

# Synthesis and Reusability Study of Sulfated Zirconia Functionalized SBA-15 for Esterification of Oleic Acid to Produce TMPTO as Biolubricant

Fully Resha Rangganita, Lilis Hermida\*, Archealin Angraeni, and Destiara Khoirunnisa

Department of Chemical Engineering, Universitas Lampung, Lampung, 35145, Indonesia

corresponding author : \*lilis.hermida@eng.unila.ac.id

**Abstract.** The synthesis of SZr-SBA-15 catalyst was prepared by using urea hydrolysis method. SBA-15 incorporated with sulfated zirconia was prepared by reacting SBA-15 with Zirconiumoxychloride and urea at 90°C to form ZrO<sub>2</sub>-SBA-15. Then H<sub>2</sub>SO<sub>4</sub> was added to ZrO<sub>2</sub>-SBA-15 at room temperature to form SZr-SBA-15. This SZr-SBA-15 catalyst was used to synthesize biolubricant oil via esterification of oleic acid with TMP. The catalyst were characterized in terms of adsorption-desorption nitrogen analysis, SEM-EDX, FTIR and biolubricant oil was characterized by FTIR analysis. Based on SEM-EDX and adsorption-desorption nitrogen analysis result, it was found that Zr had been incorporate in SBA-15. The SZr-SBA-15 catalyst was an efficient solid catalyst for esterification of oleic acid with TMP with high conversion (85,9%) and selektivty (63,7%) to Trimethylolpropanetrioleat. From the study of reusability, it was found that SZr-SBA-15 catalyst could be used up to 3 rounds reaction without significant decrease in activity.

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

## A. INTRODUCTION

Lubricant oil based on petroleum industry has caused enviroment pollution. Beside that, the lubricant oil is unrenewable technology and the source is limited. The searching for using alternative source has begun. Biolubricant production based on vegetables oil has become the solution to overcome the problem. Biolubricant oil gives the various advantages that is the source of renewable, 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. However, the solid acid catalyst has offer more advantages in the process. It is no need additional separation, netralization and purification steps of the final product.

In this research, mesoporous silica SBA 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 (garg et al, 2009), fatty acid with metanol for biodiesel (Chen et al, 2007) and glycerol with fatty acid to monoglyceride (Hermida et al, 2010). Those research suggested that sulfated zirconia functionalized SBA-15 as a catalyst gaves high activity and high converssion caused by it large surface area. However, SZr-SBA-15 for esterification of oleic acid with trimethylolpropane to trimethylolpropane trioleat (TMPTO) and the study of the catalyst reusability has not been reported so far. Herein, we studied the effectiveness of catalyst in esterification of oleic acid with TMP and it reusability.

## B. METHODE

**Preparation of SBA-15.** The sinthesis of SBA-15 in this paper was prepared according to the methode as described in literature (Zhao et al, 1998 ; Hermida et al, 2010). Four grams of pluronic as a template was dissolved in 30 ml destillated 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 conditons for futher 20 h. The solution then was transferred to erlenmeyer and aged at 80°C for 48 h in a waterbath. After that, the solid product was filtered, 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.

**Preparation of SZr-SBA-15.** SZr-SBA-15 catalyst was prepared by using hydrolysis method as describe in literature (garg et al, 2009) except the amount of sulfated zirconia that was incorporated in SBA-15. 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<sub>2</sub>-SBA-15. The mixture was filtered, 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<sub>2</sub>SO<sub>4</sub> was added to ZrO<sub>2</sub>SBA-15 at room temperature to form SZr-SBA-15. Finally, the solid product was filtered, dried at 110°C and calcined at 550°C for 3 h in a furnace.