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REMOVAL OF Cd^{2+} IN WASTEWATER USING ZEOLITIC IMIDAZOLATE FRAMEWORK-8 (ZIF-8) AS SYNTHESIS ADSORBENT

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ABSTRACT

Zeolitic Imidazole Framework-8 (ZIF-8) has been synthesized by dissolving $Zn(NO_3)_2.4H_2O$ and 2-MeIM into a methanol solvent and solvothermal method. The synthesized ZIF-8 will be applied as a cadmium (Cd) adsorbent in Polluted water. Several characterization methods were conducted, including XRD, FTIR, and AAS. The XRD analysis shows that the prominent characteristic peaks of ZIF-8 at an angle of 2θ = 7,25°; 10,26°; 12,57°; 16,22°; and 17,86°. At the same time, the FTIR analysis showed the presence of functional groups of Zn-N, C-N, C=N, C=C, and C-H on aromatic 2-Methyl Immidazole as an indication that ZIF-8 has been completely established. Based on the result, the AAS method performed to analyze ZIF-8 ability to adsorb Cd in the water showed that Cd ions adsorption increased at pH 6 and 8 while decreasing at pH 7.

Keywords: Cd, Adsorption, Adsorbent, ZIF-8.

INTRODUCTION

Water is essential to life, and as the population grows quickly and industries develop, there is a daily rise in the demand for clean water (Li et al., 2021). Water pollution is one of the problems that occur in various regions. Polluted water is hazardous to use by living things. Some of the causes of water pollution are heavy metal contamination (Riskirana, 2018). The main threats to human health from heavy metals are lead, cadmium, nickel, mercury, and arsenic exposure. These metals have been extensively studied, and global bodies such as the World Health Organization (WHO) regularly review their effects on human health (Pap et al., 2016). Human lead poisoning results in severe damage to the brain system, kidney, liver, and reproductive system, as well as decreased hemoglobin production, mental retardation, infertility, and abnormalities in pregnant women (Malkoc & Nuhoglu, 2005).

One of the heavy metals found in water is cadmium (Cd). Cd metal is an inorganic metal with a reasonably high toxicity level and is non-biodegradable (Purnamawati et al., 2015). Cd is a hazardous pollutant metal and poisonous. The Cd has a high affinity for sulfhydryl and binds with sulfur (sulfhydryl groups), making the enzyme *immobile.* Carboxyl groups (-COOH) and amino ($-NH_2$), which are abundant in the body, can also react with Cd. In the body, Cd will bind to these groups and accumulate and cause health problems for humans, such as bronchitis, bone fractures, hypertension, lung and liver damage, and kidney disorders (Riani et al., 2017). Certain metals are carcinogenic, result in birth abnormalities, damage many organs, and impair learning. Due of these dangers, heavy metal ions in wastewater are a major global concern (Adewumi et al., 2021). In quite a while, in order to meet the Sustainable Development Goals (SDGs) 3 and 6, we must identify innovative remediation techniques that are affordable, environmentally responsible, and energy-efficient to get rid of trace toxins (Ping et al., 2013).

Previous studies reported that several methods were used to reduce heavy metal contamination, including ion exchange, evaporation electrochemical, and membrane separation. However, this method is uneconomical and has high energy costs (Li et al., 2021). Effective, simple, economical, and nontoxic side effects methods are required, such as adsorption (Zhang et al., 2016). Adsorption is the process of absorption of a substance (molecule/ion) on the surface of the adsorbent (Megasari et al., 2019).

In this study, the adsorbent used was Zeolitic Imidazolate Framework-8 (ZIF-8). The ZIF-8 is a variation of ZIF, a subclass of Metal-Organic Framework (MOF) with a sodalite-shaped framework structure (SOD). ZIF is a porous material containing divalent metal connected with an imidazole ligand (Aulia et al., 2020). The ZIF-8 skeleton is formed from the Zn^{2+} cations and the 2-methylimidazole ligand (2-MeIM), which is tetrahedrally coordinated to create three dimensions (Jofrishal et al., 2020). ZIF-8 reported has a pore size of 3.4 in diameter, a pore cavity of 11.1, a large surface area reaches 1300-1600 m^2/g (Ordoñez et al., 2010), and high thermal stability and resistance (Jofrishal et al., 2020). Considering several fundamental properties of the ZIF-8, the ZIF-8 can be used as an adsorbent for polluted water. Several investigations demonstrate that ZIF-8 effectively removed organic and arsenic contaminants from aqueous solutions As(III) and As(V) were of 49.49 and 60.03 mg/g (Jian et al., 2015). Regarding adsorption isotherms, kinetics, thermodynamics, desorption, and adsorbent regeneration, (Jiang et al., 2013) showed that ZIF-8 was a new adsorbent for the quick removal of 1H-benzotriazole and 5-tolyltriazole in aqueous solution with the adsorption capacity of 298.5 and 396.8 mg/g. This research is expected to determine the ability of ZIF-8 material as an adsorbent for Cd(II) metal to increase the potential of ZIF-8 material to reduce the effectiveness of heavy metals because it will severely affect our environment and water ecosystem (Tan et al., 2017). The design of selective adsorbents for efficient removal of ions with fast kinetics and high adsorption capacities, using simple and versatile synthetic routes for economic production at scale, is thus critical (Bui et al., 2020).

RESEARCH METHOD

Equipment and Material

The equipment used X-Ray Diffraction (XRD) by scanning an angle of 2θ at 5º-70º. The X-ray source is CuKα (λ = 1.5418) at 40 kV and a current of 30 mA. For analysis with Fourier Transform Infra-Red (FTIR) and the absorbance was measured at a wave number of 500–4,000 cm^{-1} (Shimanzu), oven (Gallenkamp England), analytical balance, and magnetic stirrer. The material was zinc nitrate butahydrate ($Zn(NO_3)_2.4H_2O$) (Sigma-Aldrich), 2-Methylimidazole ($C_4H_6N_2$) (Sigma-Aldrich), methanol (CH_3OH) (Sigma-Aldrich) and Whatman filter paper.

Synthesis Zeolitic Imidazolate Framework-8 (ZIF-8)

$Zn(NO_3)_2.4H_2O$ as much as 3.12 g was dissolved with 42.4 mL of MeOH to produce a solution ($Zn(NO_3)_2.4H_2O$). Then 3.92 g of 2-MeIM was dissolved with 42.4 mL of methanol to produce a 2-MeIM solution. Then two solutions were mixed and stirred using a magnetic stirrer at room temperature for an agitation speed of 250 rpm for 1 hour (Tan et al., 2017). The mixture is heated at 70°C for 4 hours in the next process. After heating, the solution was allowed to stand for 24 hours. Then the solution was decanted to separate the residue and filtrate. The formed precipitate was washed with methanol and stood for 24 hours. The sediment was filtered and washed again with methanol. The solid obtained was dried at 100°C using an oven for 2 hours (Jofrishal et al., 2020).

ZIF-8 Materials were characterized using X-Ray Diffraction (XRD) by scanning an angle of 2θ at 5º-70º. The X-ray source is CuKα (λ = 1.5418) at 40 kV and a current of 30 mA. For analysis with Fourier Transform Infra-Red (FTIR), ZIF-8 material is mixed with KBr and ground, then formed into pellets. The pellet was put into the sample holder, and the absorbance was measured at a wave number of 500– 4,000 cm^{-1}.

Determination of Cd(II) Metal Adsorption Ability to Percentage of Adsorption

The pH was determined using pH variations of 6, 7, and 8. The $CdSO_4$ solution of 50 ml with a concentration of 10 ppm was put into a beaker, and then H_2SO_4 and NaOH were added to adjust the variation predetermined pH. Then 0.1 g of adsorbent was added and stirred using a shaker for 60 minutes. Cd(II) solution that has been mixed with adsorbent and then filtered. The concentration of Cd(II) in The filtrate was determined using the Atomic Absorption Spectrophotometer (AAS). The equation can calculate the percentage of Cd(II) metal adsorption:

$$\% \text{ Adsorption} = \frac{\text{Ci - Ce}}{\text{Ci}} \times 100\% \qquad (1)$$

Where, Ci = initial concentration of metal (mg/L),

Ce = final concentration of metal (mg/L)

Determination of The Adsorption Capacity of Cd(II) Metal

The adsorption capacity was determined based on the data obtained from the results of the AAS analysis. The data obtained is then calculated as adsorption capacity using the equation:

$$\text{Qe} = \frac{\text{Co - Ce}}{\text{W}} \times \text{V} \qquad (2)$$

Where Qe is adsorption capacity (mg/g), Co = initial concentration (mg/L), Ce = final concentration (mg/L), W = adsorbent mass (g), and V = volume of solution (L).

RESULTS AND DISCUSSION

Synthesis Zeolitic Imidazolate Framework-8 (ZIF-8)

The synthesis of ZIF-8 was carried out by dissolving the solid ($Zn(NO_3)_2.4H_2O$) and 2-MeIM each into a methanol solvent. Using methanol solvent aims to produce ZIF-8 material maximally by using room temperature during the reaction process (Ordoñez et al., 2010). Over 24 hours, the ZIF-8 remained stable in aqueous solutions with pH values ranging from 3 to 12. At this pH, ZIF-8 is able to maintain its crystallinity properly, which means that the structure of ZIF-8 is not damaged (Thanh et al., 2018).

Based on the observations, the mixture turns cloudy white after the two are mixed. The cloudy white indicates that the bond between the metal ion and the ligand has formed. Then the solution is heated in an oven at a temperature of 70°C. The temperature used is a temperature close to the boiling point of methanol.

Then the solution was allowed to stand for 24 hours at room temperature to obtain a precipitate which would be washed with methanol. The washing process with methanol aims to enlarge the pores of the ZIF-8 material. Then a further drying process was carried out at

100^oC for 2 hours to remove the remaining methanol in the ZIF-8 material. The synthesis results are in the form of white ZIF-8 material (Figure 1).

(a) **(b)**

Figure 1. (a) Washing Process (b) Material ZIF-8

Characterization of Synthesis Result

ZIF-8 material was characterized using XRD to determine the crystallinity of ZIF-8 material. The X-ray diffraction pattern can be seen in Figure 2. The XRD diffraction pattern showed the presence of main characteristic peaks at angle $2\theta = 7.25^o$; 10.26^o; 12.57^o; 16.22^o; and $17.86^{o,}$ with the highest intensity at $2\theta = 7.25^o$. The XRD pattern of ZIF-8 showed a clearly defined diffraction (011) at two theta = 7.25, which suggests that ZIF-8's crystallinity in this study was reasonably high (Thanh et al., 2018). However, at an angle of $2\theta = 6.60^o$, there is another peak that indicates the presence of a compound $Zn(OH)(NO_3)(H_2O)$ formed (Nadifah & Ediati, 2015). The ZIF-8 material produces an X-ray diffraction pattern with a small peak width and high intensity. The result indicates that the ZIF-8 material has a large particle size and high crystallinity. The highest crystallinity of ZIF-8 is at $2\theta = 7.25^o$. This is proven by the appearance of the highest intensity at that angle.

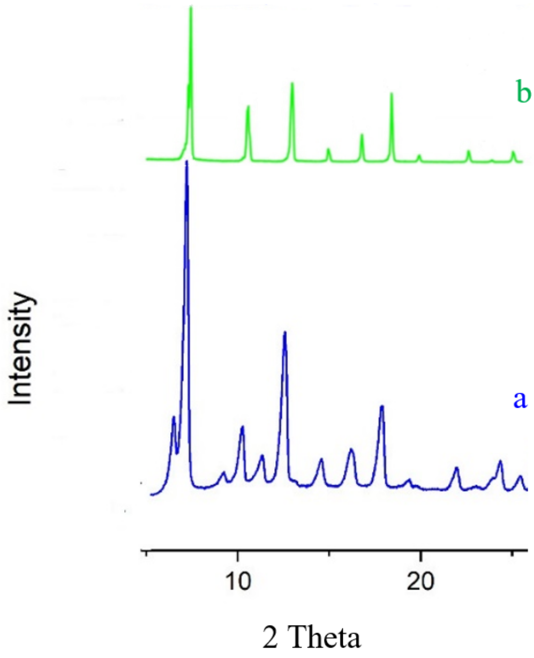

Figure 2. X-Ray Diffraction Pattern (a) ZIF-8 (b) ZIF-8 of Reference ((Nadifah & Ediati, 2015)

ZIF-8 material was characterized using FTIR to determine the presence of functional groups from ZIF-8 material. The structure and spectrum of the ZIF-8 material can be seen in Figures 3 and 4. In the FTIR spectrum, there is an absorption band at a wave number of 422 $cm^{-1,}$ which shows the stretching vibration of Zn-N, indicating that the ZIF-8 material has formed a bond between Zn and N on the 2-MeIM ligand. In the absorption band, the wave number of 1.159 cm^{-1} indicates C-N stretching vibrations. In the absorption band, wavenumber 1.581 cm^{-1} with weak intensity indicates stretching vibrations C=N. The weak absorption band is because the Zn atom has bonded to the N atom in the 2-MeIM ligand, so the number of C=N bonds becomes less and less. In the absorption band, wave number 1.730 cm^{-1} indicates the presence of stretching vibrations C=C, and the absorption band at wave number 3.128 cm^{-1} indicates the presence of C-H bonds in the aromatic group 2-MeIM (He et al., 2014). The stretching N-H vibration of the imidazole is responsible for the peaks at 1.681 and 1.591 cm^{-1}, respectively. The spectrum obtained from FTIR produces functional groups similar to the structure of the ZIF-8 material. This indicates that the ZIF-8 material has been successfully characterized.

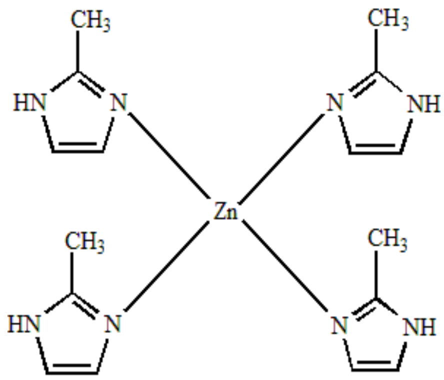

Figure 3. ZIF-8 Material Structure

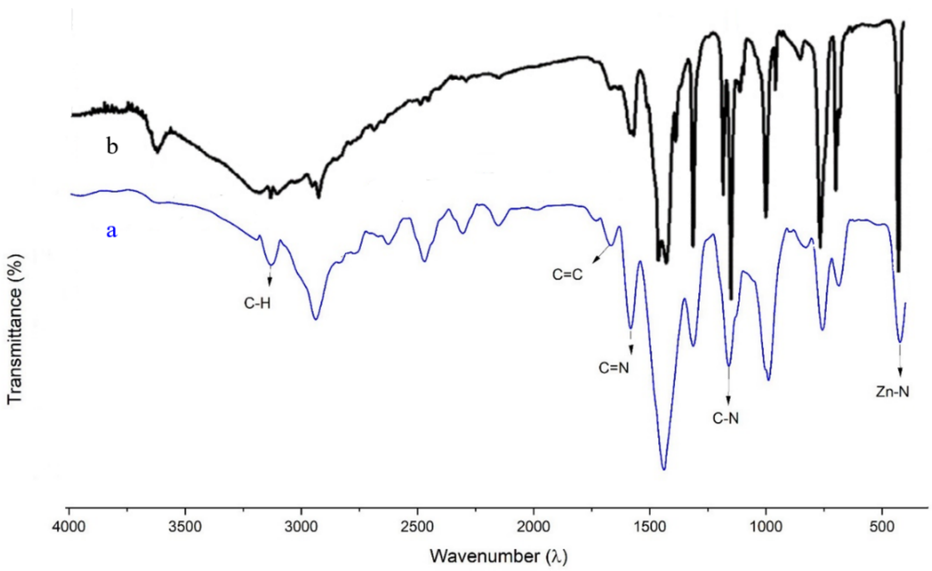

Figure 4. FTIR Spectrum of ZIF-8 Material (a) ZIF-8 (b) ZIF-8 of Reference (He et al., 2014)

Determination of Cd (II) Metal Adsorption Ability to Percentage of Adsorption

One application of ZIF-8 material is as an adsorbent for heavy metals, namely Cd metal. The pH of the solution can influence the ability of the adsorbent ZIF-8 to adsorb Cd metal. The solution used was pH 6, 7, and 8. The curve of pH variation to the adsorption percentage is shown in Figure 5.

Figure 5 shows that at pH 6, it becomes a good condition in the adsorption process. This happened because the ability of ZIF-8 to absorb Cd metal ions increased with the highest adsorption percentage value of 93.84%. This condition occurs due to reduced competition between protons (H^+) and Cd metal ions, resulting in low repulsion of Cd metal ions which causes Cd metal ions to enter the surface of ZIF-8 to replace Zn (Binaeian et al., 2020) (Wijaya & Ulfin, 2015). The adsorption mechanism of Cd metal using ZIF-8 is a cation exchange reaction seen in Figure 6. In the Cd metal adsorption mechanism, Zn^{2+} in ZIF-8 will be replaced by metal ions Cd^{2+} to form a new complex. This is because Zn^{2+} and Cd^{2+} have the same charge, so Cd^{2+} can easily replace Zn^{2+} in ZIF-8.

At pH 7, the adsorption percentage decreased to 88.99%. The decrease in adsorption percentage occurred at pH 7 (neutral) because, at neutral pH, Cd metal ions can undergo hydrolysis reactions in solution so that they are unstable and cause the ability of the adsorbent to absorb Cd metal ions to decrease (Stiyati Prihatini & Syauqiah, 2017). The hydrolysis reaction that occurs can be seen as follows:

$$Cd^{2+} + 2H_2O \rightleftharpoons Cd(OH)_2 + 2H^+$$

At pH 8, the adsorption percentage increased to 89.85% because the Cd metal ions in the solution reacted with hydroxide ions to form a precipitate, causing the Cd metal ions in the solution to decrease. The precipitation can affect the interaction of the adsorbent with metal ions Cd in the solution. As more Cd metal ions settle, the number of Cd metal ions remaining in the solution decreases, so the measured absorption increases (Rahman et al., 2012). The precipitate hydroxide reaction can be seen as follows:

$$Cd^{2+} + 2OH^- \rightarrow Cd(OH)_2$$

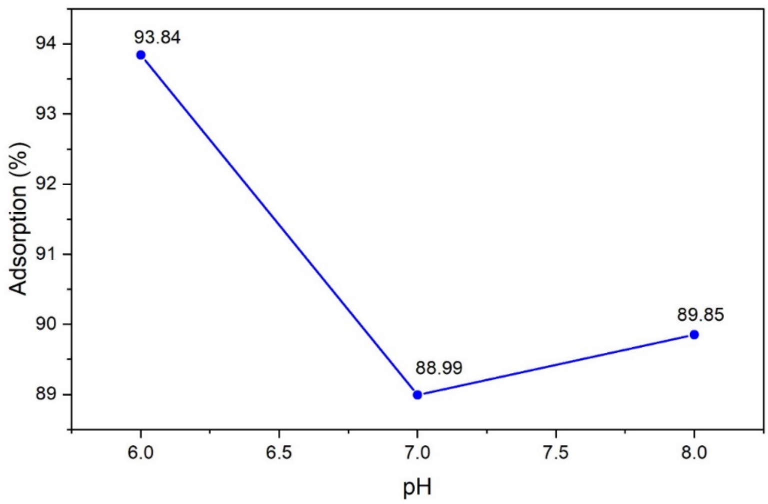

Figure 5. The curve of pH Variation to Percentage of Adsorption

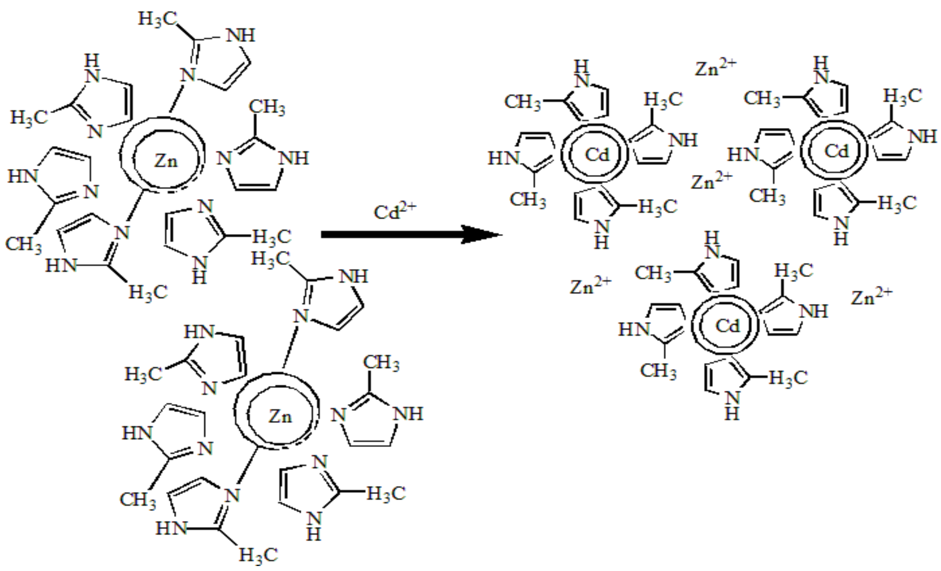

Figure 6. Cd(II) Metal Adsorption Mechanism Using ZIF-8

Determination of The Adsorption Capacity of Cd(II) Metal

Adsorption capacity is an adsorbent's ability to adsorbate (Side & Herawati, n.d.). Determination of adsorption capacity aims to determine the number of metal ions Cd^{2+} adsorbed on the surface of the adsorbent. The curve of pH variation on adsorption capacity is shown in Figure 7. Figure 6 shows that at pH 6, the highest adsorption capacity value was obtained at 4.692 mg/g. At the same time, the lowest adsorption capacity value occurred at pH 7, which was 4.450 mg/g. At pH 8, the adsorption capacity value increased again to 4.493 mg/g. Based on the results obtained, the maximum adsorption of metal pb^{2+} takes place in acidic conditions (Huang et al., 2018).

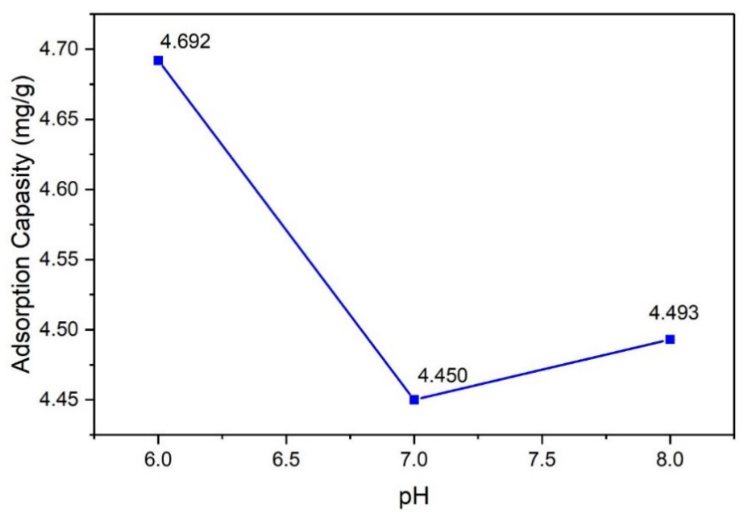

Figure 7. The curve of pH Variation on Adsorption Capacity

Conclusion

This study aimed to synthesize ZIF-8 and observe its ability to adsorb Cd metal. Some conclusions can be drawn: Cd metal ions adsorbed are increased at pH 6 and 8 while decreased at pH 7. This phenomenon is attributed to the Cd metal ions at pH 8 being able to form hydroxide deposits, causing the Cd metal ions contained in the solution to decrease. ZIF-8 material can be used as an adsorbent for Cd metal with the optimum adsorption conditions at pH 6 with an adsorption percentage of 93.84% and an adsorption capacity value of 4.692 mg/g.

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