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Natural Zeolite as An Adsorbent for Pb (II) and Cu (II) Metal ions: Study of the Effect Acid Types on Zeolite Activation

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Abstract: *Rapid industrial development causes increasing industrial waste, such as Pb (II) and Cu (II) metal ions. Porous adsorbents such as natural zeolite can reduce the concentration of both metal ions in waste. This study aims to improve the ability of natural zeolites to adsorb Pb (II) and Cu (II) metal ions through acid activation. Natural zeolite with homogeneous size is activated using HCl and* HNO_3 *acids. Natural zeolite and acid-activated zeolite are characterized by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray diffractometer (XRD). Furthermore, activated zeolite reduces Pb (II) and Cu (II) metal ions. The results of the study showed that zeolite that had been activated by HCl and* HNO_3 *acids did not experience structural damage because the diffractogram pattern did not change, namely at* $2\theta = 22.34°$; $25.72°$; $26.39°$; *and* $27.77°$. *The FTIR spectra support the data with the emergence of Si-O-Si groups at wavenumbers 1000 - 1200* cm^{-1}. *Meanwhile, the adsorption capacity of Pb(II) metal ions and Cu(II) metal ions by natural zeolite is smaller than acid-activated zeolite.*

Keywords—natural zeolite; acid type; Pb (II); Cu (II); activation

1. INTRODUCTION

Rapid industrial development is one of the causes of the increasing release of heavy metals into the environment. The primary sources of heavy metals are wastewater from modern industrial sources including chemical industries such as mining (Li et al., 2024), metallurgy (Nocoń & Korus, 2023), sulfonation process waste (Tran et al., 2023), waste incineration and gasification(Wu et al., 2014), Disposal of Metal-finishing wastewater (Ingle & Patel, 2025), surface coating, battery manufacturing, leather tanning, fossil fuels, paper and different plastic production (İnce & Kaplan İnce, 2017). Heavy metal ions are released into the environment, causing serious soil and water pollution. Mercury (Hg), lead (Pb), copper (Cu), iron (Fe), and chromium (Cr) are common metals that tend to accumulate in organisms, causing various diseases and disorders (Mudasir et al., 2016), (Sriatun et al., 2008), (Qomaruzzaman et al., 2020).

Lead (Pb) has the highest affinity for sulfur and attacks its bonds in enzymes. As a heavy metal, Pb is classified as a hazardous pollutant. Pb metal is widely used in industry and piping. Leaded gasoline is a major source in the atmosphere and on the earth's surface. Most of the Pb on earth enters the natural water system and accumulates, which can eventually enter the bodies of animals and humans. If absorbed into the human body, lead (Pb) can cause decreased intelligence in children, stunted growth, and even paralysis. Other symptoms of Pb metal poisoning are nausea, anemia, and abdominal pain (Widayatno et al., 2017).

In addition to Pb metal, Cu metal is one of the heavy metal ions that often pollute the aquatic environment. Cu metal is widely used in the textile industry for the dyeing and printing. High water use in wet processes can produce significant amounts of liquid waste if the process efficiency is not optimal. The concentration limit of Cu that affects water ranges from 1 - 5 mg / L. Based on the Decree of the Minister of Health No. 907 / MENKES / SK / VII / 2002, safe concentrations for humans should not be more than one ppm if it exceeds the safe limit it is very toxic to the nervous system and affects kidney function (Siti, 2018). Handling heavy metals can be done with various techniques, such as conventional methods, usually involving coagulation, precipitation, ion exchange, electrochemical methods, membrane processes, extraction, adsorption, and others. The adsorption method is very suitable for wastewater treatment because of its simplicity and cost-effectiveness. Some adsorbents widely used for heavy metal adsorption are activated carbon, clay minerals, biomaterials, and zeolites (Zendelska et al., 2015).

Currently, natural zeolites are widely used to purify wastewater. Zeolites are effective adsorbents for a range of pure metals, including copper. The advantages of zeolites are high selectivity, cation exchange, relatively low cost, and accessibility. Investigation of heavy metal adsorption by natural sorbents involves the adsorption capacity, isotherms, and adsorption equations describing the statics process, studying the kinetic order, dynamics of adsorption in the sorbent layer and sorbent recycling (Sabadash et al., 2017).

Zeolites are crystalline aluminosilicates with uniform molecular-sized pores. Zeolites exhibit unique physical and chemical properties such as hydrophilicity, hydrophobicity, solid acidity, and molecular sieving effects. Due to these properties, zeolites have a wide range of applications in industrial processes. For example, zeolites can be used in separation and catalysis due to their unique pore structure, adsorption properties, and high thermal, mechanical, and chemical stability. Each advantage stems directly from its crystal structure (Jamal et al., 2025).

The direct utilization of natural zeolite can be done but is not yet optimal because natural zeolite contains many impurities, such as quartz or other minerals and other cations that are less beneficial, so specific treatments are needed first to remove or separate them from impurities (Sanzana et al., 2024). Efforts made to increase the efficiency and effectiveness of natural zeolite include activation. This activation process is needed to improve the special properties of zeolite and remove impurities. The activation process can also change the type of cation and zeolite characteristics to suit the adsorbed material (Renni et al., 2018). Activation of zeolite with acid causes the dealumination of Al from the framework structure and decation. This process increases the surface area of zeolite due to the reduction of impurities that cover the pores of the zeolite so that the absorption that occurs is greater. Renni et al. reported that zeolite activated with HNO_3 acid has a larger surface area, and the Si/Al ratio also increases, so it can better adsorb metals.

This article reports the activation of natural zeolite using HCl and HNO_3 to determine the difference in its adsorption capacity on Pb and Cu metal ions. From the absorbance data obtained with AAS, the adsorption capacity of natural zeolite and acid-activated zeolite on Pb and Cu metal ions will be known.

2. METHODOLOGY

2.1 Materials and equipments

The materials used are natural zeolite from Klaten, Central Java, HF 40% (Merck), HCl 37% (p.a, Merck),), $AgNO_3$ (Merck), $Pb(NO_3)_2$, $Cu(NO_3)_2 3H_2O$. Aquades.

Meanwhile, the equipment and instruments used were glassware, sieves, magnetic stirrers, Merck pH indicator, analytical balances (Ohaus), Oven (BINDER), porcelain crucible, desiccator, Vulcan 3-130 Furnace, atomic absorption spectrophotometer (Perkin Elmer 3110), Fourier Transform Infrared Spectroscopy (FTIR 8201PC Shimadzu), shaker (MAXQ 2000) and X-ray diffractometer (Shimadzu XRD 6000). UV-vis spectrophotometer Shimadzu.

2.2 Preparation and Activation of Natural Zeolite

Natural zeolite is ground until smooth using a porcelain crucible. The zeolite is sieved 150 mesh to get a homogeneous size. Furthermore, it is soaked in distilled water, stirred, and left for 24 hours. The soaked zeolite is filtered, and the residue is removed. Then, it is dried using an oven at a temperature of 120°C for 24 hours. The dried zeolite is finely ground and sieved 150 mesh.

Zeolite powder of as much as 100 grams was put into two 1000 ml beakers, each adding 500 mL of hydrochloric acid (HCl) with a concentration of 3 M and 500 mL of nitric acid (HNO_3) with a concentration of 3 M and then, stirred using a magnetic stirrer for 120 minutes. Then left for one day. After that, filter and wash with distilled water until neutral. Then, it is filtered and dried using an oven at a temperature of 110°C for 24 hours. The dried zeolite was ground and sieved 150 mesh. The activated zeolite was characterized using XRD and FTIR.

2.3 Adsorption of Pb (II) and Cu (II) metal ions by acid-activated zeolite

The $Pb(NO_3)_2$ solution 10 ppm from 3.99 mg $Pb(NO_3)_2$ was dissolved in 250 ml of distilled water, while $Cu(NO_3)_2$ solution came from 9.35 mg dissolved in 250 ml of distilled water. Each solution was taken 20 ml and put into 6 Erlenmeyer flasks. Furthermore, it was adsorbed with 0.2 grams of zeolite. Adsorption was carried out at various times, namely 5,10, 15, 30, 60, and 120 minutes. To determine the amount absorbed, the remaining filtrate was analyzed by Atomic Adsorption Spectroscopy (AAS).

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3. RESULTS AND DISCUSSIONS

The activation of natural zeolite aims to clean pores and remove impurities by modifying its properties, such as increasing surface area and acidity. Activation can be done physically and chemically. Physical activation takes the form of heating at high temperatures and reducing the grain size. Physical activation aims to expand the surface and enlarge the pores. Meanwhile, chemical activation involves adding chemicals that aim to remove inorganic impurities.

Based on previous research, natural zeolite from Klaten, Central Java, consists of mordenite, quartz, and clinoptilolite (Sriatun et al., 2024). Usually, this type of mineral has a medium Si/Al ratio, so it is suitable for cation exchange between the cations in the zeolite and the proton. Chemical activation with acid (HCl or HNO_3) aims to dissolve impurity oxides. The active zeolite undergoes an ion exchange process with proton (H^+) using HCl 3 M or HNO_3 3 M. Hydrochloric acid and nitric acid are potent acids that dissolve Al_2O_3 and other oxides. The Cl^- anions can easily interact with metal oxides. The Cl^- anions are hard base groups that easily bond with hard acid groups of metal oxides, namely: Al^{3+} from Al_2O_3, Mg^{2+} from MgO, K^+ from K_2O, Ca^{2+} from CaO, Ti^{4+} from TiO_2, and Fe^{3+} from Fe_2O_3. Acid washing also functions as a cation exchanger of zeolites so that the cation content becomes uniform. Proton H+ plays a role in the exchange of cations, such as Na^+, Ca^{2+}, and others, as a charge balancer in the tetrahedral framework structure of aluminosilicate.

During chemical activation, cations in zeolite non-framework such as Na^+, K^+, Ca^{2+} and Mg^{2+} are exchanged with H^+, resulting in zeolite-H. The cation exchange process can be accelerated by stirring. This stirring will cause the cation exchange active groups on the surface of the zeolite to move more actively, increasing the intensity of collisions between cations from the zeolite and H^+. This is because, with stirring, the irregularities in the solution increase and cause the kinetic energy in the solution to become greater, thereby accelerating the achievement of activation energy.

3.1 FUNGSIONAL GROUPS OF NATURAL ZEOLITES AND ACID-ACTIVATED ZEOLITES

One characterization to identify the compound content and functional group analysis was carried out using an FT-IR spectrophotometer. Qualitative analysis is obtained by looking at the spectral character where typical peaks will be seen, which can be read as groups contained in the compound being analyzed, and quantitative analysis is by reading the value of the wavenumber peaks—identification of functional groups in natural and acid-activated zeolite using Fourier Transform Infrared (FTIR) analysis. The analysis was carried out at a wavelength of 600 cm^{-1} - 1300 cm^{-1}. The infrared spectra between zeolite particles are shown in Figure 1. The absorption results show that the Si-O group is at a wave number of 900 cm^{-1} - 1300 cm^{-1}.

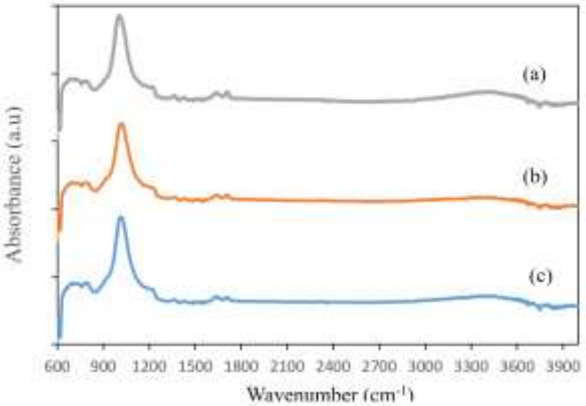

Fig. 1 *FTIR spectra of (a) Natural Zeolite (b) Activated Zeolite by HNO_3 (c) Activated Zeolite by HCl*

Qualitatively, the FTIR spectra can detect the absorption of –OH groups, Si-OH, Si-O-Al and Si-O-Si groups from natural zeolites and zeolites activated by HCl and HNO_3 acids.

Table 1: Types of vibration of natural and acid-activated zeolite

Types of vibration	Wavenumber (cm^{-1})		
	Z/HCl	Z/HNO_3	Natural Zeolite
Si-O-Si Asymmetric stretching vibration	1014	1211	1007
Bending vibration –OH of Si-OH	1639	1678	1639
Si-O-Si symmetrical bend	690	707	690
Symmetric stretching vibration of Si-O-Si	795	822	795

Based on Figure 1 and Table 1, it is obtained that the wave number of the asymmetric stretching vibration of Si-O-Si against acid-activated zeolite is greater than the wave number of natural zeolite, so it can be seen that zeolite activation successfully removes impurities in the pores of the zeolite. The shift in the wave number indicates a decrease in Al in the zeolite framework. Dealumination causes Al atoms to be released from the zeolite framework, causing the required binding energy to increase. The greater the binding energy, the greater the wave number. This dealumination is also reinforced by a significant reduction in absorption intensity in the asymmetric stretching vibration range in the 950 - 1250 cm^{-1} regions.

However, quantitative analysis is needed to determine the magnitude of the intensity of the absorbance changes that occur. Quantitative analysis can be done by analyzing the FTIR spectra using Fityk software to obtain information about changes in composition by measuring the absorbance intensity related to the type of chemical group being measured, such as Si-OH and Si-O-Si in the silica structure. Analysis using Fityk is done by equating the HWHM (Half-Width at Half Maximum) at the peak of the existing FTIR spectrum, then calculating the ratio of the area of the Si-OH group to Si-O-Si so that the Si-OH/Si-O-Si ratio is obtained. Deconvolution of FTIR data with the application of Fityk obtained changes in the ratio of Si-OH to Si-O-Si in natural zeolite and acid-activated zeolite, which are shown in Table 2.

Table 2: Deconvulation of Si-OH/Si-O-Si peaks and Si–OH/Si-O-Si ratios in zeolites

Adsorbent type	Area		Si-OH/Si-O-Si Ratio (%)
	Si-OH	Si-O-Si	
Natural Zeolite	1212.33	2913.72	41.61
Z/HNO_3	1201.31	2572.25	46.70
Z/HCl	1046.45	2389.3	43.80

Based on Table 2, it can be seen that the one with the highest Si-OH to Si-O-Si ratio is HNO3 acid-activated zeolite, which is 46.70% compared to HCl acid-activated zeolite and natural zeolite. From the results above, it can also be seen that the Si-OH/Si-O-Si ratio in acid-activated zeolite is greater than that of non-activated natural zeolite. The large ratio's effect is that the Si-OH group's area in acid-activated zeolite has a smaller area than that of natural zeolite. It indicates that the Si/Al bond is broken during activation and replaced by the OH bond. It indicates that this acidification causes the exchange of cations with H^+.

3.2 CRYSTAL STRUCTURE OF NATURAL ZEOLITES AND ACID-ACTIVATED

The structure and crystal size of natural zeolite and acid-activated zeolite were characterized using XRD. Figure 2 shows the XRD diffractogram pattern of natural zeolite and acid-activated zeolite HCl and HNO_3.

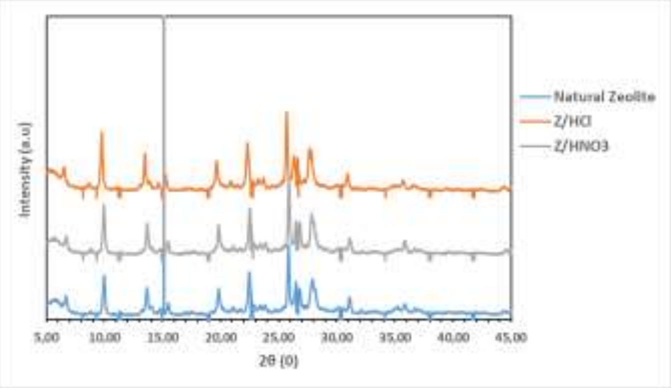

Fig. 2 XRD diffractograms of natural zeolite, Z/HCl, and Z/HNO_3

The X-ray diffraction pattern shown provides information that there are peaks at 2θ = 22.34°; 25.72°; 26.39°; 27.77°, which indicate the presence of mordenite minerals according to rruff.info id R070524. Rruff.info.id R070524 shows peaks at 2θ = 22.34°; 25.72°; 26.39°; 27.77°. In Figure 2 it can be seen that no new peaks appear and the position of 2θ after activation does not experience significant changes. It indicates that there is no change in the structure of the activated zeolite.

The X-ray diffraction pattern also provides information that the intensity of acid-activated zeolite increases from zeolite without activation. This is because the crystallinity of natural zeolite after activation is higher than that of natural zeolite before activation. The activation process has removed impurities in the zeolite framework and made it more regular so that its crystallinity increases. The increase in crystallinity makes activated zeolite more optimal in adsorbing metal ions. In contrast, natural zeolite usually gives less than optimal results in the adsorption process because it still contains many impurities in its constituent framework. The intensity of hydrochloric acid (HCl) activated zeolite is higher than nitric acid (HNO_3) activated zeolite. Hydrochloric acid (HCl) has more acidity than nitric acid (HNO_3).

3.3 ADSORPTION ABILITY OF PB (II) METAL IONS ZEOLITE

Adsorption of ion Pb (II) was carried out at a fixed concentration of 3M in various acids to determine the adsorption capacity of each adsorbent.

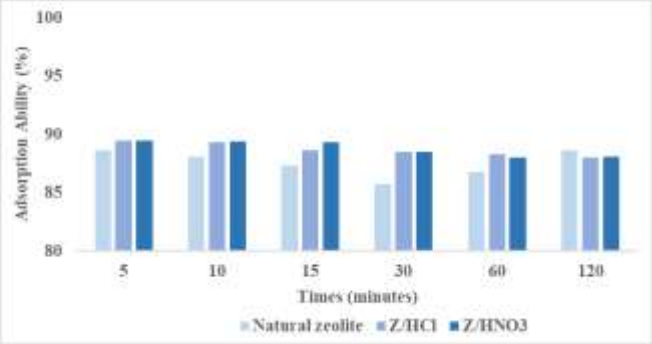

Fig. 3 Adsorption Ability of Pb (II) Metal Ions on Natural Zeolites and Acid-Activated Zeolites.

Figure 3 shows that the highest adsorbed concentration in ion Pb (II) occurs in hydrochloric acid (HCl) activated zeolite. Moreover, the optimum time required to adsorb Pb (II) metal on natural zeolite is longer, which is 120 minutes. In comparison, it takes five minutes for acid-activated zeolite to reach the optimum time. Moreover, adsorption using acid-activated zeolite is greater than zeolite without activation. This shows that adsorbing Pb (II) metal ions requires a slightly complicated method, where natural zeolite must be activated first to adsorb it so that impurities no longer cover the pores in natural zeolite and make the adsorption process easier and faster. In addition, Pb (II) metal ions have small selectivity properties compared to Cu (II) metal ions (Zendelska et al., 2015). In acid-activated zeolite, it does not show many different adsorption results. Because HCl and HNO_3 are included in the strong acid group, during the activation process, they use the same concentration, which is 3M.

3.4 ADSORPTION ABILITY OF CU (II) METAL IONS ZEOLITE

Adsorption of Cu (II) metal ions was carried out at a fixed concentration of 3M in various acids to determine the adsorption capacity of each adsorbent.

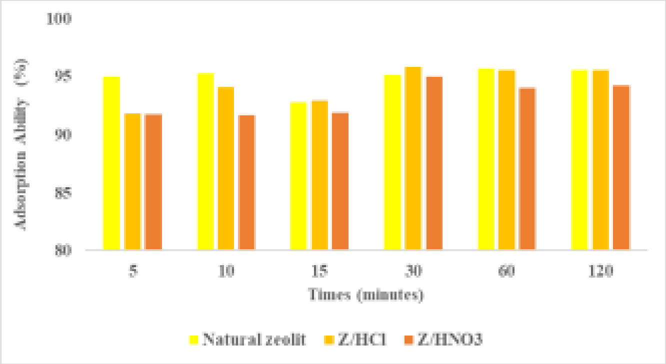

Fig. 4 Adsorption Ability of Cu (II) Metal Ions on Natural Zeolites and Acid-Activated Zeolites.

Based on Figure 4, adsorption on Cu (II) metal ions, the highest adsorbed concentration occurs in nitric acid

(HNO_3) activated zeolite. The optimum time required to adsorb Cu (II) metal ions on nitric acid (HNO_3) and hydrochloric acid (HCl) activated zeolite is 30 minutes, while natural zeolite requires 60 minutes. However, the adsorption concentration on acid-activated zeolite does not differ significantly. This is because nitric acid (HNO_3) and hydrochloric acid (HCl) are classified as strong acids. In addition, the acid concentration is the same, namely 3M, so the results are not much different. Another factor that can be considered is that the radius of the Cu (II) metal ion is smaller than Pb (II), so natural zeolite also easily absorbs Cu (II) metal. The selectivity properties of Cu (II) metal ions are greater than those of Pb (II) metal ions. Therefore, the adsorption of Cu (II) metal ions is easier than that of Pb (II) metal ions.

The adsorption of Pb (II) metal ions is smaller than Cu (II) metal ions, apart from the selectivity properties of both; this is also due to the nature of the atom itself, namely its radius, where the radius of the Cu (II) metal ion is smaller than the Pb (II) metal ion, which causes Cu (II) metal to have greater acidity than Pb (II). Thus, its polarizability is smaller than Pb (II) ions. Another factor that influences this is the presence of -OH groups in zeolites, which are hard bases. Based on the concept of soft, hard acid bases (HSAB), Cu (II) and Pb (II) ions are classified as medium acids. In contrast, the adsorbent is classified as a hard base, meaning that Cu (II) and Pb (II) ions will interact well with the adsorbent.

4. CONCLUSION

The adsorption capacity of natural zeolite is smaller than acid-activated zeolite for Pb (II) and Cu (II) metal ions. The adsorption ability of HCl-activated zeolite is similar to that of HNO_3-activated zeolite. Natural zeolite requires an optimum time of 10 minutes to adsorb Pb (II) ions of around 89%, while acid-activated zeolite only takes 5 minutes. To adsorb Cu (II) ions of around 95%, natural zeolite requires an optimum time of 60 minutes, while acid-activated zeolite only takes 30 minutes. The adsorption capacity for Cu (II) ions is greater than Pb (II) ions for all adsorbents.

5. ACKNOWLEDGEMENT

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