**Study on Electrowinning Parameters for Nickel Extraction from Sulfate Solutions**

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**ABSTRACT.** Increasing demand for nickel, driven by the expanding electric vehicle battery and steel sectors, has highlighted the importance of efficient extraction methods. Indonesia possesses abundant laterite ore, a primary source of nickel that often contains cobalt as an associated metal. Electrowinning, an electrometallurgical process, is used to extract nickel and cobalt from leach solutions, typically after selective precipitation. This study simulates nickel electrowinning using a synthetic 0.1 M NiSO4​ solution to avoid interference from impurity ions and ensure more accurate results. Key experimental parameters, including potential, solution pH, and duration, were precisely controlled to obtain a nickel deposit that meets industrial requirements. The deposits were characterized using X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy. Applying a potential of 2.8 V yielded highly crystalline Nickel (Ni), whereas a 3.8 V potential resulted in an amorphous β-Ni(OH)₂, as confirmed by XRD and FTIR analysis. At a pH of 3.0, no nickel deposition was observed; instead, only H2​ gas evolution occurred. Conversely, at pH levels of 4.0 and 5.0, Ni metal was deposited at durations of 15 and 30 minutes. A longer duration led to the formation of β-Ni(OH)₂ and promoted the hydrogen evolution reaction. This research provides insight into the importance of fine-tuning electrowinning parameters to produce nickel deposits with a specific phase, a crucial step for optimizing process efficiency and product quality.

**Keywords**: *Nickel, Extraction, Electrowinning, Potential, Research*

# INTRODUCTION

Nickel plays a pivotal role in modern infrastructure and technology, leading to an escalation in demand for its application in batteries, stainless steel, and superalloys [1] [2]. This trend correlates with the intensified exploitation of nickel ores, including those of low grade [3]. The world's nickel resources are predominantly contained within sulfide and laterite ores. Nickel sulfide, primarily sourced from pentlandite ((Ni,Fe)₉S₈), accounts for only about 27% of global nickel reserves. In contrast, low-grade nickel laterite constitutes approximately 70% of global reserves and contributes to 40% of the world's nickel production [4], [5], [6], [7]. As the supply of high-grade sulfide ores diminishes, laterite ores have become an increasingly attractive resource.

Laterite deposits are typically classified into two main types: limonite and saprolite. Limonite is generally found at depths of 0-3 meters, with typical compositions of Ni: 0.76–1.78%, Co: 0.1–0.2%, Fe: 34.10–48.21%, and SiO₂: 9.42–18.02%. Saprolite occurs at greater depths of 3–9 meters, containing Ni: 1.79–2.98%, Co: 0.02–0.1%, Fe: 10.27–34.52%, and SiO₂: 22.0–49.63% [8]. Additionally, the exploration and exploitation costs for laterite ores are lower compared to sulfide ores [9]. However, not all laterite deposits possess nickel grades that are economically viable for processing. The extraction from low-grade ores (Ni content <1.45%) presents a significant challenge, as the resulting nickel product is often associated with cobalt, and conversely, cobalt is often associated with nickel [10]. Cobalt is a high-value metal that is frequently present alongside nickel, making laterite ores a potential secondary source of cobalt [11]. Both nickel and cobalt can be extracted from sulfide and laterite ores using pyrometallurgical and hydrometallurgical methods. In recent years, the application of hydrometallurgical technology for processing nickel and cobalt-bearing ores has gained considerable traction. In general, this process begins with acid leaching, followed by solution purification, which yields intermediate products such as carbonates, hydroxides, and sulfides. These intermediates are then further processed via hydrometallurgy, with the final production of pure metals typically achieved through electrowinning[12].

Electrowinning is an electrometallurgical process that utilizes electric current and voltage to extract metals from their ore solutions. Conventionally, it serves as the final stage in the extraction of valuable metals. However, the standard reduction potentials of nickel and cobalt are remarkably close (E Ni2+/Ni0​ = -0.250 V vs. SHE; E Co2+/Co0​ = -0.277 V vs. SHE), which renders their separation challenging. Although nickel electrowinning has been widely investigated, studies that specifically address the influence of pH, overpotential, and current density on the morphology of nickel deposits from pure sulfate solutions remain limited. This study aims to fill this gap by systematically examining these key parameters to establish the optimum conditions for producing high-quality nickel deposits. In this study, nickel sulfate solutions were used as electrolytes for the electrowinning process to avoid interference from other impurity ions and to provide more accurate results. The research was conducted in two stages. The first stage involved the electrowinning of nickel, where key parameters—including potential (overpotential), solution pH, and electrolysis duration—were precisely controlled (fine-tuned) to determine the optimum conditions for depositing metallic Ni onto the cathode. With optimal parameter conditions, the deposit on the cathode will be characterized to determine the metal phase after the electrowinning process.

# EXPERIMENTAL SECTION

**Materials**

The reagents used in this study included nickel sulphate (NiSO4​), sulfuric acid (H2​SO4, Sigma Aldrich 98%), and deionized water (aquadest)

**Electrowinning Procedure**

The electrowinning process was conducted in an electrolytic bath containing 350 mL of a 0.1 M NiSO4​ solution. A pair of stainless steel plates with dimensions of 3 cm × 5 cm × 0.2 cm were employed as the cathode and anode, with the inter-electrode distance set at 2 cm. The electrolysis process was carried out under a range of applied potentials, namely 2.4, 2.6, 2.8, 3.0, 3.2, 3.4, 3.6, 3.8, and 4.0 V. The process efficiency was evaluated by the change in mass of the cathode, which was weighed before and after electrowinning. For each potential test in the series, the electrolyte solution from the preceding run was reused for the subsequent experiment.

**Materials Characterization**

The solid deposits produced from the electrowinning process were analyzed to identify their mineral phases and functional groups. Crystalline phase identification was performed using X-ray Diffraction (XRD) with an X-ray diffractometer operated at a voltage of 40 kV and a current of 30 mA. The measurement used Cu Kα radiation (λ = 1.5406 Å) over a 2θ diffraction angle range from 5° to 90° with a scan interval of 0.0170° [13], [14]. Subsequently, functional group analysis of the samples was conducted using Fourier Transform Infrared (FTIR) Spectroscopy. Samples were prepared using the KBr pellet method and analyzed within the wavenumber range of 4000–400 cm−1 to identify the existing chemical bonds [15].

# RESULT AND DISCUSSION

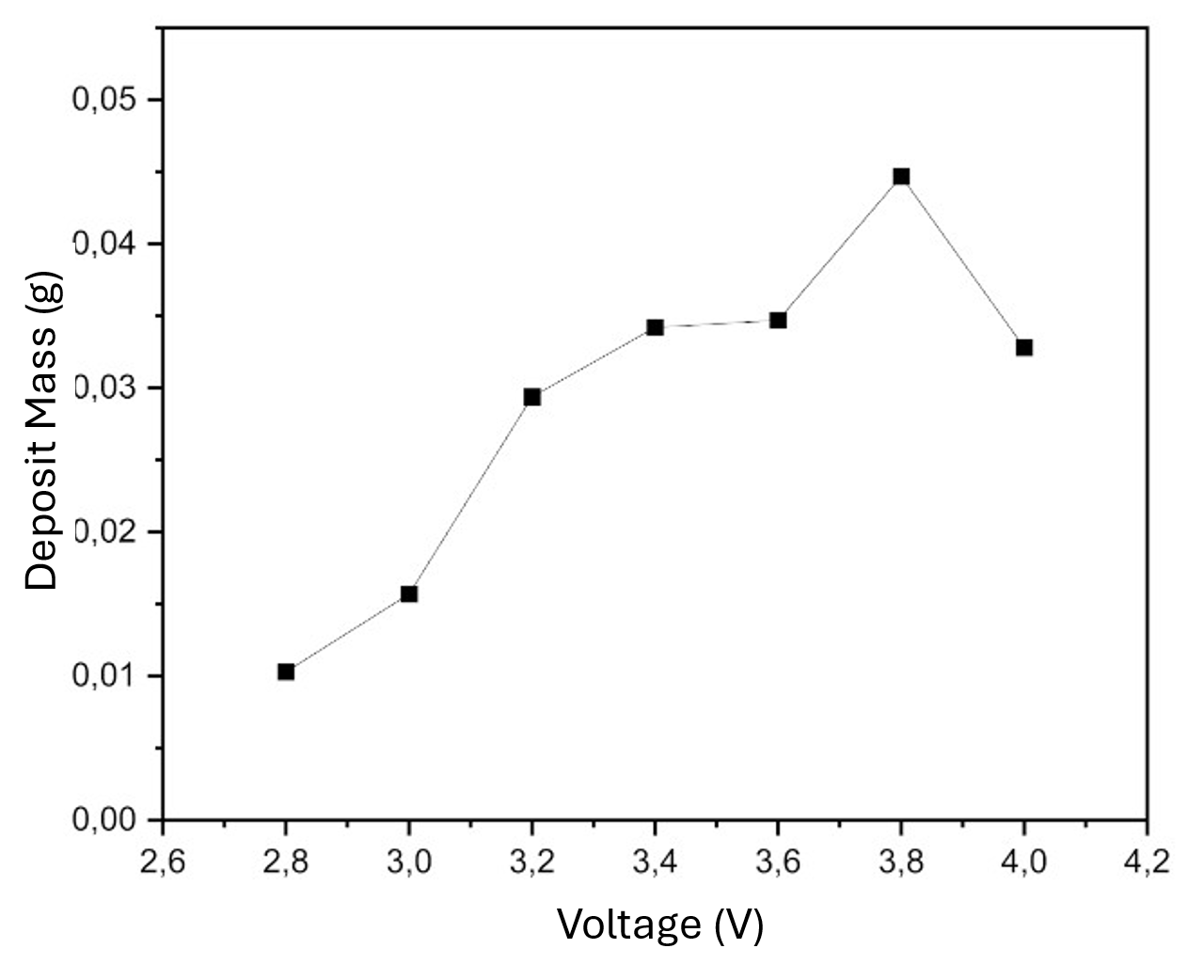
**Effect of Potential on Nickel Electrowinning**

An investigation into the influence of potential on the nickel electrowinning process from a 0.1 M NiSO4​ solution at pH 5 revealed a positive correlation between the applied voltage and the deposit mass up to a certain threshold. The mass of the nickel deposit increased with rising voltage, achieving an optimal value of 0.0447 g at 3.8 V, as depicted in Figure 1.

The nature of the resulting product varied depending on the applied potential. At 2.8 V, a black powder deposit (Ni) weighing 0.0103 g was formed through the reduction of Ni2+ to metallic Ni (Equation 1) [16]. However, in the voltage range of 3–4 V, the product formed was a green powder identified as Ni(OH)2​ (Equation 2). The formation of this nickel hydroxide is attributed to the accelerated rate of water electrolysis at higher potentials, which leads to the local generation of OH− ions in the vicinity of the cathode [16]. This phenomenon occurs due to the competitive reaction between Ni2+ reduction and the hydrogen evolution reaction (HER) (Equation 3) at the cathode [17]. Consequently, the decrease in deposit mass observed at 4 V is attributed to the dominance of the hydrogen evolution reaction, which effectively hinders the nickel deposition process.

|  |  |
| --- | --- |
|  | (1) |
|  | (2) |
|  | (3) |

To drive an electrochemical reaction (such as the reduction of Ni²⁺ or H⁺) at a given rate (i.e., a specific current density), the cathode potential must be shifted from its equilibrium value. The difference between the applied potential and the equilibrium potential is referred to as the overpotential. A higher overpotential (more negative in the case of cathodic reactions) provides a stronger driving force for the reaction, thereby resulting in an increased reaction rate (current density) [18].

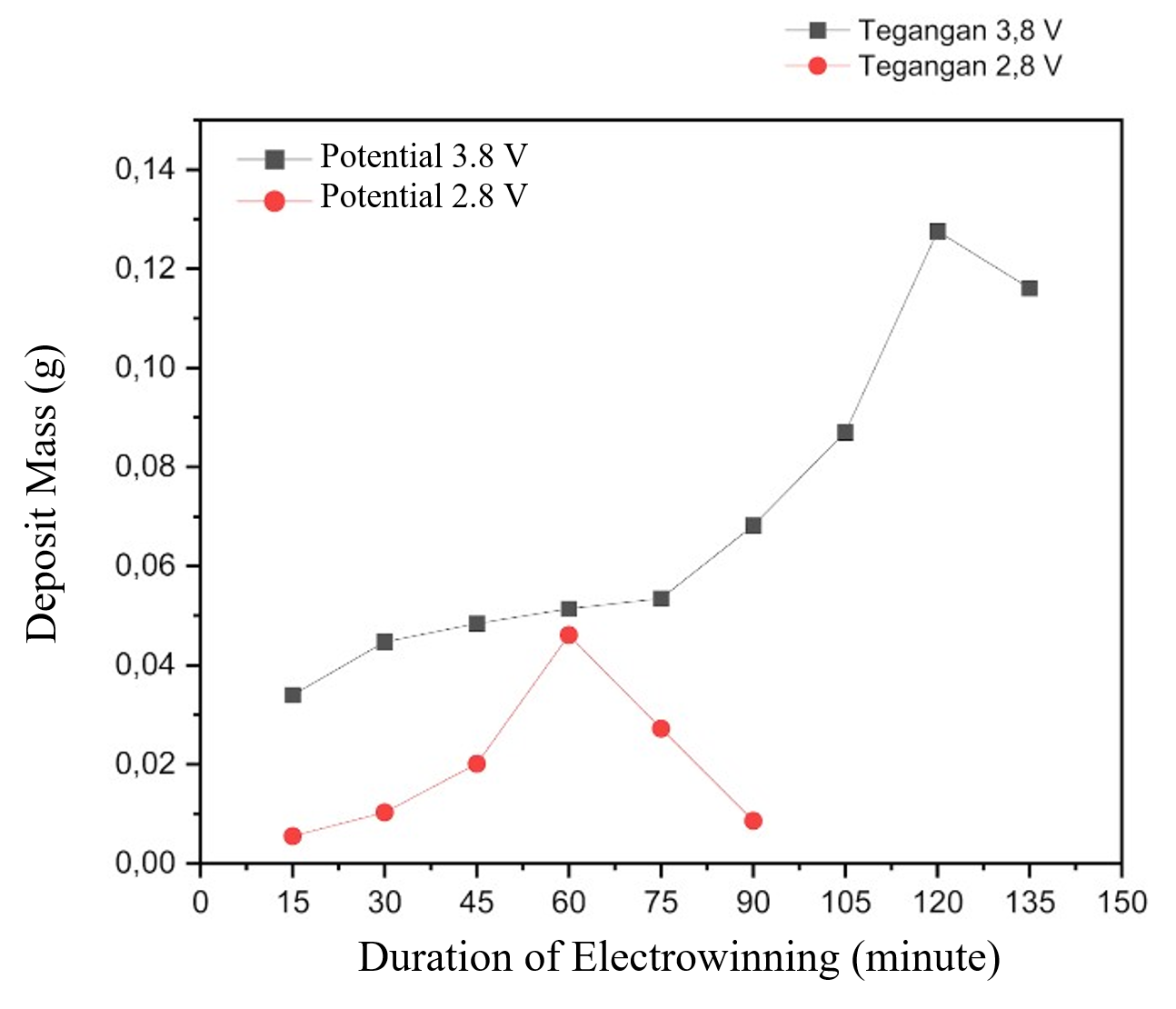


**FIGURE 1.** Effect of Voltage on the Mass of Nickel Deposit

**Effect of Duration on Nickel Electrowinning**

The effect of process duration was evaluated at two key potentials, 2.8 V and 3.8 V, which were previously shown to yield different products. At a potential of 2.8 V, the type of deposit formed was dependent on the duration; a black powder (Ni) was deposited at durations of 15 and 30 minutes, which then transitioned to a green powder (Ni(OH)2​) at 45 minutes and beyond. This transformation is attributed to the accumulation of OH− ions near the cathode over time, which facilitates the precipitation of Ni(OH)2​ [16]. The deposit mass increased with duration, peaking at 60 minutes, as shown in Figure 2.

Conversely, at a potential of 3.8 V, the deposit produced was consistently a green powder (Ni(OH)2​) across all duration variants. The optimum deposit mass at this voltage was recorded at 0.1276 g at a duration of 120 minutes. A decrease in deposit mass was observed at longer durations (75 minutes for 2.8 V and 135 minutes for 3.8 V). This is attributed to the overall decrease in the solution's pH over time, which increases the H+ concentration, thereby intensifying the hydrogen evolution reaction and causing partial dissolution of the previously formed Ni(OH)2​ deposit. In the study by Kazem-Ghamsari et al., time was found to have an influence on the mass of the metal deposit. The increase in deposit mass observed in the present work is consistent with the findings reported by Kazem-Ghamsari et al [19].

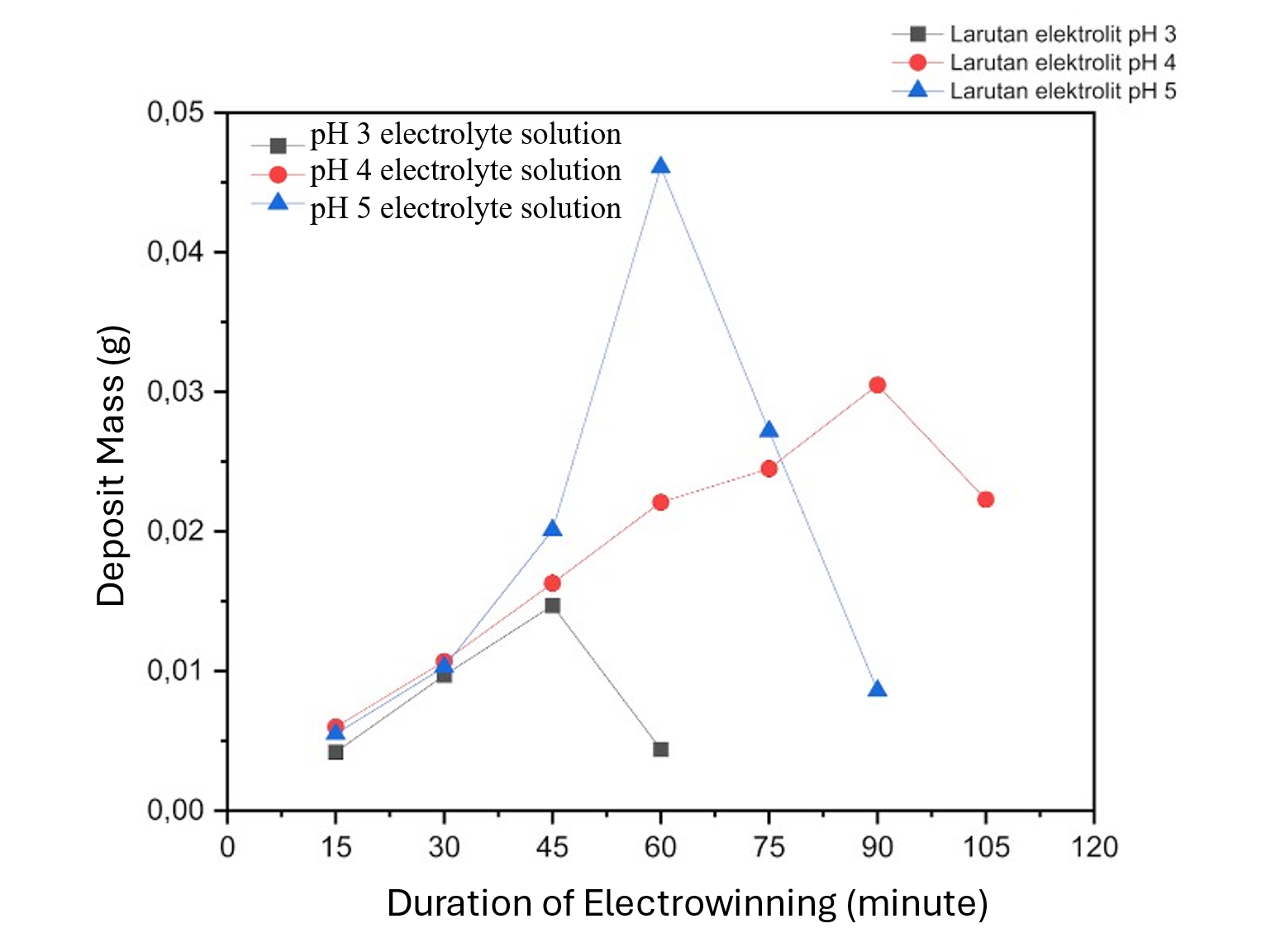


**FIGURE 2.** Effect of Duration Electrowinning on the Mass of Nickel Deposit

**Effect of pH on Nickel Electrowinning**

The influence of the initial electrolyte pH (pH 3, 4, and 5) on the efficiency of nickel electrowinning was studied at a constant potential of 2.8 V. At pH 4 and 5, a similar pattern was observed wherein the initial deposit (at 15 and 30 minutes) was a black powder (Ni), which subsequently transformed into a green powder (Ni(OH)2​) as the duration increased, as shown in Figure 3. This phenomenon is explained by an increase in the local pH at the cathode-electrolyte interface, caused by the consumption of H+ and production of OH− ions during electrolysis, which in turn triggers the precipitation of Ni(OH)2​ [16]. The optimum deposit mass at pH 5 was 0.0461 g (60 minutes), whereas at pH 4, it was 0.0395 g (90 minutes).

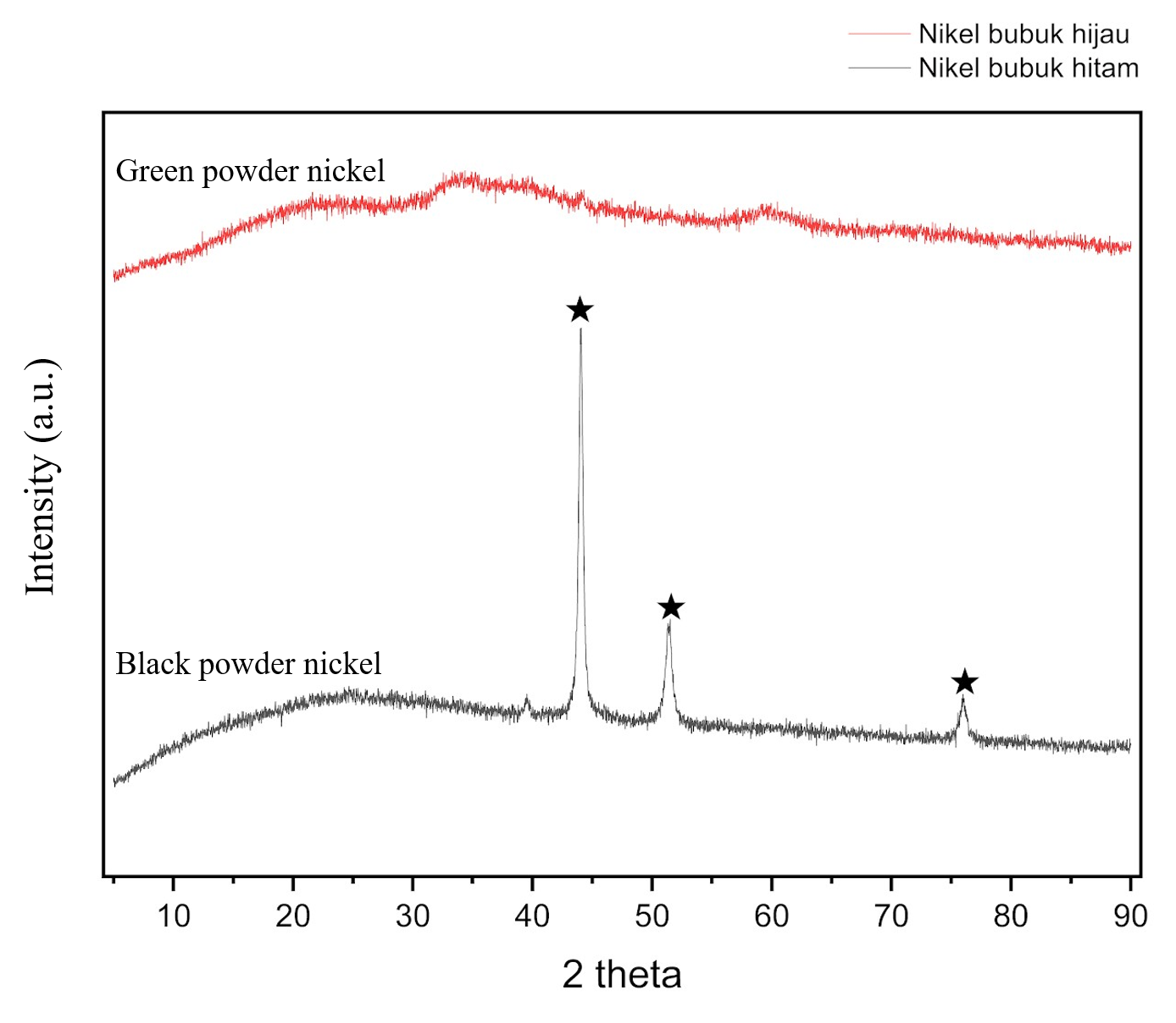
At pH 3, the highly acidic environment inhibited Ni(OH)2​ formation and significantly promoted the hydrogen evolution reaction [19]. As a result, the deposit obtained was of poor quality—consisting only of black patches and a white layer—with a significantly lower maximum mass of 0.015 g achieved at 45 minutes. Under highly acidic conditions, the elevated concentration of H⁺ ions significantly accelerates the kinetics of the hydrogen evolution reaction (HER), making it a strong competitor for cathodic current. Consequently, a substantial portion of the current is diverted to hydrogen gas formation, resulting in a markedly low cathodic current efficiency (CCE). The enhanced HER rate is therefore the primary factor responsible for the inefficiency and poor quality of deposits under these conditions[20][19], [21]. In addition to the main reaction, the electrowinning of nickel is accompanied by a side reaction involving hydrogen reduction [22]. This occurs because the standard potential of hydrogen is more positive than that of nickel deposition, making hydrogen reduction thermodynamically more favorable. Such a reaction is undesirable as it leads to energy and current losses while reducing the quality of the nickel deposit. Furthermore, the formation of Ni(OH)₂ decreases the overall process efficiency, as the desired product is pure metallic nickel.



**FIGURE 3.** Effect of pH on the Mass of Nickel Deposit

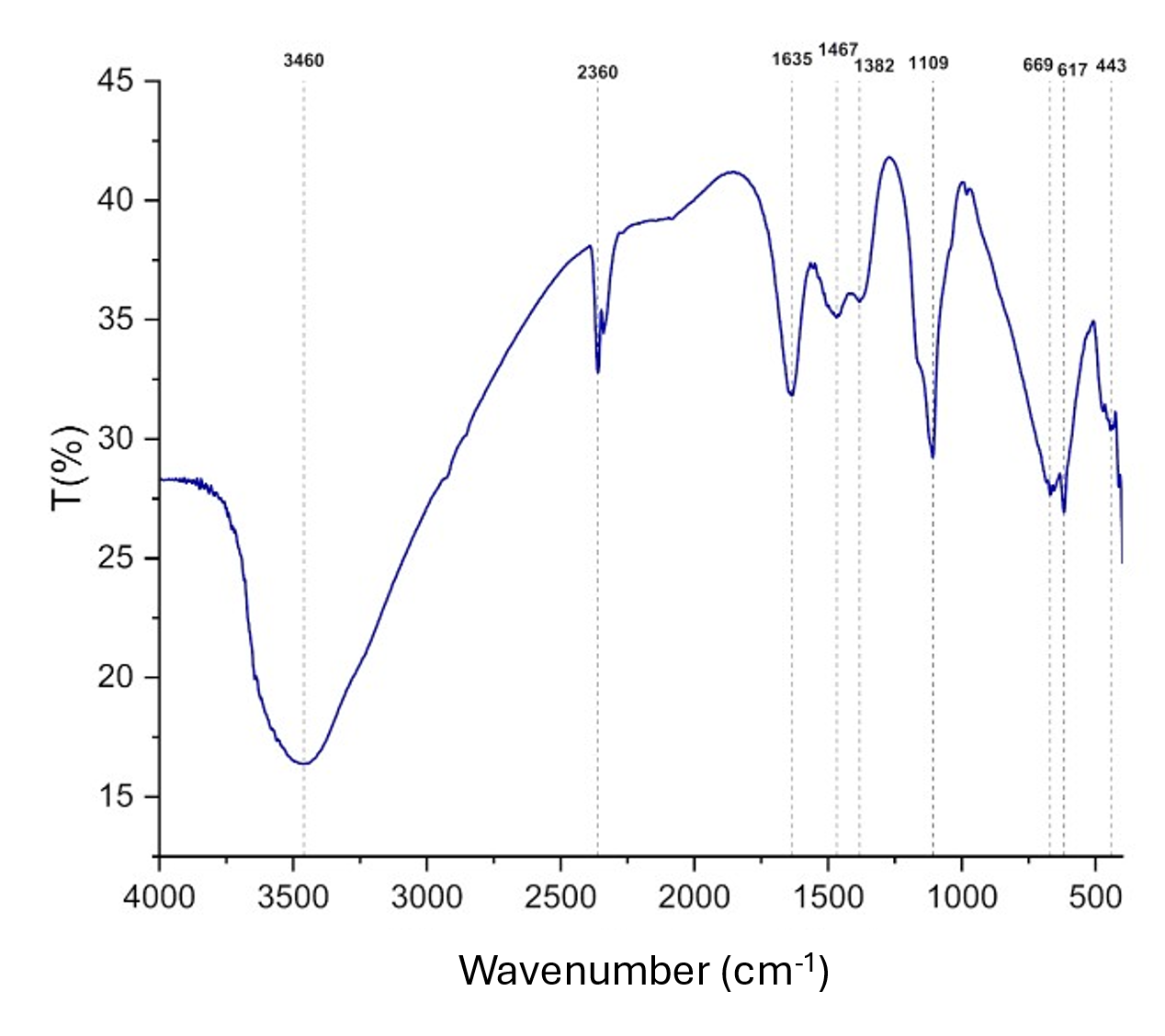
**Characterization of Material**

Characterization of the deposits was performed to identify the phase and composition of the nickel products. X-ray Diffraction (XRD) analysis of the black powder deposit obtained at 2.8 V exhibited sharp diffraction peaks at 2θ = 44.036°, 51.410°, and 75.993°, as shown in Figure 5. These peaks are consistent with the JCPDS standard No. 00-004-0850, confirming that the deposit is metallic nickel (Ni) with a high degree of crystallinity. In contrast, the diffractogram of the green powder deposit (obtained at 3.8 V) did not display any significant diffraction peaks, indicating its amorphous nature.



**FIGURE 4.** XRD Pattern of Nickel Deposited at 2.8 V and 3.8 V

To identify the composition of this amorphous deposit, a Fourier Transform Infrared (FTIR) Spectroscopy analysis was conducted. The FTIR spectrum, presented in Figure 6, reveals absorption bands at ~600 cm⁻¹ (corresponding to -OH stretching in the β-phase) and ~400 cm⁻¹ (Ni-O-H bending vibration). Additionally, a peak at 1109 cm⁻¹ indicates the presence of residual SO42−​ anions, while broad bands at ~3460 cm⁻¹ and 1635 cm⁻¹ are characteristic of H2​O molecules. Collectively, these spectroscopic data confirm that the green powder product is amorphous β−Ni(OH)2​ [23].



**FIGURE 5.** FTIR Spectrum of the Green Nickel Powder Product

# CONCLUSION

This study investigated the electrowinning conditions for nickel by evaluating parameters including potential (overpotential), electrolyte pH, and electrowinning duration. At a potential of 3.8 V with pH values of 4 and 5 for 45 minutes, the obtained solid exhibited a green color corresponding to the Ni(OH)₂ phase. In contrast, a black solid was produced under conditions of 2.8 V at pH 5 for 30 minutes. Considering the focus of this study that mainly focus on applied potential and pH, further investigations are necessary to evaluate the effects of other parameters, such as electrode spacing and electrolyte temperature on nickel and cobalt electrowinning. Therefore, a comprehensive understanding of the electrowinning process of Ni in sulfate solution can be obtained.

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