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ORIGINAL ARTICLE

Enhanced Nickel Recovery from Mixed Hydroxide Precipitate Through Selective Leaching with $KMnO_4$ Oxidant

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ABSTRACT – A mixed hydroxide precipitate (MHP), a metal precipitate with the dominant nickel and cobalt content in hydroxide compounds, can be leached as a lithium battery precursor. In this study, potassium permanganate ($KMnO_4$) was used as an oxidant agent to increase the solubility of nickel (Ni) and cobalt (Co). The variation of the sulfuric acid concentration (0.5–1.5 M) as a leachate reagent, the concentration of $KMnO_4$ (2.5–7.5 g/L), and the selective leaching temperature (60–80°C) were investigated. Solvent extraction using CYANEX-272 (bis (2,4, 4-trimethylpentyl) phosphinic acid) and D2EHPA (Di-(2-ethylhexyl) phosphoric acid) was performed to separate the Ni, Co, and (manganese) Mn. Atomic absorption spectrometry (AAS), inductively coupled plasma mass (ICP-OES), and X-ray fluorescence (XRF) were used to analyze the chemical compositions. At the same time, crystallographic analysis was observed using X-ray diffraction. It was observed that potassium permanganate increased the dissolution of Ni and Co to 91.3% and 85.4% but decreased the dissolution of Mn (37.53%) under the following conditions: 1.75 M sulfuric acid, 7.5 g/L potassium permanganate, and 60°C temperature. High purity of nickel crystal (99.64%) was observed with spontaneous nucleation due to the supersaturated nickel solution after solvent extraction with CYANEX-272. Thus, using permanganate ion as selective leaching of Ni and Co from Mn is promising.

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INTRODUCTION

Since the introduction of lithium batteries for electric vehicles, particularly nickel-based batteries such as nickel cobalt aluminium (NCA) and nickel manganese cobalt (NMC), the process of refining nickel from mining resources has been conducted to produce precursor raw materials. The significant benefits of employing nickel in batteries are that they transfer energy more efficiently and have a bigger storage capacity at a lower cost [1]–[3].

Nickel laterite as mining resources is typically processed using various methods, such as high-pressure acid leaching (HPAL), step temperature acid leaching (STAL), and reduction roasting to produce an intermediate product known as mixed hydroxide precipitate (MHP) [4]–[6]. MHP is a high-purity nickel intermediate that contains about 34–55% nickel (Ni) and other metals such as cobalt (Co), copper (Cu), manganese (Mn) and zinc (Zn) [7]. It is frequently used as a feedstock to produce nickel compounds such as nickel sulfate, nickel chloride, and nickel carbonate, which are utilized in various applications like electroplating, catalysts, and batteries.

A local Indonesian business, PT. Hydrotech Metals Indonesia, which invented the STAL method, processed the MHP used in this investigation. This process is claimed to be one of the most efficient technologies for extracting Ni from all laterite deposit profiles of both low-grade limonite and saprolite.

Recovering nickel, cobalt, and manganese from a mixed hydroxide precipitate presents several challenges, primarily due to their similar chemical properties and the complex nature of the ore processing involved [4]. Nickel, cobalt, and manganese often occur in mineral deposits and are chemically similar, making it challenging to achieve efficient separation [8], [9]. Through a hydrometallurgical process, the precursor products from MHP, such as Ni, Co, and Mn, are obtained in several important stages, including acid leaching, extraction, stripping, and precipitation processes [8], [10]. Acid leaching is used to dissolve the metals that need to be extracted so that the contaminants can be removed. Ni, Co,

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and Mn dissolved in acidic solutions are separated through an extraction procedure utilizing commercial products such as CYANEX-272 (bis (2,4, 4-trimethylpentyl) phosphinic acid) and D2EHPA (Di-(2-ethylhexyl) phosphoric acid) [11].

Several studies have investigated the selective separation of nickel from other metals such as Co and Mn to achieve optimal nickel recovery by employing oxidants such as hydrogen peroxide (H_2O_2), ozone, and sodium persulfat ($Na_2S_2O_8$), which have been shown to increase the selectivity of nickel leaching [12]–[14]. The role of oxidants in the leaching process for mixed hydroxide precipitates is to facilitate the dissolution of metal species by promoting redox reactions, enhancing solubility, forming soluble complexes, and aiding in acid regeneration [12], [15]. However, since using commercial extractant CYANEX-272 in the extraction process, which has a pH range that is far enough in the Ni and Co recovery (pH 4–5 for Co and around 7 for Ni), it is possible to separate Mn at the beginning to get Ni and Co optimal results in the leaching process. From an economic standpoint, the acquisition of Co is preferable to that of Mn.

A selective leaching procedure with potassium permanganate ($KMnO_4$) oxidant was applied in this work to optimize nickel and cobalt recovery by eliminating some of the manganese in the leaching solution. The addition of crystalline oxidant $KMnO_4$ can stabilize the solid Mn phase so that it is not easily decomposed. After determining the ideal concentration of sulfuric acid in the leaching process, the concentration of the oxidant $KMnO_4$ and the leaching temperature were varied, with the highest concentration and leaching temperature values at 7.5 g/L and 80°C. Following that, the phenomena were observed.

EXPERIMENTAL METHOD

Materials and Preparation

The use of mixed hydroxide precipitate (MHP) in this study was provided by an Indonesian company named PT. Hydrotech Metals Indonesia. The leaching reagents, sulfuric acid (H_2SO_4) and potassium permanganate ($KMnO_4$) were purchased from Sigma-Aldrich. The pH of the aqueous solution was adjusted by dropping the ammonium hydroxide (NH_4OH) (Sigma, 28%). The solvent extraction was prepared by mixing the extractants in kerosene. The commercial extractant CYANEX-272 (bis (2,4, 4-trimethylpentyl) phosphinic acid) and D2EHPA (Di-(2-ethylhexyl) phosphoric acid) were provided by Solvey. All chemicals were of analytical grade and used as received without further purification.

The MHP powder was sieved (200 mesh) and dried (oven at 80°C for 8 h) before being used in the leaching stage to get the homogeneous and dried samples. The chemical composition was determined by AAS (Atomic Absorption Spectrometry, Thermo Fisher Scientific), which is presented in Table 1. At the same time, X-ray diffraction (XRD, Rigaku Smartlab) analysis was obtained to get the crystallographic analysis from MHP before and after calcination at a temperature of 400°C for 2h.

Table 1. Chemical composition of MHP

Composition of MHP (Wt%)					
Ni	**Fe**	**Co**	**Mn**	**Mg**	**Al**
40,937	0,210	1,256	3,199	1,087	0,183

The leaching experiment was carried out using atmospheric pressure under several parameters: 0.5–1.5 M sulfuric acid (H_2SO_4), solid/liquid ratio 100 g/L, temperature 70ºC, leaching duration 1 h and stirring speed 400 rpm. Filtrate was measured using AAS to examine the chemical composition.

A selective leaching experiment was used after finding the best leaching from the previous leaching. The sulfuric acid was mixed with the oxidizing agent of $KMnO_4$ with a concentration variation of 2.5 to 7.5 g/L and a temperature concentration of 60°C–80°C. The filtrate was examined using AAS to analyze elemental compositions. The leached residue was observed using XRD to analyze the by-product after the leaching process.

Solvent Extraction and Stripping Procedures

The first solvent extraction was accomplished to extract nickel (Ni) metal in the MHP. The extraction tests were performed using a CYANEX-272 at 50°C, A/O ratio 1/1 in 500 mL beaker glass for 15 minutes to obtain phase disengagement. The NH_4OH solution was dripped in the aqueous-organic mixture during the test until the desired equilibrium pH (pH 6). The sample was filtered using filter paper, and the filtrates were subjected to inductively coupled plasma mass spectrometry (ICP) for chemical analysis.

The stripping test was carried out using H_2SO_4 1M contacting with the loaded organic solution for 30 minutes. After extraction, the concentration of metals in the aqueous solution was calculated quantitatively by ICP. The sulphate crystal from the reaction was examined using X-ray fluorescence analysis (Fischerscope X-ray XAN 250, Germany).

The second solvent extraction was done by contacting the aqueous solution from the previous stripping with D2EHPA at 50°C for 15 min. The pH was maintained constant at 3 by wisely dripping NH_4OH in the solution and stripping it with sulfuric acid for 30 minutes. The distribution coefficient (D), the separation factor (S) and the percentage of extraction (%E) were calculated in Equation (1) [16].

$$D = \left\{\frac{[X]o-[X]a}{[X]o}\right\} x \ \frac{Va}{Vo} D = \left\{\frac{[X]o-[X]a}{[X]o}\right\} x \ \frac{Va}{Vo} \tag{1}$$

$$E = \left\{\frac{D}{D+(\frac{Va}{Vo})}\right\} x \ 100 \ \%E = \left\{\frac{D}{D+(\frac{Va}{Vo})}\right\} x \ 100 \ \% \tag{2}$$

where
[X]o: concentration of metal X in the organic phase,
[X]a: concentration of metal X in the aqueous phase,
Dx: distribution ratio of metal x,
Dy: distribution ratio of metal y,
Va: volume of aqueous solution, and
Vo: volume of organic solution.

RESULT AND DISCUSSION

Characterization of Mixed Hydroxide Precipitate (MHP)

X-ray diffraction (XRD) patterns of MHP before and after the calcination process are shown in Figure 1. The XRD patterns from MHP before calcination show the amorphous solid structure. At the same time, the XRD peaks from MHP after calcination shows a crystalline structure. The major peaks of MHP before calcination have a similar pattern to MHP studied by Hussaini et al. and Mubarok et al. [9], [10], which observed the peaks from nickel hydroxide hydrate (card no:38–0715-$Ni(OH)_2.0.75H_2O$), manganese oxide hydrate (card no: 44–0140 $MnO_2 \cdot 0.3H_2O$), cobalt hydroxide (card no: 30–0443 $Co(OH)_2$), and manganite (card no: 41–1379 $Mn^{+3}O(OH)$). The peaks in calcined MHP can be indexed as NiO (ICDD 01-089-7101), Co_3O_4 (ICDD 01-.76-1802) and Mn_4O_4 (COD 96-900-6671). These peaks agree with the chemical composition from Table 1.

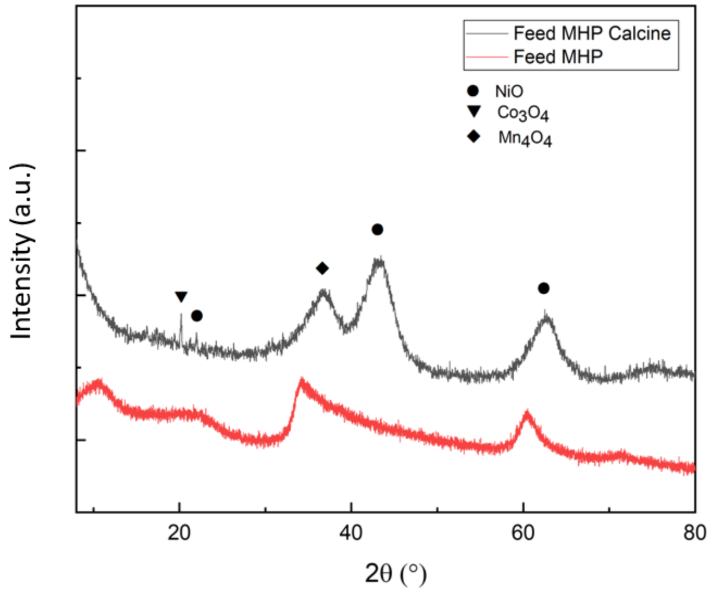

Figure 1. X-ray diffraction patterns of MHP after and before calcination

Leachability of Nickel, Cobalt, and Manganese without Oxidant

The optimum leachability of nickel, cobalt, and manganese from MHP products was investigated using a variety of sulfuric acid concentrations. Figure 2 shows the effect of acid concentration on nickel, cobalt, and manganese leaching efficiencies. The leaching efficiencies of MHP products were increased along with the increasing sulfuric acid concentration. The higher metals dissolve in higher acid concentrations due to the rise of hydrogen ions in the solution that reacted with the divalent metals. To achieve complete dissolution, each mole of the divalent metal hydroxide needs two moles of hydrogen ion [7], [17]. The dissolution of cobalt reached optimum when the acid concentration was at 1.25 M and dropped when the concentration was at 1.5 M. These phenomena, probably because of the higher sulfuric acid concentration, made the solution that reached inside the MHP products difficult to diffuse out [18], [19].

On the other hand, the incomplete dissolution of nickel (72.38%) and cobalt (50.08%) at 1.5 M sulfuric acid was found in this study. The incomplete dissolution of nickel and cobalt was also discovered by Kursunoglu et al. [15], [20]. They observed the partial dissolution of nickel and cobalt due to the trapped within highly oxidized manganese. Therefore, the acid concentration at 1.5 M was determined as the optimum concentration leaching.

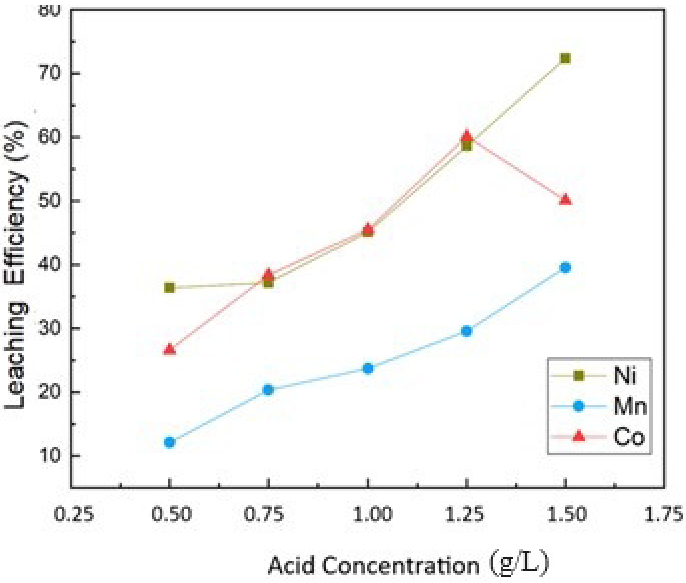

Figure 2. Effect of sulfuric acid concentration on leaching efficiencies

Selective Leaching of MHP Products with Oxidant

The effects of additive leaching oxidant, potassium permanganate, on nickel (Ni), cobalt (Co), and manganese (Mn) selective leaching efficiencies were performed in 1.5 M sulfuric acid solutions for 1 hour and the solid-to-liquid ratio of 1/10. The oxidant dosage varied from 2.5 to 7.5 g/L, and the leaching temperature ranged between 60°C to 80°C. Figure 3 presents the effect of potassium permanganate dosage and temperature on Ni dilution. The results show that adding the oxidant in the leaching process significantly improved the leaching efficiency of nickel from 72.38% to 91.3%.

Generally, increasing potassium permanganate ($KMnO_4$) dosage increased the nickel dissolution except at a temperature of 60°C, wherein the nickel dissolution was slightly lower from 88.86% (2.5 g/L) to 80.55% (7.5 g/L). The optimum dissolution was found at 70°C and 7.5 g/L concentration of potassium permanganate. Increasing the temperature and oxidant dosage enhances the kinetics of the leaching process. Higher temperatures provide more energy to the system, leading to greater molecular movement and increased collision frequency between reactant particles [14]. Higher oxidant concentrations provide more reactive species to oxidize the metal compounds in the ore or precipitate, leading to faster dissolution of metals into the solution [8]. As a result, the rate of dissolution of metal species from the ore or precipitate into the leach solution is accelerated.

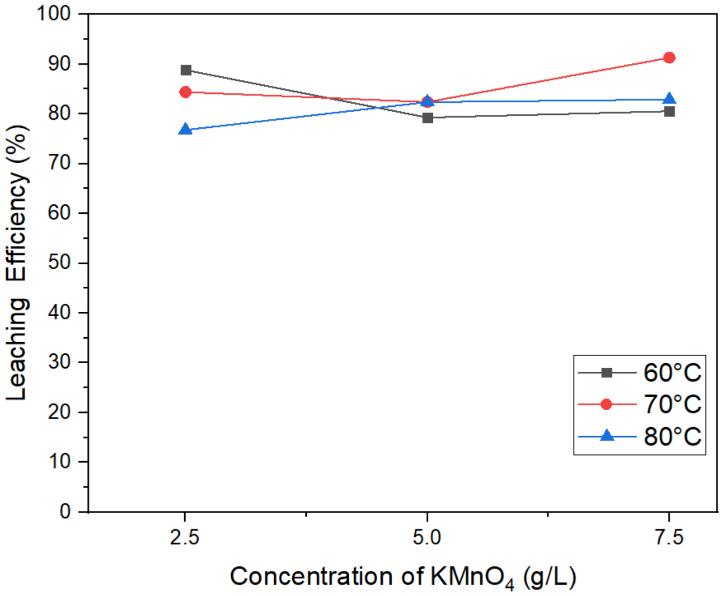

Figure 3. Effect of $KMnO_4$ concentration and temperature leaching on the dissolution of nickel (Ni)

The effect of temperature on the cobalt leaching efficiency in 1.5 M sulfuric acid as a function of potassium permanganate dosage between 2.5 M to 7.5 g/L was illustrated in Figure 4. The optimum dissolution of cobalt (89.13%) was obtained at a concentration of 5 g/L at 70ºC. However, at the same concentration for temperature 60°C, the cobalt dilution was dropped at the lowest acquisition of Co at 47%, assuming that divalent Co can be partially oxidized into its trivalent at that temperature because of the redox potential of MnO_{4-} [9], [21]. On the other hand, a temperature of 80ºC shows the constant leaching efficiency in all variations of $KMnO_4$ concentration.

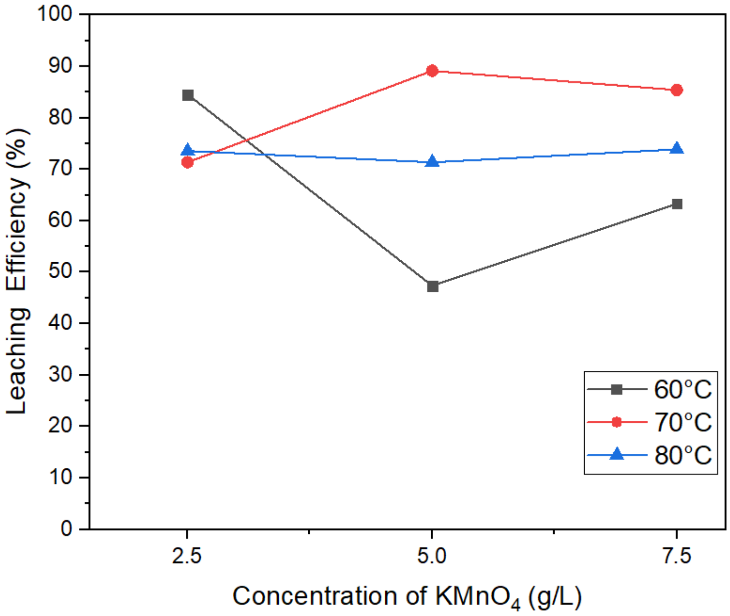

Figure 4. Effect of $KMnO_4$ concentration and temperature leaching on the dissolution of cobalt (Co)

The results of the selective leaching experiment on manganese dilution at various temperatures as a function of $KMnO_4$ concentrations ranging from 2.5 to 7.5 g/L are shown in Figure 5. The graphics show that increasing potassium permanganate concentration reduces the leaching efficiency of manganese, and higher temperature will reduce the dilution of manganese in the solution [22], [23]. The lowest leaching efficiency (31.46%) was observed at 60°C with a $KMnO_4$ concentration of 7.5 g/L. The reduction of manganese leaching efficiency due to the high amount of oxidant addition that reacted with manganese ions to produce manganese solid-phase MnO_2(s) [9].

Adding $KMnO_4$ does not oxidize Ni^{2+} ions as it has a higher reduction potential than MnO_4-. However, it will affect the solubility of Ni because the stabilization of Mn in the solid phase due to $KMnO_4$ oxidation will increase the number of leaching reagents that react with Ni. The ability to oxidize $KMnO_4$ can be seen from the redox potential. It is effective for oxidizing Mn because it has a potential magnitude greater than Mn but smaller than Co and Ni [7], [9]. Solution-phase oxidation is defined as the interaction of aqueous-phase Mn (II) ions with oxidant to produce the manganese solid-phase MnO (s). The stoichiometry of Mn (II) oxidation by $KMnO^4$ based on balanced half-reaction is as follows.

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

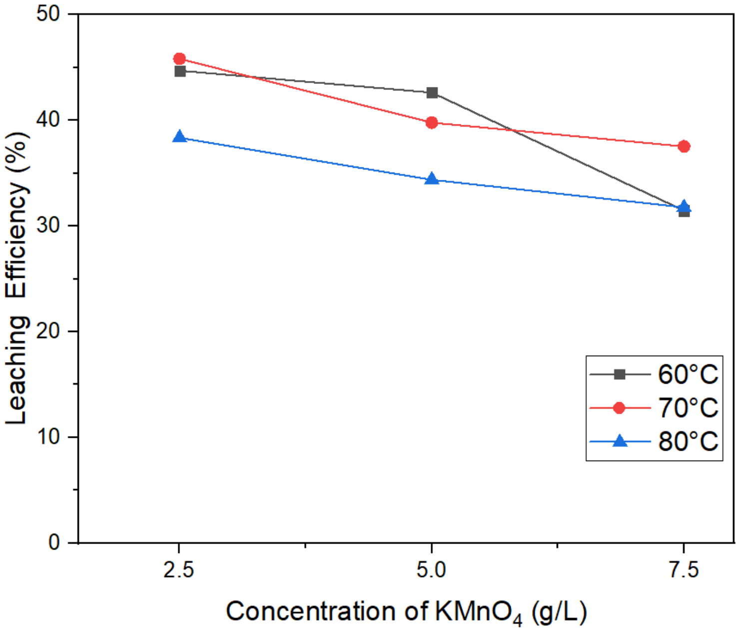

Figure 5. Effect of $KMnO_4$ concentration and temperature leaching on the dissolution of manganese (Mn)

The XRD analysis of leached residue is shown in Figure 6. The patterns were observed as manganese oxide (MnO_2: 01-072-1982), cobalt oxide hydroxide ($HCoO_2$: 01-073-0497), and manganese oxide (Mn_2O_7: 01-079-0083). The dominant phase composition in the residue was tetragonal MnO_2 and monoclinic Mn_2O_7. The manganese solid phase was produced during the leaching. These results agreed with the theoretical review that the permanganate ion can be used as selective leaching to separate manganese from nickel and cobalt.

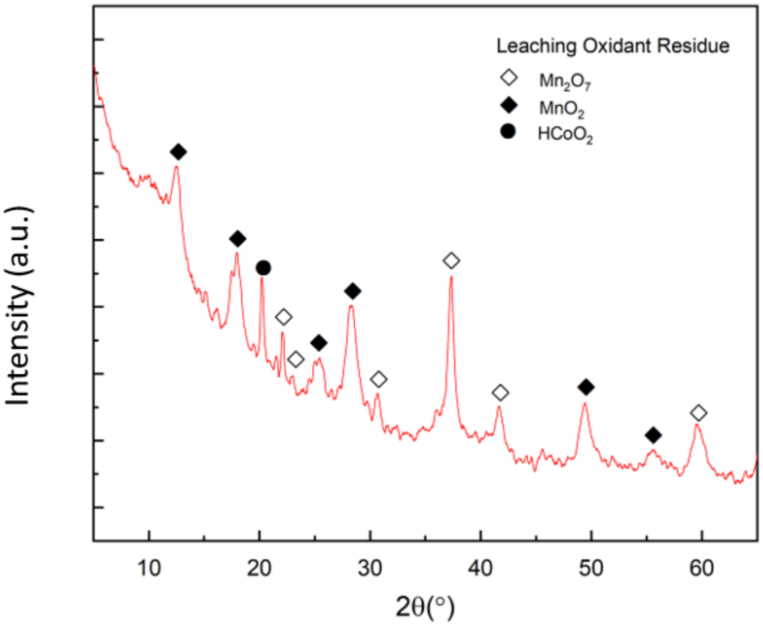

Figure 6. X-ray diffraction analysis of leached residue

Solvent Extraction (SX) of Metals Solution

The most optimum leaching conditions from the previous leaching were used for solvent extraction. The CYANEX-272 (bis (2,4, 4-trimethylpentyl) phosphinic acid) diluted in kerosine was mixed with the leach solution (PLS) at 50°C and 400 rpm. Then 28% NH_4OH was dropped wisely to raise the pH of the PLS acid to pH 6, where the extractant selectively separates Ni. The phase formations were observed between the organic phase that contains cobalt and manganese and the aqueous phase that contains nickel. The inductively coupled plasma (ICP) test was carried out to determine the extraction percentage of the organic phase. Table 2 presents the extraction percentage from SX through the calculation of the formula in equation (1) and equation (2); in this table, the E for Ni, Co, and Mn is 9.04%, 99.30% and 99.64%, respectively. The maximum selective extraction is achieved at pH 6 for separating the Ni and Co-Mn.

Table 2. Extraction and stripping percentage

Metals Ion	Ni	Co	Mn
Solvent Extraction 1			
Distribution	0.63	276.72	140.90
% SX	9.04%	99.30%	99.64%
Stripping			
% Stripping	1,22%	92,41%	90,35%
Solvent Extraction 2			
% SX	9,12%	46,62%	45,63%

The X-ray diffraction of the leached residue result (Figure 6) shows that all the nickel hydroxide in the MHP materials has already been converted into nickel sulfate. The solvent extraction process that is presented in Table 2 could remove 99.30 % of Co and 99.64 % of Mn from the aqueous phase, but this extraction process also took 9% of nickel into the organic phase. Therefore, the concentration of nickel components in the aqueous phase was 372.5267 g/L (91%). Because the concentration of nickel component at this aqueous phase achieved 372.5267 g/L, and the water solubility of nickel sulfate is about 293 g/L at 20ºC, the nickel sulfate crystals could be nucleated spontaneously. The nickel sulphate crystal was separated from the solution and dried, while the solution was cooled down using refrigeration at low temperatures for future crystallization processes. The composition of nickel sulphate, as measured by X-ray fluorescence (XRF), is detailed in Table 3.

Table 3. X-ray fluorescence (XRF) analysis of crystal precursor

Samples	Ni (%)	Co (%)	Mn (%)	Ag (%)	Cu (%)	Zn (%)	Fe (%)	Pt (%)
$NiSO_4$ crystal	99.46	0.116	-	0.031	0.035	0.062	-	0.156

Figure 7 illustrates the XRD patterns of nickel crystal. It was detected as $NiSO_4.7H_2O$ (01-088-0279), α-$NiSO_4.6H_2O$, β-$NiSO_4.6H_2O$ (01-081-1048), and (Ni (H_2O)6) SO_3 (01-074-1859). The formation of $NiSO_4.7H_2O$, α-$NiSO_4.6H_2O$, and β-$NiSO_4.6H_2O$ depends on the concentration of nickel sulfate and the temperature of nucleation processes. Nucleation process at a temperature below 31.5–32°C, the $NiSO_4$. $7H_2O$ (morenosite) is the most stable phase in the acid solution, and above this temperature, $NiSO_4$ $6H_2O$ will form in this solution [24], [25].

Jenssen et al. [24] also reported that the X-ray diffraction characteristic peaks of $NiSO_4.7H_2O$ at 16.7° and 21.1°; α-$NiSO_4.6H_2O$ at 19.4° and 20.9°; β-$NiSO_4.6H_2O$ at 20.4° and 22.3°. The X-ray diffraction result in Figure 7 also shows the formation of $NiSO_4.7H_2O$ and $NiSO_4.6H_2O$ at spontaneous nucleation of the aqueous phase (residue of first solvent extraction) with nickel concentration ± 372.5267 g/L, and this nucleation prose was done at room temperature.

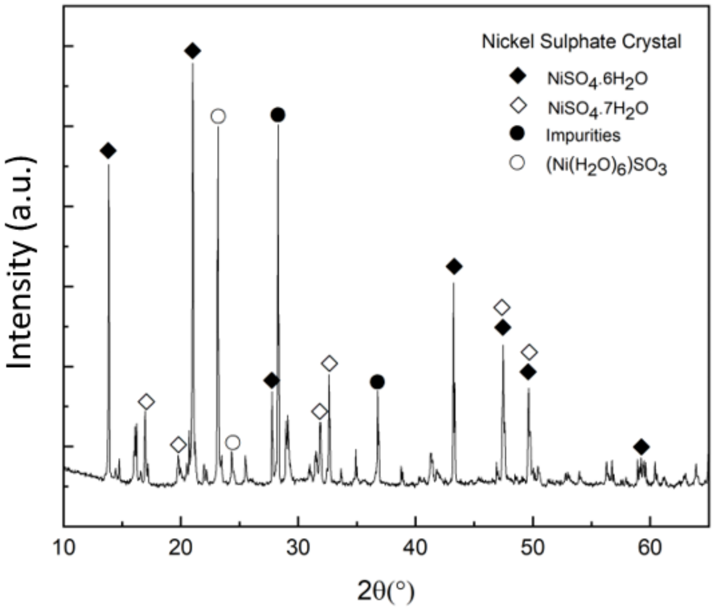

Figure 7. X-ray diffraction analysis of nickel crystal

In the stripping process, the organic phase of the SX containing Co and Mn will be mixed with 1 M sulfuric acid to release Co and Mn in the aqueous phase. The low acquisition of Co and Mn in the solution is due to the influenced concentration of sulfuric acid and the O/A ratio that could be more optimal [26]. The results of stripping percentages are shown in Table 2.

The cobalt was separated by mixing the aqueous phase from stripping 1 with D2EHPA (Di-(2-ethylhexyl) phosphoric acid) extractant at pH 3. After completion, the phase transformation will be observed, the organic phase at the top part containing Mn and the aqueous phase containing Co at the bottom level; the entire phase is carried out by ICP test. Different from nickel, the precipitation of Co needs the cooling process to accelerate the crystallization process; the the cobalt sulphate ($CoSO_4$) crystal precipitate is then dried.

Table 2 shows that the percentage of solvent extraction 2 for Co is only 45.63%, indicating that Co is low enough to be extracted into the organic phase. At the same time, Mn has a low percentage of 46.62 % in the organic phase. These results indicate that the extractant concentration is still not good enough to bind Mn ions; when compared to some literature that has Mn levels in aqueous solutions similar to this study, the concentration of D2EHPA is very low because, in other studies, a concentration of 0.2 M was used [27].

CONCLUSION

The selective leaching of mixed hydroxide precipitate (MHP) with oxidant agent potassium permanganate was successfully investigated. Sulfuric acid was used as initial leaching conditions, and observed that higher acid concentrations increased the leaching efficiency of nickel, cobalt, and manganese. The optimum leaching conditions of MHP were found at 1.5 M sulfuric acid solutions for 1 hour in the solid-to-liquid ratio of 1/10, with leaching efficiency for nickel, cobalt, and manganese are 72.38%, 50.08%, and 39.58%, respectively.

It was observed that the presence of the X-ray diffraction (XRD) analysis of leached residue also confirms that the manganese is incompletely dissolved into metal sulphate and proposes that the permanganate ion can be used as selective leaching to separate manganese from nickel and cobalt. It was determined that 1.75 M sulfuric acid, 7.5 g/L potassium permanganate, and 60°C temperature were the most optimum leaching conditions.

CYANEX-272 was used to extract 99.30 % and 99.64% of the cobalt and manganese into the organic phase at pH 6. Supersaturated nickel in the aqueous phase made the nickel sulfate crystals nucleate spontaneously at room temperature with a high purity of nickel content (99.46 %).

Developing an efficient and cost-effective process for recovering nickel, cobalt, and manganese requires extensive optimization of parameters such as pH, temperature, reagent concentrations, and residence time. Finding the optimal conditions for precipitation, leaching, and separation steps can be time-consuming and resource-intensive.

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