separations

MDPI

Article

Separation and Characterization of Nickel Hydroxide from Waste Solution Using $Ca(OH)_2$ Precipitation in Chloride Media

Mothepane Happy Mabowa [1,2,*], Andile Mkhohlakali [1,*], Luke Chimuka [2] and James Tshilongo [1,2]

[1] Analytical Chemistry Division, MINTEK, 200 Malibongwe Drive, Randburg 2155, South Africa
[2] School of Chemistry, University of the Witwatersrand, Private Bag x3, Johannesburg 2050, South Africa
* Correspondence: happyc@mintek.co.za (M.H.M.); andilem@mintek.co.za (A.M.)

Abstract: Nickel hydroxide ($Ni(OH)_2$) is a valuable compound derived from nickel, widely utilized across various fields because of its versatile properties. This study aimed to characterize as-prepared β-$Ni(OH)_2$ via chemical precipitation using various analytical techniques. X-ray diffraction (XRD) analysis confirmed the formation of a β-$Ni(OH)_2$ hexagonal crystalline structure, validating the successful precipitation. Fourier-transform infrared spectroscopy (FTIR) spectra exhibited main characteristic peaks of (υOH) and υ(NiO), corresponding to the formation of nickel hydroxide ($Ni(OH)_2$). Subsequently, X-ray photoelectron spectroscopy (XPS) revealed a prominent peak for Ni^{2+} oxidation, confirming the successful precipitation of nickel hydroxide at pH 6.5, which identifies the existence of impurities, such as chlorine and calcium, from the waste matrix. Scanning electron microscopy (SEM) micrographs demonstrated stratified granules with a nearly pure brucite crystalline phase, typical of β-$Ni(OH)_2$. Furthermore, the surface morphology revealed a coarse texture and uneven clustering, suggesting possible elevated oxide levels on the Ni surface. Energy-dispersive X-ray spectroscopy (EDX) confirmed the presence of nickel (Ni) and oxygen (O), with Ca impurities attributed to the chemical precipitation process. Particle size distribution analysis estimated an average particle size of 2.0 μm. Additionally, the precipitation of nickel was investigated using inductively coupled plasma optical emission spectroscopy (ICP-OES). Ni was observed in decreasing order, 62.7 g/L, 0.8 g/L, and 0.501 g/L in the pregnant leach, precursor solution, and solid precipitate (cake), respectively. The separation of $Ni(OH)_2$ through the precipitation process from the waste (acidic chloride media) enabled efficient recycling and re-use of nickel, which provides a cost-effective and environmentally friendly method for the highly efficient utilization of waste (acidic chloride media).

Keywords: nickel hydroxide; precipitation; elemental composition; acidic chloride media

check for updates

Citation: Mabowa, M.H.; Mkhohlakali, A.; Chimuka, L.; Tshilongo, J. Separation and Characterization of Nickel Hydroxide from Waste Solution Using $Ca(OH)_2$ Precipitation in Chloride Media. *Separations* **2024**, *11*, 96. https://doi.org/10.3390/separations11040096

Academic Editor: Mohamed Khayet

Received: 4 March 2024
Revised: 17 March 2024
Accepted: 22 March 2024
Published: 26 March 2024

1. Introduction

Nickel (Ni) can be acquired or recycled through various methods, with conventional approaches typically involving smelting or leaching techniques [1]. Following these initial steps, a refining or finishing process is employed to yield a pure nickel product or derivatives. The demand for Ni sources, especially for nickel-based battery applications, has surged in recent years. At the same time, due to its exceptional performance, nickel—also referred to as the "vitamin of the steel industry"—is widely utilized in a variety of industries as a national strategic resource. In addition, nickel is mostly utilized in the manufacturing of no-ferrous alloys, stainless steel, and superalloys with a high resistance to corrosion and high temperatures. However, traditional methods of nickel extraction often entail environmental consequences and significant costs. As a result, there is growing interest in alternative approaches to Ni recovery, particularly from waste streams. These waste streams, often produced from pyrometallurgy processes from fluxes (nickel sulphide) for platinum group element (PGE) collections, represent both an attractive secondary source and an environmental contaminant. Despite containing valuable resources, they also harbor harmful contaminants. Harnessing waste to produce nickel hydroxide ($Ni(OH)_2$) presents a compelling solution. Not only does it hold significant worth as a material with various applications, but it also presents an eco-friendly approach to recovering Ni. However, accessing $Ni(OH)_2$ from these unconventional sources poses challenges, such as impurities like sodium (Na) or calcium (Ca), which are commonly introduced from the choice of precipitating agent. These impurities can affect the purity and properties of the synthesized $Ni(OH)_2$, necessitating careful optimization of the precipitation process. The waste, if discarded from these unconventional sources, poses challenges, such as strict regulations on Ni concentrations in the effluent, which can constitute up to 10% of the municipal waste stream. This work analyzed the optimal conditions for precipitating Ni compounds from pyrometallurgy process (fire assay) waste, which is generated daily in large volumes.

Among the derivatives, nickel hydroxide holds particular significance due to its widespread applications. The focus on studying the precipitation of nickel hydroxide ($Ni(OH)_2$) stems primarily from its crucial role as a positive electrode material in alkaline rechargeable batteries [2]. Currently, the entire demand for $Ni(OH)_2$ is met through the chemical precipitation technique [2–4]. Consequently, this method of preparation has garnered significant attention and has been extensively researched. Several investigations have been conducted to explore the characteristics of $Ni(OH)_2$, ranging from its structural and morphological properties to its electrochemical behavior, and to understand its properties for application, particularly in the realm of rechargeable battery technology [2–7].

The latter holds significant importance in various fields due to its versatile properties, including catalysis, energy storage, and environmental remediation [8]. Its application in electrochemical devices, such as rechargeable batteries and supercapacitors, has drawn considerable attention owing to its high theoretical capacity and excellent electrochemical performance. In recent years, the synthesis and characterization of β-$Ni(OH)_2$, the most stable phase of the latter, have been extensively studied due to its superior electrochemical properties compared with other phases. This method involves the precipitation of nickel ions (Ni^{2+}) from an aqueous solution, typically using a precipitation agent, such as sodium hydroxide (NaOH) or calcium hydroxide ($Ca(OH)_2$), followed by subsequent washing and drying steps.

The precipitation method stands out as a prominent technique for synthesizing β-$Ni(OH)_2$ due to its simplicity, scalability, and cost-effectiveness. However, achieving precise control over the synthesis parameters, encompassing pH, temperature, and choice of precipitation agent, is crucial to obtaining β-$Ni(OH)_2$ with desired properties. Comprehensive characterization of the synthesized material is essential to understand its structural and functional groups, surface properties, and chemical composition, which directly influence its performance in various applications.

The introduction of alkaline compounds raises the pH of the solution, creating an environment where metal ions exhibit low solubility in the aqueous phase, typically during the hydroxide precipitation process, particularly $Ni(OH)_2$. The chemical reaction that takes place is indicated in Equation (1) [8,9].

$$Ni^{2+} + 2OH^{-} \leftrightarrow Ni(OH)_2 \tag{1}$$

Every metal ion possesses an optimal pH level to achieve the maximum precipitation of metal hydroxides. The pH condition significantly influences the concentration of dissolved metal ions within the solution during the precipitation process [10–13]. To the best of our knowledge, a lack or no optimal conditions for the precipitation of nickel hydroxide from high acid media, such as nickel sulphide, collection of processed waste in different stages have been reported in the literature.

In this context, this study aimed to characterize β-$Ni(OH)_2$ synthesized through a precipitation process. Various analytical techniques, including X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and inductively coupled plasma optical emission spectroscopy (ICP-OES), were employed to elucidate the structural and functional groups, surface properties, and chemical composition of the synthesized $Ni(OH)_2$. This work underpins the significance of $Ni(OH)_2$, its synthesis methods, and the importance of comprehensive characterization. Additionally, the present work holds significant practical implications for enhancing the chemical precipitation method's treatment efficiency, decreasing its operating expenses, and lowering the generation of secondary pollutants throughout the Ni recycling procedure.

2. Materials and Method

2.1. Reagents and Material

The main raw material utilized for this experiment was the waste solution from Analytical Chemistry Division, NiS Laboratory, Mintek, Randburg. This waste solution contains various metal elements as quantified using ICP-OES; the data are presented in Table 1. 5,8-diethyl-7-hydroxydodecan-6-oxime solvent was used as an extractant for Cu during the extraction of metal ions from the pregnant waste solution. All other chemicals are of analytical grade and were used without further purification: diphenylamine, phosphoric acid (H_3PO_4), potassium dichromate ($K_2Cr_2O_7$) (Sigma Aldrich, St. Louis, MO, USA), sulphuric acid (H_2SO_4, 98% *v*/*v*) (Sigma Aldrich), and calcium hydroxide ($Ca(OH)_2$) (Merc (Pty) Ltd., Modderfontein, South Afric).

Table 1. The composition of the pregnant waste solution.

Element Composition	**Al**	**Ca**	**K**	**Mn**	**Na**	**Co**	**Pb**	**S**	**Zn**	**Cu**	**Fe**	**Cl**	**Ni**
Concentration (ppm)	7.3	4.53	22.5	6.7	210	9.8	10	162	73.9	3900	4720	80,000	50,130

2.2. Determination of Ferric (Fe^{3+}) and Ferrous (Fe^{2+}) Ions

The determination of Fe^{3+} and Fe^{2+} was executed as described in [14]. The precursor solution underwent titration to quantify the presence of ferric and ferrous ions. The concentration of ferrous (Fe^{2+}) ions was determined using the dichromatometric method, relying on the reaction described by Equation (2).

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \leftrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O \tag{2}$$

This is an oxidation–reduction process in which an oxidizing agent, potassium dichromate ($K_2Cr_2O_7$), reacts with a reducing agent, Fe^{2+} ions. Diphenylamine serves as a reversible oxidation–reduction indicator in various chemical reactions and analytical methods. Fe^{3+} was determined by the iodometric method, which is based on Equations (3) and (4).

$$2Fe^{3+} + 2I^- \rightarrow 2Fe^{2+} + I_2 \tag{3}$$

$$I_2 + 2S_2O_6^{2-} = S_4O_6^{2-} + 2I^- \tag{4}$$

This is an indirect oxidation–reduction process in which an oxidizing agent, ferric ions (Fe^{3+}), reacts with a reducing agent (iodide ions). The observed results from the above titration method indicated the Fe^{3+}-bearing waste, which gives an insight into the precipitate of the Fe ion at a lower pH, around 2 to 2.5.

2.3. Synthesis/Preparation Process of $Ni(OH)_2$

The synthesis of $Ni(OH)_2$ involved two distinct steps: (1) preparation of the precursor solution (2) and synthesis of $Ni(OH)_2$. As outlined in the referenced literature [15], with minor adjustments, the pregnant waste solution (WPS) harbored valuable copper (Cu) that could be extracted using solvent extraction (SX). In this method, WPS was introduced into a 1000 mL separating flask at an organic/aqueous ratio of 1:5. Utilizing 5,8-diethyl-7-hydroxydodecan-6-oxime as the organic solvent facilitated the extraction of Cu into the organic phase before the precipitation step. This process is illustrated in Figure 1.

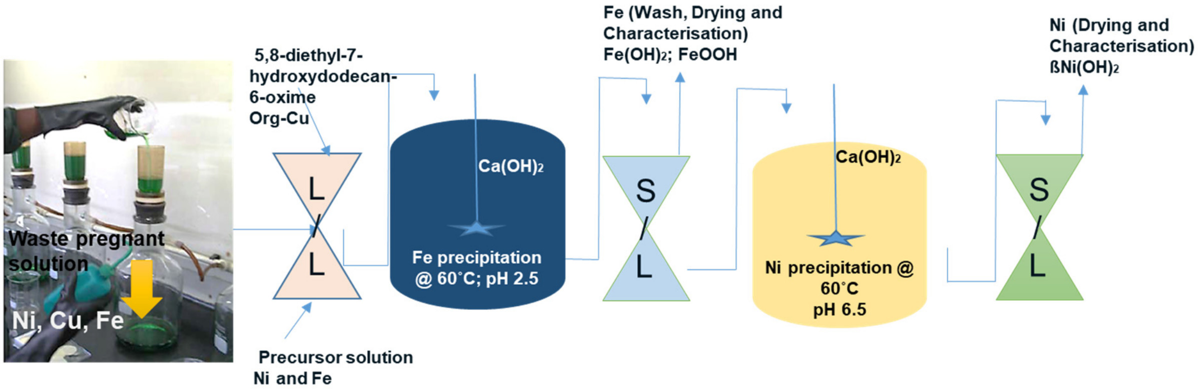

Figure 1. The workflow of $Ni(OH)_2$ synthesis from the waste pregnant solution.

Following the SX process, the raffinate was obtained, and the aqueous phase was identified as the precursor solution of the PWS. The precursor solution measured approximately 5000 mL and underwent a chemical precipitation reaction involving the metal ions of the WPS, particularly the Ni^{2+} ions. To prepare for the reaction, a 25% $Ca(OH)_2$ solution was separately created based on the approximate concentration of the chloride media in the WPS. The precipitation reaction occurred at a temperature of 60 °C and pH levels of 2.5 and 6.5, where the precursor solution was blended with the 25% $Ca(OH)_2$.

A glass reactor with an overhead stirrer was utilized during the extraction process to ensure that the seeding material was effectively suspended in the solution. During the process, the temperature was continually monitored utilizing a thermometer, while pH levels were measured using a pH meter. The operation maintained a temperature of 60 °C, with heating supplied by passing hot water through the reactor's jacket.

Once the level of pH reached the target range of 6.5 to 7, the stirring was stopped, and the fluid was filtered using a Buchner funnel. The cake was subsequently dehydrated for 24 h at a temperature of 55 °C. Throughout this phase of evaporation, the phenomenon of precipitation persisted under its fundamental mechanism. Subsequently, the liquid phase was examined to ascertain the residual concentration of Ni^{2+} ions by employing analytical techniques to yield vital insights into the efficacy and efficiency of the precipitation process.

2.4. The Effect of Precipitation

This experimental study was conducted under conditions designed at pH 6.5, which represents the optimal pH for precipitating Ni^{2+} ions [15] from PWS. After precipitation, the solution was characterized by ICP-OES. As reported in previous studies, [12] reported the optimum pH for precipitating Ni^{2+} ions at pH 10. Similarly, studies in the literature such as [13] observed that an alteration in the stability of metal ions might arise at increased operational temperatures, notably at 60 °C, throughout the hydroxide precipitation procedure. This occurrence was substantiated by formation of metal oxides and hydroxide deposits at reduced pH levels. The precipitation reaction involved in synthesizing $Ni(OH)_2$ is reversible due to the formation of complex compounds that are insoluble in liquids. Typically, this reaction is exothermic, meaning it releases heat. At higher temperatures, the precipitation reaction occurs, and the equilibrium tends to shift toward the reactants, as explained in the literature [13]. Consequently, this inhibits the formation of precipitates, resulting in a smaller amount being produced. Thus, controlling the temperature during the precipitation process was crucial to ensure optimal yield and efficiency in the synthesis of $Ni(OH)_2$.

2.5. Characterization

The characterization of $Ni(OH)_2$ involved the utilization of several analytical techniques to elucidate its structural, vibrational, surface properties, and chemical composition. The analytical techniques employed included XRD analysis, Panalytical Empyrean Diffractometer, Cu anode kα = 1.5406 Å, data acquired at 45 kV: 40 mA, 2θ scan range of 5 to 100 degrees, step size 0.01 degrees was performed to ascertain the crystalline structure of the synthesized $Ni(OH)_2$ and validate its phase purity. FTIR (SHIMADZU) spectroscopy was employed to analyze the vibrational modes of $Ni(OH)_2$ and identify functional groups present in the compound. XPS analysis provided insights into the surface chemistry of $Ni(OH)_2$, allowing for the identification of surface species and elemental composition. SEM imaging, Zeiss Cross Beam 540 Morphology utilizing an InLens detector, imaging was conducted at 2 kV, 79 pA, and a 5 mm working distance. Utilizing a backscatter electron detector, cross-section imaging was obtained at 20 kV and 5 nA. to visualize the morphology and microstructure of the synthesized $Ni(OH)_2$, providing information on shape and surface morphology. ICP-OES, Agilent 5110 Vertical dual view (VDV) USA was utilized to quantitatively analyze the chemical composition of $Ni(OH)_2$, providing data on the elemental composition and concentration of impurities.

3. Results and Discussion

3.1. The Elemental Composition of Waste Pregnant Leach

The elemental composition of waste pregnant leach is displayed in Table 1, indicating the major and minor elements. It can be seen that the waste pregnant leach contains higher Ni (50,130 ppm), followed by Cl (80,000 ppm), Fe (4720 ppm), and Cu (3900 ppm). The highest concentration of Nickel and Cl is attributed to nickel sulphide (NiS) flux present in the waste (acidic chloride) and hydrochloric acid (HCl) leaching acid, respectively.

3.2. The Effect of Pre-Loading of Copper Concentration

The copper (Cu) loading was applied onto the organic material, demonstrating the effectiveness of the oxime utilized, as stated by the report [16]. The 5,8-diethyl-7-hydroxydodecan-6-oxime had a significant attraction toward metal cations, specifically copper, which resulted in a high extraction efficiency, as documented in the literature [17,18]. In the precursor solution, the aqueous phase exhibited a Cu concentration of 801 ppm at pH 2.5 and 501 ppm at pH 6.5, as evaluated by ICP-OES. Additionally, the Cu concentration at pH 6.5 was found to be 95.7 ppm in Figure 2. As indicated in Table S1 (Supplementary Information), copper was trapped (loaded) in the organic phase, exhibiting a high concentration (3097.96 ppm), indicating the high efficiency of 5,8-diethyl-7-hydroxydodecan-6-oxime. Furthermore, after the precipitation at pH 6.5, the filtrate during the wash showed the lowest concentration of 7.43 ppm, as illustrated in Table S1 (Supplementary Information).

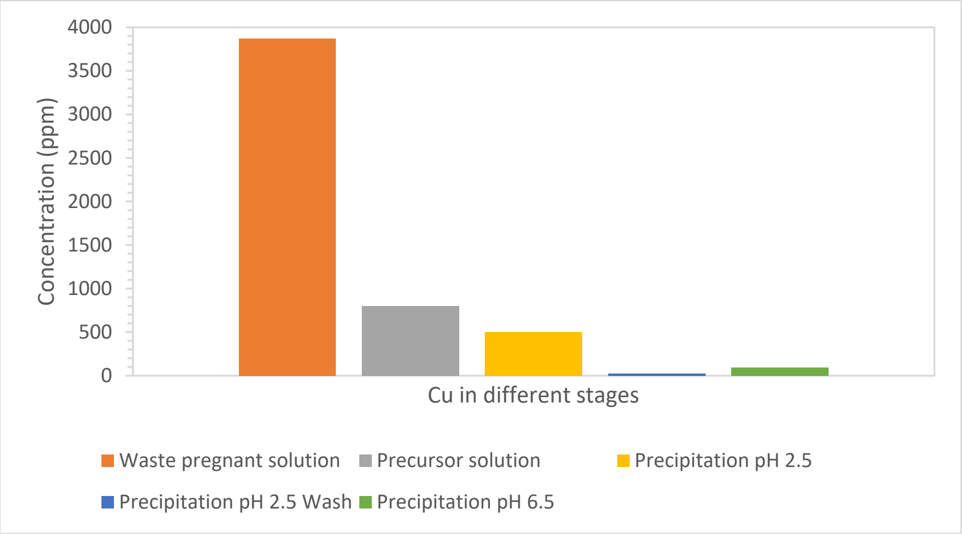

Figure 2. The copper concentration in subsequent stages was higher (concentrated) in waste solution (pregnant solution), followed by precursor solution, and filtrate after precipitation at different pH (2.5 and 6.5).

Figure 2 represents the copper concentration in different steps (stages), as depicted in Figure 1. After the pre-loading stage of Cu removal, the solution was analyzed, and the precursor solutions contained 500,130 ppm of Ni^{2+} ions, 20,197 ppm of Fe^{3+}, and 801 ppm of Cu^{2+} ions and other impurities shown in Table 2. It can be seen that there is a significant drop in Cu concentration, from 3870 ppm to 801 ppm to 501 ppm, in the waste pregnant solution and precursor solution, respectively. This could be ascribed to Cu being trapped in the organic phase by oxime functional groups, as expressed in Equation (5).

$$2RH_{(Org)} + Cu^{2+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow R_2Cu_{(org)} + 2H^{+}_{(aq)} + Cl^{-}_{(aq)} \quad (5)$$

Table 2. Solution after precipitation at pH 2.5 and 6.5 at 60 °C.

		Solution Sample			
Element		**Precursor Solution**	**pH 2.5**	**Wash**	**pH 6.5**
Cu	ppm	801	501	23.3	95.7
Fe		2197.5	2.3	1.3	0
Ni		50,130	3700	1800	600
Co		43	26.4	1.5	0.7
Ca		0	47,400	2300	57,200
Al		1	10.8	1.2	1.1
Zn		72.7	40.2	2.2	0
S		134.1	446	19.7	456.3
Mg		0	461.9	23.9	375
Pb		14	8	0.2	0

The observed trend in this study aligns with the findings in Ref. [19]. The aforementioned study demonstrated that the optimal conditions for investigating the precipitation process of other metal ions, such as Fe, in a mixture with nickel (Ni) is achieved at higher temperatures. Specifically, the utilization of elevated temperatures has an impact on decreasing the amount of Ni precipitated (Ni loss) during the Fe precipitation procedure at pH 2.5, as illustrated by this group in Ref. [15]. Thus, this study confirmed the precipitation process of Ni^{2+} ions operates optimally at a temperature of 60 °C and a pH of 6.5, as indicated in Table 2, which resulted in 600 ppm Ni in solution from 62,700 ppm Ni in the pregnant waste solution (Table 1). The decrease in Ni concentration from 3700 ppm to 600 ppm is as expected; it means that at pH 6.5, only 600 ppm is left in the solution. A high concentration of Ni is in the precipitate (cake). This is the key part of this study: at pH 2.5, there is a nickel loss co-existing with Fe precipitation. Indeed, at pH 2.5, Ni co-exists with Fe, as explained by this group in Ref. [15]. In addition, the EDX is shown in Figure S3, which verifies the presence of Ni and Fe in the precipitate. Moreover, a lower concentration of nickel (600 ppm) in the solution and a higher concentration in the solid cake indicates the effectiveness of the precipitation process using $Ca(OH)_2$.

3.3. *The Effect of Precipitating Agent on Decreasing Ni^{2+} Ion Concentration*

The precipitating agent $Ca(OH)_2$ was used, and it showed a high efficiency for the precipitation of $Ni(OH)_2$, as displayed in Table 2. The results are in strong agreement with Ref. [12]. Wanta and co-workers identified alkaline media (KOH) as the most effective precipitating agent. Calcium shows an exponential increase from 0.0 ppm to 47,400 ppm and 57,200 ppm in a precursor solution and is a precipitate at pH 2.5 and a precipitate at pH 6.5, respectively [12]. The Ca increase is due to the addition of lime in the precipitate. The Fe loss at pH 6.5 indicates the selective precipitation of Ni at higher pH. In addition, pH 2.5 indicates the co-existence of Ni and Fe as impurities.

3.4. Structure–Morphology Characterization of Ni(OH)$_2$ Precipitates

Figure 3a illustrates the SEM imaging, a stratified granule composed of approximately pure brucite crystalline phase in spherical morphology particles, which is characteristic of β-Ni(OH)$_2$. This finding supports the findings of prior research [15]. The coarse texture may be indicative of elevated levels of oxides on the surface of Ni, while the uneven clustering is also characteristic of the typical formations that occur during the precipitation process. Furthermore, the aggregation may be ascribed to factors other than post-treatment. EDX in Figure 3b verifies the presence of nickel (Ni) and oxygen (O) derived from the oxides, namely hydroxides. The presence of Ca impurities can be ascribed to the lime originating from the chemical precipitating process with Ca(OH)$_2$. The average particle size was estimated from the PSD plot and was found to be 2.0 µm; see the Supplementary Information.

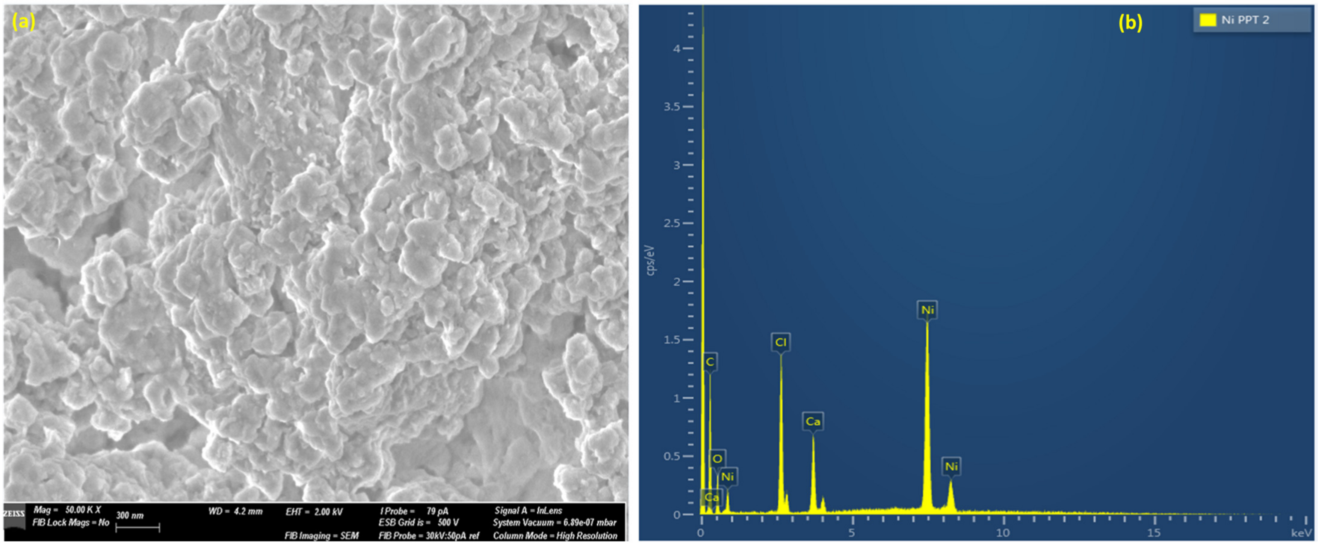

Figure 3. (**a**) SEM image and (**b**) EDX of Ni(OH)$_2$.

The X-ray diffraction (XRD) pattern depicted in Figure 4a corresponds to the formation of β-Ni(OH)$_2$. All diffractogram peaks of Ni(OH)$_2$ indicate the presence of the hexagonal phase of β-Ni(OH)$_2$ at 24°, 29°, 30°, 45°, 50°, and 60° and correspond to the crystal planes (001), (100), (101), (102), (103), and (110), respectively [20,21].

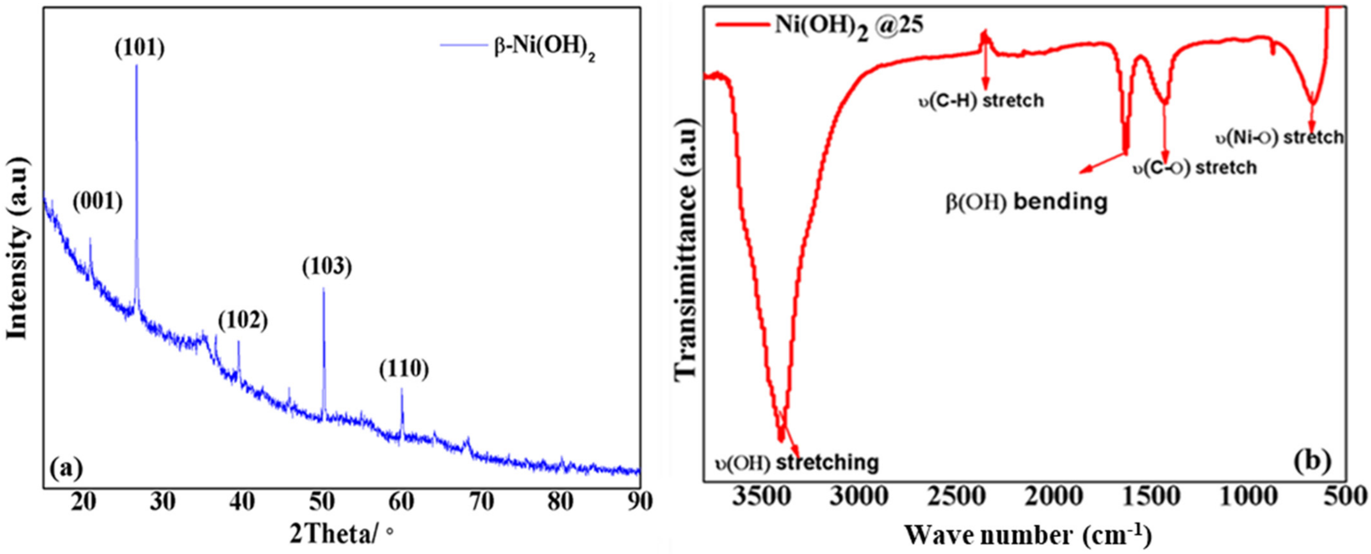

Figure 4. (**a**) XRD pattern of Ni(OH)$_2$ and (**b**) FTIR of Ni(OH)$_2$.

In Figure 4b, the FTIR spectra of $Ni(OH)_2$ exhibit a prominent functional group between 450 and 600 cm^{-1} and 1100 cm^{-1}, which correspond to υ(Ni-O) stretching vibrations of β-$Ni(OH)_2$ and υ(O-Ni-O), correspondingly. The presence of υ(Ni-O-Ni) is indicated by a stretched vibration peak at around 1000 cm^{-1}. These observations align with the findings in the literature [21]. The prominent bending vibration band seen at approximately 1600 cm^{-1} is ascribed to the presence of a -OH moiety in $Ni(OH)_2$. The peak at 3450 cm^{-1} corresponds to the asymmetric stretching mode of the υ(OH) vibration, which is attributed to the presence of water or metal hydroxide in the α-phase v(Ni-OH). The peak observed at 1620 cm^{-1} is attributed to the δ(OH) bending on the surface of Ni. The abovementioned findings validate the successful precipitation of $Ni(OH)_2$. The additional vibration bands at approximately 2200 cm^{-1} and 1330 cm^{-1} are ascribed to the stretching of v(C-O) and υ(C-H) caused by the presence of small contaminants, which are attributed to carbon species in the waste stream.

The electronic structure and oxidation state of nickel hydroxide were determined using XPS, as shown in Figure 5a. Spectroscopic analysis of $Ni(OH)_2$ ranges between 0 and 1300 electron volts. Photoelectron lines seen at 861 eV, 510 eV, and 300 eV correspond to the Ni 2p, O 1s, and C 1s orbitals, respectively. This suggests that the successful precipitation of $Ni(OH)_2$ occurred at a pH of 6.5. The photoelectron peaks observed at around 200 eV and 350 eV can be attributed to the presence of chlorine (Cl) and calcium (Ca) impurities originating from the waste matrix of hydrogen chloride (HCl) and lime, respectively. The photoelectron line of oxygen peak (O 1s) can be identified as distinct peaks, as illustrated in Figure 5d. The presence of hydroxyl species on the surface of $Ni(OH)_2$ is verified by the oxygen photoelectron line (O 1s) peak of MeO (metal-oxide peak), which appears at around 533 eV. The presence of CO_2 impurities found with FTIR in Figure 5 is indicated by the other O 1s peaks. Table 3 summarizes the elemental composition of β-$Ni(OH)_2$; high chloride (metal chloride) (22.4 wt%) can be observed at 199.3 eV binding energy, which can be due to acidic chloride metal and $CaCl_2$ formed during the precipitation steps. In addition, the XPS spectra line for $CaCl_2$ is situated at 388.1 eV. The oxygen of the hydroxyl species is located at 531 eV and 533 eV binding energy, which is ascribed to metal-hydroxyl formation, particularly β-$Ni(OH)_2$. The results are in strong agreement with EDX (Figure 3), ICP-OES (Table 2), and FTIR (Figure 4b).

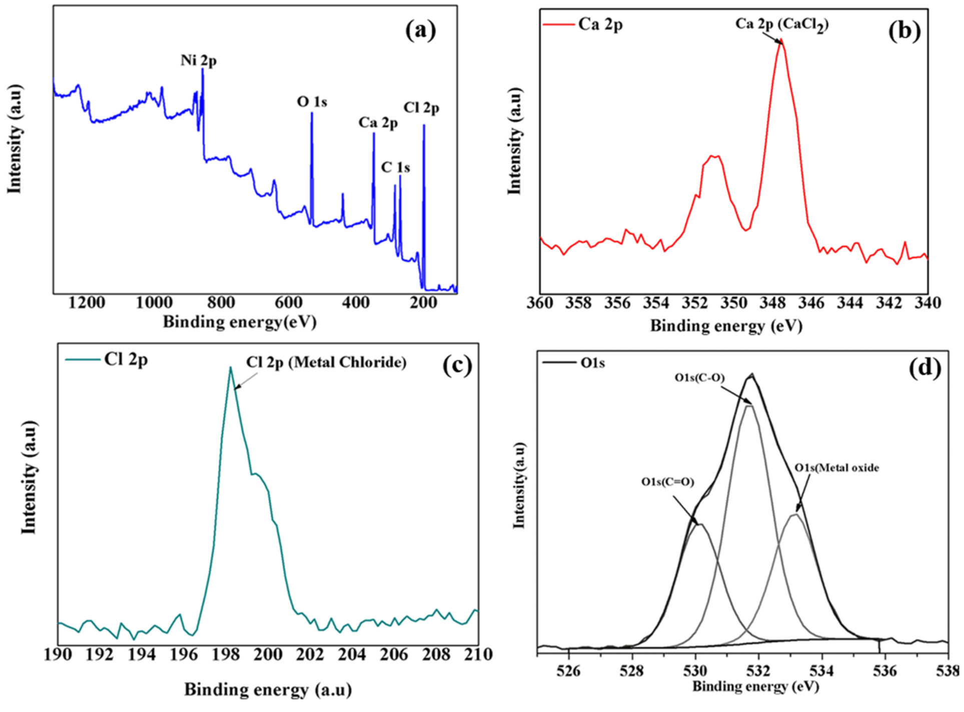

Figure 5. (**a**) XPS spectra survey, (**b**) Ca 2p spectra, (**c**) Cl 2p spectra, and (**d**) O 1s spectra of β-$Ni(OH)_2$.

Table 3. Elemental composition of β-$Ni(OH)_2$ at pH 6.5.

Element	Peak BE (eV)	Atomic %
C 1s	285.2	36.1
O 1s	531.7	23.5
Cl 2p	199.3	22.4
Ni 2p	856.2	11.8
Ca 2p	348.1	5.2

4. Conclusions

In this study, the preparation and comprehensive characterization of β-$Ni(OH)_2$ from waste (chloride media) synthesized via a precipitation process and advanced analytical techniques was conducted successfully. XRD exhibited and FTIR provided the existence of a -OH moiety, which is the attribute of $Ni(OH)_2$ at 3450 cm^{-1}, 450 and 600 cm^{-1}, and υ(Ni-O) stretching vibrations of β-$Ni(OH)_2$. XPS analysis confirmed the formation of a Ni^{2+} oxidation state, which corresponds to the presence of $Ni(OH)_2$. Furthermore, SEM imaging revealed granular morphology consistent with β-$Ni(OH)_2$, indicating the successful chemical precipitation from the waste. The major elements Ni, Cu, and Fe, quantified by EDX and ICP-OES, showed consistency in chemical composition. Overall, this study enhances our understanding of β-$Ni(OH)_2$ synthesis and properties, which are crucial for its potential applications, and suggests avenues for further research to optimize synthesis conditions and explore specific applications. These findings underscore the efficacy of the chemical precipitation method for synthesizing $Ni(OH)_2$ and highlight lime as a promising precipitating agent for future applications in the production of high-quality nickel hydroxide compounds. The precipitation process using $Ca(OH)_2$ for $Ni(OH)_2$ preparation from waste (chloride media) enables effective recycling and re-use of nickel, which provides an economical method for the highly efficient utilization of waste (acidic chloride media).

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/separations11040096/s1: The utilization of advanced analytical techniques and high-resolution instruments are essential for the synthesis of nickel hydroxide from waste solution using $Ca(OH)_2$ precipitation from a chloride media. The supporting information provided in Figures S1 and S2 supports the comprehensive analysis of the PSD and elemental composition of the sample by ICP-OES. Figure S1. Particle Size Distribution (PSD) $Ni(OH)_2$, Figure S2. Elemental composition of the sample by ICP-OES $Ni(OH)_2$, and Figure S3. EDX precipitate at pH 2.5. Table S1. Concentration of Cu in the organic phase in consequent stages.

Author Contributions: M.H.M.: Conceptualization, Methodology, Validation, Writing original draft. A.M.: Formal analysis, Investigation, Data curation, Writing original draft, L.C.: Supervision, Data curation, and statistic validation, J.T.: Supervision, editing, visualization, and acquire funding. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Mintek grant number: ASR-00002313 (HM-05).

Data Availability Statement: Data will be available upon the request.

Acknowledgments: The authors wish to extend heartfelt thanks to Mintek for their financial support granted through Science Vote grant number ASR-00002313 (HM-05), as well as for granting permission to publish this research.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Odegbemi, F.; Idowu, G.A.; Adebayo, A.O. Nickel recovery from spent nickel-metal hydride batteries using LIX-84I-impregnated activated charcoal. *Environ. Nanotechnol. Monit. Manag.* **2021**, *15*, 100452. [CrossRef]
2. Watanabe, K.; Kikuoka, T.; Kumagai, N. Physical and electrochemical characteristics of nickel hydroxide as a positive material for rechargeable alkaline batteries. *J. Appl. Electrochem.* **1995**, *25*, 219–226. [CrossRef]
3. Avena, M.J.; Vazquez, M.V.; Carbonio, R.E.; De Pauli, C.P.; Macagno, V.A. A simple and novel method for preparing $Ni(OH)_2$ Part I: Structural studies and voltammetric response. *J. Appl. Electrochem.* **1994**, *24*, 256–260. [CrossRef]
4. Fierro, C.; Zallen, A.; Koch, J.; Fetcenko, M.A. The Influence of Nickel-Hydroxide Composition and Microstructure on the High-Temperature Performance of Nickel Metal Hydride Batteries. *J. Electrochem. Soc.* **2006**, *153*, A492. [CrossRef]
5. Ramesh, T.N.; Kamath, P.V. Synthesis of nickel hydroxide: Effect of precipitation conditions on phase selectivity and structural disorder. *J. Power Sources* **2006**, *156*, 655–661. [CrossRef]
6. Cabanas-Polo, S.; Suslick, K.S.; Sanchez-Herencia, A.J. Effect of reaction conditions on size and morphology of ultrasonically prepared $Ni(OH)_2$ powders. *Ultrason. Sonochem.* **2011**, *18*, 901–906. [CrossRef] [PubMed]
7. Solomane, N.; Ajibade, P.A.; Omondi, B. Crystal structure of sodium morpholine-4-carbodithioate, ($C_5H_{12}NNaO_3S_2$). *Open Access* **2019**, *234*, 605–607. [CrossRef]
8. Matthews, J.A. Chemical Precipitation. *Encycl. Environ. Chang.* **2014**, *3*, 141–142. [CrossRef]
9. Song, Q.; Tang, Z.; Guo, H.; Chan, S.L.I. Structural characteristics of nickel hydroxide synthesized by a chemical precipitation route under different pH values. *J. Power Sources* **2002**, *112*, 428–434. [CrossRef]
10. Ludwig, R.D.; McGregor, R.G.; Blowes, D.W.; Benner, S.G.; Mountjoy, K. A permeable reactive barrier for treatment of heavy metals. *Ground Water* **2002**, *40*, 59–66. [CrossRef]
11. Endo, M.; Yoshikawa, E.; Tamaki, Y.; Hara, A.; Hikichi, K.; Sasaki, A. Variations in the Concentration of Dissolved Metal Ions and their Buffering Effect in an Acidified River Environment. *J. Water Environ. Technol.* **2012**, *10*, 463–471. [CrossRef]
12. Wanta, K.C.; Tanujaya, F.H.; Putra, F.D.; Susanti, R.F.; Gemilar, G.P.; Astuti, W.; Petrus, H.T.B.M. Synthesis and Characterization of Nickel Hydroxide From Extraction Solution of Spent Catalyst. *Metalurgi* **2020**, *35*, 111. [CrossRef]
13. Hidmi, L.; Edwards, M. Role of temperature and pH in $Cu(OH)_2$ solubility. *Environ. Sci. Technol.* **1999**, *33*, 2607–2610. [CrossRef]
14. Asakai, T.; Suzuki, T. Reliability in Standardization of Iron(III) and Titanium(III) Solutions in Volumetric Analysis. *ACS Omega* **2021**, *6*, 21147–21152. [CrossRef] [PubMed]
15. Mabowa, H.M.; Mkhohlakali, A.; Mokoena, S.; Tshilongo, J.; Chimuka, L. Removal of Nickel from Nickel Sulfite-Fire Assay Dissolution Filtrate Through Precipitation. *ACS Omega* **2023**, *9*, 5592–5600. [CrossRef]
16. Nozari, I.; Azizi, A. An Investigation into the Extraction Behavior of Copper from Sulfate Leach Liquor Using Acorga M5640 Extractant: Mechanism, Equilibrium, and Thermodynamics. *Min. Metall. Explor.* **2020**, *37*, 1673–1680. [CrossRef]
17. Younas, M.; Druon-Bocquet, S.; Romero, J.; Sanchez, J. Experimental and Theoretical Investigation of Distribution Equilibria and Kinetics of Copper(II) Extraction with LIX 84 I and TFA. *Sep. Sci. Technol.* **2015**, *50*, 1523–1531. [CrossRef]
18. Basturkcu, H.; Acarkan, N. Selective nickel-iron separation from atmospheric leach liquor of a lateritic nickel ore using the para-goethite method. *Physicochem. Probl. Miner. Process.* **2017**, *53*, 212–226. [CrossRef]
19. Kettaf, S.; Guellati, O.; Harat, A.; Kennaz, H.; Momodu, D.; Dangbegnon, J.; Manyala, N.; Guerioune, M. Electrochemical measurements of synthesized nanostructured β-$Ni(OH)_2$ using hydrothermal process and activated carbon based nanoelectroactive materials. *SN Appl. Sci.* **2019**, *1*, 34. [CrossRef]
20. Qin, Z.; Wang, Y.; Huang, X.; Shen, W.; Yu, J.; Li, J. A Facile Synthesis of Three Dimensional β-$Ni(OH)_2$ Composed of Ultrathin Nanosheets for High Performance Pseudocapacitor. *J. Inorg. Organomet. Polym. Mater.* **2020**, *30*, 2089–2097. [CrossRef]
21. Narayan, R.T. Effect of Crystallinity of β- and β bc-Nickel Hydroxide Samples on Chemical Cycling. *Indian J. Mater. Sci.* **2015**, *2015*, 820193. [CrossRef]