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Synthesis and Chelating Properties of Polystyrene Supported Schiff Base (N,N′-disalicylidenepropylenetriamine) Resin Toward Some Divalent Metal Ions

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A new polystyrene-supported Schiff base resin, N,N-bis(salicylidenepropylenetriamine)- aminomethyl polystyrene**,** has been synthesized through a reaction between the commercially available 4-chloromethyl polystyrene polymer and the Schiff base, N,N′-disalicylidenepropylenetriamine. The chelation behavior of this resin toward the divalent metal ions Cu^{2+}, Ni^{2+}, Zn^{2+}, and Pb^{2+} in aqueous solutions was investigated. Batch equilibration experiments were carried out as a function of contact time, pH, amount of metal-ion, polymer mass, and temperature. The amount of metal-ion uptake of the polymers was determined by using atomic absorption spectrometry (AAS). Results of the study revealed that the resin exhibited higher capacities and a more pronounced adsorption toward Cu^{2+} and that the metal-ion uptake follows the order: $Cu^{2+} > Zn^{2+} > Ni^{2+} > Pb^{2+}$. The adsorption and binding capacity of the resin toward the various metal ions investigated are discussed.

Keywords: polystyrene, FT-IR, Schiff bases, thermogravimetric analysis (TGA), atomic absorption spectrometry

1 Introduction

Metal chelating polymers, which are characterized by reactive functional groups containing O, N, S, and P donor atoms, constitute an important class of versatile polymeric materials that found widespread applications in environmental remediation, monitoring and separation of trace heavy metal ions from aqueous solutions. These polymeric matetrials are capable of coordinating to different transition metal ions and have been extensively studied (1). Chelating polymers are normally produced by incorporating active chelating groups into a polymeric matrix (2). Such chelating groups may be covalently bound to a polymer matrix as pendent groups or incorporated into the repeating units of the polymer backbone by polymerization of a suitable monomer containing the required chelating group. It has been demonstrated that the nature of intervening groups connecting the active chelating ligands in chelating polymers plays an important role in the chelation process (2, 3).

In the interaction between a chelate-forming resin and metal ions, the electrostatic exchange is accomplished by a rapid chemical reaction, leading, in many cases, to strong metal ligand bonds. The intensity of the chelating interaction is governed by such properties of the metal ion as their oxidation state, electronic configuration, stereochemistry, basicity and the polarization of the ligand on the resin (4). The nature of the metal species, the functional group and/or donor atom capable of forming complexes with metal ions are also of fundamental importance in metal extraction by polymers.

Schiff bases with multi-dentate coordination sites are known to readily form complexes with transition-metal ions; they are expected to show affinity and selectivity towards metal ions when present in a polymeric matrix (1). Samal and coworkers have synthesized a number of phenol-formaldehyde-type polymers by condensing the phenolic Schiff bases with formaldehyde and furfuraldehyde and used these polymers for the separation of metal ions (1). In addition, the same researchers have prepared polymers from the condensation of phenolic Schiff

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bases derived from 4,4′-diaminodiphenylmethane and *o*-hydroxyacetophenone with formaldehyde and furfuraldehyde. On the other hand, Kumar and Mathew (5) prepared a polystyrene-supported Schiff base through the reaction of the Schiff base derived from diethylenetriamine and salicylaldehyde with 4-chloromethyl polystyrene. They studied the catalase-like activity of the metal complexes of various cross-linked polystyrene-supported Schiff bases and correlated these activities with the nature and degree of crosslinking in the polymer support. Additionally, Reddy and Reddy investigated the metal ion uptake properties of polystyrene-supported chelating polymer resins functionalized a number of chelating groups including Schiff bases (6).

Heavy metals are commonly found in large quantities in industrial wastewaters. For this reason, the recovery of the metal ions present in these wastewaters is necessary for environmental protection and economical reasons (7). The most common metals found in wastewater are copper, cadmium, nickel, lead and zinc which are toxic at high concentrations (7). The presence of heavy metals in the environment is of great concern because of their increased discharge, toxic nature and other natural water adverse effects on receiving water (8). Metal contamination of various water resources is also of great concern because of the toxic effect to the human beings and other animals and plants in the environment (9). Therefore, the removal of those heavy metal ions the presence of which in the aquatic environment pose heavy risks to human health may be considered an important research activity. The major sources of heavy metal pollutants are usually from many industries, including mining, metal plating, and electric device manufacturing among others (10). Solid organic and inorganic ion exchangers constitute the basis of widely employed chemical separation procedures, with applications ranging from analytical and environmental chemistry research to water purification, waste management and material technologies (such as in nuclear and electroplating industries) (7).

In connection with our recent work on the synthesis and sorption properties of chelate-forming polymers (7, 11–19), we report, herein, on the synthesis and characterization of a new polystyrene supported Schiff base (*N*, *N*′-disalicylidenepropylenetriamine) resin. In addition, the chelation properties of the new resin towards the divalent metal ions, Cu^{2+}, Zn^{2+}, Ni^{2+}, and Pb^{2+}, in aqueous solutions under different experimental conditions of contact time, pH, mass of resin, concentration of metal ions, and temperature are investigated.

2 Experimental

2.1 Reagents

Unless otherwise indicated, all chemicals were obtained from commercial sources and were used as received; the chloromethylpolystyrene (Merrifield Polymer) crosslinked with 5.5% DVB; porous;16–50 mesh, 5.5 mmol Cl/g resin from Fluka (Buchs, Switzerland), dipropylenetriamine and salcylaldehyde 98% from Acros Organics (Geel, Belgium). The following metal-ion salts were also used without further purification: copper(II) acetate dehydrate (98%), from Fluka, nickel(II) acetate tetrahydrate from BDH (Poole, England), zinc(II) acetate dehydrate from Riedel de Haen (Seelze, Germany), and lead(II) acetate trihydrate from S. D. Fine Chemicals, Ltd (Mumbai, India).

2.2 Instrumentation

Infrared spectra of the polymers were recorded with a Nicolet Impact 400 Fourier transform infrared Spectrophotometer (Madison, WI) from 400 to 4000 cm^{-1}. KBr discs were used for all of the solid samples by the mixture of 1.0 mg of the sample with about 100 mg of KBr.

Atomic absorption data were obtained with the aid of a Varian Atomic Absorption spectrophotometer (Mulgrave, Victoria, Australia) model AA-250 plus. Samples were shaken and thermostated using GFL-1083 shaker. A Metrohm pH meter model 525A was used for pH measurements. Elemental analyses were acquired with a Euro EA3000 CHNS-O elemental analyzer (Milian, Italy). High resolution mass spectral data were obtained with a Brucker APEX (IV) mass spectrometer (Bremen, Germany). The thermal stabilities of the polymer samples were studied by thermogravemetric analysis (TGA) with the aid of a Netzch STA 409 PG/PC thermal analyzer (Selb Bavaria, Germany). Measurements were performed at a heating rate of 20°C/min under dry nitrogen atmosphere purging at a flow rate of 50 mL/min in the temperature range of 20–1000°C.

2.3 Preparation of Schiff Base

3,3′-Diaminodipropylamine (30 mL, 0.208 mol) was added dropwise to a solution of salicylaldehyde (50.7 g, 0.416 mol) in absolute ethanol (250 mL); the solution became instantly yellow. The mixture was heated for 1 h at 50°C. Evaporation of the solvent under reduced pressure afforded the desired compound in 95% yield as yellow oil. IR (KBr, cm^{-1}): 1633 (C=N), ^{1}H-NMR ($CDCl_3$) δ (ppm): 1.91 (m, 5H, CH_2CH_2NH + NH), 2.76 (m, 4H, CH_2NH), 3.63 (m, 4H, $CH{=}NCH_2$), 6.80–7.30 (m, $10H_{arom}$+ 2OH), 8.36 (s, 2H, CH=N). ^{13}C-NMR ($CDCl_3$) δ (ppm): 31.51 (CH_2-CH_2-CH_2), 47.42 (NH-CH_2), 56.66 (=N-CH_2), 116.85, 121.78, 124.43, 131.93, 132.55, and 161.27 (C_{aroma}), 166.14 (HC=N); HRMS (CI) *m*/*z*: Calcd for $C_{20}H_{26}N_3O_2$ $[M{+}H]^+$340.20195; found 340.20253. Anal. Calcd. for $C_{20}H_{25}N_3O_2$: C, 70.77; H, 7.42; N, 12.38. Found: C, 70.69; H, 7.38; N, 12.32.

2.4 Preparation of N,N-bis(salicylidenepropylenetriamine) aminomethyl Polystyrene Resin

The titled polymer was prepared according to a procedure outlined by Kumar and Mathew (5) which involved allowing the commercially available chloromethyl polystyrene (10 g) to swell in dioxane (100 mL). The Schiff base was then added (3 molar excess of the chlorine capacity) and the mixture was stirred for about 48 h at 90°C. The polymer was collected by filtration and was thoroughly washed with water. After Soxhlet extraction with dioxane for 24 h, the resulting solid resin was dried in vacuum. Satisfactory elemental analyses were obtained: Anal. Cald. for $[C_{29}H_{33}N_3O_2]_n$: %C 76.45, %H 7.30, %N 9.22. Found: %C 76.32, %H 7.18, %N 9.09.

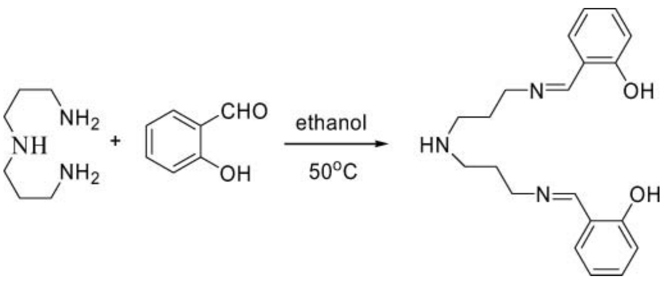
2.5 Sorption of the Metal Ions on the Polymer

The metal chelation characteristics of the resin for each metal ion were studied by the batch equilibrium technique. Duplicate experiments involving 0.100 g of dry, 35–60 mesh size, polymer samples were suspended in 25 mL of sodium acetate-acetic acid buffer and with a total ionic strength of 0.20 M (with sodium perchlorate) with continuous shaking and left for 2 h to equilibrate. To this mixture, 25 mL of metal ion solution containing a total of 25 mg of metal-ion were added. After being shaken for a definite period of time at 25°C, the mixture was filtered, and the amount of metal ion remaining in the filtrate was determined by atomic absorption spectrometry (AAS) using standard solutions for calibration.

The extent of metal-ion uptake was studied under similar experimental conditions, where the contact time was varied from 0.08 to 24 h at 25°C after the solution was equilibrated with distilled water. Similar experiments were also carried out in buffered solutions, in which the pH was varied between 3.0 and 7.0 for a fixed contact time of 6 h.

The effect of resin mass on the metal-ion uptake was also studied using the same general procedure by shaking a suspension of 0.1, 0.2, 0.3, 0.5, 0.7, or 1.0 g of the dry polymer in 25 mL of the acetate buffer solution at pH 6.0 for 2 h. To this mixture, 25 mL of buffer solution containing 25 mg of metal-ion were added. The mixtures were then shaken at 25°C for 6 h, filtered, and the amount of metal ion remaining in solution was determined by AAS.

The effect of metal-ion concentration was investigated in a similar fashion in buffer solutions containing 0.10 g of dry polymer and variable amounts of metal ions at 25°C and a fixed contact time of 6 h. Similarly, the effect of temperature on metal ion uptake was investigated by suspending 0.1000 gram of dry resin in 25.0 mL of sodium acetate-acetic acid buffer of pH = 6.0 for 2 h of continuous shaking. Then 25 ml samples of copper(II) solutions of different concentrations were added to produce a total of 50 mL solutions containing 4.16–41.67 mg of Cu(II) ion. The mixtures were shaken at different temperatures of (30.0, 40.0, and 50.0°C) for a fixed contact time of 24 h. The samples were filtered and the Cu(II) concentration remaining in solution was determined with an atomic absorption spectrophotometer.



Scheme 1. Prepration of salicylaldehyde-dipropylenetriamine Shiff base.

3 Results and Discussion

3.1 Preparation of N,N′-bis(salicylidenepropylenetriamine) aminomethyl Polystyrene

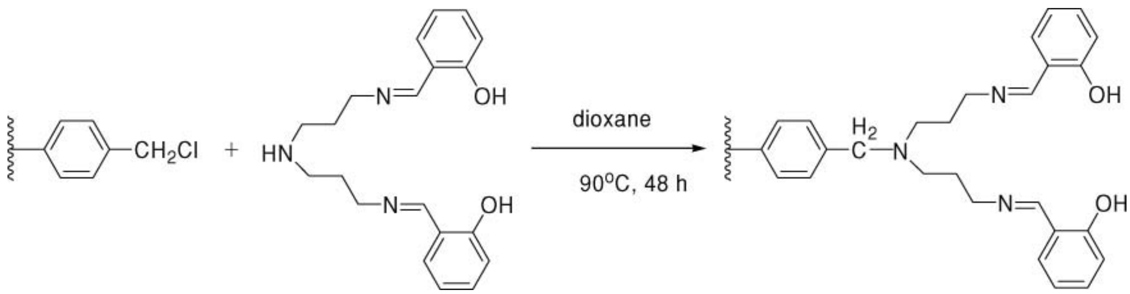
The Schiff base derived from salicylaldehyde and dipropylenetriamine was prepared according to the procedure outlined by Nishat and coworkers (20) as depicted in Scheme 1, salicylaldehyde and dipropylenetriamine were condensed in a 1:2 molar ratio. The structural characterization of the Schiff base was based on FT-IR, NMR spectroscopy, mass spectrometry, and elemental analysis. The IR spectrum of the Schiff base exhibits a strong absorption band at 1629 cm^{-1} attributed to azomethine (C=N), and a broad band at 3432 cm^{-1} due to the phenolic-OH. Moreover, the disappearance of the salicylaldehyde C=O band and the NH_2 band of dipropylenetriamine are consistent with the formation of the Schiff base. Similar results were obtained by El-Asmy and coworkers (21). The ^{1}H-NMR spectrum of the Schiff base showed the azomethine protons (HC=N) as singlets at 8.36 ppm along with a complex set of multilplet in the range 6.80 to 7.30 ppm corresponding to the ring and the phenolic protons.

The polystyrene supported Schiff base resin was prepared by treating the commercially available 4-chloromethylpolystyrene resin with the freshly prepared Schiff base as shown in Scheme 2.

The extent of incorporation of the ligand function was followed by estimation of the residual chlorine content of the obtained resin by the modified Volhard's method (5). In addition, elemental analysis of the prepared resin indicated that there was total chlorine displacement.

3.2 Characterization of the Polymer

The IR spectrum of the prepared resin is in agreement with the structure assigned to it. The broad bands at 3432 cm^{-1}



Scheme 2. Preparation of polystyrene-supported salicylaldehyde- dipropylenetriamine Schiff base.

is attributed to phenolic OH vibrations. These bands were also observed in the metal chelates because only a fraction of the functional groups on the polymer are involved in chelate formation. Absorption bands at 2925 cm^{-1} indicate intramolecular hydrogen bonding resulting from lowering of OH-vibration (6). In addition, bands that appear between 1020–1280 cm^{-1} are due to the aliphatic N while the strong and sharp band at 1632 cm^{-1} is ascribed to C=N vibration. The C=C absorption bands appear at 1580, 1507, and 1457 cm^{-1} (1). On the other hand, the sharp decrease in intensity of the band near 3400 cm^{-1} and 2900 cm^{-1} supports the involvement of the phenolic oxygen in metal ion coordination; this was observed by other researchers (5). Additionally, the appearance of two new bands in the Cu-loaded resin around 623 cm^{-1} and 558 cm^{-1} are due to metal coordination of the metal ions with phenolic oxygen (M-O) and azomethine nitrogen (M-N), respectively. This has been reported by other workers (20, 22). The IR spectra of the resin and the Cu-loaded resin are shown in Figure 1.

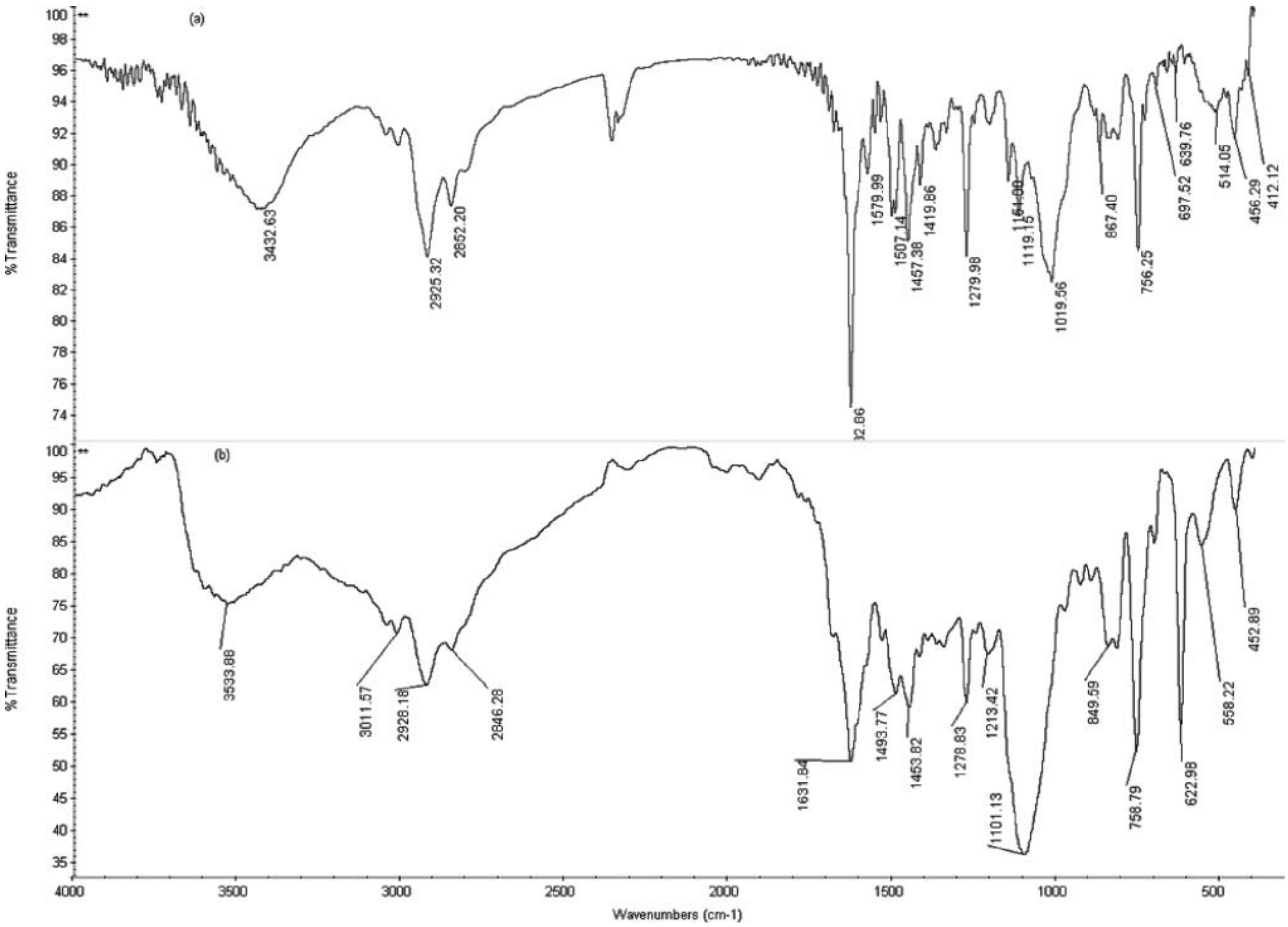


Figure 1. a) IR spectrum of resin, N,N-bis(salicylidenepropylenetriamine)- aminomethyl polystyrene, b) IR spectrum of Cu^{2+} loaded resin.

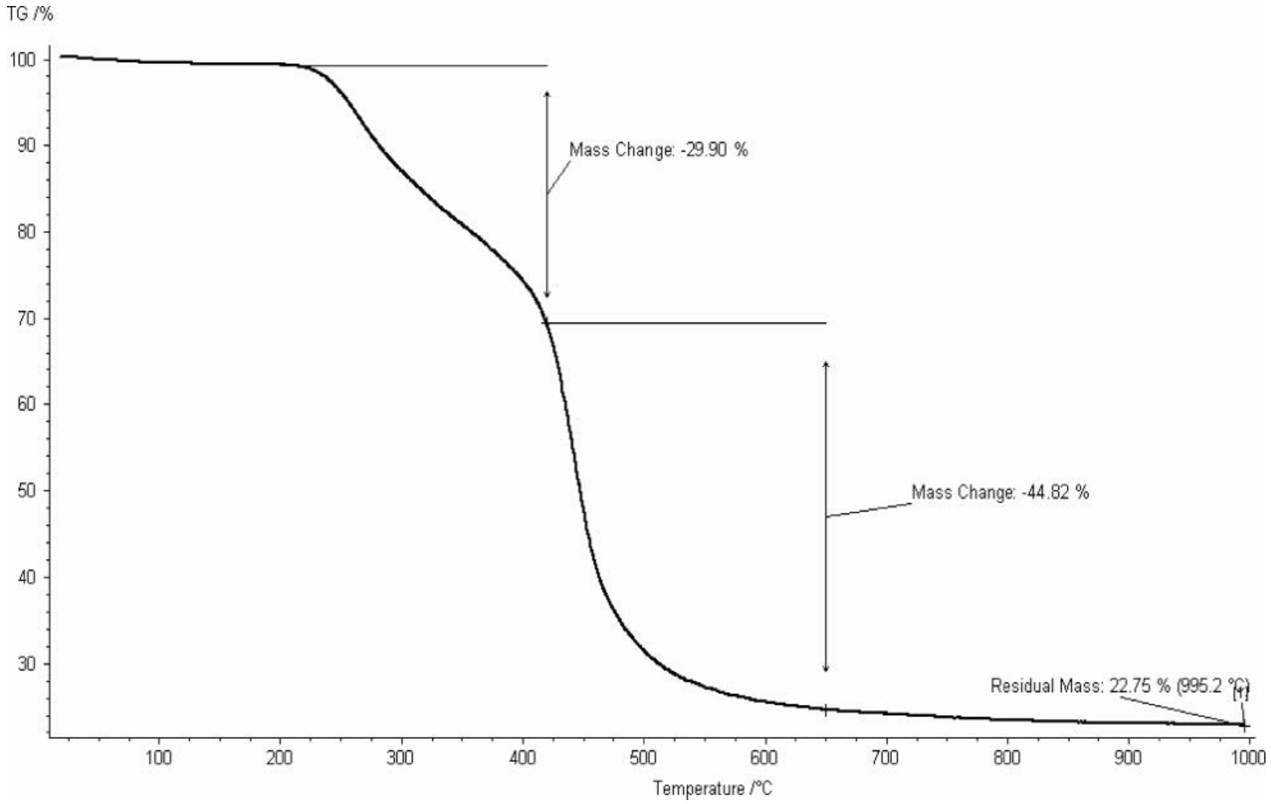


Figure 2. TGA thermogram of the resin. TG % is the mass percentage of the polymer sample remaining after heating the polymer to a certain temperature.

3.3 Thermal Stability of the Polymer

The thermal stability of the resin was investigated by TGA under dry nitrogen. Shown in Figure 2 is the TGA thermogram of the resin; it displays two-stage degradation with a slow loss of mass starting from ~ 220°C to 420°C with a 30% weight loss followed by a relatively faster loss of mass between 420°C and 650°C with an additional 45% weight loss. The residual mass percentage remaining after the resin was heated to 995°C was about 23%. These results show that the prepared resin has good thermal stability.

3.4 Rate of Metal-Ion Uptake as a Function of Contact Time

The sorption of various divalent metal ions (Ni^{+2}, Cu^{+2}, Pb^{+2}, and Zn^{+2}) on polymer as a function of contact time was investigated by a batch equilibrium technique. Results for the dependence of the metal ion uptake on contact time for polymer are presented in Figure 3. The results indicate fast rates of equilibration; the rates of metal-ion uptake increase in the first 3 h and a steady state is reached within 5–10 h. Results also revealed that the metal-ion uptake follows the order: $Cu^{+2} > Zn^{+2} > Ni^{+2} > Pb^{+2}$; resin shows highest uptake capacity toward Cu(II) and lowest for Pb(II). This trend may be attributed to a combination of factors including metal-ligand stability constants, metal ionic radii, and stereochemical configuration of active chelating sites among others (18). Apparently, the formation constant of Cu^{2+} ions-polymer is higher than those of other metal ions; this behavior has been observed by other researchers (23). In addition, the ionic radius for Cu^{+2} is 71 pm and for Pb^{+2} it is 112 pm. The stability of the chelate is expected to be less favorable for ions of larger size; this is consistent with earlier investigations (7). Moreover, variation of contact time showed a maximum loading capacities of the different metal ions in mg/g resin for the polymer of 203.1, 180.2 153.2, and 136.1, for Cu^{+2}, Zn^{+2}, Ni^{+2}, Pb^{+2}, respectively. These are relatively large amounts and indicate that this polymer can be effectively used to remove heavy

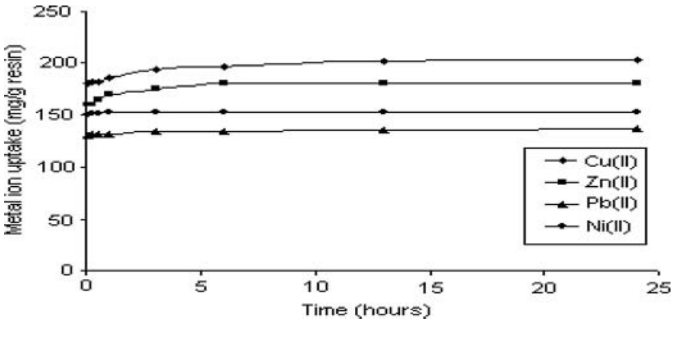


Figure 3. Metal-ion uptake by resin as a function of contact time.

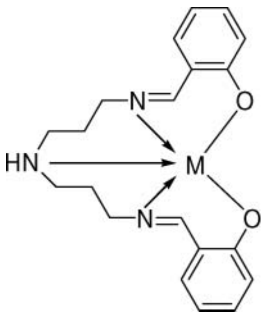
metal ions from the aquatic environment. In addition, the metal ion uptake capacities of the present resin are comparable to those of resins of comparable structures prepared by other researchers; Samal and coworkers have reported maximum loading capacities of close to 250 mg/g resin for Cu and 178 mg/g resin for Ni using *o*-phenylenrdiamine-salicylaldehyde Schiff base resin (24). In an investigation conducted by Kumar and Mathew (5) which primarily deals with the effect of the nature and degree of crosslinking on the catalase-like activity of polystyrene-supported Schiff base-metal complexes, they found that for the copper ion uptake of EGDMA (polystyrene-supported Schiff base crosslinked with ethyleneglycol) crosslinked with 2% ethylene glycol was 2.62 meq/g resin which is equivalent to 166.4 mg/g resin which is less than the copper ion uptake of our resin.

Recently, we have investigated the sorption properties of the commercially available iminodiacetate ion exchange resin, best known as Amberlite IRC-718 toward a number of heavy metal ions; it showed a maximum loading capacity of about 170–180 mg/g resin for Cu and about 100 mg/g resin for Ni (7); this indicates that the newly prepared resin is at least as good as Amberlite IRC-718 in removing heavy metal ions from aqueous solutions.

3.5 Effect of pH on Metal-Ion Uptake by the Polymers

The binding capacity of the resin toward the investigated metal ions was studied in the pH range 3–7 under continuous shaking for a fixed contact time of 6 h at 25°C and at an ionic strength of 0.20 M. At higher pH values, hydrolysis of the metal ions investigated becomes significant and may compete with polymer chelate formation. Typical pH-binding capacity profiles are displayed in Figure 4.

Results reveal that metal-ion uptake slightly increased with pH of the medium and approached a steady state at about pH 6.0. This behavior could be explained by the nature of the chelating group; the hydroxyl groups existed in equilibrium, that involves protonated and unprotonated forms in the investigated pH range. The degree of protonation and deprotonation will critically affect the ability of the resin to bind metal cations. Therefore, at higher pH values, the divalent metal ions competed favorably toward donor sites compared with hydrogen ions, in accordance with our recent work (18). In addition, metal ions were coordinated with the polymer through the oxygen of phenolic –OH group and nitrogens as illustrated in Scheme 3.



Scheme 3. Coordination of the polymer to metal ions.

3.6 Effect of Metal-Ion Concentration on Metal-Ion Uptake

The effect of metal-ion concentration on metal-ion uptake was studied by suspending 0.10 g of the dry polymer in 25 mL of the acetate buffer solution at pH 6.0 for 2 h followed by the addition of 25 mL of buffer solution containing different amounts of metal-ion. Results shown in Figure 5 reveal that the metal-ion uptake capacity of the resin increases with the increase in the initial metal ion concentration; this has been reported by other workers (7, 24).

3.7 Effect of the Polymer Mass on the Metal Ions Uptake

Effect of resin mass on metal-ion uptake The effect of resin's mass on the rate of metal-ion uptake was investigated using a batch equilibration technique by suspending different masses (0.1, 0.2, 0.3, 0.5, 0.7, or 1.0 g) of the dry resin in 25 mL of the acetate buffer solution at pH 6.0 for 2 h. Then, 25 mL of buffer solution containing 25 mg of metal

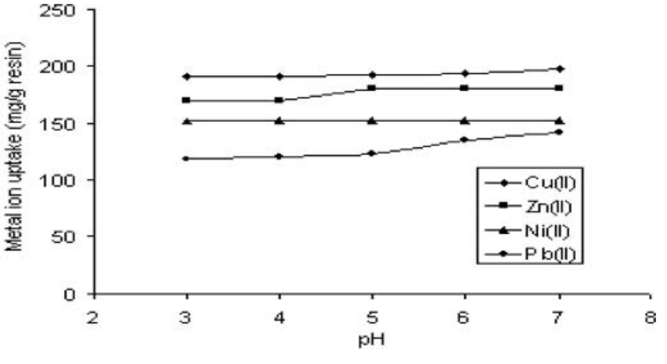


Figure 4. Effect of pH on metal-ion uptake by the resin

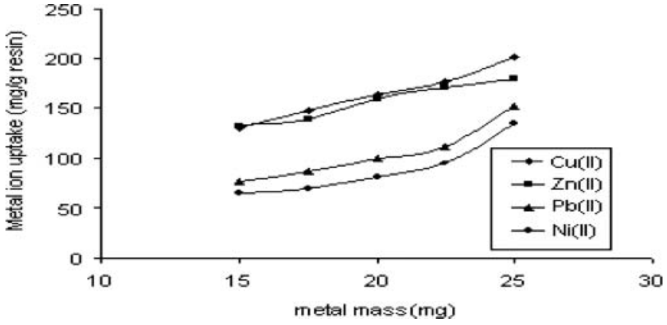


Figure 5. Effect of initial amount of metal ions on metal ion uptake by resin.

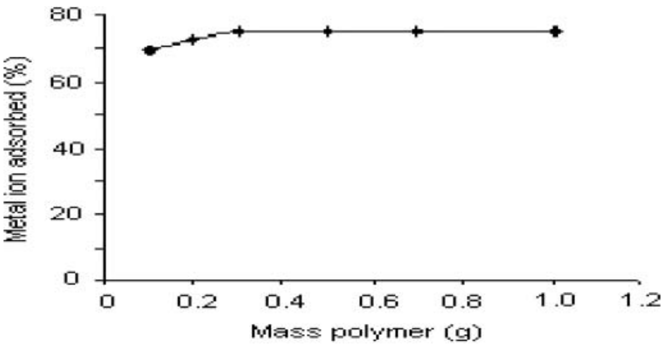


Figure 6. Effect of the polymer mass on the Cu(II) uptake.

ion was added at 25°C under continuous shaking for 24 h. Figure 6 shows the dependence of Cu(II) ion uptake on mass of resin expressed as % adsorption of metal ions vs. mass of polymer in mg. Results show that the amount of metal ions adsorbed on the resin slightly increases with the increase of the mass of resin used and reaches a steady state at about 0.3 g resin. The increase in metal-ion uptake may be explained by the increase of the polymer sites available for chelation when fixed amounts of metal ions are available in solution. In all cases, metal-ions are about 75% taken out of solution with the presence of 0.3–0.4 g of resin.

3.8 Effect of Temperature on the Metal Ions Uptake

The significance of the adsorption isotherms is that they show how the adsorbate molecules are distributed between the solution and the adsorbent at the equilibrium conditions and the effect of equilibrium concentration on the loading capacity at different temperatures. Listed in Table 1 are the Cu^{2+} uptake by the resin at different temperatures. Results show that for the same equilibrium concentration, the loading capacity of the resint from Cu^{+2} ions slightly increases as the temperature increases. For example, when the amount of added Cu^{+2} is 41.67 mg/50 mL, the loading capacity, of the resin is 20.94, 21.25, and 22.2 mg for the temperatures of 30, 40 and 50°C, respectively. The increase in loading capacity, however, is not significant which makes it rather difficult to draw conclusions about the kinetics of adsorption of heavy metals onto the resin. We strongly believe that the uptake of metal ions is mainly through chelation and complex formation rather than through adsorption.

Table 1. Effect of temperature on Cu^{2+} uptake by the resin

Concentration of Cu^{+2} (mg)	*Uptake at 30°C (in mg)*	*Uptake at 40°C (in mg)*	*Uptake at 50°C (in mg)*
4.16	2.031	2.188	2.343
8.33	4.297	4.375	4.531
16.66	7.8125	8.593	8.83
25	12.10	12.968	13.437
41.66	20.937	21.25	22.20

4 Conclusions

In this investigation, the chelation properties of the polystyrene-supported Schiff base, N,N′-disalicylidenepropylenetriamine, resin obtained through the reaction between the commercially available polymer, 4-chloromethyl polystyrene polymer, and the chemically synthesized Schiff base, N,N′-disalicylidenepropylenetriamine, toward some divalent metal ions in aqueous solution were studied with a batch equilibration method. The effect of exposure time on the metal-ion uptake was studied by a batch equilibrium technique and showed that a time of 5–10 h was enough to achieve maximum metal-ion sorption and that the extent of metal-ion uptake followed the order: $Cu^{+2} > Zn^{+2} > Ni^{+2} > Pb^{+2}$. The pH binding capacity profiles showed that the metal-ion uptake of the resin increased with increasing pH and reached a maximum at pH 6.0. The effect of resin mass, metal-ion concentration, and temperature on the extent of metal-ion uptake were also investigated.

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