Original Article

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THE STUDY OF CHELATING RESIN DOWEX XUS 43605 FOR COPPER RECOVERY FROM NICKEL LATERITE LEACH SOLUTION

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Abstract

The extraction of metals from nickel laterite ores has been increasing, due to the demand for copper, nickel and cobalt. HPAL is a hydrometallurgical process which uses sulfuric acid as leaching agent. After the leaching step, ion exchange techniques are used to recover metals selectively, as well as chelating resins. The goal of this work was to study the use of Dowex XUS 43605 for copper recovery from nickel laterite leach solution. Mono- and multielementary solutions were studied and results compared the effect of other metal ions on copper adsorption. As previous study showed, iron is the main contaminant in solution. However, the use of Dowex XUS 43605 is not explored in the literature. The effect of time, pH, copper concentration, mass of resin and temperature were evaluated. Results showed that copper adsorption was 100% using monoelementary solutions and 72% in multielementary solution at pH 1.5 after 120min, where the resin was more selective for copper. The use of high quantity of resin decreased the adsorption capacity. Temperature had no effect on copper adsorption. These results are important to develop industrial process of nickel laterite extraction.
Keywords: Hydroxypropyl-picolylamine; HPPA; ion exchange; hydrometallurgy.

1 INTRODUCTION

Nickel laterite represents 70% of nickel resources and 40% of nickel production, which it is possible to recover copper and cobalt [1,2]. The main problem of metals extraction from those resources is the high content of iron, which may make the process unfeasible [3,4]. The process to extract nickel, cobalt and copper from nickel laterite resources is The High Pressure Leaching Process (HPAL). Sulfuric acid is used as leaching agent at 240°C and 40bar. The liquor obtained needs to be purified [4].

Among the techniques that can be used to purified the liquor, the ion exchange process has few advantages, such as the high concentration of iron. The solvent extraction can be used, as Aliprandini [5] showed. A solvent extraction process was developed and 100% of nickel from the liquor was purified. However, cobalt and high concentration of iron were extracted using Cyanex 272 20%. Copper extraction was possible using Acorga M5640 5%.

Another ion exchange technique is the use of chelating resins, which are selectivity to specific metals present in solution [4,6,7]. Botelho et al. [3] showed that it is possible to recover nickel and cobalt using chelating ion exchange resins. The continuous experiments were performed in columns, where the first column was used to remove copper from solution. Nickel and cobalt recovery efficiency was 79.3% and 77.9%, respectively [8]. Nonetheless, process efficiency has been hampered by the first column, where the chelating resin Lewatit TP 207 adsorbed 17% of nickel. For this reason, the study of chelating resin to recover copper with high efficiency and low adsorption of other metals must be considered.

The goal of this work was to study the use of chelating resin Dowex XUS 43605 for copper recovery from nickel laterite leach solution and the effect of other metals. Experiments with monoelementary solutions were performed to evaluate copper adsorption by the chelating resin. The effect of time reaction, the pH and concentration of copper were evaluated. Multielementary solution was prepared to simulate the real nickel laterite leach solution. Then, experiments were performed to study the effect of other metal ions on copper recovery. The effect of pH, mass of resin and temperature were studied. Samples were analyzed on X-ray fluorescence equipment (EDXRX).

2 MATERIALS AND METHODS

2.1 Materials

Synthetic solution was prepared to simulate the real nickel laterite leach solution. Sulfate salts of each metal was dissolved in deionized water. Metals concentration is presented in Table 1. The pH was adjusted to 0.5 using sulfuric acid P.A.

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Copper monoelementary solutions were prepared to study the ion adsorption by the resin and multielementary solution with all metals presented in leach solution to evaluate the copper adsorption efficiency.

The chelating resin studied was Dowex XUS43605 with hydroxypropyl-picolylamine functional group (HPPA) and particle size 0.32. The theoretical selectivity order is Cu(II)>>Ni(II)>Fe(III)>Zn(II)>Co(II)>Cd(II)>Fe(II) [9-11]. Figure 1 shows the HPPA functional group [12]. The resin was washed with hydrochloric acid 6mol/L and sodium hydroxide 2mol/L in three steps, following the order HCl-deionized water-NaOH-water deionized. Then, the resin was died in stove at 60°C during 24 hours.

2.2 Methods

Ion exchange experiments were performed in erlenmeyer flasks with 1g of resin and 50mL of solution in shaker at 200rpm. Experiments with copper monoelementary solution were performed to study the ions adsorption by the resin. The effect of time was studied between 30-1440min and the effect of pH was studied between 0.5-2.0. The concentration of copper was studied between 70-400mg/L.

Experiments with multielementary solution were performed to study the effect of other ions from nickel laterite leach solution on copper adsorption. The effect of pH between 0.5-2.0 and mass of resin between 0.055-7g were evaluated. Thermodynamics experiments were performed between 25-60°C. Samples were analyzed in FRX. Equation 1 was used to quantify the mass of ions adsorbed per mass of resin (mg/g). Equation 2 was used to quantify of ion was adsorbed, in percentage [3,13,14].

Table 1. Concentration of elements present in synthetic solution of nickel laterite leach solution, in mg/L

Element	Al	Co	Cr	Cu	Fe	Mg	Mn	Ni	Zn
Conc. mg/L	4,101	78	195	146	18,713	7,774	397	2,434	36

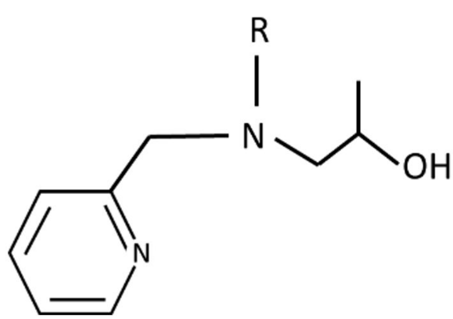

Figure 1. The hydroxypropyl-picolylamine functional group of Dowex XUS 43605 chelating resin [12].

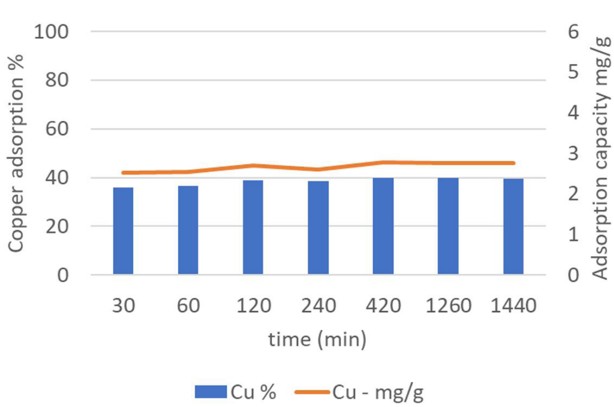

Figure 2. Percentage of copper ions adsorbed and adsorption capacity (mg/g) during the time (30-1440min) using 1g of resin, 50mL of solution and pH 0.5 at 25°C.

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

$$\%S = \frac{(C_0 - C_t)}{C_0} \times 100\% \quad (2)$$

qt: capacity of ion adsorbed in time t in mass of ion per mass of resin (mg/g);
C_0 and C_t: concentration of ions in time = 0 and time = t (mg/L);
v: volume of solution (L); m: the mass of resin (g)

The pH was studied to verify the effect of H^+ ions on metals adsorption, where the H^+ ions concentration decreases when the pH increases. As a result, the metals adsorption increases. The kinetics of adsorption was studied to verify when the reaction react the equilibrium. The effect of copper concentration was studied in order to determine the adsorption limit. The temperature was studied to verify if the reaction is endothermic or exothermics.

3 RESULTS AND DISCUSSION

3.1 Copper Adsorption in Monoelementary Solution

3.1.1 Effect of time on copper monoelementary solution

The effect of time on copper adsorption was studied at pH 0.5, 1g of resin and 50mL of solution at 25°C. The time reaction studied were 30min, 60min, 120min, 240min, 420min, 1260min and 1440min. Results are shown in Figure 2. Copper adsorption increased from 30min to 120min, where reached the equilibrium. In 120min, the adsorption capacity was 2.7mg/g, which represents 38.8% of copper adsorption.

The difference on copper adsorption at 120min and 240min, 420min, 1260min and 1440min was less than 5%. Botelho et al. [8] verified that copper adsorption reached the equilibrium in 120min. The chelating resin studied by the authors has an iminodiacetate functional group (TP 207). For this reason, the following experiments were performed in 120min.

3.1.2 Effect of pH on ions adsorption

The effect of pH was studied in 120min, using 1g of resin and 50mL of solution at 25°C. Figure 3 shows the percentage of copper recovery. Copper adsorption increased from 38.8% at pH 0.5 to 100% at pH 1.5 and 2.0. The increase on metal adsorption when pH increases occurs due to the decrease of H^+ concentration in solution. The H^+ ions competes with metal ions for the functional group of chelating resin [15], and in pH 1.5 all copper ions were adsorbed.

However, these experiments were performed with monoelementary solutions, and other metal ion present in solution should decrease copper recovery due to the competition among them. Also, previous studies depicted that iron precipitates in pH above 2.00 which causes the co-precipitation of copper and cobalt [5,11]. For this reason, experiments in pH above 2.00 were not performed.

3.1.3 Effect of concentration on ions adsorption

Figure 4 shows the effect of copper concentration on ions adsorption. The concentrations studied were 70mg/L, 147mg/L, 270mg/L and 400mg/L. All copper was adsorbed in solutions with 70mg/L and 147mg/L. However, ions adsorption decreases when copper concentration increased, where 69.5% of copper was adsorbed in solution with 400mg/L. It may have occurred due to the limit of activated sites of the resin, saturating the ions adsorption on the resin surface.

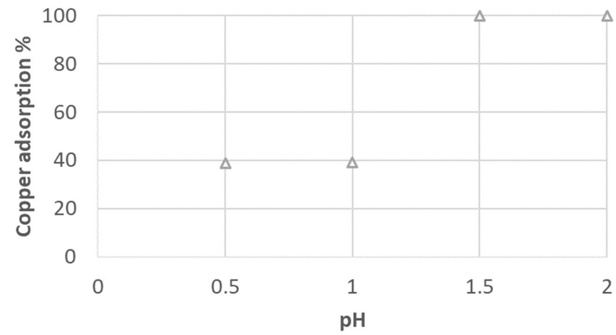

Figure 3. Percentage copper adsorbed for each pH studied in 120min, using 1g of resin, 50mL of solution at 25°C. Δ = Percentage of copper ions adsorption.

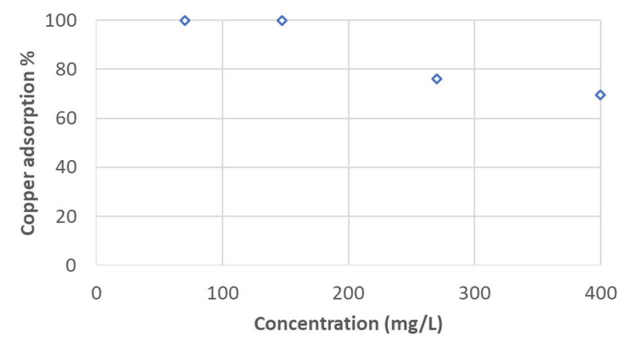

Figure 4. The effect of copper concentration in 120min, at pH 1.5, using 1g of resin and 50mL of solution at 25°C. ◊ = Percentage of copper ions adsorption.

3.2 Metals Adsorption on Synthetic Solution of Nickel Laterite Leach Solution

The effect of copper adsorption in multielementary solution were performed to study the effect of contaminants of process efficiency. As previous study showed, iron is the main contaminant in solution [16]. However, the use of Dowex XUS 43605 is not explored in the literature. For this reason, the effect of contaminants was studied.

3.2.1 The effect of pH on metals adsorption

The effect of pH was studied to evaluate the effect of concentration of H^+ on metals adsorption. The presence of other contaminants for copper recovery was evaluated. Figure 5 shows the percentage of copper, iron and nickel adsorption. Aluminum, manganese, cobalt, chromium, magnesium and zinc were not adsorbed by the resin. Copper adsorption was 6.1mg/g at pH 2.0, while in monoelementary experiments it was 7.6mg/g. Iron and nickel adsorption were respectively 6.3 and 1.3 times higher than copper adsorption at pH 2.0, due to the selectivity order of the chelating resin and concentration of those metals in solution.

Copper adsorption decreased from 100% in monoelementary experiments to 72% in multielementary experiments at pH 1.5. Jiménez Correa et al. [17] verified that copper recovery was 80% in pH between 0.5 and 2.0 using chelating resin Dowex M4195 (bis-picolylamine functional group). It occurs due to the presence of other ions competing for the functional group of the chelating reins. Abbasi et al. [18] studied a chelating resin with iminodiacetate functional group and showed that iron is the main contaminant on nickel laterite leach solution, which decreases metals adsorption.

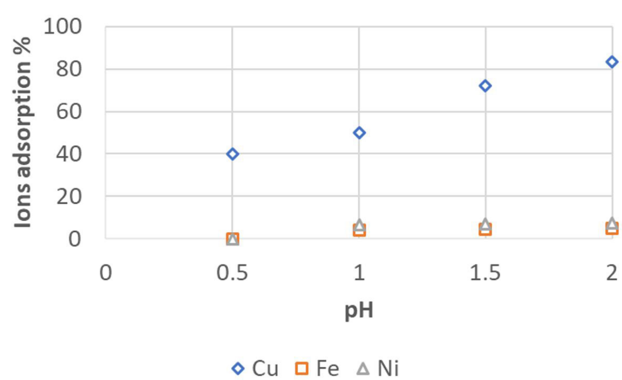

Figure 5. Percentage ions adsorbed for each pH studied in 120min, using 1g of resin, 50mL of solution at 25°C.

3.2.2 The effect of mass of resin on metals adsorption

The effect of mass of resin was studied for 0.055g, 0.25g, 0.5g, 1g, 2.5g, 4g and 7g. In batch industrial process, the quantity of resin per volume of solution is important

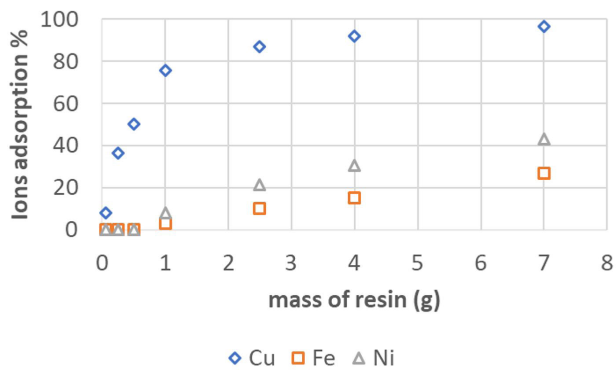

Figure 6. Percentage ions adsorbed for each mass of resin studied in 120min, pH 1.5, 50mL of solution at 25°C.

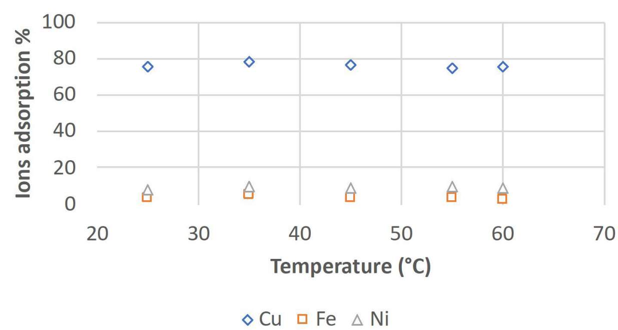

Figure 7. Percentage ions adsorbed varying the temperature in 120min, 1g of resin, pH 1.5 and 50mL of solution.

for the economic and technical feasibility [19]. Experiments were performed at pH 1.5, 50mL of solution, during 120min at 25°C. Figure 6 presents the percentage of copper, iron and nickel adsorption. Aluminum, magnesium, manganese and zinc were not adsorbed. Metals adsorption increases when mass of resin increased. Chromium and cobalt were adsorbed using 2.5g, 4g and 7g of resin. Iron and nickel were adsorbed in experiments with more than 1g of resin.

Copper adsorption increased from 7.9% (0.055g) to 96.6% (7g). However, the increase of mass of resin decreased the adsorption capacity from 10.9mg/g to 1.1mg/g, which may occurred due to the fact that not all activated sites were saturated. The increase of quantity of resin in contact of solution increased the quantity of metals adsorbed by the resin due to the activated sites available, which did not saturated the resin. By the reason of copper adsorption increased from 50% (0.5g) to 75% (1g) and iron and nickel adsorption increased from 3% and 8% (1g) to 10.1% and 21.5% (2.5g), respectively, the use of 1g of resin had better results.

3.2.3 The temperature on metals adsorption

The thermodynamics of copper adsorption by Dowex XUS 43605 were studied at 25-60°C. Chelating resins can be endothermic (metal adsorption increases when temperature increase) or exothermic (metal adsorption decreases when temperature increase) [16].

Figure 7 depicts the results of nickel, copper and iron adsorption varying the temperature. Iron adsorption had no effect when temperature increased until 55°C, but decreased at 60°C, from 29.5mg/g to 23.4mg/g. Copper adsorption had no effect when temperature increased. Consequently, there is no reason to increase the temperature in industrial process to recover copper using Dowex XUS 43605 from nickel laterite leach solution.

Copper adsorption slightly decreases in chelating resins with iminodiacetate functional groups when temperature increases [8,20]. For this reason, the effect of temperature must be studied for each chelating resin and solution.

4 CONCLUSION

The goal of this work was to evaluate the chelating resin Dowex XUS 43605 for copper recovery from nickel laterite leach solution. Monoelementary and multielementary experiments were performed to study the effect of other metals in solution on copper recovery efficiency. Results showed that the reaction reached the equilibrium at 120min. Copper adsorption was 100% using monoelementary solutions and 72% in multielementary solution at pH 1.5. The pH where the resin was more selectivity for copper was 1.5. The increase of quantity of resin in solution decreased the adsorption capacity and the better result was using 1g of resin. Temperature has slightly effect on metals adsorption. Future studies must be focus on continuous experiments to simulate an industrial process.

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REFERENCES

1 Mudd GM. Global trends and environmental issues in nickel mining: sulfides versus laterites. Ore Geology Reviews. 2010;38(1-2):9-26. http://dx.doi.org/10.1016/j.oregeorev.2010.05.003.

2 Norgate T, Jahanshahi S. Assessing the energy and greenhouse gas footprints of nickel laterite processing. Minerals Engineering. 2011;24(7):698-707. http://dx.doi.org/10.1016/j.mineng.2010.10.002.

3 Botelho Junior AB, Dreisinger DB, Espinosa DCR. A review of nickel, copper, and cobalt recovery by chelating ion exchange resins from mining processes and mining tailings. Mining, Metallurgy & Exploration. 2019;36(1):199-213.

4 Crundwell FK, Moats MS, Ramachandran V, Robinson TG, Davenport WG. Extractive metallurgy of nickel, cobalt and platinum-group metals. Oxford: Elsevier; 2011. 583 p. [cited 2018 Jan 3]. Available at: http://linkinghub.elsevier.com/retrieve/pii/B9780080968094100012.

5 Aliprandini P. O uso da extração por solventes para tratamento de licor de lixiviação de minério limonítico de níquel. São Paulo: Universidade de São Paulo; 2017.

6 Sole KC, Mooiman MB, Hardwick E. Ion exchange in hydrometallurgical processing: an overview and selected applications. Journal Separation & Purification Reviews. 2018;47(2):159-178. http://dx.doi.org/10.1080/15422119.2017.1354304.

7 Inamuddin ML. Ion exchange technology I. Vol. 10. New York: Springer; 2012. 350 p. [cited 2018 Jan 3]. Available at: http://www.degruyter.com/doi/10.1524/zpch.1957.10.5_6.350

8 Botelho Junior AB, Espinosa DCR, Dreisinger D, Tenório JAS. Recovery of nickel and cobalt from nickel laterite leach solution using chelating resins and pre-reducing process. Canadian Journal of Chemical Engineering. 2019;97(5):1181-1190.

9 Perez ID, Espinosa DCR. Efeito do pH na adsorção de metais de uma solução sintética utilizando resina quelante Dowex XUS43605. In: Associação Brasileira de Metalurgia, Materiais e Mineração. Anais do ABM Week 2016; 2016; Rio de Janeiro, Brazil. São Paulo: ABM; 2016. p. 1-9.

10 Dow Chemical Company. Ion exchange resins for chemical processing [cited 2018 Jan 3]. Available at: http://msdssearch.dow.com/PublishedLiteratureDOWCOM/dh_07c8/0901b803807c8488.pdf?filepath=liquidseps/pdfs/noreg/177-02437.pdf&fromPage=GetDoc.

11 Perez ID. Recuperação de cobre de uma solução sintética baseada no licor de lixiviação atmosférica de minério limonítico de níquel por troca iônica utilizando a resina quelante Dowex XUS43605 [dissertação]. São Paulo: Universidade de São Paulo; 2018.

12 Squadrone S, Burioli E, Monaco G, Koya MK, Prearo M, Gennero S, et al. Human exposure to metals due to consumption of fish from an artificial lake basin close to an active mining area in Katanga (D.R. Congo). The Science of the Total Environment. 2016;568:679-684. http://dx.doi.org/10.1016/j.scitotenv.2016.02.167.

13 Rudnicki P, Hubicki Z, Kołodyńska D. Evaluation of heavy metal ions removal from acidic waste water streams. Chemical Engineering Journal. 2014;252:362-373.

14 Yu Z, Qi T, Qu J, Wang L, Chu J. Removal of Ca(II) and Mg(II) from potassium chromate solution on Amberlite IRC 748 synthetic resin by ion exchange. Journal of Hazardous Materials. 2009;167(1-3):406-412.

15 Zainol Z, Nicol MJ. Ion-exchange equilibria of Ni2+, Co2+, Mn2+ and Mg2+ with iminodiacetic acid chelating resin Amberlite IRC 748. Hydrometallurgy. 2009;99(3-4):175-180. http://dx.doi.org/10.1016/j.hydromet.2009.08.004.

16 Botelho Junior AB, Vicente ADA, Espinosa DCR, Tenório JAS. Effect of iron oxidation state for copper recovery from nickel laterite leach solution using chelating resin. Separation Science and Technology. 2019 [cited 2018 Jan 3]:1-11. Available at: https://www.tandfonline.com/doi/full/10.1080/01496395.2019.1574828.

17 Jiménez Correa MM, Aliprandini P, Silvas FPC, Tenório JAS, Dreisinger D, Espinosa DCR. Nickel and copper adsorption from acidic sulfate medium by ion exchange. In: Proceedings of the Conference of Metallurgists Hosting World Gold & Nickel Cobalt; 2017; Vancouver, BC, Canada. Vancouver: Canadian Institute of Mining, Metallurgy and Petroleum; 2017.

18. Abbasi P, McKevitt B, Dreisinger DB. The kinetics of nickel recovery from ferrous containing solutions using an Iminodiacetic acid ion exchange resin. Hydrometallurgy. 2018:333-339. https://doi.org/10.1016/j.hydromet.2017.11.002.

19 Alexandratos SD. Ion-exchange resins: a retrospective from industrial and engineering chemistry research. Industrial & Engineering Chemistry Research. 2009;48:388-398. http://dx.doi.org/10.1021/ie801242v.

20 Kuz'Min VI, Kuz'Min DV. Sorption of nickel and copper from leach pulps of low-grade sulfide ores using Purolite S930 chelating resin. Hydrometallurgy. 2014;141:76-81. http://dx.doi.org/10.1016/j.hydromet.2013.10.007.

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