b). In the samples calcined at 700°C (Figure 1c) and 900°C (Figure 1d), on the other hand, this peak associated with Lewis acid disappears, but there is an indication that these two samples contain Brönsted acid site, suggested by the presence of absorption band at around 1640 cm^{-1} [29].

3.3. Structure of zeolites

The XRD difractograms of the samples are presented in Figure 2. The difractograms presented in Figure 2, clearly display that both amorphous and crystalline phases are present in all samples. This amorphous phase is in agreement with the nature of rice husk silica as amorphous material up to 900°C [27]. Crystalline phase identification was carried out using search and match procedure with the aid of software PCPDF for Window 1997. Using this computer program, several crystalline phases are identified, include cristobalite (PDF File: 39-1425), nepheline (PDF File: 35-0424), jadeite (PDF File: 22-1338), and albite (PDF File: 09-0466).

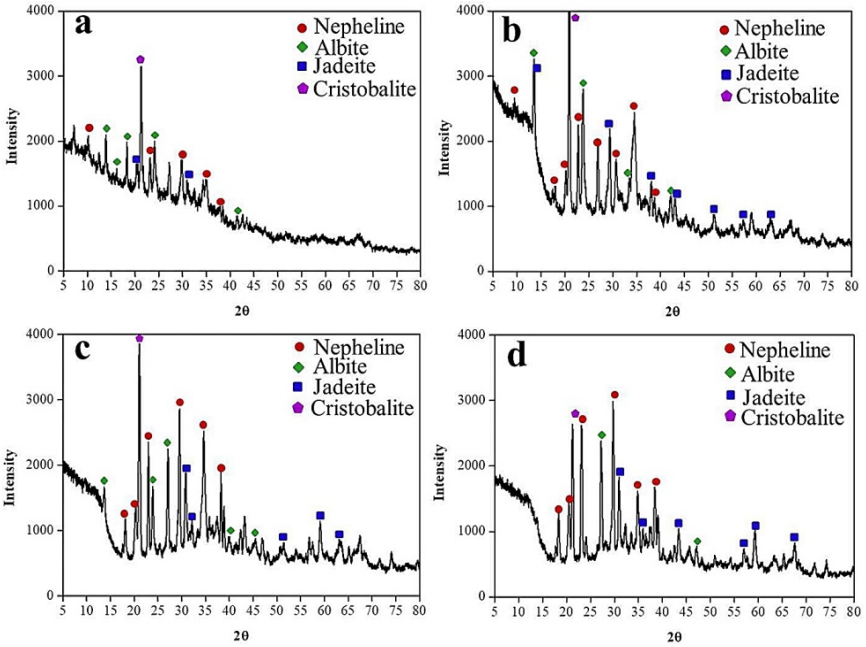

Figure 2. The X-ray difractograms of zeolite-A samples calcined at 600 (a), 700 (b), 800 (c), and 900°C (d).

The four difractograms in Figure 2, suggest that to some extent, the formation of phases in the samples is influenced by the temperatures of calcination treatment applied. In the sample calcined at 600°C, cristobalite appears as the dominant phase, as suggested by significantly higher relative intensity of the peak representing this phase compared to those of the peaks representing the other phases.

The difractograms also display that increased calcination temperatures resulted in gradual growth of crystalline phases, as indicated by increased intensities of the peaks. In addition, it can be seen that up to calcination temperature of 800°C, the cristobalite remains as the dominant phase, however significant change was observed in the sample calcined at 900°C, in which nepheline becomes the dominating phase. Overall, the XRD data reveal that the samples are multiphasic material, with cristobalite and nepheline as the most abundant phases.

3.4. Microstructure of zeolites

To asses the effect of calcination temperatures on microstructure, the micrographs of three samples, with 10000x magnification are compiled in Figure 3. The SEM images in Figure 3, clearly indicate the heterogeneous morphology of the surface of all samples, characterized by the formation of clusters having different shapes and sizes. The essence of the samples as porous materials is clearly reflected by the images, suggesting the potential use of the samples as catalyst. The images also display the presence of several crystalline phases as has also been indicated by the XRD results, include tetragonal-shaped cristobalite, hexagonal-shaped nepheline, monoclinic-shaped jadeite, and triclinic-shaped albite. In overall, characterization using SEM evidently suggests the existence of the samples as multiphasic material.

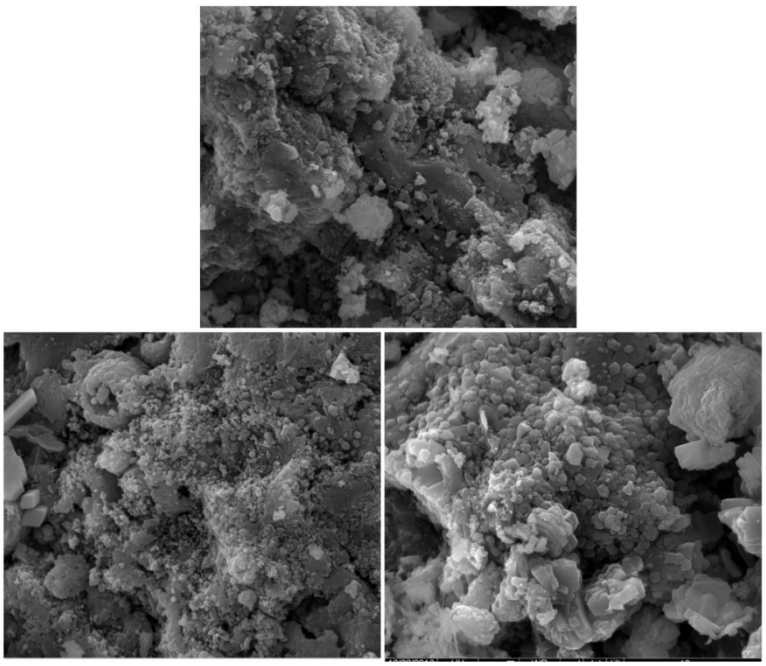

Figure 3. SEM images of zeolite-A samples calcined at 700 (a), 800 (b), and 900°C (c).

3.5. Analysis of transesterification product

The products of transesterification reaction using the zeolites were analyzed with GC-MS spectroscopy, and an example of GC chromatogram is shown in Figure 4.

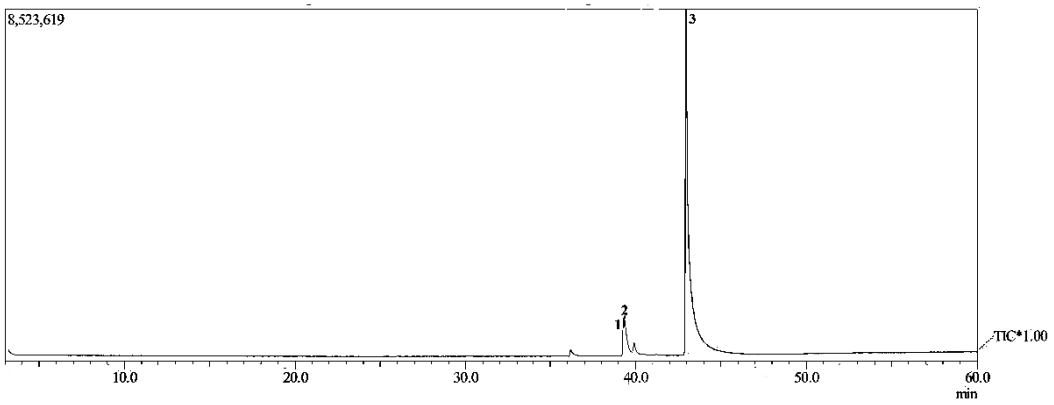

Figure 4. The GC chromatogram of *Ricinius communis* oil transesterification product.

Figure 4 shows the existence of 3 separate peaks in the chromatogram, indicating that the sample composed of three different compounds. With the aid of NIST62 Library System, the three compounds were identified as methyl linoleate, methyl elaidate, and methyl ricinoleate. These three compounds are methyl esters of glycerides contained in *Ricinius communis* oil commonly reported in literatures [30-31]. Formation of these methyl esters confirmed that transesterification took place, confirming the catalytic activity of the zeolites synthesized. The relative composition of the sample was calculated by dividing the relative area of each of the peaks with the total area off all peaks. Using this calculation method, it was found that methyl ricinoleate contributes 87.21%, methyl elaidate contributes 6.7%, and methyl linoleate contributes 4.12%, to the sample. Another finding related to the activity tests is the achievement of reaction yields of 69 to 82%, which also support the existence of zeolite-A as a potential solid catalyst to support biodiesel industry.

4. Conclusions

This study demonstrates that zeolite-A could be prepared using silica extracted from rice husk and aluminum metal, through sol-gel process. Development of zeolite-A structure was revealed by the FTIR and XRD analyses, and the existence of the samples as porous and multiphasic materials by SEM analysis. The zeolites were found to exhibit catalytic activity as revealed by the results of transesterification of *Ricinius communis* oil. Conversion of glycerides contained in *Ricinius communis* oil into methyl esters with appreciable reaction yields indicate that zeolite-A synthesized exhibits good activity as catalyst for transesterification reaction.

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FORMULIR EVALUASI MAKALAH

No. **:**

Judul Makalah **: Characteristics and Catalytic Activity of Zeolite-A Synthesized from Rice Husk Silica and Aluminium Metal by Sol-Gel Method**

A. Poin Evaluasi:

	Ya	Tidak	Periksa Kembali
1. Apakah isi makalah original?	☐	☐	☐
2. Apakah judul makalah mewakili isi keseluruhan makalah?	☐	☐	☐
3. Apakah intisari menggambarkan isi dari makalah?	☐	☐	☐
4. Apakah kata kunci mengindikasikan cakupan penelitian?	☐	☐	☐
5. Apakah pendekatan dan metodologi penyelesaian masalah dijelaskan dengan baik?	☐	☐	☐
6. Apakah data yang digunakan valid dan masuk akal?	☐	☐	☐
7. Apakah penggunaan tabel dan gambar membantu dalam penjelasan permasalahan dan pembahasan?	☐	☐	☐
8. Apakah pembahasan atau analisis berkaitan dengan hasil penelitian yang dihasilkan?	☐	☐	☐
9. Apakah acuan yang digunakan adalah relevan?	☐	☐	☐

	Sangat Baik	Baik	Cukup	Kurang
10. Kontribusi terhadap pengetahuan	☐	☐	☐	☐
11. Keaslian	☐	☐	☐	☐
12. Sistematis	☐	☐	☐	☐
13. Bahasa	☐	☐	☐	☐
14. Akurasi penulisan	☐	☐	☐	☐

B. Keputusan Reviewer

Makalah ini:	Ya	Tidak
i. Dapat diterbitkan secara langsung	☐	☐
ii. Dapat diterbitkan dengan sedikit perbaikan (*MINOR REVISION*)	☐	☐
iii. Dapat diterbitkan dengan banyak perbaikan (*MAJOR REVISION*)	☐	☐
iv. Harus dikembalikan kepada reviewer setelah dilakukan perbaikan	☐	☐
v. Apakah nama reviewer boleh disampaikan pada penulis?	☐	☐
vi. Layak masuk prosiding IOP Conference	☐	
atau JURNAL	☐	

FORMULIR EVALUASI MAKALAH

No. :

Judul Makalah : **Characteristics and Catalytic Activity of Zeolite-A Synthesized from Rice Husk Silica and Aluminium Metal by Sol-Gel Method**

A. Poin Evaluasi:

	Ya	Tidak	Periksa Kembali
1. Apakah isi makalah original?	☑	☐	☐
2. Apakah judul makalah mewakili isi keseluruhan makalah?	☑	☐	☐
3. Apakah intisari menggambarkan isi dari makalah?	☑	☐	☐
4. Apakah kata kunci mengindikasikan cakupan penelitian?	☑	☐	☐
5. Apakah pendekatan dan metodologi penyelesaian masalah dijelaskan dengan baik?	☑	☐	☐
6. Apakah data yang digunakan valid dan masuk akal?	☑	☐	☐
7. Apakah penggunaan tabel dan gambar membantu dalam penjelasan permasalahan dan pembahasan?	☑	☐	☐
8. Apakah pembahasan atau analisis berkaitan dengan hasil penelitian yang dihasilkan?	☑	☐	☐
9. Apakah acuan yang digunakan adalah relevan?	☑	☐	☐

	Sangat Baik	Baik	Cukup	Kurang
10. Kontribusi terhadap pengetahuan	☐	☑	☐	☐
11. Keaslian	☐	☑	☐	☐
12. Sistematis	☐	☑	☐	☐
13. Bahasa	☐	☑	☐	☐
14. Akurasi penulisan	☐	☑	☐	☐

B. Keputusan Reviewer

Makalah ini:	Ya	Tidak
i. Dapat diterbitkan secara langsung	☐	☐
ii. Dapat diterbitkan dengan sedikit perbaikan (*MINOR REVISION*)	☑	☐
iii. Dapat diterbitkan dengan banyak perbaikan (*MAJOR REVISION*)	☐	☐
iv. Harus dikembalikan kepada reviewer setelah dilakukan perbaikan	☐	☐
v. Apakah nama reviewer boleh disampaikan pada penulis?	☐	☑
vi. Layak masuk prosiding IOP Conference	☑	
atau JURNAL	☐	

C. Komentar mengenai makalah (silahkan menggunakan lembar tambahan jika diperlukan).

1. Introduction is very general and remove unwanted and known theory. Please include important research finding from the present research.
2. The introduction is also completely missing to address the proper research gap. The objective of the present research is not properly highlighted.
3. Similarity of this manuscript need to be checked by the software.
4. For production of biodiesel, this type of catalyst has been used to treat different raw materials, such as waste cooking oil [14], microalga oil [15], palm oil [12], triglycerides [16], butter [17]. (This sentence should be written as “For production of biodiesel, this type of catalyst has been used to treat different raw materials, such as waste cooking oil [14], microalga oil [15], palm oil [12], triglycerides [16], and butter [17].”
5. In the introduction of paragraph 5, linolic or linoleic?
6. Figure 4. shows the existence of 3 separate peaks in the chromatogram, …… (This sentence should be written as “Figure 4 shows the existence of 3 separate peaks in the chromatogram)
7. Please check the consistency in reference writing again.

Tanda tangan reviewer:

(Suharso)

D. Catatan dari editor