Investigating the Extraction Process of Nickel and Cobalt from Low-grade Nickel Laterite Ore Using Sulfuric Acid Leaching Method

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**Abstract.** The extraction of nickel and cobalt from laterite ores is predominantly performed using hydrometallurgical methods due to their efficiency in processing low-grade ores. In this study investigates the leaching behavior of low-grade laterite ore from Konawe, Southeast Sulawesi, was investigated using sulfuric acid (H2SO4). The experimental procedure consisted of three main stages sample preparation, leaching, and precipitation. Initially, ore samples with particle sizes smaller than 200 mesh were characterized by X-ray fluorescence (XRF) and X-ray diffraction (XRD) to determine their mineralogical composition, particularly the nickel and cobalt contents. Subsequently, leaching experiments were conducted by varying the concentration of sulfuric acid and leaching duration. The optimum leaching conditions were achieved at 7 M H2SO4 and 6 hours, resulting in extraction efficiencies of 97.73% for nickel and 73.32% for cobalt. In the precipitation stage, different reagents were evaluated, including ammonium oxalate ((NH4)2C2O4), ammonium hydroxide (NH4OH), and sodium hydroxide (NaOH). Ammonium oxalate facilitated the selective precipitation of cobalt and manganese, while nickel remained largely in solution. In contrast, ammonium hydroxide promoted the partial precipitation of nickel and manganese as oxalates. Meanwhile, sodium hydroxide successfully precipitated both metals directly as nickel hydroxide and cobalt hydroxide. These results confirm the critical role of pH adjustment in enhancing the efficiency of nickel and cobalt recovery from laterite ores using sulfuric acid leaching.

# INTRODUCTION

Laterite ore serves as the primary source of nickel, commonly occurring in oxide form due to extended weathering and deposition processes [1]. Many of these ores are found in tropical regions, including Indonesia, the Philippines, and Australia [2]. The Geological Agency of the Ministry of Energy and Mineral Resources reports that Indonesia possesses approximately 72 million tons of nickel reserves, representing nearly 52% of global reserves. The most significant laterite nickel deposits in Indonesia are found in Sulawesi, Halmahera, Papua, and Kalimantan . Indonesian laterite ores are primarily composed of goethite ((Fe, Ni, Al)O(OH)) at approximately 80%, with silicate minerals such as lizardite ((Mg, Fe, Ni)₃Si₂O₅(OH)) and olivine ((Mg, Fe, Ni)₂SiO₄)) constituting around 15%. Minor phases include hematite (Fe₂O₃), chromite ((Fe, Mg)O(Cr, Fe, Al)₂O₃), maghemite, and quartz [3]. The ores are categorized into several zones, the ferricrete (top soil) zone, the limonite zone, the saprolite zone, and the bedrock zone, each characterized by unique mineralogy and varying thickness based on the depositional environment [4]. The top soil/ferricrete zone represents the uppermost layer in mining regions, characterized by a reddish-brown to dark clay-like texture and a thickness ranging from 1 to 4 meters. The minerals present in the topsoil zone include hematite, geothite, and manganese. The second zone is the limonite zone, characterized by a thickness of 2-3 meters and a reddish-brown color attributed to hematite oxide presence. Additionally, the limonite zone includes the mineral goethite, resulting in a significant magnetic intensity. The saprolite zone typically exhibits a greenish-yellow hue, possesses a low magnetic intensity, and has a thickness ranging from 3 to 4 meters. The final zone is the bedrock zone, the lowest layer, characterized by rocks containing laterite nickel deposits with a thickness of 2-3 meters. The thickness of each zone varies based on the specific area of laterite ore deposition.

The nickel in the ore is unable to be processed directly due to the presence of various other minerals in the natural ore [5]. Consequently, extracting and purifying nickel ore is essential to achieve a higher concentration of nickel. The purification of nickel from ore is carried out through a hydrometallurgical process that employs chemical solutions, including sulfuric acid, to extract nickel metal from laterite ore [6]. This method is appropriate for laterite ore due to its low nickel content, which exists as oxides that can be solubilized in acidic solutions [7]. The hydrometallurgical process comprises three stages: leaching, extraction, and purification through precipitation, aimed at achieving purer nickel [8]. The leaching process generally employs a reagent, typically a strong acid like sulfuric acid (H2SO4), nitric acid (HNO3), or hydrochloric acid (HCl), along with a reducing agent such as hydrogen peroxide (H2O2) (Larouche et al., 2020). All types of acids can be used as leaching reagents. It is important to recognize the existence of leaching waste, which has the potential to contaminate the environment. Hydrochloric acid exhibits corrosive properties and high volatility, contributing to air pollution, particularly in industrial applications [9]. Sulfuric acid demonstrates effective leaching capabilities due to its high reactivity, enabling the dissolution of metals into their ionic forms. The separation and purification of nickel and cobalt from the leachate is a critical step following leaching. The use of sodium hydroxide for precipitation is a widely utilized method for the selective recovery of nickel and cobalt in the form of hydroxide precipitates. This method enables the effective separation of valuable metals from impurities, thereby facilitating their refining and utilization.

Previous studies have shown that utilizing solid waste as a source of metals through chemical treatment is a strategic approach in industrial waste management. Metal precipitation techniques using acid treatment enable the release of metal ions from solid matrices and selective precipitation based on pH conditions and reagent types. Hartanto et al. (2023) examined the phase transformation of rice husk ash as a silica precursor under acidic conditions and found that changes in material structure were greatly influenced by acid concentration and reaction temperature, which affected the efficiency of metal element release [10]. Another study by Hartanto et al. (2025) showed that the synthesis of metal-based composites from waste precursors can be controlled by varying chemical conditions, including the type of acid and reaction time, which affect the morphology and purity of the precipitation results. These findings provide a strong scientific basis for the development of efficient and sustainable metal precipitation methods, and support the integration of chemical approaches in metal recovery from solid waste.

This study methodically investigates the extraction of nickel and cobalt from low-grade nickel laterite ore through sulfuric acid leaching, followed by precipitation using various reagents, including ammonium oxalate, ammonium hydroxide, and sodium hydroxide.

## MATERIALS

Materials used in this study are laterite ore from Konawe, Southeast Sulawesi, sulfuric acid (H2SO4, Smartlab Merck, 98%), sodium hydroxide (NaOH, Merck, ≥ 98 %), oxalic acid (H2C2O4, SAP, ≥ 98 %), ammonium oxalate ((NH4)2C2O4, Supelco Merck), ammonium solution (NH4OH, Supelco Merck, 25%), and sodium hydroxide (NaOH, Supelco Merck) and deionized water.

## EXPERIMENTAL

**Sample Preparation and Characterization**

The laterite ore needed crushing, grinding, and sieving to obtain a particle size of less than 200 mesh. The prepared samples were characterized to identify the mineral phases and determine the elemental and mineral composition using X-ray diffraction (XRD) and X-ray fluorescence (XRF) analyses.

**Leaching experiments**

The leaching experiments were conducted by mixing 5 g of the prepared laterite ore with 100 mL of sulfuric acid at varying concentrations (1-10 M). The mixtures were stirred continuously at 250 rpm and maintained at a temperature of 95 ℃ for 6 hours. After the leaching process, the solid residues were separated by filtration, and the concentrations of nickel and cobalt in the leachate were analyzed using atomic absorption spectroscopy (AAS) to calculate the metal extraction efficiencies.

**Nickel and Cobalt Precipitation**

Precipitation experiments were conducted on the leachate samples using three different reagents, ammonium oxalate (0.3 M), ammonium hydroxide (6%), and sodium hydroxide (2%). For each experiment, the leachate was placed in a Duran, and the reagent was added dropwise while continuously stirring at 250 rpm until the mixture reached pH 4. The temperature was maintained between 45 ℃ throughout the reaction, and the stirring continued for 2 hours. After the precipitation process, the solid residues were separated from the filtrate by vacuum filtration, washed with deionized water until neutral, and then dried at 60 ℃ for approximately 3 hours. The dried residues were subsequently characterized using X-ray fluorescence (XRF) to determine elemental composition and X-ray diffraction (XRD) to identify the mineral phases.

## Materials Characterization

The mineral content of the laterite ore was characterized using X-ray fluorescence (XRF). The XRF analysis was conducted using a PANalytical instrument at an accelerating voltage of 30 kV. This analysis offered critical insights into the elemental composition and content of the examined specimen[10,11]. The mineral phases were characterized using X-ray diffraction (XRD) with a PANalytical instrument under Cu Kα irradiation (λ = 1.5406 Å), applying an accelerating voltage of 40 kV and a current of 40 mA [12]. Additionally, the concentrations of nickel and cobalt in the leachates were determined using an Atomic Absorption Spectrophotometer (AAS; Thermo Scientific iCE 3000 series), which allowed precise quantification of the dissolved metals during the leaching experiments[13].

## RESULT AND DISCUSSION

**Identification of Laterite Ore**

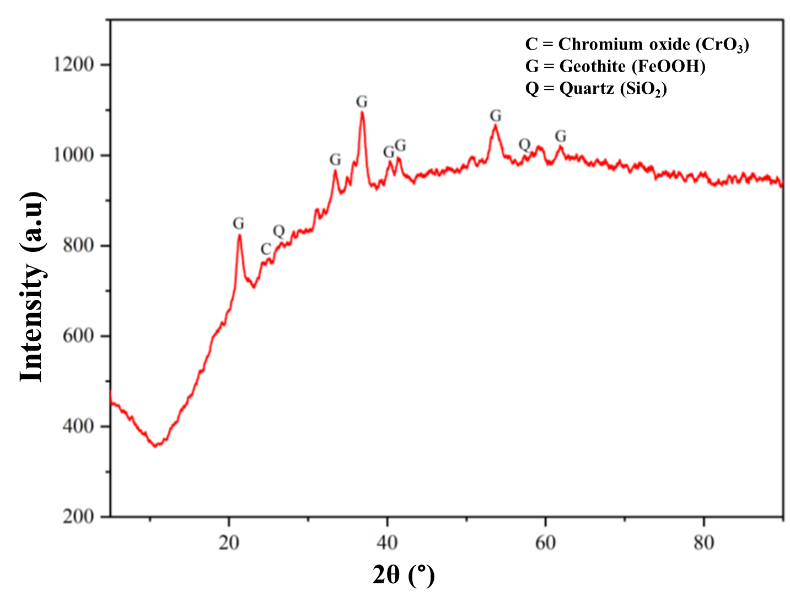
The laterite ore was characterized to determine its elemental composition and mineralogical phases using XRF and XRD analyses. As shown in Table 1, the XRF results revealed that the ore contained significant amounts of iron (Fe), nickel (Ni), cobalt (Co), aluminum (Al), and silicon (Si), with minor contents of magnesium (Mg), manganese (Mn), and other trace elements.

**TABLE 1**.  Elemental composition of ore

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Element | Ni | Co | Mg | Fe | Si | Mn | Cr | Al | P |
| Content  (wt. %) | 1.27 | 0.04 | 0.35 | 40.52 | 2.75 | 0.5 | 1.48 | 5.41 | 0.02 |

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Element | Cu | Ca | Ti | S | W | Zn | Pb | V |
| Content  (wt. %) | 0.02 | 0.05 | 0.08 | 0.33 | 0.01 | 0.03 | 0.01 | 0.03 |

The results presented in Table 1 show that the laterite ore samples primarily consist of iron, representing 40.52% of the total sample weight, whereas nickel contains only 1.27%. Based on the nickel content, ranging from 0.8 to 1.5% allows for classification of the ore as limonite-type laterite [14]. Limonite-type ores are typically enriched in iron oxides with moderate nickel content, rendering that suitable for hydrometallurgical extraction processes. The low nickel concentration indicates that effective leaching techniques are essential for optimizing nickel recovery from these samples, whereas the high iron content may affect the selection of reagents and processing conditions.



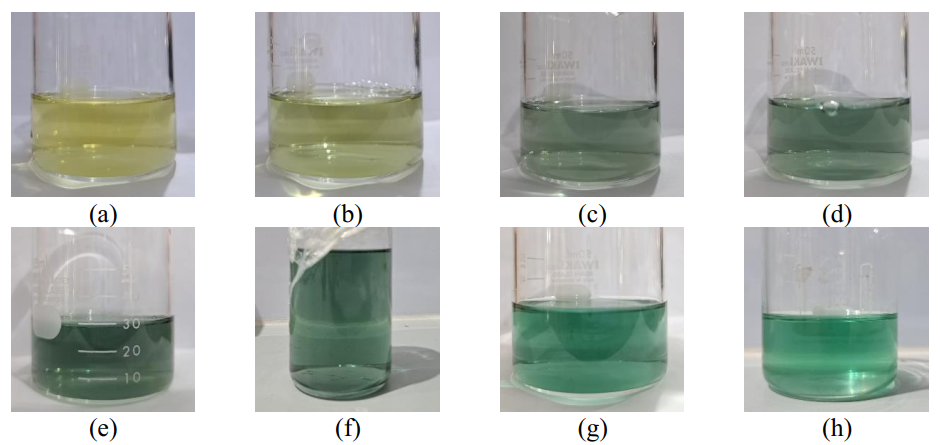
**FIGURE 1**. X-ray diffraction pattern of the ore sample

As shown in Figure 1, XRD analysis showed that the dominant mineral phases in the laterite ore were goethite (FeOOH), and silicate minerals such as quartz (SiO2). The presence of these iron oxide and silicate phases correlate with the XRF elemental composition and confirms that the ore exhibits a lateritic profile characterized by high Fe content and moderate Ni and Co concentrations. Minor reflections corresponding to gibbsite (Al(OH)3) and magnetite (Fe3O4) were also observed, potentially contributing to the overall metal content and affecting leaching behavior.

The integration of XRF and XRD data offers a thorough analysis of the laterite ore, emphasizing its chemical composition and crystalline structure. The characterization is essential for optimizing leaching conditions, as the ore's reactivity is significantly affected by the mineral phases present and the distribution of nickel and cobalt within the iron and silicate matrices. The results indicate that the ore from the studied site is appropriate for subsequent hydrometallurgical extraction of nickel and cobalt.

**Acid Leaching of Laterite Ore**

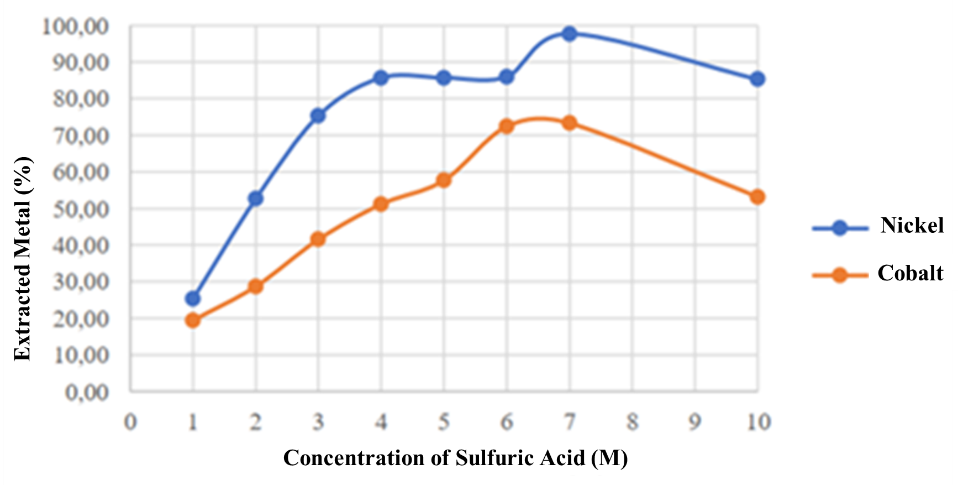
The acid leaching of laterite ore using sulfuric acid (1-10 M) produced leachates with noticeable differences in colour, which reflected the extent of metal dissolution shown in Figure 1. At low acid concentrations (1-3 M), the leachate was pale yellow to light brown, indicating limited extraction of nickel and cobalt from the ore matrix. As the acid concentration increased to 5-7 M, the leachate turned dark green, suggesting higher concentrations of dissolved nickel and cobalt ions. At concentrations above 7 M, the leachate colour became slightly darker but did not change significantly, implying that the extraction had approached its maximum efficiency. The colour variations are closely related to the mineralogical composition of the ore and the solubilization of iron, nickel, and cobalt. Limonite-type ores contain iron oxides such as goethite and hematite, which can impart brownish tones to the solution at low acid concentrations. The green coloration at higher acid concentrations is primarily due to dissolved nickel ions, while cobalt contributes slightly to bluish-green hues.



**FIGURE 2.** Filtrates produced under different sulfuric acid concentrations (a) 1M,

(b) 2M, (c) 3M, (d) 4M, (e) 5M, (f) 6M, (g) 7M, (h) 10M

The concentration of sulfuric acid influences the leaching process, as the H+ ions in sulfuric acid react with nickel oxide in the ore [15]. Higher sulfuric acid concentrations provide more H+ ions, which facilitates greater nickel dissolution and results in more efficient leaching. The effect is observable in the leachate colour, transforming from yellow at low acid concentrations to green at elevated concentrations. The AAS analysis of the leachate, shown in Figure 3, indicates that the observed colour variations correlate with variations in nickel concentration resulting from differing sulfuric acid concentrations.

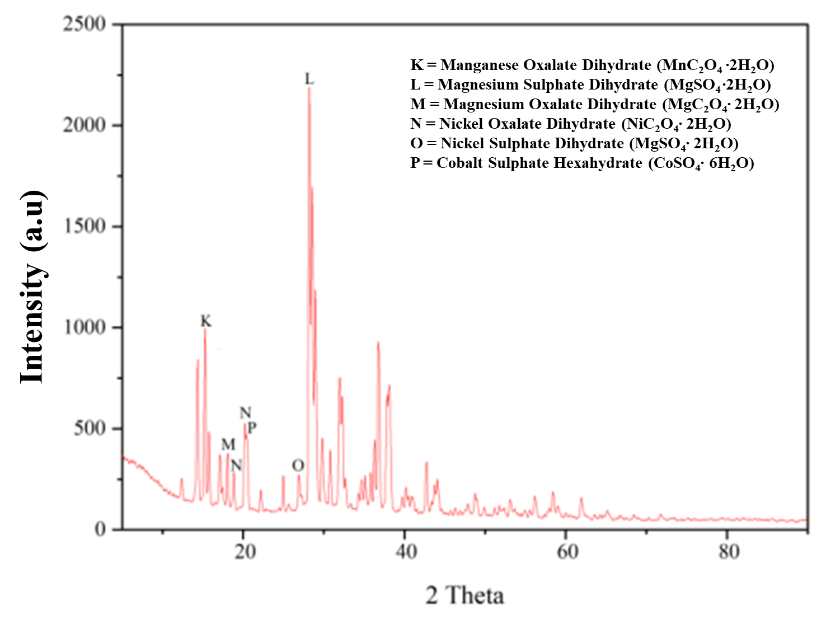


**FIGURE 3.** Effect of sulfuric acid concentration on the extraction efficiencies of nickel and cobalt

Figure 3 shows that the extraction efficiencies of nickel and cobalt enhance as the concentration of sulfuric acid increases. This study identified the optimal acid concentration as 7 M, resulting in nickel and cobalt recoveries of 97.73% and 73.32%, respectively. A decrease in metal recovery was detected at 10 M H₂SO₄, obviously due to the leaching solution achieved saturation.

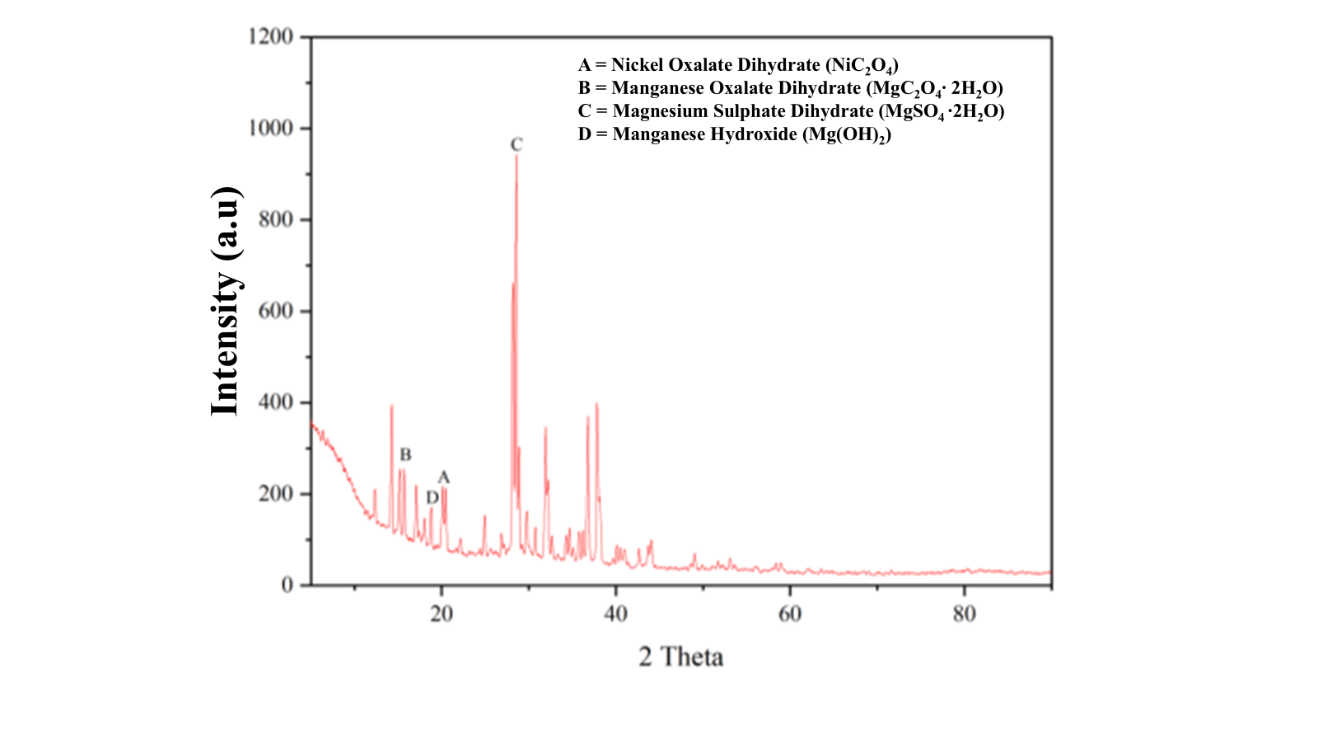
**Effect of Precipitation Reagents on Metal Recovery**

The precipitation of nickel and cobalt from laterite leachates was investigated using three reagents: ammonium oxalate, ammonium hydroxide, and sodium hydroxide. The aim was to identify an effective and environmentally friendly method for metal recovery. The results showed differences in performance among the reagent. Ammonium oxalate exhibited limited efficiency, likely due to incomplete complex formation and insufficient pH adjustment. Ammonium hydroxide improved metal precipitation by forming metal hydroxides, though some nickel and cobalt remained in solution. Sodium hydroxide proved the most effective, enabling nearly complete precipitation of both metals through the formation of nickel hydroxide and cobalt hydroxide. The substantial difference in Ksp values between nickel (1.2 × 10-3 ) and cobalt oxalates (2.7 × 10-9) is facilitates the selective separation and purification of these metals. Nickel, that has a higher solubility product constant (Ksp), is more resistant to precipitation compared to cobalt under analogous conditions. Ammonium oxalate effectively separates nickel and cobalt into distinct phases, with nickel remaining in the liquid phase and cobalt forming a solid precipitate [16]. XRD analysis (Figure 4) revealed peaks corresponding to manganese oxalate dihydrate (MnC2O4·2H2O) at 15.67°, magnesium oxalate dihydrate (MgC2O4·2H2O) at 18.12°, and nickel oxalate dihydrate (NiC2O4·2H2O) at 18.78° and 20.17°. Additional peaks were observed for magnesium sulfate dihydrate (MgSO4·2H2O) at 28.58°, nickel sulfate dihydrate (NiSO4·2H2O) at 26.83°, and cobalt sulfate hexahydrate (CoSO4·6H2O) at 20.29°. The results indicate that oxalate ions can precipitate metals as the associated oxalates; however, the amount of oxalate added was insufficient for the complete precipitation of all metals in the filtrate.



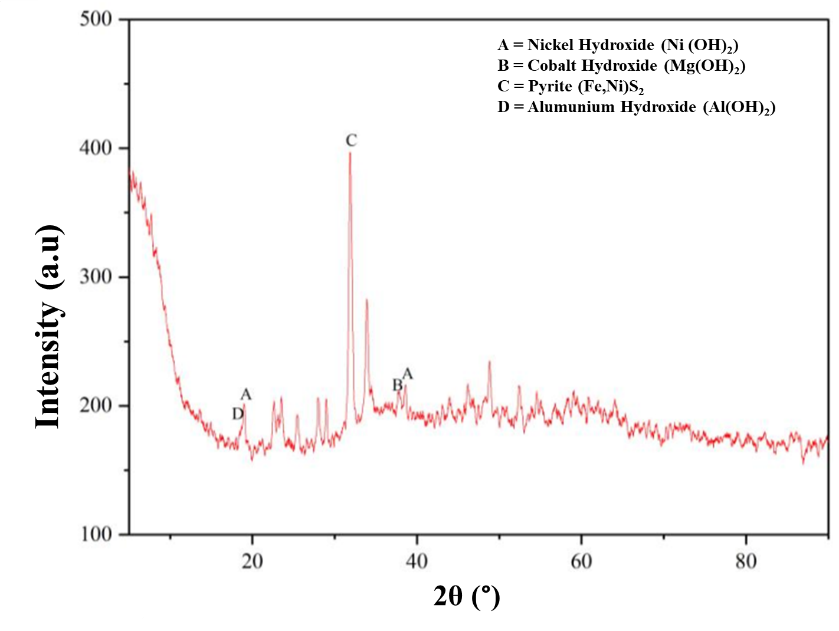
**FIGURE 4.** X-ray Diffraction Analysis of Ammonium Oxalate Precipitation Residue

The XRD diffractogram (Figure 5) of the precipitation using ammonium oxalate exhibited peaks corresponding to nickel oxalate (NiC₂O₄) at 20.16° and manganese oxalate dihydrate (MnC₂O₄·2H₂O) at 15.67°, confirming that oxalate ions effectively precipitated nickel and manganese. A peak corresponding to manganese hydroxide (Mn(OH)₂) at 18.2° indicated the presence of residual hydroxide ions still participating in the reaction despite the addition of oxalic acid. Furthermore, magnesium sulfate dihydrate (MgSO₄·2H₂O) was detected at 28.77°, suggesting that not all dissolved metals in the filtrate were successfully precipitated by the hydroxide and oxalate ions.



**FIGURE 5.** Diffractogram XRD of precipitation residue from ammonium hydroxide

The precipitation process using NaOH can be carried out in stages to separate nickel and cobalt from other metals such as iron and magnesium. The precipitation residue was characterized using XRD to determine the metal that was successfully precipitated by sodium hydroxide. Figure 6 shows that the XRD peaks corresponding to nickel hydroxide (Ni(OH)2) at 19.25° and 38.54°, cobalt hydroxide Co(OH)2 at 37.76°, and aluminum hydroxide (Al(OH)3) at 18.51°. A prominent peak was also observed, identified as pyrite (Fe,Ni)S2, indicating that not all metals present in the leachate were successfully precipitated by sodium hydroxide.



**FIGURE 6.** Diffractogram XRD of precipitation residue from sodium Hydroxide

The effectiveness of three precipitation reagents ammonium oxalate, ammonium hydroxide, and sodium hydroxide was evaluated for the recovery of nickel, cobalt, and other metals from laterite leachates. Ammonium oxalate selectively precipitated cobalt and manganese as oxalates, while a significant portion of nickel remained in solution due to its higher solubility, enabling potential metal separation. Ammonium hydroxide, followed by oxalic acid addition, facilitated the formation of nickel and manganese oxalates, although residual hydroxide ions indicated only partial metal precipitation. Sodium hydroxide efficiently precipitated nickel, cobalt, and aluminum as hydroxides, achieving higher overall metal recovery; however, the detection of a pyrite peak ((Fe,Ni)S₂) suggested that a fraction of metals remained unprecipitated. These results indicate that sodium hydroxide is most effective for comprehensive recovery, whereas ammonium oxalate and ammonium hydroxide can be applied for selective separation and purification of metals.

# CONCLUSION

This study demonstrated the effective extraction and recovery of nickel and cobalt from low-grade laterite ore using sulfuric acid leaching followed by chemical precipitation. Optimal leaching conditions were achieved at 7 M H**2**SO**4** and 95°C for 6 hours, resulting in maximum nickel and cobalt recoveries of 97.73% and 73.32%, respectively. Precipitation experiments using ammonium oxalate, ammonium hydroxide, and sodium hydroxide revealed distinct metal recovery behaviors. Ammonium oxalate enabled selective precipitation of cobalt and manganese, leaving nickel predominantly in solution, whereas ammonium hydroxide facilitated partial precipitation of nickel and manganese as oxalates. Sodium hydroxide was the most effective reagent for comprehensive recovery, precipitating nickel, cobalt, and aluminum as hydroxides, although traces of metals remained unprecipitated. Overall, the sequential use of optimized leaching and precipitation processes provides a promising, efficient, and environmentally compatible approach for nickel and cobalt recovery from laterite ores.

# Acknowledgments

# The authors greatly acknowledge Institut Teknologi Sepuluh Nopember (ITS) for funding this work under the Ministry of Education, Culture, Research, and Technology (Kementerian Pendidikan, Kebudayaan, Riset, dan Teknologi) of Indonesia with the scheme of the Doctoral thesis (Penelitian Disertasi Doktor) based on contract number 1189/PKS/ITS/2025.

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