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Synthesis of Fe(III)-IIPs (Ion Imprinted Polymers): Comparing Different Concentrations of HCl and HNO_3 Solutions in the Fe(III) Polymer Extraction Process for Obtaining the Largest Cavities in Fe(III)-IIPs

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Abstract
This study was conducted to synthesize Fe (III)-IIPs by free radical polymerization using the cooling-heating method. Cooling process at -5°C for 1 hour, as well as heating at 75°C, 80°C, and 85°C maintained for 3 hours, 2 hours and 1 hour, respectively. The Fe (III)-IIPs synthesis process involved $Fe(NO_3)_3$ with an average diameter of 18.23 nm, methacrylic acid (MAA), ethylene glycol dimethacrylate (EGDMA), benzoyl peroxide (BPO) and ethanol, each of which plays a role as an analyte, functional monomer, cross-linker, initiator, and porogen. The result of the polymerization process was a polymer containing ions namely Fe(III) polymer. The ions need to be removed by the extraction process to produce Fe(III)-IIPs, which act as absorbents. Furthermore, the extraction process is very influential in the process of losing ions and the formation of cavities or templates in the polymer body. The number of cavities formed tends to affect the ability of Fe(III)-IIPs to identify the target ion which has similar physical and chemical properties to the shape of the Fe(III)-IIPs cavity. The extraction process was carried out on Fe(III) polymer samples using HCl and HNO_3 solutions with varying concentrations of 3 M and 6 M, respectively. The transmission percentage of FTIR analysis showed that for samples of Fe(III)-IIPs HCl 3 M and 6 M were 94.258% and 95.666%, while for Fe(III)-IIPs HNO_3 3 M and 6 M were 92.735%, respectively. The largest percentage was shown in the 6 M HCl IIPs sample, which indicated that there were several ions lost from the polymer body after the extraction process. This is also reinforced by the results of the SEM analysis processed with Matlab, which showed 498 cavities with a distribution of voids on a scale of <100 nm, totaling 470.

Keywords
Imprinted Polymer, Fe(III), Cooling-Heating, Extraction, HCl, HNO_3, Cavity

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1. INTRODUCTION

The earth's surface is covered by 29% land and 71% water (Domingo, 2012), which includes 97.5% (approximately 1.41 billion km^3) is saltwater and only 2.5% is fresh water, respectively, consisting of polar ice, groundwater, and water vapor (Petersen et al., 2016). However, only 0.014% can be directly utilized, while the rest need physical and chemical processes to become suitable for use (Trisnaini et al., 2018). The river is one of the water sources used by humans to fulfill all their life activities. However, the water quality has become deteriorated and polluted by chemical waste or heavy metals. One of the heavy metals that is often found in river water is Fe(III) (Ayu and Taufik, 2021; Grząbka-Zasadzińska et al., 2021). In general, iron is divided into two forms, namely solid Fe^{2+} and water-soluble Fe^{3+}. Elemental iron dissolved in water has a high redox potential (Ara et al., 2018; Corral-Bobadilla et al., 2021).

The level of iron dissolved will cause a change in the color of the water to yellow and even reddish, with smells of rust and can form an oil-like layer. According to the Regulation of the Minister of Health (Permenkes) No. 492 of 2010, the limit of iron dissolved must not exceed 0.3 mg/L in water for use or consumption by humans. When the iron content in the water exceeds 1 mg/L, it will have a direct impact on a person's physique, causing irritation to the eyes and skin, while levels up to 10 mg/L directly change the color and smell of water (Ayu and Taufik, 2021). High levels of Fe(III) dissolved in water when accidentally consumed and continuously accumulated in the body, leads to various health problems ranging from loss of consciousness, respiratory disorders, as well as damage to several organs and even death (Ara et al., 2018; Kusumkar

Table 1. Use of Fe(III)-IIPs in Water Treatment and Iron Trace Analysis

Utility	References
Absorbent extraction of iron ions from food and water samples	(Roushani et al., 2016)
Selective solid phase extraction from mixed solutions of iron ions and many other metal ions	(Ara et al., 2018)
Determination of Fe(II)/Fe(III) in wine samples	(Mitreva et al., 2017)
Removes traces of Fe(III) ions from solutions containing Cr(III)	(Zhu et al., 2019)
Removes Fe^{3+} from highly concentrated alkaline chromium sulfate solutions	(Zhu et al., 2020)
Absorbent extraction of iron ions from food and water samples	(Roushani et al., 2016)

et al., 2021).

Considering the problems mentioned above, it is important to have a method of separating Fe^{3+} ions. There are various methods commonly used to remove Fe(III) dissolved in water, including extraction of neutral solvents (Mishra et al., 2011; Spathariotis et al., 2020; Tavakoli and Dreisinger, 2014) such as ethers, ketones, amines, and tributylphosphates, as well as cationic reagents, namely quaternary ammonium ions. However, these methods are not adequately effective. Apart from being too expensive, the use of mineral acid is also high, the performance is slow and the level of selectivity is low, causing the need for other suitable methods. Solid phase selection methods such as Ion Imprinted Polymers (IIPs) provide several advantages, such as lower costs, more effectiveness, faster performance and high selectivity (Ara et al., 2018; Cao et al., 2021). Through the uniqueness and advantages of these IIPs, many researchers have developed a variety of methods and processes for removing or extracting Fe(III) metal ions in aquatic sites or the environment as shown in Table 1.

The determining factors for the success of Fe(III)-IIPs include the polymerization process used. In this study, the cooling-heating method was selected because it is more effective and does not require liquid nitrogen, which is expensive to remove oxygen levels. In addition, this method only takes 7 hours, while others require up to 24 hours (Ara et al., 2018; Mitreva et al., 2017; Roushani et al., 2016; Zhu et al., 2019). Effective cooling-heating methods have previously been successfully used in the synthesis of molecularly imprinted polymers as atrazine (Royani et al., 2014) and caffeine recognition sites (Royani et al., 2021). Aside from polymerization, the extraction process plays a significant role in the final result of IIPs cavity formation. The process is carried out by heating at a temperature of 60°C for 18 hours repeatedly to avoid losing more ions (Ara et al., 2018; Mitreva et al., 2017; Zhu et al., 2019). The solution with a certain concentration is the determining factor to extract ions from the polymer body. HCl was selected for the Fe(III) extraction because it causes splits between Fe(III) ions and the polymer network. Meanwhile, (HNO_3) was selected because its high oxidizing ability significantly increases the dissolution of Fe(III) ions.

Some of the reference sources adopted did not discuss in depth the concentration and acid solutions used in the metal ion extraction process. Therefore, this study aims to examine the effect of the acid solution concentration on the Fe(III) ion extraction process to determine the number of cavities formed in Fe(III)-IIPs. It is hoped that more cavities will be produced during the Fe(III) polymer extraction process, the final result of the IIPs Fe(III) material can be used as a reference sensor material that can overcome water pollution in the future.

2. EXPERIMENTAL SECTION

2.1 Materials

$Fe(NO_3)_3$ as analyte, ethanol as porogen solvent, methacrylic acid as functional monomer, ethylene glycol dhymethacrylate as crosslinker, benzoil peroxide as initiator, deionized water as thinners and washers, HCl and HNO_3 as extraction solution.

2.2 Methods

The synthesis of Fe(III)-IIPs was processed through 3 stages including the first stage of Fe(III) polymer preparation using the cooling heating method. The second was the extraction of Fe^{3+} ions in the polymer body, while the third was the characterization of Fe(III)-IIPs samples by FTIR and SEM. The stages of Fe(III)-IIPs synthesis are illustrated in Figure 1.

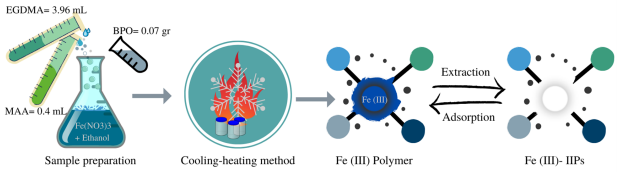

Figure 1. Illustration of Fe (III)-IIPs Synthesis

2.2.1 Fe(III) Polymer Preparation Using Cooling Heating Method

In this procedure, Fe(III) polymer was synthesized by free radical polymerization using the cooling-heating method. A total of 0.404 g of $Fe(NO_3)_3$ was dissolved in 40 mL of ethanol, then added with 0.4 mL of methacrylic acid (MAA), 3.96 mL of ethylene glycol methacrylate (EGDMA) and 0.07 g benzoyl peroxide (BPO). The mixture was homogenized with

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a magnetic stirrer on a hot plate at 25°C for 90 minutes. The homogeneous solution was transferred to a vial with a dose of about 1.5 mL of solution added to remove the oxygen content trapped, while the cooling process was carried out in a freezer at a temperature of -5°C for 60 minutes. This was followed by a heating stage with a furnace at a temperature of 75°C, 80°C, and 85°C maintained for 3 hours, 2 hours and 1 hour, respectively (Royani et al., 2020). A similar preparation was carried out for the synthesis of NIP (Non Imprinted Polymers) without the addition of $Fe(NO_3)_3$ analyte.

2.2.2 Extraction Process of Fe^{3+} Ions on Polymer Body

The process removal of Fe(III) in the polymer body is basically carried out in a way that is commonly done by previous studies. The Fe(III) polymer was in the form of a clear brown acrylic solid which was ground with a mortal cup until it became a powder. The obtained powder was then washed with ethanol and deionized water for five repetitions to remove unreacted compounds during the polymerization process. Afterward, the extraction was carried out using two types of acid solutions namely HCl and HNO_3 which will be compared with variations in the concentration of 3 M and 6 M in each sample of Fe(III) polymer powder. This extraction process was carried out for 18 hours by heating at 60°C while stirring with a magnetic stirrer and repeated 8 times until the powder sample with brown color turns to whitish-orange. The final step is to obtain a sample of Fe(III)-IIPs which is then washed with deionized water, repeated 8 times for 24 hours until a neutral state was achieved. This was followed by drying in the furnace at a temperature of 60°C for 60 minutes.

2.2.3 FTIR and SEM Characterization

Several samples obtained from the synthesis were characterized using FTIR characterization to determine the functional groups, compounds, chemical bonds, as well as percent transmittance and absorbance. Meanwhile, the SEM characterization was carried out to determine the morphological structure (Darmawan et al., 2020), as well as the size and number of cavities formed in the Fe(III) IIPs sample. Table 2 shows a list of samples tested in a series of characterization stages.

3. RESULTS AND DISCUSSION

3.1 FTIR Characterization

Through the stages and cooling methods that have been carried out, the sample variations are obtained as shown in Figure 2. Furthermore, each of these samples was analyzed by FTIR. FTIR analysis for various samples was carried out in the wave number range of 3000-500 cm^{-1} as shown in Figure 3. the acid solution with the greatest ability to remove Fe^{3+} ions more effectively in polymer samples for the acquisition of Fe(III)-IIPs produced the highest number of voids. Figures 3(a) and (b) show that there is a match and uniformity between the range of wave numbers and functional groups of each sample. The functional groups C=O (1750-1705 cm^{-1}) and C-O (1300-1000 cm^{-1}) derived from the carboxylic acid group imply that there were compounds containing EGDMA and BPO. The C-H functional group (1484-1445 cm^{-1}) from the methlene bend group indicates the presence of a Methacrylic Acid (MAA) compound (Koriyanti et al., 2020; Royani et al., 2020). Furthermore, the C-I functional group (600-500 cm^{-1}) of the stretch aliphatic iodo compounds suggests the presence of metal ions (Kousalya et al., 2010; Nandiyanto et al., 2019). This is supported by the presence of a functional group NO_3^- (1386-1350 cm^{-1}) from the common inorganic ions group which indicates the presence of Fe^{3+} ions (Huang et al., 2010).

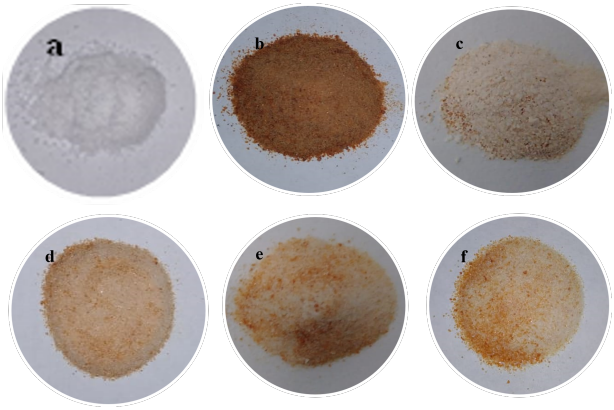

Figure 2. Synthesis Results (a) NIP, (b) Fe(III), (c) Fe(III)-IIPs HCl 3 M, (d) Fe(III)-IIPs HCl 6 M, (e) Fe(III)-IIPs HNO_3 3 M, (f) Fe(III)-IIPs HNO_3 6 M

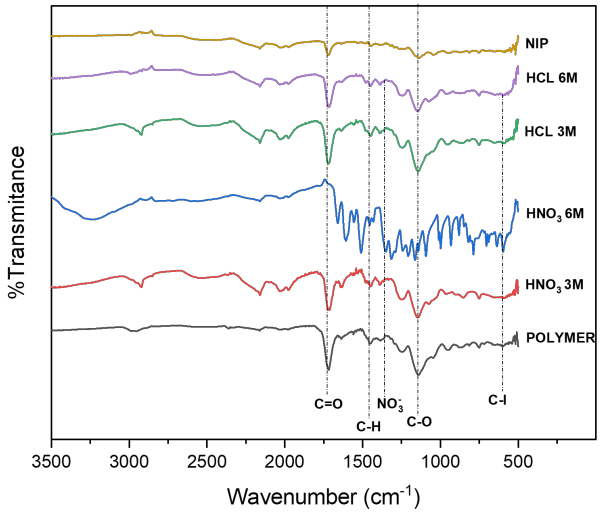

Figure 3. Spectrum FTIR NIP, Fe(III) polymers, Fe(III)-IIPs HCl 3 M, Fe(III)-IIPs HNO_3 3 M and Fe(III)-IIPs HCl 6 M, Fe(III)-IIPs HNO_3 6 M

The transmittance percentage values listed for the NO_3^- functional group which suggest the presence of Fe^{3+} ions from the six existing samples is further supported by the Fe(III) polymer

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Table 2. Sample Data on FTIR and SEM Characterization Tests

Sample	Compositions	Characterization
NIP	Ethanol, MAA, EGDMA, BPO	FTIR
Fe(III) polymer	$Fe(NO_3)_3$, Ethanol, MAA, EGDMA, BPO	FTIR
Fe(III)-IIPs HCl 3 M	$Fe(NO_3)_3$, Ethanol, MAA, EGDMA, BPO, Deionized Water, HCl 3 M	FTIR
Fe(III)-IIPs HCl 6 M	$Fe(NO_3)_3$, Ethanol, MAA, EGDMA, BPO, Deionized water, HCl 6 M	FTIR and SEM
Fe(III)-IIPs HNO_3 3 M	$Fe(NO_3)_3$, Ethanol, MAA, EGDMA, BPO, Deionized water, HNO_3 3 M	FTIR
Fe(III)-IIPs HNO_3 6 M	$Fe(NO_3)_3$, Ethanol, MAA, EGDMA, BPO, Deionized water, HNO_3 6 M	FTIR and SEM

sample with a value of 88.349%, while 11.651% represents the IR frequency absorbed by the sample or referred to as the absorbance value. From these data, the Fe(III) polymer has the smallest transmittance percentage of the four samples. This means that the absorbance value of the sample is large, presumably due to the strong Fe^{3+} ionic bond, hence, the highest ion content was found in the Fe(III) polymer sample. In contrast, for the NIP sample synthesized without $Fe(NO_3)_3$ analyte, the absence of the NO_3^- functional group was confirmed in the wave number range of 1386-1350cm^{-1}.

The functional group NO_3^- in the four samples of Fe(III)-IIPs extracted using HCl and HNO_3 solutions with concentrations of 3 M and 6 M labeled as Fe(III)-IIPs HCl 3 M, Fe(III)-IIPs HCl 6 M, Fe(III)-IIPs HNO_3 3 M, and Fe(III)-IIPs HNO_3 6 M had a percentage of 94.258%, 95,666%, 92.735%, and 92.735%, respectively. The largest percentage transmittance value was found in the Fe(III)-IIPs HCl 6 M sample. Consequently, the absorbance value in this sample is smaller than the other four, at 4.334%, the small value is related to the unbound Fe^{3+} ion which implies that they have been released from the polymer body due to the extraction process. Based on the available data for the Fe(III)-IIPs HNO_3 3 M and Fe(III)-IIPs HNO_3 6 M samples, there was no difference even though the concentration of the solvent used varied. The HNO_3 solution used in the extraction process can still remove Fe^{3+} ions from the polymer body with reference to the percentage transmittance value suggesting that Fe^{3+} ions must be larger than the Fe(III) polymer sample. Therefore, the 6 M HCl solution was more effective than HCl 3 M or HNO_3 3 M and HNO_3 6 M in removing Fe^{3+} ions. The use of hydrochloric acid (HCl) causes a split in the interaction between Fe^{3+} ions with polymer particles while the increase in concentration is proportional to the eluting rate of the Fe(III) polymer. The final results obtained from the FTIR analysis indirectly illustrate that the most voids are in the Fe(III)-IIPs HCl 6 M sample, this was also reviewed further in the SEM analysis.

3.2 Cavity Analysis on Fe(III)-IIPs HCl 6 M and Fe(III)-IIPs HNO_3 6 M Samples Based on SEM Imaging

The SEM imaging results of the two Fe(III)-IIPs samples were compared to determine the morphological structure, cavity size, and the number of cavities produced from the extraction with two different solvents HCl and HNO_3 at the same concentration of 6 M. The extraction process was carried out manually with repeated heating to optimize the loss of Fe^{3+} ions from the polymer body (Ghassa et al., 2020; Zhu et al., 2019). This is also evident from the SEM imaging results in Figure 4, showing a distribution of cavities formed in the Fe(III)-IIPs sample. SEM data processing was supported with the Matlab 2010 application programmed with Poredize software. The output obtained represents the size of the cavity diameter in nanometer scale.

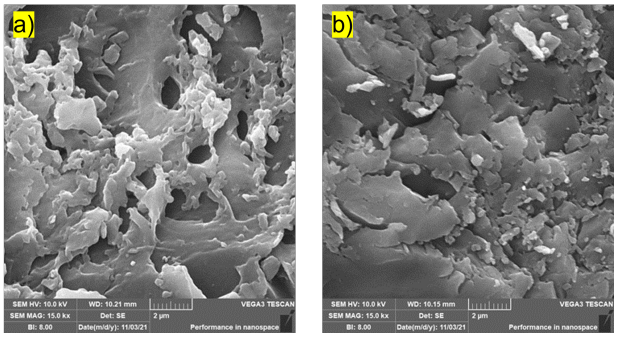

Figure 4. SEM Imaging Results 15.0 kx a) Fe (III)-IIPs HCl 6 M and b) Fe(III)-IIPs HNO_3 6 M

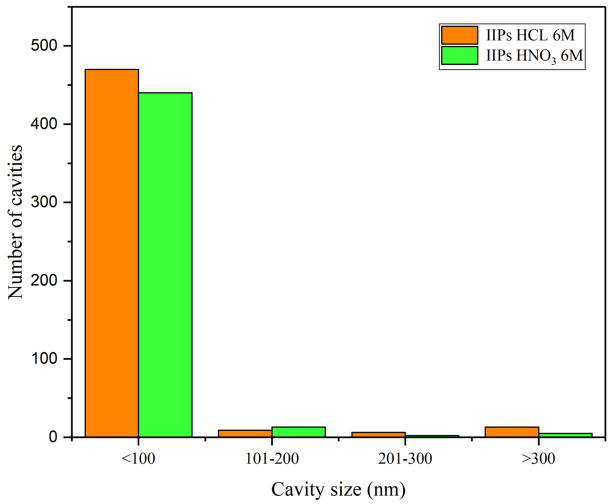

Figure 5. Cavity distribution of Fe(III)-IIPs HCl 6 M and Fe(III)-IIPs HNO_3 6 M

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Based on the output data results from Matlab processing in Figure 5, the distribution of voids in Fe(III)-IIPs HCl 6 M and Fe(III)-IIPs HNO_3 6 M samples was obtained, each with a total void of 498 and 460, respectively, with the highest distribution on a scale of <100 nm for Fe(III)-IIPs HCl 6 M at 470, while Fe(III)-IIPs HNO_3 6 M was 440. The FTIR and SEM analysis results showed that HCl solution with a concentration of 6 M has the ability to remove Fe^{3+} ions more effectively than HNO_3 at 6 M.

4. CONCLUSION

The extraction process carried out with 6 M HCl solution formed the largest distribution of voids in the Fe(III) sample, amounting to 498 in total, and on a scale of <100 nm reaching 470. Based on the results, the use of hydrochloric acid (HCl) with increasing concentration is more effective than HNO_3 6 M in the removal of Fe^{3+} ions in Fe(III) polymer samples.

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