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Effect of Solution Concentration on the Adsorption Capacity of Ionic Imprinted Polymer (IIP) for Fe(III) Separation

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Abstract: Iron (Fe) contamination in Indonesian waters frequently exceeds the drinking water quality standard (0.3 mg/L) and poses risks to human health and aquatic ecosystems, highlighting the need for a selective and effective removal method. This study aimed to analyze the effect of solution concentration on the adsorption capacity of an EDTA-based Ion Imprinted Polymer (IIP) for Fe(III) ions. The research employed an experimental design through the synthesis of IIP using the precipitation polymerization method, with methacrylic acid (MAA) as the functional monomer, ethylene glycol dimethacrylate (EGDMA) as the crosslinker, and benzoyl peroxide (BPO) as the initiator. Data were collected through characterization using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy with Energy Dispersive X-ray (SEM-EDX), while Fe(III) concentrations were measured using Atomic Absorption Spectrophotometry (AAS). $Fe(NO_3)_3$ solutions at concentrations ranging from 5 to 35 ppm were tested at neutral pH. Data analysis was conducted by calculating the adsorption capacity as the difference between the initial and final concentrations. The results showed that increasing the initial concentration increased adsorption capacity until equilibrium was reached, with a maximum of 19.06 mg/g. FTIR analysis confirmed the presence of characteristic functional groups and the removal of Fe–O bonds after extraction, while SEM analysis revealed the formation of specific cavities in the IIP structure. In conclusion, the synthesized IIP demonstrates good selectivity and effectiveness for Fe(III) adsorption from aqueous solutions, indicating its potential application as a selective adsorbent for heavy metal-contaminated water treatment.

Keywords: Adsorption; Concentration; Fe(III); Ionic Imprinted Polymer; Precipitation.

Introduction

Indonesia, as a maritime country with 65% of its territory consisting of water, faces significant challenges related to iron (Fe) contamination in aquatic environments [1]. However, several reports indicate that Indonesian waters are affected by Fe metal pollution. The iron metal content in well water in Muara Sungai Tallo, Makassar City, was 0.8507 mg/L [2], and the iron metal content in the Belumai Deli Serdang River was 2.65 mg/L [3]. Then, it was stated that the iron metal content in the groundwater of Keputih Village, Sukolilo District, Surabaya City was 1.108 mg/L [4], while the water quality in five rivers in Surabaya, namely Canggu, Cangkir, Bambe, Karang Pilang, and Jagir, was found to be polluted and did not meet quality standards [5]. The iron content in the water ranged from 0.7486 to 39. 2775 mg/L, while in sediments it is 16944.24-83096.96 mg/kg. This exceeds the quality standard for iron (Fe) in drinking water, which is 0.3 mg/L, in accordance with Minister of Health Regulation (Permenkes) No. 492/Menkes/Per/VII/2010.

Iron (Fe) is one of the essential heavy metals that living things, including humans, need in small amounts. If the amount exceeds the tolerance limit, iron metal can cause cancer in humans. In freshwater bodies such as lakes, dissolved iron in the nanomolar range regulates the fertility and growth of phytoplankton, thereby affecting the aquatic food chain [6]). In addition, Fe plays an important role in the biogeochemical cycle, particularly through changes in its valence between Fe(II) and Fe(III), which affect nutrient availability and water chemical reactivity [7]. However, in high concentrations, Fe(II) ions can be toxic. Excessive exposure to iron in the blood can reduce haemoglobin levels, which can lead to anaemia [8]. Iron in water usually originates from various human activities, such as household and industrial activities [9]. To reduce excessive Fe levels, a removal method is needed.

The adsorption method has several advantages, namely flexibility, simple design, low cost, practical application, and resistance to reaction with toxic substances, making it non-toxic [10]. In general, adsorption is a phenomenon that occurs at the surface of a material [11]. There are many types of adsorption, distinguished by the adsorbent. Among others, there are activated carbon, zeolite, and IIP (Ionic Imprinted Polymer). Activated carbon is a type of graphite that is coarse and not perfectly structured. Activated carbon has a wide pore spectrum, ranging from obvious cracks and crevices to molecular dimensions. Due to its significant surface area, activated carbon is often used for various purposes, including removing impurities from air and water [12]. Activated carbon has limitations, including high cost, reliance on expensive raw materials, difficulty separating the powder from waste, and the need for expensive regeneration methods [13]. One method is adsorption with natural zeolite. Zeolite is a versatile non-metallic mineral or industrial mineral commodity used as an adsorbent or adsorbent medium [14]. Natural zeolite is known to have several shortcomings, including high levels

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of impurity ions such as Na^+, K^+, Ca^{2+}, Mg^{2+}, and Fe^{3+}, as well as low crystallinity. The presence of these impurity elements can reduce the activity and effectiveness of zeolite in its application [15]. In this study, we chose the IIP method because it can effectively identify ions dissolved in water, particularly heavy metals and radioactive elements, which are of increasing concern [16]. In addition to exhibiting high selectivity, Ion Imprinted Polymers (IIP) also have a very strong affinity for target ions thanks to the formation of specific cavities during the imprinting stage. These cavities form after the polymer extraction process and serve as recognition sites, enabling the polymer to bind target molecules with similar properties [17].

In this study, the precipitation method was used because it was considered simpler. This method involves the direct mixing of functional monomers, cross-linkers, initiators, and template ions in a solvent, followed by polymerisation. This process does not require additional steps such as grinding or re-moulding, making it more efficient and easier to perform [18]. Ethanol and acetonitrile are commonly used as porogens because they can effectively dissolve functional monomers and template ions, and they also play a role in forming the porosity and the final structure of the polymer. The use of ethanol as a porogen can produce IIP with high adsorption capacity for Fe(III) ions [19]. EGDMA is often used as a crosslinker because of its ability to form thermally and mechanically stable polymers, as well as its ability to enable rapid mass transfer during synthesis [20] The use of BPO has been proven effective for the precipitation-based synthesis of MIPs, producing polymer particles with uniform size and high polymerisation efficiency. In IIP research, it is very important to know the limit capacity of an IIP in adsorbing target ions. It is mentioned that an increase in the concentration of target ions can increase the adsorption capacity until it reaches a saturation point, where all imprint sites are filled [21]. Therefore, this study will discuss the adsorption capacity of IIP in binding target ions.

Research Methods

Tools and Materials

The equipment used in this study included spatulas, watch glasses, laboratory bottles, micropipettes, 100 mL measuring cups, 250 mL beakers, 10 mL volumetric pipettes, vortex mixers, hotplate stirrers, magnetic stirrers, ovens, thermometers, analytical balances, vacuum pumps, Whatman 42 filter paper, aluminium foil, Petri dishes, Erlenmeyer flasks with black lids, test tubes, filter paper, 100 mL glass bottles, and glass funnels. For the characterisation analysis of NIP and IIP, Fourier Transform Infrared (FTIR) and Scanning Electron Microscope (SEM) instruments were used. Meanwhile, characterisation and metal content testing were carried out using Atomic Absorption Spectrophotometry (AAS) instruments.

For NIP synthesis, the materials used include $Fe(NO_3)_3$, ethylenediaminetetraacetic acid (EDTA), benzoyl peroxide (BPO), methacrylic acid (MAA), ethylene glycol dimethacrylate (EGDMA), and solvents such as ethanol, acetonitrile, and aquabidest, with nitrogen gas used as a purge gas. In the extraction stage to obtain IIP, a 3M HNO_3 solution is used. Meanwhile, during the adsorption stage, a standard $Fe(NO_3)_3$ solution is used, along with a NaOH or HNO_3 solution.

Synthesis of Non-Imprinted Polymer

The preparation of NIP begins by mixing 0.0404 grams (0.1 mmol) of $Fe(NO_3)_3$ with 0.09306 grams (0.25 mmol) of EDTA, which acts as a ligand. After that, 60 mL of a 2:1 ethanol:acetonitrile mixture is added as a porogen. The mixture is then stirred using a magnetic stirrer for 30 minutes. The next step is to add 340 μL (0.4 mmol) of MAA as a functional monomer, 0.0484 grams (0.2 mmol) of BPO as an initiator, and 3.9644 grams (20 mmol) of EGDMA as a crosslinker. This mixture was then homogenised using a vortex mixer for 5 minutes. After homogenisation, the reaction vessel was sealed with aluminium foil and flushed with nitrogen gas for 5 minutes to create an inert atmosphere. The mixture was heated on a hot plate stirrer at 70°C until it formed a paste.

The resulting paste was then separated through vacuum filtration, and the residue was washed using ethanol and aquabidest to remove any unreacted material. Finally, the residue was dried in an oven at 60°C until its weight stabilised, yielding NIP powder as the final product of the synthesis process.

Non-Imprinted Polymer Extraction

Weigh 0.5 grams of NIP and place it in a sealed Erlenmeyer flask. Next, add 100 mL of 3 M HNO_3 solution, then stir with a magnetic stirrer at 800 rpm for 1 hour. After that, the residue is separated from the filtrate under vacuum. The residue is then rinsed with distilled water and dried in an oven at 60°C until completely dry, to obtain IIP.

IIP Adsorption At Varying Concentrations Of Standard Solution

The maximum adsorption capacity was determined to understand the adsorption kinetics of IIP. The test was carried out using varying concentrations of the $Fe(NO_3)_3$ standard solution: 5, 10, 15, 20, 25, 30, and 35 ppm. A total of 50 mL of solution at each concentration was mixed with 0.05 g of IIP and stirred at 800 rpm for 1 hour under neutral pH conditions (pH 7). The adsorption capacity of IIP at each concentration was then analysed using an AAS instrument.

Characterization of NIP-Fe and IIP-Fe

Fe–NIP and Fe–IIP polymers synthesised by precipitation polymerisation were characterised using various analytical techniques. Fourier Transform Infrared Spectroscopy (FTIR) was used to identify functional groups in the 4000–400 cm^{-1} range. Meanwhile, Atomic Absorption Spectroscopy (AAS) was used to determine the Fe(III) content in the samples. In addition, Scanning Electron Microscopy (SEM) was used to observe the surface morphology and elemental composition of the polymers, including the presence of Fe before and after the leaching process.

Results and Discussion

Adsorption of Fe IIP with Variations in Concentration

The adsorption results from varying concentrations of iron(II) ions are shown in Table 1 and Figure 1. The test results show that at the initial concentration, adsorption of iron(II) ions increased, but as the analyte concentration increased, the EDTA-modified IIP adsorbent reached equilibrium. The adsorption capacity obtained on the EDTA-modified IIP adsorbent was 19.06 mg/g. This adsorption capacity is relatively low, indicating that complexes between iron(II) ions and EDTA ligands did not form fully; only some iron(II) ions successfully formed template complexes. This condition is likely caused by an insufficient amount of mmol MAA to form optimal hydrogen interactions with the iron–EDTA complex, so that some of the complex dissolved in aqua and ethanol during the NIP washing process.

Table 1. Adsorption capatity

Co (mg/L)	Ce (mg/L)	Volume (V)	Wheigt (g)	Adsorption capatity (mg/g)
5.96	1.50	0.05	0.05	4.46
11.42	3.21	0.05	0.05	8.21
15.92	5.56	0.05	0.05	10.35
21.75	7.77	0.05	0.05	13.98
28.96	13.44	0.05	0.05	15.52
35.79	16.73	0.05	0.05	19.06
39.33	20.35	0.05	0.05	18,98

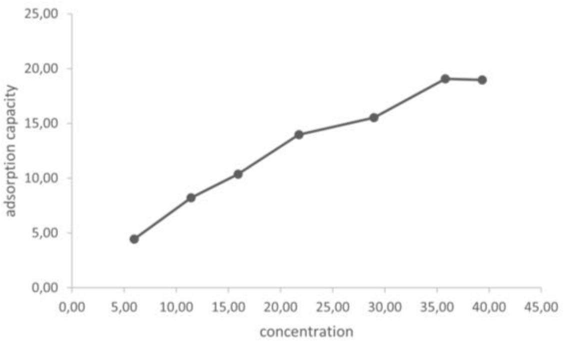

Figure 1. Large Adsorption Capacity at Each Concentration

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Characterization of NIP-Fe and IIP-Fe

Characterization FTIR

The FTIR spectra of the empty polymer, NIP, and IIP synthesised via the precipitation polymerisation method were obtained in the wavenumber range of 400–4000 cm⁻¹ (Figure 2). In the NIP spectrum, the characteristic peak at 468.55 cm⁻¹ is related to Fe–O bond vibrations, confirming the presence of Fe(III) ions in the polymer matrix. Conversely, this peak is absent in the IIP spectrum, indicating that Fe(III) ions have been successfully removed by HNO_3 washing, thereby forming specific recognition sites in the polymer structure.

The peak at 1728.33 cm⁻¹ is related to the strain vibration of the carbonyl group (C=O), while the peak at 1638.80 cm⁻¹ indicates the presence of a C=C double bond. The absorption band at 1160.96 cm⁻¹ corresponds to the C–O–C stretching vibration, characteristic of EGDMA cross-linking.Additionally, the weak band at 3463.10 cm⁻¹ indicates the stretching vibration of the –OH group from the carboxyl group, while the strong peak at 1728.22 cm⁻¹ further confirms the presence of the C=O group originating from EDTA.

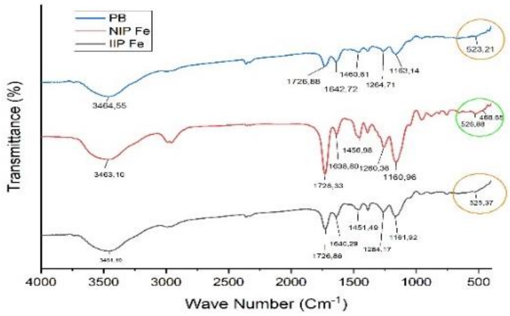


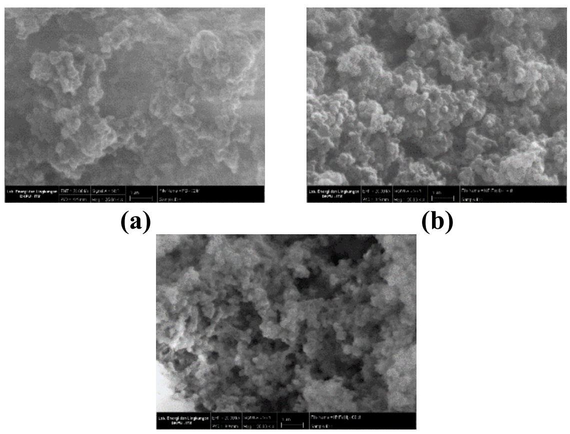
Figure 2. FTIR Spectra of BP, NIP, and IIP

Table 2. Wavelength of The Function Group

Function Group	Wavelengt reference	Wavelengt Result
Fe-O	468 [22]	468.55
–OH	2650-3538 [23]	3463.10
C=O	1651-1740 [24]	1728.33
C=C	1636[25]	1638.30
C–O–C	1156 [24]	1160.96

Characterization SEM

SEM characterisation at 25,000x magnification of the blank polymer, NIP, and IIP revealed the morphology of the materials. In Figure b, the surface morphology of the IIIP Fe(III) adsorbent showed the formation of a number of cavities and a clear porous structure, in which the particles appeared to be dispersed without agglomeration. This condition indicates that the synthesis process and the leaching or Fe(III) template-removal stage have proceeded smoothly, leading to the formation of pores on the adsorbent material's surface. The average particle size of the IIIP Fe(III) adsorbent was approximately 227 nm. Compared to NIP, which has a particle size of approximately 495.6 nm, this difference indicates a higher specific surface area for IIIP, potentially improving the adsorption process efficiency.



(a) **(b)** **(c)**

Figure 3. SEM micrographs of (a) Blank Polymer (BP), (b) Non-Imprinted Polymer (NIP), and (c) Ionic Imprinted Polymer (IIP) at a magnification of 25,000×

In the recovery percentage test, the result was 107,05%, as shown in Table 3. According to Sukmawati[26], this recovery percentage is acceptable because it meets the accuracy requirement, which is an average recovery percentage range of 90-110. The inaccuracy of the results may be due to several factors, including human error, sample contamination, and measurement and instrument calibration errors.

Table 3. Percentage Recovery

calculation concentration	concentration obtained	percentage recovery	average percentage recovery
0.151	0.155	103.08	107.05
0.108	0.119	110.29	

Conclusion

Ion-imprinted polymer (IIP) adsorbents containing EDTA-based ligands were successfully synthesised through a precipitation polymerisation method to obtain selective adsorption capabilities for Fe(III) ions. In the synthesis process, methacrylic acid (MAA) was used as a functional monomer, ethylene glycol dimethacrylate (EGDMA) as a crosslinking agent, and benzoyl peroxide (BPO) as a free radical initiator. The success of the IIP synthesis was confirmed by Fourier Transform Infrared Spectroscopy (FTIR), which revealed characteristic functional groups, including Fe–O bonds. Morphological and elemental composition analysis using SEM-EDX also showed significant differences between non-imprinted polymers (NIP) and ion-imprinted polymers (IIP). The structure of IIP displays more developed pores following the removal of Fe(III) ions, thereby creating specific recognition sites for these ions. In addition, the test results show that the maximum adsorption capacity of IIP for Fe(III) ions is 19.06 mg/g, confirming its high selectivity.

Author's Contribution

A.T. Wibowo: Conceptualization, methodology, writing the original draft. M. M. S. Basukiwardojo: Validation, Conceptualization

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