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## Nickel Extraction From Solid Waste From The Nickel Smelting Industry Through An Environmentally Friendly Organic Acid Leaching Process

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### Abstract

*This study aims to analyze the effect of the type and concentration of organic acids on nickel extraction results. The analysis shows that the type of organic acid has a significant effect on the effectiveness of nickel leaching. Citric acid produces higher extraction yields than acetic acid due to its polyprotic nature, which enables it to form stronger complexes with nickel ions. In addition, the concentration of organic acids plays an important role in the extraction process. The optimum concentration was achieved at 7 M, resulting in the highest nickel percentage of 0.78%. At a concentration of 4 M, the extraction yield was lower due to the limited availability of H<sup>+</sup> ions, while at 10 M, a decrease in extraction efficiency was observed, which is likely caused by the formation of secondary complexes or precipitates that inhibit nickel solubility. Therefore, the appropriate selection of organic acid type and concentration is crucial for optimizing the nickel extraction process.*

**Keywords:** Nickel Extraction, Organic Acids, Citric Acid, Acetic Acid, Acid Concentration.

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## INTRODUCTION

The rapid growth of industry, particularly in the mining and metal processing sectors, has resulted in large amounts of solid waste that have the potential to pollute the environment. One type of waste that has economic value but is often discarded is solid waste that still contains heavy metals, such as nickel. Nickel is a strategic metal widely used in various industries, ranging from stainless steel manufacturing to battery production (Kotok & Kovalenko, 2018).

To date, nickel extraction from natural raw materials has generally been carried out through pyrometallurgical and hydrometallurgical methods. Although these methods are effective, they often have negative environmental impacts due to the use of hazardous chemicals and high energy consumption. Therefore, the development of more environmentally friendly extraction methods has become an urgent necessity to support the principles of sustainable development (Sudalto, 2022).

One promising approach is the leaching process using organic acids, which are biodegradable and less toxic than conventional inorganic acids. The use of organic acids such as citric acid, acetic acid, and lactic acid has demonstrated potential in dissolving heavy metals from various types of solid waste without causing significant environmental impacts (Lo et al., 2024).

Several previous studies have explored organic acid-based leaching as a more environmentally friendly alternative. Research conducted by Rhamdhani et al. (2021) showed that citric acid was able to dissolve nickel from laterite with relatively high efficiency. Similar results were reported by Wahyudi and Siregar (2022), who found that acetic acid could extract nickel from industrial solid waste with lower energy consumption compared to conventional methods. Meanwhile, a more recent study by Lo et al. (2024) confirmed that organic acids such as citric acid, acetic acid, and lactic acid are capable of dissolving heavy metals from various types of waste without causing significant environmental impacts.

This study aims to evaluate the efficiency of nickel extraction from industrial solid waste through a leaching process using environmentally friendly organic acids. It is expected that the results of this research will provide an alternative and more sustainable metal recovery technology and contribute to reducing environmental pollution caused by industrial waste.

### **Problem Formulation**

1. How does the type of organic acid affect the nickel extraction yield?
2. How does the concentration of organic acid affect the nickel extraction yield?

### **Research Objectives**

1. To determine the effect of the type of organic acid on nickel extraction yield.
2. To determine the effect of organic acid concentration on nickel extraction yield.

### **Scope and Limitations**

1. The types of organic acids used are limited to citric acid and acetic acid.
2. The leaching process is conducted under limited temperature and time conditions according to predetermined parameters.

## **RESEARCH METHODS**

### **Research Location and Duration**

This research is planned to be conducted for approximately three months in a laboratory.

### **Materials and Equipment**

#### 1. Materials

The materials used in this study include industrial waste, citric acid, acetic acid, and distilled water.

#### 2. Equipment

The equipment used in this study consists of a jaw crusher, ball mill, sieve, drying oven, beaker glass, hot plate with magnetic stirrer, water bath, thermometer, pH meter, burette, filter press, filter paper, and XRF Epsilon 4.

### **Research Variables**

#### 1. Controlled Variables

The controlled variables in this study are:

- a. Mass of industrial waste : 100 grams
- b. Leaching temperature : 90°C
- c. Leaching time : 4 hours

#### 2. Independent Variables

The independent variables in this study are:

- a. Type of organic acid : citric acid and acetic acid
- b. Organic acid concentration : 4 M, 7 M, and 10 M
- c. Waste sampling location : Company A (PT A) and Company B (PT B)

### **Research Procedure**

#### 1. Sample Preparation

Representative samples of industrial solid waste (such as spent batteries, spent catalysts, or slag) were collected. The samples were size-reduced using a jaw crusher or hammer mill to obtain smaller particle sizes, followed by further grinding using a ball mill or disc mill to achieve the desired particle size (e.g., below 100 microns or according to research standards). The ground samples were dried in an oven at approximately 105°C for at least 24 hours or until a constant weight was achieved to remove moisture. The dried samples were then sieved to obtain a uniform particle size fraction, if necessary.

#### 2. Preparation of Leaching Agent Solutions

Citric acid solutions with concentrations of 4 M, 7 M, and 10 M were prepared using demineralized/distilled water by dissolving solid citric acid to achieve the desired concentrations. Acetic acid solutions with concentrations of 4 M, 7 M, and 10 M were prepared by diluting liquid acetic acid with demineralized/distilled water to obtain the required concentrations. All solutions were prepared accurately and homogenized thoroughly.

#### 3. Leaching Process

A total of 100 grams of the prepared solid waste sample was weighed and placed into a beaker glass or Erlenmeyer flask equipped with a hot plate and magnetic stirrer. A predetermined volume of one of the organic acid solutions (citric acid or acetic acid) at varying concentrations (4 M, 7 M, or 10 M) was added to the reaction vessel. A solid-to-liquid ratio of 1:10 was maintained consistently for all experiments. The temperature of the hot plate or water bath was adjusted to reach 90°C and maintained throughout the leaching process. The magnetic stirrer was set at a constant stirring speed of 500 rpm to ensure effective contact between the solid and liquid phases. The leaching process was carried out for 4 hours. After leaching, the mixture was filtered using filter paper, and the residue was dried in an oven for 2 hours before being analyzed using XRF Epsilon 4.

$$\text{Leaching Efficiency} = \frac{\text{Nickel Mass in Leaching Solution}}{\text{Nickel Mass in Waste}} \times 100 \dots (3.1)$$

**Diagram Alir**

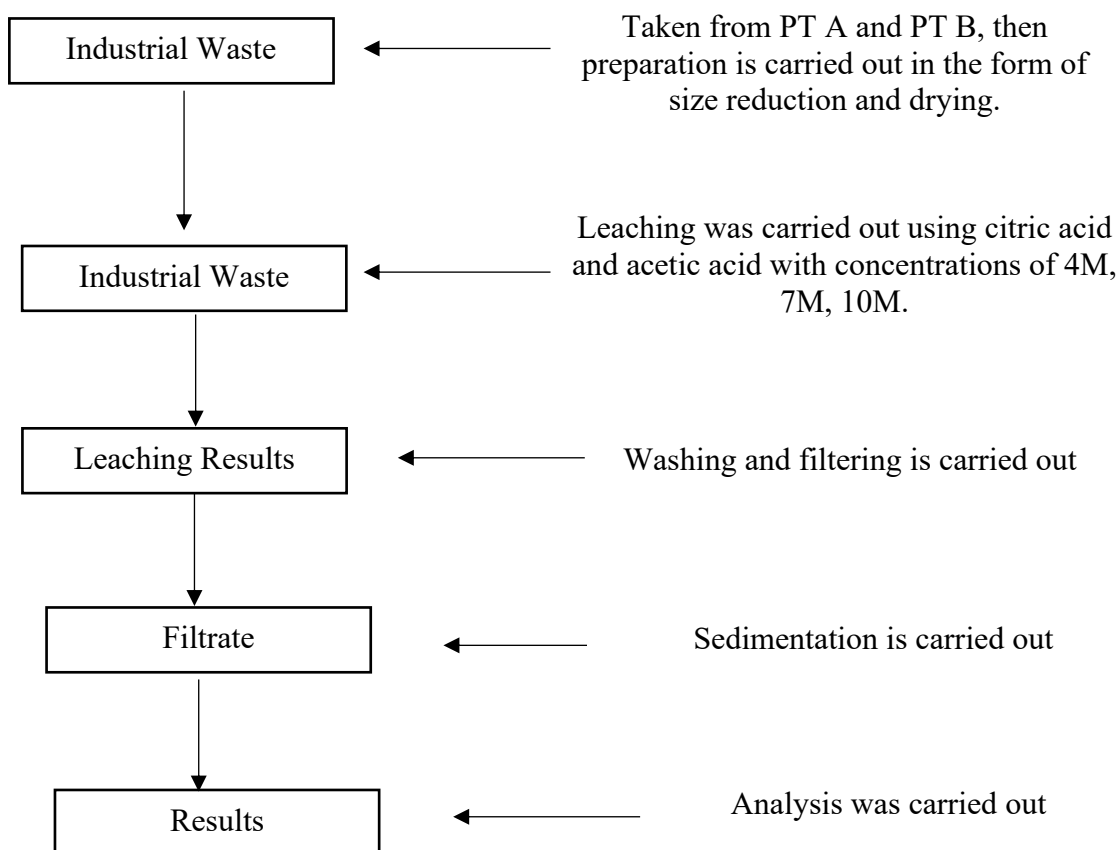


Figure 1. Research Flowchart

**RESULTS AND DISCUSSION**

Solid waste management from the nickel smelting industry is a critical concern because it still contains valuable metals like nickel and has the potential to pollute the environment. Efforts to reuse this nickel content require effective and environmentally friendly methods. One widely developed alternative is the leaching process using organic acids, which is considered safer, more biodegradable, and has a lower pollution impact than inorganic acids. This approach is expected to enable sustainable nickel extraction from waste while also supporting industrial waste reduction. Samples for this study were taken from two different locations, PT A and PT B.

The following are the results of nickel extraction from solid waste from the nickel smelting industry through the environmentally friendly organic acid leaching process:

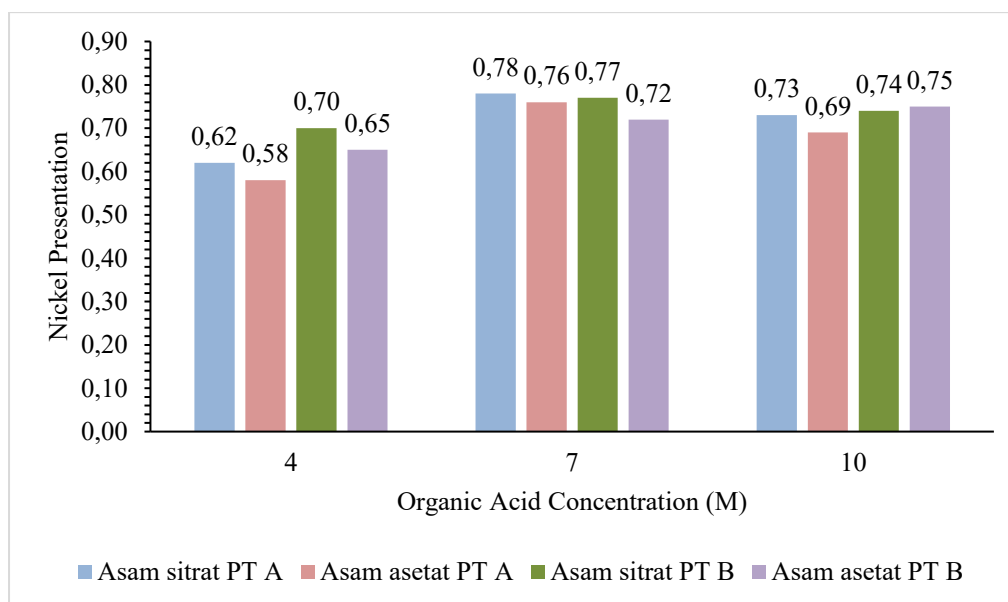


Figure 2. Nickel Extraction Results from Solid Waste from the Nickel Smelting Industry

The graph illustrates the effectiveness of the leaching process using organic acids on solid waste from the nickel smelting industry with varying concentrations of 4 M, 7 M, and 10 M at two sample locations, PT A and PT B. In general, increasing the acid concentration significantly affected the percentage of dissolved nickel, with optimum conditions achieved at a concentration of 7 M. At this concentration, nickel extraction yields were higher than at 4 M and 10 M, using both citric and acetic acids. For example, at PT A, citric acid yielded 0.78% Ni, higher than 0.62% at 4 M and 0.73% at 10 M. A similar pattern was also observed at PT B, where 7 M citric acid yielded 0.77% Ni, compared to 0.70% at 4 M and 0.74% at 10 M. This indicates an optimum acid concentration, where 7 M provides ideal conditions for dissolving Ni ions without causing the formation of stable complexes or secondary deposits that could hinder the leaching process (Zulyansyah, 2018).

Comparing the acid types, citric acid tends to be more effective than acetic acid. This is due to citric acid's nature as a polyprotic acid with three carboxylic groups that can form strong complexes with metal ions, thus facilitating the release of nickel from the solid waste matrix. Conversely, acetic acid, being monoprotic, exhibits lower extraction capacity due to its relatively weaker complexation interaction with nickel ions. Nevertheless, both still yielded significant results, with Ni concentrations ranging from 0.65 to 0.78% (Fathoni & Mubarak, 2024).

When compared with the nickel concentrations found in the field, particularly in solid waste from nickel smelting, a significant discrepancy is apparent. Generally, nickel levels in saprolite or limonite smelting waste can range from 1.5% to over 2% Ni, depending on the ore source and the process used. Therefore, the values obtained from leaching using organic acids (maximum 0.78%) are still slightly lower than the initial nickel content in the waste. This is understandable, considering that leaching using organic acids is more selective and environmentally friendly, meaning not all nickel is dissolved simultaneously, unlike leaching using strong inorganic acids (e.g., H<sub>2</sub>SO<sub>4</sub> or HCl), which can extract larger amounts of nickel (Wahab, Deniyatno, et al., 2021).

However, this is precisely where this method excels. The use of organic acids not only extracts nickel at significant levels but also reduces the environmental pollution impacts caused by the use of hazardous inorganic acids. Furthermore, this approach offers the opportunity to utilize solid waste from the nickel smelting industry sustainably with reduced environmental risks. From a practical perspective, optimum results at a concentration of 7 M with citric acid reaching 0.78% can be considered as efficient operating conditions. Although the results are slightly below the nickel content found in the field, the effectiveness of this method remains promising for development as an alternative environmentally friendly nickel waste processing technology.

### **Analysis of the Effect of Organic Acid Concentration**

Data shows that increasing the concentration from 4 M to 7 M increases dissolved Ni in almost all treatments. At 4 M, the number of H<sup>+</sup> ions and organic ligands is still limited, resulting in slower dissolution of Ni from the particle surface. At 7 M, the solution provides more H<sup>+</sup> to dissolve the NiO phase or other Ni phases, and the number of carboxylate ligands also increases. These ligands form Ni-organic complexes, thus maintaining Ni stability in solution. This condition typically accelerates the achievement of leaching equilibrium and increases the solute yield (Widiatmoko et al., 2020; Nurfaidah et al., 2020).

The decrease in yield at 10 M indicates an inhibitory effect at high concentrations. Highly concentrated solutions can increase viscosity, slowing the diffusion of reagents toward the solid surface. High ionic strength can also reduce the effective ion activity and alter the complexation equilibrium. Furthermore, high concentrations have the potential to dissolve more impurities. Impurities can form a passive layer on the particle surface or trigger the formation of secondary deposits that block pores. These two effects reduce effective contact between the leaching agent and the Ni phase. Therefore, the optimum concentration is not always at the highest value (Tyassena et al., 2022; Ikmal et al., 2021).

### **Comparison of Citric Acid and Acetic Acid**

Citric acid produced higher yields than acetic acid in PT A and PT B. Citric acid is polyprotic and has three carboxylic groups. This structure enhances its chelating ability towards Ni ions. The stable Ni-citrate complex keeps Ni dissolved and suppresses precipitation, especially when the local pH rises due to H<sup>+</sup> consumption during the reaction. Acetic acid has only one carboxylic group, so its complexation is weaker. Ni dissolution relies more on proton attack, so under the same conditions the yield tends to be lower (Nurfaidah et al., 2020; Fathoni & Mubarok, 2024).

Nevertheless, acetic acid is still worth considering. Its performance is quite stable and, at 7 M, it can produce 0.72% to 0.76% dissolved Ni. In practice, the selection of a leaching agent must consider reagent cost, availability, work safety, and the cost of processing the residual solution. A slightly weaker agent can be advantageous if the total process cost is lower or if Ni recovery from the complex is simpler (Wahab, Deniyatno, et al., 2021).

### **Differences in sample characteristics between PT A and PT B**

PT B showed slightly higher dissolved Ni under some conditions, for example, at 4 M citric acid. This difference may stem from different initial Ni levels, Ni mineral forms, and slag matrix composition. Smelting wastewater often contains silicates and iron oxides, which can bind Ni strongly. If more Ni is trapped in the stable silicate phase, the acid solution takes longer to break down the structure. If the Ni phase is predominantly a simple oxide, dissolution occurs more rapidly. Variations in smelting processes and raw materials at different locations can result in different residue structures, resulting in varying leaching responses (Masyrurroh et al., 2022).

Further research is needed to add initial characterization data to support this explanation. Researchers can measure the initial Ni content of the solids before leaching using XRF, then measure the Ni in the residue after leaching. This data allows for mass balance-based recovery calculations. Phase analysis using XRD also helps identify whether the Ni is predominantly present as NiO, nickel silicate, or a mixed phase. If possible, particle size and surface area analysis should also be included to quantify the effect of preparation on leaching rates (Tyassena et al., 2022).

### **Relationship to Fixed Process Variables**

The fixed process variables used, namely 90°C, 4 hours, a solid-to-liquid ratio of 1:10, and stirring at 500 rpm, already support leaching. High temperatures typically increase reaction rates and accelerate diffusion in solution. Stirring reduces the boundary layer thickness around particles, thereby accelerating mass transfer. However, the leaching rate can still be inhibited if the particles are too large or agglomeration occurs during heating. Therefore, reports should include the sieve size fraction used and ensure that the suspension remains homogeneous for 4 hours (Widiatmoko et al., 2020).

Changes in pH during the process can also affect results. At the beginning of leaching, low pH promotes dissolution of the Ni phase. Over time, the pH can increase as H<sup>+</sup> reacts with the oxide or as the solution dissolves basic components of the slag matrix. This increase in pH can decrease the leaching rate and affect the stability of the Ni-organic complex. Monitoring the pH at regular intervals will help clarify when the system begins to slow down and whether side reactions that reduce dissolved Ni occur (Herlina et al., 2021).

### **Implications for Nickel Recovery and Solution Management**

The leached filtrate contains dissolved Ni. The next step must be to recover the Ni to produce a valuable product. Common methods include selective precipitation with pH adjustment, the formation of specific nickel salts, or separation through solvent extraction if higher purification is required. The choice of recovery method must consider other dissolved metals to ensure the process remains selective and easy to operate (Rao et al., 2020).

The use of organic acids offers safety and environmental benefits, but the process still generates liquid waste that must be treated. The treatment process can include neutralization, separation of dissolved metals, and evaluation of the recyclability of the acid solution. Recycling is important because reagent consumption impacts operating costs. If the solution can be reused after Ni recovery, the process will be more efficient and support the concept of utilizing waste as a secondary metal source (Shofi, 2020).

### **Data Limitations and Room for Improvement**

The graphs shown do not include replicates and standard deviations. This condition makes statistical conclusions weak. Future research should replicate each condition at least three times and display error bars. Researchers should also measure other metals, such as Fe and Mg, to assess selectivity. Selectivity data is important because dissolving impurities can complicate Ni purification and increase chemical requirements during the recovery stage (Wahab, Deniyatno, et al., 2021).

## **CONCLUSION**

Based on the analysis results, it can be concluded that:

- a. The type of organic acid significantly affects nickel extraction yield. Citric acid exhibits higher leaching capacity than acetic acid due to its polyprotic nature, which allows it to form stronger complexes with nickel ions, resulting in a higher percentage of Ni under the same conditions.
- b. The concentration of organic acid also plays a significant role in extraction effectiveness. The optimum value was achieved at a concentration of 7 M, with the highest nickel percentage reaching 0.78%. At a concentration of 4 M, extraction yields were lower due to limited H<sup>+</sup> ions, while at 10 M, the yield decreased again due to the possible formation of complexes or secondary precipitates that inhibit nickel solubility.

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