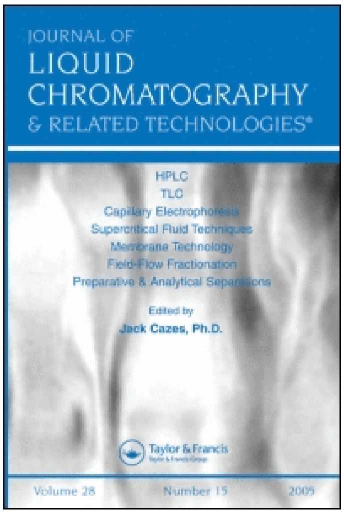

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Selective Separation and Determination of Copper and Gold in Gold Alloy Using Ion Exchange Polyurethane Foam

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Selective Separation and Determination of Copper and Gold in Gold Alloy Using Ion Exchange Polyurethane Foam

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Abstract: A sensitive and selective determination of copper and gold in gold alloys was achieved using a strong anion exchange polyurethane foam (PUFIX) and polyurethane foam after hydrolysis (white-PUF). The total sorption capacity of the resin was 4.22 and 0.85 mmol g^{-1} for copper and gold, respectively. The maximum sorption of Cu (II) and Au (III) were in 0.02–0.2 and 0.06 M HCl, respectively. The half-life values of sorption ($t_{1/2}$) was 2.2 and 0.5 min for Cu (II) and Au (III), respectively. The thermodynamic parameters were also determined. The red color formed on the surface of the white-PUF is used for quantitative and/or semiquantitative determination of trace amounts of copper and gold ions spectrophotometrically or visually, respectively. As low as 4.8 ng cm^{-2} of copper and gold is easily detected and Beer's law is obeyed in the concentration range of 0.025–0.21 μg mL^{-1} with molar absorbtivity of 3.5×10^4 L mol^{-1} cm^{-1}. PUFIX and white-PUF was verified as a good sorbent by Cu (II) and Au (III) determination in gold alloy.

Keywords: Copper (II), Gold (III), Ion exchanger, Polyurethane foam, Determination

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INTRODUCTION

Developing highly sensitive and selective methods for copper and gold determination is of interest due to the economic value of the gold alloys used in the production of medicinal and scientific devices and gold jewellery. Several methods have been described for the determination of Cu (II) and Au (III).[1–6]

The simplest and most cost effective technique for the trace and ultratrace determination of these metal ions is separation and preconcentration using solid phase extraction. Many types of adsorbents, e.g., Amberlite XAD-16,[7] Dowex 1-X8,[8] silica gel,[9] divinylbenzene homopolymer,[10] and polyurethane foam[11–15] have been developed for the recovery of copper and gold from aqueous media.

Polyurethane foam (PUF) is a quite useful new class of solid, polymeric sorbents for batch and/or flow solid-liquid systems for the separation and preconcentration of inorganic and organic species.[11,16,17] Some advantages in the use of PUF include very low cost, simple preparation, high available surface area, and cellular structure and stability in acidic and basic media.[11] Considerable attention has been directed in recent years to the use of PUF after special treatment (grafted or functionalized) with a suitable reagent for simple and rapid separation and preconcentration processes.[18–21] Also, PUF functionalized with a tertiary amine and quaternary ammonium group (PUFIX) was prepared by coupling of ethyl iodide with the primary amine of the PUF matrix; it is applied for the determination of palladium in environmental samples.[22]

In the present work, PUFIX, as a new sorbent, is used for the separation, preconcentration, and determination of copper (II) and gold (III), and comparison with the white-PUF sorbent. The sorption behavior of tested metal ions onto the PUFIX has been studied to optimize the best conditions for determination in gold alloys. Data have been obtained for kinetic, thermodynamic, and equilibrium situations. Finally, the red color rapidly produced in the white-PUF membranes is directly proportional to the amount of Cu (II) and Au (III) present, and based on the solid phase spectrophotometrically determination of these metal ions. The main advantage of the proposed method over the other methods is the low detection limit, high selectivity, and short analysis time.

EXPERIMENTAL

Reagents and Materials

All reagents were prepared from analytical reagent grade chemicals and bidistilled water. A stock solution containing 1 mg mL^{-1} of copper ions was prepared by dissolving 0.3928 g of $CuSO_4 \cdot 5H_2O$ in water containing 1 mL of concentrated H_2SO_4 and dilution of the solution to 100 mL in a measuring flask. A 1 mg/mL stock solution of Au (III) was prepared by dissolving 0.1 g of pure gold in 5 mL of aqua regia and dilution of the solution to 100 mL with doubly distilled water. A 4 M of thiocyanate solution was prepared by dissolving 97 g of KSCN in distilled water and diluting to 250 mL with distilled water. Commercial thin film of white-PUF sheets (Saudi Chemical Ind. Co. Ltd, SCIC) was used. White-PUF was cut into a thin film (25 mm × 8 mm × 2 mm). A portion of 10 g of PUF was soaked in 100 mL of 2 mol/L HCl with stirring 24 h, washing with distilled water, followed by acetone and allowed to dry at room temperature. The earlier published procedure[20] was synthesized and characterized as PUFIX.

Apparatus

All spectrophotometric measurements were performed using a spectro UV-Vis RS Digital spectrophotometer (Labomed, Inc). The pH measurements were carried out using a pH meter from Microprocessor pH Meter (HANNA Instruments). Glass columns of about 25 cm long and 1.5 cm in diameter were employed in the chromatographic separation experiments.

General Procedures

In the batch technique, a 0.2 g portion of the PUFIX or white-PUF is mixed with a 25 mL aliquot solution containing 1 $\mu g\ mL^{-1}$ of metal ions and 1 M of KSCN in a shaker thermostated to the desired temperature and adjusted to a desired shaking speed. After fixed intervals, the aliquots of solution were withdrawn and the concentration of Cu (II) or Au (III) ions was determined spectrophotometrically.[22]

In the solid phase spectrophotometric determination of metal ions, the white-PUF parallelepiped after shaking 30 minutes was separated from the sample solution and washed twice with water. For quantitative experiments, the absorbance of the foam was measured at appropriate wavelengths against a blank of the foam in ethanol. The net absorbance of the colored species in the foam is used to construct a calibration graph for metal ions. This method gives a considerable improvement in the sensitivity and precision of the PUF thin-layer method[23] by eliminating the attenuation effects caused by the foam matrix and reagents. Semiquantitative comparison with foam cubes was employed using a procedure described previously in a test tube.[23]

RESULTS AND DISCUSSION

Optimum Condition for Separation and Determination of Copper and Gold

The effect of pH and acidity on the separation and determination of anionic complexes, $[Cu\ (SCN)_4]^{2-}$ and $[Au\ (SCN)_4]^{-}$ onto white-PUF and PUFIX was examined using a batch mode. The distribution coefficient for the sorption of Cu (II) and Au (III) onto the solid-phase sorbents was plotted against the HCl concentration as shown in Figure 1. The maximum value of distribution coefficient (K_d) for the sorption of copper (II) thiocyanate complex onto white-PUF and PUFIX sorbents was 1.3; the maximum values of K_d for gold (III) thiocyanate sorbed onto white-PUF and PUFIX sorbents was 0.9 and 1.2, respectively. The uptake of metal ion complexes was decreased with increasing pH values (1.7–12) and HCl concentration (0.3–1 M). The similarity of the curves of the K_d for the sorption of $[Cu\ (SCN)_4]^{2-}$ onto white-PUF and PUFIX could reasonably suggest that the sorption of copper (II) thiocyanate complex onto white-PUF and PUFIX has the same sorption mechanism. This confirms that the primary amine salt group of white-PUF, forms ion pairs with Cu (II) thiocyanate complex. The distribution coefficients sequence was in the order of PUFIX > white-PUF, which indicates that the PUFIX sorbent is more efficient than white-PUF. The K_d value for sorption of $[Cu\ (SCN)_4]^{2-} > [Au\ (SCN)_4]^{-}$ onto PUFIX, confirms that the strong anionic exchange process depends on the size of metal ions and the charge of anionic complexes.[21] The value of K_d for sorption of $[Cu\ (SCN)_4]^{2-} \leq [Au\ (SCN)_4]^{-}$ onto white-PUF, due to the sorption of metal ions onto white-PUF, may proceed via both mechanism, weak anion exchange, and ion association mechanism.[19] Finally, the value of separation factor (α) of the tested metal ions was calculated ($\alpha = K_{d(Cu)}/K_{d(Au)}$), which show that the selectivity sequence is in the order Cu (II) > Au (III) at pH range 1.3 ($\alpha = 2.63$).

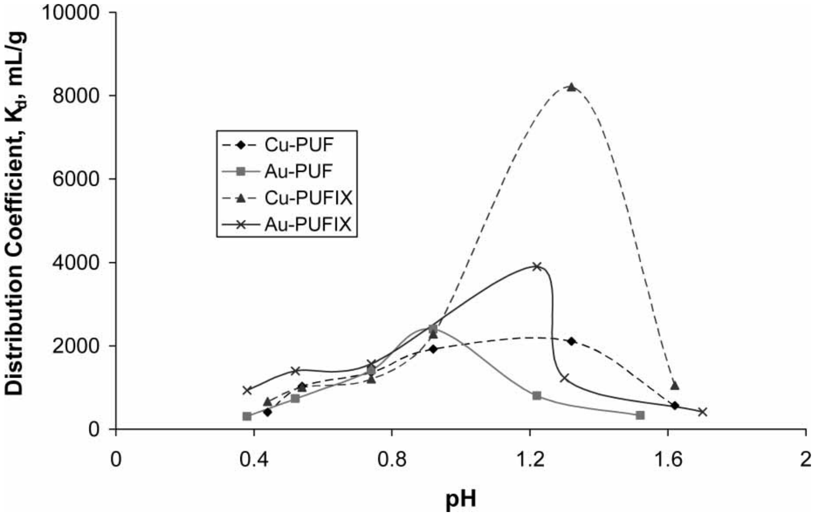

Figure 1. Effect of pH on the distribution coefficient of the sorption of copper (II) and gold (III) ions onto white-PUF and PUFIX.

The effect of shaking time on the sorption Cu (II) and Au (III) onto white-PUF and PUFIX was studied. From the results obtained, the time required for sorption equilibrium was found to be 5–20 minutes. The kinetic data for the sorption of copper and gold ions was calculated according to first order reaction equation: $\log (Q_e - Q_t) = \log Q_e - k_1 t/2.303$ where k_1 is the rate constant of the sorption. Also, the half time ($t_{1/2}$) required for sorption of Cu (II) and Au (III) onto PUFIX was calculated ($t_{1/2} = 0.693/k_1$) and found to be 2.2 and 0.5 min, respectively. The values of $t_{1/2}$ for the sorption of copper and gold ions onto white-PUF were 9.12 and 1.11 min, respectively (Table 1). These results show that the sequence of sorption rate of the metal ions onto the sorbents was in order of gold (III) > copper (II) and the PUFIX is more efficient than white-PUF sorbent.

The effect of thiocyanate concentration on the sorption of copper (II) and gold (III) ions onto white-PUF and PUFIX sorbents was examined at a concentration range from 0.1 up to 3.0 mol L^{-1} KSCN. From the results obtained, the optimum thiocyanate concentration for the maximum uptake of metal ions was 0.8–1.5 mol L^{-1}.

The effect of temperature on the sorption of Cu (II) and Au (III) ions onto white-PUF and PUFIX was studied in the range of 25–80°C. The results obtained show that the sorption of tested metal ions was better at low temperature than at high temperature. Ln K was plotted against 1/T (Figure 2); the plot is linear over the entire range of temperature investigated. The thermodynamic parameters of the metal ions sorbed onto white-PUF and PUFIX were estimated using the equations: (Table 2).

$$\ln K_C = -(\Delta H/RT) + (\Delta S/R) \quad \text{and} \quad \Delta G = \Delta H - T\Delta S$$

The negative values of ΔG indicate that the sorption process is energetically favorable. Similarly, the negative values of ΔH are interpreted as the exothermic chemisorptions process. Negative values of entropy may be indicative of the faster sorption of tested metal ions onto PUFs. The values of ΔH and ΔS of Cu (II) sorbed onto PUFs >Au (III) are due to the size of metal ions.

Table 1. Kinetic parameters for the sorption and desorption of copper (II) and gold (III) onto PUFIX and white-PUF

Sorbent	Metal ions	Rate constant of sorption k_1 (min^{-1})	Rate constant of desorption k_{-1} (min^{-1})	Overall rate constant $\grave{k}$ (min^{-1})	Half life of sorption $t_{1/2}$ (min)
PUFIX	Cu (II)	0.315	0.013	0.328	2.20
	Au (III)	1.337	0.062	1.399	0.52
White-PUF	Cu (II)	0.076	0.001	0.077	9.12
	Au (III)	0.624	0.022	0.646	1.11

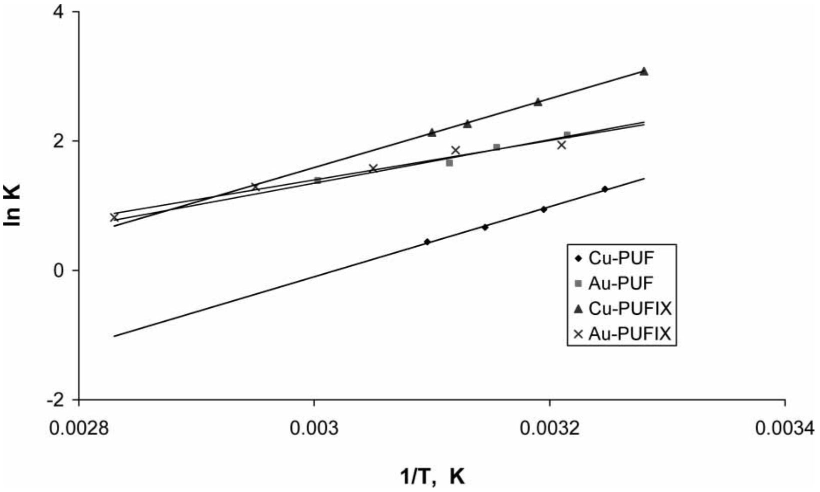

Figure 2. Effect of temperature on the sorption of copper (II) and gold (III) ions onto white-PUF and PUFIX.

The effect of the sample volume (10–100 mL) to the weight of PUF (0.2 g) on the distribution coefficient (K_d) of copper (II) and gold (III) ions was studied using a batch technique. The data indicate that the maximum sorption of Cu (II) and Au (III) in batch factor (V/m) ranges from 100 to 150. The K_d values of the metal ions sorbed was decreased with the increasing of the sample volume (from 8200 to 1400) in batch factor, $V/m \approx 200$.

The distribution coefficient values of metal ions sorbed onto PUFs was determined as a function of salt concentration (0.02–0.1 M) of NaCl, KCl and NH_4Cl. The results obtained show that the K_d value was slightly increased with increasing of sodium and potassium salt concentration, while the K_d value was decreased with increasing of the ammonium concentration. This result indicates that the ammonium salts can be used in the stripping of

Table 2. Thermodynamic parameters of the sorption copper (II) and gold (III) onto PUFIX and white-PUF

Sorbent	Metal ions	Free energy ΔG (kJ mol^{-1})	Enthalpy ΔH (kJ mol^{-1})	Entropy ΔS (J K^{-1} mol)
PUFIX	Cu(II)	−8.61	44.12	119.15
	Au (III)	−6.12	25.26	64.18
White-PUF	Cu(II)	−4.51	44.95	135.70
	Au (III)	−6.47	27.93	72.61

Cu (II) and Au (III) from the white-PUF or PUFIX matrix to form [M $(NH_3)_4]^{n+}$.

Absorption Spectrum and Calibration Curve

A simple and sensitive spectrophotometric method for the microdetermination of copper (II) gold (III) has been developed. This method is based on the sorption of Cu (II) and Au (III) thiocyanate complexes onto white-PUF. The red color formed on the surface of the foam material is used for quantitative and/or semiquantitative determination of submicrogram amounts of gold (III) spectrophotometrically or visually, respectively. White-PUF gave better color reaction results than PUFIX, because the color of white-PUF was changed from white to red (while the colour of PUFIX was changed from orange to red). The UV/Vis spectrum of white-PUF after adsorbing the copper (II) and gold (III) thiocyanate complexes was compared with that of $[Cu(SCN)_4]^{2-}$ and $[Au(SCN)_4]^{-}$ in aqueous solution. From the results obtained, the two peaks appear in the white-PUF after adsorbing the metal ionic thiocyanate complexes (Figure 3). The suitable wavelength for spectrophotometric measurements for the absorption spectra of the white-PUF after absorbing $[Cu(SCN)_4]^{2-}$ and $[Au(SCN)_4]^{-}$ are 410 and 440 nm, respectively (Figure 3). The method obeyed Beer's law in the concentration range 0.03–0.1 and 0.02–0.32 μg mL^{-1} for Cu (II) and

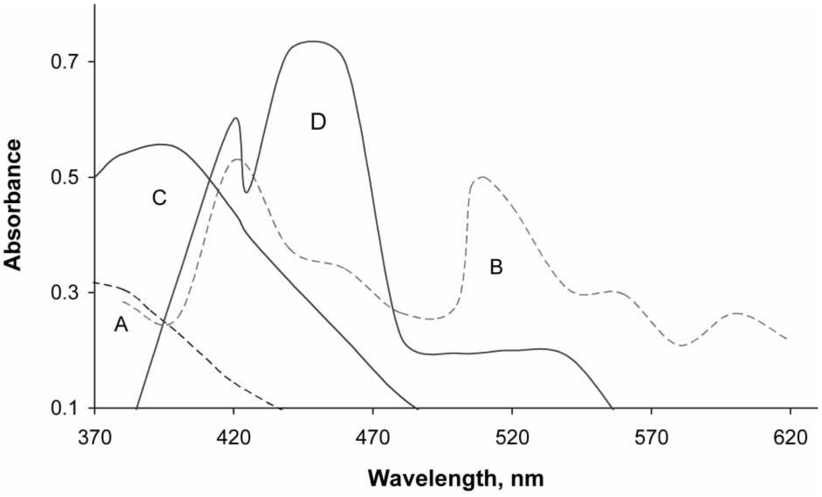

Figure 3. Spectra of (A) $[Cu(SCN)_4]^{2-}$ in aqueous solution, (B) white-PUF after sorbed copper (II) thiocyanate complex, (C) $[Au(SCN)_4]^{-}$ in aqueous solution and (D) white-PUF after sorbed gold (III) thiocyanate complex.

Au (III), respectively. The values of molar absorbtivity (ε) are 1.3×10^{-4} and 5.1×10^{4} L mol^{-1} cm^{-1} for Cu (II) and Au (III), respectively. The sensitivity sequence in the order, Au (III) > Cu (II) which has an average value of the lower detection limit of Sandell's sensitivity is 4.8 ng cm^{-2}, $S = 0.001/a$ where "a" is specific absorbtivity (Table 3). The results obtained show that the white-PUF method is more sensitive than the direct method for the determination of copper and gold by using thiocyanate solution as reagent (the values of ε are 1.1×10^{3} and 4.7×10^{3} L mol^{-1} cm^{-1} for Cu (II) and Au (III), respectively). Finally, from the result obtained, the proposed method is one of the simple and rapid spectrophotometric methods for the determination of copper and gold compared to other spectrophotometric methods.[1]

The absorbance of the PUF was measured after immersing the PUF parallelepiped in different media (water, benzene, ethanol, and diethyl ether) in a spectrophotometer cell. A good linear relationship between the absorbance of metal in the white-PUF phase and gold in dilute aqueous solution is observed. Considerable improvement in the absorbance values were noticed in diethyl ether compared with the other media (correlation factor, $r = 0.999$). The sensitivity sequence was in order of, water > ethanol > diethyl ether > benzene. The low relative standard deviation values (RSD $\approx$ 5.85 %, n =5) for the analysis of five sample replicates of the Au (III) indicates a good precision and accuracy of the proposed method. Finally, the color of the PUF after absorbance of $[Au(SCN)_4]^-$ was stable over 120 minutes while the intensity of the color of $[Au(SCN)_4]^-$ in aqueous solution was decreased after 5 minutes ~11.8%.

As low as 1.1 and 0.1 μg mL^{-1} of copper and gold were detected after shaking one cube of the white-PUF with 2.5 mL of the acidic solution of Cu (II) and Au (III) thiocyanate complexes in test tube. The color intensity on the white-PUF was found to be proportional to the concentration of metal ions in aqueous solution. The metal ions were determined semiquantitatively by a visual comparison for the color of the white-PUF cube with a standard scale of copper and gold prepared under the same experimental conditions. The results obtained indicate that the sensitivity sequence was in order, Au > Cu (Table 3).

Table 3. The detection limit (Sandell's sensitivity, S and semiquantitative determination, Se), maximum wavelength (λ_{max}) and molar absorbtivity (ε) for spectrophotometric measurements for Cu (II) and Au (III) using white-PUF

Metal ions	Sensitivity, LDL S (ng cm^{-2})	Se (μg mL^{-1})	λ_{max} (nm)	ε (L mol^{-1} cm^{-1})
Cu (II)	5.63	1.1	410	1.3×10^{4}
Au (III)	3.86	0.1	440	5.1×10^{4}

The effect of interfering ions for the detection of 20 μg of tested metal ions in the presence of 1 mg of each Na^+, K^+, Mg^{2+}, Ca^{2+}, Pb^{2+}, Al^{3+}, Ni^{2+}, Fe^{2+}, Fe^{3+}, Ag^+, SO_4^{2-}, Cl^-, and NO_3^- was examined using batch technique. The results obtained are successful using the straight forward method. In the presence of Ni^{2+} and Ag^+ cause an interference which reduces the color intensity. Fe^{2+} and Fe^{3+} cause an interference, which increases the color intensity and bathochromic shift. The masking, agent, e.g., tartaric acid, citrate, or phosphate ions were used to eliminate their interferences and to obtain the selective and sensitive detection of Cu (II) and Au (III).

ANALYTICAL APPLICATION

For assaying the gold metal in gold alloys (Gold grad 18), the copper content (Cu/Au∼1:3) was separated and determined. A set solution (25 mL) containing 1 g of gold alloys was passed through the column containing 1.5 g of PUFIX at a flow rate of 2–3 mL min^{-1}. Au (III) and Cu (II) were eluted using 1 M NH_4OH at flow rate of 2–3 mL min^{-1} (Figure 4). It is evident from Figure 4, that a resolution value $[R_S = 2(V_{R-Cu} - V_{R-Au})/(W_{Cu} + W_{Au})]$ is 3.06, which gives complete separation of gold from copper. This selectively technique allows the determination of each metal ion from the other ions in mixture.

For assaying the gold metal in gold alloys (Gold grad 21), the copper content (Cu/Au∼1:7) was separated and determined. A set solution

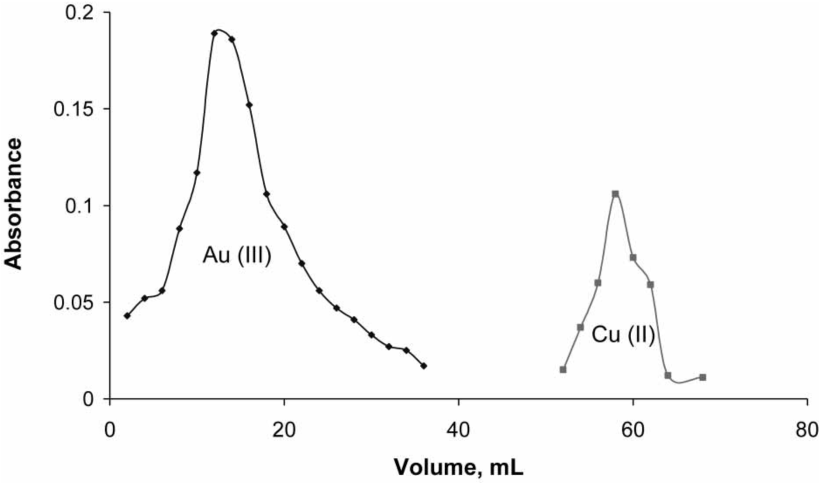

Figure 4. Separation of copper from gold alloy (Gold grade 18) using PUFIX column with 1 M NH_4OH solution.

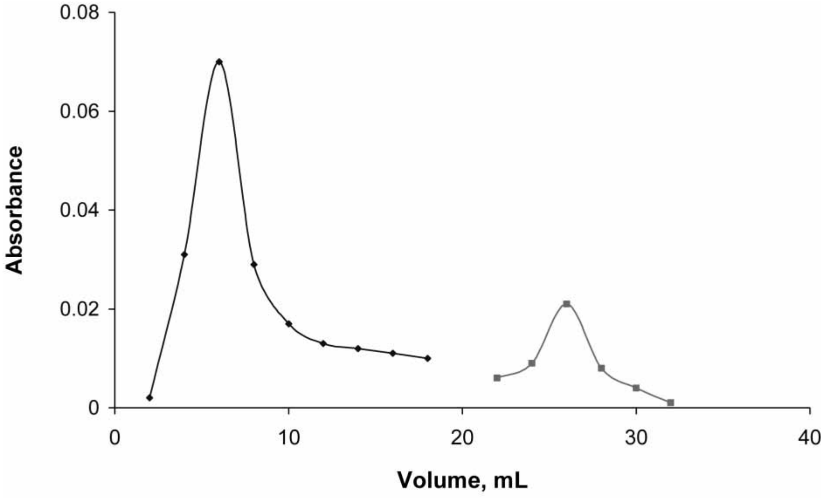

Figure 5. Separation of copper from gold alloy (Gold grade 21) using white-PUF column with 1 M NH_4OH and 1 M NaOH solutions.

(25 mL) containing 1 mg of gold alloys was passed through the column containing 1.0 g of white-PUF at a flow rate of 2–3 mL min^{-1}. Au (III) was eluted using 1 M NaOH then, copper was eluted using 1 M NH_4OH at flow rate of 4 mL min^{-1} (Figure 5). It is evident from Figure 5, that a resolution value is 2.24, which gives complete separation of gold from copper.

CONCLUSION

The present work deals with the separation and preconcentration of copper (II) and gold (III) metal ions from thiocyanate media using strong anion exchange polyurethane foam (PUFIX) and white polyurethane foam (white-PUF). The factors effecting sorption condition, e.g., pH, shaking time, thiocyanate concentration, sample volume, and temperature were studied. From our study, we conclude that PUFIX is more efficient than the white-PUF for the separation and preconcentration of copper and gold metal ions. Also, white-PUF has the ability to determine Cu (II) and Au (III) for assaying the gold metal as solid-phase spectrophotometry.

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