

## UTILIZATION OF NATURAL ZEOLITE AS AN ADSORBENT IN ETHANOL PURIFICATION

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### ABSTRACT

Research about application of natural zeolite (ZA) as adsorbent to absorb water in ethanol has been started. The purpose is, to evaluate the ability of ZA that have been activated in order to absorb water in ethanol. The activator that used is H<sub>2</sub>SO<sub>4</sub> 1N and HCl 1N solution. ZA crushed and sieved using a sieve 60, 80, and 100 meshes. A total of 35 grams of sifted ZA mixed with the activator and stirred for 30 minutes and then precipitated for 24 hours. Residue washed until neutral and heated using a furnace at a temperature of 400°C for 2 hours. ZA which has activated used to adsorb the water content in ethanol having a concentration of 60 %, 65 %, 70 %, and 80 %. The results showed that the activated zeolite by using H<sub>2</sub>SO<sub>4</sub> 1N solution to absorb water content in the ethanol have the efficiency until 19.7 %. However, the adsorption of water content in ethanol which has more than 60 % concentration, ethanol tends to adsorbed by ZA so that the water content in ethanol to be slightly higher compared with ethanol before adsorption.

**Keywords:** Adsorben, zeolit, water content, activated, adsorbtion.

### 1. INTRODUCTION

Ethanol derived from biomass has been proven to be a viable alternative energy source to replace fossil fuels. However, a common issue is that high-purity ethanol requires costly distillation processes. Additionally, ethanol purification through distillation typically yields ethanol with a purity of 96% (Harjono, 2004). To address this challenge, natural zeolite (ZA), which possesses adsorbent properties, can be used. Research on water adsorption in bioethanol has resulted in ethanol with high purity. Further studies can be conducted to support bioethanol purification to achieve purity levels of >99.95% (Dwi Karsa AR, 2007).

Ethanol purification using zeolite is based on the principle of surface adsorption; the larger the zeolite surface area, the greater its adsorption capacity. Natural zeolite (ZA) is a group of minerals with very small pores, typically found filling cracks or crevices in rocks. To date, there are over 150 types of synthetic zeolites. In nature, zeolite is formed from volcanic ash and eruption materials (D. Novitasari et al., 2012). Additionally, zeolite also results from volcanic activity deposits, rich in silica content. The pore structure of zeolite is shown in Figure 1.

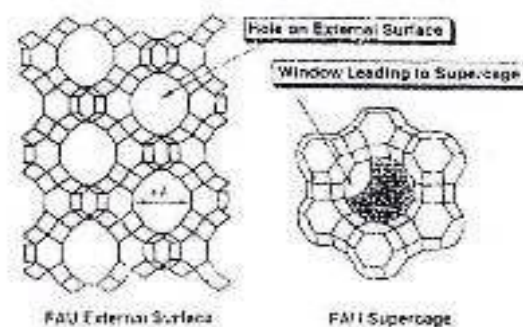


Figure 1. Pore Structure in Zeolite (Sari, 2006)

Natural zeolite is found in various provinces in Indonesia, such as Lampung, West Java, Central Java, Yogyakarta, and Aceh. Currently, ZA is only used in limited industrial applications, making it relatively inexpensive. The use of zeolite minerals is increasingly growing in large-scale industrial applications. In developed countries, such as the United States, zeolite is fully utilized in industry. Due to its unique properties, zeolite is used in various fields, including as a material for industrial waste treatment. Natural zeolite has several properties, including dehydration, adsorption, ion exchange, catalysis, and separation (Dian Kusuma, 2010).

HCl and H<sub>2</sub>SO<sub>4</sub> have been widely used in purifying adsorbent materials, such as for activating carbon and zeolite. These solutions help remove impurities that clog the pores of adsorbents. This study aims to determine the most effective activation solution between H<sub>2</sub>SO<sub>4</sub> and HCl for activating ZA by assessing water absorption efficiency in ethanol. It also considers the effect of ZA particle size on its water absorption capability in ethanol.

This research focuses on the initial processing of ZA using activation methods. The activation of ZA using two types of solutions, HCl and H<sub>2</sub>SO<sub>4</sub>, both at a 1N concentration, is expected to expand the surface area of ZA, allowing for maximum adsorption to occur.

## **2. RESEARCH OBJECTIVES, BENEFITS AND LIMITATIONS**

The primary objectives of this research are to evaluate the effectiveness of HCl and H<sub>2</sub>SO<sub>4</sub> solutions in activating natural zeolite (ZA) for enhanced water adsorption in ethanol purification, assess ZA's water absorption efficiency, examine the effect of particle size on water absorption, and ultimately optimize ethanol purification to achieve a purity level greater than 99.95%. This research could contribute significantly to sustainable energy production by utilizing biomass-derived ethanol as a renewable fuel alternative to fossil fuels. Moreover, using ZA for ethanol purification could reduce the costs associated with traditional distillation methods, enhancing the economic feasibility of ethanol production. The study also promotes the industrial value of ZA, potentially increasing its demand and expanding its applications in various sectors. However, certain limitations must be considered. This research focuses specifically on HCl and H<sub>2</sub>SO<sub>4</sub> activation solutions at a single concentration level, which may restrict the scope of the findings. Additionally, results obtained in the lab may not directly scale to industrial applications, making further studies necessary for large-scale implementation. While ZA is currently affordable, scaling its use in ethanol purification may require additional economic

analysis. Finally, the acid activation process itself may introduce environmental considerations, as it could generate waste products and necessitate added safety protocols, impacting the overall environmental footprint of this purification method.

## **3. RESEARCH METHODS**

This research was conducted in the Process Unit Laboratory of the Chemical Engineering Department at the Lhokseumawe State Polytechnic.

### **3.1 Materials**

Materials that used in this research are natural zeolite (ZA) stones, 1.0 N H<sub>2</sub>SO<sub>4</sub> solution, 1.0 N HCl solution, and Ethanol solutions with varying concentrations (60%, 65%, 70%, 75%, and 80%)

Equipment: Beaker glass, filter paper, porcelain crucible, furnace, desiccator, and refractometer.

### **3.2 Methodology**

#### **1. Mixing Stage of Natural Zeolite (ZA) with 1.0 N H<sub>2</sub>SO<sub>4</sub> and HCl Solution**

The ground ZA was weighed to 35 grams and mixed with 150 ml of 1.0 N H<sub>2</sub>SO<sub>4</sub> solution in a beaker. The mixture was stirred for 2 hours until homogenous and then allowed to settle for 24 hours. It was then filtered using filter paper and washed to a neutral pH.

#### **2. Activation Stage of Natural Zeolite**

The filtered sample was placed in a porcelain crucible, and activation was performed in a furnace for 2 hours at a temperature of 200°C. The sample was then cooled in a desiccator and weighed after cooling. ZA was then prepared for testing.

#### **3. Refractive Index Analysis**

The refractive index of ethanol was analyzed using a refractometer. Several ethanol concentrations were also analyzed for refractive index calibration, enabling the determination of ethanol concentration in the adsorbed samples.

### 3.3 Sample Preparation

#### 1. Adsorbent Preparation Stage

ZA stones were ground to particle sizes of 60, 80, and 100 mesh.

#### 2. Variation of Ethanol Solutions for Testing

ZA samples prepared from the activation stage were tested for water absorption in ethanol solutions of different concentrations: 60%, 65%, 70%, 75%, and 80%.

### 4. RESULTS AND DISCUSSION

The ethanol purification process using natural zeolite (ZA) is based on the principle of surface adsorption. This study examines the effects of the type of activation solution and the particle size of ZA on the water adsorption capacity in ethanol. The aim of this research is to test the ability of activated ZA to adsorb water contained in ethanol. The use of 1 N HCl and H<sub>2</sub>SO<sub>4</sub> solutions is intended to remove impurities within the ZA material.

The adsorption process of ZA on water in ethanol is conducted at room temperature

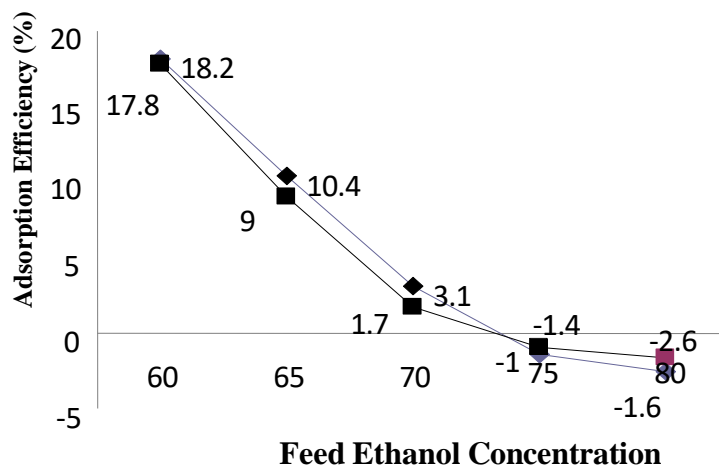
with a stirring time of 30 minutes. The efficiency of ethanol absorption is calculated based on the initial ethanol concentration and the concentration after the process.

#### Effect of Ethanol Concentration on Adsorption Efficiency by 60 Mesh Active Zeolite

The adsorption of ethanol using 60 mesh ZA with two types of activation solutions, namely H<sub>2</sub>SO<sub>4</sub> and HCl at a concentration of 1.0 N, has been carried out. The variation of these two activation solutions aims to compare the adsorption capacity of ZA activated in acidic and basic conditions. The results of the study can be seen in Figure 2. Based on Figure 2, the ZA activated with H<sub>2</sub>SO<sub>4</sub> can adsorb slightly more water in ethanol compared to ZA activated with HCl. At an input solution concentration (ethanol-water) of 60%, the adsorption efficiency of ZA activated with H<sub>2</sub>SO<sub>4</sub> is 18.2%.

**Table 1.** Ethanol concentration after the adsorption process

Activation Solution	Activation Solution	Activation Solution			Activation Solution		
Particle Size (mesh)	60	60	60	100	60	80	100
H <sub>2</sub> SO <sub>4</sub>	60	70.9	70.9	71.4	18.2	18.2	19.1
	65	71.8	71.9	72.1	10.4	10.7	10.9
	70	72.2	72.3	72.6	3.1	3.3	3.7
	75	73.9	73.7	73.7	-1.4	-1.7	-1.7
	80	78.1	77.9	77.8	-2.6	-2.6	-2.7
HCl	60	70.7	70.7	70.9	17.8	17.9	18.2
	65	70.8	70.8	70.9	9	9	9.14
	70	71.2	71.4	71.6	1.7	2.1	2.3
	75	74.3	74.1	73.9	-1	-1.3	-1.41
	80	78.72	78.17	77.94	-1.6	-2.58	-2.6



**Figure 2. The effect of ethanol concentration on adsorption efficiency by 60 mesh active zeolite. Activation with H<sub>2</sub>SO<sub>4</sub> (◆), Activation with HCl (■).**

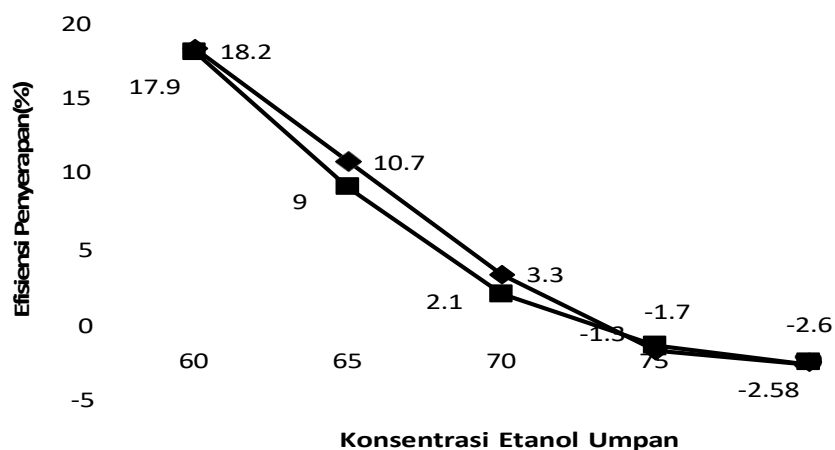
The ZA activated with HCl can absorb water in ethanol by 17.8%. At a feed solution concentration (ethanol-water) of 65%, the adsorption efficiency of ZA activated with H<sub>2</sub>SO<sub>4</sub> is 10.4%, while the efficiency of ZA activated with HCl is 9%. In the adsorption test using a feed solution concentration (ethanol-water) of 70%, the water adsorption efficiency in ethanol for ZA activated with H<sub>2</sub>SO<sub>4</sub> is 3.1%, whereas for ZA activated with HCl, the efficiency is 1.7%.

From the perspective of the feed solution concentration (ethanol-water), as the concentration of the feed solution increases,

the adsorption efficiency by ZA tends to decrease. This is because the water content in the feed solution (ethanol-water) is lower, making it more difficult for ZA to adsorb it.

#### **Effect of Ethanol Concentration on Adsorption Efficiency by 80 Mesh Active Zeolite**

The results of the adsorption study using 80 mesh ZA with two types of activation solutions, H<sub>2</sub>SO<sub>4</sub> and 1.0 N HCl, are presented in Figure 3.



**Figure 3. The effect of ethanol concentration on adsorption efficiency by 80 mesh active zeolite. Activation with H<sub>2</sub>SO<sub>4</sub> (◆), Activation with HCl (■).**

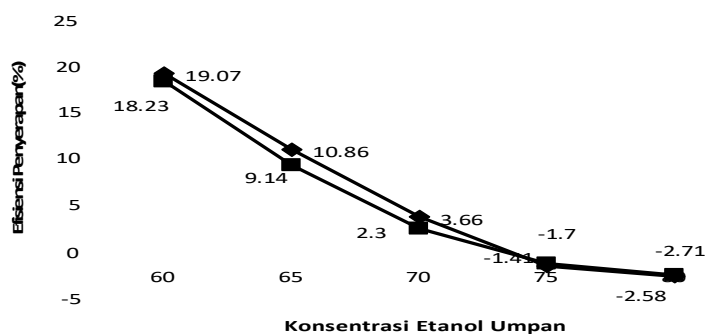
Based on Figure 3, the ZA activated with H<sub>2</sub>SO<sub>4</sub> is slightly better at adsorbing

water in ethanol compared to the ZA activated with HCl. This is similar to the

adsorption results for the 60 mesh particle size ZA discussed in the previous section. At a feed solution concentration (ethanol-water) of 60%, the adsorption efficiency of ZA activated with H<sub>2</sub>SO<sub>4</sub> is 18.2%, while ZA activated with HCl can absorb water at a rate of 17.9%.

In contrast, using a feed solution (ethanol-water) concentration of 70% results in an adsorption efficiency of 3.3% for ZA activated with H<sub>2</sub>SO<sub>4</sub>, while ZA activated with HCl shows an adsorption efficiency of 2.1%. In this experiment, the water absorption efficiency in ethanol by the 80 mesh particle size ZA tends to be better compared to the 60 mesh particle size ZA.

#### **Effect of Ethanol Concentration on Adsorption Efficiency by 100 Mesh Active Zeolite**



**Figure 4.** The effect of ethanol concentration on adsorption efficiency by 100 mesh active zeolite. Activation with H<sub>2</sub>SO<sub>4</sub> (◆), Activation with HCl (■).

The ZA activated with H<sub>2</sub>SO<sub>4</sub> is slightly better at adsorbing water in ethanol compared to ZA activated with HCl. However, the difference in adsorption capacity between ZA using these two types of activation solutions is not very significant.

The surface area of the adsorbent has a significant effect on its adsorption capacity; the larger the surface area of the adsorbent, the greater its adsorption capacity. The use of 100 mesh ZA can absorb water in ethanol up to 19.7%, compared to 18.2% for both 60 mesh and 80 mesh ZA.

At feed solution concentrations (ethanol-water) of 75% and 80%, the zeolite is less effective in absorbing the water content present in ethanol. In this study, the

The results of the study using 100 mesh ZA activated with H<sub>2</sub>SO<sub>4</sub> and 1.0 N HCl for adsorbing water in ethanol are shown in Figure 4. Similar to the findings for 60 mesh and 80 mesh particle sizes, the ethanol concentration obtained from adsorption using 100 mesh zeolite with H<sub>2</sub>SO<sub>4</sub> activation demonstrates better adsorption efficiency compared to the 60 mesh and 80 mesh ZA, achieving a maximum absorption efficiency of 19.07% from the 60% ethanol feed. In contrast, the adsorption efficiency with HCl activation is 18.23% from the 60% ethanol feed. As with the larger particle sizes (60 mesh and 80 mesh), the adsorption efficiency tends to decrease when using higher ethanol concentrations.

ethanol concentration after adsorption is observed to be lower than the concentration before adsorption.

The phenomenon of increased water content in ethanol after the adsorption process can be explained as a natural occurrence. According to the research by Silvia and Darmawan (2008), ZA is capable of absorbing ethanol in the solution, which can lead to an increase in the water concentration in ethanol. Generally, the use of ZA for purifying ethanol is less effective due to the characteristics of ZA itself. The use of synthetic zeolite (4A) could be a consideration for ethanol purification, as per the study conducted by D. Novitasari et al.

(2012), which showed an increase in ethanol concentration up to 86.36%.

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## CONCLUSION

Based on the results of the research conducted, it can be concluded that H<sub>2</sub>SO<sub>4</sub> solution is generally more effective as an activation solution for ZA adsorbent compared to HCl solution. The 100 mesh particle size of ZA tends to provide better water adsorption compared to the 60 mesh and 80 mesh ZA. Additionally, ZA is less effective as a water adsorbent in ethanol.

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