Zeolite Impregnated with Ag as Catalysts for Glycerol Conversion to Ethanol Assisted by Ultrasonic

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Abstract. The objectives of this study are to investigate the effectiveness of activated zeolite with and without impregnated Ag on the conversion of glycerol to ethanol. Zeolite catalyst used in this study was synthesized zeolite from silica extracted from local sand and glycerol conversion using zeolite with and without impregnated Ag done at various temperatures of sonication. Product conversion analyzed using Gas Chromatography. The results showed that the impregnation of Ag on the activated zeolite catalyst increase the acidity from 0.0774 to 3.6253 mmol/g. The surface area slightly decreases from 1339 becomes 1279 m²/g. The process of converting glycerol to ethanol, the use of ultrasonic waves increases the effectiveness of glycerol conversion. The higher the sonication temperature, the greater ethanol produced. The highest ethanol product with a yield of 13.6% was obtained at a sonication temperature of 60 °C when the active Ag/zeolite catalyst was used. The use of Ag/zeolite catalyst makes glycerol conversion can be carried out at relatively low temperatures and ambient pressures so that it has more promising future prospects.

Keywords: Zeolite, Ag, catalysts, glycerol conversion, ultrasonic.

1. Introduction
The use of biodiesel as an alternative fuel has been a concern for the last decades. Biodiesel is produced through the transesterification reactions between plant oils or animal fats with alcohol. The production of biodiesel with a transesterification reaction produces 10% glycerol residue from the weight of the biodiesel produced [1]. Accordingly, for every 100 kg biodiesel production, 10 kg of glycerol residue is obtained. It is estimated that in 2020 biodiesel will contribute as much as 20% of fuel needs for transportation [2]. The increasing biodiesel production causes glycerol production to increase. This will cause a decrease in the price of glycerol in the market. Therefore, further research on the conversion of glycerol into other more useful products needs to be done.

Several studies on glycerol conversion have been carried out including using the pyrolysis and steam reforming methods. In addition, a glycerol conversion has also been carried out with the help of microwaves. Glycerol conversion by the pyrolysis method was carried out with variations in the nitrogen
gas flow rate at temperatures of 650-800 °C in an atmospheric pressure reactor [3]. The products produced are hydrogen, carbon dioxide, carbon monoxide, methane, and ethane. Another study converts glycerol through steam reforming at a temperature of 600-1000 K and a pressure of 1-5 atm [4]. The conversion of glycerol with the help of microwaves in batches system produced ethanol with the highest yield of 6.764% obtained when using the power of 600 watts for 15 minutes [5]. These studies were done at high temperatures, high pressure, or high electric power so that these processes are less safe and less economical. Therefore, it is necessary to find alternative methods of glycerol conversion that can be carried out at room temperature and pressure so that they are easy to apply and safe and have more promising future prospects.

Zeolite is a solid that has excellent properties to be used as a catalyst and as a carrier. Zeolite is used as catalysts because the pores are large, have a broad surface, and there is an active side. Zeolites are used as carriers because they are heat resistant and capable of forming bifunctional catalysts. The use of zeolite as a heterogeneous catalyst has the advantage of being able to easily separate catalysts and products [6]. The use of zeolite as a catalyst requires activation and modification of zeolites by impregnation or by distributing the group metal transition into the pores and surface of the zeolite. The metal catalyst which is applied to zeolite through impregnation makes zeolite a bifunctional catalyst, namely the function of the metal and its carrier as a catalyst [7]. The impregnated metal acts as a Lewis acid site while the zeolite is active as Bronsted acid [8]. Thus it is necessary to try the impregnation of transition group metals to increase the effectiveness of zeolites as catalysts.

Transition metals that can be used as catalysts and have the best catalytic properties including Pd and Pt [9]. However, the two metals have relatively expensive prices. So that in this study Ag metal is used. Metal Ag has a price that is relatively cheaper than Pd and Pt. Several studies have been conducted on the use of Ag as the catalyst's active core. Ag metal can be used as the active core of the catalyst impregnated into zeolite Y. The zeolite Y can be used as a catalyst in ammonia oxidation [10]. Zeolite with impregnation of Ag metal is also used as rapid degradation of methyl orange [11]. So that in this study Ag metal was impregnated into activated zeolite to increase the active site of the catalyst and increase the conversion of glycerol to ethanol.

An alternative technology is needed to degrade glycerol at room temperature and atmospheric pressure. It has been known that ultrasonic waves produce large energy because of cavitation effect. Cavitation produces high temperatures and pressures [12]. In these extreme conditions, radicals will arise which can initiate chemical reactions. Radicals are formed due to decomposition of monomers, solvent decomposition or breakdown of polymer chains [13]. Ultrasonic waves from sonochemical processes are expected to accelerate the reaction rate and increase conversion products [14]. The use of Ag/ZA catalyst with the help of ultrasonic waves is expected to be an alternative method for the process of converting glycerol to ethanol.

2. Methods

2.1 Materials

Synthesis of zeolite from local sand, HF pure analysis (pa) solution, HCl solution (pa), NH4Cl solid (pa), 100 ppm methylene blue solution (pa), concentrated ammonia (pa), demineralized water, AgNO3 (pa), glycerol (technical quality).

2.2 Zeolite activation

Before the activation process, zeolite was prepared by pounding it using mortar and pestle until zeolite become smaller then sifted using a 100-mesh sieve. Zeolite activation was carried out using Zeolite which will be used to degrade glycerol is zeolite NaY made from local sand as a source of silica. This type of zeolite is zeolite NaY. NaY zeolite before being used in the degradation process of glycerol is activated chemically and physically first. The chemical activation used HF (aq) 1 wt%, HCl (aq) 1 M at various soaking times of 6, 12, and 24 hours, and NH4Cl (aq) 1M. Physical activation was done by
means of zeolite calcined at 500 °C for 4 hours in the furnace. The activated zeolite catalyst was then characterized using XRF (MiniPal), the test of the surface area, and catalyst acidity.

2.3 Impregnation of ZA with Ag Metal from AgNO₃ (aq)
Activated zeolite was taken as 14.25 g and put in a 200 mL beaker, then added 100 mL of AgNO₃ (aq) 5 M. The solution was then stirred for 24 hours at 65 °C, washed using demineralized water until neutral and dried in the oven for 2 hours. The dried zeolite was calcined at 500 °C for 4 hours. This catalyst was called Ag/ZA was then characterized through the test of XRF, surface area, and catalyst acidity.

2.4 Catalyst Characterization
Catalyst characterization consisted of the determination of catalyst surface area with methylene blue adsorption method, acidity testing by ammonia mass adsorbed by the sample, the elemental composition of zeolite analysis using XRF.

2.5 Glycerol Conversion
Glycerol and demineralized water were put into a 250 mL enclosed Erlenmeyer with a volume ratio of 1/4. Then ZA catalyst was added as much as 3 wt% of the glycerol. The mixture was then vibrated with an ultrasonic bath (Branson 1510) at 30 °C with a sonication time of 2 hours with a frequency of 42 kHz. The mixture was cooled to room temperature. After reaching room temperature, the solution mixture was filtered. The filtrate was put into a vial bottle for further testing using GC (HP 5890). The same procedure was carried out at sonication temperature of 40, 50, and 60 °C for the conversion of glycerol using Ag/ZA catalyst.

3. Results and Discussion
3.1 Activation of Zeolite
Activation with HF (aq) 1 wt% is also called desilication, i.e., removal of silica free from zeolite pores. Soaking zeolite in HF solution was only carried out for 30 minutes. The reaction that occurred between free silica and HF solution is shown at reaction Equation 1.

$$\text{SiO}_2(s) + 4\text{HF(aq)} \rightarrow \text{SiF}_4(g) + 2\text{H}_2\text{O(l)}$$  \hspace{1cm} (1)

The activation of zeolite with HF solution dissolves silica and produces SiF₄ gas. Furthermore, the zeolite is activated with HCl 1 M. The process of activation of zeolite with HCl is also called dealumination which is the reduction of free Al₂O₃ from the zeolite framework to increase the Si/Al ratio. The higher the Si/Al ratio, the higher the thermal stability of zeolite. Activation is carried out with various of immersion time in HCl (aq) for 6, 12, and 24 hours. Activation with HCl causes loss of Al₂O₃ free oxide. Dealumination caused zeolite surface area increased due to reduced inorganic compounds that covered zeolite pores [15]. The reaction that occurred between Al₂O₃ and HCl is shown at reaction Equation 2.

$$\text{Al}_2\text{O}_3(s) + 6\text{HCl(aq)} \rightarrow 2\text{AlCl}_3(aq) + 3\text{H}_2\text{O(l)}$$  \hspace{1cm} (2)

HCl concentration affects the crystallinity of zeolite so that in this study HCl (aq) 1 M was used to avoid a decrease in zeolite crystallinity due to dealumination. Dealumination of zeolite as aluminosilicate with HCl was a substitution of aluminum with hydrogen which resulted in a change of the structure of Si-O-Al aluminosilicate to Si-OH silanol. In the activation process, H⁺ ions would break down the Al atomic bonds which were in the zeolite structure. This H⁺ ion will attack oxygen atoms bound to Si and Al. Based on the dissociation bond energy of the Al-O (116 kcal.mol⁻¹), which was much lower than the dissociation bond energy of Si-O (190 kcal.mol⁻¹), the Al-O bond was more fragile than Si-O. So that H⁺ ions would tend to cause the breakdown of Al-O bonds and silanol groups would be formed. Substitution of the structure of aluminosilicate into silanol caused a decrease in bond length.
which is from 1.69 to 1.61 Å [16]. The reaction of dealumination of zeolite by acid is shown at reaction Equation 3.

Zeolite which has been activated with 1\% HF and 1 M HCl was then activated with a solution of NH4Cl (aq) 1 M. Cation exchange occurs between impurity metal cations and NH4\(^+\). Weak bonds of metal cations cause these cations to be easily replaced with NH4\(^+\) through ion exchange [17]. The ion exchange mechanism that occurs in zeolites with NH4\(^+\) cations is shown at reaction Equation 4.

\[
\text{NH}_4^+ + \text{M}^+ \rightarrow \text{NH}_4^+ \cdot \text{M}^+ + \text{H}^+ (4)
\]

NH4\(^+\) ions from ammonium chloride solution replaced the position of impurity metal cations in zeolites. The ion exchange took place gradually and was proportional to the number of cations NH4\(^+\), so the higher the concentration of ammonium chloride solution, the more impurity metal cations that can be substituted by NH4\(^+\). After an exchange with ammonium ion, H-zeolite and NH3 gas will be formed. During heating, the ammonium cation bound to the zeolite decomposes into an NH3 gas as shown at reaction Equation 5.

\[
\text{NH}_4^+ \rightarrow \text{NH}_3 + \text{H}^+ (5)
\]

3.2 Impregnation of Active Zeolites with Metal Ag from Salt of AgNO3

Impregnation was carried out by immersing active zeolite in a metal salt solution. Ion exchange was almost always applied to include transition group metals such as Cu, Co, Pd, Pt, and so on [18]. Transition metals such as Ag are impregnated in their ion form, Ag\(^+\), through exchange with H\(^+\) on active zeolite so that Ag\(^+\) ions can be distributed evenly into zeolites.

Impregnation was done by soaking the active zeolite in AgNO3 solution which was accompanied by heating and stirring. This was done so that the Ag metal can be distributed evenly in order to increase catalyst activity. The mechanism of impregnation of active zeolite with Ag from AgNO3 salt is shown at reaction Equation 6.
3.3 Catalyst Characterization

To find out the types of elements contained in zeolites and their percentage compositions were analyzed by XRF. The results of the characterization of acid-activated zeolites using the XRF method are presented in Table 1.

| Elemental Composition | ZA | ZA-HCl 6 hours | ZA-HCl 12 hours | ZA-HCl 24 hours |
|-----------------------|----|----------------|----------------|----------------|
| Na                    | 3.90 | -              | -              | -              |
| Mg                    | 0.30 | 0.40           | 0.30           | 0.30           |
| Al                    | 21.12| 14.99          | 15.80          | 15.30          |
| Si                    | 53.10| 69.00          | 73.20          | 71.70          |
| P                     | 0.90 | 1.48           | -              | 2.30           |
| K                     | 2.22 | 1.76           | 1.72           | 1.79           |
| Ca                    | 3.90 | 2.73           | 2.62           | 2.31           |
| Ti                    | 2.20 | 1.80           | 1.70           | 1.60           |
| V                     | 0.30 | 0.28           | 0.26           | 0.30           |
| Cr                    | 1.10 | 0.63           | -              | 0.84           |
| Mn                    | 6.40 | 2.00           | 1.70           | -              |
| Zr                    | 4.10 | 3.20           | 2.10           | 1.86           |
| others                | 0.26 | 3.20           | 2.10           | 1.86           |

Based on the results of XRF, it can be seen that the activation can reduce the percentage of impurity metals such as Ca, Ti, V, Cr, and Mn. The calculation of the Si/Al ratio was obtained data as presented in Table 2.

| Type of Catalyst | Mass Ratio (Si/Al) |
|-----------------|--------------------|
| ZA              | 2.51               |
| ZA.HCl 6 jam    | 4.60               |
| ZA.HCl 12 jam   | 4.63               |
| ZA.HCl 24 jam   | 4.69               |
The data show that there is an increase in the Si/Al ratio after activation. The highest mass ratio of Si/Al is a catalyst with acid activated for 24 hours. The catalyst is then impregnated using Ag metal. After impregnation, an analysis using the XRF was conducted to determine the percentage of elemental composition. The results of the XRF analysis of zeolite impregnated Ag are presented in Table 3.

**Table 3. Elemental composition of Zeolite impregnated Ag**

| Elemental composition | (wt%) |
|-----------------------|-------|
| Na                    | -     |
| Al                    | 12.2  |
| Si                    | 57.2  |
| P                     | 0.52  |
| K                     | 1.2   |
| Ca                    | 3.76  |
| Ti                    | 1.8   |
| V                     | 0.33  |
| Mn                    | 1.01  |
| Mo                    | 1.5   |
| Ag                    | 18.3  |
| others                | 2.1   |

The activated zeolite impregnated Ag metal has a mass ratio of Si/Al 4.69. It can be seen that impregnation relatively does not change the Si/Al mass ratio of zeolite. The previous study revealed that the higher the Si/Al ratio, the more stable catalyst against high temperatures [19]. The catalyst of Ag/ZA has Si/Al mass ratio as high as ZA HCL 24 hours and it should have the highest thermal stability compared to other catalysts.

The acidity of the catalyst is analyzed from its ability to adsorb alkaline. The acidity that was analyzed was total acidity, namely the amount of Bronsted and Lewis acids. In general, to test the acidity of the catalyst using the base of ammonia or pyridine, but in this study base of ammonia was used. The calculation of the acidity of the catalyst is shown at equation (Equation 7).

Based on Figure 1, it can be seen that there is an increase in acidity of acid-activated zeolite. The ZA HCl 24-hour catalyst sample had the highest acidity among other samples. This is caused by the maximum removal of impurities in zeolites so that the surface area and active site of zeolite have increased. The greater the active site that is owned, the higher ammonia is adsorbed. While the acidity of the catalyst decreases after the impregnation of metal Ag. This is caused by the formation of aggregates which can cover the zeolite pores so that it closes the catalyst acid site. The closure of the catalyst acid site causes a decrease in the adsorption of the ammonia. The ZA HCl 24 hours catalyst is the highest catalyst with total acidity. The acidity of the catalyst in acid-activated zeolite reaches optimum at the time of immersion of zeolite in HCl for 24 hours with total acidity of 3.8036 mmol/g.

\[
\text{adsorbed ammonia (mmol)} = \frac{(b-a) \times 1000 \, \text{mg}}{M \, \text{NH}_3}
\]

\[
\text{total acidity (mmol/gram)} = \frac{\text{mmol absorbed ammonia}}{a}
\]

**Description:**

a = mass of sample before absorbing ammonia gas (gram)
b = sample mass after absorbing ammonia gas (gram)

Based on the acidity test of catalyst acidity, the obtained data are shown in Table 4 and the graph of the total acidity of several types of catalysts are shown in Figure 1.
Table 4. Data of catalyst acidity

| Catalyst          | a    | b    | (b-a) | Total acidity (mmol/g) |
|-------------------|------|------|-------|-----------------------|
| ZA                | 0.3033 g | 0.3037 g | 0.0004 g | 0.0774               |
| ZA.HCl 6 hours    | 0.3023 g | 0.3183 g | 0.0016 g | 3.1131               |
| ZA.HCl 12 hours   | 0.3064 g | 0.3253 g | 0.0189 g | 3.6282               |
| ZA-HCl 24 hours   | 0.3076 g | 0.3275 g | 0.0199 g | 3.8036               |
| Ag/ZA             | 0.3018 g | 0.3243 g | 0.0225 g | 3.6253               |

Figure 1. Total Acidity of Several Types of Catalysts

The surface area of the catalyst can be determined by its ability to adsorb methylene blue. This adsorption consider has a type of Langmuir isotherm where only a monolayer layer was formed at the time of maximum adsorption of adsorbate on the surface of the adsorbent [20]. In this study, measurements were taken at the maximum wavelength of methylene blue solution that is 660 nm. Data of catalyst surface area are presented in Table 5.

Based on the data obtained, it can be seen that the activation using a 1% HF solution, 1 M HCl, and NH₄Cl 1 M can increase the specific surface area of the catalyst. This was caused by the loss of impurities in zeolites. However, the impregnation of active zeolite with Ag metal causes the surface area to decrease. This is due to the closure of zeolite pores by the metal of Ag. Ag metal is not dispersed evenly on the pores and surface of zeolites so that the metal only accumulates on one of the active sides of the catalyst. This causes the specific surface area of the catalyst to decrease [21].

Table 5. Surface area of the catalyst

| Type of Catalyst | Surface area (m²/g) |
|------------------|---------------------|
| ZA               | 1251                |
| ZA-HCl 6 hours   | 1286                |
| ZA-HCl 12 hours  | 1307                |
| ZA-HCl 24 hours  | 1339                |
| Ag/ZA HCl 24 hours | 1279               |
3.4 Glycerol degradation

The glycerol degradation process was carried out using ultrasonic waves for 2 hours with various sonication temperatures of 30, 40, 50, and 60 °C. Degradation of glycerol used ZA and Ag/ZA catalysts. Glycerol degradation reactions occur through the mechanism of free radicals [22]. Radicals were formed directly or indirectly during isomerization of primary radicals. The mechanism of glycerol to ethanol degradation reaction is presented in Figure 2.

Glycerol degradation was caused by the breakdown of glycerol molecule bonds and solvent decomposition. Solvent decomposition would form radicals [23]. The catalyst functions to reduce the bonding force between or within the glycerol molecule so that it was easy to react with H⁺ [24]. The catalyst is separated from the degradation product by centrifuge and filtering is carried out. After being analyzed using GC, it was found that degradation products contained ethanol with different yield percentages. The results of the glycerol degradation are shown in Figure 3.

Based on Figure 3, the highest ethanol product is formed at 60 °C. This is because the higher the temperature of glycerol degradation, the faster the reaction takes place. The increase in temperature caused an increase in vapor pressure on the media. At high temperatures, cavitation was easier to form [25]. Cavitation created a very extreme environment that easily forms radicals that initiated chemical reactions.

The catalyst of Ag/ZA has better characteristics than ZA seen from the yield percentage of ethanol produced. The degradation process of glycerol using Ag/ZA catalysts resulted in ethanol with the highest yield of 13.56 vol% at 60 °C. This is caused by the ability of the transition metal to catalyze the reaction related to the presence of d-orbitals which is not occupied electron so that they can provide Lewis acid sites and capable of adsorbing the reactants on the surface of the catalyst [26]. The Ag metal acts as a catalyst because of the presence of 5s orbital with unpaired electrons and 5d orbitals that have not been filled with electrons. Impregnation can increase total acidity and increase catalyst activity; this is due to the presence of Lewis acid sites (electron pair acceptor species) on the catalyst [27]. The results of this study indicate that there is an increase in %yield obtained compared to previous studies when carried out with microwave assistance in batches with a power of 400-600 watts for 5 to 15 minutes where the highest yield ethanol produced of 6.8 v% obtained at 15 minutes with a power of 600 watts [5]. Another research also showed that the conversion of non-catalytic glycerol at varying temperatures of 200-400 °C obtained the highest ethanol yield of 2.74 v% [28]. Therefore, this study that uses a relatively low temperature and ambient pressure on the glycerol conversion process is safe and has more promising prospects.
Figure 3. Graph of effect of catalyst type and sonication temperature on yield of ethanol

4. Conclusion

The results showed that the impregnation of Ag on the zeolite catalyst increased the Si/Al ratio from 2.5 to 4.6; Acidity increased from 0.0774 to 3.6253 mmol/g, the surface area slightly decreased but did not change the structure of the zeolite. In the process of converting glycerol to ethanol, the use of ultrasonic waves increased the effectiveness of glycerol conversion. The higher the sonication temperature, the greater ethanol produced. The highest ethanol product with a yield of 13.6% was obtained at a sonication temperature of 60 °C when the active Ag/zeolite catalyst was used.

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