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EDITORIAL TEAM

PREFACE

This is the inaugural issue of JESR contains several articles in the field of engineering and applied science research. We would like to endow our grateful, for public attention, consideration and suggestions regarding the manuscript quality of this inaugural issue.

This issue contains 8 research articles on interdisciplinary works. Most of the articles are industrial research in the field of geophysical engineering, agriculture, chemical engineering, and applied sciences. A review paper in biomedical science is also included. We believe this issue, and forward, JESR will contribute in scientific and technology world as well as engineering developemnt and needs. During the processing of the publications, we received tremendous supports and services from the reviewers and all contributors, in which we expressed our appreciation and grateful.

We do hope this issue will provide some significant for the readers.

We are getting better, but we have to get better.

Best regards,

Editorial Boards

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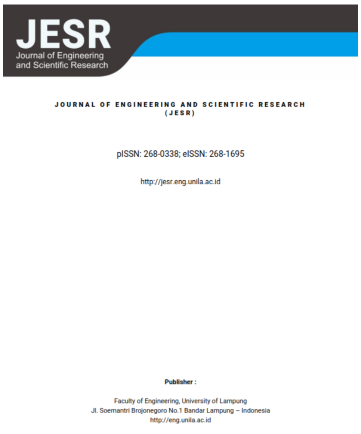
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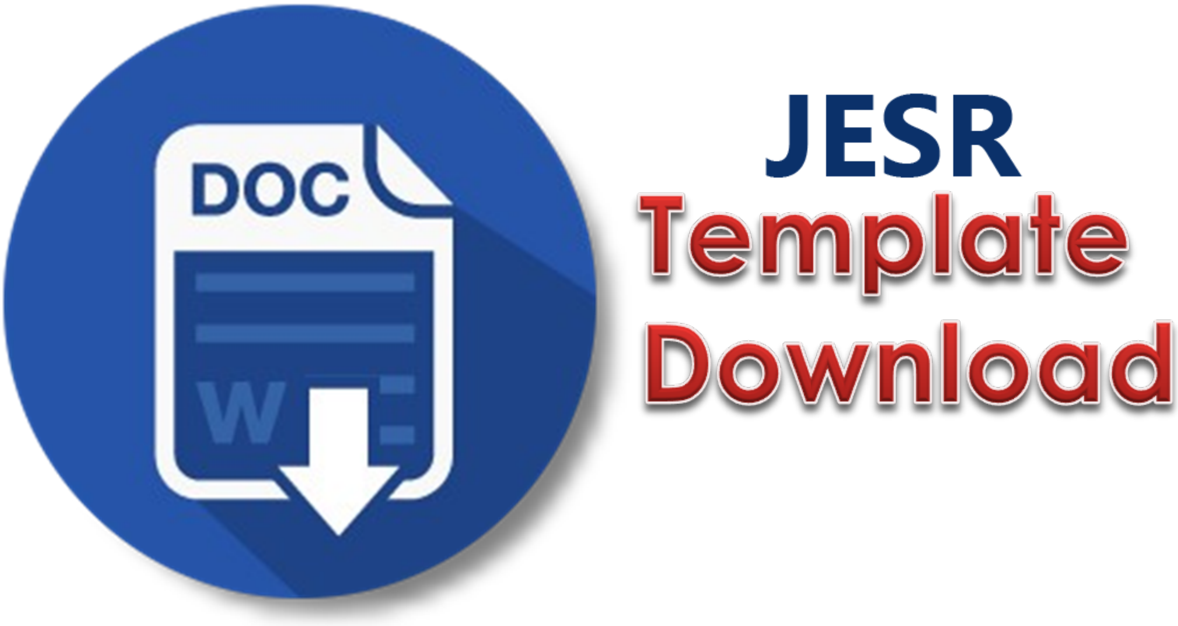

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INDEXING

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Reusability Study of Sulfated Zirconia Functionalized SBA-15 Catalyst for Biolubricant Oil Production from Oleic Acid

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Abstract

Sulfated zirconia functionalized SBA-15 catalyst (SZr-SBA-15) was prepared by reacting SBA-15 with Zirkoniumoxychloride and urea at 90°C to form ZrO_2-SBA-15. Then, ZrO_2-SBA-15 was reacted with H_2SO_4 at room temperature to produce SZr-SBA-15 catalyst. The catalyst was characterized in terms of adsorption-desorption nitrogen analysis, SEM-EDX and FTIR. Based on SEM-EDX and adsorption-desorption nitrogen analysis results, it was found that Zr had been incorporated in SBA-15. By using the SZr-SBA-15 catalyst, esterification reaction of oleic acid with TMP to produce biolubricant oil of Trimethylolpropanetrioleat achieved 85% oleic acid conversion and selectivity of 63,7%. Reusability study of SZr-SBA-15 catalyst was carried out for 3 rounds of reaction. It was found that the catalyst could be used up to 3 rounds without significant decrease in activity

Keywords: *biolubricant oil, catalyst reusability, sba-15, sulfated zirconia.*

I. Introduction

LUBRICANT oil based on petroleum industry has caused environment pollution. Besides that, the lubricant oil is made from unrenewable source and the source is limited. As such, biolubricant production based on renewable sources, for example: vegetables oil, has become the solution to overcome the problem. In addition, biolubricant oil gives other advantages such as cheap, biodegradable and no effect on environmental. Currently, the esterfication of oleic acid with trimethylolpropane (TMP) is used in the industry for the synthesis of biolubricant. This reaction is generally catalyzed by homogeneous acidic catalyst that usually need additional processes i.e. separation, neutralization and product purification [1]. Solid acid catalyst offers more advantages than homogeneous acidic catalyst in the process as it is no need the additional processes and reusable.

In this research, mesoporous SBA-15 has been incorporated to sulfated zirconia to produce solid acid catalyst that has thermal stability with large surface area and large pore size. This catalyst has been studied for esterification of cyclohexanol with acetic acid [2], fatty acid with methanol for biodiesel [3] and glycerol with fatty acid to monoglyceride [4]. It was reported that sulfated zirconia functionalized SBA-15 catalyst (SZr-SBA-15) gave high activity and high conversion caused by it large surface area. Various SZr-SBA-15 catalysts with different structures have been studied for reaction of oleic acid esterification with trimethylolpropane to produce biolubricant oil of trimethylolpropane trioleat (TMPTO) [1]. However, reusability study of SZr-SBA-15 for the oleic acid esterification has not been reported, in this study, reusability of SZr-SBA-15 was carried out up to three times for reaction of oleic acid esterification with trimethylolpropane to produce the biolubricant oil.

II. Materials and Methods

A. Preparation of SBA-15

The synthesis of SBA-15 was prepared according to the method as described in literature [4-5]. Four grams of pluronic as a template was dissolved in 30 ml distillated water and 120 ml HCl 2M at room temperature. Then, the solution was heated to 60°C on a hotplate. TEOS as silica source was added to the solution and stirred for 30 min with stirring rate of 750 rpm. When the colour of the solution change to white, the stirring rate was decreased to 300 rpm and kept this condition for further 20 h. The solution then was transffered to Erlenmeyer and aged at 80°C for 48 h in a waterbath. After that, the solid product was filltered, washed with distilleted water and dried in an oven at 120°C for 12 h. Calcination was carried out in a furnace at 550°C for 6 h to produce SBA-15.

B. Preparation of SZr-SBA-15

SZr-SBA-15 catalyst was prepared by using a method as describe in literature [2] except the amount of sulfated zirconia. Four grams of SBA-15 (heated in oven at 120°C for 4 h) was added to solution of 0.5508 g zirconium oxychloride, 1.083 g of urea and 120 ml distillated water. The mixture was stirred and refluxed at 90°C for 6 h to form ZrO_2-SBA-15. The mixture was filltered, washed, dried at 100°C for 24 h in an oven and calcined in a furnace at 550°C for 6 h. The solution of H_2SO_4 was added to ZrO_2SBA-15 at room temperature to form SZr-SBA-15. Finally, the solid product was filltered, dried at 110°C and calcined at 550°C for 3 h in a furnace.

C. The Reusability Test of SZr-SBA-15 catalyst for the Esterification of Oleic Acid with TMP

The reaction was performed in a necked flat bottom flask equipped with water separator and thermometer on a stirring hotplate. Oleic acid and Trimethylolpropane (TMP) with molar rasio 3:1 were placed in the flask, then, 5% w/w catalyst (based on weight of oleic acid) was added to it. The reaction was taken place at 150°C for 5 h with stirring rate 750 rpm. After the reaction was completed, the samples of product were taken out and filtered to take the catalyst. The former catalyst was washed with hexane solution to remove the oil that still stack on the catalyst and dried at 80°C for 24 h in an oven. The former catalyst then used to the same procedure of esterification (reusability) up to 3 times reaction.

III. Results and Discussions

A. Characterization of Catalyst.

The result of N_2 adsorption-desorption analysis are shown in Fig.1. Based on the result, the adsorption-desorption isotherms curves for SBA-15 and SZr-SBA-15 had the same type, a curve that shown the mesostructure of the material. It is shown that the process of SZr incorporation in SBA-15 did not change the mesostructural characteristic.

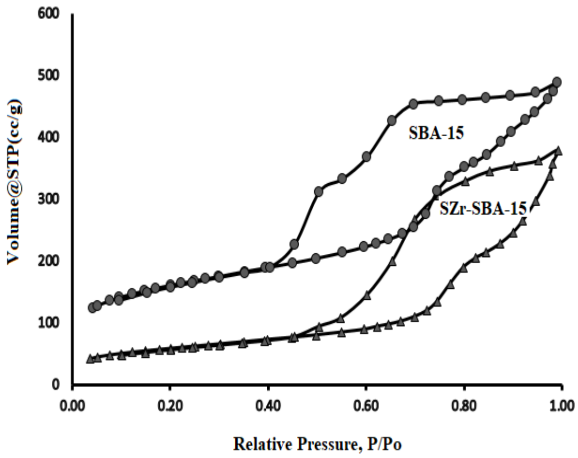

Figure 1. Adsorption/desorption curves of SBA-15 and SZr-SBA-15

The properties such as surface area, pore volume and pore size of SBA-15 and SZr-SBA-15 are shown in Table 1.

Table 1. The Properties of Materials

No	Materials	Surface Area (m^2/g)	Pore Volume (cc/g)	Pore Size (Å)
1	SBA-15	672.097	0.795	38.584
2	SZrSBA15	413.336	0.693	65.781

The pore size and surface area for SBA-15 are 529.219 m^2 g^{-1} dan 38.584 Å, it was comparable to a previous publication [4,6] suggesting that SBA-15 has surface area with range 500-1500 m^2 g^{-1} and pore size 20-500 Å. Besides that, the pore size increase after the incorporation, indicated that SZr is mainly incorporate into the pore of SBA-15. The increasing of pore size was caused by silica dissolution reaction when the incorporation taken place [7].

SEM image of SBA-15 and SZr-SBA-15 are shown in Fig 2. All of SEM images shown the cylinder hexagonal forms as suggested in the literature [5].

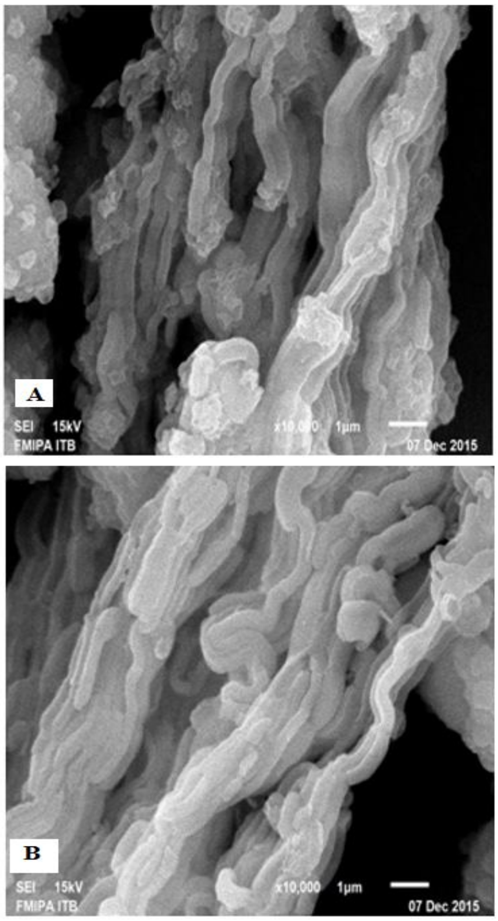

Figure 2. SEM Images of a) SBA-15 ; b) SZr-SBA-15

Incorporation of sulfated zirconia in SBA-15 was monitored by EDX spectrum. According to the analysis result, there were Zr peaks in SZr-SBA-15 spectrum as shown in Fig 3 at around 0.5 and 2 keV. The Zr peak location were agreed as reported in the literatures [Bhanjana,2016]. This indicated that sulfated zirconia could be successfully incorporated in SBA-15.

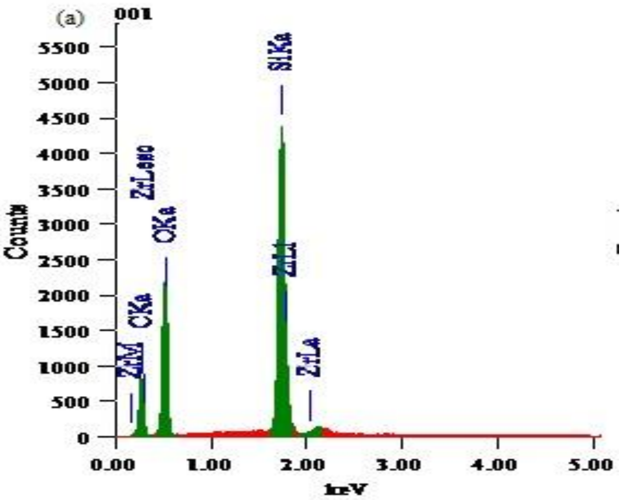

Figure 3. EDX Spectrum SZr-SBA-15

The presence of C-SZr bond in SBA-15 was also confirmed by FTIR Spectrum as shown in Fig. 4. The vibration at 800 cm^{-1} to 1260 cm^{-1} are attributed to Si-O-Si bond that presented in SBA-15 while the vibration at 613 cm^{-1} is related to C-SZr bond.

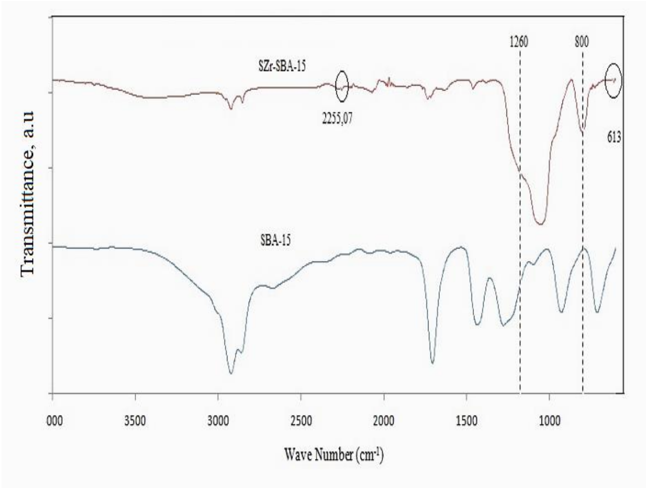

Figure 4. FTIR Spectrum of SBA-15 and SZr-SBA-15

B. Esterification of Oleic Acid with TMP and The Reusability .

Conversion and selectivity of TMPTO was determined by GC-MS analysis. Based on data in Figure 5, it shows that esterification of oleic acid with TMP using SZr-SBA-15 offered 85,9% conversion with 63,7% selectivity. This conversion did not decrease significantly in the second cycle (83,3%) and third cycle (82,02%) of usage in the esterification to produce TMPTO. It was shown that SZr-SBA-15 was the effective catalyst for the esterification of oleic acid with TMP to produce biolubricant of TMPTO and also had the ability to be reused. The decreasing of selectivity would be caused by carbon that closed the pore of catalyst during the reaction.

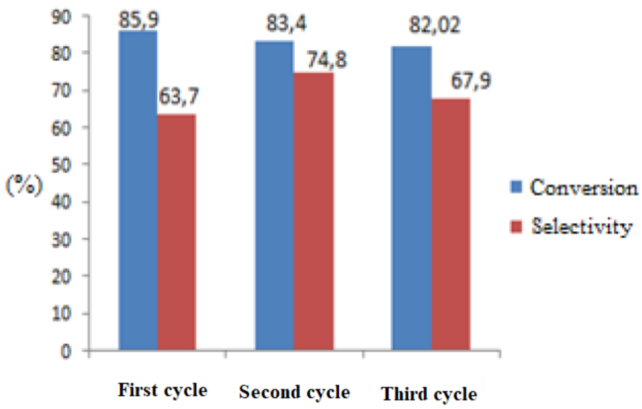

Figure 5. Oleic acid conversion and TMPTO Selectivity

IV. Conclusion

We have shown that the synthesis of catalyst SZr-SBA-15 has successfully based on N_2 adsorption-desorption analysis and SEM-EDX analysis results. Sulfated Zirconia functionalized SBA-15 was efficient catalyst for esterification of oleic acid with trimethylolpropane to produce TMPTO and gave conversion more than 85% and selectivity more than 60%. The catalyst also can be reused up to 2 times in the esterification without significant decrease in activity.

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