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Adsorption of Copper Ion from Acidic Wastewater by Local Natural Zeolite

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Abstract

Natural zeolites are available in Indonesia in very large quantities and are available in the market at very affordable prices. The use of zeolite has developed in various fields of industry, water treatment and wastewater treatment. The study on the ability of local natural zeolite to adsorb copper from acidic wastewater from a laboratory in Tangerang has been conducted. XRD data show that the local natural zeolite consists of mordenite as main mineral and quartz as an accessory mineral. Acid activation of zeolite with HCl 3 M could remove quartz impurities from natural zeolite based on XRD data. Mordenite is one of the most abundant zeolite minerals and commercially used for many purposes including adsorbents, catalyst, fertilizer, and gas separation. The crystallinity of mordenite decreases throughout the process of acid activation and calcination. The calcined zeolite has better copper adsorption ability compared to local natural zeolite without any treatment. The optimum copper adsorption percentage is 14.554% at solution pH of 0.3 by using 10 grams of calcined zeolite in 200 mL of wastewater solution.

Keywords: *acidic wastewater; copper; zeolite.*

Introduction

Heavy metals can be bioaccumulated in living organisms and human tissues through various processes that cause toxic effects. In the human body, these heavy metals are absorbed into the cells and binding to proteins and nucleic acids, then destruct these macromolecules and damage many cellular functions, and cause cells death. Thus, heavy metals have several consequences in the human body such as affecting central nervous function leading to mental disorders, damaging blood constituents and can damage lungs, liver, kidneys, and other vital organs promoting several disease conditions (Jaishankar et al., 2014). Accumulation of heavy metals in the body can result in the development of physical, muscular, and neurological degenerative processes such as Parkinson's disease and Alzheimer's disease (Bhat et al., 2019). Along with that, repeated long-term contact with some heavy metals or their compounds can even damage nucleic acids, cause mutations, disrupt the endocrine and reproductive systems and cause cancer (Jaishankar et al., 2014).

Wastewater is commonly known as the source of heavy metal contamination. The spill or improper treatment of wastewater is the main problem of heavy metal contamination in land and aquatic environments. Wastewater containing heavy metals must be ensured that the concentration of heavy metals is below the threshold value set by the Ministry of Health of the Republic of Indonesia before it can be discharged into the environment (Simbolon et al., 2014).

Based on its toxicology, heavy metals can be divided into essential heavy metals and non-essential heavy metals. Essential heavy metals in certain quantities are needed by living organisms, but in excessive amounts, they can cause toxic effects, for instance Zn, Cu, Fe, Co, Mn, and Se. Non-essential heavy metals are toxic metals whose benefits in the body are still unknown, like Hg, Cd, Pb, Sn, Cr, and As (Samoila et al., 2019). These heavy metals can cause adverse effects on human health, so they are often referred to as toxic metals. These compounds are non-degradable in nature and do not change into other forms (Tahya et al., 2019).

Copper (Cu) is essential heavy metal but excess of copper in human body is life-threatening (Irawati et al., 2019; Irawati & Tahya, 2020, 2021; Nurlaila et al., 2021). Several studies have indicated that an imbalance in copper levels can contribute to Alzheimer's disease and cardiovascular disease (Sacco et al., 2022). Copper is also toxic to amphibians, fish and invertebrates. Amphibians are relatively sensitive to copper chronic exposure (Azizishirazi et al., 2021).

Zeolite is a hydrated alumino-silicate chemical compound with sodium, potassium and calcium cations. Zeolite is also often referred to as a 'molecular sieve' or 'molecular mesh' because zeolites have molecular-sized pores so that they can separate/filter metal molecules or ions of a certain size (Atikah, 2017). The use of zeolite has developed in various fields of industry, water treatment and wastewater treatment. Natural zeolite has long been known as an effective adsorbent for a variety of designations including for sewage treatment. Natural zeolite can be used as heavy metal adsorbent, but in this research, we observe how local natural zeolite are able to adsorb copper ion from strong acidic wastewater. This study aims to observe the ability of local natural zeolite in the removal of copper from acidic wastewater.

Methodology

Tools and Material

Analytical balance (Ohaus), furnace (Thermolyne), pH meter (Ohaus) to determine pH, ICP-OES instrument for determination of copper concentration in solution, XRD and FTIR for mineral and functional groups analysis, and standard glass tools. Chemicals used are hydrochloric acid p.a (Merck), silver nitrate (Merck), natural zeolite from local market, and the wastewater was collected from one of laboratory in Tangerang.

Procedure

Preparation of Zeolite

Natural zeolite (NZ) was purchased from one of material store in Tangerang city, Indonesia. The NZ was washed with distilled water twice, rinsed and let dry in oven at 95° C for 48 hours. The dried zeolite was ground to powder and sieved. The sample was taken for further characterization with XRD and FTIR.

Acid Activation and Calcination of Zeolite

A total of 200 grams of clean zeolite powder were mixed with 3 M HCl, stirred for 5 x 60 minutes at room temperature, then filtered through a Buchner filter, then washed with distilled water until free of Cl^- according to the test with 0.1 M $AgNO_3$ solution. Acid-activated zeolite was dried at 81°C for 2 days, then a sample was taken for characterization with XRD. Around 150 g of sample were ground and calcined in a furnace at temperature of 400°C for 5 hours. Then,

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sample was taken for characterization with XRD.

Adsorption Process

Wastewater obtained from the laboratory was 1 L then diluted by adding 4 L of distilled water so that the total volume of the solution is 5 L. The pH of this solution was measured by a pH meter. About 100 mL of this solution was taken for ICP-OES analysis to observe the heavy metals content of Cd, Cr, Cu, Pb, and Mn. Weight the zeolite, about 1 g, 5 g, 10 g, 20 g and 50 g, and put it into Erlenmeyer flask 500 mL. Into every flask added 200 mL of wastewater. Then the stirring process was carried out for 5 hours at room temperature, around 27°C. Filter the mixtures, and took 100 mL of each filtrate for the ICP-OES analysis of Copper.

Result and Discussion

Characterization of Natural Zeolite

Natural zeolite was characterized by XRD dan FTIR analysis. The XRD diffractogram of natural zeolite is shown in Figure 1. The phase identification from XRD shows the natural zeolite consists of Mordenite and Quartz. Mordenite has chemical formula of (Ca, Na_2, K_2)$Al_2Si_{10}O_{24}$. $7H_2O$. Mordenite is one of the most abundant zeolite minerals and commercially used for many purposes including adsorbents, catalyst, fertilizer, and gas separation (Klunk et al., 2020; Trisunaryanti et al., 2018; Wahono et al., 2019). Mordenite has shown better chemical resistance, a larger quantity of pore volume, good thermal stability, and high surface area (Narayanan et al., 2020). Mordenite shows potential ability in adsorption of heavy metals, such as Hg (Murthy et al., 2013), Pb (Turkyilmaz et al., 2014), Tl, As and Co (Pourahmad et al., 2010).

Based on the XRD of local natural zeolite, we can observe the present of one accessory mineral which is quartz. Natural zeolite usually consist of many accessory minerals such as quartz, kaolinite, illite, feldspar, smectite and mica (Alshameri et al., 2019). The XRD data also give us information about crystallinity of mordenite is 31.0% and the crystallite size of mordenite phase is 143 Å. While crystallinity of quartz is 31.0% and the crystallite size of quartz phase is 261 Å.

The FTIR analysis of local natural zeolite is shown in Figure 2 and the functional groups' determination was shown in Table 1. Strong and broad vibrational stretch is found at 1042 cm^{-1} related to the Si–O group in mordenite and quartz which was similarly observed by Klunk et al., (2020). The H-OH vibration was observed in 1644 cm^{-1}.

Table 1. Peak assignment of functional group in local natural zeolite.

Wavenumber (cm^{-1})	Peak Assignment
529	T-O bending (T= Al or Si)
794	T-O-T symmetric stretching (T= Al or Si)
1042	T-O-T assymmetric stretching (T= Al or Si)
1644	H-O-H bending
3432	O-H stretching

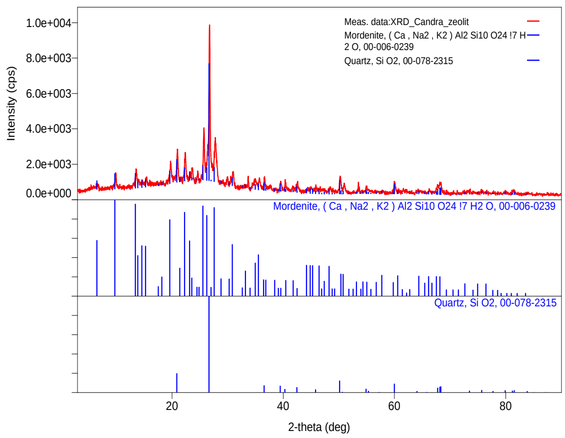

Figure 1. XRD data of local natural zeolite and phase identification.

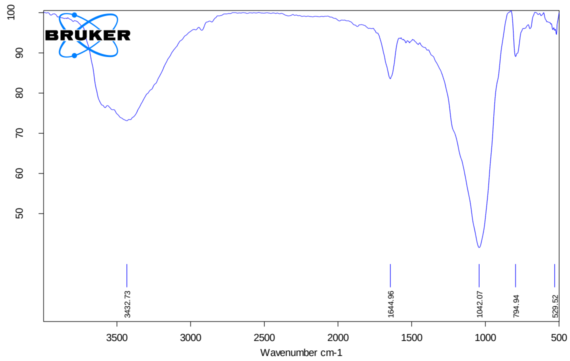

Figure 2. FTIR data local natural zeolite.

Characterization of Acid-activated and Calcined Zeolite

Natural zeolite was activated by 3 M HCl to remove any organic and inorganic impurities from natural zeolite such as iron, sulfur or phosphorous, cleanse the pores, and leading to decationization-dealumination process of zeolite framework without destroying it (Al Muttaqii et al., 2019; Kadirbekov et al., 2017). The purpose of dealumination is to optimize selectivity, acidity control, and stability at high temperature of natural zeolite (Al Muttaqii et al., 2019). The acid-activated zeolite was characterized by XRD shown in Figure 3.

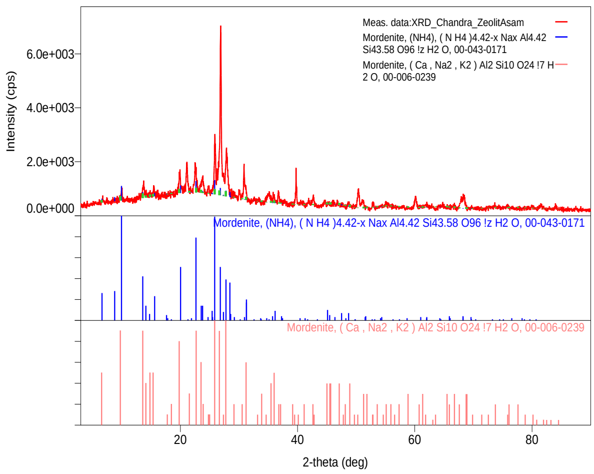

Figure 3. XRD data of acid-activated zeolite and phase identification.

The XRD phase identification shows that it is only single phase identified, which is mordenite. Quartz phase has been removed by the acid activation process. High concentration of HCl (3 M) and duration of stirring (5 h) could be determining factor for the removal of quartz, because Muttaqii et al., (2019) used HCl concentration less than 3 M and only 2 hours stiring duration, and still observed the present of quartz in acid-activated zeolite. The using of HCl 3 M was also good in decationization of zeolite as what was reported by Tišler et al., (2019) that they observed the increasing in the silicate modulus parameter which indicated that Na and K were rapidly leached from the zeolite so the acid activation process promotes the removal of most of sodium and potassium (Tišler et al., 2019). The XRD data of activated zeolite also give us information about crystallinity of mordenite is 23.8% and the crystallite size of mordenite phase is 112 Å. Here we observe the decreasing of crystallinity and crystallite size of mordenite acid-activated compared to natural zeolite, however the basic zeolite structure remains intact. The smaller the crystallite size, the more efficient the sintering or calcination (A. Ruys, 2019). The crystallinity decreased with increasing concentration of acid, leaching time, temperature, during the activation of zeolite (Tišler et al., 2019).

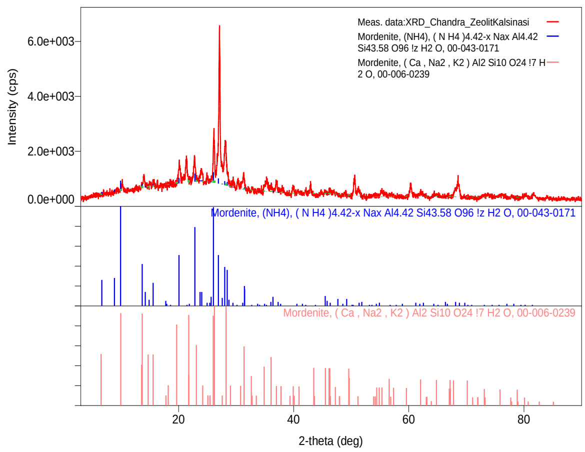

Figure 4. XRD data of calcined zeolite and phase identification.

The acid-activated zeolite was then calcined at 400°C for 5 hours. This temperature was referred to the previous studies of calcination treatment of natural zeolite for organic compound decomposition and purification of the pores (Wahono et al., 2019; Wahono & Rizal, 2014). The XRD data of calcined zeolite is shown in Figure 4.

Mordenite is the only phase that identified in calcined zeolite XRD data, so there is no change in mineral phase of zeolite, the structure remains intact after calcination. This data support the reports which state that calcination treatment above 500 °C could change zeolite into siliceous phase (quartz or cristobalite) or a compact aluminosilicate (labradorite) or amorphous solid, which means the lost porosity (Tišler et al., 2019; Trisunaryanti et al., 2018; Wahono et al., 2019).

The Crystallite size commonly corresponds to the size of the grains of a powder sample, or thickness of polycrystalline thin film or bulk material (Bishnoi et al., 2017). The crystallite size of mordenite was increased to 202 Å in calcined zeolite compared to 112 Å of acid-activated zeolite. However, the crystallinity of mordenite was decreased to 16.6 % in calcined zeolite compared to 23.8% in acid-activated zeolite. Generally, calcination resulted in reduced zeolite crystallinity. This is indicated by a reduction in the heights of XRD peaks associated to characteristic zeolite reflections (Burris & Juenger, 2020).

ICP-OES Analysis of The Wastewater

About 100 mL of diluted wastewater was taken for Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES) analysis to determine the heavy metals content of Cd, Cr, Cu, Pb, and Mn. All these metals are dangerous to human life and environment so must be treated properly (Bhat et al., 2019). Table 2 shows the ICP-OES result. The pH of this solution was 0.3 (acidic solution) and was calculated by using pH meter with tool's standard operation was followed.

Table 2. ICP-OES result of acidic wastewater.

No	Metal or Major Cations	Concentration (mg/L)
1	Cadmium	<0.02
2	Chromium	5.60
3	Copper	182
4	Lead	0.44
5	Manganese	0.17

The highest heavy metal content in the wastewater is copper, 182 mg/L. This concentration is quite high far from allowed regulation. The copper metal threshold in wastewater must not exceed 0.5 mg/L based on the Regulation of Indonesian Minister of the Environment. In human body, copper in high doses can cause vomiting, anemia, dizziness, weakness, kidney and liver failure, and dead (Indah et al., 2021).

Adsorption of Copper from acidic Wastewater by Natural and Calcined Zeolite

Adsorption process was carried out with every 200 mL of acidic wastewater and various mass of zeolite added into each flask. The average pH of solution before and after adsorption is 0.3. The pH before and after adsorption was determined and was shown as the same value. The adsorption proses does not change the pH. The percent of adsorption was calculated with equation 1. Table 3 and Figure 5 show the adsorption results.

$$\text{Adsorption (\%)} = (C_o - C_a)/C_o \times 100\% \qquad (1)$$

Adsorption capacity

$$AC\ (ppm/g) = (C_o - C_a)/m \qquad (2)$$

Where: C_a is the concentration of copper in solution after adsorption (mg/L), C_o is the concentration of copper in solution before adsorption (mg/L). The m is mass of zeolite in gram.

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Table 3. The adsorption of copper by natural zeolite.

Mass of zeolite (g) per 200 ml of solution	Conc. of Cu in ww before treatment (mg/L)	Conc. of Cu in ww after adsorption (mg/L)	Percent of adsorption of natural zeolite (%)
1	182	176	3.297
5	182	168	7.692
10	182	168	7.692
20	182	176	3.297
50	182	177	2.747

Note: ww = wastewater

Table 4. The adsorption of copper by calcined zeolite.

Mass of zeolite (g) per 200 ml of solution	Conc. of Cu in ww before treatment (mg/L)	Conc. of Cu in ww after adsorption (mg/L)	Percent of adsorption of calcined zeolite (%)
1	213	199	6.573
5	213	196	7.981
10	213	182	14.554
20	213	194	8.920
50	213	184	13.615

Note: ww = wastewater

Table 5. The adsorption capacity of copper by natural- and calcined-zeolite based on eq 2.

Dosage of zeolite (g/L)	Adsorption capacity of natural zeolite (ppm/g)	Adsorption capacity of calcined zeolite (ppm/g)
5	5.263	12.281
25	2.729	3.244
50	1.345	3.088
100	0.296	0.919
250	0.100	0.578

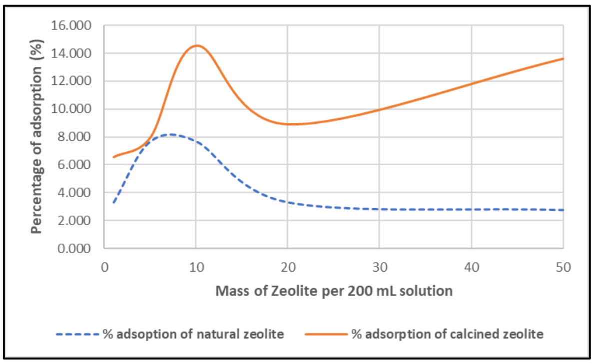

Figure 5. The graph of copper adsorption by natural zeolite and calcined zeolite.

The adsorption effectivity (%) of Copper by natural zeolite is smaller than the calcined zeolite and the adsorption capacity of calcined zeolite is higher than the natural zeolite. There is an increase in the adsorption capacity of calcined zeolite compared to the natural zeolite. The calcined zeolite would have cleaner pores to bind copper and trapped it in the inner framework of zeolite. The optimum adsorption reached by using 10 gram of calcined zeolite per 200 mL of wastewater solution (Figure 5). However, we observe here that the more mass of zeolite used in adsorption does not mean the more copper adsorbed. High amount of zeolite will limit the movements of zeolite particles in solution, with a constant speed of stirring, resulting less interaction of copper to zeolite and less copper was adsorbed. This phenomenon is observed in figure 6, as the dosage of zeolite increased the adsorption of copper per gram of zeolite decreased.

The pH of the solution also influences the ability of zeolite to adsorb copper from solution. Concentration of H^+ in solution also play important role in zeolite ability to bind Copper. The lower of pH due to the higher of the concentration of H^+, the smaller of the adsorption of copper was observed (Ahmad et al., 2017; Wang et al., 2007). From this study, we can clearly observe that the calcined zeolite is still able to adsorb Copper at a very low pH.

Conclusion

This study concludes that mordenite is the main phase mineral in local natural zeolite. The calcined zeolite has better Copper adsorption ability compared to local natural zeolite. The optimum Copper adsorption percentage at pH 0.3 is using 10 g of acid activated-calcined zeolite, in 200 mL of wastewater solution. Acid activation with HCl 3 M could remove quartz impurity from natural zeolite. The calcination at 400 ˚C did not destroy zeolite framework but only decreasing its crystallinity.

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