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Selective leaching of a mixed nickel-cobalt hydroxide precipitate in sulphuric acid solution with potassium permanganate as oxidant

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ABSTRACT

Selective leaching of a mixed nickel-cobalt hydroxide precipitate was investigated using potassium permanganate as oxidant in sulfuric acid solution. 94.9% Ni, 50% Co and 0.6% Mn were dissolved under the following conditions: sulfuric acid concentration of 0.75 M, potassium permanganate of 5 g/L, temperature of 30°C, leaching duration of 60 min, solid-to-liquid ratio of 1/10, and stirring speed of 400rpm. The pregnant leach solution was subjected to a solvent extraction process. 98% Co and 99% Mn were extracted at pH 4.84 with 30% (v/v) Cyanex 272, leaving essentially all nickel in the raffinate. Based on the experimental results, a flowsheet is proposed.

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KEYWORDS
Selective leaching; potassium permanganate; mixed nickel-cobalt hydroxide precipitate; solvent extraction; nickel

Introduction

Nickel is an increasingly essential metal in modern life given its important role to produce stainless steel and various nonferrous alloys with high impact strength, high corrosion resistance, and other desirable electrical, thermal, and magnetic properties. Currently, more than half of the global nickel production is derived from laterite ores, which account for approximately 60% of the global land-based nickel resources, following the depletion of nickel sulfide ores, the traditional source of the metal.[1–4] The processing of laterite nickel ores, however, is more difficult than that of sulfide ores as they are not readily amenable to flotation requiring the bulk of the ore to be processed to recover the nickel. Hence, a hydrometallurgical route is more preferred than a pyrometallurgical route to treat laterite nickel ores. In addition, it is the only route that allows the recovery of cobalt – a valuable by-product – from the ores.

Depending on the downstream processing, there are currently three commercial hydrometallurgical routes to process laterite nickel ores, namely mixed sulfide precipitation (MSP), mixed hydroxide precipitation (MHP), and direct solvent extraction (DSX). The MHP route is increasingly favored over the other two routes particularly for smaller operations.[5] This is because this route circumvents the technical complexity and intensive capital that are required by the other two routes. The DSX route that is used in the Goro process, for example, requires strict control of minor impurities such as copper and iron that can cause irreversible reaction with the extractant causing loss of the expensive reagent.[6,7] The MSP process uses hazardous H_2S gas to selectively precipitate nickel and cobalt, along with impurities such as copper and zinc, from the major impurities especially manganese, magnesium, and calcium.[8,9] The MHP route, however, is not highly selective against manganese. The refining process of the MHP product, therefore, needs to incorporate a measure to separate manganese from the nickel and cobalt before their recovery.

The use of sulfuric acid solution to leach MHP products has been deemed unsuitable as the lixiviant dissolves also high amounts of manganese. This makes the recovery of the nickel and cobalt difficult because the current commercially available SX extractant, such as bis (2,4,4-trimethylpentyl)phosphinic acid (Cyanex 272), has very limited selectivity to separate manganese from cobalt. Recently, however, there has been an increasing interest in the development of selective leaching processes using oxidizing agent in sulfuric acid solution to separate nickel in the mixed hydroxide precipitate from manganese and cobalt. Researchers from the University of Queensland have used crystalline sodium persulfate ($Na_2S_2O_8$) as an oxidant to selectively oxidize manganese and cobalt during leaching in a weakly sulfuric acid solution to stabilize the two metals in the solid phase while allowing nickel to dissolve according to Equation

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(1), where M is Mn or Co, and (2).[5,10–12] Others have studied a similar process but using ozone as oxidant.[13] These processes have the potential to circumvent the need for SX altogether. The dissolved hydrogen persulfate and ozone, however, are highly corrosive even for stainless steel materials requiring the process to be carried out in a sophisticated material of construction.

$$M)OH(_{2)s(} = MOOH_{)s(} + H^{+}_{)aq(} + e^{-} \qquad (1)$$

$$Ni)OH(_{2)s} + (2H^{+})_{aq(} = Ni^{2+}{}_{(aq(} + 2H_2O_{)l)} \qquad (2)$$

In the present study, the use of crystalline potassium permanganate as an alternative oxidant to stabilize manganese in their solid phases to allow its separation from nickel and cobalt was investigated. Dissolved permanganic acid is much less corrosive than hydrogen persulfate and ozone, and at a concentration higher than 150 ppm, it causes passivation on a carbon steel surfaces leading to negligible corrosion rate of the material.[14] This approach allows the production of high purity nickel and cobalt streams via SX with Cyanex 272 in the subsequent refining step. This study is, therefore, aimed to evaluate the separation performance of this approach.

Materials and methods

Materials and reagents

The MHP product used in this study was supplied by Meta Nickel Plant in Gordes, Turkey. The precipitate was crushed with a jaw crusher and ground in a laboratory ball mill. The particle size distribution of the product was determined by Master Sizer 2000 (Malvern). The elemental composition was measured using X-ray fluorescence (XRF) spectrometer (Zetium, PANalytical), while its mineralogical composition was examined using X-ray diffraction (XRD) (Bruker Discover) with CuKa1 (wavelength 1.5406 A) operating at 40 kV and 40 mA. The XRD 2θ diffraction angle was calibrated using 2θ/d spacing calibration standard. Subsequently, the mineral phase compositions were identified using Diffrac Suite EVA software equipped with the current ICDD PDF-2/Minerals database. To confirm the XRD data before the oxidative acid leaching, the representative sample was examined by field emission scanning electron microscopy (FE-SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) (Zeiss GeminiSEM 300).

The following analytical grade reagents were used to prepare the inorganic solutions required for the experiments: sulfuric acid (95–97% purity, Merck), potassium permanganate (>99% purity, Sigma-Aldrich), and sodium hydroxide (>99%, Merck). Deionized water was used for dilution when needed. The organic solutions for the SX experiments were prepared by dissolving the extractant bis (2,4,4-trimethylpentyl) phosphinic acid, Cyanex 272 (Industrial Grade, Cytec), in analytical grade grade n-Hexane (>99% purity, Merck). The extractant Cyanex 272 was provided by Cytec Russia. The prepared organic solutions were then pre-treated by manually shaking them with equal volumes of deionized water in a separation funnel to remove any water-soluble constituents. The organic solutions were then filtered through Whatman 1PS phase separation paper to prevent any water entrainment.

Experimental method

The leaching tests were performed in a 500-mL glass beaker covered with aluminum foil to minimize evaporation losses. The leach slurry was mixed by a digital overhead mechanical stirrer (MTOPS-MS3040D) with a three-blade stainless steel impeller (45 mm diameter) at 400 rpm. The beaker and slurry were maintained at the required temperature using a heating mantle (MTOPS-MSDSM). For each experiment, 50 g sample was transferred into the beaker after adding 500 mL of sulfuric acid at a predetermined concentration. The slurry was heated to the desired temperature and then, a pre-determined amount of potassium permanganate was added into the slurry. After the leaching experiment was finished, the pH of the slurry was measured using a pH meter (Hach, HQ40d) equipped with a pH probe (IntelliCAL PHC 28101). Subsequently, the slurry was filtered with Whatman 1 filter paper and the filtered residues were washed several times with deionized water. The filtrates were then subjected to atomic absorption spectroscopy (AAS, Thermo Scientific ICE 3300) for elemental analysis. In all leaching tests, the solid-to-liquid ratio was kept constant at 1/10 (w/v). The leaching tests were triplicated to assess the reproducibility of the test results. The average result of the three experiments was given with ± 0.5% confidential level.

The SX experiments were carried out in a 250-mL glass beaker equipped with a heating mantle. The temperature of the solution was maintained at 40°C. The aqueous solution for the SX experiments was obtained from the optimum oxidative leaching experiment. The PLS was then mixed with an equal volume of the organic solution (A/O = 1, 25 mL/25 mL) using also a digital overhead mechanical stirrer (MTOPS-MS3040D) at 400 rpm for 10 min. The pH of the PLS was controlled by small additions of NaOH (5 mol L^{-1}). After mixing, the mixture was allowed to stand for 5 min to disengage and some aqueous solution (10 mL) was taken by a syringe for pH measurement. The sample was filtered using a 0.45-µm syringe membrane filter and then, the filtrate was subjected to AAS analysis for nickel, cobalt, and manganese analysis. The concentration of metal ions in the organic phase was calculated via mass balance.

Results and discussion

Materials characterization

Prior to experimental tests, the particle size distribution of the MHP sample was measured. The results showed that 90% of the product is less than 43.1 μm. The nickel, cobalt, and manganese concentrations in the MHP product, as shown in Table 1, were obtained via hot aqua regia digestion using a hydrochloric to nitric acid ratio of 3 to 1. The elemental analysis from the liquor was performed using AAS. The XRD pattern of the sample (Fig. 1) exhibited major peaks from nickel hydroxide hydrate (card no: 38–0715-Ni(OH)2 · 0.75H2O), manganese oxide hydrate (card no: 44–0140 MnO2 · 0.3H2O), manganite (card no: 41–1379 Mn+3O(OH)) and cobalt hydroxide (card no: 30–0443 Co(OH)2). The XRD results revealed the data and peak of the sample were widened which is indicative of amorphous solid structure. The peaks at 2θ of 12 and 23 are also determined as characteristics of α-$Ni(OH)_2.0.75H_2O$, which is consistent with findings of previous studies.[5,11,15] Other means of solid analysis including energy-dispersive X-ray (EDX) with a scanning electron microscope (SEM) were also carried out and the results showed closed similarity to that found with the AAS analysis (Fig. 2).

Table 1. Elemental composition of the MHP sample.

Elements (MHP)	Concentration (wt. %)
†Ni	34.50
†Co	1.90
†Mn	5.40
*Mg	2.42
*Ca	0.68
*Al	0.90
*Cr	0.03
*Fe	0.56
*Sc	0.01
*Zn	0.45
*Cu	0.21
*Na	0.10
*S	5.39

*: XRF analysis; †AAS analysis.

Leachability of metals from the MHP product without oxidant

The leachability of nickel, cobalt, and manganese in the absence of an oxidant, which will be used as the benchmark for the subsequent oxidative leaching experiments, was assessed by carrying out a leaching experiment using 0.75 M sulfuric acid at 70°C. The solid-to-liquid ratio and stirring speed were kept constant at 1/10 and 400 rpm, respectively. The results as presented in Fig. 3 show the dissolutions of the metals from the MHP product as a function of time. The dissolution of nickel, cobalt, and manganese significantly increased with increasing leaching duration in the first 60 min and then reached a plateau. After 60 min of leaching, 90.7% of nickel was dissolved whereas the dissolution of cobalt and manganese were 86.4% and 41.9%, respectively. Note that even after 60 min, theoretically, there should be more than 1.0 M hydrogen ion remained in the solution to dissolve the remaining nickel and cobalt from the solid. Williams et al. (2013), who also performed oxidative leaching of an

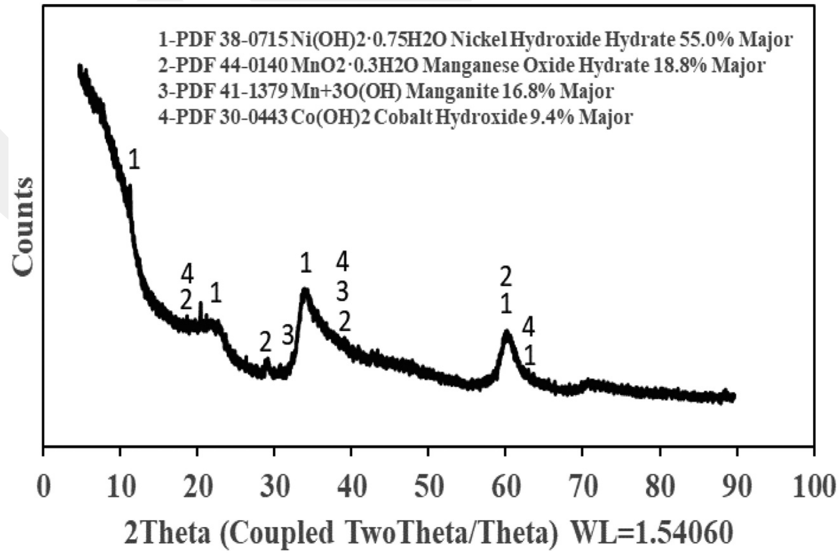
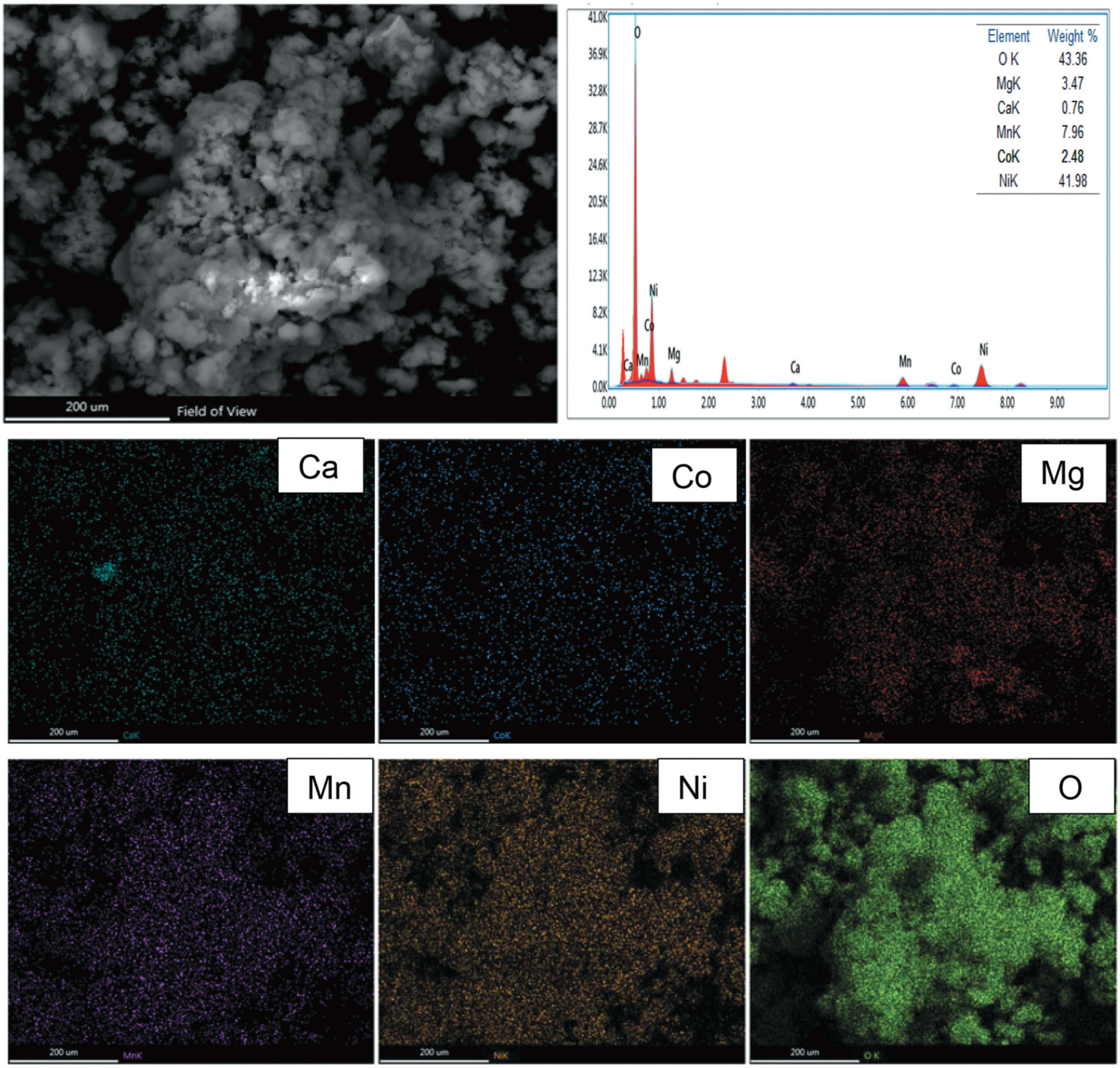


Figure 1. XRD pattern of the MHP sample.



Element	Weight %
O K	43.36
MgK	3.47
CaK	0.76
MnK	7.96
CoK	2.48
NiK	41.98

Figure 2. SEM mapping analysis of the representative MHP product.

MHP product containing α-Ni(OH)$_2$, attributed the incomplete dissolution of nickel in their study to Ostwald ripening, wherein the atoms in the metastable solids slowly rearrange into a more crystalline configuration.[16] Kursunoglu (2019), who performed reductive leaching of the same MHP product as used in this study, showed that the incomplete dissolution of the nickel and cobalt may also partly because the two metals were trapped within highly oxidized manganese. He showed that as the manganese dissolution was increased when citric acid was added into the sulfuric acid solution, the dissolution of nickel and cobalt was also increased. The presence of higher states of manganese such as oxide hydrate (Mn^{4+}) and manganite (Mn^{3+}), which were dominant host mineral phases in the MHP product, was apparent from the relatively low manganese dissolution in this study.[17]

Selective leaching of the MHP product with oxidant

Manganese exists in a number of oxidation states, but only the Mn(II), Mn(III), and Mn(IV) species contribute significantly to the aqueous chemistry of the metal. Under reducing conditions, Mn(II) is the most stable species. Mn(II) oxidation with strong oxidant addition has been practiced in the water industry for many years. As used in the current study, solution-phase oxidation is defined as the interaction of aqueous-phase Mn(II) ions

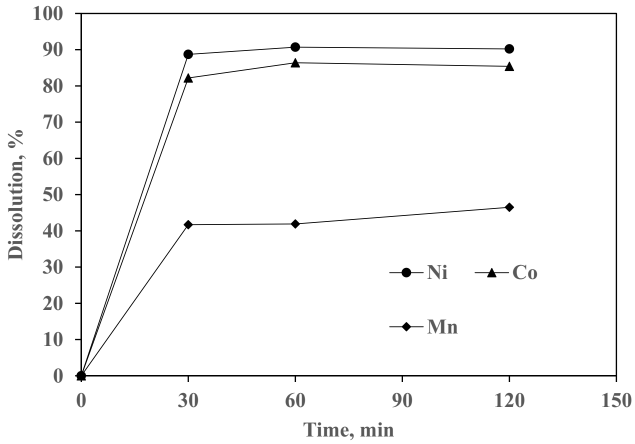


Figure 3. Effect of leaching duration on the metal dissolutions (Conditions: sulfuric acid concentration: 0.75 M, solid-to-liquid ratio: 1/10 and temperature: 70°C).

with oxidant to produce the manganese solid-phase $MnO_2(s)$. The stoichiometry of Mn(II) oxidation by $KMnO_4$ based on balanced half-reaction is as follows:

$$3Mn^{2+} + 2MnO_4^- + 4OH^- = 5MnO_2(s) + 2H_2O \tag{3}$$

The standard reduction potential (E^0) of permanganate anion to manganese dioxide according to Eq. (4) is higher than the E^0 value of manganese(IV) dioxide to manganese(II) (Eq. 5) but lower than the E^0 values of cobalt(III) hydroxide to cobalt (Eq. 6) to cobalt(II) and nickel(III) hydroxide to nickel (Eq. 7). Theoretically, therefore, permanganate ion can be used as an oxidant to separate manganese from nickel and cobalt as apparent from the thermodynamics prediction for oxidative precipitation of divalent manganese, cobalt, and nickel shown in Fig. 4. Also included on the diagram are oxidative precipitation of divalent iron and a dashed line showing the reduction of oxygen to water for perspective. Note that while the oxidation of water is thermodynamically favored, the oxidation of manganese and cobalt are more kinetically favored.[11] No gas evolution was observed during the experiments suggesting that no significant water oxidation was occurred.

$$MnO_4^- + 4H^+ + 3e^- = MnO_2(s) + 2H_2OE^0 = 1.706\ V \tag{4}$$

$$MnO_2(s) + 4H^+ + 2e^- = Mn^{2+}(aq) + 2H_2OE^0 = 1.216\ V \tag{5}$$

$$Co(OH)_3(s) + 3H^+ + e^- = Co^{2+}(aq) + 3H_2OE^0 = 1.741\ V \tag{6}$$

$$Ni(OH)_3(s) + 3H^+ + e^- = Ni^{2+}(aq) + 3H_2OE^0 = 2.212\ V \tag{7}$$

The oxidative leaching tests to observe the effect of $KMnO_4$ dosage to separate nickel and cobalt from manganese were performed in 0.5 M sulfuric acid solutions at a temperature of 70°C and a solid-to-liquid ration of 1/10. The oxidant concentration was varied between 0.0 g/L to 10 g/L. The results,

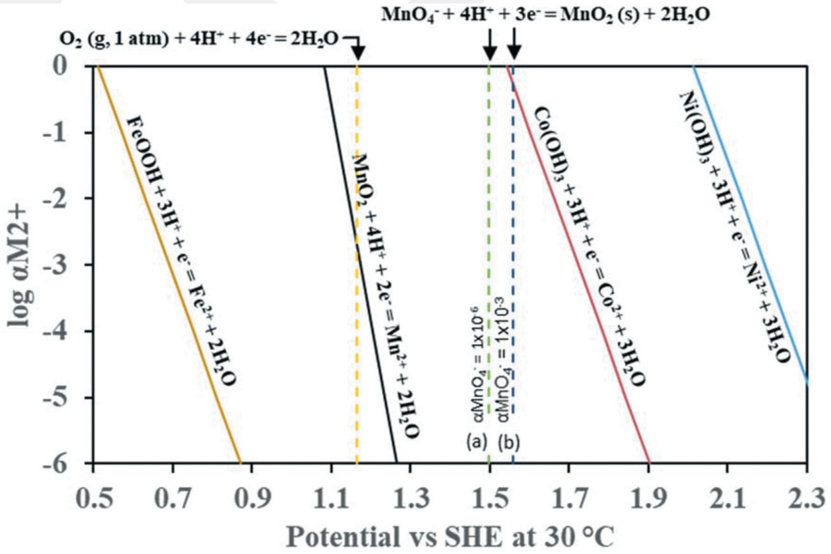


Figure 4. Oxidative precipitation diagram at 30°C and pH = 1.1. Dashed lines showing the reduction of oxygen to water and permanganate to manganese dioxide with (a) $\alpha MnO4^- = 1x10^{-6}$ and (b) $\alpha MnO4^- = 1x10^{-3}$. Standard potential data from HSC software version 6.00 (2006).

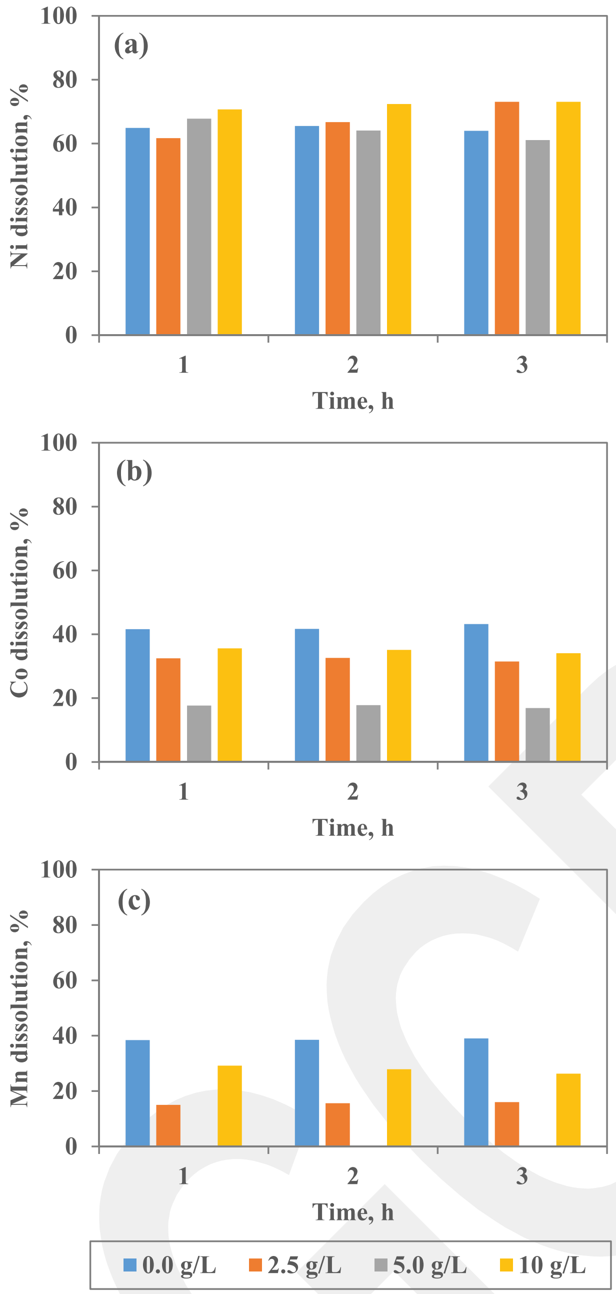


Figure 5. Effect of $KMnO_4$ dosage on the dissolutions of (a) nickel, (b) cobalt and (c) manganese (Conditions: sulfuric acid concentration: 0.5 M, solid-to-liquid ratio: 1/10, temperature: 70°C).

presented in Fig. 5, show that the nickel dissolution were generally increased with increasing $KMnO_4$ concentration except at the third hour using 5.0 g/L $KMnO_4$, wherein the nickel dissolution (61.1%) was slightly lower to that when no oxidant was added (64.0%). On the other hand, the increase in the oxidant concentration reduced the cobalt and manganese dissolution but only until 5.0 g/L, after which the dissolutions of the two metals were increased significantly. A comparison of the leaching performance in 0.75 M sulfuric acid solution without and with the addition of oxidant at the optimum concentration is given in Table 2.

It was understood from the oxidative precipitation diagram shown in Fig. 4 that some divalent cobalt may be oxidized into its trivalent species but the results obtained in this study showed that the co-precipitation of cobalt was much higher than its thermodynamic prediction. This can be attributed to cobalt adsorption on the MnO_2 matrix, which was formed in a significant amount throughout the slurry. The proclivity of cobalt adsorption to MnO_2 matrix was evident in the work of Nathsarma et al. (2013) who showed cobalt co-precipitation during a manganese precipitation process using air/oxygen gas from an ammonia-ammonium sulfate leach solution, in which solution cobalt should be stable in the aqueous phase as ammine complexes.[18]

The effect of temperature on the metal dissolutions in 0.75 M sulfuric acid solution as a function of $KMnO_4$ dosage ranging from 2.50 to 5.00 g/L was presented in Fig. 6. This acid concentration was selected because 0.5 M was not sufficient to provide high nickel dissolution. The results show that temperature only had small effect on the dissolutions of the three metals. Notably, the increase in temperature improved the selectivity of the nickel and cobalt against manganese as manganese dissolution was reduced from 0.6% at 30°C to 0.2% at 70°C, but this improvement was not sufficient to justify the operation at 70°C considering the energy required to increase the temperature. Hence, it was determined that the most optimum conditions for the selective leaching with oxidant was using 0.75 M sulfuric acid at 30°C with a solid-liquid-ratio of 1/10 and leaching duration of 60 min. The reduction in dissolution was more pronounced with manganese as apparent that less than 1% dissolution was observed in 5.0 g/L $KMnO_4$. This was owing to the oxidation of divalent manganese to its trivalent or tetravalent species during the leaching process as evident from the XRD result of the leach residue shown in Fig. 7.

Solvent extraction (SX) of nickel and cobalt from the leach solution

Before the SX experiments, leaching tests were conducted at using 0.75 M sulfuric acid, 5.0 g/L g $KMnO_4$

Table 2. A comparison of the extraction performance at fixed conditions without and with oxidant.

		Without oxidant		With 5 g/L $KMnO_4$	
Conditions		Element	Dissolution, %	Element	Dissolution., %
H_2SO_4, M	0.75	Ni	90.7	Ni	93.7
T, °C	70	Co	86.4	Co	53.5
t, min	60	Mn	41.9	Mn	0.2

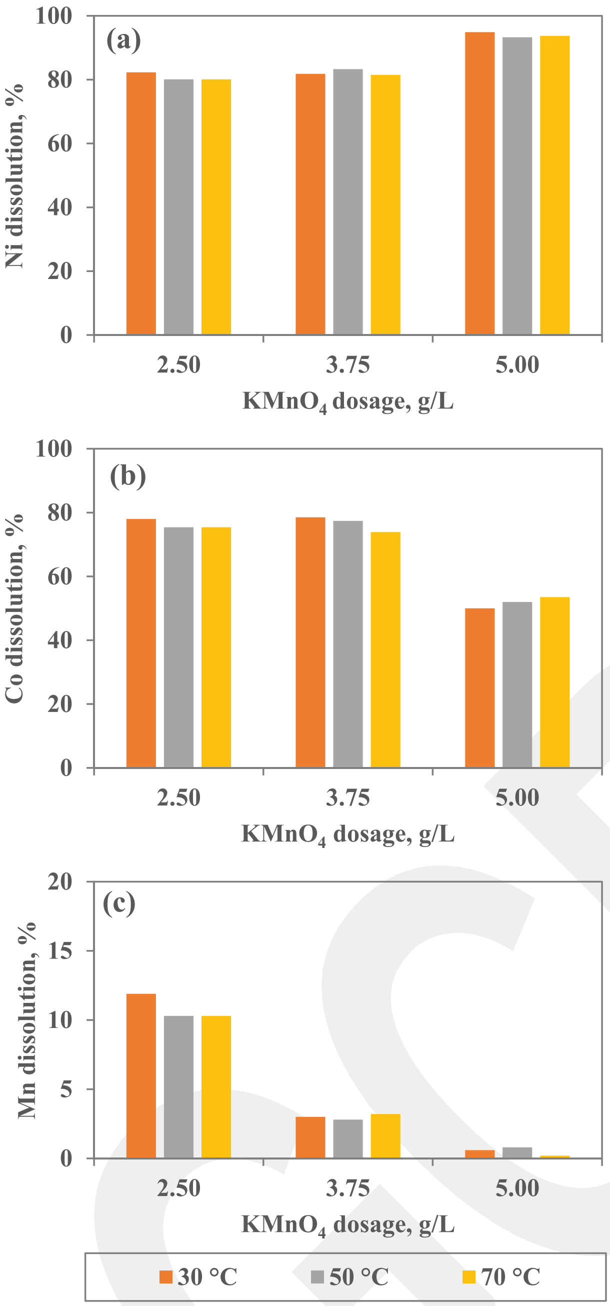


Figure 6. Effect of temperature and KMnO$_4$ dosage on the dissolution of (a) nickel, (b) cobalt and (c) manganese. (Conditions: sulfuric acid concentration: 0.75 M, solid-to-liquid ratio: 1/10, leaching duration: 60 min).

as oxidant at 30°C for 60 min, which condition was determined to be the most optimum. The produced PLS consisted of 32710 mg/L Ni, 1020 mg/L Co and 43.60 mg/L Mn. This PLS was used for the SX experiment. The SX tests were carried out using Cyanex 272 at concentrations in the range of 20–30% (v/v) in n-hexane as diluent. The extraction percentage (E) of metal ions was calculated according to the following equation:

$$E = \frac{[M]_{org}}{[M]_{aq}} \times 100 \tag{8}$$

where [M]org and [M]aq represent the concentration of metal ions in the organic and aqueous phase (mg L^{-1}), respectively.

Third phase formations were observed using 20% Cyanex 272 at pH 4.96, whereas good phase separations were occurred using 30% Cyanex 272 at pH 4.84. Figure 8 shows the effect of equilibrium pH and the concentration of Cyanex 272 on the extraction of nickel and cobalt from aqueous solution. The increase in pH and concentration of Cyanex 272 resulted in higher cobalt extraction. The organic system containing 30% Cyanex 272 was determined as the best composition at an A/O of unity. Using 30% Cyanex 272 concentration, 98% of cobalt along with 99% of manganese was extracted at pH 4.84 in a single contact, leaving all of the nickel in the raffinate. Based on the experimental results, a flowsheet is proposed for the selective leaching and separation of nickel and cobalt from the mixed nickel-cobalt hydroxide precipitate (Fig. 9). Using this flow sheet, the precipitate was firstly leached with sulfuric acid in the presence of KMnO$_4$ as an oxidizing agent at 30°C for 60 min to remove the bulk of manganese. The leach slurry was filtered and the filtrate was sent to an SX unit. In this unit, 30% of Cyanex 272 diluted in n-Hexane was used. 999.60 mg/L of cobalt was loaded onto the organic solution along with 43.10 mg/L of manganese, leaving nickel in the raffinate. As evident from the experimental results, this flowsheet is able to separate the bulk of manganese from nickel and cobalt to produce separate streams of the two metals with very low manganese concentrations.

Conclusions

Selective leaching of a mixed nickel-cobalt hydroxide precipitate in the presence of potassium permanganate as an oxidant in sulfuric acid solution was experimentally investigated. Preliminary leaching experiments revealed that 90.7% and 86.4% of nickel and cobalt dissolutions, respectively, were achieved within 60 min but 41.9% of manganese was also dissolved using 0.75 M sulfuric acid at 70°C in the absence of potassium permanganate. It was observed that increases in potassium permanganate dosage in sulfuric acid solution sharply decreased manganese dissolution. This was due to the oxidation of divalent manganese ions to trivalent or quadrivalent ions. Cobalt divalent ions were also partially oxidized to trivalent ions. It was determined that the following conditions were the most optimum for the selective leaching process resulting in 94.9%, 50%, and 0.6% of nickel, cobalt, and manganese dissolution, respectively: 0.75 M sulfuric acid, 5 g/L potassium permanganate,

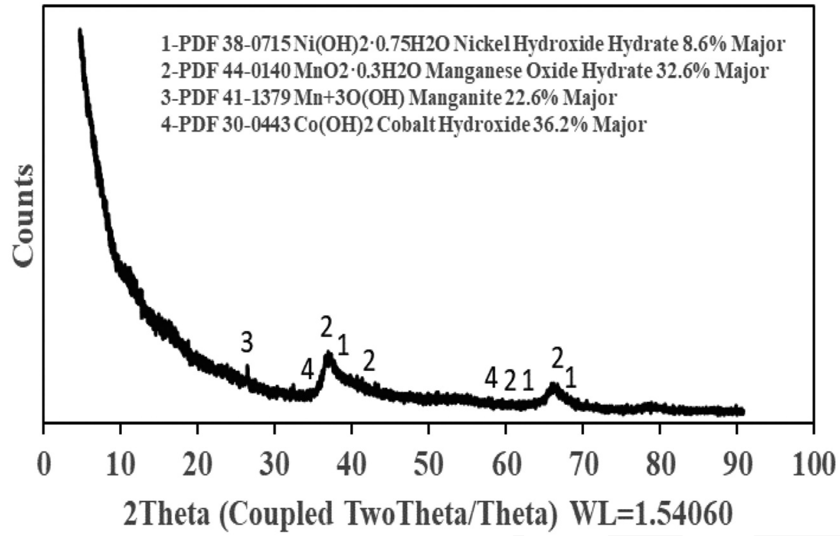


Figure 7. XRD pattern of the leached residue (sulfuric acid concentration: 0.75 M, $KMnO_4$ dosage: 5 g/L, solid-to-liquid ratio: 1/10, temperature: 30°C).

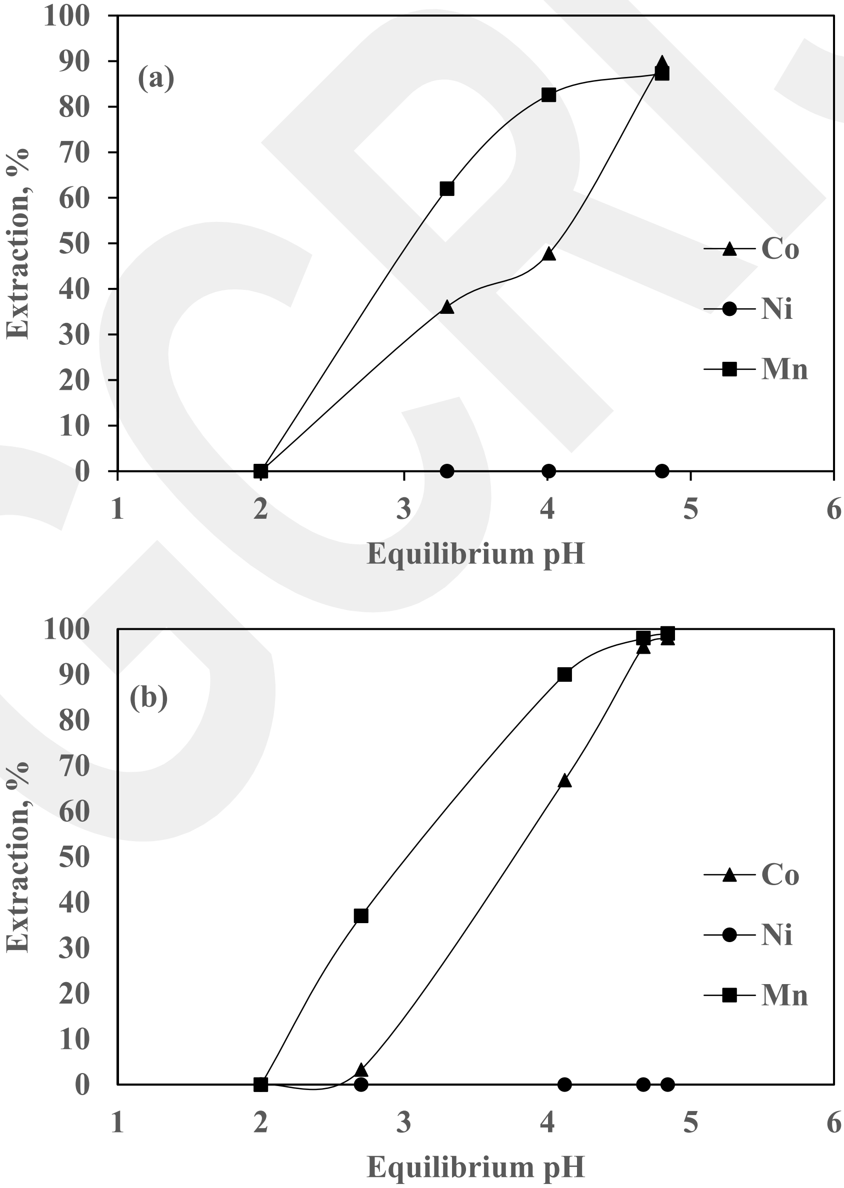


Figure 8. Extraction of nickel and cobalt with Cyanex 272 concentration of (a) 25% v/v and (b) 30% v/v.

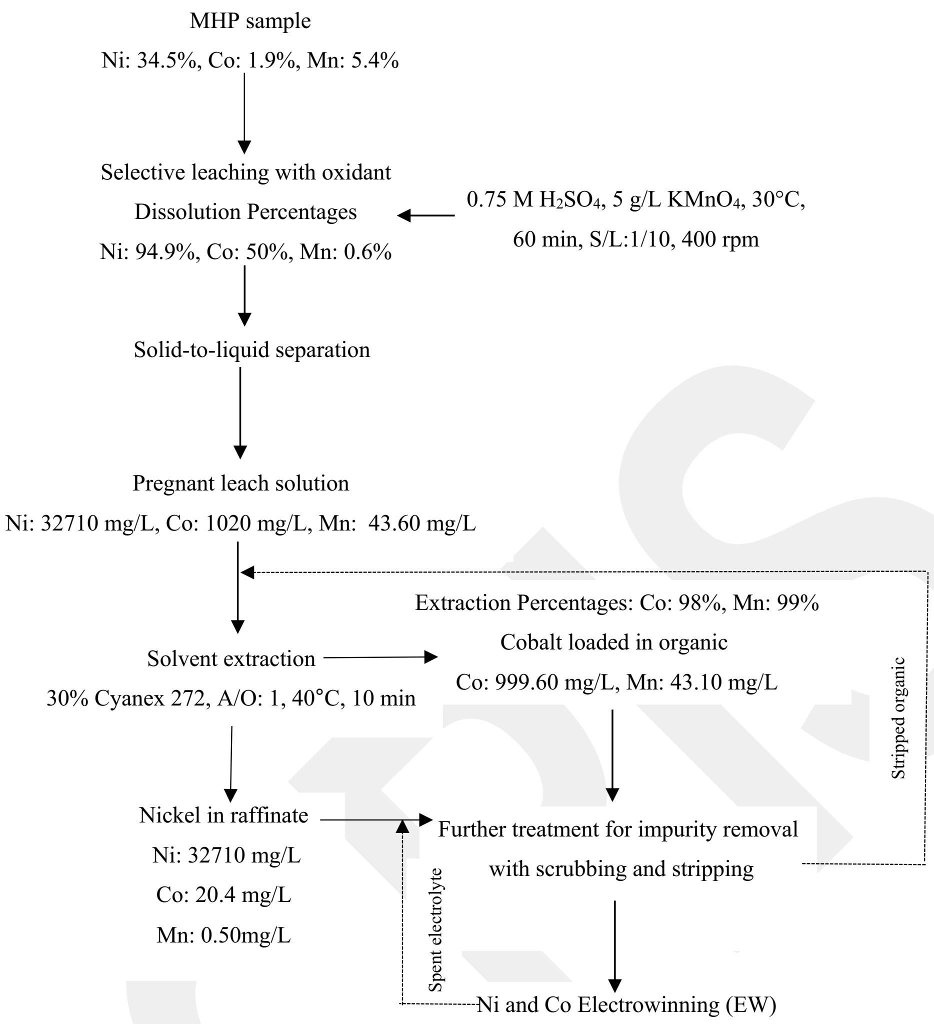


Figure 9. Proposed flowsheet for the oxidative leaching and separation of nickel and cobalt from the mixed nickel-cobalt hydroxide precipitate.

30°C temperature, 60 min leaching time, 1/10 solid-to-liquid ratio and 400 rpm stirring speed. The obtained leach solution contained 32710 mg/L Ni, 1020 mg/L Co and 43.60 mg/L Mn. By using an organic system comprising 30% (v/v) Cyanex 272, 98% and 99% of cobalt and manganese, respectively, were extracted into the organic phase at pH 4.84 in a single contact, leaving nickel in the raffinate. Based on the experimental results, a flow sheet was proposed for the selective leaching and separation of nickel and cobalt from mixed nickel-cobalt hydroxide precipitates.

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