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RESEARCH ARTICLE

Nickel recovery from nickel-containing spent catalyst using atmospheric leaching and oxalate precipitation

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OBJECTIVES A study has been done to investigate and optimize the recovery of nickel from nickel-containing spent catalyst through sulfuric acid (1 - 2 mol/L) atmospheric leaching in different operation conditions. METHODS From the leaching experiments, it was possible to extract 78% nickel under the conditions of 1 mol/L H_2SO_4, reaction time of 5 h, 100 g/L pulp density, and temperature of 80°C. RESULTS In the next step Ni was selectively precipitated from a sulfuric acid-leached solution using 1 mol/L oxalic acid. The nickel content in the product was 97.29% Ni. Based on the study, sulfuric acid was found to be a suitable leaching agent to extract Ni from the nickel-containing spent catalyst. CONCLUSIONS The study also indicated the effective extraction and recovery of nickel which was well supported by characterization studies using XRD technique.

KEYWORDS nickel; spent catalyst; atmospheric leaching; precipitation; oxalic acid

1. INTRODUCTION

Catalysts are substances that increase the reaction rate of a chemical reaction without being consumed in the process. The application of catalysts in large-scale chemical and petrochemical processes has started at the beginning of the twentieth century in area ranging from pharmaceuticals and specialty chemicals to polymers and petroleum processing (Staszak 2018). Commercial catalyst has limited durability, usually from several months to 10 years, depending on the process and type of catalyst (Staszak 2018). Catalysts gradually lose their catalytic activity, usually through structural changes, poisoning, overheating or the deposition of extraneous material such as coke. When the activity of the catalyst declines below the acceptable level, it is usually regenerated and reused, but regeneration is not always possible (Akcil et al. 2015). After a few cycles of regeneration and reuse, the catalyst activity may decrease to very low levels and further regeneration may not be economically feasible (Marafi and Stanislaus 2008).

A catalyst is "spent" when it no longer exhibits the necessary activity or specificity required by the user. The spent catalysts are discarded as solid wastes (Trimm 2001; Eijsbouts 1999; Furimsky 1996). The accumulation of spent catalysts causes severe pollution problems, and it is estimated that more than 150000-170000 t/year of spent catalyst discharged as waste worldwide (Batti and Mandre 2020). Recycling of spent catalysts has become an unavoidable task not only to lower their costs but also to reduce the catalysts waste to prevent the environmental pollution. Besides, these metals command a significant price in the market.

In many cases, spent catalysts are considered to be hazardous wastes due to their metals contain such as Ni, Co, Mo, Cr, Cu, Zn, Fe, Ti, V, W, Pd, Ru, Rh (Staszak 2018). The spent catalyst may also be considered as a secondary ore, due to their high metal concentrations. Therefore metals recovery from spent catalyst is important from the viewpoint of environmental protection and resource recycling.

Nickel is a strategic metal with new industrial and metallurgical applications (Sadat et al. 2016). Today, nickel is widely used in various industries such as alloying in steel making, chemical and spatial applications, magnets, and recharge-

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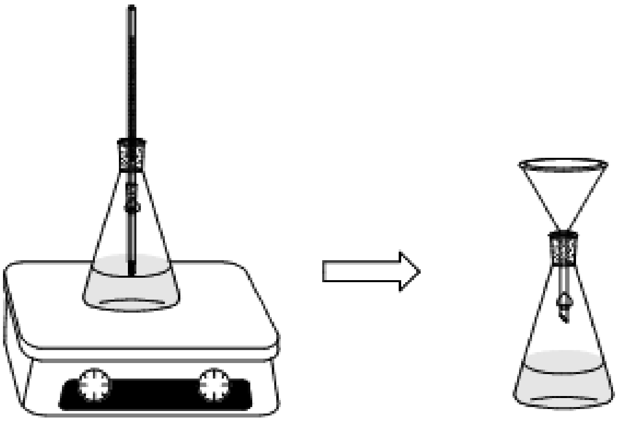


FIGURE 1. Apparatus sketch for the leaching process.

able batteries. Increasing demand for nickel requires further intensive studies of its extraction methods from secondary resources. Extraction of nickel can be effectively performed from spent catalysts.

To deal with the spent catalyst problem several methods are proposed, generally based on pyrometallurgy (using a heat treatment) and hydrometallurgy (with chemical and microbial leaching) methods. In both cases the metals are recovered as mixed solutions, and then separated by conventional separation techniques (solvent extraction, selective precipitation, and ion-exchange).

Although different pyro-based processes have been used earlier, recent developments involve hydrometallurgical processes for their recovery. Hydrometallurgy processes have been widely applied to metal recovery from industrial wastes, due to their flexible and environmental friendly (low energy requirements, low gas emissions and waste generations) and also complete recovery of metals (Oza et al. 2011; Szymczycha-Madeja 2011; Kinoshita et al. 2003). Compared to direct disposal of spent catalysts, hydrometallurgical treatment significantly reduces the risk of hazardous metal contamination in the environment. The acidic leachates generated during metal extraction can be managed through neutralization, precipitation, or adsorption processes, minimizing the potential for soil and water pollution (Reynier et al. 2015). Additionally, the selective precipitation techniques used in metal recovery help reduce the volume of hazardous residues, making disposal safer and more manageable.

In this regard many reagents, such as HCl, H_2SO_4, NH_3 and HNO_3 have been employed for the extraction of nickel. Vicol et al. (1986) studied leaching of spent catalyst with an aqueous solution of 15 –23% ammonia at 60– 90°C and at pH 7.5 –9. Chaudhary et al. (1993) reported the extraction of nickel (~73%) at 80°C from a spent catalyst (17.7% Ni) employing HCl leaching. Abdel-Aal and Rashad (2004) studied nickel leaching kinetics on spent nickel catalysts (Al_2O_3-NiO) with 50% H_2SO_4 at 358 K extracted 94% NiO. Sahu et al. (2005) achieved a high Ni leaching (~98%) with H_2SO_4 from a spent fertilizer catalyst ($NiO–SiO_2–Fe_2O_3–Al_2O_3$). A combination acid ($HNO_3/H_2SO_4/HCl$) was used by Lai et al. (2008), indicated a recovery of 99% V and Ni, and ~90% Mo. Batti and Mandre (2022) recovered 98.5% NiO under the condition of 5.5 mol/L H_2SO_4 at 358 K. In addition, organic acids are also a choice worth considering because they have the advantages of milder treatment conditions, avoiding secondary pollution and corrosiveness. Mouna et al. (2021) found that among different organic acids (citric acid, gluconic acid, oxalic acid), oxalic acid has the highest efficiency for leaching metals (Ni and V) from a spent hydrotreating catalyst.

From those references, it may be seen that there is a need to develop further intensive studies of nickel leaching and separation. Therefore, in the present study, the hydrometallurgical methods for Ni recovery from spent catalyst of petrochemical industry in Indonesia have been addressed in detail. Chemical leaching tests were performed using various solutions of sulphuric acid, hydrochloric acid, nitric acid, as well as citric acid in some leaching parameters followed by precipitation by using oxalic acid to get the best leaching reagent and selective precipitation of nickel from nickel-containing spent catalyst. The results obtained from the investigation were supported by the characterization studies.

2. MATERIALS AND METHOD

2.1 Materials

The spent catalyst in this study was originated from petrochemical industry in Indonesia. The chemical compositions of the catalyst were obtained using X-ray fluorescence (XRF; Epsilon 3XLE, PANalaytical, Netherland) and Induced Couple Plasma Optical Emision Spectroscopy (ICP-OES; Plasma Quant PQ 9000 Elite, analytikjena). The mineral phases of the raw samples were identified by X-ray Diffraction (XRD; X'Pert 3 Powder, PANalytical, Netherland with X'celerator detector and Highscore Plus software) using Cu $K\alpha$ radiation, in the 2θ range from 5°to 80°. The accelerating voltage and applied current were 40 kV and 40 mA, respectively, with a scanning speed of 2°/min and a scanning step of 0.02°.

2.2 Experimental method

To evaluate the most selective leaching reagent over to nickel, atmospheric leaching was carried out using various leaching reagents (sulfuric acid, hydrochloric acid, nitric acid, and citric acid) in the same leaching conditions. Leaching filtrate was further processed using oxalate precipitation to produce nickel oxalate. After leaching processes and filtration, nickel ion from leaching filtrate was precipitated using 1M of oxalic acid, 1:1 of volume ratio, at temperature of 80°C for 1 h to produce nickel oxalate precipitate. After filtration, nickel oxalate precipitate was dried at at temperature of 80°C for 12 h. Leaching reagent that produce high purity nickel will be used in further leaching study.

To evaluate the optimum leaching condition in various leaching parameters, spent catalysts were leached using var-

TABLE 1. Chemical composition of nickel-containing spent catalyst by XRF.

Elements	Al2O3	NiO	CaO	K2O	Fe2O3
wt. %	70.36	15.17	13.99	0.13	0.20

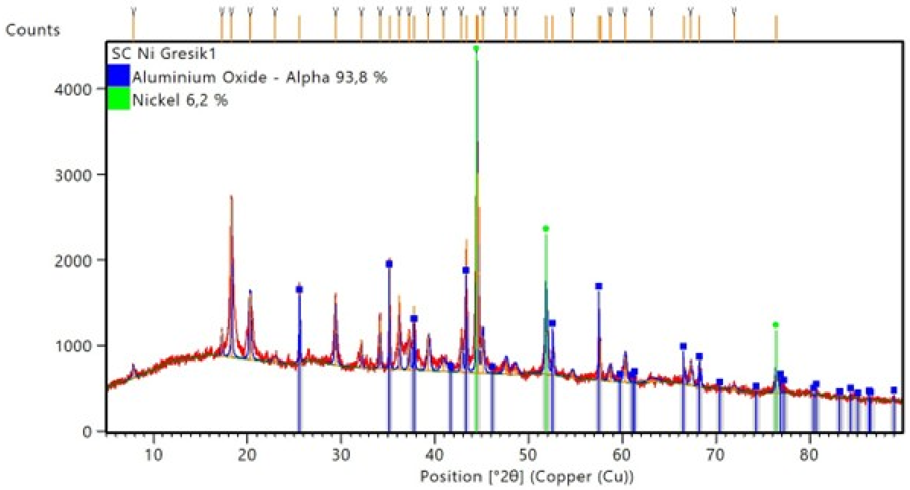


FIGURE 2. XRD spectra of nickel-containing spent catalyst.

ious pulp densities (50; 100; and 200 g/L), various acid concentration (1; 1.5; and 2M), various leaching temperature (30, 60, 80°C), and various leaching period (1, 3, 5 hours) at 200 rpm shaker speed. Analytical-grade of all leaching reagents (Merck, Germany) were used to prepare the solutions. Ultrapure water with a resistivity of 18 ΩM was used throughout this work. The concentration of nickel in the leachate was analyzed using Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES). The nickel recovery percentage (% Ni Recovery) was calculated using the equation 1

$$\%\, Ni\ Recovery\ = \left(\frac{C_{leachate} \times V}{m_{sample} \times\ Ni_{initial}}\right) \times 100\% \qquad (1)$$

Where $C_{leachate}$ is the nickel concentration in the leachate (mg/L), V is the leachate volume (L),

m_{sample} is the initial sample mass (g), and $Ni_{initial}$ represents the initial nickel content in the spent catalyst (wt%).

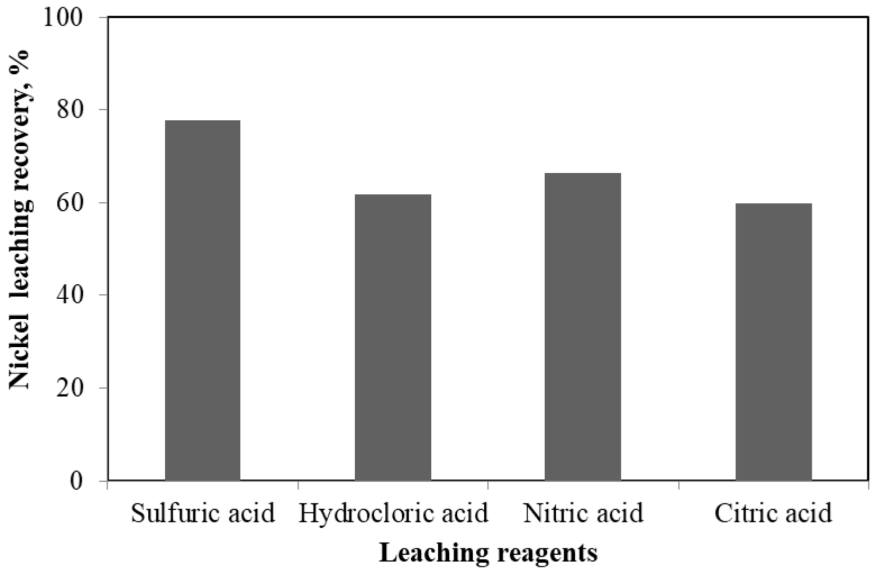
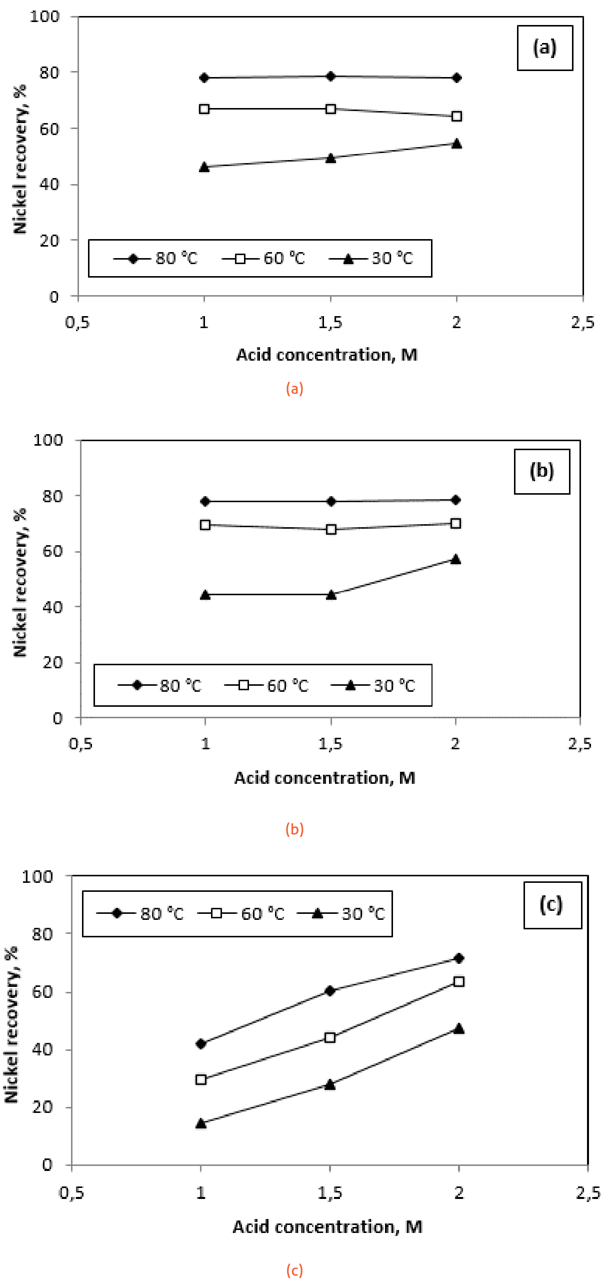


FIGURE 3. Nickel leaching recovery of nickel-containing spent catalyst by various acids with an acid concentration of 1 mol/L, a pulp density of 50 g/L, and a temperature of 80°C.

TABLE 2. Chemical composition of nickel-containing spent catalyst by XRF.

Elements	Ni-oxalate from sulfuric acid	Ni-oxalate from nitric acid	Ni-oxalate from hydrocloric acid	Ni-oxalate from citric acid
Ni	97.29%	49.30%	31.90%	33.06%
Ca	0.99%	50.09%	67.61%	66.24%
Fe	0.52%	0.15%	977.8 ppm	165.6 ppm
Al	0.32%	0.21%	0.18%	0.16%
Mn	0.32%	943.6 ppm	0.11%	0.31%



(a)

(b)

(c)

FIGURE 4. Effect of sulfuric acid concentration and leaching temperature on nickel recoveries from sulfuric acid leaching of spent catalyst at different leaching temperatures (30; 60; 80°C) and pulp density (a) 50 g/L; (b) 100 g/L; (c) 200 g/L.

This equation was used to evaluate nickel extraction efficiency under different leaching conditions, allowing the determination of optimal parameters for maximum nickel recovery. The leaching apparatus used in this study is shown in Figure 1.

3. RESULT AND DISCUSION

3.1 Characterization of raw spent catalyst

Table 1 provides the typical chemical composistion of nickel-containing spent catalyst used in this study. These were obtained through the use of XRF analysis of the major and minor elements of samples. From the analysis, it is also clear that aluminium, nickel, and calcium are the major constituents. Iron, which is usually an undesirable impurity in downstream metallurgical processes, is low. Figure 2 shows the XRD spectra of raw nickel-containing spent catalyst. Powder XRD showed that the major elements are Al_2O_3, Ni, and CaO. This spectra confirms the XRF results.

3.2 Effect of leaching reagents on the nickel leaching recovery using various acids

Leaching experiments were performed with various acids in an isothermal reaction vessel. Figure 3 shows that the highest nickel recovery of 77.74% was achieved when sulfuric acid was applied to the leaching process. In the same condition, hydrochloric acid, nitric acid, and citric acid can dissolve nickel at 61.81%, 66.29%, and 59.74% respectively. The superior performance of sulfuric acid in nickel dissolution can be attributed to its strong acidic nature and the solubility of nickel sulfate in aqueous solutions. The reaction mechanism for nickel oxide leaching with sulfuric acid is as Equation 2.

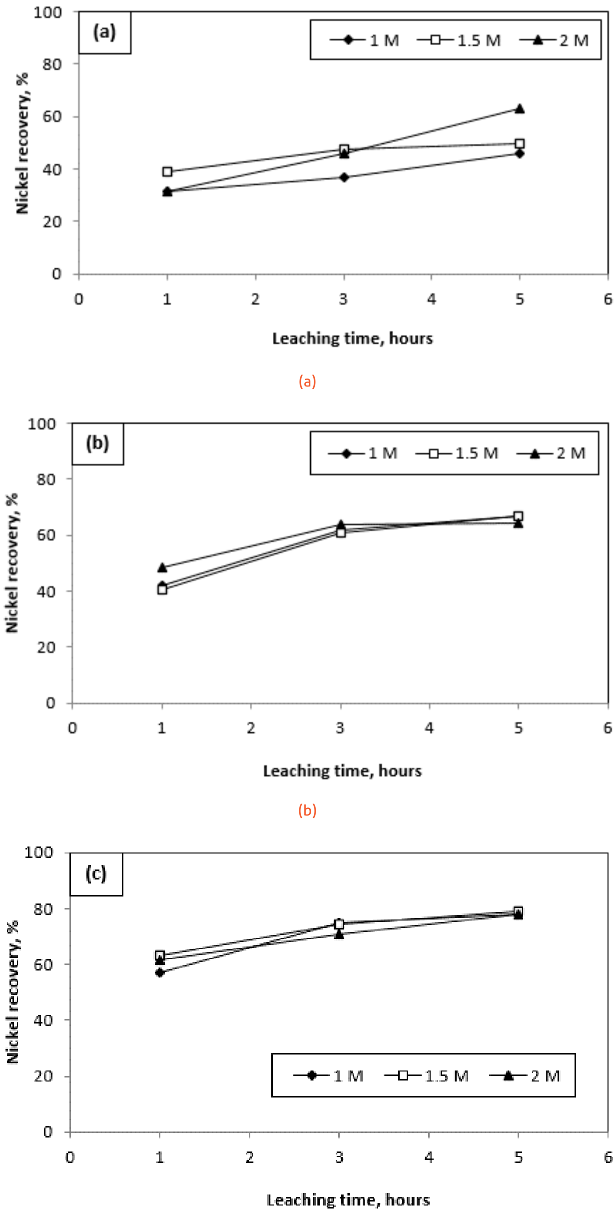
$$NiO_{(s)} + H_2SO_{4(aq)} + 5H_2O \rightarrow NiSO_4.6H_2O_{(aq)} \tag{2}$$

The efficiency of leaching is governed by the proton activity, oxidizing potential, and complexation ability of the acid used. Acids catalyze the dissolution of metal oxides by donating protons (H^+) to break the metal-oxygen bonds, thereby solubilizing the metal ions into the solution. The general leaching reaction for metal oxides can be represented as Equation 3.

$$MeO_{(s)} + 2H^+ \rightarrow Me^{2+}_{(aq)} + H_2O \tag{3}$$

where MeO represents a metal oxide such as NiO, and H^+ is provided by the acid. The effectiveness of different acids in the leaching process depends on their ability to supply H^+ ions, their oxidation strength, and their tendency to form soluble complexes with metal ions. Sulfuric acid is a strong diprotic acid that effectively dissociates in solution, providing a high concentration of H^+ ions, which enhances the dissolution of nickel oxide. Additionally, nickel forms highly soluble $NiSO_4$ complexes, which prevent precipitation and enhance nickel recovery. In contrast, nitric acid has a strong oxidizing property that facilitates the dissolution of nickel oxide by oxidizing Ni(II) to Ni(III) species, which are more soluble, but the formation of nitrate salts with varying solubility can sometimes reduce extraction efficiency. Hydrochloric acid dissolves nickel oxide primarily through proton attack, but the formation of $NiCl_2$ complexes in solution may limit dissolution due to chloride complexation effects. Furthermore, Cl_2 gas evolution at high concentrations can lead to volatilization losses. Citric acid, an organic acid, relies on both proton activity and complexation, but its lower dissociation and weaker acidic strength result in lower metal dissolution efficiency compared to strong mineral acids. The formation of Ni-citrate complexes, though beneficial for solubility, is less efficient in breaking metal-oxygen bonds compared to sulfate and nitrate ions.

The findings of this study align with previous research on nickel leaching from spent catalysts. For instance, a study by Muntaqin et al. (2022) reported a nickel recovery of 96.43% using sulfuric acid at a concentration of 1 M in atmospheric



(a)

(b)

(c)

FIGURE 5. Effect of leaching period on nickel recoveries from sulfuric acid leaching of spent catalyst at different acid concentration (1; 1.5; 2 M) and leaching temperatures (a) 30°C; (b) 60°C; (c) 80°C.

acid leaching of nickel laterite ores, which demonstrates the high efficiency of sulfuric acid in nickel dissolution. Furthermore, a study by Mystrioti et al. (2018) on counter-current leaching of low-grade laterites with hydrochloric acid found that the overall nickel recovery was 40%, indicating the varying effectiveness of hydrochloric acid depending on ore type and leaching conditions. The lower recovery obtained using citric acid corresponds with findings presented by Muntaqin et al. (2022), who noted that organic acids generally exhibit lower metal dissolution efficiency due to their moderate acidity and weaker metal-ligand interactions. The comparison with these studies reinforces the conclusion that sulfuric acid is the most effective leaching reagent for nickel recovery from spent catalysts under atmospheric conditions.

3.3 Effect of leaching reagents on the nickel precipitation using oxalic acid

In the second stage, metals were selectively precipitated from acid-leached solutions by addition of oxalic acid solution. Metal oxalate was generated. Table 2 shows that the highest Ni recovery of 97.29% was achieved when oxalic acid added into leaching solution of sulfuric acid. This result inline with Mouna et al. (2021) that reported oxalic acid has high efficincy for nickel leaching.

$$NiSO_4 + H_2C_2O_4.2H_2O \rightarrow NiC_2O_4.2H_2O + H_2SO_4 \qquad (4)$$

Considering those results, sulfuric acid was used as the leaching agent for further variation of the parameters, such as acid concentration, temperature, leaching time, and the pulp density. Hydrochloric acid has issues such as volatilization and difficulty utilizing waste acid, while nitric acid has a high cost. Sulfuric acid is therefore often selected as an inorganic leachate in the industry (Wang et al. 2022).

Acid concentration and leaching temperature are significant parameters influencing metal extraction. The effects of sulfuric acid concentration and leaching temperature on the nickel recovery were studied for concentrations of 1, 1.5 and 2 mol/L at 5 hours leaching time; 50 g/L; 100 g/L and 200 g/L of pulp densities; and three different leaching temperatures (30°C, 60°C and 80°C). Temperature is an important factor affecting the kinetics of leaching in hydrometallurgical processes. It is generally considered that higher temperatures are used to increase the leaching rate.

Figure 4 shows that nickel recoveries at all pulp densities increase with increasing leaching temperature. However, nickel recovery was not strongly influenced on increasing acid concentration except for 200 g/L of pulp density. The results indicate that temperature has an appreciable effect on nickel extraction. The optimum nickel recovery of 78% was achieved at 1 mol/L H_2SO_4, 100 g/L of pulp density and 80°C (Fig.4(a)). The lowest is 14% at 1 mol/L H_2SO_4, 30°C, 200 g/L of pulp density. The trend of nickel recovery between 50 g/L and 100 g/L of pulp density is similar.

In addition, these experiments also show that nickel recovery is low at higher pulp density. This might be caused by the high amount of solid that need to dissolve by an acid. The amount of acid was not enough to dissolve all metals-containing spent catalyst. Fig.4c shows that nickel recovery at 200 g/L pulp density was lower than the results of 50 g/L and 100 g/L of pulp density.

The effect of temperature on the leaching process can be analyzed using a kinetic approach. One of the most commonly used kinetic models for leaching reactions is the Shrinking Core Model (SCM), which describes the dissolution of solid particles in a liquid phase. According to this model, leaching occurs through a sequence of steps: diffusion of the reactant through the liquid film surrounding the particle, diffusion through the solid product layer, and chemical reaction at the unreacted core of the particle. The overall leaching rate is often controlled by one of these steps. For nickel leaching from laterite ores, the process is typically reaction-controlled, meaning that the dissolution rate is determined by the chemical reaction at the solid-liquid interface.

The increase in temperature reduces the activation energy barrier, thereby increasing the reaction rate and enhancing nickel dissolution. This trend is consistent with studies on acid leaching of nickel, which report higher leaching efficiencies at elevated temperatures due to faster reaction kinetics (Wanta et al. 2016). The results of this study align with previous research which also reported that nickel leaching follows shrinking core model with controlled by diffusion through the solid product layer and that the rate increases with temperature (Astuti et al. 2015). The strong temperature dependence suggests that activation energy calculations and kinetic modeling would be useful for further optimizing the process conditions.

3.4 Effect of leaching time on nickel recovery

The leaching test were carried out with 1, 1.5 and 2 M sulfuric acid; 100 g/L of pulp density; leaching temperatures (30°C, 60°C and 80°C) and three different leaching time (1, 3 and 5 h). The results shown in Figure 5. From the experiment, it was observed that the nickel recovery increased with increasing leaching time. The highest nickel recovery of around 78% was achieved after 5 hours leaching time, at 80°C, 100 g/L at all acid concentration (1-2 M). The leaching time effected to nickel recovery by the period of interaction between nickel-containing spent catalyst and sulfuric acid.

4. CONCLUSION

1. Sulfuric acid was found to be the most selective leaching reagent of nickel from the nickel-containing spent catalyst for further nickel oxalate precipitation.
2. Nickel was selectively precipitated and separated as $NiC_2O_4.2H_2O$ with the purity of nickel >97% from the sulfuric acid-leached solution.
3. The optimum leaching conditions can be obtained under the condition of 1-2 M H_2SO_4, 100 g/L of pulp density, at 80°C of temperature for 5 h of reaction time which around 78% of nickel recovery.

5. DECLARE

All the authors have no interest conflict.

6. AUTHOR STATEMENT

Widi Astuti: Corresponding author, supply idea, give the finance, and draft the manuscript; Aulia Pertiwi Tri Yuda and Slamet Sumardi: do experiments, characterize the samples; analyze data; Mutia Dewi Yuniati, Kevin Cleary Wanta, Jilda Sofiana Dewi and Himawan Tri Bayu Murti Petrus: threat some data and edit the manuscript.

7. DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

8. ACKNOWLEDGEMENT

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