

Separation of Gold(III) Ions from Copper(II) and Zinc(II) Ions Using Thiourea–Formaldehyde or Urea–Formaldehyde Chelating Resins

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ABSTRACT: Thiourea–formaldehyde (TF) and urea–formaldehyde (UF) chelating resins were synthesized and these resins were used in the separation of gold(III) ions from copper(II) and zinc(II) base metal ions. In the experimental studies, the effect of acidity on gold(III) uptake and gold(III) adsorption capacities by batch method, and loading and elution profiles of gold(III) ions, gold(III), copper(II), and zinc(II), dynamic adsorption capacities and the stability tests of TF and UF resins by column method were examined. By batch method, the optimum acidities were found as pH 2 and 0.5M HCl, and gold(III) adsorption capacities in the solutions including copper(II) and zinc(II) ions were obtained as 0.088 and 0.151 meq Au(III)/g for UF and TF resins, respectively. On the other hand, by col-

umn method, the dynamic adsorption capacities were calculated as 0.109 meq Au(III)/g with TF, 0.023 meq Au(III)/g with UF, 0.015 meq Cu(II)/g with TF, 0.0057 meq Cu(II)/g with UF, and under 6.1×10^{-5} meq Zn(II)/g with TF or UF. TF resin was more effective in the separation and the concentration of gold(III) ions from copper(II) and zinc(II) ions than UF resin. It was seen that sulfur atoms contributed the gold(III) adsorption comparing with oxygen atoms. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 111: 2798–2805, 2009

Key words: thiourea–formaldehyde resin; urea–formaldehyde resin; gold(III) adsorption; gold(III) separation; chelating resin

INTRODUCTION

Recovery of gold as a precious metal attracted the attention of many researchers over many years. Gold is recovered from a wide variety of sources such as primary sources constituted by ores, and secondary sources usually more complex such as copper nickel sulfide leach residues, copper and silver anode slimes, as well as recycled scraps.¹

Recently, chelating resins with various functionalities have also been widely used for concentrating and retrieving of gold. A chelating resin or polymer essentially consists of two components: the chelate forming functional group and the polymeric matrix or the support.^{2,3} 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. In these 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).^{2–7}

The chelating resins with functional groups including sulfur and nitrogen donor atoms are very efficient at chelating with precious metals according to hard-soft acid-base (HSAB) theory by Pearson.⁸ Soft metal ions, for instance Au^{3+} , Ag^+ , and Pd^{2+} , show affinity to soft bases with donor atoms as $\text{O} < \text{N} < \text{S}$. On the other hand, hard metal ions, for instance Cu^{2+} , Fe^{3+} , and Al^{3+} , show affinity hard bases with donor atoms as $\text{O} > \text{N} > \text{S}$.^{2,3,8–10}

Moreover, amino resins as chelating resins have ionic interaction properties by protonated amines donor atoms as well as coordination. N and O atoms in a chelating resin can be protonated and then they can show ionic interaction with anionic metal ions such as AuCl_4^- . S is poor in protonation and it is effective in coordinating with soft metal ions.^{9–11}

A number of sulfur-containing chelating resins were synthesized and used widely in preconcentration, separation or purification, recovery of noble metals, as well as gold. Polymers with covalently bonded functional groups containing donor sulfur atoms, such as thiol,^{5,12} thiourea,^{6,7,10,13} bithiourea,⁴ dithizone,¹⁴ thiosemicarbazide,^{15,16} and other sulfides,^{5,9,17–22} have been applied for the selective sorption of noble metal or gold ions. In addition, there are also nitrogen-containing chelating resins, which are used in the separation or recovery of gold ions. Some example functional groups of these chelating

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resins are amines,^{5,23} aminoguanidyl,²⁴ quaternary ammonium,^{1,25,26} azole,^{19,27} nitrile, and hydroxylamine.²⁸

This study was undertaken to characterize the adsorption and separation of gold(III) ions from copper(II) and zinc(II) ions by batch and column methods, using urea–formaldehyde (UF) and thiourea–formaldehyde (TF) chelating resins. UF resin includes N and O donor atoms, whereas TF resin includes N and S atoms. In the batch adsorption studies, effect of acidity, gold(III) adsorption capacities of these resins were examined. Moreover, column method was used to investigate the loading, the elution and the separation of gold(III) from copper(II) and zinc(II) ions, and dynamic adsorption capacities and stability tests of the resins.

EXPERIMENTAL

Chemicals

The reagents used in the synthesis of UF and TF are formaldehyde (solution 37% w/w), urea, and thiourea supplied from Fluka, Switzerland and they were used without further purification. Gold atomic absorption standard solution (1000 mg/L) was purchased from Merck, Germany. Copper and zinc solutions were prepared from copper(II) nitrate and zinc(II) nitrate supplied from Merck, Germany. The other reagents were analytical grade.

Instruments

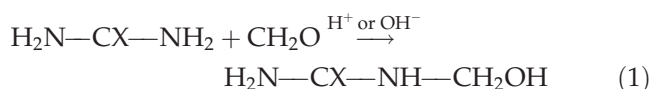
Elemental analyses of UF and TF resins were performed with the use of a LECO CHNS 932 elemental analyzer (Leco Co. USA) at Tubitak in Ankara, Turkey. The column studies were performed with a column of length 10 cm and inner diameter 0.8 cm. A constant flow rate during adsorption or elution in the column was provided with a peristaltic pump (Masterplex, Cole-Polmer Ins. Co., UK). Metal ion concentrations were analyzed by atomic absorption spectrophotometer (Shimadzu, AA-6701F, Japan). pH measurements were done with a Hanna pH-meter (pH211, Italy). IR spectra were recorded on a Shimadzu-IRPrestige-21 FTIR spectrophotometer

Syntheses of the chelating resins

UF and TF resins were synthesized by amine–formaldehyde reactions. These reactions include hydroxymethylation [eq. (1)] and condensation [eqs. (2)–(4)]. Formation of hydroxymethyl compounds occur in acidic or basic conditions at low temperature (50–100°C) [eq. (1)]. Condensation of hydroxymethyl compounds, to form methylene or dimethylene ether

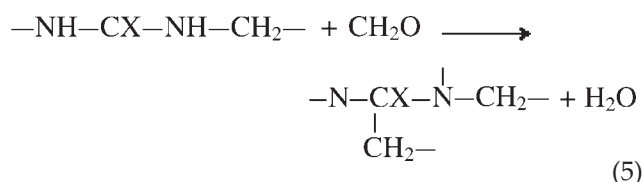
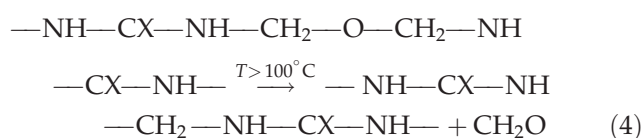
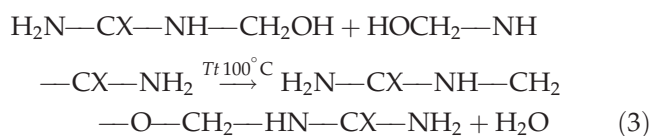
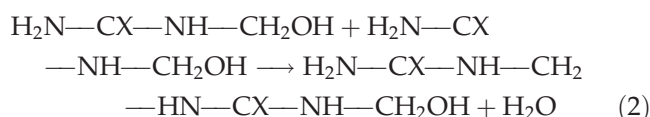
bridges, is faster in acidic media.^{10,11,29–34} TF or UF resin has very complex structure. When urea (or thiourea)/formaldehyde mole ratio is between 1 : 2 and 1 : 4, highly crosslinked UF or TF resin can be synthesized [eq. (5)].

Hydroxymethylation:



X: S (thiourea) or O (urea)

Condensation:



X: S (thiourea) or O (urea)

Both UF and TF resins were synthesized considering amine–formaldehyde reactions. Amounts of reactants taken were as follows: 60 g (1 mol) of urea or 76 g (1 mol) of thiourea and 96.67 g (1 mol CH₂O) of formaldehyde solution (of 37% aqueous). In this study, more linear or less crosslinked resin was the aim in the synthesis for the adsorption studies. Thiourea or urea was dissolved in 50 mL water in a beaker and then pH was adjusted to between 8 and 10 by adding 1M NaOH solution. Formaldehyde was added in to the basic urea solution. The mixture was heated for ~ 30 min at the temperature of 80°C. At this time, amine groups will form hydroxymethyl groups. The synthesis was continued by acid-catalyzed condensation by adding 1M HCl solution. When the mixture was acidified, TF or UF resin condensates began to precipitate. The obtained condensates were filtered, washed with acid and water, dried at 105°C, and they were ground to below particle size of 150 μm. These powder resins were used

TABLE I
Elemental Analyses of UF and TF Resins

Resin	C%	H%	N%	S%	O% (Calculated)
Experimental					
Urea-formaldehyde (UF)	30.34	5.90	32.35	–	31.41
Thiourea-formaldehyde (TF)	26.77	4.16	28.69	33.26	7.12
Theoretical					
Urea-formaldehyde (UF)					
$[-NH-CO-NH-CH_2-]_n$ or	33.33	5.56	38.89	–	22.22
$[-NH-CO-NH-CH_2-O-CH_2-]_n$	35.29	5.88	27.45	–	31.37
Thiourea-formaldehyde (TF)					
$[-NH-CS-NH-CH_2-]_n$ or	27.00	4.54	31.81	36.36	–
$[-NH-CS-NH-CH_2-O-CH_2-]_n$	30.50	5.08	23.72	27.11	13.55

in all experimental studies. They were characterized by elemental analysis.^{10,29–34}

Uptake measurements

Preparation of the solutions

The gold(III) solutions used in the experimental studies were prepared by diluting from AAS standard solution having 1000 mg/L concentration. The solutions of gold(III) or mixtures with copper(II) and zinc were adjusted to requested pH value with HCl or NaOH solution after enough chloride concentration (0.1M) was provided by HCl solution for $AuCl_4^-$ complex.

Batch method studies

Metal ions uptake tests by batch method were performed to determine the optimum initial acidity and gold(III) adsorption capacities of the resins. Effect of initial acidity was studied with 0.1 g TF or 1 g UF resin in 100 mL gold solution including 100 mg/L gold(III), copper(II), and zinc(II) ions at 0.5, 1, 3M HCl concentrations and pH 1–6. Gold(III) adsorption capacities were studied with 0.1 g resin in 100 mL solution at different concentrations of gold(III) ions in the competitive conditions with copper(II) and zinc(II). The concentrations of the metal ions in the samples collected before adsorption and at equilibrium time were determined by atomic absorption spectrophotometer. The adsorption values were calculated according to initial concentrations.

Column method studies

A glass column of 10 cm length and 0.8 cm inner diameter was packed with UF or TF resin (1.0 g each of both resins). The column bed was conditioned first with 0.1M KNO_3 for ionic effect exposure. The feed solutions including 200 mg/L gold(III), copper(II), and zinc(II) metal ions were passed through the column. The adsorption and the elution studies of the metal ions carried out at a constant flow rate

using a peristaltic pump. The metal ions loaded on to the resins were eluted by 0.5M thiourea and 0.5M HCl solution. Metal ion concentrations in each 10 mL solutions passed through from the column were analyzed by atomic absorption spectrophotometer.

RESULTS AND DISCUSSION

Characterization of UF and TF resins

Elemental analysis

The elemental analyses of UF and TF chelating resins are given in Table 1. It was thought that the resins may have been included $-CH_2-O-CH_2-$ polyether structure as an intermediate product. Moreover, theoretical constituents were calculated with and without $-CH_2-O-CH_2-$ polyether structure in Table 1. The elemental analyses confirm nearly the compositions of UF and TF resins.

FTIR spectra

The structures of UF and TF resins were confirmed by FTIR spectra. The spectra of the resins are given in Figure 1. The characteristic peaks of UF resin were assigned at 3329 cm^{-1} for N–H stretching, 1632 cm^{-1} for C=O stretching. In addition, the peak at 1136 cm^{-1} was seen for C–O–C stretching and the peak at 1387 cm^{-1} is related to C–H stretching of $CNHCH_2$. On the other hand, the peaks recorded with TF resin were at 1518 cm^{-1} for thiourea functional group N–C=S stretching and at 1072 cm^{-1} for C=S stretching. The peak at 1130 cm^{-1} for C–O–C stretching was also seen with TF resin. Additionally, the peaks at 1238 cm^{-1} for UF resin spectrum and at 1260 cm^{-1} in TF resin spectrum were assigned for C–H stretching of $>N-CH_2-N<$. These peaks show the crosslinking in the resins via N atoms. The degree of crosslinking is lower in TF resin because of peak intensities. The FTIR results emphasize the occurrence of UF and TF reactions. Moreover, C–O–C peaks in the both resins show

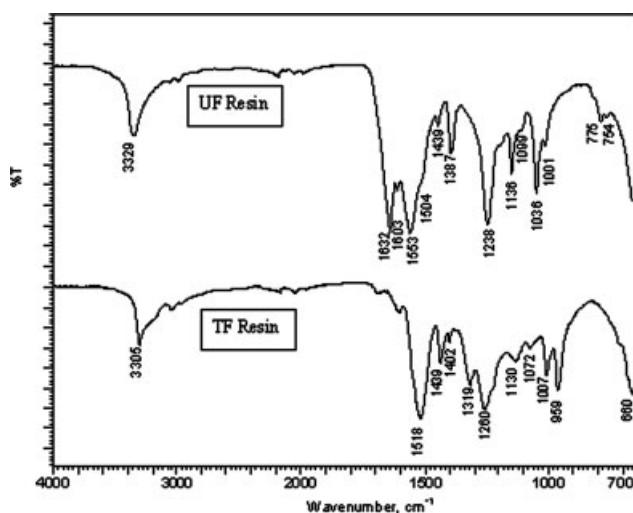


Figure 1 FTIR spectra of UF and TF resins.

polyether product.^{32,35–37} The suggested structures of the resins considering FTIR spectra and elemental analysis results were shown in Scheme 1.

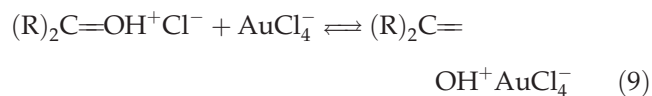
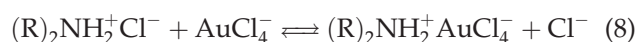
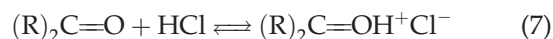
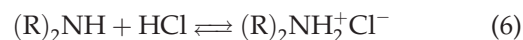
Batch method studies

Effect of acidity on the uptake

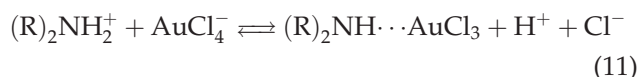
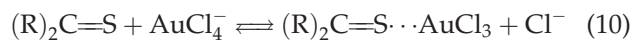
Gold(III) ions have different complexes in aqua solution according to chloride and acid concentrations. The coexistence of hydroxide, chloride, and hydroxide–chloride complexes makes gold speciation very complex. At low chloride concentrations (below $10^{-6}M$), gold is present in the solution in hydroxide forms: $Au(OH)_2^+$ (below pH 1), $Au(OH)_3$ (between pH 1 and 12), and $Au(OH)_4^-$ (above pH 12). For high chloride concentrations (above $0.01M$), $AuCl_4^-$ predominates in acidic solutions, whereas in alkaline solutions, both $Au(OH)_4^-$ and $Au(OH)_3$, $Au(OH)_3Cl^-$ coexist and/or predominate. In the near neutral region, $AuOHCl_3^-$ (pH 3.5–6.5) and $Au(OH)_2Cl_2^-$ (pH 4.5–8) exist. In acidic solutions containing enough chloride, gold forms anionic $AuCl_4^-$ complex. It can be adsorbed on to UF and TF resin via ionic interac-

tion [eqs. (8) and (9)], chelation [eqs. (10) and (11)], or both mechanisms.^{9,10,38}

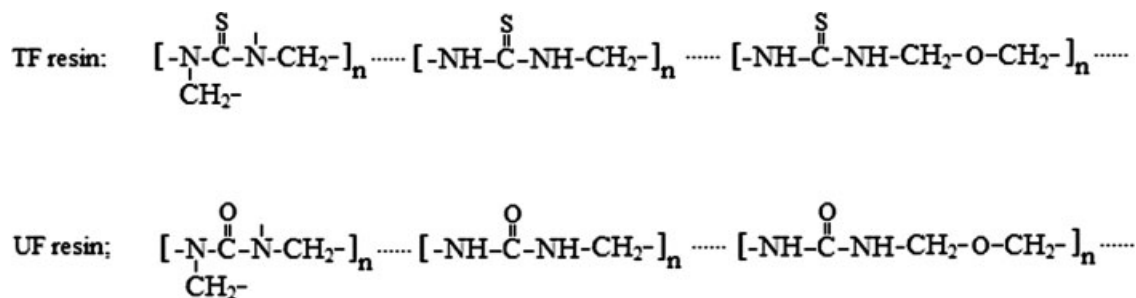
Ionic interaction;



Chelation;



The investigation of the effect of acidity on the uptake was performed by batch method, and the obtained results are presented in Figure 2 for TF and in Figure 3 for UF resin. In the uptake studies with TF resin, the best uptake was recorded at $0.5M$ HCl concentration. On the other hand, UF resin had the best adsorption values at pH 2. At $3M$ HCl concentration, the amounts of the adsorption with the both resin decreased. This is because of the strong interaction between the chloride and gold(III) ions to form chloro-complexes and competition of chloride ions with $AuCl_4^-$. At higher pH values, the adsorptions resulted in less amounts, because protonated nitrogen and oxygen were neutralized.³⁹ In general, similar adsorption values at different acid concentrations were found when UF or TF resin was used. However, acidity is indispensable in gold(III) adsorption by UF and TF resins because of ionic interaction. The initial acidities of the solutions used in the later experimental studies were adjusted to the determined optimum acid concentration of $0.5M$ HCl with TF and pH 2 with UF.



Scheme 1 The suggested structures of UF and TF resins.

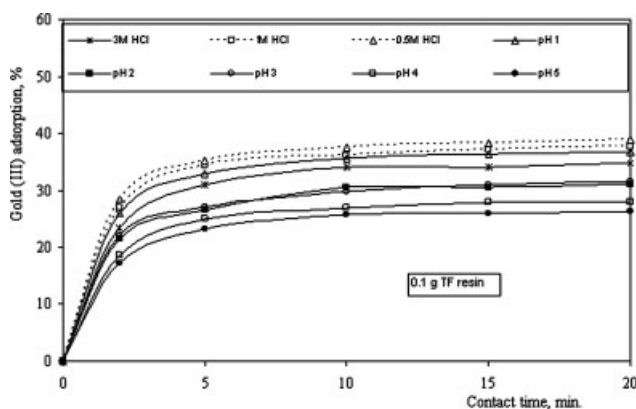


Figure 2 Effect of acidity on the gold(III) uptake by TF resin (100 mg/L Au(III), Cu(II), and Zn(II), 100 mL solution, 0.1 g TF resin).

Gold(III) adsorption capacities

Gold(III) adsorption capacities of UF and TF chelating resins were determined through Langmuir isotherm; [eq. (12)] where C_e is the concentration of gold(III) at equilibrium in the solution (mg/L); q_e is the amount of gold(III) adsorbed per unit weight of the adsorbent (mg/g); the constant Q is the adsorption capacity (mg/g); and b is related to the energy of adsorption (L/mg).^{5,17,18,20} The plots of the experimental q_e and C_e values as specific sorption (C_e/q_e) against the equilibrium concentration (C_e) for adsorption of gold(III) ions on TF and UF adsorbents are shown in Figure 4. The values of Q , b , and R^2 (regression coefficient) for gold(III) were calculated from the linear plot, C_e/q_e vs. C_e and they are represented in Table 2.

$$\frac{C_e}{q_e} = \frac{1}{bQ} + \frac{C_e}{Q} \quad (12)$$

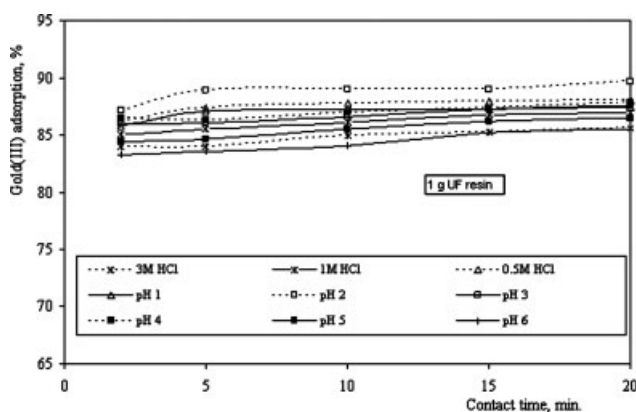


Figure 3 Effect of acidity on the gold(III) uptake by UF resin (100 mg/L Au(III), Cu(II), and Zn(II), 100 mL solution, 1 g UF resin).

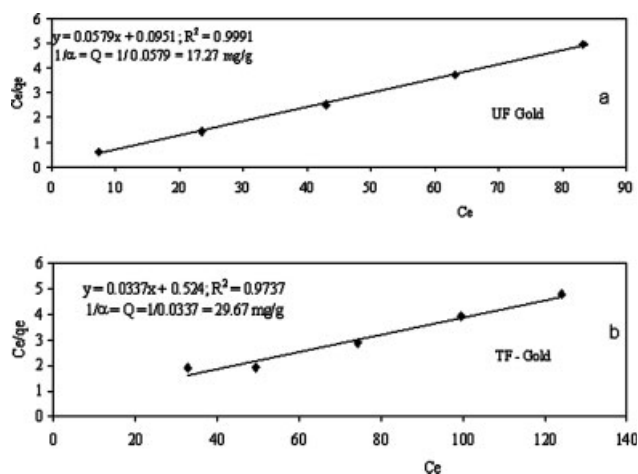


Figure 4 Langmuir isotherms: (a) urea-formaldehyde resin and (b) thiourea-formaldehyde resin.

Gold(III) adsorption capacities were calculated as 17.27 and 29.67 mg/g or 0.088 and 0.151 meq/g for UF and TF resins, respectively. It was found that TF resin had higher gold(III) adsorption capacity than UF resin. From the result, sulfur atoms in TF resin should be dominant for the higher gold(III) adsorption capacity.

Column studies

Loading of gold(III) ions

After the batch studies, column method was performed to examine the loading or the adsorption and the elution of gold(III), copper(II), and zinc(II) metal ions. During the loading step, the feed solution including 200 mg/L gold(III), copper(II), and zinc(II) ions was allowed to pass through the column packed with TF or UF resins. The obtained breakthrough curves of gold(III) ions were plotted as dimensionless concentration factor, C/C_0 (C is the concentration of the metal ions in the solution outcoming the column and C_0 is the concentration of the same metal ions in the feed solution), versus effluent volume. The gold(III) ions breakthrough curves are given in Figure 5. Moreover, the dynamic adsorption capacities (C/C_0 : 0.90) of the resins were calculated. It was found that TF resin had the dynamic adsorption capacity of 21.56 mg (0.109 meq) Au(III)/g, whereas UF resin was 4.57 mg

TABLE II
Gold(III) Adsorption Capacities of UF and TF Resins

	UF-Gold	TF-Gold
Q (mg/g)	17.27	29.67
Q (meq/g)	0.088	0.151
b (L/mg)	0.609	0.064
R^2	0.9991	0.9737

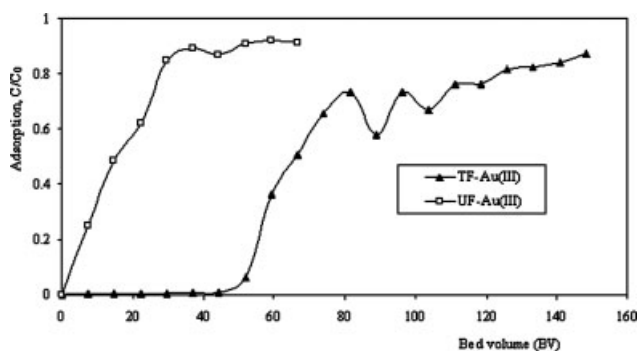


Figure 5 Breakthrough curves of gold adsorption (feed solution: 200 mL, 200 mg/L Au(III), Cu(II), and Zn(II), 1 g resin, flow rate: 0.74 BV/min).

(0.023 meq) Au(III)/g. It was seen that TF resin was more effective in the adsorption of gold(III) than UF resin.

Elution of gold(III) ions

The adsorbed gold(III) ions on to TF or UF resin were eluted using the solution including 0.5M thiourea and 0.5M HCl. Thiourea is a complexing agent and HCl is both protonating agent and competitive agent with chloride ions in the elution of gold(III) ions. The obtained elution profiles for TF and UF resins are given in Figure 6. It was seen that more concentrated gold(III) solution could be obtained when TF resin was used. It was found that TF resin was more convenient for the concentration of the gold(III) ions. UF resin was poor in the concentration of gold(III) ions.

Dynamic adsorption capacities

All batch and column studies were performed in competitive conditions, which included gold(III), copper(II), and zinc(II) metal ions. In first cycles tests, copper(II) and zinc(II) metal ions were also analyzed and the dynamic adsorption capacities (C_0

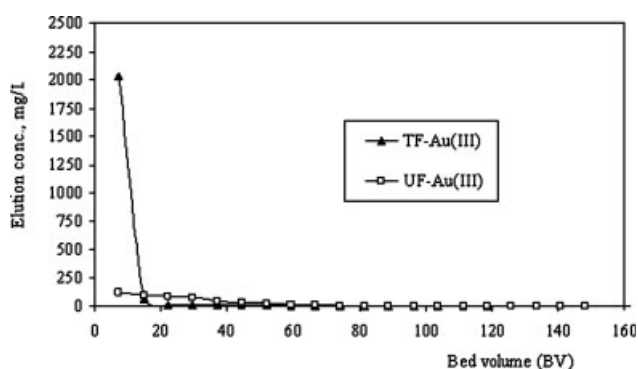


Figure 6 Elution of gold(III) (loaded resin: 1 g, elution solution: 0.5M thiourea and 0.5 HCl, flow rate: 0.74 BV/min).

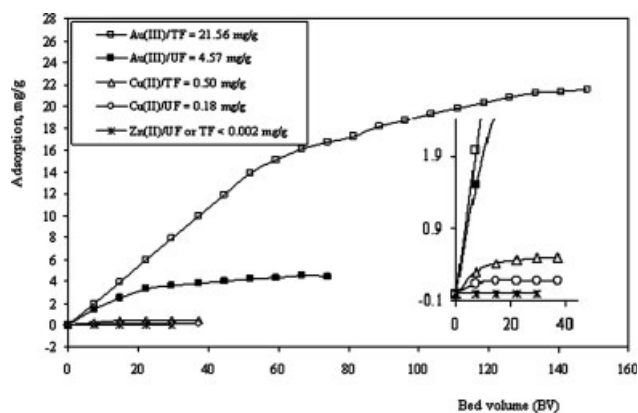


Figure 7 Dynamic adsorption capacities (feed solution: 200 mL, 200 mg/L Au(III), Cu(II), and Zn(II), 1 g resin, flow rate: 0.74 BV/min).

C_0 : 0.90) of TF and UF resins were examined (Figs. 7 and 8). The dynamic adsorption capacities were found as 21.56 mg Au(III)/g (0.109 meq Au(III)/g), 0.50 mg Cu(II)/g (0.015 meq Cu(II)/g), and under 0.002 mg Zn(II)/g (6.1×10^{-5} meq Zn(II)/g) with TF resin and 4.57 mg Au(III)/g (0.023 meq Au(III)/g), 0.18 mg Cu(II)/g (0.0057 meq Cu(II)/g), and under 0.002 mg Zn(II)/g (6.1×10^{-5} meq Zn(II)/g) with UF resin. If the results are compared, it can be seen from Figures 7 and 8 that the resins have high gold(III) and less copper(II) and zinc(II) dynamic adsorption capacities. Gold(III) ions are very competitive in the adsorption on TF resin.

On the other hand, gold(III) concentrations in the effluent solutions after first cycles were measured as 2030 and 121.8 mg/L, while the copper(II) concentrations were 30.3 and 12.4 mg/L for TF and UF resins, respectively, and the zinc(II) concentration was under 0.2 mg/L for TF or UF resin. If the metal ion concentrations of 200 mg/L before the loading are considered, TF resin is more effective in the concentration of gold(III) ions and the separation of gold(III) ions from copper(II) and zinc(II) ions.

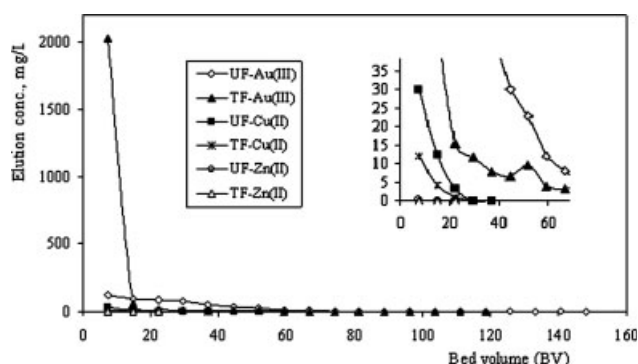


Figure 8 Elution of metal ions (feed solution: 0.5M thiourea and 0.5M HCl, 1 g resin, flow rate: 0.74 BV/min).

Stability tests

The stability tests of TF and UF resins were examined by measuring gold(III) ion concentrations after and before three or four cycles. The obtained experimental results are given in Figure 9 for TF resin and in Figure 10 for UF resin. The stability tests were performed up to the chromatographic elution volumes (C/C_0 : 0.50) of the resins for the metal ions. TF resin showed increasing chromatographic or breakthrough capacity by increasing cycles. On the other hand, UF resin had less chromatographic or breakthrough capacity in two cycles but third cycle is different from the others. Both resins continued to be conditioning by the elution. Two resins were used in similar experimental procedure to compare with each other. The elution solution containing 0.5 thiourea and 0.5M HCl contributed the conditioning of the resins. Also, thiourea, a good chelating ligand with gold(III) ion, was impregnated in to the UF resin.

CONCLUSIONS

In this study, TF and UF chelating resins were used in the separation and the concentration of gold(III) ions from copper(II) and zinc(II) metal ions. Some important results can be drawn as following.

Batch method studies showed that initial acidities of gold(III) solutions were 0.5M HCl and pH 2, and gold(III) adsorption capacities were 0.151 meq/g and 0.088 meq/g with TF and UF resins, respectively. By column method, it was found that the dynamic adsorption capacities were calculated as 0.109 meq Au(III)/g, 0.015 meq Cu(II)/g, and under 6.1×10^{-5} meq Zn(II)/g with TF resin, and 0.023 meq Au(III)/g, 0.0057 meq Cu(II)/g, and under 6.1×10^{-5} meq Zn(II)/g with UF resin in the competitive conditions. It was concluded that sulfur atoms

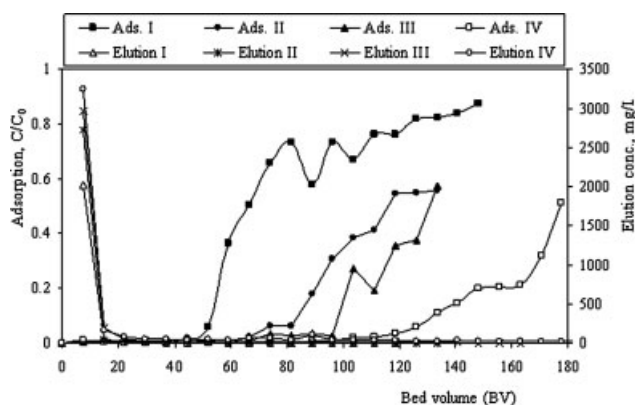


Figure 9 Stability test of TF resin (feed solution: 200 mL, 200 mg/L Au(III), Cu(II), and Zn(II), 1 g resin, flow rate: 0.74 BV/min, elution solution: 0.5M thiourea and 0.5M HCl).

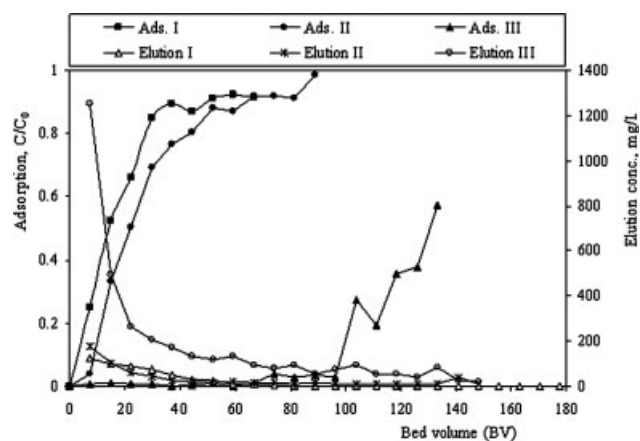


Figure 10 Stability test of UF resin (feed solution: 200 mL, 200 mg/L Au(III), Cu(II), and Zn(II), 1 g resin, flow rate: 0.74 BV/min, elution solution: 0.5M thiourea and 0.5M HCl).

in TF resin contributed the gold(III) adsorption comparing with oxygen atoms in UF resin. TF chelating resin is more effective in the separation and the concentration of gold(III) ions from copper(II) and zinc(II) ions than UF resin.

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