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SCIENTIFIC PAPER

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INVOLVEMENT OF AMINOALKYLATED MERRIFIELD RESINS RETRACTED BY BIS(CHLORODIETHYL) ETHER IN METAL EXTRACTION

Highlights

- Merrifield resin is robust, resisting water and environmental degradation.
- MR-EDA and MR-TETA were synthesized by grafting EDA and TETA onto Merrifield resin.
- Crosslinking with bis(chlorodiethyl) ether produced MR-EDA-BCEE and MR-TETA-BCEE.
- Polymers extracted 50%-85% of Pb^{2+} and Li^{+}, with MR-TETA-BCEE performing best.
- Modified resins show promise for metal ion recovery and environmental depollution.

Abstract

Merrifield resin, a cross-linked polystyrene resin with chloromethyl functional groups, is an extremely resilient material resistant to water and environmental degradation. The objective was to develop novel substituted polymers with functional groups capable of chelating heavy metals for depollution applications. Cross-linked polymer networks, which are predominantly insoluble and adaptable to diverse chemical environments, are invaluable for pollution management. A key area of environmental significance is the selective retention of organic contaminants for purifying polluted water sources. This work focused on creating new chelating polymeric sorbents for metal extraction by grafting ethylenediamine (EDA) and triethylenetetramine (TETA) onto Merrifield resin, resulting in two polymers: MR-EDA and MR-TETA. These modified polymers were further reticulated with bis(chlorodiethyl) ether to produce two novel reticulated polymers, MR-TETA-BCEE and MR-EDA-BCEE. To characterize the polymers, differential thermal analysis (DTA) and infrared (IR) spectroscopy were employed. The resins were subsequently tested for their ability to extract lead (Pb^{2+}) *and lithium* (Li^{+}) *ions from aqueous solutions using the solid-phase extraction (SPE) method. Conductivity and atomic absorption spectrometry (AAS) analyses showed high extraction efficiencies, ranging from 50% to 85% for both* Pb^{2+} *and* Li^{+} *ions. These results demonstrated the potential of MR-EDA-BCEE and MR-TETA-BCEE as effective polymeric sorbents for environmental remediation and heavy metal removal from water.*

Keywords: Merrifield resin, ethylenediamine (EDA), chlorodiethyl, metal extraction.

INTRODUCTION

The extraction of metals from water remains a critical area of research, driven by the need to address water pollution caused by toxic metals and their harmful effects on the environment and human health [1], as well as the necessity to recover and recycle certain metal ions that play vital roles in various applications [2]. For instance, lithium has garnered particular attention due to the increasing demand for energy storage materials, such as lithium batteries, and the gradual depletion of natural reserves of this element [3]. Among the various separation techniques, solid-phase extraction (SPE) has emerged in recent years as a preferred method, owing to its technical and economic feasibility [4]. The chemical modification of polymeric resins through the incorporation of functional groups and polar compounds enhances the efficiency of extraction by increasing the surface interaction between the sorbent and

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aqueous samples [5]. Organic resins based on styrene-divinyl-benzene copolymers are especially valued for their mechanical stability, resistance to hydrolysis, pH variations, and relatively high-temperature conditions [6]. In this context, several studies have focused on functionalizing Merrifield resins (chloromethylated polystyrene). For example, Slimi *et al* [7] grafted piperazine-based chelating units onto the resin for extracting metals such as Fe^{3+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, and Pb^{2+}. Similarly, Lukashova *et al* [8] substituted tetrahydroxy-thiacalixarenes and tetraphosphonates on Merrifield resin to selectively remove europium (Eu^{3+}) from aqueous solutions. Puig *et al* [9] functionalized Merrifield resin with 2,2-pyridylimidazole, enabling selective separation of nickel from other base metals in synthetic sulfate solutions. Furthermore, the immobilization of bioactive compounds, such as quercetin, on these resins has led to the development of adsorbents capable of recovering Pb^{2+} ions. Other modifications, such as the introduction of polyamines and phosphorus derivatives, have shown effectiveness in extracting metals like Cd, Cu, and Fe [10]. Recent studies have also highlighted the efficiency of a polychloromethylstyrene-based resin modified with heterofluorenone pendant groups and polymers like poly(6-acryloylamino-N-hydroxyhexanamide), which exhibited remarkable adsorption capacities for Cu^{2+} and Pb^{2+} ions under optimal pH conditions [11].

Duan *et al.* [12,13] reported that a poly(6-acryloylamino-N-hydroxyhexanamide) resin effectively adsorbed Cu^{2+} and Pb^{2+} ions. Duan *et al.* [12] reported that a poly(6-acryloylamino-N-hydroxyhexanamide) resin effectively adsorbed Cu^{2+} and Pb^{2+} ions. Ana-Laura *et al.* [13] synthesized three Merrifield-based chelating resins (MR-PDA, MR-DPA, MR-AMP) functionalized with bidentate amines. These resins effectively remove toxic metal ions (Ag^{+}, Cu^{2+}, Pb^{2+}, Fe^{3+}) from water and are selective, reusable, and pH-tolerant—making them a sustainable solution for water treatment. In this study, two polyamine-supported polymers were prepared from Merrifield resin, modified with ethylenediamine (EDA) and triethylene-tetramine (TETA), and subsequently crosslinked using bis(chlorodiethyl) ether. The complexing properties of these novel sorbents were then studied for the extraction of a heavy metal (lead) and an alkali metal (lithium)

EXPERIMENTAL

Materials

Merrifield resin (MR reticulated by 2% divinylbenzene, 200–400 mesh, 2.1 mmol Cl. g^{-1}, Fluka), EDA, TETA, bis(chlorodiethyl) ether (BCEE), and dimethylformamide (DMF) were purcha-sed from Sigma-Aldrich. Absolute ethanol, triethylamine (TEA), and diethyl ether were purchased from Prolabo. Lithium bromide (LiBr) and lead(II) nitrate ($Pb(NO_3)_2$) were purchased from Fluka.

Synthesis of Merrifield resin with EDA group (MR-EDA)

MR-EDA was synthesized following the procedure reported in the literature [14]. Commercially available Merrifield resin (2 g) was suspended in 30 cm^3 of dimethyl-formamide (DMF) and allowed to swell for 10 hours. EDA (8.4 mmol) was then added to the suspension. An excess of EDA was used to act as a base and accelerate the reaction. The reaction mixture was stirred at room temperature for 1.5 hours, followed by heating at 70-80 °C for 24 hours. Upon completion of the reaction, the resin was thoroughly washed multiple times with deionized water, followed by absolute ethanol, to remove unreacted reagents. The final product (MR-EDA) was dried under vacuum at 50 °C for 48 hours. The resulting material is depicted in Figure 1.

Synthesis of Merrifield resin with TETA group (MR-TETA)

The reaction involved 2 g of MR, 30 cm^3 of DMF, and an excess of TETA (8.4 mmol). The reaction conditions of the product were similar to those of MR-EDA (Figure 1).

Synthesis of MR-EDA reticulated by bis(chlorodiethyl) ether (MR-EDA-BCEE)

MR-EDA-BCEE was synthesized according to the method described in the literature [15]. The starting material, MR-EDA (2 g), was dissolved in 30 cm^3 of dimethylformamide (DMF), a polar aprotic solvent that facilitates efficient dissolution and ensures the homogeneous mixing of reactants. Bis(chlorodiethyl) ether (BCEE, 6 cm^3) was then added to the solution. BCEE was used as a crosslinking agent to introduce additional functional groups, enhancing the resin's chemical properties for its intended application. The reaction mixture was stirred to ensure uniform distribution of BCEE throughout the solution and heated to 125 °C for 24 hours. The elevated temperature was necessary to activate the reaction and promote covalent bonding between MR-EDA and BCEE, ensuring effective crosslinking. Upon completion of the reaction, the resulting product was isolated by filtration to separate the modified polymer from the reaction mixture. The resin was thoroughly washed with distilled water to remove any residual reactants or by-products, ensuring the purity of the final Product. The modified polymer was initially dried at room temperature for 24 hours to remove surface moisture. It was subsequently washed with diethyl ether to remove non-polar impurities and rinsed again with distilled water to ensure all solvents and residual chemicals were eliminated. Finally, the obtained resin (Figure 1) was dried under controlled conditions. It was first oven-dried at 60 °C for 12 hours to remove residual solvents and stabilize the polymer. To achieve complete drying, the resin was subjected to vacuum drying at 70 °C for 48 hours, which removed any remaining moisture or volatile components, resulting in a stable and ready-to-use product.

Synthesis of MR-TETA reticulated by bis(chloroethyl) ether (MR-TETA-BCEE)

The reaction medium contained MR-TETA, DMF, BCEE, and TEA. The reaction conditions of the product were similar to those of MR-EDA-BCEE (Figure 1).

Extraction procedure of metal cations

The work consists of using the modified resins with amino and bis(chlorodiethyl) ether groups for the removal of metal ions (Pb^{2+} and Li^{+}) using the solid phase extraction method (SPE).

Metal ion extraction method

The kinetics of metal extraction in this experiment involves the interaction between the metal ions (Pb^{2+} and Li^{+}) and the polymer, where the rate of chelation depends on factors like polymer and metal ion concentration, temperature, pH, and the incubation time. During the incubation, the polymer binds to the metal ions, reducing their concentration in the solution. This chelation process is typically time-dependent, and a time study could be performed to model the kinetics, often following first- or second-order reaction models depending on the reaction dynamics. After the incubation, the filtrate is analyzed for the remaining metal ions using Flame Atomic Absorption Spectrometry (FAAS), which detects the metal concentration by measuring the absorption of light at characteristic

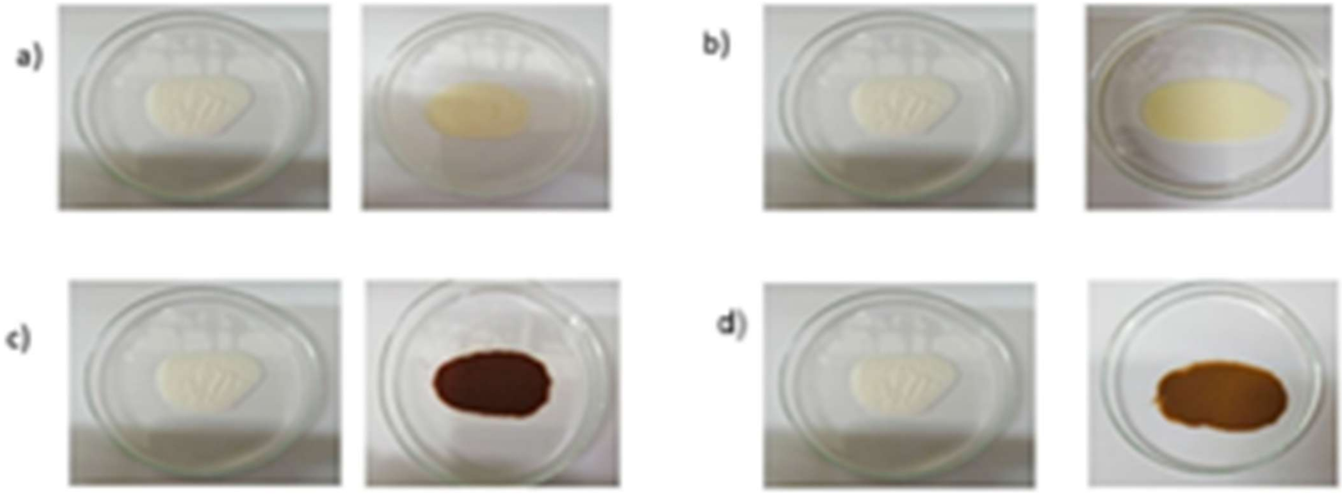

Figure 1 a) Aspect of M_0 and M_1, b) M_0 and M_2, c) M_0 and M_3, and d) M_0 and M_4.

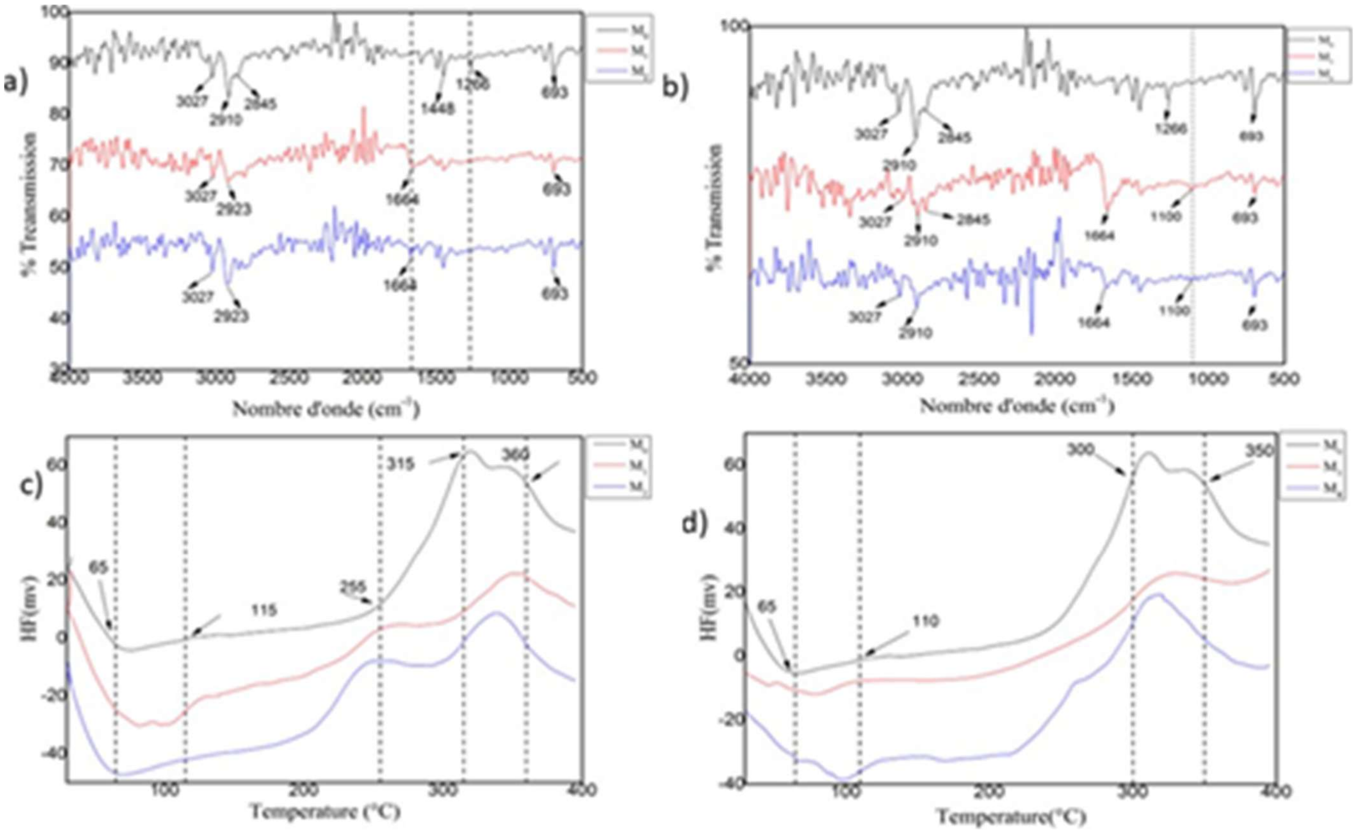

Figure 2 a) ATR-FTIR of M_0, M_1, M_2, b) ATR-FTIR of M_0, M_3, M_4, c) DTA curves of M_0, M_1 M_2, and d) DTA curves of M_0, M_3, M_4.

wavelengths. In this method, the sample is aspirated into a flame where the metal ions are atomized, and their absorption of light is proportional to their concentration. A calibration curve is created using known concentrations of the metal, allowing for the determination of the metal concentration in the filtrate. FAAS detection is sensitive, with detection limits typically in the range of 10^{-9} to 10^{-8} M, and potential interferences such as spectral, ionization, and chemical effects must be considered to ensure accurate measurements. The results were expressed as percentages of extraction of the metal (Table 1, Figure 2). The percentage was calculated using the following expression:

$$\%E=(\sigma_i-\sigma_f)\div\sigma_i \qquad (1)$$

where σ_i is the initial conductivity of the metal in aqueous solution and σ_f is the final conductivity of the metal.

Methods of testing

The infrared analysis using the (ATR FT-IR) technique was carried out on a Thermo Scientific Nicolet FTIR 200 spectrophotometer with a scanning range between 4000 and 400 cm^{-1}. Differential thermal analysis (DTA) was performed on a SETARAM TGA 92 device. The pH was measured with a pH and conductivity meter, VWR/CO 3000L.

RESULTS AND DISCUSSION

IR spectroscopy

The structures of the resulting chelating resins, MR-EDA and MR-TETA, were confirmed using ATR-FTIR spectroscopy, as shown in Figure 2. The ATR-FTIR spectra provided evidence for the successful functionalization of the Merrifield resin. A key observation was the disappearance of the characteristic CH_2-Cl bond, which appears at 1266 cm^{-1} in the spectrum of the Merrifield resin [7]. This disappearance indicates the loss of the chloromethyl functional group during the substitution reaction. In addition, new peaks were observed at 1644 cm^{-1} [16], corresponding to the deformation vibration of NH groups, confirming the incorporation of EDA or TETA into the resin structure. Amines exhibit characteristic bands in infrared spectroscopy, enabling their identification. The N-H stretching vibrations typically appear in the region 3300-3500 cm^{-1}. Primary amines ($-NH_2$) show two bands in this region, corresponding to symmetric and asymmetric stretching, while secondary amines (−NH) display only one. These bands are usually broad and of moderate intensity. Another characteristic band, associated with N-H bending vibrations, is found in the region 1500-1650 cm^{-1}. Lastly, the C-N stretching vibration appears between 1200 and 1350 cm^{-1}. These features help distinguish between primary, secondary, and tertiary amines in a mixture or compound. This observation is consistent with findings by Kadous *et al.* [12], who reported a similar peak at 1602 cm^{-1} in polystyrene resin grafted with EDA. These spectroscopic changes collectively support the substitution of the chloro group by polyamine groups. For the resins MR-EDA-BCEE and MR-TETA-BCEE, the ATR-FTIR spectra (Figure 2) displayed all the characteristic bands observed for MR-EDA and MR-TETA. Additionally, a new valence band was detected at 1100 cm^{-1}, corresponding to the C-O-C stretching vibration. This band indicates the successful grafting of the bis(chlorodiethyl) ether group onto the amino-functionalized Merrifield resins. Similar findings were reported by Mbarki *et al.* [15], who observed a C-O-C band in the range of 1050-1170 cm^{-1} for amino-PVC modified with bis(chlorodiethyl) ether.

DTA analyses

The DTA curves of MR-EDA, MR-TETA, MR-EDA-BCEE, and MR-TETA-BCEE (Figure 2) showed that all resins present two peaks, an exothermic peak between 300 and 360 °C that could indicate their decomposition, and an endothermic peak between 65 and 115 °C that can be explained by the evaporation of residual water. Dardouri *et al.* [10] observed the same decomposition peaks at 305, 330, and 509 °C.

Metal ion extractions by the synthesized polymers

The extraction was also performed using the atomic absorption spectrometry technique. Table 1 shows the heavy metal extraction determined by conductivity measurements and by AAS. The difference between the extraction percentages determined by these two methods (the absorption and conductivity methods) is between 8% and 12%.

Effect of initial pH on modified Merrifield resins

The pH of the aqueous phase plays a crucial role in the extraction of Pb^{2+} and Li^+ ions by an extracting agent. It influences both the chemical behavior of the functional groups in the functionalized resin and the nature of the species present in the solution. Studying the retention efficiency of lead and lithium ions as a function of pH helps determine the optimal conditions for the extraction process. The effect of pH on the extraction of these ions was examined within the pH range (1.0-6.0), using an aqueous solution containing 2 × 10^{-4} M of Pb^{2+} and Li^+. A volume of 30 mL of this solution was treated with 0.1 g of resin, while the agitation speed was kept constant at 250 rpm using a vibrating platform. The results show that the extraction efficiency of Pb^{2+} and Li^+ ions increases with pH, reaching a maximum of 80% at pH 6.1 (Table 2). At low pH, the high concentration of H^+ ions in solution leads to competition between protons and Pb^{2+}/Li^+ ions for extraction. Additionally, in an acidic medium, the lone electron pairs of the nitrogen and oxygen atoms in the Merrifield resin are blocked by H^+ ions, reducing their availability for complexation with metals. Furthermore, in an environment rich in H^+, these ions strongly adsorb onto the resin, giving it a positive charge that creates electrostatic repulsion, thereby limiting the approach of Pb^{2+} and Li^+ ions.

The extraction percentages of metal ions Pb^{2+} and Li^+ with the chelating resins M1, M2, M3, and M4 are shown in Table 1 and Figure 3. The values are between 50% and 85%, which indicates that all resins are of a high metal nature for both Pb^{2+} and Li^+. Figure 3 shows that the order of adsorption capacity of the chelating resins against Pb^{2+} and Li^+ ions is as follows: MR-TETA > MR-EDA, which is similar to other investigations [9,11]. This result was explained by the higher content of nitrogen groups (% N) in modified resins, which improves coordination with metallic ions. The extraction percentages of Pb^{2+} and Li^+ with the synthesized polymers MR-EDA, MR-TETA, MR-EDA-BCEE, and MR-TETA-BCEE varied between 65.6 and 85.5% and 50 and 87%, respectively. All the synthesized polymers showed more selectivity for the Pb^{2+} which has a higher size than Li^+, these results can be explained by the compatibility factor between the size of the Pb^{2+} ion and the size of the complexing cavity in the polymers, which could be influenced by the cross-linking degree, the length of amine chain and the cross-linking by the bis(chlorodiethyl) ether is directly proportional. However, the active sites are less readily available, and the efficiency of the resin is reduced. The orders of adsorption capacity for Pb^{2+} were respectively: MR-TETA-BCEE > MR-EDA-BCEE > MR-TETA > MR-EDA. Besides, the orders of adsorption capacity for Li^+ are as follows: MR-TETA-BCEE > MR-TETA> MR-EDA-BCEE> MR-EDA. When looking at all the results, as an average of all polymers, we can observe that the synthesized polymer that is the best extractant and the most selective for both Pb^{2+} and Li^+ is MR-TETA-BCEE, respectively, with 85.5% and 78%. This high adsorption selectivity could be explained by the high affinity of Pb^{2+} and Li+ ions to the polyamine and oxygen groups in the studied resins, which can fix the metal through non-cova-lent bonds (van der Waals bonds) as shown in Figure 5. This extraction efficiency could also be partially explained by the electro-negativities of the substituted nitrogen of the polyamine and oxygen groups. In the case of Li+, the polymer M4 (78 %) is the better extractant; Li+ is less voluminous than Pb^{2+}. Based on the hard-soft acid-base theory, Li^+ has affinities to hard ligands, which contain oxygen atoms like in M_4 more than Pb^{2+} [18-19].

Kinetic study

Figure 3 shows variation curves of average electrical conductivity with time for metal cations Pb^{2+} and Li^+ in contact with the modified polymers (M1, M2, M3, and M4). The kinetic study showed that the optimum extraction time obtained was approximately 35 h and 22 h for Pb^{2+} and Li^+ extraction, respectively. The extraction results are the average of three experiments. These results show that all modified resins M_1, M_2, M_3, and M_4 give better results (shorter extraction durations) with Li^+ cation. However, the development of new chelating ion exchange materials with special chelating properties can provide better kinetics of interaction between metal ions and chelating groups [6].

Table 1 Extraction percentage results of metal ions (Pb^{2+}, Li^+) by the synthesized polymers.

Metal cation	%with M_1 Conductivity	AAS	%with M_2 Conductivity	AAS	%with M_3 Conductivity	AAS	%with M_4 Conductivity	AAS
Pb^{2+}	65.6	57.6	73	61	82.6	72.6	85.5	75
Li^+	50	42	68.3	55.7	57.4	47	78	68

Table 2. Extraction Percentages of Pb^{2+} and Li^+ Ions as a Function of pH (M1 to M4).

Metal cation	%with M_1 to PH=2.0	%with M_2 to PH=3.0	%with M_3 to PH=5.1	%with M_4 to PH=6.1
Pb^{2+}	62	70	80	83
Li^+	45	63	55	75

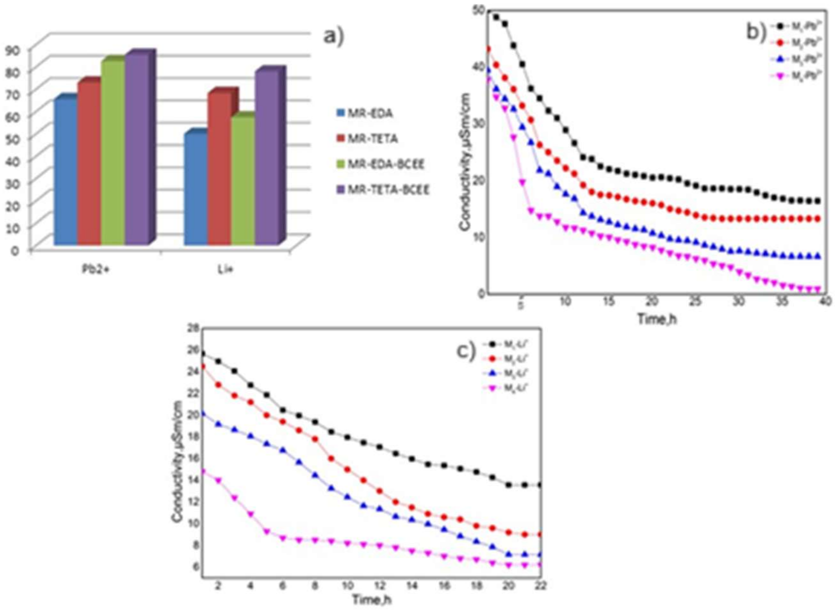

Figure 3 a) Percentages of metal ions extraction for the polymers M_1, M_2, M_3, and M_4, b) curves of the optimal duration of Pb^{2+} extractions, and c) curves of the optimal duration of Li^+ extractions.

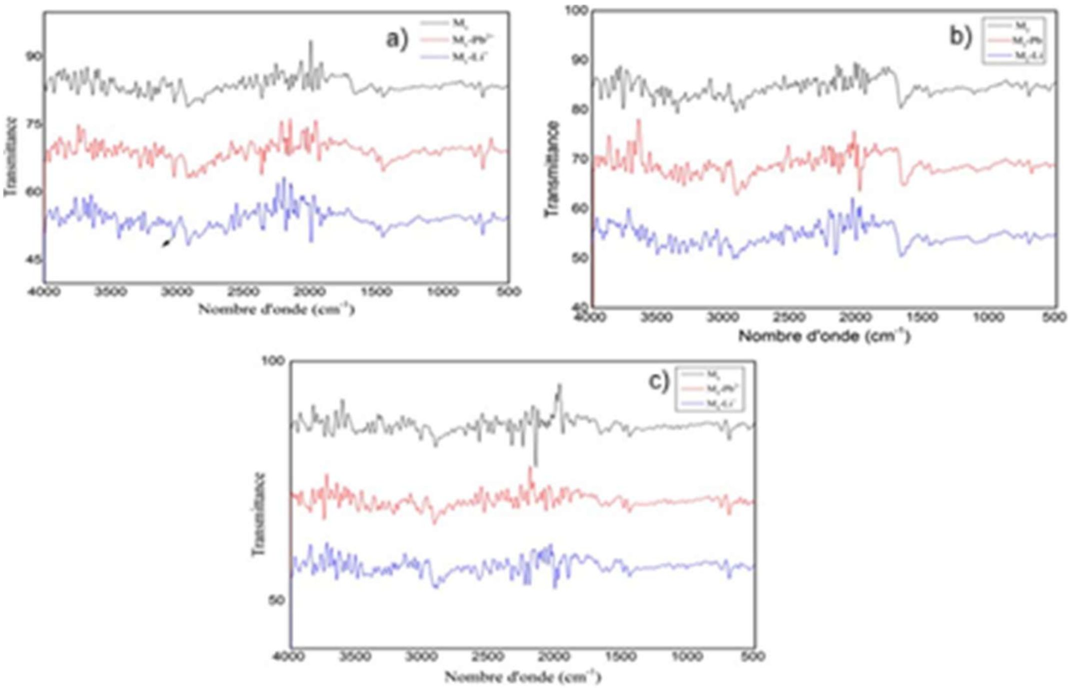

Figure 4 IR spectra of (a) polymer M_1 and complex M_1-Pb^{2+}, M_1-Li^+; (b) polymer M_2 and complex M_2-Pb^{2+}, M_2-Li^+; and (c) polymer M_4 and complex M_4-Pb^{2+}, M_4-Li^+.

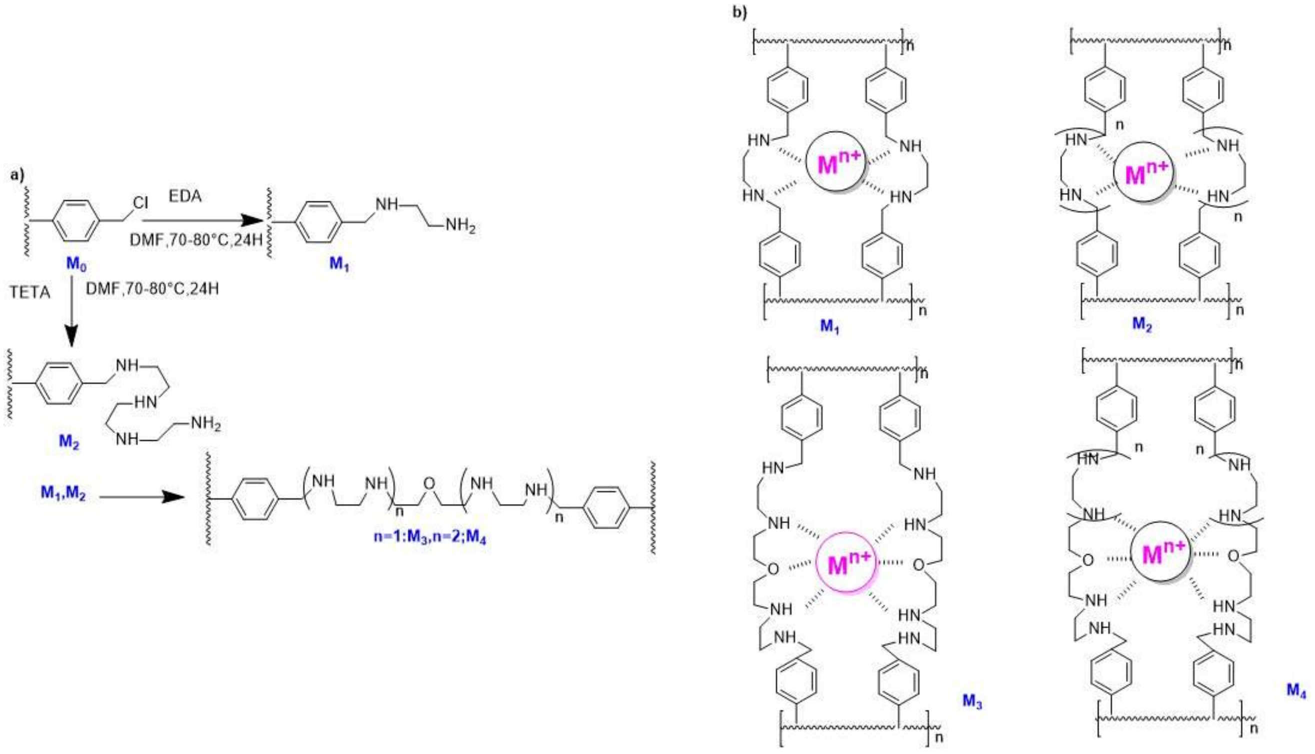

Figure 5 (a) Proposed structure of polyamine resin, and (b) Proposed complex structures of M_1, M_2, M_3, and M_4 with M^{n+}: Li^+ or Pb^{2+}

The demonstration that metal coordination occurs through amines and not simply via "entanglement" in the Merrifield resin is based on several observations from Figure 5. Graph (a) shows that resins functionalized with amines (MR-EDA, MR-TETA, and their derivatives) exhibit higher adsorption rates for Pb^{2+} and Li^+, suggesting a specific chemical interaction involving amine groups. Graphs 4 (b) and (c) illustrate a progressive decrease in solution conductivity over time, indicating the extraction of metal ions. The difference in kinetics among the resins (M_1, M_2, M_3, and M_4) and their superior performance for Pb^{2+}, which has a higher affinity for amines due to its electronic properties, supports the hypothesis of metal-amine coordination. This selective behavior and the dependence on the type of amine confirm that the interaction is chemical and specific, rather than a mere physical entanglement.

Influence of extracted metals on the IR spectra of new resins

The IR absorption spectra of the studied complexes in Figure 4 indicate that the influence of free polymers and their complexes on the IR spectra is not very significant. Ammari *et al.* [17] found the same observation with functionalized PVC, explaining it by the effect of the counter-anions of the used salts.

CONCLUSION

In this study, we demonstrated the efficiency of modified Merrifield resin-based polymers for extracting Pb^{2+} and Li^{+} ions from aqueous solutions. The integration of polyamine groups, followed by cross-linking with bis(chlorodiethyl) ether, enhanced the adsorption capacity of the polymers, with a particularly high performance for lead extraction. Among the synthesized materials, MR-TETA-BCEE proved to be the most effective, highlighting the influence of functional group type and structure on selectivity and extraction efficiency. These findings suggest that the developed polymers could be promising candidates for water decontamination and the selective recovery of industrially valuable metals. Further investigations, particularly on the reusability of the resins and their efficiency under real-world conditions, will be necessary to fully assess their potential for industrial and environmental applications.

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NAUČNI RAD

UČEŠĆE AMINOALKILIRANIH MERIFILD SMOLA RETRAKCIRANIH BIS(HLORODIETIL)-ETROM U EKSTRAKCIJI METALA

Merifild smola, umrežena polistirenska smola sa hlorometil funkcionalnim grupama, izuzetno je otporan materijal na vodu i degradaciju u životnoj sredini. Cilj rada je bio razvijanje novih supstituisanih polimera sa funkcionalnim grupama sposobnim da heliraju teške metale za primene u prečišćavanju. Umrežene polimerne mreže, koje su pretežno nerastvorljive i prilagodljive različitim hemijskim sredinama, neprocenjive su za upravljanje zagađenjem. Ključno područje od ekološkog značaja je selektivno zadržavanje organskih zagađivača za prečišćavanje zagađenih izvora vode. Ovaj rad je fokusiran na stvaranje novih helatnih polimernih sorbenata za ekstrakciju metala kalemljenjem etilendiamina (EDA) i trietilentetramina (TETA) na Merifild smolu, da bi se dobila dva polimera: MR-EDA i MR-TETA. Ovi modifikovani polimeri su dalje retikulirani bis(hlorodietil) etrom, pri čemu se dobijaju dva nova retikulisana polimera, MR-TETA-BCEE i MR-EDA-BCEE. Za karakterizaciju polimera korišćene su diferencijalna termička analiza i infracrvena spektroskopija. Smole su potom testirane na sposobnost da ekstrahuju jone olova i litijuma iz vodenih rastvora korišćenjem metode ekstrakcije iz čvrste faze. Analize provodljivosti i atomske apsorpcione spektrometrije pokazale su visoku efikasnost ekstrakcije, u rasponu od 50% do 85% za jone Pb^{2+} i Li^{+}. Ovi rezultati pokazuju potencijal MR-EDA-BCEE i MR-TETA-BCEE kao efikasnih polimernih sorbenata za sanaciju životne sredine i uklanjanje teških metala iz vode.

Ključne reči: Merifildova smola, etilendiamin (EDA), hlorodietil, ekstrakcija metala.