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Preparation of layered nickel aluminium double hydroxide from waste solution of nickel



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ABSTRACT

The separation of nickel has been carried out from a waste solution containing 3.18 g/L Ni with other impurities such as Fe, Zn, Cu and As. Iron was removed by precipitation and Cu and Zn were removed by solvent extraction using LIX 622N and NaTOPS-99, respectively. After removal of all these impurities nickel was extracted by 1.5 M NaTOPS-99 in two counter-current stages at A:O ratio of 3:1 and the loaded organic was stripped with 30 g/L H_2SO_4 at phase ratio of unity. The strip solution of nickel was treated with $Al_2(NO)_3 \cdot 9H_2O$ for co-precipitation by increasing the pH of solution with 1 M NaOH up to 10. The Ni–Al layered double hydroxide was confirmed through XRD characterization.

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1. Introduction

The demand of nickel has been increasing day by day because of its use in various products such as electronic devices, magnets, rechargeable batteries, stainless steel, super alloy coins and catalysts. So any waste containing nickel should be treated for recovery of nickel. In the copper plant, during electropurification of copper, the bleed stream obtained from liberated cell contains significant amount of Cu, Ni, H₂SO₄ and trace amount of As, Fe etc. (Agrawal et al., 2008). From such a bleed stream Cu and Ni were recovered by solvent extraction.

Solvent extraction is the most suitable technique to obtain pure Ni from a mixed solution. Many authors have worked on extraction of Ni using various extractants such as D2EHPA (Reddy and Bhaskara Sarma, 2001; Cheng, 2000), PC88A (Lin et al., 2006), Cyanex 272 (Sarangi et al., 1999) and LIX84I (Tanaka and Alam, 2010; Parija and Bhaskara Sarma, 2000). The extraction of nickel was enhanced by the synergistic effect of LIX 860 and D2EHPA (Zhang et al., 2001). The mixed extractant extracted nickel and cobalt very effectively from an acidic aqueous solution with pH < 3.0. Separation of nickel and cobalt was carried out from 1 M chloride solution using Na-Cyanex 272 and the mixture of Na-Cyanex 272 with Na-PC88A (Sarangi et al., 1999). The separation factors in the case of Na-PC88A as extractant and Na-Cyanex 272 as synergist was 5.6 times higher than that using Na-Cyanex

272 alone. Nickel extraction was studied from ammoniacal medium by LIX 54 in an aliphatic diluent (Alguacil and Cobo, 1998). The effect of different parameters such as pH, equilibration time, temperature, counter anion sulphate and extractant concentration on extraction was studied. The sulphuric acid was used as the stripping agent. The extraction of nickel decreased with increase of temperature. Also due to the presence of sulphate as ammonium sulphate nickel extraction decreased. The nickel extraction was studied from ammoniacal-ammonium carbonate solution using LIX 87QN (Bhaskara Sarma and Nathsarma, 1996). The extraction of nickel decreased with increasing equilibrium pH from 8.6 to 10.0 and with increasing ammonium carbonate concentration in aqueous phase. The complete nickel extraction was possible from a solution containing 5.21 g/L Ni and 60 g/L ammonium carbonate in two counter current stages at an A:O ratio of 2:3. The loaded organic contained 0.15 g/L ammonia, which was removed by single stage scrubbing with dilute sulphuric acid at an equilibrium pH > 7.0.

The layered double hydroxides (LDHs), also known as anionic clays or hydrotalcite (HT)-like constitute a family of layered materials where Mg^{2+}/M^{3+} substitution takes place. The unique properties of LDHs are that they have anionic exchange capacity as well as the ability to capture organic and inorganic anions. The application of these materials includes a wide range of fields such as catalysts, ion-exchange, adsorption, pharmaceutics, photochemistry and electrochemistry (Li and Duan, 2006). The layered Nickel–Aluminium double hydroxide (Ni–Al LDH) is one such layered double hydroxide and much work has been reported on preparation of



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these materials (Kooli et al., 1995; Ehlsissen et al., 1993; Abdolmohammad-Zadeh et al., 2011; Perez-Bernal et al., 2009; Bienvenu and Minoru, 2010).

After recovery of valuable metals from the bleed streams of copper refining plant the solution is sent to the effluent treatment plant. But this solution also contains 2–3 g/L Ni along with other impurities such as Cu, Fe, Zn and As. The objective of the present work is to recover this small amount of nickel from the waste solution and to prepare a valuable product such as Ni–Al layered double hydroxide.

2. Experimental

2.1. Chemicals and reagents

The commercial extractant TOPS-99 which is di-(2-ethyl hexyl) phosphoric acid was obtained from Heavy Water Plant, Talcher, Odisha, India. Tri *n*-butyl phosphate (TBP) was obtained from s.d. Fine-CHEM LTD BOISAR and LIX 622N (which is a mixture of 5-dodecylsalicylaldoxime and tridecanol) was obtained from Cognis Corporation, USA and was used as such without any purification. Distilled kerosene (b.p. 180–240 °C) was used as the diluent. All other chemicals such as H₂SO₄, HCl, NaOH, Al₂(NO₃)₃9H₂O, Ca(OH)₂ used in this study were from AR/Qualigens, India.

2.2. Apparatus

A digital Systronics µpH Meter Model 361 provided with a combined glass electrode was used to measure the pH of aqueous solutions. The metal ion concentration in aqueous solution was measured by Atomic Absorption Spectrophotometer (Perkin-Elmer Model AAnalyst-200). The mixer-settler unit used in this work was a box-type mixer-settlers made of PVDF. The active volume of one mixer-settler or stage was 600 mL while the ratio of the mixer and settler volumes was 1:4 (120 and 480 mL, respectively). The mixer and settler have dimensions $L \times W \times D$ as: 5.5 cm \times 4.0 cm \times 8.5 cm and 14.0 cm \times 4.0 cm \times 8.5 cm, respectively. Each mixer unit was provided with a pump-mixer impeller also made of PVDF, which ensured the flow of both phases between the stages. The diameter of impeller was 3.5 cm. The peristaltic pumps of Watson Marlow, Model 505S were used for pumping feed solution, solvent and strip solution. X-ray diffractometer model RIGAKU ULTIMA IV in the 2θ range of 10–80° with voltage of 30 kV and Cu Ka was used for characterization.

2.3. General extraction procedure

The extractant, TOPS-99 was neutralized up to 70% by intimately mixing it with stoichiometric quantity of concentrated NaOH solution to form a single phase and was used as extractant. Tri *n*-butyl phosphate (5 vol%) was added as phase modifier. The pH of aqueous phase was adjusted to the desired value by adding NaOH/H₂SO₄ solution. For extraction and stripping experiments the organic and aqueous phases were contacted for 5 min in separating funnel and allowed for complete phase disengagement. After phase separation, the raffinate was collected and the equilibrium pH was measured. The concentration of metal ion in the raffinate was determined by AAS after proper dilution. The concentration of metal ion in the organic phase was calculated from the difference between the concentration of metal ion in aqueous phase before and after extraction. When ever necessary, the organic phase was stripped with H₂SO₄ after filtering through 1PS filter paper and was analysed for metal ion concentration.

The solution of spent electrolyte contains 3.18 g/L nickel, 0.42 g/L copper, 0.6 g/L iron, 0.27 g/L zinc and 7.89 g/L arsenic with 252 g/L free acid. As the purpose of the study is to prepare layered nickel aluminium double hydroxide from this waste solution, the impurities such as copper, zinc and iron were removed from the solution leaving behind only nickel and arsenic. From nickel and arsenic solution, nickel was selectively extracted with NaTOPS-99 and was stripped with H_2SO_4 to obtain pure nickel solution and from this nickel solution layered nickel aluminium double hydroxide was prepared by co-precipitation with aluminium salt.

3. Results and discussions

3.1. Removal of iron by precipitation

The waste solution of the bleed stream was treated with lime for removal of iron and neutralisation of acid. The initial pH of the solution was 0.61. This solution was taken in a beaker and Ca(OH)₂ was added till the pH increased to 3.5. The solution was agitated with a mechanical stirrer to have proper mixing. The samples were collected at different pH, filtered and the filtrates were analysed for metal ion concentration. The percentage of metal precipitated at different pH values was shown in Fig. 1. The figure indicated an increase of iron precipitation from 95.3% to 99.5% with increase of pH from 2.5 to 3.5. The co- precipitation of other metal ions such as copper, zinc, nickel and arsenic also increased with increase of pH from 2.5 to 3.5. At pH 3.5, the co-precipitation of copper, zinc, nickel and arsenic was 37.32%, 43.75%, 29.94% and 46.48%, respectively. As the loss of nickel was not desired, the precipitation of iron was done at pH 2.5 where loss of nickel was only 8.4%. After precipitation, the solution was filtered with Whatman 42 filter paper and the residue was washed repeatedly with distilled water. The filtrate contains 0.03 g/L iron, 0.26 g/L copper, 0.20 g/L zinc, 2.91 g/L nickel and 4.47 g/L arsenic.

3.2. Extraction of copper

The filtrate after iron precipitation contains 0.26 g/L copper and to extract this copper from the solution LIX 622N was used. From the literature it was found that LIX 622N is very selective for copper extraction and it extracts copper at pH ~ 2.5 (Panigrahi et al., 2009). So without any optimization study copper was extracted from the solution by stirring it with 5% LIX 622N with a mechanical stirrer at A:O phase ratio of 10:1 for 5 min. The analysis of raffinate showed 4.8 mg/L of copper indicating 98.15% copper extraction. The equilibrium pH of the solution decreased to 2.3 and



Fig. 1. Effect of pH on precipitation of metal ions.

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