e-ISSN: 2682-8588

PENERBIT PRESS
UNIVERSITI TEKNOLOGI MARA

Available online at
https://journal.uitm.edu.my/ojs/index.php/MJCET

Malaysian Journal of Chemical Engineering & Technology 9(1) 2026, 65–78

Malaysian Journal of Chemical Engineering and Technology

Nickel recovery from Raney nickel by leaching and precipitation: Towards resource valorisation

Muhammad Hairul Haizan Azizan[1], Norhaslinda Nasuha[2], Muhammad Syafiq Hazwan Ruslan[3], Hawaiah Imam Maarof [4*]

[1,2,3,4]*Faculty of Chemical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*
[2,4]*Hybrid Nanomaterials, Interfaces & Simulation (HYMFAST), Faculty of Chemical Engineering, Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, 13500 Permatang Pauh, Pulau Pinang, Malaysia*
[3]*Integrated Separation Technology Research Group (i-STRONG), Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

ARTICLE INFO

Article history:
Received 17 November 2025
Revised 06 January 2026
Accepted 09 March 2026
Online first 30 April 2026
Published 30 April 2026

Keywords:
Leaching
Precipitation
Metal recovery
Spent catalyst
Nickel
Hydrometallurgy

DOI:
10.24191/mjcet.v9i1.8976

ABSTRACT

Spent catalysts are often rich in valuable metals and are typically discarded, leading to both resource loss and environmental issues. Recovering nickel from spent catalysts is thus crucial for improving resource efficiency and sustainable resource use. In the present study, nickel was recovered from a Raney Nickel catalyst via acid leaching with sulphuric acid and precipitation with sodium hydroxide. The effects of acid concentration on leaching process and the effect of pH on precipitation process were investigated. The leaching process was carried out using sulphuric acid at concentrations of 3, 4, 4.5 and 5.5 M, and the solid residue from the leachate solution was separated by filtration. Later, the pH of the leach solution was adjusted to 5 M NaOH to precipitate nickel. Nickel recovery was investigated as a function of sulphuric acid concentrations in the leaching process and the solution pH in the precipitation process. It was found that 4.5 M H_2SO_4 yielded the highest nickel recovery of 68.13%. Using 4.5 M H_2SO_4 for leaching and pH 14 of NaOH precipitation, the presence of nickel and nickel oxide was confirmed using XRD in the precipitate. The leaching rate was further increased by using 4.5 M H_2SO_4 ($k = 0.1805\ h^{-1}$), with greater acid strength and pH control than 3 M ($k = 0.0466\ h^{-1}$), confirming the strong relationship between these key parameters to optimise nickel recovery. Overall, these findings indicate that acid leaching–alkali precipitation will be an efficient and scalable technique for nickel recovery from spent catalysts, facilitating resource recycling and waste reduction.

[4*]Corresponding author. *E-mail address*: hawaiah162@uitm.edu.my

1. INTRODUCTION

Nickel is widely utilised in many industries, including steel production, battery production, and chemical catalysis (Ijomone et al., 2020). It is widely used in high-load and erosive conditions applications, including gas turbines and engines (Xu et al., 2024), due to its excellent corrosion and mechanical performance as well as good ductility. Its melting and boiling points are approximately 1455 °C and 2913 °C, respectively. Hence, nickel is extensively used in making stainless steel, as well as for creating renewable energy products, like lithium-ion batteries for electric vehicles and electronics (Mudd & Jowitt, 2022). Nickel is also a crucial metal in the process of electroplating, as it provides both aesthetic and protective coatings on the materials in electronics and automotive industries (Gugua et al., 2024). Due to the high demand and limited high-grade ores, it has been increasingly vital to recover nickel from secondary sources such as spent catalyst (Kusuma et al., 2025; Moosakazemi et al., 2024). Spent nickel-based catalysts are classified as hazardous waste and must be incinerated or safely landfilled. In Malaysia, spent nickel-based catalysts are designated as Scheduled Wastes (SW202), thereby imposing a significant disposal burden with disposal costs that could be highly variable. Improper handling can also pose serious environmental risks. Thus, the recycling of nickel from used catalysts is economically highly valuable and plays a significant role in minimising environmental pollution and dependency on primary metal resources

Hydrometallurgical methods effectively dissolve nickel-based material (Aboody et al., 2024; Gomes et al., 2024; Lim et al., 2021; Muscetta et al., 2023). Optimising parameters such as acid concentration, temperature, and time is important for efficient recovery (Al-Mansi & Abdel Monem, 2002). Li et al. (2018) reported 87.7% nickel was recovered from laterite nickel ore with ammonium chloride-hydrochloric acid solution at 90 °C and 90 min of the leaching process, while a lower percentage (75.8%) was obtained when only hydrochloric acid was employed at 75 °C and 70 min (Permana et al., 2020). Elsewhere, nitric acid leaching reached 97.5% at 80 °C in 2 h (Sheik et al., 2013). Sulphuric acid, however, remains the most efficient, giving up to 99% recovery at 85 °C in 4 h with fine particles and proper stirring (Batti & Mandre, 2022). On the other hand, a lower percentage (94.3%) was reported for the recovery of nickel from spent catalyst at 100 °C and 8 h of leaching (Gomes et al., 2024). Sulphuric acid is highly effective at dissolving nickel and forming soluble sulphate, making it the preferred leaching agent. The amount of acid used is an important factor in the leaching process (Idris et al., 2010; Zulkifili et al., 2022), influencing the efficiency of nickel recovery. It influences the availability of protons and anions that dissolve nickel oxide (NiO). Lower concentrations lead to slower reaction due to fewer reactive species, and higher concentrations will improve kinetics and recovery. Higher acid concentration not only promotes the solubility of NiO but also inhibits passivating layer formation on the surface of the catalyst. Nazemi and Rashchi (2012) noted that maintaining the right acid concentrations increases selectivity, while further dropping impurity solubility. Lee et al. (2010) also claimed that adjusting acid strength can significantly reduce the production of undesirable byproducts. Furthermore, Alajoki et al. (2024) recommended moving towards sustainable leaching practices and concluded that water leaching is a particularly environmentally friendly and cost-effective option.

Quantitative analysis of nickel recovery kinetics during acid leaching provides insight into mass transfer considerations and refines the overall process design. Kinetic studies on nickel leaching from laterite nickel ores in acid solutions often use shrinking-core or diffusion models to fit experimental data using empirical constants. As an example, Agacayak and Zedef (2012) reported an apparent rate constant of approximately 2.77×10^{-3} h^{-1} for the sulphuric acid leaching of lateritic nickel ore at 90 °C, with

diffusion through the product layer being identified as the controlling mechanism. Elsewhere, in studies on spent nickel oxide catalysts leached with sulphuric acid, the kinetic analyses (typically from plots of fractional extraction vs time) yield apparent rate constants between 0.6 to 0.06 h^{-1} for nickel dissolution based on linear fits to diffusion-controlled rate equations. Mulak et al. (2005) suggested faster overall kinetics under atmospheric conditions. Additionally, Abdel-Aal and Rashad (2004) obtained a rate constant of 0.822 h^{-1} for sulphuric acid leaching at 85 °C . These kinetic parameters, expressed as rate constants, k, quantify the impact of acid concentration, particle size, and temperature on nickel leaching. These parameters are essential for modelling and scaling hydrometallurgical recovery processes.

The nickel-rich solution obtained from leaching is then subjected to precipitation (generally sodium hydroxide) for the precipitation of $Ni(OH)_2$. Over 99.7% recovery can be achieved with precipitation at 60 °C, pH 8 to 14, with a leaching period of up to 30 minutes (Razavian et al., 2020). However, pH control is crucial to prevent the co-precipitation of impurity (Nazemi & Rashchi, 2012). Additionally, ammonia is often added to recover nickel by forming pure $Ni(OH)_2$ in aqueous solution. This method operates at 25 ± 2 °C and in a narrow pH range of 1 to 5, producing ammonium ions (NH_4^+) as a byproduct. Although suitable for the type of applications requiring high-purity products, it requires strict pH control and safe handling, due to ammonia's volatility and environmental concerns (Nazemi & Rashchi, 2012). On the other hand, sodium carbonate reacts with nickel at a pH range of 6 to 8 to form nickel carbonate ($NiCO_3$), which can be calcined to produce nickel oxide (NiO). At room temperature, it can take 30 minutes to a few hours to produce nickel oxide. However, excessive use of sodium carbonate leads to waste and byproducts (Lee et al., 2010). Apart from it, pH must be controlled in order to reduce other undesired products, including aluminium hydroxide (Razavian et al., 2020). Of all these precipitation agents, sodium hydroxide is the most effective (Sahiruddin et al., 2025) and practical choice for nickel recovery owing to its superior efficiency and broad base pH range, which can be implemented on industrial scales. The precipitation of nickel in metal-containing solutions is highly pH dependent due to its effects on ionic speciation. This affects nickel ion solubility, favouring the precipitation of solid phases including hydroxides, carbonates and ammonia complexes.

Nickel recovery from spent catalysts is vital for sustainable resource management, but the inherent characterisation of these materials, due to their variability and degradation prevents fundamental understanding of leaching and precipitation that elucidates key process parameters. Therefore, this study demonstrates this gap by exploring nickel recovery from virgin Raney nickel, allowing uniform composition to control sulphuric acid leaching and precipitation. By optimising parameters such as acid concentration and precipitation pH, the work provides mechanistic insights into nickel dissolution and recovery, establishing a foundation from which to rationalise efficient recovery strategies applicable to more complex spent catalysts, whilst promoting sustainable metal valorisation.

2. METHODOLOGY

2.1 Material and chemical

Commercial virgin Raney nickel catalyst (Sigma-Aldrich) is an alloy of nickel and aluminium, consisting of approximately 50% nickel and 50% aluminium. Sulphuric acid (H_2SO_4) (95–97%, Merck) was used as the primary leaching agent in this study, while distilled water was utilised for solution preparation, equipment cleaning, and other purposes. Sodium hydroxide (NaOH) (>98%, Sigma-Aldrich) was used to adjust the pH of the leachate during precipitation.

2.2 Leaching and precipitation process setup

The catalyst was used without any modification. The catalyst (2.2 ± 0.01 g) was mixed with 110 mL of sulphuric acid. The solution was heated to 50 °C using a heating mantle, and the reaction was stirred at 350 rpm for 5 h in a sealed glass reactor. Fig. 1(a) shows the leaching process setup adapted from Gomes et al. (2024). After leaching, the hot slurry was filtered at 50 °C using filter paper, and the remaining solid residue was washed with hot distilled water to recover any residual nickel. The filtrate varying in concentration, which was 3, 4, 4.5, and 5.5 M was collected, and nickel concentration was measured at intervals of 1, 2, 3, 4, and 5 h for the kinetic analysis. The nickel concentration at each time point was measured using inductively coupled plasma optical emission spectroscopy (ICP-OES) (Thermo Scientific, iCAP 6000 Series).

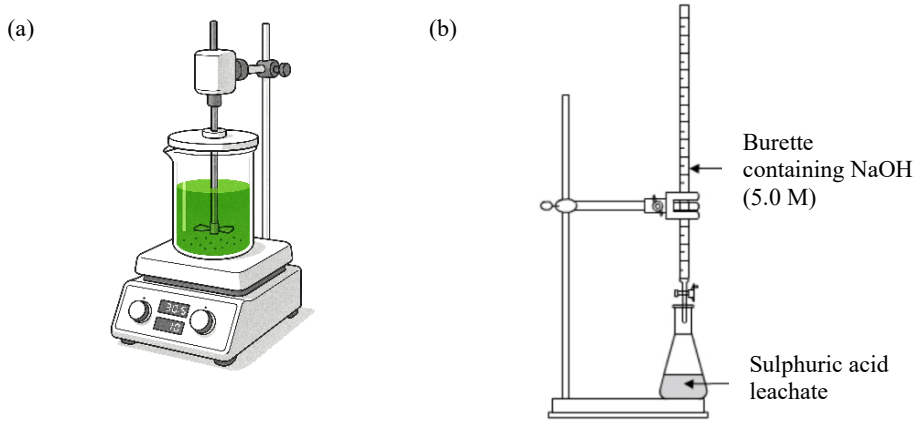

Fig. 1. Process setup of (a) leaching (b) precipitation

Source: Author

Next is the precipitation step, where sodium hydroxide (NaOH) was added to the leachate to recover nickel in the form of nickel hydroxide ($Ni(OH)_2$). Fig. 1(b) illustrates the precipitation process setup. Prior to precipitation, the pH of the leachate was adjusted to 4. The initial pH was set at 4 to ensure that there no premature nickel precipitation occurs before the controlled addition of a precipitating agent (NaOH). Then, the leachate was stirred for 20 minutes, followed by a 5-minute resting period. The volume of NaOH added was recorded at each stage. At pH 4, the solution was divided into four portions, each of which was further titrated to achieve target final pH values of 8, 10, 12, and 14, respectively. At the initial preset pH of 4, 5 M NaOH was used to control the pH more accurately, as the solution was not a strongly acidic solution and the pH was easier to adjust without sudden changes. NaOH was added dropwise using a burette, and the pH was measured periodically and monitored until it reached 14. Samples from the solution were collected for each pH level (8, 10, 12, and 14). It is then filtered through a filter cartridge to eliminate any particles. On the other hand, the solution sample was measured for the remaining nickel concentration, and the efficiency of nickel precipitation at each pH stage was evaluated. After precipitation (when the pH reached 14), the obtained nickel hydroxide was collected by filtration and rinsed with distilled water to remove residual contaminants. After washing, each filtered sample, which is the solid precipitate ($Ni(OH)_2$), was labelled according to its leaching acid concentration and final pH for the precipitation (e.g.

4.5 M pH 8, 3 M pH 14). The precipitates were further dried in an oven at 75 °C for 24 hours. Constant mass was verified to ensure complete removal of moisture. The dried solids were then ground, sieved, and stored in labelled polyethylene bags. Next, the sample was analysed using x-ray diffraction (XRD) to identify the nickel-containing phases. The efficiency of nickel recovery was calculated by comparing the amount of nickel recovered from the leachate to the initial nickel content in the catalyst, according to Eq. (1).

$$\text{Recovery efficiency (\%)} = \frac{\text{Nickel leach (ppm)}}{\text{Initial nickel (ppm)}} \times 100 \quad (1)$$

3. RESULT AND DISCUSSION

3.1 Leaching efficiency

During leaching process, a green colouration of the leachate was consistently observed, indicating the presence of Ni^{2+} ions in the aqueous phase. This is in accordance with previous literature, where green hues are typically attributed to hydrated nickel complexes (Greenwood, 2012; Petrucci et al., 2017). Table 1 lists the leaching efficiency of nickel, which was found to be highest with 4.5M sulphuric acid, reaching approximately 68% after 5 h. Increasing acid molarity (from 3 to 4.5 M) lowers the pH of the leaching solution and raises the concentration of hydrogen ions (H^+) available which promotes dissolution of nickel-bearing phases into solution. More H^+ accelerates the chemical dissolution of nickel compounds into Ni^{2+} in solution by shifting the reaction equilibrium in favour of dissolution. A higher proton (H^+) concentration also helps prevent the formation of insoluble intermediate species (e.g., nickel hydroxide precipitates) that can form at higher pH, so a lower pH (higher acidity) keeps Ni in solution more effectively. As a result, leaching efficiency typically increases with acid molarity up to an optimised range because of enhanced kinetics and thermodynamic driving force for nickel release into solution. This explains the observed increase in Ni extraction when the acid concentration rises from around 3 to 4.5 M. A similar trend has been observed in multiple hydrometallurgical studies where nickel extraction increases with rising sulphuric acid concentration due to improved reaction kinetics and proton availability, facilitating nickel solubilization. As an example, Abdel-Aal and Rashad (2004) stated that nickel extraction increased considerably (20–86%), which was associated with the increase of sulphuric acid concentration (low to high percentage acid). This is in line with the observation that higher acid molarity increases the leaching efficiency via proton activity, further driving dissolution equilibrium toward soluble nickel species. Elsewhere, Ali et al. (2023) reported that increasing the sulphuric acid concentration from 1.5 to 3.0 M significantly increased the recovery of Ni^{2+} by 28%, indicating better nickel dissolution at higher molarity. This indicates that the higher the molarity, the more readily nickel dissolves.

On the other hand, increasing the acid concentration to 5.5 M resulted in a 62% decrease in efficiency. This reduced performance could be due to the occurrence of side reactions between excess acid and other components of the catalyst (particularly aluminium), which could hinder nickel dissolution (Sheik et al., 2013). Conversely, the lowest concentration (3 M) tested achieved only around 40% leaching efficiency. Based on the observed leaching efficiencies, sulphuric acid concentrations of 3 M and 4.5 M were selected for further kinetic studies. These two conditions represent the highest and lowest leaching efficiencies observed, making them suitable for investigating the effect of acid strength on nickel dissolution rates.

Table 1: Leaching efficiency using various acid molarity

Acid molarity (M)	Leaching efficiency (%)
3	48.27
4	65.85
4.5	68.13
5.5	62.16

Source: Author

3.2 Leaching kinetics

Kinetic studies were conducted using 3 M and 4.5 M sulphuric acid (H_2SO_4) to evaluate the dissolution behaviour of nickel from the spent catalyst. A first-order kinetic model was employed because the experimental data showed a better linear relationship than other kinetic models. This also implies that the leaching rate is proportional to the concentration of unreacted nickel in the solid phase, which agrees with Targanov and Troshkina (2021) findings on nickel leaching in sulphuric acid systems. Eq. (2) is the first-order kinetics model that was used in this study.

$$\ln\left(\frac{C_o}{C_t}\right) = \mathrm{k}t \tag{2}$$

where, C_o is the initial concentration Ni^{2+}, C_t is the Ni^{2+} concentration at time, t. The kinetic rate constant k was determined from the slope of the plot of $\ln\left(\frac{C_o}{C_t}\right)$ vs t.

The nickel leaching rate at 3 M H_2SO_4 was relatively low as shown in Fig. 2. There was a gradual increase in Ni^{2+} concentration over time, though after the last four hours, the concentration fluctuated slightly. The rate constant was determined to be 0.0466 h^{-1}, indicating a lower leaching efficiency and slower kinetics under this acid concentration (3 M). In comparison, the leaching behaviour improved significantly at 4.5 M H_2SO_4, as shown in Fig. 3. The nickel concentration increased rapidly, especially during the first three hours, after which it approached equilibrium. The corresponding rate constant was 0.1805 h^{-1}, which is nearly four times higher than the value obtained at 3M. This confirms that leaching kinetics are enhanced with increased acid molarity, primarily due to the greater availability of hydrogen ions, which promote faster dissolution of the catalyst matrix. Elsewhere, Abdel-Aal and Rashad (2004) reported the use of 50%w/w (7.1 M) H_2SO_4 at 85 °C resulted in a nickel efficiency ~86%, with kinetic rate constant of 0.822 h^{-1} after 150 minutes of leaching. Their study employed a chemical-controlled surface reaction model. Hence, a higher efficiency and rate constant were observed, showed that both acid concentration and temperature have a significant role in the leaching process.

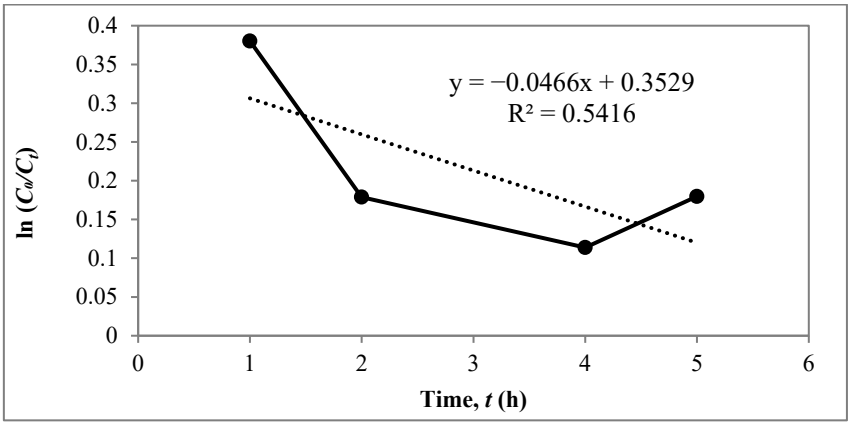

Fig. 2: First-order kinetics for the leaching process using 3 M H_2SO_4

Source: Author

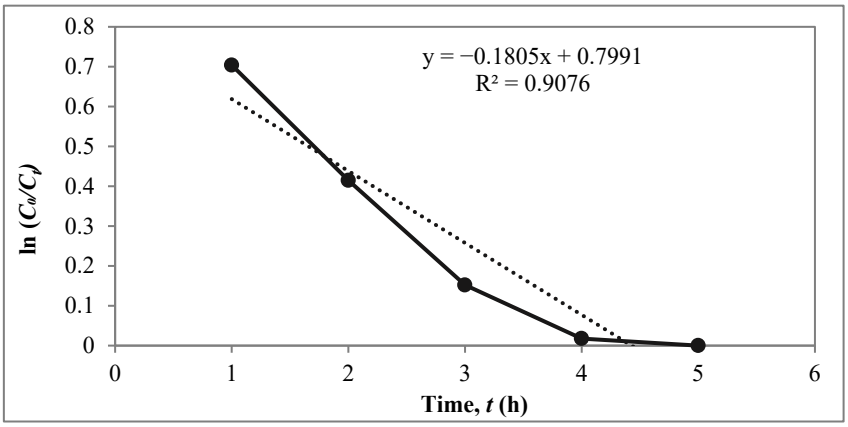

Fig. 3: First-order kinetics for the leaching process using 4.5 M H_2SO_4

Source: Author

3.3 pH adjustment and precipitation of nickel hydroxide

After the leaching process, the raw leachate was found to have extremely low pH values, ranging from 0.1 to 0.3. Attempts to precipitate directly from this solution resulted in uncontrolled precipitation, and the formation of a viscous green slurry was observed at pH 4–5. This could have happened because the nickel and aluminium in the solution started forming solid particles too quickly, causing them to mix and settle out as an uneven jelly-like solid (Chernyaev et al., 2023). To reduce this occurrence, the leachate was diluted with distilled water in a 1:1 ratio prior to neutralisation. Generally, the dilution was performed for two reasons: the first is to decrease the concentration of free Ni^{2+}ions and the second is to have better control over the precipitation environment (Tsai et al., 2020).

The pH was neutralised in a step-wise manner by adding NaOH solutions of varied molarities (0.5 M to 2 M, then finalised with 5 M). For more resistant systems or when it was necessary to adjust the pH quickly, 10 M and/or 20 M were also applied. But this should be applied carefully, so as not to cause local overheating. The exothermic neutralisation of the H_2SO_4 and NaOH resulted in distinct temperature spikes and transient crystal formation. Hence, a necessary adjustment to the stirring speed and a temporary termination of NaOH addition is required until the system re-stabilises.

Fine solids were observed at the bottom of each vessel, as precipitation became apparent. Part of the supernatant was taken for metal concentration analysis using ICP. Next, the precipitate was filtered and washed several times with distilled water to remove residual sulphate and sodium salts. The wash water pH stabilised at neutral (~7), which indicated that the impurities were removed sufficiently.

3.4 Precipitation of nickel using NaOH

For x-ray diffraction (XRD) studies, four samples were selected to compare the effect of precipitation pH at different leaching acidities: two from 3 M H_2SO_4 (pH 8 and pH 14) and two from 4.5 M H_2SO_4 (pH 8 and pH 14). They were labelled as 3 M pH 8, 3 M pH 14, 4.5 M pH 8 and 4.5 M pH 14. These samples were chosen to represent precipitation regimes at both low and high pH and to evaluate what crystalline phases formed under different leaching intensities. During the leaching-to-precipitation transition, a stable visual change of solution colour was observed. The leachates were green during the leaching stage, consistent with Ni^{2+} in aqueous solution, whilst the visually dewatered solutions displayed aquatic blue or bluish shades. This transformation is related to nickel coordination geometry and hydration state changes in precipitation, depending on whether nickel hydroxide or basic nickel salts are formed. This colour change is thus associated with changes in nickel coordination geometry and hydration state during precipitation, particularly during the formation of nickel hydroxide or basic nickel salts (Greenwood, 2012). The colour change thus supports successful precipitation and phase transformation of dissolved nickel species.

3.5 Effect of solution pH during the precipitation process

Nickel was recovered from the leachate by adjusting the pH using sodium hydroxide (NaOH). The leachates obtained from 3 M and 4.5 M of sulphuric acid at pH 8, 10, 12, and 14 were compared in terms of precipitation efficiency as well as the volume of NaOH consumed, as shown in Fig. 4. Nickel precipitation was complete in all instances, as evidenced by 100% recovery efficiency and a trace level of nickel found in the leachate. Thus, this confirms that the precipitation worked under these conditions. However, the 4.5 M leachate required more NaOH volume. At pH 8, it requires 204.2 mL as compared to 145.4 mL for 3 M, and at pH 14, it uses 251.8 mL compared to 182.6 mL. Although the 4.5 M H_2SO_4 system required a greater volume of NaOH for neutralisation than the 3 M counterpart, its substantially higher rate constant (0.1805 h^{-1} vs 0.0466 h^{-1}) reflects markedly enhanced leaching kinetics. The results were in agreement with those of Xiao et al. (2020), indicating that increasing sulphuric acid concentration markedly enhances nickel leaching rates and rate constants. Collectively, these results confirm that effective kinetic performance and higher practical efficiency for nickel recovery can be achieved using 4.5 M H_2SO_4 concentration.

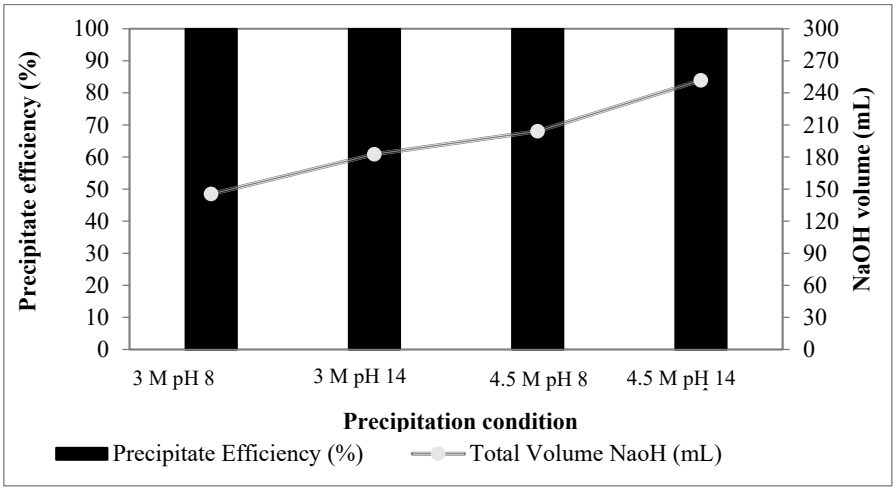

Fig. 4: Effect of NaOH volume and pH on nickel precipitation efficiency at different sulphuric acid concentrations

Source: Author

3.6 Precipitation of nickel: XRD analysis

X-ray diffraction (XRD) was used to identify the solid phases formed in each sample. The intensity versus 2θ graphs for 3 M pH 8, 3 M pH 14, 4.5 M pH 8, and 4.5 M pH 14 are shown in Fig. 5. In the Fig. 5(a), 3 M pH 8 sample, several strong peaks appeared, but none matched the standard peaks for nickel metal or nickel oxide. The 3 M pH 14 sample, illustrated by Fig. 5(b) showed peaks for possibly zinc oxide (ZnO) and titanium dioxide (TiO_2), but no nickel peaks were detected. The 4.5 M pH 8 sample, as shown in Fig. 5(c), exhibited weak and broad peaks. A small peak near 63.2° was close to nickel oxide, but it was not enough to confirm its presence. Only Fig. 5(d), which represents a 4.5 M pH 14 sample, showed clear peaks for nickel metal, confirming its formation.

Among all the samples, only the 4.5 M pH 14 sample, as shown by Fig. 5(d), exhibited clear peaks at 44.5° and 51.8°, corresponding to the Ni (111) and Ni (200) planes, confirming the presence of metallic nickel. As shown in Table 2, this sample is the most effective condition for recovering nickel in metallic form. By contrast, the other samples either lacked a nickel peak or showed only weak signals associated with nickel oxide, suggesting incomplete or less efficient recovery of nickel metal. Although precipitation produced nearly total nickel recovery from solution, the XRD patterns did not show distinct peaks corresponding to metallic nickel (111, 200), NiO or any other crystalline nickel phases. This absence is due to the amorphous or poorly crystalline nature of nickel hydroxide produced upon oxidation, which does not exhibit long-range structural order that can be detected by the XRD. In comparison, the other samples show no nickel peak or only weak nickel oxide signals, indicating partial recovery and incomplete recovery of metallic nickel. Even if precipitation resulted in nearly complete solution nickel recovery, the XRD patterns did not exhibit peaks characteristic of metallic nickel (111, 200), NiO or a great variety of crystalline nickel phases. This absence is due to the amorphous or poorly crystalline nature of nickel hydroxide produced upon oxidation, which does not exhibit long-range structural order that can be detected by the XRD. This is consistent with previous studies on nickel hydroxide precipitated under specific conditions by Ramesh and Kamath (2006), which also found significant structural disorder or amorphous, resulting in weak or no diffraction peaks in XRD despite quantitative recovery of nickel.

After acid leaching using 4.5 M H_2SO_4, metallic nickel was present in pH 14 precipitation but absent in pH 8 precipitation. pH is critical for controlling the stability of nickel in the aqueous phase. Thermodynamically stable regions of nickel metal, dissolved Ni^{2+} species, and solid nickel hydroxides or oxides are represented as a function of pH and redox potential as depicted in Pourbaix (potential–pH) diagrams for the Ni–H_2O system. The diagrams indicate, especially at neutral to moderately alkaline pH (~9–12), nickel tends to form passivating hydroxide/oxide phases. Under strongly reducing conditions (i.e. at high pH such as pH 14), this is expected to enlarge the range of nickel metal domain relative to oxidised species.

Table 2: XRD Analysis of selected samples

Sample	Ni (111)@ 44.5°	Ni (200)@ 51.8°	NiO Peaks	Nickel Phase
3 M pH 8	No	No	No	None
3 M pH 14	No	No	No	None
4.5 M pH 8	No	No	Weak	NiO
4.5 M pH 14	Yes	Yes	No	Ni

Source: Author

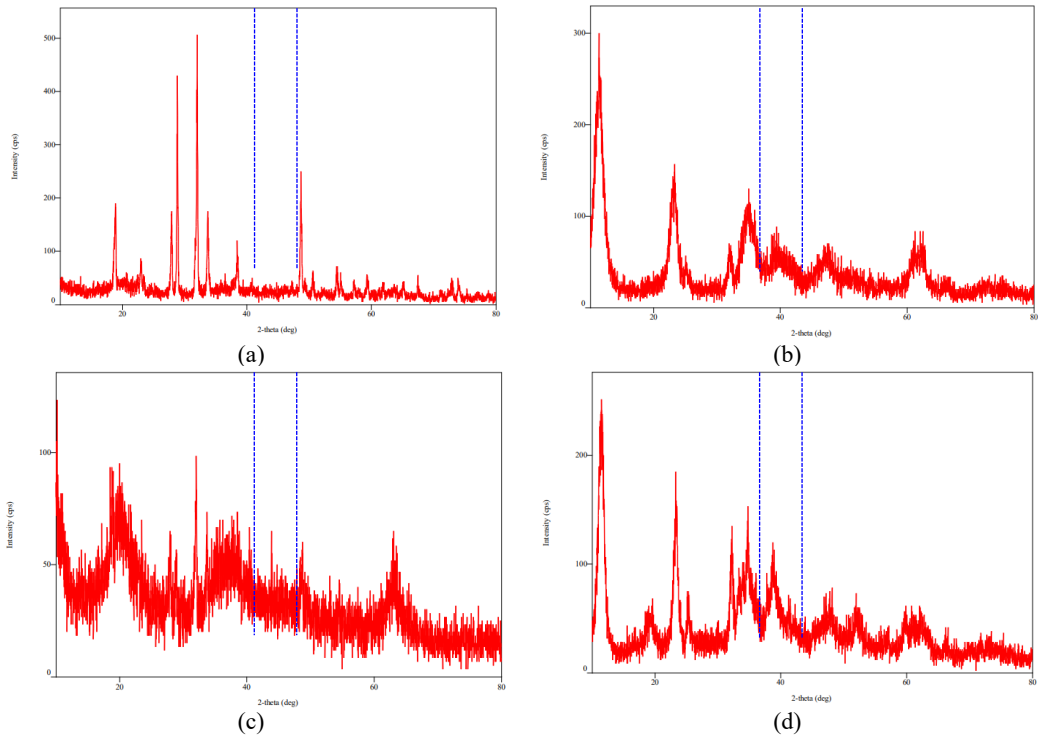

Fig. 5: XRD pattern of (a) 3 M pH 8 (b) 3 M pH 14 (c) 4.5 M pH 8 (d) 4.5 M pH 14, identification of Ni peaks (blue lines) at 44.5° and 51.8°

Source: Author

4. CONCLUSION

Nickel was recovered from the Raley nickel catalyst by acid leaching (H_2SO_4) and alkali precipitation (NaOH). The highest nickel recovery of 68.13% was achieved with leaching by using 4.5 M H_2SO_4 at 50 °C out of the four different acid concentrations (3, 4, 4.5, and 5.5 M) studied. Meanwhile, 5 M NaOH was used to adjust the pH of the leachate for precipitation. Nickel was completely precipitated as Ni(OH) at pH 10 and above. This confirms nickel precipitation is favoured under alkaline conditions without requiring extreme pH values. The leaching kinetics were evaluated by fitting the experimental data to a first-order model. The dissolution of nickel in 4.5 M H_2SO_4 exhibited a rate constant of 0.1805 h^{-1}, confirming a faster leaching rate and higher efficiency relative to 3 M H_2SO_4. In conclusion, the optimal leaching conditions at 4.5 M and precipitation at pH 14 proved to be efficient for nickel recovery from Raney nickel catalyst. A systematic investigation of sulphuric acid concentrations within the range of 4 to 5 M is recommended to optimise recovery efficiency and refine chemical utilisation. Finally, it is recommended to perform the process at pilot scales under continuous-flow conditions to evaluate its potential for industrial implementation.

ACKNOWLEDGEMENT

The authors would like to acknowledge the Faculty of Chemical Engineering, Universiti Teknologi MARA Shah Alam, for providing the facilities that made this research possible.

FUNDING

The project receives no funding.

CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts.

AUTHORS' CONTRIBUTIONS

Muhammad Hairul Haizan Azizan: Conceptualisation, methodology, formal analysis, investigation and writing-original draft; **Norhaslinda Nasuha**: Formal analysis and validation; **Muhammad Syafiq Hazwan Ruslan**: Writing-review and editing, and validation; **Hawaiah Imam Maarof**: Conceptualisation, methodology, formal analysis, supervision, writing-review and editing, and validation.

DECLARATION OF GENERATIVE AI IN THE WRITING PROCESS

During the preparation of this work, the author used generative artificial intelligence (AI) tools, specifically ChatGPT developed by OpenAI, to assist in improving language clarity, grammar, sentence structure, and overall readability of the manuscript. The AI tool was used solely for language refinement and drafting support, while all technical content, data analysis, interpretation of results, and final conclusions remain the sole responsibility of the author.

The author carefully reviewed and edited all AI-generated suggestions to ensure accuracy, originality, and compliance with academic integrity standards. No AI tool was used to generate original research findings, manipulate data, or replace critical scientific judgment. The author takes full responsibility for the content of this manuscript.

DATA AVAILABILITY/SUPPLEMENTARY MATERIALS

All data generated or analysed during this study are included in this published article.

ETHICAL COMPLIANCE STATEMENT

The authors confirm that this study was conducted in accordance with established ethical standards and institutional guidelines. All procedures complied with the Safety, Health, and Environmental (HSE) protocols of Universiti Teknologi MARA. The research was conducted with integrity, and all data were reported accurately without fabrication, falsification, or inappropriate manipulation

ETHICS APPROVAL STATEMENT

The authors declare that this study did not involve human or animal subjects and complies with institutional ethical guidelines.

REFERENCES

Abdel-Aal, E. A., & Rashad, M. M. (2004). Kinetic study on the leaching of spent nickel oxide catalyst with sulfuric acid. *Hydrometallurgy*, *74*(3–4), 189–194. https://doi.org/10.1016/j.hydromet.2004.03.005

Aboody, H., Najafabadi, A. T., Mohammadi, S., & Hashemnezhad, S. E. (2024). Valorization of spent catalyst of sponge iron production industries into high-purity metallic nickel via a novel three-step method of leaching, cementation and purification. *Process Safety and Environmental Protection*, *188*, 1145–1159. https://doi.org/10.1016/j.psep.2024.06.021

Agacayak, T., & Zedef, V. (2012). Dissolution kinetics of a lateritic nickel ore in sulphuric acid medium. *Acta Montanistica Slovaca*, *17*, 33–41.

Al-Mansi, N. M., & Abdel Monem, N. M. (2002). Recovery of nickel oxide from spent catalyst. *Waste Management*, *22*(1), 85–90. https://doi.org/10.1016/S0956-053X(01)00024-1

Alajoki, J., Karppinen, A., Rinne, T., Serna-Guerrero, R., & Lundström, M. (2024). Leaching strategies for the recovery of Co, Ni, Cu and Zn from historical tailings. *Minerals Engineering*, *217*, 108967. https://doi.org/10.1016/j.mineng.2024.108967

Ali, I., Gaydukova, A., Kon'kova, T., ZA, A. L., & Sillanpaa, M. (2023). Kinetics and optimization of metal leaching from heat-resistant nickel alloy solid wastes. *Molecules*, *28*(14). https://doi.org/10.3390/molecules28145545

Batti, N. R., & Mandre, N. R. (2022). Nickel and aluminium recovery from spent reforming catalyst through selective leaching, crystallization and precipitation. *Transactions of Nonferrous Metals Society of China (English Edition)*, *32*(1), 345–353. https://doi.org/10.1016/S1003-6326(22)65799-6

Chernyaev, A., Zhang, J., Seisko, S., Louhi-Kultanen, M., & Lundström, M. (2023). Fe^{3+} and Al^{3+} removal by phosphate and hydroxide precipitation from synthetic NMC Li-ion battery leach solution. *Scientific Reports*, *13*(21445), 1–12. https://doi.org/10.1038/s41598-023-48247-6

Gomes, N., Garjulli, F., Botelho, A. B., Jr., Espinosa, D. C. R., & Baltazar, M. D. G. (2024). Recycling of spent catalysts from the petrochemical industry by hydrometallurgy to obtain high-purity nickel products for electroplating. *The Journal of The Minerals, Metals & Materials Society*, *76*(3), 1372–1382. https://doi.org/10.1007/s11837-023-06290-8

Greenwood, N. N., & Earnshaw, A. (2012). *Chemistry of the elements*. Butterworth-Heinemann. https://books.google.com.my/books?id=EvTI-ouH3SsC

Gugua, E. C., Ujah, C. O., Asadu, C. O., Von Kallon, D. V., & Ekwueme, B. N. (2024). Electroplating in the modern era, improvements and challenges: A review. *Hybrid Advances*, *7*, 100286. https://doi.org/10.1016/j.hybadv.2024.100286

Idris, J., Musa, M., Yin, C. Y., & Hamid, K. H. K. (2010). Recovery of nickel from spent catalyst from palm oil hydrogenation process using acidic solutions. *Journal of Industrial and Engineering Chemistry*, *16*(2), 251–255. 10.1016/j.jiec.2010.01.044

Ijomone, O. M., Olayemi K., Iroegbu, J. D., Ifenatuoha, C. W., Olung, N. F., & Aschner, M. (2020). Epigenetic influence of environmentally neurotoxic metals. *NeuroToxicology*, *81*, 51–65. https://doi.org/10.1016/j.neuro.2020.08.005

Kusuma, H. S., Putri, N. A., Farahita, A. M., Manullang, H. B., Christa Jaya, D. E., Amenaghawon, A. N., Darmokoesoemo, H., & Mahfud, M. (2025). Sustainable nickel recovery: Advancing bioleaching techniques for secondary sources in renewable energy and circular economy applications. *Sustainable Chemistry and Pharmacy*, *46*, 102094. https://doi.org/10.1016/j.scp.2025.102094

Lee, J. Y., Rao, S. V., Kumar, B. N., Kang, D. J., & Reddy, B. R. (2010). Nickel recovery from spent Raneynickel catalyst through dilute sulfuric acid leaching and soda ash precipitation. *Journal of Hazardous Materials*, *176*(1), 1122–1125. https://doi.org/10.1016/j.jhazmat.2009.11.137

Li, J. H., Li, D. S., Xu, Z. F., Liao, C. F., Liu, Y., & Zhong, B. (2018). Selective leaching of valuable metals from laterite nickel ore with ammonium chloride-hydrochloric acid solution. *Journal of Cleaner Production*, *179*, 24–30. https://doi.org/10.1016/j.jclepro.2018.01.085

Lim, M. S. W., Yang, T. C. K., Yap, Y. H., Pan, G. T., Chong, S. E. H., & Tiong, T. J. (2021). Intensification and optimisation of nickel recovery from spent hydrogenation catalysts via ultrasound-augmented hydrometallurgy. *Journal of Environmental Chemical Engineering*, *9*(4), Article 105771. https://doi.org/10.1016/j.jece.2021.105771

Moosakazemi, F., Entezari-Zarandi, A., Ghassa, S., & Larachi, F. (2024). Recovery of magnetic Ni particles from spent catalyst leachate by direct cementation. *Sustainability*, *16*(19), 8630. https://www.mdpi.com/2071-1050/16/19/8630

Mudd, G., & Jowitt, S. (2022). The new century for nickel resources, reserves, and mining: Reassessing the sustainability of the devil's metal. *Economic Geology*, *117*. https://doi.org/10.5382/econgeo.4950

Mulak, W., Miazga, B., & Szymczycha, A. (2005). Kinetics of nickel leaching from spent catalyst in sulphuric acid solution. *International Journal of Mineral Processing*, *77*(4), 231–235. https://doi.org/10.1016/j.minpro.2005.06.005

Muscetta, M., Andreozzi, R., Clarizia, L., Marotta, R., Palmisano, G., Policastro, G., Race, M., Yusuf, A., & Di Somma, I. (2023). Recovery of nickel from spent multilayer ceramic capacitors: A novel and sustainable route based on sequential hydrometallurgical and photocatalytic stages. *Separation and Purification Technology*, *326*, Article 124780. https://doi.org/10.1016/j.seppur.2023.124780

Nazemi, M., & Rashchi, F. (2012). Recovery of nickel from spent NiO/Al_2O_3 catalyst through sulfuric acid leaching, precipitation and solvent extraction. *Waste Management & Research*, *30*(5), 492–497. https://doi.org/10.1177/0734242x11417984

Permana, D., Kumlasari, R., Wahab, & Musnajam. (2020). The leaching of low-grade nickel laterite ore using atmospheric acid leaching method in hydrochloric acid. *Riset Geologi Dan Pertambangan*, *30*(2), 203–214. https://jrisetgeotam.brin.go.id/index.php/jrisgeotam/article/view/1097

Petrucci, R. H., Herring, F. G., Madura, J. D., & Bissonnette, C. (2017). *General chemistry: Principles and modern applications*. Pearson Education.

Ramesh, T. N., & Kamath, P. V. (2006). Synthesis of nickel hydroxide: Effect of precipitation conditions on phase selectivity and structural disorder. *Journal of Power Sources*, *156*(2), 655–661. https://doi.org/10.1016/j.jpowsour.2005.05.050

Razavian, M., Fatemi, S., & Najafabadi, A. T. (2020). Extraction of highly pure nickel hydroxide from spent NiO/Al_2O_3 catalyst: Statistical study on leaching by sulfuric acid lixiviant and selective precipitation. *Journal of Environmental Chemical Engineering*, *8*(2), 103660. https://doi.org/10.1016/j.jece.2020.103660

Sahiruddin, S., Liu, W., Li, B. Q., Zhang, K., Zhang, Z. L., & Liu, F. Y. (2025). Thermodynamics analysis and experimental optimization of mixed hydroxide precipitation from nickel laterite ores. *Mining Metallurgy & Exploration*. https://doi.org/10.1007/s42461-025-01276-3

Sheik, A. R., Ghosh, M. K., Sanjay, K., Subbaiah, T., & Mishra, B. K. (2013). Dissolution kinetics of nickel from spent catalyst in nitric acid medium. *Journal of the Taiwan Institute of Chemical Engineers*, *44*(1), 34–39. https://doi.org/10.1016/j.jtice.2012.08.003

Targanov, I. E., & Troshkina, I. D. (2021). Kinetics of sulfuric-acid nickel Leaching from the grinding wastes of rhenium-containing superalloys. *Russian Journal of Non-Ferrous Metals*, *62*(5), 508–513. https://doi.org/10.3103/S1067821221050163

Tsai, T. H., Chou, H. W., & Wu, Y. F. (2020). Removal of nickel from chemical plating waste solution through precipitation and production of microsized nickel hydroxide particles. *Separation and Purification Technology*, *251*, 117315. https://doi.org/10.1016/j.seppur.2020.117315

Xiao, W. H., Liu, X. H., & Zhao, Z. W. (2020). Kinetics of nickel leaching from low-nickel matte in sulfuric acid solution under atmospheric pressure. *Hydrometallurgy*, *194*, Article 105353. https://doi.org/10.1016/j.hydromet.2020.105353

Xu, H. Y., Huang, T. W., Ai, C., Miao, L. K., Zhang, J., & Liu, L. (2024). Research progress on thin-wall effect of nickel-based single crystal superalloy turbine blades. *Rare Metal Materials and Engineering*, *53*(5), 1486–1492. https://doi.org/10.12442/j.issn.1002-185X.20230568

Zulkifili, F. S., Maarof, H. I., Nasuha, N., & Puasa, S. W. (2022). Leaching of electric arc furnace slag for selective recovery of iron: Effect of temperature, H_2SO_4/HCl acid, and oxidant concentration. *Pertanika Journal of Science and Technology*, *30*(3), 2023-2032. https://doi.org/10.47836/pjst.30.3.14