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The Effectiveness of Bulk Polymerization and Precipitation Polymerization on the Adsorption Capacity of Pb(II) Metal Ions Using Ionic Imprinted Polymer (IIP)

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Abstract. Lead (II) metal is a heavy metal with a high level of toxicity that easily decomposes, potentially causing long-term adverse effects on the environment and human health. Therefore, it is important to develop an effective separation method to detect lead (II) metal ions in water, in order to reduce its negative impact on humans, organisms, and aquatic biota. In this study, Lead Ion Imprinted Polymer (Pb-IIP) was developed to detect Pb(II) metal ions in water sample. The commonly used polymerization methods are bulk polymerization and precipitation polymerization. Bulk polymerization and precipitation polymerization were tested for their efficiency in creating IIP capable of adsorbing Pb(II) ions. Therefore, this study aims to determine the effectiveness of these methods in enhancing the adsorption ability Pb(II) metal ions in water. The IIP was successfully synthesized using $Pb(NO_3)_2$ as the template, methacrylic acid (MAA) as the functional monomer, ethyleneglycoldimethacrylate (EGDMA) as the crosslinker, benzoyl peroxide (BPO) as the initiator, and ethylenediaminetetraacetic acid (EDTA) as the chelating ligand. The results showed that the precipitation polymerization method produced an adsorbent with a higher adsorption capacity, smaller particle size, and better quality, although both methods were selective for Pb(II) metal ions. The adsorption capacity achieved using precipitation polymerization was (56,23 mg/g), while bulk polymerization achieved (46,24 mg/g). This method has great potential to be applied in treating water contaminated with heavy metals, offering an efficient solution to reduce environmental pollution and protect human health.

Introduction

Lead (Pb) is a heavy metal that is widely known and easily recognized by the general public due to its extensive use in non-food industries, such as additives in gasoline, cables, batteries, and mining (Charkiewicz and Backstrand, 2020). Lead is classifed as a heavy metal due to its high toxicity and prevalence in industrial waste and is one of the most common causes of poisoning in living organism, affecting the immune system, cardiovascular, respiratory, reproductive, digestive, and nervous systems. Lead also contaminates the environment even at trace concentrations (Lazar et al., 2023).

Lead(II) ions have high toxicity, decompose easily, and accumulate, leading to long-term adverse effects on the environment and human health (Fahruddin, 2020). Lead and its products are commonly disposed of in water bodies. Lead waste discharged into water can contaminate groundwater or surface water and affect living organisms around the river (Xie et al., 2020). Lead(II) ions can be effectively targeted in river water to mitigate harmful effects on humans, organisms, and aquatic life in rivers (Xia et al., 2023). Various methods and strategies are currently used to capture lead(II) ions in water, including membrane separation, ion exchange, electrochemical remediation, and

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adsorption. Ion exchange and electrochemical methods have drawbacks such as high costs and significant disadvantages. Therefore, adsorption is a simple method, easy to operate, cost-effective, highly efficient, and shows greater potential for development (Roushani, 2018).

Simple adsorption methods, such as activated carbon and zeolite, commonly used to adsorb heavy metals, have limitations, such as low adsorption efficiency and poor selectivity (Chaipuang et al., 2021) . Therefore, it is necessary to develop adsorbents with high selectivity, high adsorption capacity, environmental friendliness, and reusability (Wang et al., 2024). To address this issue, Ionic Imprinted Polymer (IIP) is used as an adsorbent that shows a higher affinity for target ions compared to other ions with similar structures due to its specific ion recognition sites. Additionally, IIP is also cost-effective and reusable (Çıtlakoğlu and Yolcu, 2023).

In the polymerization process, several methods are commonly used in synthesis, including bulk polymerization and precipitation polymerization (Pratiwi et al., 2018). Both methods have their advantages and disadvantages. Bulk polymerization is the most frequently used method in the production of IIP due to its simplicity and ease of operation, requiring no complex equipment, simple operation, good adsorption capability, and selectivity toward target ions (Kusumkar et al., 2021). However, the resulting material has irregular particle sizes, requiring grinding and sieving to achieve the desired size (Deng et al., 2019). In contrast, precipitation polymerization can be classified as homogeneous polymerization, where all components are dissolved in a solvent, and polymerization begins in a homogeneous solution (Wirawan et al., 2019). The resulting adsorbent is in the form of particles within the nano, submicron, and micron size ranges, making metal ion removal easy with this method. However, precipitation polymerization requires a large amount of solvent and a longer polymerization time compared to bulk polymerization, making it costly and time-inefficient (Hasanah et al., 2019).

Previous research on the synthesis of atenolol-imprinted polymers using the precipitation polymerization method yielded better affinity and imprinting factor values compared to bulk polymerization (Hasanah et al., 2019). On the other hand, Pratiwi (2019) found that adsorption using the bulk polymerization method was more selective compared to precipitation polymerization in the application of Molecularly Imprinted Polymer (MIP) for the selective extraction of atenolol in blood samples. Therefore, the novelty of this study is to compare the bulk polymerization and precipitation polymerization methods regarding the selective adsorption capability of lead(II) ions using IIP in river water. Therefore, the aim of this research is to compare the bulk polymerization and precipitation polymerization methods in terms of their adsorption capabilities for lead(II) ions using IIP.

Experimental

Material and Methods

The equipment used in this experiment includes a spatula, reagent glass, beaker glass, measuring cylinder, volumetric flask, test tubes, dropper pipettes, thermometer, glass funnel, measuring pipette, volumetric pipette, reagent bottles, magnetic stirrer, watch glass, vacuum membrane filtration apparatus, micropipettes, analytical balance (Ohaus), AAS (Shimadzu AA-7000), FTIR (PerkinElmer) and SEM (ZEISS Evo 10).

The materials used in this study are $Pb(NO_3)_2$ (Merck), Benzoyl Peroxide, Methacrylic Acid (MAA) (Sigma-Aldrich), Ethylenediaminetetraacetic Acid (Merck), Ethylene Glycol Dimethacrylate (Sigma-Aldrich), fuming HNO_3, HCl 37% (Merck), Ethanol (Merck), Acetonitrile (Merck), NaOH, Aquades, Aquabidest and Aquademin.

Procedures

Synthesis of IIP-Pb using Bulk Polymerization

The synthesis of bulk polymerization was carried out by mixing 0.06624 g $Pb(NO_3)_2$ (0.2 mmol) and 0.04653 g (0.125 mmol) EDTA in 15 mL of porogen ethanol : acetonitrile (2:1). The solution was stirred using a magnetic stirrer for 30 minutes. Finally, 680 µL (8 mmol) methacrylic acid (monomer), 7.9288 g (40 mmol) ethylene glycol dimethacrylate (crosslinker), and 0.016 g (0.06 mmol) benzoyl peroxide (initiator) were added. The beaker was covered with aluminum foil and purged with nitrogen gas for 5 min. Bulk polymerization was carried out by heating in a water bath at 70 °C until the mixture became a paste, then it was oven-dried at 60 °C until a constant weight was achieved. This adsorbent called as NIP. This adsorbent called as NIP (Non-Ionic Imprinted Polymer).

Synthesis of IIP-Pb using Precipitation Polymerization

The synthesis of pecipitation polymerization was carried out by mixing 0.0331 g $Pb(NO_3)_2$ (0.1 mmol) and 0.09306 g (0.25 mmol) EDTA in 60 mL of porogen ethanol : acetonitrile (2:1). The solution was then stirred using a magnetic stirrer for 30 minutes. Finally, 340 µL (4 mmol) methacrylic acid (monomer), 3.9644 g (20 mmol) ethylene glycol dimethacrylate (crosslinker), and 0.0484 g (0.2 mmol) benzoyl peroxide (initiator) were added. The beaker was covered with aluminum foil and purged with nitrogen gas for 5 min. Precipitation polymerization was

carried out by heating in a water bath at 70 °C until the mixture became a paste, then it was oven-dried at 60 °C until a constant weight was achieved. This adsorbent called as NIP (Non-Ionic Imprinted Polymer).

Determination of Extaction Percentage

The extraction process was carried out using 100 mL of 1M HNO_3 with the aim of removing template metal ions that had bound to specific polymer binding sites during the synthesis process to improve reproducibility. The adsorbent was washed with distilled water and dried at 60°C and then the adsorbent called as IIP. The filtrate from the extraction was tested using AAS to determine the concentration of dissolved or extracted metal. The test results were then applied to Equation 1.

$$\text{Extraction (\%)} = \frac{(Ci\text{-}Cf)}{Ci} \text{ x } 100\% \qquad (1)$$

where Ci (mg/L) is the initial amount of template ion, Cf (mg/L) is amount of residual template ions.

Determination of Optimum pH

IIP works specifically at a certain pH, so it is necessary to investigate the optimum pH that is effective for the template. The experiment was conducted using IIP precipitation polymerization method by adding 0.05 grams of IIP to a 50 ppm $Pb(NO_3)_2$ standard solution in a reagen bottle and adjusting the pH to 5, 6, 7, 8 using 0,1 M HNO_3 and 0.1 M NaOH. The experiment was performed two repetitons.

Determination of Adsorption Capacity

The dried IIP was then weighed to 0.05 grams and placed into a reagent bottle. After that, 50 mL of a 50 ppm Pb standard solution, optimized at pH 7, was added. The solution was stirred using a magnetic stirrer for 3 hours at a speed of 1000 rpm. After 3 hours, the solution was filtered, and the filtrate was tested using AAS to determine the adsorption capacity of the Pb metal. The test results were then applied to the adsorption capacity formula in Equation 2.

$$Qe = \frac{(Co\text{-}Ce)V}{m} \text{ x } 100\% \qquad (2)$$

where Qe (mg/g) is adsorption capacity, Co (mg/L) is initial concentration of adsorbat, Cf (mg/L) is final concentration of the adsorbate after adsorption, V (L) is volume of the soluton and m (g) is mass of the adsorbat.

Characterization analysis of Pb-IIP and Pb-NIP

The characterization of IIP-Pb and NIP-Pb compounds using bulk polymerization and precipitation polymerization methods was performed with FTIR (Fourier Transform Infrared) instruments at a wavelength range of 500-4000 cm^{-1}, morphological analysis using SEM (Scanning Electron Microscopy) at a magnification of 25,000x, and metal content analysis using AAS (Atomic Absorption Spectroscopy).

Result and Discussion

Characterization of IIP-Pb using FTIR

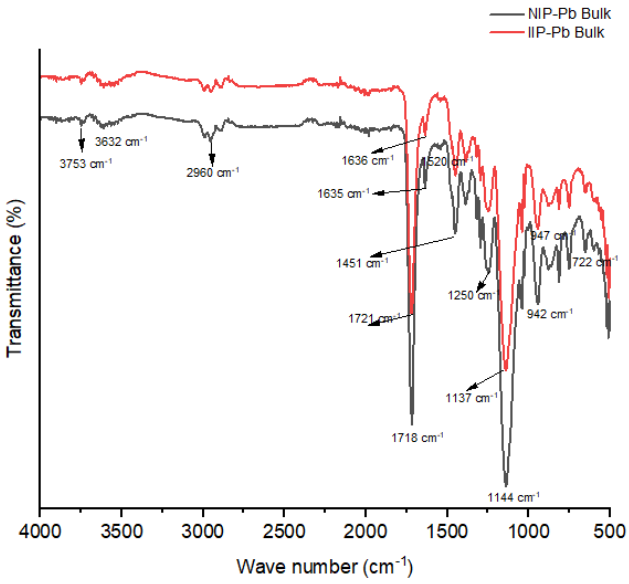

Figure 1. FTIR spectra from IIP-Pb and NIP-Pb of bulk polymerization.

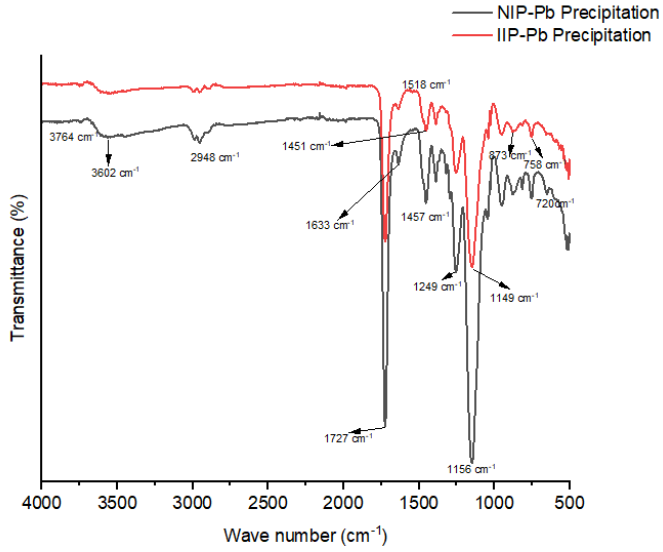

Figure 2. FTIR spectra from IIP-Pb and NIP-Pb of precipitation polymerization.

The characterization of IIP was conducted through chemical characterization using an FTIR instrument, which aims to identify the functional groups in the IIP adsorbent. The FTIR analysis is carried out in the wavelenght range of 500-4000 cm^{-1}. The FTIR spectrum of IIP by bulk and precipitation polymerization are presented in Figures 1 and 2.

Based on the FTIR results of Pb-IIP and Pb-NIP synthesis using bulk polymerization and precipitation polymerization methods, there were no specific differences observed, and the spectra produced were generally identical. As shown in Figure 1, the peaks at wavelengths of 3764 cm^{-1} and 3632 cm^{-1}, as well as in Figure 2 at 3764 cm^{-1} and 3602 cm^{-1} are stretching vibration characteristics from the O-H groups then 2960 cm^{-1} and 1710 cm^{-1}, as well as in Figure 2 at wavelengths of 2940 cm-1 and 1727 cm^{-1}, are characteristic of the stretching vibrations of the C-H and C-O groups from EGDMA. These results are consistent with G. Supriyanto (2019), who found that the peaks at 2954 cm-1 and 1722 cm^{-1} correspond to the stretching vibrations of the C-H and C-O groups from EGDMA.

The peaks at 1250 cm^{-1} and 1144 cm^{-1} in bulk polymerization and at 1249 cm^{-1} and 1156 cm^{-1} in precipitation polymerization are characteristic of the C-O-C stretching vibrations from EGDMA. The vibration frequency at 1627 cm^{-1} indicates the characteristic of the carbonyl group in EDTA (El Ouardi et al., 2021). The peak at 2991 cm^{-1} represents the absorption of the -O-H- vibration, which originates from the carboxylic acid group, while the peak at 1627 cm^{-1} corresponds to the C=C vibration absorption from the vinyl group in the MAA compound (Liu et al., 2019). The hydroxyl group (-OH) can serve as a binding site for Pb ions. When the oxygen atom in the C-O group coordinates with Pb ions, it forms a stable complex. This coordination increases the material's affinity for Pb ions and leads to electrostatic interactions, contributing to an increase in adsorption capacity (Pan et al., 2024).

The carbonyl group (C=O) can act as a ligand, where the oxygen atom donates lone pairs of electrons to the Pb^{2+} ion. This interaction is crucial in ion exchange and chelation processes, which enhance the adsorption of Pb ions. As Pb^{2+} binds with carbonyl groups, the adsorption capacity increases due to the formation of stable complexes (Z. Li et al., 2019). The wavelength at 1759 cm^{-1} is a characteristic absorption band of the carbonyl C=O group, and the wavelength at 1206 cm^{-1} is a characteristic absorption band of the peroxide O-O bond from BPO (Huang et al., 2024).

The success of IIP synthesis is evidenced by the absence of an O-O bond at a wavelength of 1206 cm^{-1}, which corresponds to the wavelength of the BPO compound, indicating that initiation has occurred. Additionally, the appearance of peaks at 722 cm^{-1} in bulk polymerized NIP and 720 cm^{-1} in precipitated NIP indicates the successful synthesis of IIP, corresponding to the Pb-O bond (Abdullah et al., 2019).

Characterization of IIP-Pb using SEM

The SEM-EDX instrument was used for examining the microstructure morphology and chemical composition characterization of the IIP adsorbent. In this study, surface morphology was analyzed using a magnification of 25.000x to obtain information about the surface morphology of the adsorbent. The samples analyzed were IIP adsorbents synthesized using different methods, namely bulk polymerization shown in 3(a) and 3(b) in Figure 3 then precipitation polymerization shown in 4(a) and 4(b) in Figure 4.

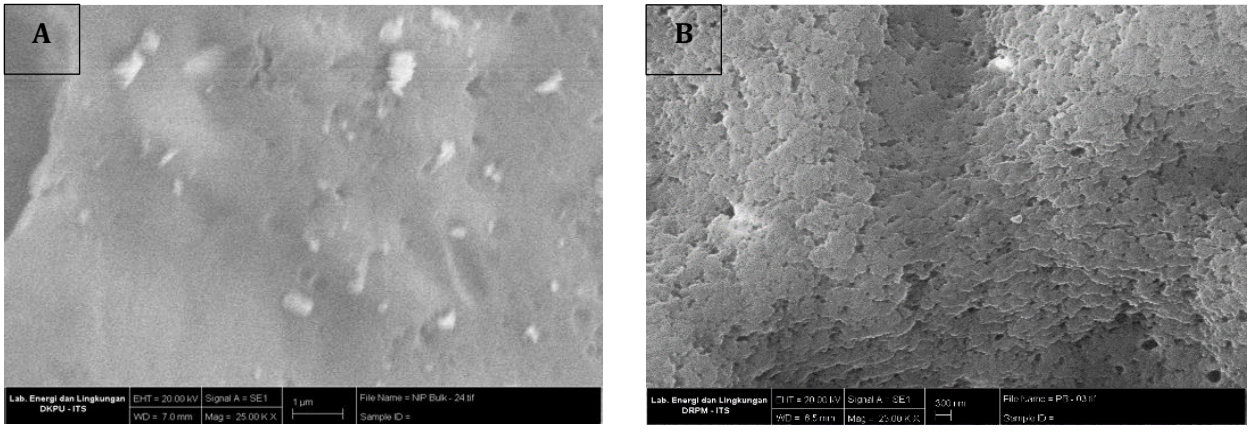

Figure 3. SEM of (a) NIP (b) IIP bulk polymerization method at 25.000x magnification.

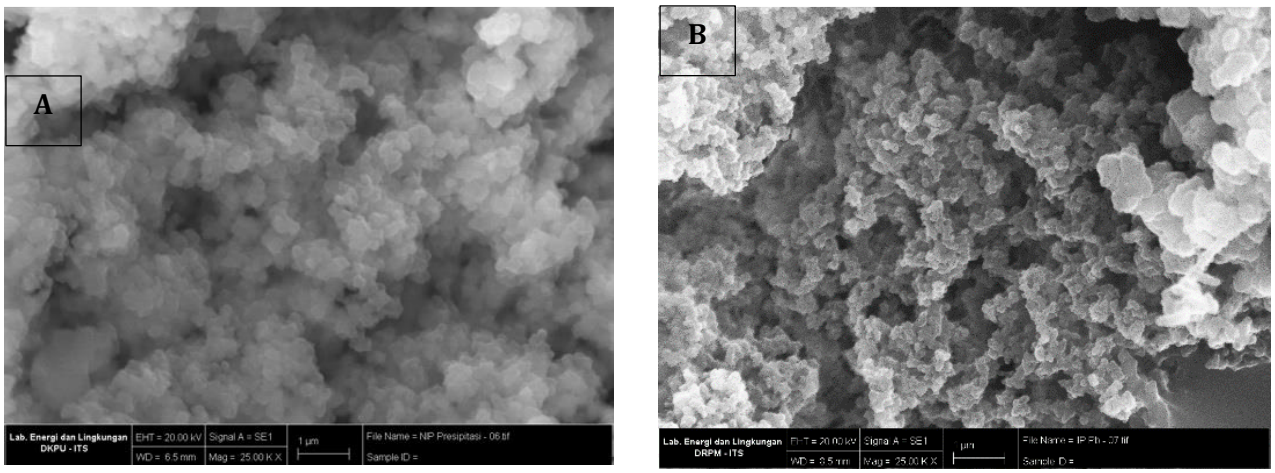

Figure 4. SEM of (a) NIP (b) IIP precipitation polymerization method at 25.000x magnification.

Based on the SEM analysis results, the morphology of the polymer synthesized using the bulk polymerization method showed that the material tended to be non-porous in both NIP and IIP compared to the polymer morphology of the precipitation polymerization method. Bulk polymerized NIP resulted in a generally non-porous morphology, which could lead to low template ion binding, thereby reducing the metal ion adsorption capacity. The morphological structure of bulk polymerized IIP showed slightly more cavities than bulk polymerized NIP, indicating that the extraction process was successful and that bulk polymerized IIP had better adsorption capabilities for target ions compared to bulk polymerized NIP. This is because porosity is one of the important factors that affects adsorption performance and is directly related to the active surface area and accessibility of target ions to the active sites of the adsorbent.

Basically, the higher the porosity of an adsorbent, the greater the number of pores that are open to adsorb target molecules or ions. Adsorbents with greater porosity have a higher surface area, so they can accommodate more molecules or ions in their pore spaces (Raji et al., 2023).

On the other hand, precipitation polymerized NIP exhibited porous material with many cavities in the form of imperfect and smooth round granules with less homogeneous particle distribution. Meanwhile, precipitation polymerized IIP showed a round, non-uniform surface with larger pores compared to precipitation polymerized NIP. This suggests that the template compound would be more easily adsorbed by the IIP material compared to the NIP (Somyanonthanakun et al., 2023)

Determination of Optimum pH

The pH of IIP can influence metal ions and the ionization of binding between functional groups at active sites, making it necessary to optimize the pH conditions. Moreover, the pH level affects the solubility of metal ions, which makes it a key factor in the adsorption process (Hidayat, 2017). In this study, the adsorption process was conducted at pH conditions of 5, 6, 7, and 8.

Table 1. Effect of pH on adsorption Pb^{2+} using IIP-Pb.

pH	Adsorption of Pb^{2+} ions		
	C_0 (mg/L)	Ce (mg/L)	Qe (mg/g)
5	50,04	7,92	42,12
6	48,33	5,04	43,29
7	50,16	1,28	48,88
8	51,44	4,48	46,64

As shown in Table 1, the higher the pH, the greater the adsorption capacity of IIP-Pb, reaching a maximum at pH 7. This is because lead at low and neutral pH will dissolve completely to form lead(II) ions, whereas in a basic environment above pH 7, it will form $Pb(OH)_2$ precipitate (Abdullah et al., 2019). At pH 5 and 6, the adsorption capacity is low because the carboxyl groups in the polymer become protonated and remain in the COOH form, which repels Pb^{2+} ions in the solution. At pH 8, the addition of HCl significantly reduces the H^+ concentration in the solution, causing the deprotonation of the functional monomer in the IIP (Qi et al., 2023). Additionally, the reduction in adsorption occurs at higher pH levels because metal ions

tend to form hydroxide complexes. This leads to fewer metal ions being able to bind with the active sites on the adsorbent, which reduces the overall adsorption efficiency (A. M. Tanasal, 2015) . Therefore, neutral pH is used as the optimum condition for Pb^{2+} ion adsorption in this study experiment.

Determination of Extraction Percentage and Adsorption Capacity

The pH of the adsorbate solution significantly affects the adsorption capacity of the IIP-Pb adsorbent, whether in bulk polymerization or precipitation polymerization. The optimum pH for IIP has been determined previously, so for the subsequent adsorption experiments, the adsorbate solution was conditioned at pH 7. The resulting adsorption capacity and percentage of extraction are presented in Table 2.

Table 2. The adsorption capacity IIP of bulk polymerization and precipitation methods.

Value	Bulk polymerization	Precipitation polymerization
	IIP-Pb	IIP-Pb
m (gr)	0,6841	1,509
Extraction percentage (%)	79,37	95,87
Adsorption capacity (mg/g)	46,24	56,23

As shown in Table 2, the IIP-Pb synthesized through precipitation polymerization exhibits higher adsorption capacity than the IIP-Pb synthesized through bulk polymerization, as well as a higher percentage of adsorption. This is because the precipitation polymerization of IIP-Pb results in a smaller and more uniform particle size distribution compared to bulk polymerization, thereby increasing the specific surface area. Additionally, the structural regularity helps provide consistent and effective binding sites for the target ions (Cabaleiro-Lago et al., 2023).

Conclusion

The IIP-Pb adsorbent was successfully synthesized using both bulk polymerization and precipitation polymerization methods. The results show that the IIP-Pb synthesized using the precipitation polymerization method although more time-intensitive, result in smaller more uniform particles, making it superior for lead ion adsorption compared to bulk polymerization method. The precipitation polymerization adsorbent achieved a higher adsorption capacity (56.23 mg/g) compared to the bulk polymerization method (46.24 mg/g). Both methods are selective for adsorbing lead metal, but the precipitation polymerization method is more selective than the bulk polymerization method.

Conflict of Interest

The authors declare that there is no conflict of interest.

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