


## Research Article

# Pre-Reducing Process Kinetics to Recover Metals from Nickel Leach Waste Using Chelating Resins

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The main problem of the separation process from nickel mining using the ion exchange technique is the presence of iron, which precipitates in pH above 2.00 and causes coprecipitation of copper and cobalt. Chelating resins have the main advantage of being selected for a specific metal present in solution. Studies have been developed to increase the efficiency of metals recovery using chemical reduction and the ion exchange process to recover metals. The aim of this work was to use sodium sulfite as a reducing agent to convert Fe(III) to Fe(II). Chelating resins Lewatit® TP 207, selective for copper, and Lewatit® TP 220, selective for nickel and cobalt, were studied. Batch experiments were performed to study the effect of pH with and without sodium sulfite. Results indicated that the industrial process has increased efficiency when the reducing process is applied.

## 1. Introduction

Nickel laterite represents 70% of nickel reserves and 40% of nickel production, mostly processed by the hydrometallurgical process, due to the fact that the nickel laterite process is more expensive and difficult than other ores [1–6]. The main problem for metals recovery, such as nickel, copper, and cobalt, from these ores is the high concentration of iron. Limonite layer, the first layer of the nickel laterite ore and processed by high-pressure acid leaching or atmospheric acid leaching using sulfuric acid, has 40–50% of iron approximately, while nickel concentration is 0.8–1.5% and cobalt concentration is 0.1–0.2% [7].

In order to separate iron from nickel laterite leach, Jiménez Correa et al. studied chemical precipitation of Fe(III) and Fe(II) in a solution of nickel laterite using hydroxides. Results obtained show that, at pH 2.50, 30% of iron and 20% of cobalt precipitate. At pH 3.00, 100% of iron, 60% of cobalt, and 20% of copper precipitate [8]. Another study realized by Chang et al. performed experiments to precipitate iron from nickel laterite leach by the oxidation

process. Results show that there was loss of nickel to the residue with all iron. Nickel can be recovered from the residue using the weak acid solution, but more steps can turn the process impracticable [9].

Ion exchange process using chelating resins can be a solution to selectively recover metals. For this reason, Jiménez Correa et al. studied copper and nickel recovery using the chelating resin Dowex M4195 from nickel laterite leach waste. The chelating resin has the bis-picolylamine functional group, and results show that copper recovery was highly influenced by iron due to its high concentration (150 mg·L<sup>-1</sup> of copper and 18000 mg·L<sup>-1</sup> of iron) [10]. Littlejohn and Vaughan [11] studied nickel and cobalt recovery using the chelating resin Lewatit® TP 220, with the same functional group of M4195, from nickel laterite tailings. Ferric iron was the most significant impurity adsorbed by resin, and results were obtained by Jiménez Correa et al. [10].

Zainol and Nicol studied five chelating resins with the iminodiacetate functional group to recover nickel and cobalt from nickel laterite leach tailings. The presence of iron, as well as chromium and aluminum, decreased resins

efficiency, due to strong adsorption of these functional groups. In spite of all resins studied having the same functional group, results were different, and TP 207 MonoPlus had better results for nickel recovery [12]. Comparing metals recovery using Lewatit® TP 207 with the iminodiacetate functional group, copper recovery is higher than nickel in all pH values studied by Rudnicki et al. [13]. Metals recovery, both copper and nickel, increased when pH increases [13].

According to Littlejohn and Vaughan and Mendes and Martins, chelating resins with the iminodiacetate functional group are better to recover copper, and resins with the bis-picolylamine functional group were more selective for nickel and cobalt than the others metals [14, 15].

In spite of chelating resins used to selectively recover metals, iron still is a problem to overcome. Botelho Junior et al. studied the difference between ferric iron and ferrous iron in nickel laterite leach waste for copper recovery using Lewatit® TP 207, in which chelating resin efficiency increases when iron is present as ferrous iron. In solution with Fe(III), copper recovery was 48.72%, while solution with Fe(II) copper recovery was 61.32% [16–18]. A way to convert ferric iron to ferrous iron is using a reducing agent. Sodium dithionite and sodium metabisulfite were studied to convert ferric iron to ferrous iron of nickel laterite leach waste reducing the potential of the solution until 590 mV (pH 0.50–2.00) and 240 mV (pH 2.50–3.50). However, the problem is these reducing agents are dangerous in acid solutions, which can release hydrogen sulfide ( $H_2S$ ), and these reducing agents can be added if dissolved in water before. The other problem is they are expensive [19–21].

Other reducing agents that can be used such as sodium thiosulfate or microorganismos have the same problem in acid solutions [22–26]. Nevertheless, sodium sulfite is a reducing agent that can be used in acid solutions. Liu et al. studied reductive stripping of ferric iron using sulfuric acid and sodium sulfite [27]. Luo et al. studied atmospheric leaching of nickel limonite with sulfuric acid using sodium sulfite as a reducing agent to facilitate nickel extraction, comparing the leaching process using only sulfuric acid. Results indicated that nickel extraction increases in presence of sodium sulfite, as well as iron extraction. Though the nickel extraction increased, the increase of the iron extraction still keeps the problem [28].

The goal of this work was to study the batch industrial process to recover copper, nickel, and cobalt using the ion exchange technique. Chelating resins Lewatit® TP 207, selective for copper with the iminodiacetate functional group, and Lewatit® TP 220, selective for nickel and cobalt with bis-picolylamine functional group, were studied. Sodium sulfite was used to reduce the potential of solution, converting ferric iron to ferrous iron, while the reducing process was studied before using sodium dithionite and sodium metabisulfite [19, 20, 29]. Three synthetic solutions were prepared to simulate real conditions of nickel laterite leach waste from the limonite ore. Solution 1 was used to study copper recovery, Solution 2 without copper was used to study nickel recovery; and Solution 3 without copper and nickel was used to study cobalt recovery. Effect of pH was studied between

pH 0.50 and 2.00, for solutions without sodium sulfite, and between pH 0.50 and 3.50, with sodium sulfite. Experiments were performed in a batch at 25°C and 150 rpm. Samples were analyzed using ICP-OES (Varian 725ES).

## 2. Materials and Methods

*2.1. Materials.* Composition of synthetic solutions is present in Table 1. Sulfate salts of each metal were dissolved in deionized water, and the pH was adjusted with sulfuric acid concentrated PA. Three different solutions were prepared: Solution 1 was prepared with all metals that compose the nickel laterite leach, Solution 2 was prepared without copper, and Solution 3 was prepared without copper and nickel. Therefore, it is a possible study effect of other metals presented in the leach solution for copper, nickel, and cobalt adsorption in three steps.

Two different chelating resins were studied: Lewatit® TP 207, for experiments performed using Solution 1, and Lewatit® TP 220, for experiments performed using Solutions 2 and 3. TP 207 is a cationic resin with the iminodiacetate functional group, crosslinked polystyrene macroporous matrix, pH range 0–14 and density  $1.17\text{g}\cdot\text{mL}^{-1}$  [30]. The theoretical selectivity order for this resin is  $\text{Fe(III)} > \text{Cu(II)} > \text{Ni(II)} > \text{Zn(II)} > \text{Fe(II)} > \text{Mn(II)} > \text{Mg(II)}$  [13]. TP 220 is also a cationic resin, but with the bis-picolylamine functional group, crosslinked polystyrene macroporous matrix, density  $1.1\text{g}\cdot\text{mL}^{-1}$ . The theoretical selectivity order for this resin is  $\text{Cu(II)} > \text{Ni(II)} > \text{Fe(III)} > \text{Co(II)} > \text{Mn(II)} > \text{K(I)} > \text{Ca(II)} > \text{Na(I)} > \text{Mg(II)} > \text{Al(III)}$  [11, 31].

### 2.2. Method

*2.2.1. Pre-Reducing Process.* Botelho Junior et al. studied the reducing process using sodium dithionite and sodium metabisulfite to convert Fe(III) to Fe(II) from the synthetic solution of nickel laterite leach. Effect of time, pH, and temperature was studied. Temperature decreased ferric iron chemical conversion, probably due to the reducing agent composition. The effect of time was studied until 96 hours, and after 120 min, the reaction reaches equilibrium, until 480 min, and then decreased from 95% to 80% after 1144 min. Conversion of Fe(III) to Fe(II) in 100% was performed decreasing the potential until 590 mV, at pH 0.50–2.00, and 240 mV, at pH 2.50–3.50. For synthetic solution with all metals of nickel laterite leach, ferric iron conversion is 80% [19, 20, 29].

Thus, the pre-reducing process was performed during 120 min adding sodium sulfite in order to decrease the potential until 590 mV SHE (standard hydrogen electrode), pH 0.50–2.00, and 240 mV SHE, pH 2.50–3.50, at 25°C in 150 rpm.

*2.2.2. Ion Exchange Experiments.* Ion exchange experiments were performed in flasks of 100 mL with 50 mL of solution and 0.50 mL of resin in 150 rpm at 25°C during 120 min. The effect of time was studied without the pre-reducing process between 30 and 480 min at pH 0.50 for Solution 1, with

TABLE 1: Metals concentration for batch experiments for Solution 1, Solution 2, and Solution 3 in  $\text{mg}\cdot\text{L}^{-1}$ .

—	Metals									
	Al	Co	Cu	Cr	Fe	Mg	Mn	Ni	Zn	
Conc. ( $\text{mg}\cdot\text{L}^{-1}$ )	Solution 1	4101	78	146	195	18713	7774	387	2434	36
	Solution 2	4101	78	—	195	18713	7774	387	2434	36
	Solution 3	4101	78	—	195	18713	7774	387	—	36

Lewatit® TP 207, and Solution 2, with Lewatit® TP 220. The effect of pH was studied between pH 0.50 and 3.50.

Resins were washed using hydrochloric acid  $6 \text{ mol}\cdot\text{L}^{-1}$  and sodium hydroxide  $1 \text{ mol}\cdot\text{L}^{-1}$  for three times using water deionized between each step. Sulfuric acid  $1 \text{ mol}\cdot\text{L}^{-1}$  was used after wash. Sulfuric acid concentrated PA and sodium hydroxide pellet were used to correct the pH [32].

In order to quantify the cations adsorbed, Equation (1) was used, where  $q_t$  is the capacity of the ion adsorbed in time  $t$  in mass of the ion per mass of resin ( $\text{mg}\cdot\text{g}^{-1}$ ),  $C_0 \cdot e \cdot C_t$  are concentrations of ions in time = 0 and time =  $t$  ( $\text{mg}\cdot\text{L}^{-1}$ ),  $v$  is the volume of solution ( $L$ ), and  $m$  is the mass of the resin ( $g$ ) [13, 33]. Equation (2) was used to quantify the coefficient of distribution of ion, which one is the measure of the effectiveness of ion adsorption from solution [32]. Equation (3) was used to quantify the adsorbed ion, in percentage:

$$q_t = (C_0 - C_t) \times \frac{v}{m}, \quad (1)$$

$$Kd = \frac{q_e}{C_t} \times 1000, \quad (2)$$

$$\%S = \frac{(C_0 - C_t)}{C_0} \times 100\%. \quad (3)$$

The pH was measured using electrode Ag/AgCl (Sensoglass), and electrode ORP (oxidation reduction potential) was used to measure the potential in solution. Samples were analyzed using ICP-EOS (Varian 725ES Optical Emission Spectrometer).

### 3. Results and Discussion

Figure 1 presents Pourbaix's diagram constructed using Hydra-Medusa software in experimental conditions for the Fe-S- $\text{H}_2\text{O}$  system, where the conversion of Fe(III) to Fe(II) is 100% after 590mV between pH 0.50 and 2.00, and it is after 240mV between pH 2.50 and 3.50. However, presence of the other metals decreases the reducing process efficiency. In acid medium with sulfuric acid, sodium sulfite reacts with  $\text{H}^+$  to form  $\text{H}_2\text{SO}_3$ , as presented in Equation (4), in which  $k_1 = 1.54 \times 10^{-2}$  and  $k_2 = 1.02 \times 10^{-7}$ . Besides, sodium dithionite and sodium sulfite are different, and both dissociate to form sodium bisulfite, which is the main responsible for the reducing process in solution, but sodium dithionite also dissociates to form sodium thiosulfate [27, 34–37].



Another problem about sodium dithionite is that acid medium can be dissociated to form sulphur and hydrogen sulfide, being the last extremely dangerous [38]. Besides,

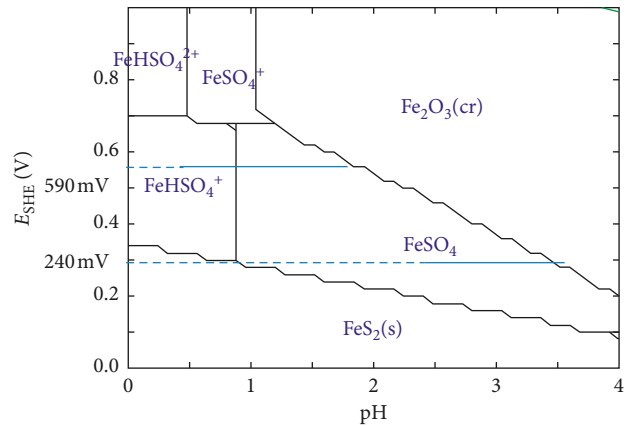


FIGURE 1: Pourbaix's diagram of Fe-S- $\text{H}_2\text{O}$  constructed using Hydra-Medusa software at  $25^\circ\text{C}$ .

sodium dithionite, sodium thiosulfate, and sodium bisulfate are dangerous because of same problem in the acid medium, using them only in the basic medium [23, 39].

**3.1. Effect of Time.** The effect of time was studied using Solution 1 and Solution 2 without the pre-reducing process at  $25^\circ\text{C}$  and pH 0.50. Results for copper (Solution 1) and nickel (Solution 2) recovery are present in Figure 2 and indicate that the reaction reached equilibrium after 120 min, in which Solution 1 was in contact of Lewatit® TP 207 and Solution 2 with Lewatit® TP 220. Iron was the metal highest adsorbed in mg per g of resin ( $151 \text{ mg}\cdot\text{g}^{-1}$ ), while copper was  $1.67 \text{ mg}\cdot\text{g}^{-1}$ , due to high concentration of  $\text{H}^+$  in solution at pH 0.50. For Solution 2, iron was also in this case the highest metal adsorbed ( $85 \text{ mg}\cdot\text{g}^{-1}$ ), while nickel was  $10.87 \text{ mg}\cdot\text{g}^{-1}$ . Experiments to study the effect of pH in the ion exchange process were performed during 120 min.

**3.2. Effect of pH.** The effect of pH in chelating resin with the iminodiacetate functional group can be seen in Figure 3. At pH 2, high concentration of  $\text{H}^+$  in the functional group repulses cations in solution due to protonation of the functional group, where high competition between  $\text{H}^+$  and cations occurs by the functional group. At pH 2–4,  $\text{H}^+$  and cations present in the solution still compete for the chelating resin functional group, the latter being deprotonated. In Figure 3, at pH 7, carboxylic acid of the functional group is deprotonated, and at pH 12, the iminodiacetate functional group is totally deprotonated. However, although the last situation is the most favorable to recover cations due to no presence of  $\text{H}^+$  in the functional group, metals in general

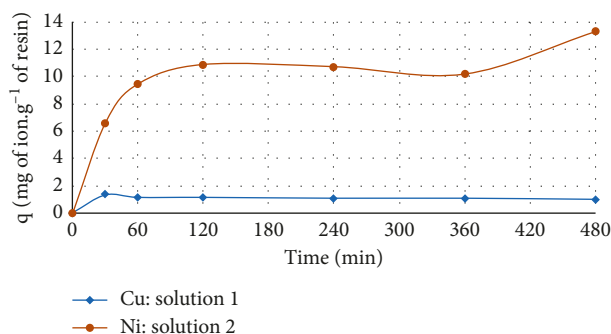


FIGURE 2: Results of effect of time of Solution 1 and Solution 2.

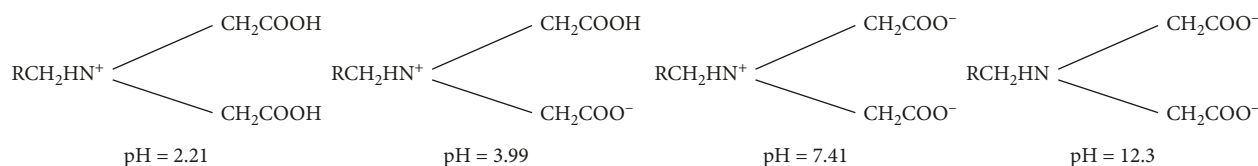


FIGURE 3: Effect of pH in the iminodiacetate functional group [40].

precipitate at pH 5, which causes working pH to be totally dependent of solution characteristics [12, 13, 40].

Results for metals recovery with and without the pre-reducing process are present below. Effect of pH without the pre-reducing process above pH 2.00 was not studied, because from this pH, iron will precipitate with copper and cobalt [8, 41]. Figure 4 presents results for copper recovery in Solution 1 by the iminodiacetate resin Lewatit® TP 207. It is observed that copper recovery increased for Solution 1 with the pre-reducing process. At pH 0.50, copper recovery was 9.66% without the pre-reducing process, while using sodium sulfite to convert Fe(III) to Fe(II), copper recovery at pH 0.50 was 16.67%. At pH 2.00, both had highest copper recovery. In solution without sodium sulfite, copper recovery was 41.43%, and with sodium sulfite as a reducing agent, copper recovery was 68.57%.

Botelho Junior et al. studied the effect of presence of Fe (III) and Fe(II) to recover copper from nickel laterite leach using resin with the iminodiacetate functional group. Results show that when iron is Fe(II), copper recovery is higher than when Fe(III) is present in solution [18]. This can occur because the iminodiacetate functional group has high affinity for Fe(III) [15, 42–44], once it was the highest metal recovery among all in  $\text{mg}\cdot\text{L}^{-1}$  in all pH studied.

Figure 5 shows results of metals recovery of Solution 2 with Lewatit® TP 220, chelating resin with the bis-picolylamine functional group. At pH 0.50–2.00, chelating resin was more selective for nickel than other metals in both solution, except for solution without the pre-reducing process at pH 0.50. At pH 2.50 and 3.00, in solution with the pre-reducing process, cobalt was the metal more selective by the resin, followed by zinc and nickel. At pH 3.50, however, chelating resin was more selective for zinc, followed by nickel and cobalt. This phenomenon was also seen in Solution 1 experiments, where chelating resins selective

order changes for different pH values and also with the pre-reducing process. The difference in the selective order for solution with and without the reducing process can be explained due to conversion of Fe(III) to Fe(II).

Chelating resin for Solution 2 with the pre-reducing process between pH 0.50 and 2.00 was more selective for nickel than cobalt and zinc. At pH 2.50 and 3.00, the resin was more selective for cobalt, and at pH 3.50, chelating resin was more selective for nickel and zinc, simultaneously.

The effect of pH for cobalt and zinc recovery from Solution 3 by the bis-picolylamine resin is shown in Figure 6. Chelating resin has high selective for cobalt between pH 0.50 and 2.00 in both situations. However, at pH 2.50 and 3.00, zinc was more selective by the resin than cobalt, and both were almost not recovered by the resin at pH 3.50. The change in the order of selectivity was also observed. Figure 7 presents coefficient distribution of copper, nickel, cobalt, and zinc in Solutions 1, 2, and 3 with the pre-reducing process.

It is possible seen that copper coefficient distribution, in Solution 1, was maximum ( $186\text{ mL}\cdot\text{g}^{-1}$ ) at pH 2.00, indicating that the chelating resin Lewatit® TP 207 with the iminodiacetate functional group was more selective for copper in this pH. In Solution 2, nickel coefficient distribution was maximum at pH 3.50 ( $121\text{ mL}\cdot\text{g}^{-1}$ ); however, at the same pH, zinc had higher coefficient distribution ( $160\text{ mL}\cdot\text{g}^{-1}$ ) than nickel, indicating Lewatit® TP 220 with the bis-picolylamine functional group was more selective for zinc than nickel. In the meantime, cobalt had higher coefficient distribution at pH 3.50 ( $61\text{ mL}\cdot\text{g}^{-1}$ ) than the other pH values, which is seen in Figure 5, where copper recovery was maximum at pH 3.5 as well as nickel and zinc. For Solution 2, the chelating resin was more selective for nickel at pH 2.00 ( $43\text{ mL}\cdot\text{g}^{-1}$  for nickel and  $15\text{ mL}\cdot\text{g}^{-1}$  for zinc and cobalt), where nickel recovery was 32.55% and zinc and

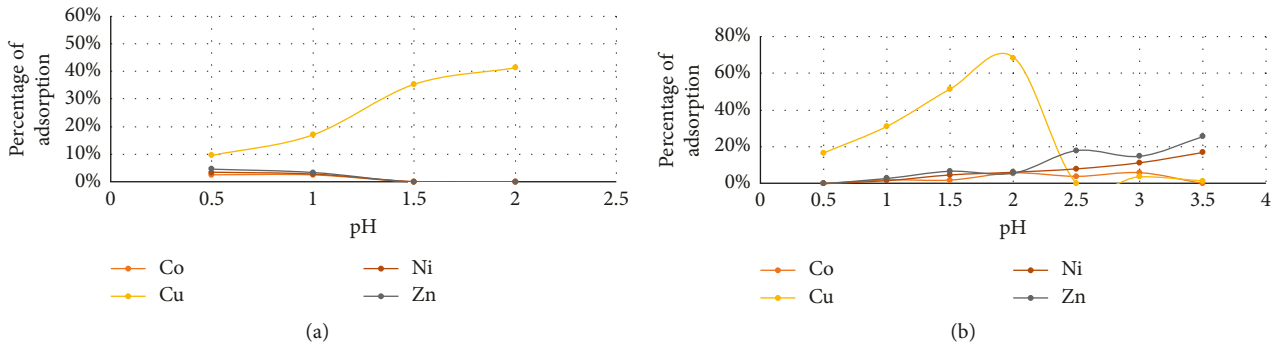


FIGURE 4: Effect of pH in Solution 1 by iminodiacetate resin (a) without and (b) with the pre-reducing process.

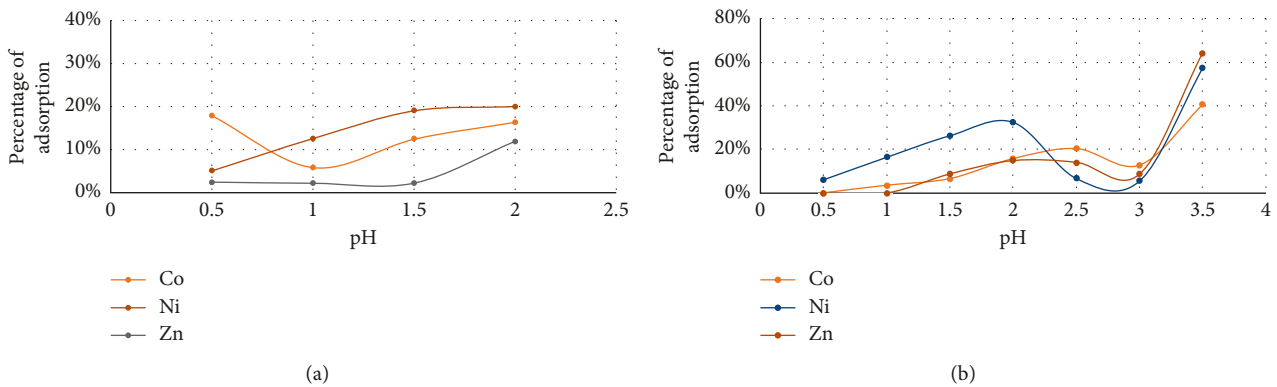


FIGURE 5: Effect of pH in Solution 2 by the bis-picolylamine resin (a) without and (b) with the pre-reducing process.

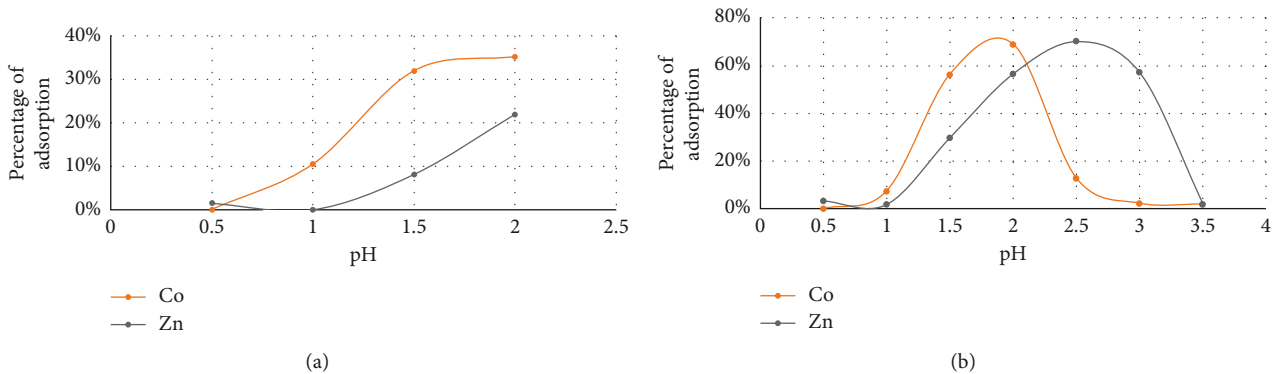


FIGURE 6: Effect of pH in Solution 3 by the bis-picolylamine resin (a) without and (b) with the pre-reducing process.

cobalt, 14.86%. For Solution 3, cobalt coefficient distribution was maximum at pH 2.00 ( $198 \text{ mL}\cdot\text{g}^{-1}$ ), being resin more selective for this metal than others, and at pH 2.50, zinc coefficient distribution was maximum ( $206 \text{ mL}\cdot\text{g}^{-1}$ ).

Studies to recover metals from nickel laterite leach using chelating resins indicated that resins with the iminodiacetate functional group are better to recover copper, while in order to recover nickel and cobalt resins with bis-picolylamine are better [45, 46]. In experiments performed using Solution 1, Lewatit® TP 207 was more selective for copper, while experiments performed with Solutions 2 and 3 using Lewatit®

TP 220 cobalt and nickel were selectively recovered. Zinc was also selectively recovered depending on the pH value.

#### 4. Conclusion

The aim of this work was to study the batch industrial process for metals recovery using two different chelating resins from synthetic solution of nickel laterite leach. Sodium sulfite was used in order to reduce Fe(III) to Fe(II). Results indicated that Lewatit® TP 207 was more selective for copper than the other metals, due to its functional group,



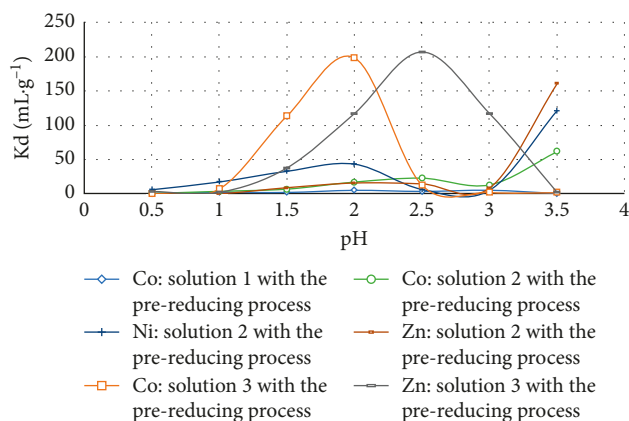


FIGURE 7: Coefficient distribution of copper, nickel, cobalt, and zinc with the pre-reducing process.

and Lewatit® TP 220 was more selective for cobalt and nickel. Sodium sulfite increased metals recovery because chelating resins were less selective for ferrous iron than ferric iron, and pH can be increased without ferric iron precipitation, due to the fact that, while increasing pH, the concentration of  $H^+$  decreases, as well as competition between  $H^+$  and metals in solution for the functional group of chelating resin. Another reason that can explain the metals recovery raise is that ferrous iron occupies less active sites on the chelating resin than ferric iron. In all the solutions studied, metals recovery was higher after the pre-reducing process. A change in the selectivity order of resins was observed comparing with and without the pre-reducing process, which may be caused by conversion of Fe(III) to Fe(II) and also by changing the pH. Pre-reducing process using sodium dithionite and sodium metabisulfite was studied before, but the use of sodium sulfite as a cheap and secure option can make the process economically viable and secure. Industrial process can be a benefit for the process involving chemical reducing and ion exchange process, in which metals recovery increases comparatively without the reducing process. Column experiments are the next step to simulate fixed-bed reactors for the continuum process.

## Data Availability

No data were used to support this study. Please feel comfortable to contact the corresponding author if any doubt comes up.

## Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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