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The Effect of pH on Adsorption Capacity Based on the Ionic Imprinted Polymer-Cd(II) Method

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ABSTRACT

Population growth and technological advancement in Indonesia have contributed to increasing environmental pollution, particularly from industrial waste containing heavy metals such as cadmium (Cd^{2+}). This pollution poses serious health risks as cadmium can enter the food chain through aquatic ecosystems. Therefore, effective separation methods are required to remove cadmium from water bodies. One such method is the use of Ionic Imprinted Polymer (IIP), which offers high selectivity toward specific metal ions. This study aimed to determine the adsorption capacity of cadmium using the IIP method, synthesized via precipitation polymerization, over pH values ranging from 3 to 8. $CdCl_2$ was used as the ion template, combined with Na_2EDTA as a chelating ligand, methacrylic acid (MAA) as the functional monomer, ethylene glycol dimethacrylate (EGDMA) as the crosslinker, and benzoyl peroxide (BPO) as the initiator. Characterization was conducted using Fourier Transform Infrared Spectroscopy (FTIR) to identify the functional groups and confirm template removal, and Scanning Electron Microscopy (SEM) to observe surface morphology. Adsorption performance was tested using Atomic Absorption Spectrophotometry (AAS). The IIP showed a specific absorption at 520 cm^{-1} (Cd–O stretch), confirming the presence of cadmium-binding sites, and exhibited a porous morphology, unlike the dense structure of the blank polymer. The results showed that the optimum adsorption of Cd^{2+} occurred at pH 6, with an adsorption capacity of 4.47 mg/g, which was higher than that of the non-imprinted polymer (3.08 mg/g at pH 4). The improved adsorption performance at pH 6 is attributed to the predominance of the deprotonated EDTA form (Y^{4-}), which forms a stable complex with Cd^{2+} ($Kf \approx 10^{16.5}$), thereby enhancing selective adsorption. These findings confirm that IIP-Cd is an effective material for cadmium removal from aqueous environments, with pH playing a critical role in optimizing adsorption capacity.

Keywords: Adsorption, Cadmium, IIP, Optimum pH, Precipitation polymerization

1. Introduction

The rapid population growth and advances in science and technology in several major cities in Indonesia have resulted in public health problems due to environmental pollution from household waste and the uncontrolled expansion of the industrial sector, which produces waste without adequate processing. Humans as consumers feel the impact of heavy metal pollution. The concentration of heavy metal pollution, which continues to increase, can have profound implications for humans. This pollution is often found in various conditions, including on the surface of seawater and other water. These toxic heavy metals easily enter the food chain through aquatic and marine species, thus threatening the ecosystem and human health that depend on these resources [1].

In general, heavy metals are naturally present in water at very low levels, namely, less than 1 µg/L. The concentration of metals in water can be categorized by level of pollution: non-polluted, lightly polluted, moderately polluted, and heavily polluted. Water that is heavily polluted generally has metal concentrations exceeding the safe threshold of ≥ 0.01 mg/L. At a moderate level of pollution, the concentrations of heavy metals in water and

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organisms are within a limited range, around 0.003mg/L –0.01 mg/L. Meanwhile, at a non-polluted level, the concentrations of heavy metals in water and organisms are very low and cannot even be detected, at ≤ 0.003 mg/L [2].

Most industrial waste contains heavy metals, including cadmium. Industries that produce cadmium waste include the textile dyeing industry, where certain dyes can contain heavy metals such as cadmium [3]. In the paint industry, the most common metal waste includes iron, lead, copper, and cadmium [4]. In the battery industry, waste can be in the form of assembly material residues, such as nickel-cadmium. In the paper printing industry, the printing process uses inks and chemicals that contain heavy metals such as cadmium [5], and in home industries, such as the batik industry, some synthetic dyes used in the batik coloring process contain heavy metals such as cadmium [6]. Welding workshop workers are also potentially exposed to heavy metal vapors, including cadmium vapor produced by the welding process [7]. Cadmium has strong bioavailability, allowing it to quickly enter the food chain, including through human consumption of contaminated vegetables and fish. Cadmium that enters the food can contaminate the human body, thereby causing cardiovascular, kidney, and cancer diseases [8].

Many methods can be used to remove heavy metal cadmium from wastewater, including coagulation, flocculation, precipitation, adsorption, membrane filtration, electrolysis, photocatalytic degradation, ion exchange, oxidation, reduction, and solvent extraction [9]. Ionic Imprinted Polymer (IIP) is a method used to reduce heavy metal levels, such as cadmium (Cd), by producing an adsorbent selective for the desired metal ion. Other methods have limitations in selectivity, efficiency at low concentrations, or require additional processing for the resulting concentrated waste. IIP has the advantage of high selectivity and easy preparation. The IIP method produces a metal-ion mold bound to the polymer, then releases it from the polymer matrix, creating a mold selective for the printed ion. The high selectivity of IIP is due to its ability to remember a polymer's interaction with specific ligands, the coordination geometry and coordination number of metal ions, the metal ion's charge, and the size of the metal ion [10]. The increase in cadmium absorption is also influenced by pH, redox state, and solution ionic strength [11].

In this research, the composition modification was carried out using the Na_2EDTA ligand, which forms hexadentate chelate complexes that coordinate to cadmium (II) ions (Cd2+). The use of Na_2EDTA as a ligand is expected to increase the selectivity and efficiency of cadmium ion adsorption due to its strong binding affinity for heavy metals. In the manufacture of this IIP, methacrylic acid (MAA) is also used as a functional monomer, benzoyl peroxide (BPO) as an initiator, and ethylene glycol dimethacrylate (EGDMA) as a crosslinker to form polymers with stable, specific structures. The adsorption pH-optimization test was conducted to determine the optimal pH for the adsorbent to capture cadmium ions. The adsorption capacity was measured using an Atomic Absorption Spectrophotometer (AAS), and the functional groups were identified using a Fourier Transform Infrared (FTIR) spectrometer.

2. Methods

This research is included in a proper experimental design with the research target to determine the effect of the pH medium adsorption on the adsorption capacity of cadmium by synthesizing IIP-Cd(II) adsorbent material as an adsorbent of Cd(II) metal ions using the precipitation polymerization method.

2.1. Materials and Tools

The materials used include $CdCl_2$ powder, disodium dihydrogen ethylenediaminetetraacetate (Na_2EDTA), benzoyl peroxide (BPO), methyl methacrylate (MAA), ethylene glycol dimethacrylate (EGDMA), nitrogen, ethanol, nitric acid, sodium hydroxide, acetonitrile, and distilled water. Equipment includes a magnetic stirrer-heater, vacuum filtration, Whatman no. 42 filter paper, aluminium foil, plastic wrap, a Fourier transform infrared (FTIR) spectrometer, and an atomic absorption spectrophotometer (AAS).

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2.2. Experiment

The first thing to do is to weigh 0.0201 grams (0.1 mmol) of $CdCl_2$ and 0.0931 grams (0.25 mmol) of EDTA into a mixture of solutions (2:1 v/v ethanol-acetonitrile), 60 mL. Then, it was stirred with a magnetic stirrer for 30 minutes, and then 340 µL (4 mmol) of MAA, 0.0484 grams (0.2 mmol) of BPO, and 3.9644 grams (20 mmol) of EGDMA were added. After that, it was flushed with nitrogen gas for 5 minutes. Then place it in a water bath at 70 ℃ with a stirrer until it becomes a paste. The polymer formed was filtered under vacuum, washed with 100 mL of ethanol and 100 mL of aquabidest, and then dried in an oven at 60 ℃ until constant weight. The resulting NIP powder will then be used for IIP extraction.

The dried NIP, with a constant weight of up to 0.5 grams, was extracted with 100 mL of 1 M HNO_3 and stirred for 5 hours at 650 rpm using a magnetic stirrer. The residue was then dried in an oven at 60 ℃ until constant weight. The dried IIP is then used to adsorb target ions at a specific pH.

In the adsorption stage, weigh 0.05 g of IIP-Cd(II), then add 50 mL of a 25 ppm $CdCl_2$ standard solution at pH 3, 4, 5, 6, 7, and 8. The solution is stirred with a magnetic stirrer for 2 hours, then filtered through filter paper, and analyzed by AAS to determine the metal content in the filtrate.

The results were then analyzed using an Atomic Absorption Spectrophotometer (AAS) to measure the metal content in the filtrate and a Fourier Transform Infrared (FTIR) instrument to determine the functional groups present in the IIP adsorbent.

3. Results and Discussion

This research aims to determine the effect of the adsorption media's pH on the adsorption capacity of cadmium using the precipitation polymerization method. Synthesis of Ionic Imprinted Polymer (IIP) Cd(II) was carried out by printing a 0.1 mmol $CdCl_2$ solution. The synthesis results will be characterized and quantified using an Atomic Absorption Spectrophotometer (AAS) and a Fourier Transform Infrared (FTIR) spectrometer to assess the measurement success by determining the product concentration and the identified functional groups.

3.1. Synthesis of PB, NIP, and IIP

The synthesis of Non-Imprinted Polymer (NIP) by the precipitation polymerization method involves selecting templates, ligands, porogenic solvents, functional monomers, crosslinking agents, and initiators. Cadmium solution ($CdCl_2$) is used as a template to form a stable complex compound with cadmium metal ions dissolved in water; a chelating agent in the form of a Na_2EDTA ligand is required. Na_2EDTA as a chelating ligand will surround the Cd(II) ion and form a stable complex. This process helps create a pattern that resembles the bond between Cd(II) and the polymer [12]. The porogenic solutions used are ethanol and acetonitrile. This porogen forms a pore structure within the polymer and a specific binding site that later facilitates the binding of target ions [13]. Methacrylic acid (MAA) is a functional monomer that forms a prepolymerization complex with a template, providing functional groups, thereby determining the strength or weakness of the interaction with the template. MAA has an effective bond with its template, both covalently and noncovalently [14]. EGDMA is one of the most commonly used crosslinkers for MIP synthesis because it contains an ester functional group that can crosslink monomers [15]. The initiator, benzoyl peroxide (BPO), initiates the polymerization of functional monomers and crosslinkers during synthesis. In this synthesis process, a white solid product is produced. The synthesis of Blank Polymer (PB) is carried out in the same way as the synthesis of NIP, but without a template ($CdCl_2$) added to the manufacturing process.

IIP is a type of cross-linked polymer that has pores and specific binding sites that are then designed to recognize and absorb targeted charged ions and molecules selectively [16]. During IIP preparation, the dried NIP is extracted with HNO_3. The purpose of giving HNO_3 is to separate the bond between the cadmium metal ion and the adsorbent so that a specific metal ion mold is formed. The result of the IIP manufacturing process is a white-colored solid.

3.2. Analysis of the effect of pH on the adsorption of IIP-Cd(II)

This research aims to determine the optimal adsorption capacity of target ions at specific pH

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values. The initial step is to weigh 0.05 grams of IIP-Cd(II) and then add 50 mL of a 25 ppm $CdCl_2$ standard solution at pH 3, 4, 5, 6, 7, and 8. The pH of the solution during adsorption affects the adsorption capacity. The solution is then stirred with a magnetic stirrer for 2 hours. After 2 hours, the solution is filtered through filter paper, and AAS analyzes the filtrate to determine the metal content. The results of the AAS reading are calculated using the equation [17] :

$$Qe = \frac{Co - Ce}{m} \times V$$

Description:
Qe = adsorption capacity (mg/g)
Co = initial concentration of Cd metal ions (mg/L)
Ce = final concentration of Cd metal ions (mg/L)
m = mass of adsorbent/IIP (g)
V = volume of solution (L)

Table 1. Comparison of PB and IIP adsorption capacity

pH	Adsorption Capacity PB (mg/g)	Adsorption Capacity IIP-Cd (mg/g)
3	2,94	3,32
4	3,08	3,52
5	2,58	3,71
6	1,49	4,47
7	1,19	1,67
8	0,54	0,87

The table shows a comparison of the adsorption capacity of the blank polymer, which increased at pH 3-4, then decreased at pH > 5. The optimal pH for the blank polymer was 4, with an adsorption capacity of 3.08 mg/g. In IIP, adsorption capacity increased over the pH range 3-6 and decreased at pH > 7. The optimal pH in IIP was 6, with an adsorption capacity of 4.47 mg/g. The pH value can affect the distribution of supply at the mineral surface by altering protonation and deprotonation at the active site [18].

At increasingly acidic pH values, ionization increases, but at more basic pH values, precipitation increases. At higher pH values, adsorption capacity decreases due to the high concentration of OH^- in the solution. Cadmium metal will bind to OH^- ions to form the compound $Cd(OH)_2$ [19].

The influence of pH on the adsorption performance of the IIP is strongly related to the stability of the cadmium-ligand complexes formed during the process. In this study, ethylenediaminetetraacetic acid (EDTA) was used as a ligand due to its ability to create highly stable chelate complexes with cadmium ions. The formation constant (Kf) of the Cd-EDTA complex is approximately $10^{16.5}$ at 25 °C, indicating a robust and stable coordination interaction under neutral to slightly alkaline conditions [11]. However, at lower pH values, competition between protons (H^+) and metal ions (Cd^{2+}) for the EDTA ligand reduces the availability of deprotonated EDTA, thereby lowering the conditional formation constant (Kf).

The conditional formation constant (K_f) is influenced by the degree of deprotonation of EDTA, which depends on pH. At low pH (e.g., pH 3–4), EDTA is mostly in protonated form (H_4Y or H_3Y^-), limiting its ability to bind metal ions. As pH increases, more EDTA exists as Y^{4-}, the fully deprotonated form that effectively chelates Cd^{2+}. This explains the increase in adsorption capacity up to pH 6 [12].

In contrast, the cadmium–cyanide complex (Cd–CN) has a lower formation constant, with $K_f \approx 10^{16}$. Still, it is typically stable in more basic solutions because it requires excess CN^-. Since CN^- is not present in this study, the formation of Cd–CN complexes does not significantly influence adsorption behavior. The simplified expression for the conditional formation constant can be given as:

$$Kf = \frac{[CdY^{2-}]}{([Cd^{2+}][Y^{4-}])}$$

But, in practice, it depends on the distribution of all protonated forms of EDTA and the free Cd^{2+} in solution. Thus, the observed optimum at pH 6 aligns with the highest effective concentration of Y^{4-}, maximizing complex formation and adsorption.

3.3. Functional Group Analysis using FTIR

In this study, the FTIR technique was used to identify functional groups in PB, NIP, and IIP in the wave-number range of 4000 cm^{-1} to 500 cm^{-1}. Figure 1 shows that all polymers have almost identical functional groups: the O-H group from alcohol, the O-

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H group from carboxylate, the C=O group resulting from EGDMA cross-linking, and the typical C-N group from the Na_2EDTA ligand.

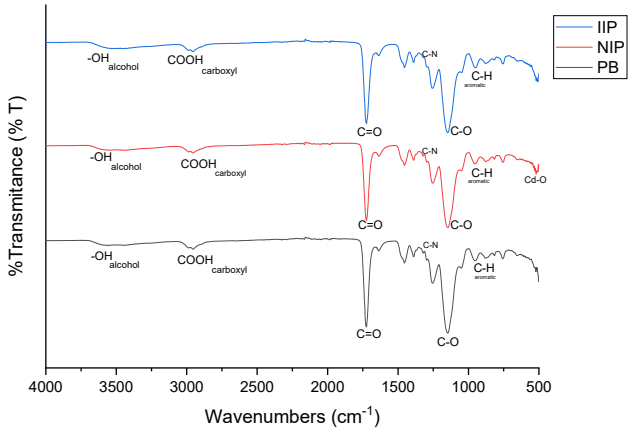

Figure 1. FTIR spectra of IIP, NIP, and PB

The difference is evident in NIP, where the number of waves changes slightly due to the presence of metal. For example, in the C=O group in PB, with a wave number of 1723 cm^{-1}, while in NIP, 1722 cm^{-1}. After the extraction process, the IIP produced from NIP showed a shift in the wave number toward that of the blank polymer, namely 1725 cm^{-1}, which was attributed to the release of the metal template from the polymer. In addition, the Cd-O group was observed at 520 cm^{-1}, indicating a bond between the metal and the ligand in the 500-700 cm^{-1} range [20].

3.4. Morphological Analysis using SEM

Figure 2 and Figure 3 show the surface morphology of the Blank Polymer (PB) and the Ionic Imprinted Polymer for Cd(II) (IIP-Cd(II)), which was analyzed using Scanning Electron Microscopy (SEM) at 25,000x magnification. The SEM image of PB shows a relatively dense, aggregated surface with overlapping particles and no clearly visible pores. This indicates that PB lacks the specific binding sites necessary for selective adsorption.

In contrast, the SEM image of IIP-Cd(II) shows a rougher, more porous surface with dispersed particles and microcavities. These pores result from the ion-imprinting process, in which Cd(II) ions act as a template during polymerization. After the template is removed by acid extraction, specific cavities matching the shape and coordination environment of Cd(II) are formed within the polymer matrix.

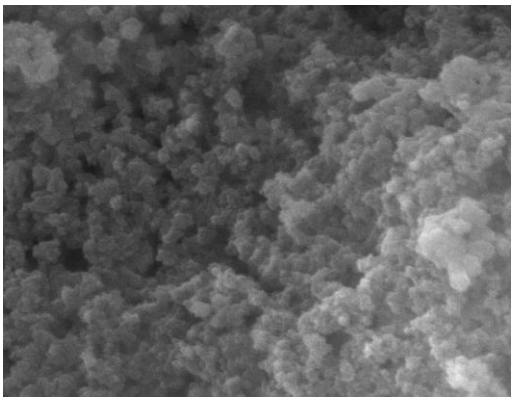

Figure 2. SEM image of PB at 25,000x magnification

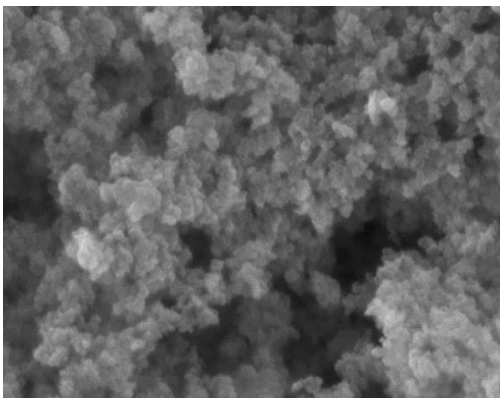

Figure 3. SEM image of IIP-Cd(II) at 25,000x magnification

The morphological difference between PB and IIP confirms the successful imprinting process, which forms selective binding sites on the polymer surface. The presence of these pores increases the specific surface area and availability of active sites, thereby enhancing the adsorption capacity of IIP compared to PB. This indicates that an increase in the IIP's porosity directly enhances both adsorption capacity and selectivity toward the target ion [21].

In addition, [22] reported that the porous surface structure resulting from the imprinting process plays a vital role in accelerating ion

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diffusion into the polymer matrix and facilitating more efficient adsorption.

4. Conclusion

Based on the research and discussion, it was concluded that the pH of the adsorption media affects the adsorption capacity of cadmium. The adsorption capacity of IIP-Cd in capturing Cd(II) metal ions at the optimum pH of pH 6 was 4.45 mg/g. While the adsorption capacity of the blank polymer, used as a control at the optimum pH of 4, was 3.08 mg/g, NIP showed an absorption at 520 cm^{-1}, corresponding to the stretching vibration of Cd-O. The increase in adsorption capacity at pH 6 is influenced by the stability of the Cd-EDTA complex, which has a high formation constant (Kf ≈ $10^{16.5}$). At this pH, EDTA predominantly exists in its deprotonated form (Y^{4-}), facilitating more effective complexation with Cd^{2+} ions and thereby enhancing selective adsorption.

The SEM analysis clearly demonstrated the morphological differences between the Blank Polymer (PB) and the Ionic Imprinted Polymer (IIP-Cd(II)). While PB exhibited a dense, aggregated surface lacking visible porosity, the IIP-Cd(II) showed a significantly more porous, rougher structure. These morphological features confirm the successful coordination of Cd(II) ions, resulting in the formation of specific cavities on the polymer surface. The enhanced porosity and surface roughness of IIP-Cd(II) are directly associated with increased active-site availability, thereby improving the material's adsorption capacity and selectivity for cadmium ions.

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