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Halil Yirikoglu[a]; Mustafa Gülfen[a]
[a] Faculty of Arts & Sciences, Department of Chemistry, Sakarya University, Sakarya, Turkey

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Separation and Recovery of Silver(I) Ions from Base Metal Ions by Melamine-formaldehyde-thiourea (MFT) Chelating Resin

Halil Yirikoglu and Mustafa Gülfen

Faculty of Arts & Sciences, Department of Chemistry, Sakarya University, Sakarya, Turkey

Abstract: Melamine-formaldehyde-thiourea (MFT) chelating resin were prepared using melamine (2,4,6-triamino-1,3,5-triazine), formaldehyde, and thiourea and this resin has been used for separation and recovery of silver(I) ions from copper(II) and zinc(II) base metals and calcium(II) alkaline-earth metal in aqueous solution. The MFT chelating resin was characterized by elemental analysis and FT-IR spectra. The effect of pH, adsorption capacity, and equilibrium time by batch method and adsorption, elution, flow rate, column capacity, and recovery by column method were studied. The maximum uptake values of MFT resin were found as 60.05 mg Ag^+/g by batch method and 11.08 mg Ag^+/g, 0.052 mg Zn^{2+}/g, 0.083 mg Cu^{2+}/g and 0.020 mg Ca^{2+}/g by column method. It was seen that MFT resin showed higher uptake behavior for silver(I) ions than base and earth metals due to chelation.

Keywords: Silver(I) ions, melamine-formaldehyde-thiourea (MFT) resin, chelating resin, separation, recovery, adsorption, elution

INTRODUCTION

Silver, a heavy, noble, and precious metal has the highest thermal and electrical conductivities among all the metals. Silver, its compounds, and

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Address correspondence to Mustafa Gülfen, Faculty of Arts & Sciences, Department of Chemistry, Sakarya University, Esentepe Campus, TR-54140, Sakarya, Turkey. E-mail: mgulfen@sakarya.edu.tr

alloys have wide applications and usage in different areas; jewelry, catalyst, dentistry, electricity, medicine, photography etc. It was reported that 25% of the world's silver needs are supplied by recycling (1–3).

Chelating resins with different functionalities have potential applications in the selective removal of metal ions. They have many practical applications in the fields of wastewater treatment, chemical analysis, and environmental protection (4–7). The importance of these types of resins comes from their high selectivity, high uptake capacity, and possible modification of their physical and chemical properties to fulfill the application needed (4, 7, 8). Chelating or coordinating resins are polymers with covalently bound functional groups containing one or more donor atoms that are capable of forming complexes directly with metal ions. These polymers can also be used for a specific separation of one or more metal ions from solutions with different chemical environment (9).

In chelating resins, functional group atoms most frequently used are nitrogen (e.g. N presents in amines, azo groups, amides, nitriles), oxygen (e.g. O presents in carboxylic, hydroxyl, phenolic, ether, carbonyl, phosphoryl groups), and sulfur (e.g. S presents in thiols, thiocarbamates, thioethers). The nature of the functional group will give an idea of the selectivity of the ligand towards metal ions. In practice, inorganic cations may be divided into three groups: – group I-'hard' cations: these preferentially react via electrostatic interactions (due to a gain in entropy caused by changes in orientation of hydration water molecules); this group includes alkaline and alkaline-earth metals (Ca^{2+}, Mg^{2+}, Na^{+}) that form rather weak outer-sphere complexes with only hard oxygen ligands. – group II-"borderline" cations: these have an intermediate character; this group contains Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+} and Mn^{2+}, They possess affinity for both hard and soft ligands. – group III-"soft" cations: these tend to form covalent bonds. Hence, Cd^{2+}, Ag^{+} and Hg^{2+} possess strong affinity for intermediate (N) and soft (S) ligands (6, 10–12).

For soft metals, the following order of donor atom affinity is observed: $0 < N < S$. A reversed order is observed for hard cations. In general, the competition for a given ligand essentially involves Group I and Group II metals for O sites, and metals of Group II and Group III for N and S sites (6). A chelating sorbent or resin essentially consists of two components: the chelate forming functional group and the polymeric matrix or the support; the properties of both components determine the features (10).

In aqueous solutions, an effective ionic radii of Ag^{+} is 2.5 A^0. A lot of metals have bigger effective ionic radii in aqueous solutions (For examples, Cu^{2+}:6, Zn^{2+}:6 Mg^{2+}:8, Fe^{3+}:9, Al^{3+}:9, Zr^{4+}:11 A^0) (10). Silver(I) is a good soft metal, which forms stable complexes with the ligands including N and S donor atoms, in solutions or on surface of solids. The chelating resin containing sulfur and nitrogen as donor atoms have great attention due to their selectivity towards silver(I) ions as soft metal (1, 7, 14–16).

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Many researchers have studied the recovery, preconcentration, and the separation of silver ions as well as other metal ions in different samples. There are a number of reports of different types of chelating resins used in the separation, preconcentration or recovery of silver(I) ions and including functional groups; 2-mercaptobenzothiazole (14), thiol, purolite thiomethyl, spheron thiol (15), thiourea (9, 16), amino-mercapto (17), polythiazaalkane (4), thioureas, dithiocarbamate (18), bisthiourea (3), S- and N- containing resins (19).

MFT resin can be prepared easily in aqueous media. Although thiourea-formaldehyde (TF) resin has been studied as chelating resin, melamine-formaldehyde-thiourea (MFT) resin has not been used enough in the adsorption of precious metals. In the present work, MFT resin containing S- and N-donor atoms was prepared and characterized. The uptake and elution behaviors of the resin obtained towards to Ag^{+}, Cu^{2+}, Zn^{2+}, and Ca^{2+}, were investigated by batch and column methods, to separate and recover silver(I) ions from base and earth metal ions.

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EXPERIMENTAL

Melamine (2,4,6-triamino-1,3,5-triazine), formaldehyde, the thiourea and other chemicals were obtained from Merck. Stock solutions for Ag^{+}, Cu^{2+}, Zn^{2+}, and Ca^{2+} were prepared from $AgNO_3$, $Cu(CH_3COO)_2$, $Zn(NO_3)_2$ and $Ca(NO_3)_2$ respectively.

The initial molar ratio of melamine, formaldehyde (as solution), and thiourea was taken as 1:5:1 in a 500-mL capacity beaker. First, melamine and thiourea and then a formaldehyde solution were added to a reaction beaker. pH was increased with NaOH until the solid reactants dissolved at 80°C temperature. After dissolution of the solid reactants or oligomers of melamine-metylol-thiourea, the HCl solution was added as acid catalyst. Reaction mixture starts developing the melamine–formaldehyde-thiourea polycondensate. For polycondensation and release of water, heating above 80°C was carried out. The obtained resin was collected by filtration and washed with HCl solution and then with distilled water. It was dried at 105°C and ground for subsequent studies (20).

For the batch tests, a known amount of the resin beads was stirred with a solution containing metal ions for a certain period at room temperature. pH was adjusted with $NaCH_3COO/CH_3COOH$ buffer or HNO_3 solution. It was though that acetate buffer can compete with complexing groups (S and N groups) of the resin for copper and zinc. The amounts of metal ions adsorbed on to the resin were calculated from the analysis of the aqueous phase. For column studies, a column with 0.80 cm inner diameter was charged by the resin. A peristaltic pump was used to pump the feed solution or the elution solution down flow through the column. The effluent solutions were periodically collected and analyzed. Elution was carried out by 0.1 M thiourea and 0.1 M HNO_3 solution.

In the characterization of the resin, elemental analysis was performed by TUBİTAK, Marmara Research Center in Turkey, and IR spectra was recorded on a Mattson FT-IR 60R spectrophotometer in KBr disk. A Shimadzu Model 6700 flame atomic absorption spectrophotometer (FAAS) was used for the determination of the metal ions in aqueous solutions before and after adsorption or elution.

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RESULTS AND DISCUSSION

Characterization of the Resin Obtained

Elemental Analysis

The elemental analysis was carried out on the synthesized MFT resin in order to determine C, N, H, and S content. The obtained results are given in Table 1. The fact that the resin includes N and S donor atoms shows that the chelating resin formed. In addition the ligand concentration was calculated as 27.71 and 4.53 mmol/g for N and S respectively.

FT-IR Spectra

The obtained MFT resin was also characterized by FT-IR spectrophotometry. FT-IR spectrum of the resin is given in Fig. 1. The structural formula of the resin was confirmed from the FT-IR spectra. The peak around 2900 cm^{-1} shows the C-H stretching absorption of metylene. 1143 cm^{-1} was the peak of -N-C=S group. N-H stretching in secondary amines was seen at 3500–3300cm^{-1} area, (3, 13, 16, 21, 22). The confirmed structure of the MFT resin is given in Fig. 2.

Table 1. Elemental analysis of MFT resin

Elemental analysis	%	mmol/g
C	35.40	—
N	38.80	27.71
H	6.20	—
S	14.50	4.53

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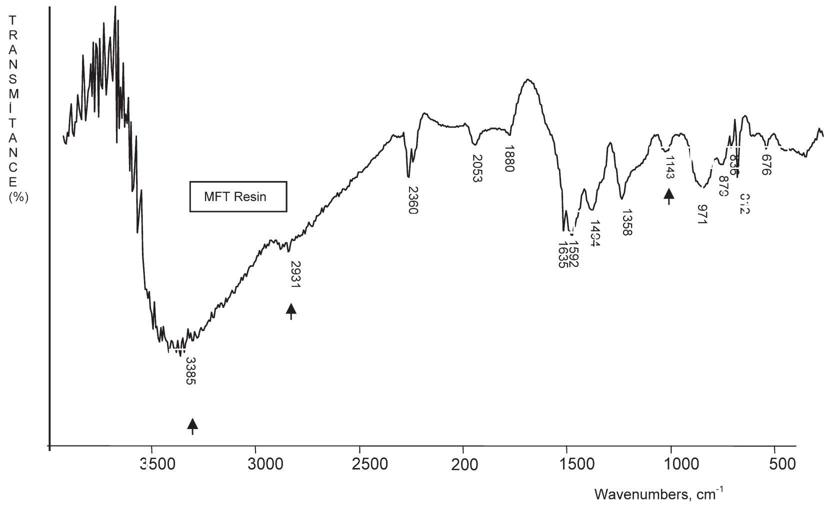

Figure 1. FT-IR spectra of MFT resin.

Metal Ions Uptake by Batch Method

Effect of pH on Silver Uptake

The investigation of the effect of pH on the uptake of silver(I) was performed at pH 1–8, by mixing 1 g of sorbent with 100 mL of 100 ppm silver solution. Since silver(I) begins to precipitate as silver hydroxide, it wasn't studied above pH 8. Initial pH's of solutions were adjusted with CH_3COOH, CH_3COONa, and NaOH. The acetate buffer may act as a complexing group with copper. The buffer can compete with complexing groups of sorbate resin. At the same time, copper acetate is used as reagent. Figure 3 shows the effect of pH on the silver uptake. In general, the sorption of silver increased by increasing pH (3, 22).

Silver(I) may interact with S or N donor atoms through chelation or ion exchange. Since it was found from the experimental data that high sorption

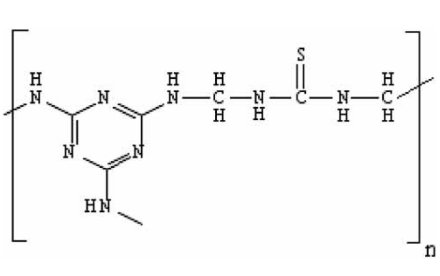

Figure 2. Melamine-formaldehyde-thiourea (MFT) resin.

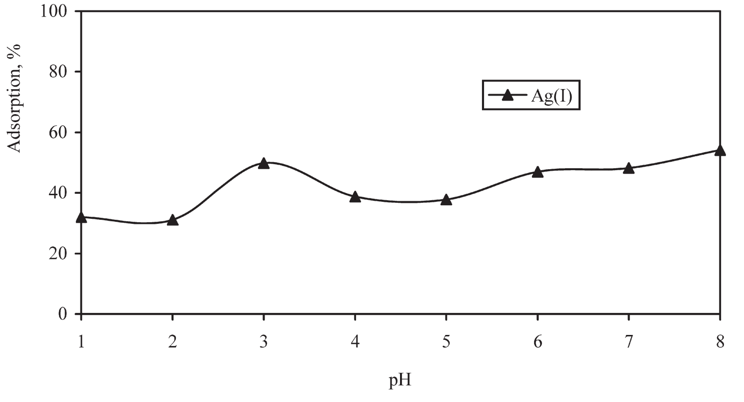

Figure 3. Effect of pH on silver(I) uptake (1 g MFT resin, 100 mL 100 ppm Ag^+ solution, 15 min contact time).

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occurred at high pH values, it may concluded that silver(I) interacts with S or N donor atoms through chelation. A chelation mechanism governs the silver sorption process.

$$RNH^+X^- + Ag^+ = RN \longrightarrow Ag^+X^- + H^+ \quad (1)$$

$$RC = S + Ag^+ = RC = S \longrightarrow Ag^+ \quad (2)$$

The optimum pH of 3 was selected. At this pH value, little more sorption efficiency for silver(I) was seen. Moreover most metal ions are soluble and they do not make the resin impure with their hydroxide precipitates or in other words, matrix metal ions can flow separately. So the subsequent studies carried out using silver solution adjusted to pH 3.

Silver(I) Adsorption Capacity of the Resin

Silver(I) adsorption capacity of the MFT resin was determined by batch method, putting 0.1 g resin in to 100 mL 100 ppm Ag^+ solution and stirring up to 120 min contact time. It was found (Fig. 4) that the resin had silver(I) adsorption capacity as 0.5567 mmol Ag^+/g resin or 60.05 mg Ag^+/g resin.

$$\text{Adsorption capacity} = \frac{Ag^+(\text{mmol}) \text{ on the resin}}{\text{Resin (g)}} \quad (3)$$

Selective Adsorption

The selective adsorption of silver(I) ions together with copper(II), zinc(II), and calcium(II) metal ions was investigated. The obtained results are given in Fig. 5.

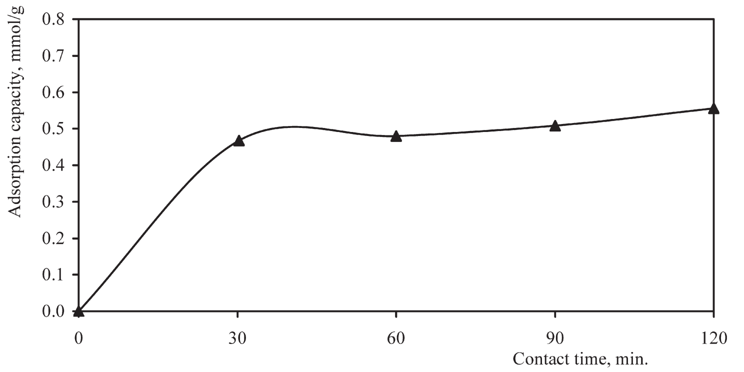

Figure 4. Silver(I) adsorption capacity of the MFT resin (0.1 g resin, 100 mL 100 ppm Ag^+ solution, pH = 3).

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It was found that a higher uptake for silver(I) ions than the other ions occurred at the same initial concentrations as 100 ppm. While copper(II), zinc(II), and calcium(II) ions adsorbed about 3–5% because of ionic interaction only, silver(I) ions adsorbed about 50–60% by batch method since silver(I) forms a chelate on the resin at same time.

In other words, using Eq. (4) (23) and the data in the selective adsorption studies, selectivity coefficients $\alpha_{Ag/Cu}$, $\alpha_{Ag/Zn}$ and $\alpha_{Ag/Ca}$ were calculated as 23.25, 25.40, and 26.54, respectively.

$$\alpha_{Ag/M} = \frac{(X_{Ag})^{ZM}(C_M)^{ZAg}}{(X_M)^{ZAg}(C_{Ag})^{ZM}} \tag{4}$$

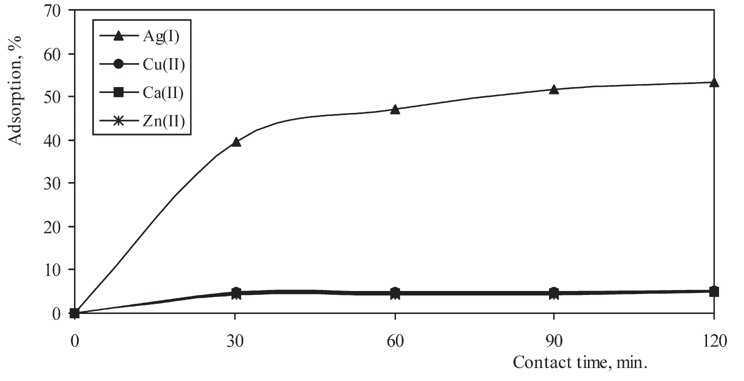

Figure 5. Selective adsorption of silver(I) ions (1 g resin, 100 mL 100 ppm initial Ag^+, Cu^{2+}, Zn^{2+}, and Ca^{2+} concentrations, pH = 3).

In Eq. (4), x and C represent the equilibrium sorption and equilibrium concentration of metal ions, respectively.

Metal Ions Uptake Using Column Method

Adsorption

In the column studies, firstly adsorption of the metal ions was examined. The obtained results are given in Fig. 6. As it can be seen from Fig. 6, it was found that the resin had very high uptake behavior for silver(I) ions, but hardly ever uptake the others.

Separation Factor

The separation factors for silver(I) ions over copper(II), Zinc(II) and calcium(II) ions were calculated from the column adsorption data using Eq. (5). The results are given in Table 2.

$$\text{Separation factor; } K_{A/B} = \frac{(C_{A1} - C_{A2})C_{B2}}{(C_{B1} - C_{B2})C_{A2}} \quad (5)$$

where,

C_{A1}; The concentration of A metal ion in feed
$C_{A2;}$ The concentration of A metal ion in effluent
$C_{B1;}$ The concentration of B metal ion feed
$C_{B2;}$ The concentration of B metal ion effluent

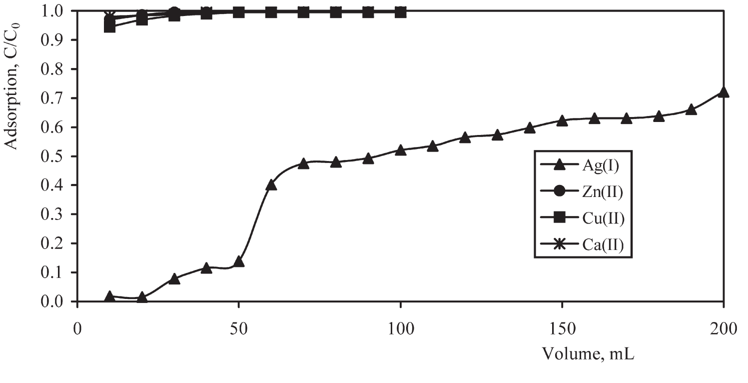

Figure 6. Adsorption by column method (1 g resin, 250 mL 100 ppm initial Ag^{+}, Cu^{2+}, Zn^{2+}, and Ca^{2+} concentrations (C_0), C: Final concentration, pH = 3, 10 mL/min flow rate).

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Table 2. Separation factor for silver(I) ions over copper(II) and zinc(II) ions

Volume mL	K(Ag/Zn)	K(Ag/Cu)	K(Ag/Ca)
0–10	1754	932	2529
10–20	4444	1972	3881
20–30	2045	678	965
30–40	1381	720	1519
40–50	1150	1194	1242
50–60	296	290	296
60–70	224	220	275
70–80	234	215	269
80–90	233	213	256
90–100	212	194	228

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Elution

Elution of silver(I) and the other ions, with 0.1 M thiourea solution acidified with 0.1 M HNO_3, from a column packed with the MFT resin was carried out at a flow rate of 10 mL/min. The curves for the elution are given in Fig. 7.

In the adsorption study, the solution having 100 ppm silver ions had been used. At the end of elution, it was seen that the first 10 mL-elution solution included 480 ppm Ag^+ concentration and below 5 ppm Cu^{2+}, Zn^{2+} and Ca^{2+} ions. In other words, the concentration of the silver ions increased from 100 to 480 ppm. Hence silver(I) ions can be concentrated and recovered selectively over base and earth metal ions.

In the elution, 130 mL effluent volume can be determined breakthrough point. Above 130 mL, Ag^+ concentration was below 5 ppm, which was five

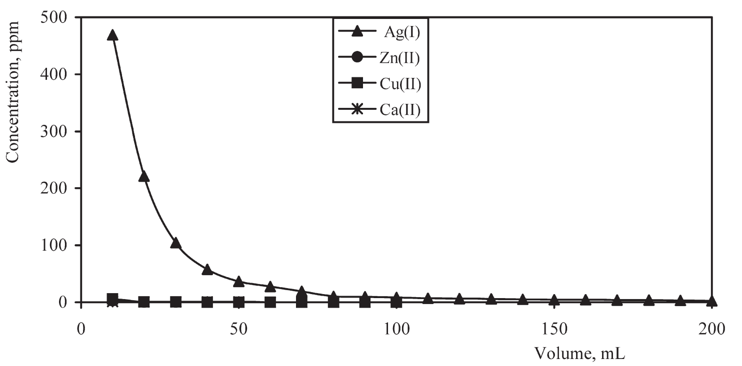

Figure 7. Elution of metal ions (0.1 M thiourea + 0.1 M HNO_3 elution solution, 10 mL/min flow rate).

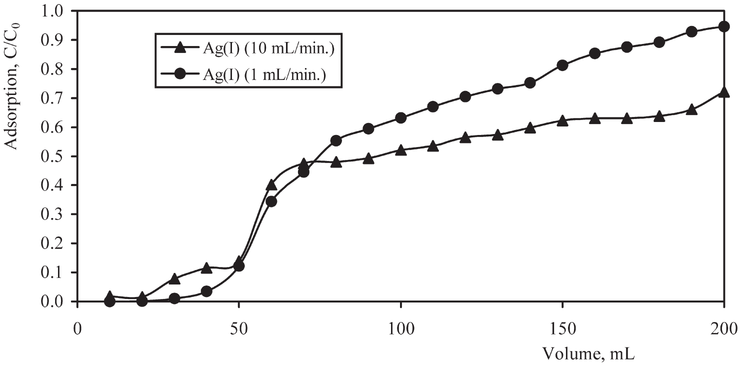

Figure 8. Effect of flow rate on the adsorption (1 g resin, 250 mL 100 ppm initial Ag^+ concentrations (C_0), C: Final concentration, pH = 3).

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percent of feed concentration. Breakthrough capacity was calculated as 9.8 mg Ag^+/g.

Effect of Flow Rate

The effect of flow rate on adsorption and elution of silver(I) ions (at flow rates 1 and 10 mL/min) were examined and the obtained results are given in Figs. 8 and 9. Similar adsorption efficiencies at 1 and 10 mL/min flow rates were obtained at different flow rates in general. A lower flow rate in the adsorption and a higher flow rate in the elution may be recommended especially. 100 ppm Ag^+ solution was adsorbed on the resin as nearly 100% up to 40 mL volume and then more concentrated silver(I) solution was eluted at high flow rate and less interval volumes.

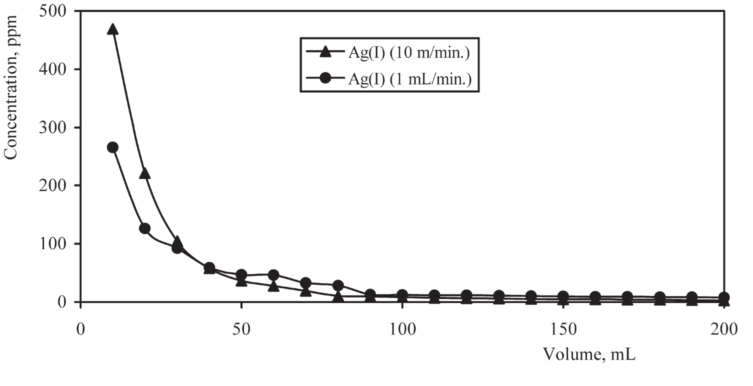

Figure 9. Effect of flow rate on the elution ions (0.1 M thiourea + 0.1 M HNO_3 elution solution).

Table 3. Column capacities for metal ions and silver recovery

	Adsorption		Elution		Recovery (%)
	(mmol/g)	(mg/g)	(mmol/g)	(mg/g)	
Ag^+ (10 mL/min)	0.1026	11.08	0.0936	10.11	91.20
Ag^+ (1 mL/min)	0.0842	9.10	0.0758	8.18	89.80
Zn^{2+} (1 mL/min)	0.0008	0.052	0.0004	0.026	—
Cu^{2+} (1 mL/min)	0.0013	0.083	0.0007	0.044	—

Column Capacity and Silver(I) Recovery

Column capacity for silver(I), copper(II), and zinc(II) ions and percent recovery of silver ions calculated from experimental data by column method. Total adsorbed and eluated metal ion quantities up to 200 mL effluent volume were calculated since all metal ions were not convenient for the breakthrough point. The results are given in Table 3. It can be seen from Table 3 that silver(I) was adsorbed and recovered highly according to the other ions. While a good separation of silver(I) from copper(II), and zinc(II) occurred, but lower adsorption capacity was calculated in column method studies due to less contact area of the resin with the solution.

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CONCLUSIONS

The separation and recovery of silver(I) ions over base and earth metal ions by melamine-formaldehyde-thiourea (MFT) chelating resin were investigated by the batch and column methods. The results have shown that the MFT chelating resin can be used to recover, separate, or concentrate silver(I) ions from base and earth metal ions. The resin has less uptake behavior for base and earth metal ions. The maximum uptake values of MFT resin were found as 60.05 mg Ag^+/g by batch method and 11.08 mg Ag^+/g, 0.083 mg Cu^{2+}/g, 0.052 mg Zn^{2+}/g and 0.020 mg Ca^{2+}/g by the column method. It was concluded that chelation governs the adsorption mechanism of silver(I) ions. In conclusion, the MFT chelating resin can be used for separation, recovery, or pre-concentration of silver(I) ions.

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