Study of the Effect of Time Variations on the Leaching Process of Ferronickel Products from Mini Blast Furnace to Yield Elements of Fe, Ni, and Co for NiSO₄.₆H₂O Synthesis

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Abstract. Indonesia, as one of the biggest contributor to air pollution, has a solution to replace petroleum-powered motorized vehicles with battery-powered electric vehicles (EVs). Indonesia has nickel reserves that can be used as one of the cathodes of a Li-ion battery called NMC. In this research, the author uses ferronickel ore from the smelting process of laterite nickel ore. Then ferronickel is leached to produce nickel sulfate. This research was conducted to determine the effect of variations in leaching time (2 hours, 4 hours, 6 hours, 8 hours, 10 hours) on the extraction rate, extraction percentage, chemical composition, yield, and the resulting compounds. The results of this study were that the highest content of nickel extract was produced in a 10-hour variation of 11530 mg/L with an extraction percentage of 94.16%, the most iron in a 10-hour variation of 4128 mg/L with 100% extraction percentage, and the most cobalt in a 2-hour variation as much as 47.3 mg/L with 100% extraction percentage. The highest nickel yield was produced at a 10-hour concentration variation of 87.37%. The compounds produced from the crystallization products were NiSO₄.₆H₂O, Na₂SO₄, CoSO₄, and FeSO₄.

1. Introduction
In recent decades, the number of electric vehicles (EVs) has continued to increase. It is estimated that worldwide, there will be more than 125 million EVs to be launched by 2030. The main component of this state-of-the-art vehicle is the lithium-ion (Li-ion) battery which provides the required energy storage. It is predicted that an increase in EVs due to the desire to reduce the negative consequences associated with the use of combustion engine-powered vehicles, and in particular to be donated to reduce carbon emissions, has led to a significant revival in the field of electric transport [1].

As a result of the increased demand for EV, it will cause an increase in demand for LiB and, thus, the raw materials needed for its production will also increase [2]. Li-ion batteries are divided into six based on their cathode constituents, including Lithium Cobalt Oxide (LCO), Lithium Manganese Oxide (LMO), Lithium Iron Phosphate (LFP), Lithium Nickel Cobalt Aluminum Oxide (NCA), Lithium Nickel Manganese Cobalt Oxide (NMC), and Lithium Titanate (LTO) [3]. Of these types of batteries, the NMC battery is a battery with the most superior properties for EV use, because it has a high energy density of up to 0.6 kWh/kg and has a moderate life cycle (2000-3000 times) [4]. One of the main components of NMC battery is nickel, thus nickel has an important role in infrastructure and
technology development, and nickel is used in a wide variety of products and has a wide range of applications, this is due to the special characterization of nickel itself [5].

In mines, nickel can be obtained from laterite and sulfide sources. About 70% of the world’s nickel ore is laterite, while 30% of it is sulfide ore [6,7]. Nickel Laterite ore has about 70% of nickel reserves. However, only 40% are processed further. It is because not easy to process laterite nickel ore when compared to process nickel sulfide ore [8,9]. Indonesia has a high potential for laterite nickel ore because Indonesia has a tropical climate. Indonesia is ranked third in the world for countries that have the most nickel ore reserves behind New Caledonia and the Philippines. There are around 1.58 million tons of laterite nickel ore spread over several large islands in Indonesia, including Papua, Sulawesi, and Maluku. These data show that Indonesia has great potential to become one of the countries producing nickel-based products in the global market. Laterite nickel has two types of ore. The two types of ore are saprolite and limonite. Limonite laterite nickel ore usually occurs at the top of nickel mines. This limonite has the dominant mineral in the form of goethite. Limonite ore contains low SiO2, which is less than 20%. In addition, limonite has a nickel content of 0.5 to 0.7% and a Fe content of 40 to 60%. On the contrary, saprolite is in a lower layer than limonite. This saprolite layer has a higher Ni content than limonite. In addition, saprolite has lower iron content and higher magnesium content than limonite coating [7]. In terms of processing, nickel laterite ore can be processed either through pyrometallurgy or hydrometallurgy routes. Limonite nickel ore, in general, can be processed via the hydro-route [10]. One technology that can process limonite laterite nickel ore is High-Pressure Acid Leaching or HPAL.

The most important process in hydrometallurgy is the leaching process. The leaching process determines the rate and efficiency of the change of metal into the solution, it affects most of the economic parameters of the whole process. The efficiency of this leaching process is determined by the thermodynamic aspect and identifying which agents interact together. There are several factors that influence the leaching process, such as the temperature during the roasting process, the concentration of reactants, sample particle size, pH, and the length of the leaching process [11]. In this study, the leaching process uses time variations because the time is required to release several base metals from the matte is very important. After all, metal ions need time to compound and also release bonds with each other per period. Therefore, it is important to study the effect of leaching time on the release of nickel and copper from matte [12].

2. Experimental Method

2.1 Materials Preparation
The ferronickel preparation stages in this study used laterite nickel ore from Southeast Sulawesi. Then the laterite nickel ore goes through a smelting process in MBF and becomes a ferronickel product. The ferronickel product through the grinding and sizing process to obtain a similar size, namely 50 mesh. Refining the particle size of the ore to be dissolved will accelerate the extraction process. This is because the area of contact between the metal particles and the leachate will be greater so the transfer of material and the smallest distance for the metal to diffuse from the solid core to the solution will be more effective [13]. Then the ferronickel powder is tested on EDX and AAS to determine the chemical composition contained in ferronickel. After that weigh the ferronickel mass as much as 5 samples with 2 grams each.

2.2 Preparation of H2SO4 Solution
To make a 98% H2SO4 solution, distilled water is needed for the dilution process. The 98% H2SO4 dilution carried out until the volume reaches 120 mL, using the following equation (1):

\[ M_1 \times V_1 = M_2 \times V_2 \] (1)

In Where \( M_1 \) is the molarity of 98% H2SO4 which is 18.4 M. \( V_1 \) is the volume of H2SO4 98% needed. \( M_2 \) is the concentration of H2SO4 that we want, which is 2M. \( V_2 \) is the volume of the solution used, which is 120 mL. So that from equation (1), the required volume of H2SO4 is 13.04 mL.
2.3 Leaching Process
Analyzed the amount of nickel-metal using the AAS test. In this leaching process, the independent variable that is varied is the leaching time. Meanwhile, the fixed variables that would keep constant were the temperature of 90°C, the volume of 150 ml, the stirring speed of 200 rpm, and the concentration of sulfuric acid 2M. The pH of the filtrate solution was controlled using a pH meter during the leaching process. The pH of the leaching solution was -0.6 with a potential of -0.209 V, this shows that the reaction under the nickel E-pH diagram where indicates the stability area of nickel compounds which is related to the pH value and potential [14].

In this leaching process, 120 mL of H₂SO₄ is added to 30mL of 30% H₂O₂ solution. The function of the H₂O₂ solution is to catalyze the reaction because the H₂O₂ solution is a strong oxidizing solution. After the leaching process is complete according to the predetermined time, the leaching solution is filtered to be separated from the impurities and then a sample of the filtrate is taken for AAS testing of each independent variable, after which the filtrate goes through the neutralization process.

2.4 Neutralization Process
The neutralization process of this study used 2.5M NaOH. Neutralization is carried out until the pH of the solution reached pH 6. pH meter is needed during the neutralization process so that the pH can be controlled as desired. The pH precipitation up to pH 6 is to form impurities, Fe(OH)₂, and Co(OH)₂ uniformly. The reference for this neutralization process uses the precipitated hydroxide precipitation graph based on the pH obtained from the relationship between pH at the precipitation of the hydroxide and the concentration of metal ions [15]. This neutralization process is carried out by titration so that the deposition process is homogeneous [4]. After that, the filtrate is filtered to be separated from the precipitate that is formed from the neutralization process.

2.5 Crystallization Process
This crystallization process aims to obtain nickel sulfate crystals from a solution of nickel sulfate that has gone through a neutralization process. Nickel sulfate filtrate solution was crystallized by heating in an oven for 2 hours at a constant temperature of 200°C. The end product of this crystallization is the crystalline solid NiSO₄.6H₂O.

2.6 Product Analysis
Atomic Absorption Spectroscopy (AAS), Energy-Dispersive X-ray Spectroscopy (EDX) testing, and X-ray Diffractometer (XRD) testing are used to determine the dissolved metal content, the percentage of extraction, the compounds formed, the elemental composition, and the yield of the product. The AAS test is used to determine the content of nickel, iron, and cobalt dissolved in the solution and to calculate the extraction percentage from the leaching process. The AAS test results from the ferronickel sample and the leaching process of each variable can be obtained by using equation (2) below [9]:

\[
\% \text{ Leaching} = \frac{\text{Metal content in solution} \times \text{Volume of leachate}}{\text{Metal content in FeNi sample} \times \text{FeNi sample weight}} \times 100
\] (2)

EDX testing aims to determine the elemental composition and yield formed from the product. The EDX test results of ferronickel samples and crystallization products from each variable can be obtained by using equation (3) below [6]:

\[
\% \text{ yield} = \frac{\text{Metals content in product} \times \text{Product Mass}}{\text{Metals content in sample} \times \text{sample weight}}
\] (3)

And XRD testing aims to determine the compounds contained in ferronickel samples and NiSO₄.6H₂O crystals. XRD testing was carried out at the 2θ position from 10° to 90° and using a CuKα wavelength of 1.54056 Å.
3. Results and Discussion

3.1 Ferronickel Sample Characterization

The ferronickel powder which has been in powder form is tested for EDX and AAS, EDX testing to determine the initial content of the element before going through the leaching process. The results of the EDX test in the form of the % chemical composition of the ferronickel sample, it was found that the ferronickel sample contained 16.47% Ni, 3.25% Co, 75.98% Fe, 0.95% S, 0.93% Na, and 2.43% O. Based on the amount of nickel content, this ferronickel is included to the low-grade ferronickel category [16]. The composition can be seen in Table 1.

| Element | Ni   | Co  | Fe    | S   | Na  | O   |
|---------|------|-----|-------|-----|-----|-----|
| % Wt    | 16.47| 3.25| 75.98 | 0.95| 0.93| 2.43|

AAS test results from ferronickel samples to determine the percentage of dissolved metal in the leaching process. Obtained from the AAS test results, this ferronickel sample contains 32.88% Ni, 0.35% Co, and 68.26% Fe content. The composition can be seen in Table 2.

| Metal Content | Ni   | Co  | Fe  |
|---------------|------|-----|-----|
| %             | 32.88| 0.35| 68.26|

3.2 Effect of Time Variation on Dissolved Metal Content and Extraction Percentage

The AAS test was carried out to determine the levels of Fe, Ni, and Co contained in the solution. This test tests the solution of time variation leaching results, namely the leaching time of 2 hours, 4 hours, 6 hours, 8 hours, and 10 hours. As well as ferronickel samples to obtain the extraction percentage of Fe, Co, and Ni. The results of the AAS test for NiSO₄ filtrate can be seen in Table 3.

| Leaching Time (hour) | Fe (mg/L) | Ni (mg/L) | mg/L) |
|----------------------|-----------|-----------|-------|
| 2                    | 8970      | 3871      | 47.3  |
| 4                    | 9900      | 3836      | 34.9  |
| 6                    | 7960      | 3589      | 32.75 |
| 8                    | 8960      | 3457      | 37.8  |
| 10                   | 11530     | 4128      | 41.4  |

A trend can be made from the extracted content of Ni, Fe, and Co in the leaching filtrate with time variations in Figure 1.
The AAS test aims to determine the levels of Fe, Ni, and Co contained in the solution. This test tests the solution of time variation leaching results, namely the leaching time of 2 hours, 4 hours, 6 hours, 8 hours, and 10 hours. As well as ferronickel samples to obtain the extraction percentage of Fe, Co, and Ni. The results of the AAS test for NiSO$_4$ filtrate can be seen in Table 3 [17].

\[
\begin{align*}
    NiO + H_2SO_4 & \rightarrow NiSO_4 + H_2O & \Delta G^\circ &= -98,283 \text{ kJ/mol} \\
    FeO + H_2SO_4 & \rightarrow FeSO_4 + H_2O & \Delta G^\circ &= -124,292 \text{ kJ/mol}
\end{align*}
\]

So that the sulfuric acid anion SO$_{4}^{2-}$ binds to the metal ion to form NiSO$_4$, FeSO$_4$. The highest extract of Fe and Ni was found in a variety of 10 hours. The highest extract of Ni can be found in the longest time because, in the process of forming compounds, metal ions need time to compound and also mutually bond with each other per period [18]. Nevertheless, the highest extraction Co content was found at a reaction time of 2 hours. It happens because with increasing time, the solvent can dissolve all the precious metals in the ferronickel. Because the contents of ferronickel are not only contained nickel, iron, and cobalt metals but also many other impurity minerals. So, the ability of the solvent that should dissolve cobalt is reduced due to it being dissolved in impurities [19]. This also explains the phenomena of decreasing levels of iron that occur at 6 hours, nickel extract content at 4, 6, 8 hours, and cobalt extraction content at 4 and 6 hours.

From the extracted content above, it can be calculated the extraction percentage. The extraction percentage shows the amount percentage that was successfully extracted from the leached ferronickel [20]. The results are shown in Table 4.

**Table 4. Percentage of time variation leaching extraction.**

| Leaching Time (hour) | Fe (%) | Ni (%) | Co (%) |
|----------------------|--------|--------|--------|
| 2                    | 98.55  | 88.29  | 100    |
| 4                    | 100    | 87.5   | 74.78  |
| 6                    | 87.45  | 81.86  | 70.17  |
| 8                    | 98.44  | 78.85  | 81     |
| 10                   | 100    | 94.16  | 88.71  |
The highest percentage of Fe and Ni occurred at 10 hours reaction time of 94.16% Ni and 100% Fe, for the highest percentage of Co was obtained at 2 hours of leaching time of 100%. These results, a graph of the extraction percentage trend can be made (Figure 2).

Figure 2. Extraction Percentage Graph

3.3 Effect of Time Variation on Chemical Composition

To find out the chemical composition that contained in the nickel sulfate crystals, the EDX test was carried out. The results of EDX testing on NiSO₄.6H₂O products with variations in leaching time can be seen in Table 5.

Table 5. The chemical composition of the product at various leaching times

| Element | 2 hour | 4 hour | 6 hour | 8 hour | 10 hour |
|---------|--------|--------|--------|--------|---------|
| Na (wt%)| 32.63  | 30.71  | 26.01  | 17.03  | 23.30   |
| S (wt%) | 34.28  | 18.08  | 17.90  | 8.79   | 16.28   |
| Fe (wt%)| 6.33   | 24.67  | 7.74   | 41.15  | 28.15   |
| Co (wt%)| 0.51   | 2.49   | 1.92   | 3.24   | 3.08    |
| Ni (wt%)| 12.01  | 13.44  | 17.81  | 18.55  | 19.19   |

From the table above, the chemical composition of the product can be obtained as a trend graph of the content Ni, Fe, and Co in NiSO₄.6H₂O crystals, the trend can be seen in Figure 3.

Figure 3. Graph of Ni, Fe, and Co content
The nickel content in the picture above tends to rise until the highest nickel content is found in the longest leaching variation, which is 10 hours at 19.19%. It is because the longer the extraction time, the longer the contact time between the solvent and nickel as a solid so that more solute is contained in the solvent [21]. However, a too long time will also result in saturated dissolved nickel, so it does not experience a significant increase, such as the variation in the leaching time of 8 hours and 10 hours, the trend is not significant. While the elements of Fe and Co, in the time variation from 2 hours to 10 hours is fluctuated, it is because of the effect of the neutralization process.

3.4 The Effect of Time Variation on Yield
From the data in Table 1 and Table 5, we can calculate the yield of Fe, Ni, and Co in the NiSO\textsubscript{4}.6H\textsubscript{2}O product. The results are in Table 6.

Table 6. The chemical composition of the product at various leaching times

| element | 2 hour | 4 hour | 6 hour | 8 hour | 10 hour |
|---------|--------|--------|--------|--------|---------|
| Fe (wt%) | 7.33   | 29.54  | 7.95   | 41.43  | 27.76   |
| Co (wt%) | 13.72  | 69.63  | 46.08  | 76.34  | 71.08   |
| Ni (wt%) | 64.17  | 74.24  | 84.35  | 86.15  | 87.37   |

A yield trend of Ni, Fe, and Co can be made on NiSO\textsubscript{4}.6H\textsubscript{2}O products, which can be seen in Figure 4. The highest yield of nickel was 87.37% at 10 hours, the highest yield of iron was 41.43% at 8 hours and the highest yield of cobalt was 76.34% at 8 hours.

![Figure 4. Graph of Ni, Fe, and Co Yields](image)

3.5 The Effect of Time Variations on the Compounds Formed
To find out the compounds contained in the nickel sulfate crystals, an XRD test was carried out. The results of the analysis can be seen in Figure 5.
Figure 5. XRD test results for NiSO$_4$.6H$_2$O crystals

The XRD test results for NiSO$_4$.6H$_2$O crystals contained Na$_2$SO$_4$, NiSO$_4$.6H$_2$O, CoSO$_4$, and FeSO$_4$ compounds at all time variables. NiSO$_4$.6H$_2$O is the main compound of crystal products, this compound can be found in each of the time variables. This shows that NiO at each time variable has reacted with H$_2$SO$_4$ to form NiSO$_4$ in solution form. The solution is then subjected to a neutralization and crystallization process to form a NiSO$_4$.6H$_2$O compound.

The presence of CoSO$_4$ and FeSO$_4$ compounds indicates that the Co and Fe samples dissolve in the leaching process by producing equations 6 and 7, as follows:

$$FeO + H_2SO_4 \rightarrow FeSO_4 + H_2O$$

$$\Delta G^\circ = -124,292 \text{ kJ/mol}$$

$$CoO + H_2SO_4 \rightarrow CoSO_4 + H_2O$$

$$\Delta G^\circ = -115,444 \text{ kJ/mol}$$

The reaction shows FeO and CoO react with H$_2$SO$_4$. The neutralization process in equations 8 and 9 shows the reaction between CoSO$_4$, FeSO$_4$ with NaOH which expect to produce deposits of Co(OH)$_2$ and Fe(OH)$_2$ completely. However, the CoSO$_4$ and FeSO$_4$ compounds have not precipitated completely.

$$FeSO_4 + NaOH \rightarrow Fe(OH)_2 + Na_2SO_4$$

$$\Delta G^\circ = -130,057 \text{ kJ/mol}$$

$$CoSO_4 + NaOH \rightarrow Co(OH)_2 + Na_2SO_4$$

$$\Delta G^\circ = -142,701 \text{ kJ/mol}$$

The existence of the Na$_2$SO$_4$ compound is because in every neutralization process there will produce a secondary product called Na$_2$SO$_4$. Such as the result of FeSO$_4$, CoSO$_4$, and NiSO$_4$ react with NaOH is sediment and by-products in the form of Na$_2$SO$_4$ [22]. Based on Figure 5, the dominant compounds are Na$_2$SO$_4$, CoSO$_4$, and FeSO$_4$, this shows that the precipitation is not maximally. Regarding this matter, it needs to be considered again in the neutralization process to produce optimal nickel.
4. Conclusions

Based on the results of testing and data analysis, it can be concluded that the best extract content and percentage extraction of Ni and Fe at the leaching time of 10 hours and the best extract content and percentage extraction of Co at the leaching time of 2 hours. NiSO$_4$.6H$_2$O compounds can be found at all variations of time. However, the resulting compounds in the NiSO$_4$.6H$_2$O crystals were dominated by Na$_2$SO$_4$, CoSO$_4$, and FeSO$_4$. The highest nickel chemical composition is at a time variation of 10 hours by 19.19%. While wt% in Fe and Co elements fluctuate. It is because the neutralization process is not optimal. The highest yield for Ni from NiSO$_4$.6H$_2$O crystals is 87.37% at a variation of the 10-hour leaching time. The highest yield of Fe was 41.43% at the variation of the leaching time of 8 hours. The highest yield of Co was 76.36% at the variation of the leaching time 8 hours.

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