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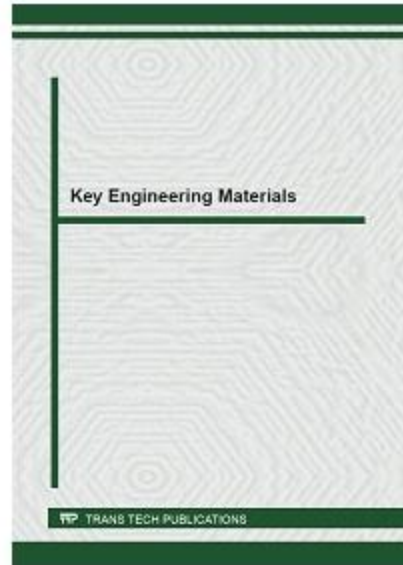
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The Formation of Local Catalyst From Zeolite, Characterization and Performance in the Reaction

Zahrul Mufrodi, Erna Astuti^{1,a} and Gita Indah Budiarti¹

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Keywords: Catalyst, zeolite, activity, density, SEM and XRD

Abstract. The existence of catalyst in a chemical reaction is very important. This catalyst can accelerate the chemical reaction. Currently there are local catalyst with the performance as good as commercial catalyst. One of those catalyst is zeolite. This paper determines the study of characterization of zeolite using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD), and explains the performance of zeolite in the synthesis of triacetin. Catalyst from zeolite was made by mixing with H₂SO₄ in various concentrations, neutralization and drying. From morphology analysis by using Scanning Electron Microscope (SEM), density and X-Ray Diffraction (XRD) obtain that mixing with H₂SO₄ of 0.5M is better than the others. From the study of catalyst synthesis from natural zeolite it can be concluded that natural zeolite before activation has fragile structure meanwhile after being activated it was formed crystal structure. The best zeolite crystal phase was obtained from zeolite activated by using 0,5 M H₂SO₄ with glycerol conversion of 94.45% and triacetin selectivity of 4.01%.

Introduction

Catalyst has important role in accelerating chemical reaction. Currently, there are many relatively high price catalysts. Solid catalysts are widely used for liquid or gas reactant. Reaction with gas or liquid reactant and solid catalyst is a good option because the product is easily separated from the catalyst. To fulfill the catalyst demand it is necessary to develop alternative catalyst with relatively cheaper price. Natural zeolite is a solution which can be utilized as catalyst, besides its abundant availability it is also relatively cheap compare to commercial catalysts.

Zeolite is a hydrated alumina silicate crystalline with three dimensional open structure which can be prepared to increase its activity [1]. Zeolite has function as catalyst due to its surface characteristic. In this study it is expected that this research make zeolite catalyst and used it for heterogeneous reaction.

Catalyst. Catalyst is a substance that increases the rate of a chemical reaction without itself undergoing any permanent chemical change and does not affect equilibrium of reaction. In industry catalyst has been widely used, especially in chemical industry. Based on its phase, catalysts are classified as homogeneous and heterogeneous catalyst. Homogeneous catalyst is a catalyst which has the same phase as the reactant. Heterogeneous catalyst is a catalyst which has different phase from the reactant. Homogenous catalyst works through interaction of reactant particles to transitional state. Furthermore transitional state merge with the other reactants to form product, after the product is formed the catalyst regenerate into pseudo substance. Heterogeneous catalyst is commonly in a form of solid which can be used for gas or liquid reactant and the reaction occur on catalyst surface. The materials used as heterogeneous catalyst is a precious metal such as platinum Rhodium and Palladium. However because it's limited amount and high prices thus it limit the usage [2]. Therefore study to produce catalyst from relatively cheap raw natural material is necessary without neglecting the quality of the catalyst.

The performance of catalyst to increase the reaction rate is affected by various factors. The factors affecting catalyst performance are physical and chemical properties of catalyst; operating condition such as temperature, pressure, flow rate, contact time; feed type; the type of supporting solid used. The catalyst prepared with different method will give different activity and selectivity. The ability of

catalyst in accelerating the reaction rate is affected by several factors. The factors affecting the catalyst performance are physical and chemical properties; operating condition such as temperature, pressure, flow rate, contact time; feed type; and the type of supporting solid used. The catalyst prepared with different method will give different activity and selectivity [3].

Zeolite as Catalyst. Zeolite is silica-alumina crystal material which has three dimensional polymer arrangement consists of tetrahedral unit SiO_4 and AlO_4^- , which merge through oxygen sharing. Zeolite structure can be seen on Fig. 1. Based on its solid structure which has pores and hollow and also has high thermal stability, zeolite-like material can be functioning as catalyst metal support or adsorbent [4].

Zeolite can be used direct waste absorbent. However, to increase the zeolite absorbance ability, activation is necessarily to be conducted. Activation process can be conducted through acidification [5]. Zeolite also has micro porous structure so that it can provide wide spaces for reaction to occur in higher pressure. High Si/Al ratio in zeolite make it hydrophobic-organophilic which will promote reactant diffusion process. Zeolite molecular dimension also makes zeolite selective toward reactant, product and transitional condition.

Triacetin. Glycerol can be made of triacetin and trinitroglycerin [6-10]. Triacetin as bio additive can be synthesized by using glycerol and acetic acid [11]. Various solid and liquid catalyst can be used in this reaction to increase the rate of reaction. Solid catalysts which has been used were Fe-Sn-Ti(SO_4^{2-}) [12], Zr-Natural Zeolite [13], and silica alumina [14], sulfuric acid [15-17]. This research used zeolite as heterogeneous catalyst in the formation of triacetin. So this paper determines the study of characterization of zeolite using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD), and explains the performance of zeolite in the synthesis of triacetin.

Methods

Catalyst was made by reduce the size up to 200 mesh, then mixed with H_2SO_4 solution for 4 hours in three neck flask equipped with condensor in the temperature of 90°C . It was then separated by using filter. The solid was neutralized with distilled water and dried by using oven. It was then decomposed by adding NaOH, stirred for 30 minutes and washed in order to make neutral. The result which will be analyzed to find out surface morphology using Scanning Electron Microscope (SEM), and to know crystalline structure using X-ray Diffraction (XRD). The density of catalyst before and after activation was also be measured. The characteristic of activated zeolite catalyst was used for triacetin formation from glycerol and acetic acid. The analysis of triacetin was conducted by using Gas chromatography–mass spectrometry.

Result and Discussion

Characteristic results of zeolites were analyzed using scanning Electron Microscope (SEM), X-Ray Diffraction (XRD) and density analysis.

Scanning Electron Microscope (SEM) Analysis. The result of morphology of catalyst surface before and after activation is shown on Fig. 1 as follow.

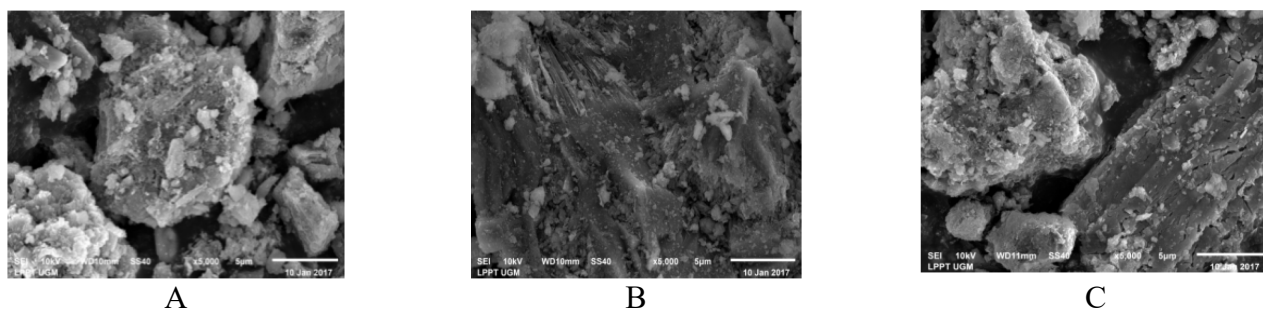


Fig. 1. SEM Analysis on a. Natural Zeolite without activation, b. Natural Zeolite activated with 0,5 M H_2SO_4 , c. Natural Zeolite activated with 1 M H_2SO_4

The result of zeolite identification by using Scanning Electron Microscope (SEM) is shown on Fig. 1 with zeolite morphology in a form of fragile porous crystal. The activated zeolite was harder and denser, as shown on Fig. 1.b and 1.c. comparatively activated zeolite with 0.5 M H_2SO_4 is stronger and has more pores than activated zeolite by using 1 M H_2SO_4

X-Ray Diffraction (XRD) Analysis. The result of X-ray Diffraction (XRD) Test before and after activation is shown on Fig. 2 as follow.

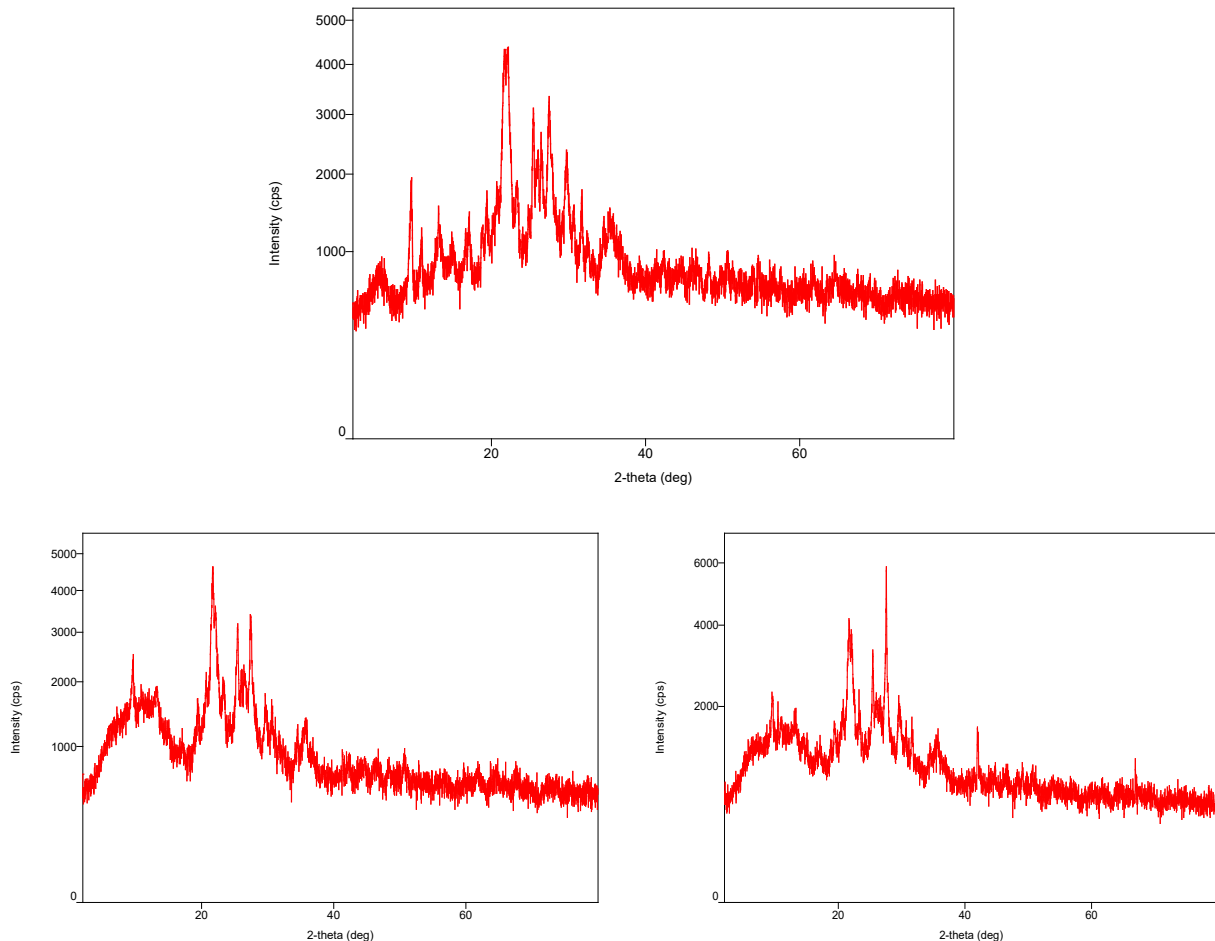


Fig. 2. Zeolite Cristal Structure : a. Natural Zeolite without activation, b. Natural Zeolite activated with 0,5 M H_2SO_4 , c. Natural Zeolite activated with 1 M H_2SO_4

Fig. 2. shows diffractogram as the analysis result by using XRD which shows that amorph phase was more dominant on non-activated zeolite and the crystal phase increased after activation. Amorphous phase can be seen from its peak in of 2 theta area around 0° - 20° . Meanwhile crystal phase can be seen on Fig. 2b and 2c indicated by the appearance of diffractogram peaks in 2 theta area around 21° - 31° . Besides it was indicated by a greater number of picks in its crystal structure. From three charts on Fig. 2 both a and c there is similarity of the existence of amorphous shape in the area around 0° - 20° and this figure is not shown on Fig. 2b. Fig. 2b shows crystal pikes formed in are around 0° - 20° .

Density Analysis. For catalyst density test it can be determined from the number of pores formed in which strong crystal structure yet has many pores. The result of density test before and after activation is shown on Table 1.

Table 1. Density of zeolite with and without activation

No	Activated zeolite using H_2SO_4	Density
1.	Before activation	20.0
2.	Activated 0.5M	40.5
3.	Activated 1M	40.0

Table 1 shows that low density was obtained from zeolite with activation of 0.5 M, which mean that pores structure is more prominent in this activation. It acentuated the result of morphology analysis by using SEM and structure analysis by using SEM and crystal analysis by using XRD.

The Role of Catalyst in the Triacetin Formation. Activated zeolite was tested in the triacetin formation with the result as shown in Table 2 as follow.

Table 2. The effect of catalyst on conversion and selectivity, monoacetin, diacetin dan triacetin

Zeolite catalyst	Glycerol conversion, %	selectivity, %		
		Monoacetin	Diacetin	Triacetin
Before activation	13.61	91.73	8.27	-
Activated 0,5 M H ₂ SO ₄	94.45	50.32	45.67	4.01
Activated 1 M H ₂ SO ₄	70.82	60.45	39.44	0.11

Table 2 exhibit that catalys usage in btriacetin bioaditif synthesizing has good effect compare to without catalyst. The usage of zeolite with the activation using 0.5M H₂SO₄ was better than zeolite withouth activation and activated 1M H₂SO₄ for both glycerol conversion (94.45%) and triacetin selectivity (4.01%).

Triacetin can be used to improve the performance of biodiesel. The previous study used homogeneous catalyst in syntesis of biodiesel [18,19]. Gnanaprakasam et al. [20] stated that CaO, CaTiO₃, CaZrO₃, CaO–CeO₂, CaMnO₃, Ca₂Fe₂O₅, Al₂O₃/KI, ETS-10, zeolite, alumina/silica-supported K₂CO₃ can be used as heterogeneous base catalyst in the production of biodiesel from waste cooking oil[20]. This research will be continued with the use of zeolite as local catalyst on the synthesis of biodiesel from waste cooking oil.

Summary

From the study of catalyst synthesis from natural zeolite it can be concluded that natural zeolite before activation has fragile structure meanwhile after being activated it was formed crystal structure. The best zeolite crystal phase was obtained from zeolite activated by using 0,5 M H₂SO₄ with glycerol conversion 94.45% and triacetin selectivity 4.01%.

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