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Adsorption of Copper from an Ammonia-Thiosulfate Media Using DOWEX 550A Ion Exchange Resin

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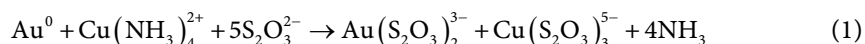Email: *cristian.vargas@usach.cl

Abstract

The study of copper adsorption onto ion exchange resins of anionic type is part of the gold recovery from ammonia-thiosulfate solutions, where copper is the main impurity of the system because it acts as a catalyst of gold dissolution reaction. A study is made of the adsorption and desorption of copper in the form of the $Cu(S_2O_3)_2^{3-}$ complex in an ammonia-thiosulfate media on an ion exchange resin, DOWEX 550A, classified as a strong base, which in its inner structure has a quaternary amine functional group. In the studied pH range copper adsorption increased with increasing pH, while the presence of thiosulfate decreased it, the same as the ammonia content, due to the greater presence of cuprotetramine, $Cu(NH_3)_4^{2+}$. Elution of the copper complexes from the resin was more efficient with sulfite than with perchlorate.
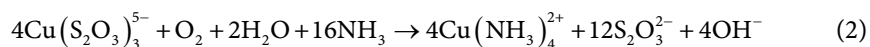
Keywords

Adsorption, Desorption, Copper, Ion Exchange Resin, Ammonia-Thiosulfate

1. Introduction

The study of copper adsorption on anion exchange resins has to do with the recovery of gold as $Au(S_2O_3)_2^{3-}$ present in the solutions produced by leaching gold ores in an ammonia-thiosulfate media, where copper is the main impurity in the system. The ammonia-thiosulfate system with copper offers high gold leaching rates compared to the cyanide system, because the reaction is catalyzed by cupric ions [1]-[23]. The main reaction in the gold leaching in a thiosulfate-ammonia media is the following:

$$Au^0 + Cu(NH_3)_4^{2+} + 5S_2O_3^{2-} \rightarrow Au(S_2O_3)_2^{3-} + Cu(S_2O_3)_3^{5-} + 4NH_3 \qquad (1)$$

With the oxidation reactions of Cu^+ to Cu^{2+} through the following equilibrium:

$$4Cu(S_2O_3)_3^{5-} + O_2 + 2H_2O + 16NH_3 \rightarrow 4Cu(NH_3)_4^{2+} + 12S_2O_3^{2-} + 4OH^- \qquad (2)$$

The solutions resulting from gold leaching contain gold-thiosulfate, copper-ammonia and copper-thiosulfate complexes, plus tetrathionate in different proportions as a function of the concentrations of copper, ammonia, thiosulfate, and pH.

Recovery of the gold dissolved in the rich leaching solution can take place by activated carbon, solvent extraction, ion exchange resins, or cementation with zinc powder [24]-[31].

The application of the ion exchange resins technique is very appropriate for solutions with low content of the element of interest, because it is a process that allows concentrating solutions very selectively. The ion ex-change is a reversible chemical reaction that takes place when an ion of a solution or a dissolved complex is exchanged for another ion (or ions) of equal value that is joined to the stationary solid particle.

When anion exchange resins are used, the adsorbed complexes will be both the aurothiosulfate anions, as well as the cuprothiosulfates, plus the trithionate, tetrathionate and other higher polythionates produced by the oxidation of the thiosulfate [32]. For that reason, it becomes necessary to know the behavior of the copper complexes separately in these kinds of resins in a thiosulfate-ammonia media, because copper will always be present in the gold leaching solutions.

The adsorption of copper on ion exchange resins in thiosulfate ammonia medium has not been studied extensively. Dreisinger and Zhang determined that with strong base anion exchange resins there is a high gold load on the resin, with a high adsorption rate due to the alkaline character of the solutions coming from the leaching [33] [34]. Most information is aimed at finding the complexes formed in the copper(I/II)-thiosulfate-ammonia system by means of speciation diagrams [35].

The speciation diagrams for the system of interest allow a better understanding of the phenomena that take place, and that is how Senanayake and Muir showed the effect of pH on the distribution of the involved species. For the copper(II)-thiosulfate-ammonia system they found that the predominant species at pH lower than 7.5 is $Cu(S_2O_3)_2^{3-}$, with 0.1 M concentrations of thiosulfate and ammonia; on the contrary, at higher ammonia concentrations and higher pH, but less than 12, the predominant species is $Cu(NH_3)_4^{2+}$.

The objective of this work was to study experimentally by means of discontinuous tests the effect of different variables such as pH, thiosulfate, ammonia and copper concentrations in the adsorption of copper complexes in the $Cu(S_2O_3)_2^{3-}$ form on the DOWEX 550A ion exchange resin. The effect of the sulfite and perchlorate ions on the desorption of gold from the resin was also studied.

2. Experimental

2.1. Materials

The ion exchange resin used was DOWEX 550A (Dow Chemical Company), classified

as a type 1 strong base. In its internal structure it has a quaternary amine functional group, and its outer appearance is as gel-type uniform particles with an average size of 590 ± 50 μm.

For the preparation of the copper solution and the eluting solutions double distilled and deionized water was used and analytical grade reagents, such as copper(II) sulfate pentahydrate [$CuSO_4 \cdot 5H_2O$], sodium thiosulfate [$Na_2S_2O_3 \cdot 5H_2O$], ammonium hydroxide [NH_4OH], sodium hydroxide [$NaOH$], sodium perchlorate [$NaClO_4 \cdot 2H_2O$], and sodium sulfite [Na_2SO_3]. All reagents were purchased from Sigma-Aldrich.

2.2. Experimental Techniques

The adsorption tests were carried out in 500-mL reactors with mechanical stirring, keeping constant the following experimental parameters: contact time: 3 h, temperature: 298 K, aqueous solution volume: 400 mL, solution/resin ratio: 833.3 mL/g.

In this stage the percentage of copper adsorbed and the resin's copper load (g Cu/kg resin) as a function of time were measured for the different experimental conditions. The studied variables and their experimentation ranges are presented in Table 1.

The experimental method used in each experiment was the same, varying only the specific conditions for each case. The following stages were used:

- The aqueous solution was added to the reactor, and the temperature and the pH were adjusted.
- The resin was added to the solution and the system was stirred mechanically in order to work with a perfect mixture.
- Samples of 3 mL were removed occasionally from the aqueous phase for the chemical analysis.
- The pH and the temperature of the aqueous solution were measured continuously.
- After 3 h the stirring was stopped, the resin was filtered from the aqueous solution, and it was washed for its later chemical analysis by atomic absorption, and the copper load on the resin was determined from the copper balance present in the solutions.

For the elution tests, the resin was loaded and the desorption experiments were performed in 500-mL reactors with mechanical stirring, keeping the following experimental conditions elutant: Cu load on the resin: 17.4 g Cu/kg resin, pH: 11, temperature: 298 K, contact time: 3 h, solution/resin ratio: 200 mL/g, volume of aqueous solution: 300 mL.

Table 1. Variables and experimental ranges in the adsorption stage.

Variable	Experimental range
pH	9 - 11
Thiosulfate concentration	0.1 - 0.5 M
Ammonia concentration	0.1 - 0.5 M
Copper concentration	50 - 200 ppm

In this stage the copper concentration in the rich solution was determined, and the percentage of desorbed copper as a function of time for the different experimental conditions was determined. The studied variables and their experimental ranges are shown in Table 2.

In this case the experimental method was the following:

- The copper bearing resin was loaded in agreement with the experimental conditions studied in the previous stage.
- The aqueous eluting solution was added and the temperature and the pH were adjusted to the required values.
- The loaded resin was added and the system was stirred mechanically.
- Samples of 3 mL were removed occasionally from the aqueous phase for the chemical analysis.
- The pH and the temperature of the aqueous solution were measured continuously.
- After 3 h the stirring was stopped, the resin was filtered from the aqueous solution, and it was washed.
- The same as in the previous stage, the analyses were performed by atomic absorption.

3. Results and Discussion

3.1. Adsorption Stage

3.1.1. Effect of pH

Figure 1 shows the results of the effect of pH on the copper adsorption on the Dowex 550A resin. In general, the adsorption did not exceed 10% at best, and the general shape of these curves indicates that initially the copper is adsorbed at a fast rate in all cases until a maximum value is reached, and then there is a small drop in the adsorption that can be attributed to a partial desorption of the copper.

The amount of adsorbed copper indicates that the process improves at pH 11 in relation to pH 9. The greater adsorption at pH 11 can be attributed to the greater stability of the thiosulfate at that pH, generating less tetrathionate ($S_2O_6^{2-}$) which acts as a competitor of the $Cu(S_2O_3)_2^{3-}$ complex in the adsorption process. The reason for the drops of these curves (due to partial desorption) can be attributed to a critical concentration of tetrathionate that causes this situation, and this agrees with the fact that at pH 9 partial desorption takes place in shorter solution-resin contact times. The chemical equilibrium that accounts for this situation can be represented by the following equation.
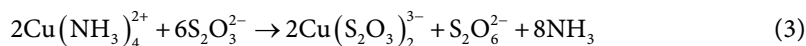
$$2Cu(NH_3)_4^{2+} + 6S_2O_3^{2-} \rightarrow 2Cu(S_2O_3)_2^{3-} + S_2O_6^{2-} + 8NH_3 \qquad (3)$$

Table 2. Variables and experimental ranges in the elution stage.

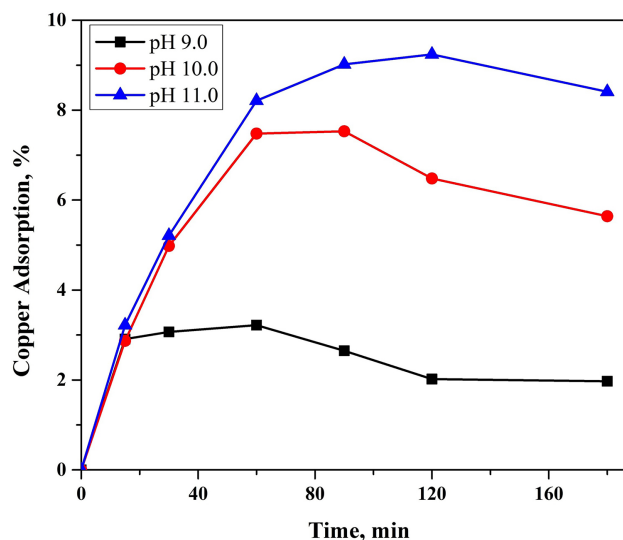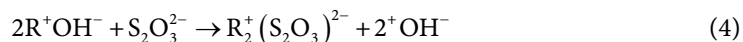| Variable	Experimental range
Sodium sulfite concentration	0.1 - 0.5 M
Sodium perchlorate concentration	0.1 - 0.5 M

Figure 1. Effect of pH on copper adsorption. Conditions: 200 ppm Cu; 0.1 M ammonia; 0.1 M thiosulfate.

3.1.2. Effect of Thiosulfate Concentration

Figure 2 shows the results of the effect of thiosulfate on the copper adsorption and load, and it is seen that as the concentration of thiosulfate increases the percentage of adsorbed copper decreases.

When the concentration of thiosulfate is between 0.3 and 0.5 M, 2% to 3% of Cu is adsorbed, achieving a copper load on the resin of 3 to 4 g Cu/kg resin. But working with a 0.1 M thiosulfate concentration the adsorption rises to 8%, with a load of approximately 17 g Cu/kg resin. This situation is explained because the thiosulfate would act as a competitor of copper for the resin's active sites. According to reaction (3), a lower concentration of thiosulfate generates less tetrathionate, which, as already mentioned, competes with the copper complex in the adsorption process.

Figure 3 shows the adsorption of copper at different thiosulfate concentrations and different contact times. It is seen that for 0.5 M thiosulfate there is no variation in the percentage of adsorbed copper for the different solution-resin contact times, but for a 0.1 M initial thiosulfate concentration the amount of adsorbed copper is a function of time. This behavior can be attributed to the coadsorption of the anionic thiosulfate complexes on the resin. The stoichiometry that represents the adsorption of thiosulfate can be described by the following equation:

$$2R^+OH^- + S_2O_3^{2-} \rightarrow R_2^+ \left(S_2O_3\right)^{2-} + 2^+OH^- \tag{4}$$

3.1.3. Effect of Ammonia Concentration

Figure 4 presents the results of copper load on the Dowex 550A resin as the ammonia concentration is varied. It is seen that as the concentration of dissolved ammonia increases, the copper adsorption and load on the resin decrease. This may be due to the fact that as the ammonia concentration increases, the stability of the $Cu\left(NH_3\right)_4^{2+}$ complex in relation to the formation of the $Cu\left(S_2O_3\right)_2^{3-}$ complex increases.

Speciation diagrams were made of the Cu-NH$_3$-S$_2$O$_3^{2-}$ system under the experimental temperature and electrochemical potential conditions. **Figure 5** shows that as the amount of ammonia in the solution increases, the stability of the copper-thiosulfate complexes decreases, the presence of the copper-ammonia complex becoming more important, so that at an NH$_3$ concentration of 500 mM the copper-thiosulfate complexes disappear.

At higher ammonia concentrations the following situations occur:

- The formation of cuprotetramine, $Cu(NH_3)_4^{2+}$, becomes more important
- There will be a greater amount of free thiosulfate, which may cause a greater production of tetrathionate, causing a decrease of the adsorption of the $Cu(S_2O_3)_2^{3-}$ complex, as shown in **Figure 5**.
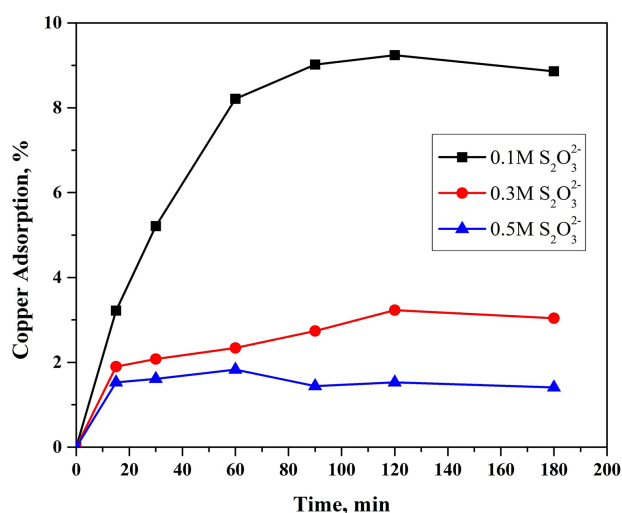

Figure 2. Effect of thiosulfate on copper adsorption. Conditions: 200 ppm Cu; 0.1 M ammonia; pH 11.
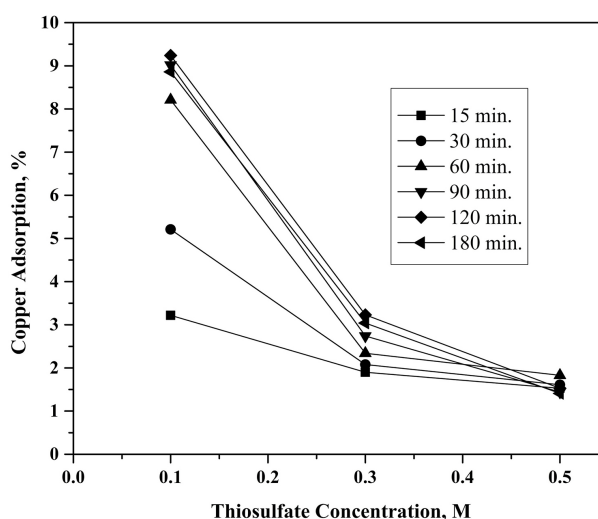

Figure 3. Variation of copper adsorption with thiosulfate concentration. Conditions: 200 ppm Cu; 0.1 M ammonia; pH 11.
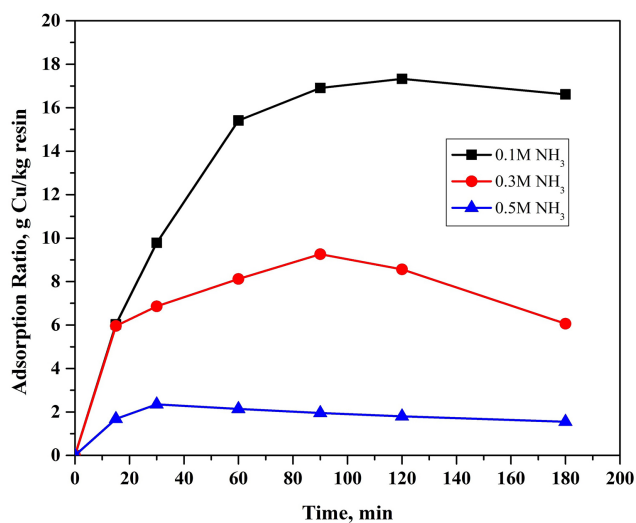
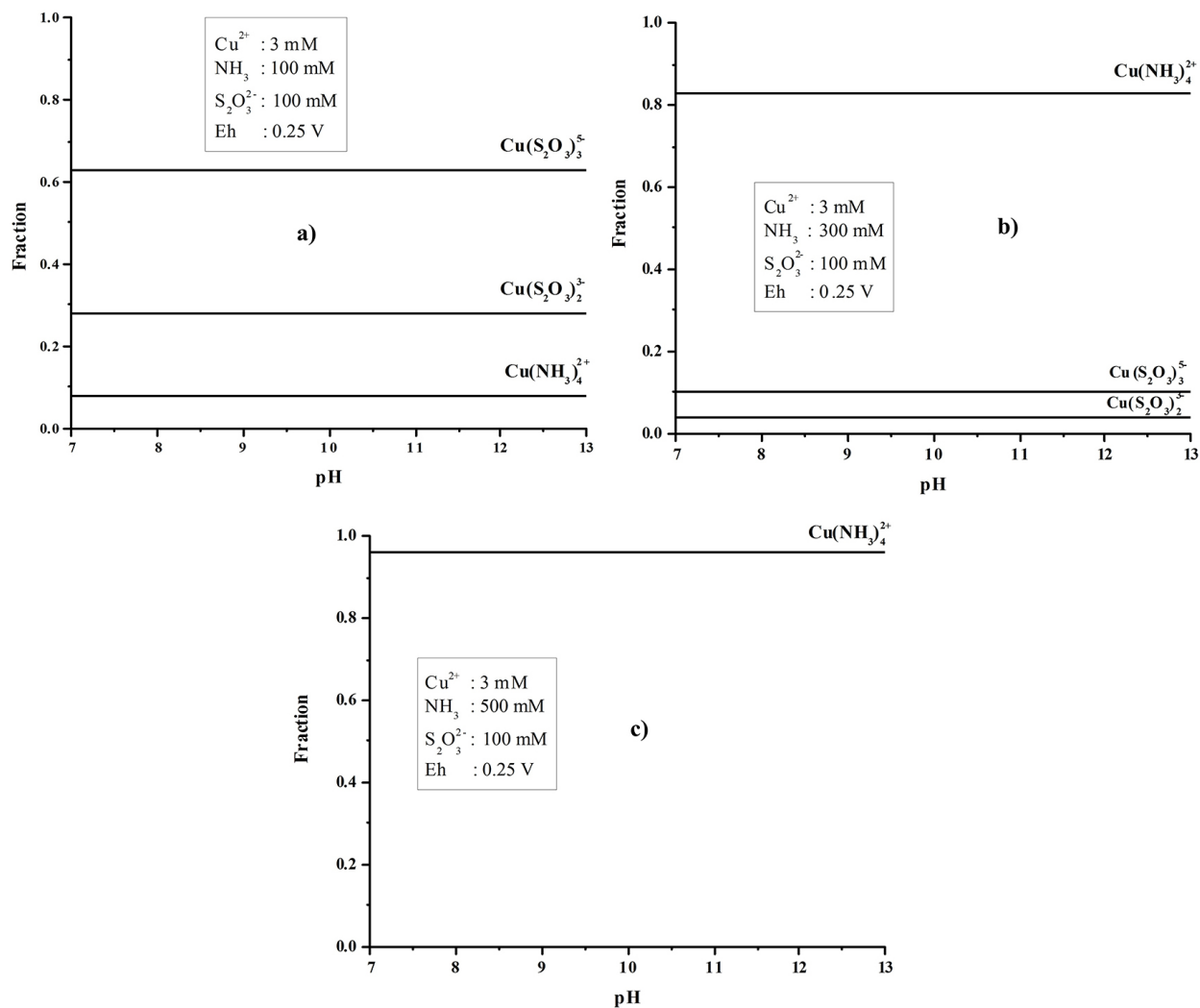**Figure 4.** Effect of ammonia on copper load. Conditions: 200 ppm Cu; 0.1 M thiosulfate; pH 11.
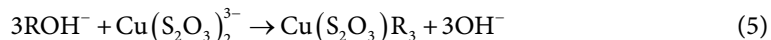

Figure 5. Speciation of the Cu-NH$_3$- SO$_3^{2-}$ system at 25°C.

3.1.4. Effect of Copper Concentration

Figure 6 shows the effect of the concentration of dissolved copper on the copper load on the resin. Under the experimental conditions such as pH, and NH_3 and $S_2O_3^{2-}$ concentration, the adsorption of copper on the resin takes place according to the following stoichiometry:

$$3ROH^- + Cu(S_2O_3)_2^{3-} \rightarrow Cu(S_2O_3)R_3 + 3OH^- \tag{5}$$

As the dissolved copper concentration increases, the copper load on the resin also increases. For the three copper concentrations studied there was a slight desorption after 90 to 120 minutes of solution-resin contact.

3.2. Desorption Stage

The elution or desorption tests were performed to replace the $Cu(S_2O_3)_2^{3-}$ complex loaded on the resin by the sulfite or perchlorate eluent anion. Initially the resin was loaded with 17.4 g Cu/kg resin. This load was obtained by contacting the resin with an aqueous solution of 200 ppm Cu; 0.1 M; and 0.1 M NH_3 at pH 11 during 2 h of resin-solution contact, at a constant temperature of 25°C.

3.2.1. Elution of Copper with Perchlorate (ClO_4^-) Solutions

Figure 7 shows the kinetics of copper elution from the loaded resin by an aqueous solution with different perchlorate concentrations. Increasing the concentration of perchlorate from 0.1 M to 0.5 M did not cause an important increase in the desorption of the copper complex from the resin after 155 minutes of solution-resin contact. The following stoichiometry represents the desorption reaction of the $Cu(S_2O_3)_2^{3-}$ copper complex from the ion exchange resin by perchlorate:

$$R_3^+\left[Cu(S_2O_3)_2\right]^{3-} + 3ClO_4^- \rightarrow 3R^+(ClO_4)_3^- + \left[Cu(S_2O_3)_2\right]^{3-} \tag{6}$$
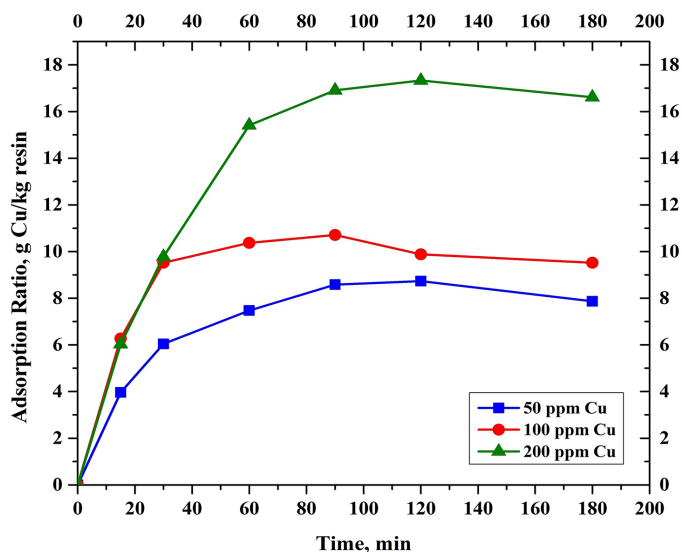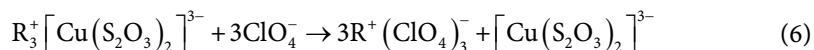

Figure 6. Effect of initial copper concentration and its adsorption in the resin. Conditions: pH 11; 0.1 M ammonia; 0.1 M thiosulfate.
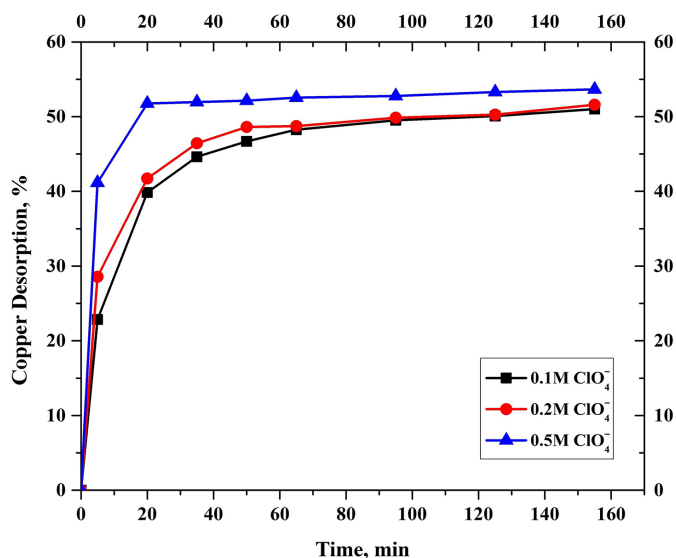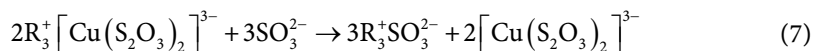
Figure 7. Effect of perchlorate in the copper elution. Conditions: resin loaded with 17.35 g Cu/kg resin; 0.1 M ammonia; pH 11.

As the perchlorate concentration was increased from 0.1 M to 0.5 M, the initial exchange rate increased considerably (reaction 6), so that working with a 0.5 M perchlorate concentration the maximum copper elution of 50% was achieved after 20 minutes of solution-resin contact. But using 0.1 and 0.2 M ClO_4^-, after the 20 minutes of contact 39% and 41% desorption of copper was achieved.

3.2.2. Elution of Copper with Sulfite (SO_3^{2-}) Solutions

Figure 8 shows the effect of sulfite concentration in the eluent solution on the elution of copper from the loaded resin. The results show that the elution of the $Cu(S_2O_3)_2^{3-}$ complex with sulfite is very efficient and fast. Working with 0.1 M and 0.2 M SO_3^{2-} concentrations, elutions close to 80% were achieved after 150 min of solution-resin contact, and 99% working with 0.5 M SO_3^{2-} after 30 min of contact. The stoichiometry of the elution of copper with sulfite is given by:

$$2R_3^+\left[Cu\left(S_2O_3\right)_2\right]^{3-}+3SO_3^{2-}\rightarrow 3R_3^+SO_3^{2-}+2\left[Cu\left(S_2O_3\right)_2\right]^{3-} \qquad (7)$$

Sulfite solutions were more efficient than those of perchlorate for eluting the $Cu(S_2O_3)_2^{3-}$ complex from the resin, which in an industrial process has the effect of working with a smaller inventory of eluent solution, in addition to yielding solutions with higher copper concentrations.

4. Conclusions

Under different experimental conditions the $Cu(S_2O_3)_2^{3-}$ complex can be adsorbed by a strong base ion exchange resin like DOWEX 550A. In the pH range studied, increasing this parameter from 9 to 11 favors the adsorption of copper, reaching a load of 5.86 kg Cu/ton resin at pH 9 and 60 minutes of solution-resin contact, and 16.91 kg Cu/ton
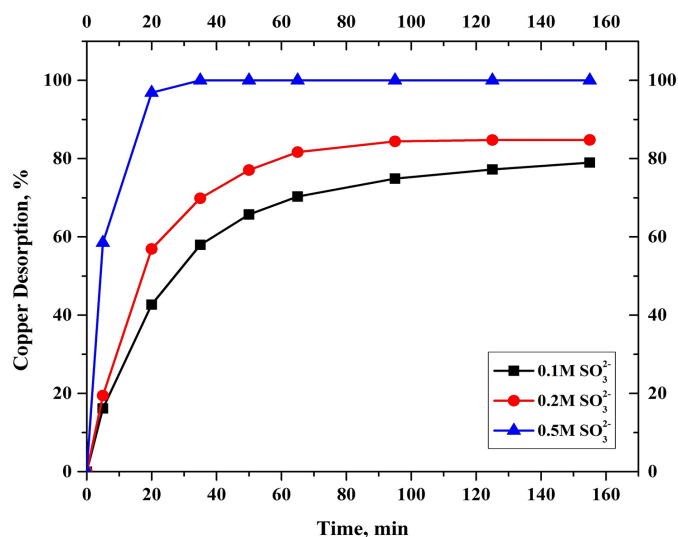
Figure 8. Effect of sulfite in the copper elution. Conditions: resin loaded with 17.35 g Cu/kg resin; 0.1 M ammonia; pH 11.

resin at pH 11 after 90 minutes of contact. Increasing the concentration of thiosulfate in the aqueous solution (0.1 M to 0.5 M) decreases the adsorption of the $Cu(S_2O_3)_2^{3-}$ complex. Increasing the ammonia content in the solution increases the stability of the cuprotetramine, decreasing the amount of copper-thiosulfate complex, producing a lower adsorption of copper on the resin.

Elution of the copper complexes from the resin with sulfite ions is better and faster than with perchlorate ions. A good elution with sulfite is beneficial because this anion is part of the thiosulfate oxidation system. Elution with sulfite ion is more sensitive to changes in concentration, achieving 100% elution for a 0.5 M and 84.79% elution for 0.2 M, while elution was 53.65% at 0.5 M perchlorate concentration and 51.59% at 0.2 M concentration.

Acknowledgements

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