

*Research Article*

Selective Nickel Recovery from the Magnetic Fraction of Chrysotile Asbestos Beneficiation Waste Using Organic Acids

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Abstract: Chrysotile asbestos beneficiation waste poses a significant environmental challenge while representing an underutilized secondary source of nickel. In this study, we investigated the selective leaching of a nickel-enriched magnetic fraction using citric, acetic, oxalic, and tartaric acids combined with comparative kinetic modeling. Citric acid achieved the highest nickel extraction (82.6%), outperforming acetic (61.8%), oxalic (39.2%), and tartaric (26.1%) acids. Kinetic analysis showed that the Drozdov–Rotinyan model best described the process ($R_{\text{avg}}^2 = 0.9837$), indicating a mixed reaction–diffusion mechanism with an apparent activation energy of 21.0 kJ mol⁻¹. Iron dissolution remained low (2.1%–13.5%), producing Ni/Fe selectivity up to 19.2. The extraction and kinetic results indicate that nickel recovery involves proton-assisted mineral dissolution, ligand stabilization of dissolved Ni²⁺, and diffusion-related resistance. This study provides mechanistic insight into selective nickel extraction from iron-rich technogenic materials and demonstrates the potential of organic-acid leaching for chrysotile asbestos beneficiation waste valorization.

Keywords: Nickel recovery; Organic-acid leaching; Reaction–diffusion mechanism; Selective dissolution; Technogenic waste

1. Introduction

The transition toward low-carbon energy systems and large-scale electrification has increased the demand for nickel, which is widely used in stainless steels, high-performance alloys, catalysts, and lithium-ion batteries (Abdirashit et al., 2025b, 2025a; Mamyrbayeva et al., 2025; Shabanov et al., 2026). The depletion of high-grade sulfide deposits has shifted increasing attention toward lateritic ores, which account for approximately 70% of the global nickel reserves

(Mamyrbayeva et al., 2024; Shayakhmetova et al., 2026; Suleimen et al., 2026). However, their complex mineralogy and high iron and magnesium contents complicate selective nickel recovery (Tauakelov et al., 2025). Although conventional high-pressure, atmospheric, and heap-leaching processes may provide high nickel recovery, they are commonly associated with substantial acid consumption, demanding operating conditions, corrosion, and secondary waste generation (Gleeson et al., 2003; Sharipov et al., 2026). These limitations have stimulated interest in less aggressive and potentially more sustainable lixiviants.

LMWOA (low-molecular-weight organic acids) have attracted attention as potentially lower-impact alternatives to strong inorganic lixiviants. Unlike mineral acids, they promote metal dissolution through both proton-assisted mineral decomposition and ligand-assisted complexation of dissolved metal ions. The formation of stable Ni–organic complexes lowers the activity of dissolved Ni^{2+} species, which thermodynamically favors continued dissolution (Chen et al., 2024). Thus, leaching performance depends not only on acidity but also on ligand structure, denticity, complex stability, and feedstock mineralogy. Citric, oxalic, tartaric, and acetic acids differ in their acidity, denticity, and complexation behavior, resulting in substantially different nickel-leaching efficiencies. Although numerous studies have investigated the organic-acid leaching of laterites and other nickel-bearing materials, the relative effectiveness of different organic acids is highly feedstock-dependent and cannot be generalized (Astuti et al., 2016; Hussaini et al., 2024). Previous work has reported promising nickel recoveries using citric and oxalic acids from laterites and serpentine-based materials (Baigenzhenov et al., 2022; Baigenzhenov et al., 2024). However, the behavior of these lixiviants toward nickel-enriched magnetic fractions derived from asbestos-processing waste remains largely unexplored.

Within circular-economy strategies, technogenic materials are increasingly considered as secondary resources or precursors for functional materials. For example, industrial residues, such as dried alum sludge, have been successfully repurposed as adsorbents for phosphate removal from wastewater (Djekoune et al., 2024). In this context, chrysotile-asbestos beneficiation waste represents a particularly large but underutilized secondary resource. These materials have already been subjected to mining, crushing, and beneficiation and are commonly accumulated near existing industrial infrastructure. Therefore, their valorization may combine resource recovery with the mitigation of long-term environmental risks associated with finely dispersed tailings (Ferrante et al., 2020; Ruff, 2017). The Zhitikara chrysotile-asbestos deposit in Kazakhstan contains more than 350 million tons of accumulated waste (Akyzbekov et al., 2023; Baigenzhenov et al., 2024; Sharipov et al., 2026). Magnetic separation can concentrate nickel-bearing and iron-rich phases from this material, transforming low-grade waste into a more suitable feedstock for selective leaching. However, the behavior of such magnetically enriched fractions in organic-acid solutions has not been systematically investigated. Previous studies have focused primarily on overall metal recovery or on magnesium and silica production (Baigenzhenov et al., 2015; Shayakhmetova et al., 2024), whereas the relationships between organic-acid chemistry, nickel selectivity, and dissolution kinetics remain poorly understood.

The leaching of complex oxide–silicate materials is a heterogeneous solid–liquid process governed by interfacial chemical reactions, ligand-assisted complexation, and mass transfer through the evolving particle structure. The relative contributions of surface reaction, internal diffusion, and external film diffusion determine the effects of temperature, particle size, agitation, and residence time. However, the rate-controlling mechanism of nickel dissolution from magnetically enriched asbestos waste has not yet been established. Its identification is necessary for rational lixiviant selection and subsequent process development. Differences in the molecular structure and complexation capacity of citric, oxalic, tartaric, and acetic acids would influence nickel dissolution by controlling proton-assisted mineral decomposition and the stability of dissolved nickel complexes. The novelty of this study lies in the systematic comparison of these four organic acids for nickel extraction from a magnetically enriched asbestos-derived feedstock, together with the evaluation of Ni/Fe selectivity and dissolution kinetics. Comparative application of the Drozdov–Rotinyan, Prout–Tompkins, shrinking-core, and film-diffusion models, combined with

Arrhenius analysis, was used to identify the dominant kinetic regime.

Accordingly, this study investigates nickel leaching from a magnetically enriched fraction of chrysotile-asbestos beneficiation waste using citric, acetic, oxalic, and tartaric acids. The effects of acid type, concentration, liquid-to-solid ratio, temperature, and leaching time on nickel extraction were evaluated, together with the dissolution of iron and magnesium and the corresponding Ni/Fe and Ni/Mg selectivity ratios. The dissolution kinetics were analyzed using the Drozdov–Rotinyan, Prout–Tompkins, shrinking-core, and film-diffusion models, and the apparent activation energy was determined using Arrhenius analysis. The study focuses on nickel because it is the principal target metal in the magnetic fraction, whereas a separate analytical and optimization strategy would be required for a substantially lower cobalt content.

2. Materials and Methods

2.1 Preparation of Feedstock

The feedstock used in this study was the asbestos processing waste generated during the dry beneficiation of chrysotile asbestos fibers. The as-received bulk waste was dried to a constant mass in an electric oven at 105°C. The mass loss due to residual moisture removal was 7.5%–8.0% of the initial wet mass. The dried material was comminuted in a laboratory ball mill (Retsch PM 100, Germany) and dry-sieved to obtain the <0.25 mm size fraction. This fraction was subjected to magnetic separation using a laboratory drum magnetic separator (ERGA SMBS-L, Russia) at a magnetic field intensity of 1500 Oersted ($\approx 119 \text{ kA m}^{-1}$). The obtained magnetic fraction was 5.0–5.2 wt% of the initial dry waste mass. The magnetic fraction appeared as a grayish-black powder and exhibited a strong response to an external magnetic field, consistent with the presence of ferromagnetic phases such as magnetite.

Representative samples of the magnetic fraction were prepared using the coning and quartering method. The material was pulverized in a hardened steel mortar and sieved through a 75 μm test sieve. The undersize fraction, representing more than 95% of the pulverized mass, was collected to ensure a homogeneous particle size distribution suitable for subsequent leaching experiments.

2.2 Characterization of the Magnetic Fraction of a Sample

The chemical composition of the magnetic fraction was determined by wavelength-dispersive X-ray fluorescence spectroscopy (WD-XRF).

Table 1 Bulk chemical composition of the magnetic concentrate determined by WD-XRF analysis

Component	Content (wt%)	Component	Content (wt%)
Fe ₃ O ₄	77.95	SiO ₂	9.75
MgO	9.90	NiO	1.84
Cr ₂ O ₃	0.56	Total	100

Table 1 presents the bulk chemical composition of the magnetic concentrate determined by WD-XRF. The concentrate was dominated by iron oxides, with total iron expressed as Fe₃O₄ equivalent accounting for 77.95 wt%, with MgO and SiO₂ contents of 9.90 and 9.75 wt%, respectively. The NiO content reached 1.84 wt%, confirming the enrichment of nickel-bearing phases during magnetic separation and demonstrating the suitability of the magnetic concentrate as a feed material for selective nickel leaching. A minor Cr₂O₃ content of 0.56 wt% was also detected. The NiO content reported in Table 1 was used as the basis for calculating the nickel leaching efficiency throughout this study.

2.3 Reagents and Leaching Solutions Used

All reagents used in this study were of analytical grade and were used without further purification. Four LMWOA were selected as alternative leaching agents: oxalic acid dihydrate ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, $\geq 99.5\%$), tartaric acid ($\text{C}_4\text{H}_6\text{O}_6$, $\geq 99.5\%$), glacial acetic acid (CH_3COOH , $\geq 99.8\%$), and citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$, $\geq 99.5\%$). These acids were selected because they differ substantially in acidity, molecular structure, number of functional groups, and metal-complexation capacity, thereby allowing systematic evaluation of the influence of ligand chemistry on nickel dissolution behavior.

Leaching solutions were prepared using deionized water with a resistivity of $18.2 \text{ M}\Omega\cdot\text{cm}$. For each organic acid, solutions with concentrations of 0.5, 1.0, 1.5, 2.0, and 2.5 M were prepared by dissolving the required amount of reagent in deionized water under continuous stirring until complete dissolution was achieved. The selected concentration range was chosen to evaluate the influence of acid concentration on nickel extraction efficiency and to identify the concentration interval that provides the most favorable balance between reagent consumption and metal recovery.

Fresh leaching solutions were prepared immediately before each experimental series to ensure the stability and reproducibility of the results. To prevent contamination, all glassware used for solution preparation and leaching experiments was thoroughly rinsed with deionized water before use. The prepared solutions were stored in sealed glass containers until further use. The selected acids represent mono-, di-, and tricarboxylic ligands with substantially different complexation abilities toward divalent metal ions, making them suitable candidates for investigating nickel leaching mechanisms.

2.4 Experimental Design

The effects of three operating parameters on nickel leaching were investigated: organic-acid concentration, liquid-to-solid ratio, and temperature. The acid concentration was varied between 0.5 and 2.5 M (0.5, 1.0, 1.5, 2.0, and 2.5 M), the liquid-to-solid ratio was varied at 3:1, 4:1, and 5:1 mL g^{-1} , and the leaching temperature was varied at 20, 50, and 80°C . A univariate experimental approach was adopted in which one parameter was varied while the others were maintained at baseline conditions. The baseline conditions were as follows: acid concentration, liquid-to-solid ratio of 4:1 mL g^{-1} , temperature of 50°C , stirring speed of 300 rpm, and leaching time of 180 min. All experiments were conducted in triplicate. The effect of leaching time was evaluated over the interval from 30 to 210 min.

2.5 Leaching Procedure

Leaching experiments were conducted in batch mode under atmospheric pressure. In a typical experiment, 20 g of the prepared magnetic fraction was placed in a 500 mL glass reactor equipped with a mechanical overhead stirrer and a liquid sampling port. The reactor was immersed in a thermostatic water bath to maintain the required temperature with an accuracy of $\pm 1^\circ\text{C}$. Before leaching, both the solid sample and the organic acid solution were separately preheated to the target temperature to minimize temperature fluctuations at the beginning of the experiment. After reaching thermal equilibrium, the preheated organic acid solution was rapidly added to the reactor containing the magnetic fraction. This moment was taken as the starting point of the leaching experiment. The suspension was stirred at a constant rate of 300 rpm throughout the experiment to maintain homogeneous solid-liquid contact. The effects of acid concentration, liquid-to-solid ratio, and temperature were investigated according to the experimental design described in Section 2.4.

Liquid samples (5.0 mL) were withdrawn at predetermined time intervals of 30, 60, 90, 120, 150, 180, and 210 min. Each aliquot was immediately diluted to 50 mL with deionized water to suppress the further reaction and then filtered through a $0.45 \mu\text{m}$ membrane filter before nickel analysis. After each experiment was completed, the leach slurry was vacuum-filtered through

pre-weighed Whatman No. 42 filter paper. The solid residue was washed twice with 50 mL of deionized water to remove residual acid, dried to a constant mass at 95°C, and stored for subsequent characterization. The filtrate and washing solutions were combined, and the final solution volume was recorded for nickel mass-balance calculations.

The leaching efficiency (LE) for Ni was calculated as the percentage of the total nickel mass transferred into the solution relative to its initial content in the solid sample, as follows:

$$\text{LE (\%)} = \frac{C_{\text{Ni}} \cdot V}{m_{\text{sample}} \cdot w_{\text{Ni}}} \cdot 100 \quad (1)$$

where LE is the nickel leaching efficiency (%), C_{Ni} is the nickel concentration in the leach solution (g L^{-1}), V is the volume of the leach solution (L), m_{sample} is the mass of the magnetic fraction used in the experiment (g), and w_{Ni} is the mass fraction of nickel in the initial magnetic fraction.

Changes in the solution volume caused by sampling were taken into account when calculating the cumulative nickel extraction. All experiments were conducted in triplicate, and the results are presented as mean values with standard deviations. Linear regression analysis was performed for kinetic fitting, and R^2 values were used to compare the applicability of the tested kinetic models. The reproducibility of the leaching experiments was assessed based on the standard deviation of the nickel extraction values obtained from parallel experiments.

2.6 Analytical Methods Used

The chemical composition of the magnetic fraction was determined by wavelength-dispersive X-ray fluorescence spectroscopy using a PANalytical Axios spectrometer. The instrument was calibrated using certified reference materials. The concentrations of Ni, Fe, and Mg in the leach solutions were measured by flame atomic absorption spectrometry (TAS-990). Nickel was determined at a wavelength of 232.0 nm using a hollow nickel cathode lamp, while Fe and Mg were analyzed under their corresponding element-specific operating conditions. Calibration curves were prepared using standard solutions obtained by dilution of certified 1000 mg L^{-1} stock solutions. Each solution was analyzed in triplicate, and the mean concentration was used to calculate the extraction efficiencies of Ni, Fe, and Mg.

2.7 Kinetic Analysis

To identify the rate-controlling mechanism of nickel dissolution, the experimentally determined extraction values were converted to fractional conversion, α , and fitted using the Drozdov–Rotinyan, Prout–Tompkins, shrinking-core, and film-diffusion models. These models were selected to evaluate mixed reaction–diffusion control, progressive exposure of reactive sites, surface-reaction control, and external mass-transfer control (Baimbetov et al., 2023; Wang et al., 2025; Yessengaziyev et al., 2025b).

The linearized form of the Drozdov–Rotinyan model can be expressed as follows:

$$\frac{1}{\tau} \ln \frac{1}{1 - \alpha} = M - \beta \frac{\alpha}{\tau} \quad (2)$$

where α is the fractional conversion (dimensionless, 0–1), τ is the interaction duration (min), and β and M are the empirical kinetic constants associated with the process retardation and the overall reaction rate, respectively.

The Prout–Tompkins model, which represents the progressive exposure of reactive sites during structural alteration of the solid, was expressed as follows:

$$\ln \left(\frac{\alpha}{1 - \alpha} \right) = k \ln \tau + b \quad (3)$$

where α is the fractional conversion (dimensionless, 0–1), k is the corresponding fitting or rate coefficient, τ is the time (min), and b is the intercept.

This model is often employed to describe solid-state transformations in which the overall reaction kinetics are accelerated by the progressive exposure of fresh reactive surfaces (Baimbetov et al., 2023).

The chemical-reaction-controlled form of the shrinking-core model was written as follows:

$$1 - (1 - \alpha)^{1/3} = k\tau \quad (4)$$

Whereas possible control by mass transfer through the liquid boundary layer was evaluated using the following film-diffusion equation:

$$-\ln(1 - \alpha) = k\tau \quad (5)$$

In these equations, α is the fractional nickel conversion, τ is the leaching time (min), k is the corresponding fitting or rate coefficient, and b is the intercept of the Prout–Tompkins equation. The model applicability was compared using the coefficient of determination (R^2). Because the Drozdov–Rotinyan model provided the best overall fit, its apparent rate constants M , determined at different temperatures, were used to calculate the apparent activation energy from the Arrhenius relationship:

$$\ln k = \ln A_{\text{exp}} - \frac{E_a}{RT} \quad (6)$$

where A_{exp} is the frequency factor, E_a is the apparent activation energy (kJ mol^{-1}), R is the universal gas constant ($R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), and T is the absolute temperature (K).

In Eq. (6), k denotes the apparent kinetic rate constant. For the Drozdov–Rotinyan model, the experimentally determined parameter M was used as the corresponding apparent rate constant. The value of E_a was calculated from the slope of the linear relationship between $\ln k$ and $1000/T$, where the M values obtained from the Drozdov–Rotinyan model were used as the apparent rate constants. The obtained E_a values were subsequently used to assess the dominant dissolution mechanism. In general, activation energies below approximately 20 kJ mol^{-1} are characteristic of diffusion-controlled processes, values between 20 and 40 kJ mol^{-1} indicate mixed control, whereas activation energies exceeding 40 kJ mol^{-1} are typically associated with chemical-reaction control (Shoshay et al., 2023; Yessengaziyev et al., 2025a).

3. Results and Discussion

3.1 Effect of Process Parameters on Nickel Leaching

The effects of organic acid concentration, leaching time, and temperature on nickel extraction from the magnetic fraction of asbestos-processing waste are shown in Figures 1–3. The results clearly demonstrate that nickel dissolution strongly depends on the type of organic acid and operating conditions. Among the investigated lixiviants, citric acid, followed by acetic, oxalic, and tartaric acids, exhibited the highest extraction efficiency under all studied conditions. This order remained unchanged regardless of the acid concentration, temperature, or leaching time, indicating that the molecular structure and complexation capacity of the organic acids play a decisive role in nickel dissolution. Figure 1 shows the combined effect of organic acid concentration and temperature on nickel extraction after leaching for 180 min.

For all organic acids, nickel extraction increased with increasing acid concentration from 0.5 to 2.5 M and temperature from 20 to 80°C . However, the magnitude of this effect differed substantially depending on the acid type. Citric acid provided the highest nickel extraction, increasing from 34.18% at 0.5 M to 82.60% at 2.5 M at 80°C and 180 min . Acetic acid showed an intermediate performance, with nickel extraction increasing from 21.36% to 61.80% over the same concentration range. In contrast, oxalic and tartaric acids were significantly less effective, reaching only 39.20% and 26.11% , respectively, at 2.5 M . Although 2.5 M gave the highest extraction for all acids, the gain over 2.0 M was marginal: for citric, acetic, oxalic, and tartaric acids, extraction increased by only 1.16 – 4.08 percentage points (from 78.82% to 82.60% , 57.72%

to 61.80%, 36.52% to 39.20%, and 24.95% to 26.11%, respectively). Considering the higher reagent consumption and practical limitations associated with more concentrated organic acid solutions, 2.0 M was selected as the rational acid concentration for further experiments. This concentration ensures high recovery while minimizing reagent costs and avoiding the practical challenges of handling highly concentrated organic acid solutions.

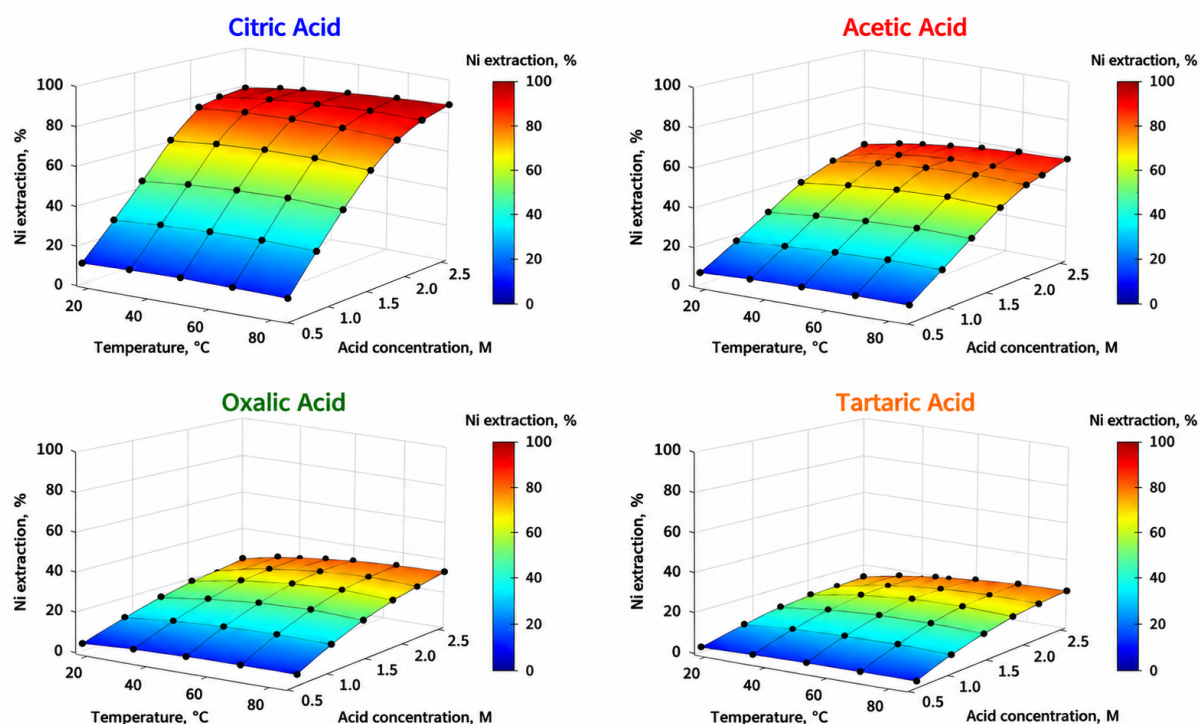


Figure 1 Effects of temperature and acid concentration on nickel extraction from the magnetic fraction of chrysotile asbestos beneficiation waste using (a) citric, (b) acetic, (c) oxalic, and (d) tartaric acid

The superior performance of citric acid can be attributed to its tricarboxylic structure and strong chelating ability toward Ni^{2+} ions. Nickel dissolution in organic acid media involves both proton-promoted breakdown of nickel-bearing phases and ligand-driven stabilisation of released Ni^{2+} in solution. Citrate anions can form stable soluble nickel–citrate complexes, thereby decreasing the activity of free Ni^{2+} in solution and promoting further dissolution of nickel-bearing phases. The superior performance of citric acid observed in the present study is consistent with previous reports showing that citrate ligands promote nickel dissolution through the combined effects of proton-assisted mineral decomposition and Ni–citrate complex formation. Astuti et al. (2016) demonstrated that the citric-acid leaching of Indonesian saprolitic ores is strongly controlled by mineralogy, with nickel hosted in serpentine being more readily dissolved than nickel associated with more refractory phases. Similarly, Kursunoglu and Kaya (2015) showed that citric acid was the most effective organic lixiviant during the initial selective leaching of nickel from lateritic ore, although their sequential citric–oxalic system also caused substantial iron dissolution.

In contrast, Hosseini Nasab et al. (2020) reported only 25.5% nickel recovery from iron-rich laterite under optimized organic-acid leaching conditions, highlighting the difficulty of extracting nickel from highly ferruginous matrices. The relative performance of organic acids observed in the present study is consistent with the findings of Tzeferis and Agatzini-Leonardou (1994), who investigated the leaching of Greek laterite ores using various organic acids. These authors reported that citric acid achieved the highest nickel recovery (up to 60%) among the organic acids tested, whereas oxalic acid dissolved negligible nickel (<3%) but extracted >60% of iron, highlighting its remarkable selectivity for iron over nickel, a pattern that closely parallels the

present results for the asbestos-derived magnetic fraction. Furthermore, Geng et al. (2020) investigated metal leaching from iron tailings using citric, oxalic, and malic acids. Consistent with the present findings, citric acid exhibited comparatively high leaching efficiency, whereas oxalic acid showed lower performance because of the formation of sparingly soluble metal oxalates. Their XPS results indicated an increased contribution of carboxylate groups after leaching, supporting the participation of organic ligands in metal–ligand interactions. XPS analysis revealed that the proportion of COO^- functional groups increased markedly after LMWOA leaching, confirming the participation of organic ligands in metal complexation. XPS analysis revealed an increased contribution of COO^- groups after leaching, supporting the participation of organic ligands in metal complexation.

Therefore, the 82.6% nickel extraction achieved in the present study cannot be attributed solely to citrate complexation; rather, it reflects the combined effect of ligand-assisted dissolution and preliminary magnetic beneficiation, which increased the abundance and accessibility of nickel-bearing domains while limiting the dissolution of the iron-rich matrix. This distinction emphasizes the central novelty of this process: selective nickel recovery is governed not only by lixiviant chemistry but also by feedstock pre-concentration and mineralogical accessibility. Despite its strong complexing capacity, oxalic acid exhibited lower efficiency, which may be associated with the formation of sparingly soluble nickel oxalate species. Such solid formation reduces the dissolved nickel concentration and passivates the reactive surface, hindering further leaching. This explains why oxalic acid produced lower nickel extraction than acetic acid, even though oxalic acid is a stronger acid and a more powerful ligand. Tartaric acid showed the lowest extraction efficiency, which may be related to its weaker ability to promote continuous dissolution of nickel-bearing phases under the studied conditions and to the lower stability of soluble nickel–tartrate complexes compared with nickel–citrate species.

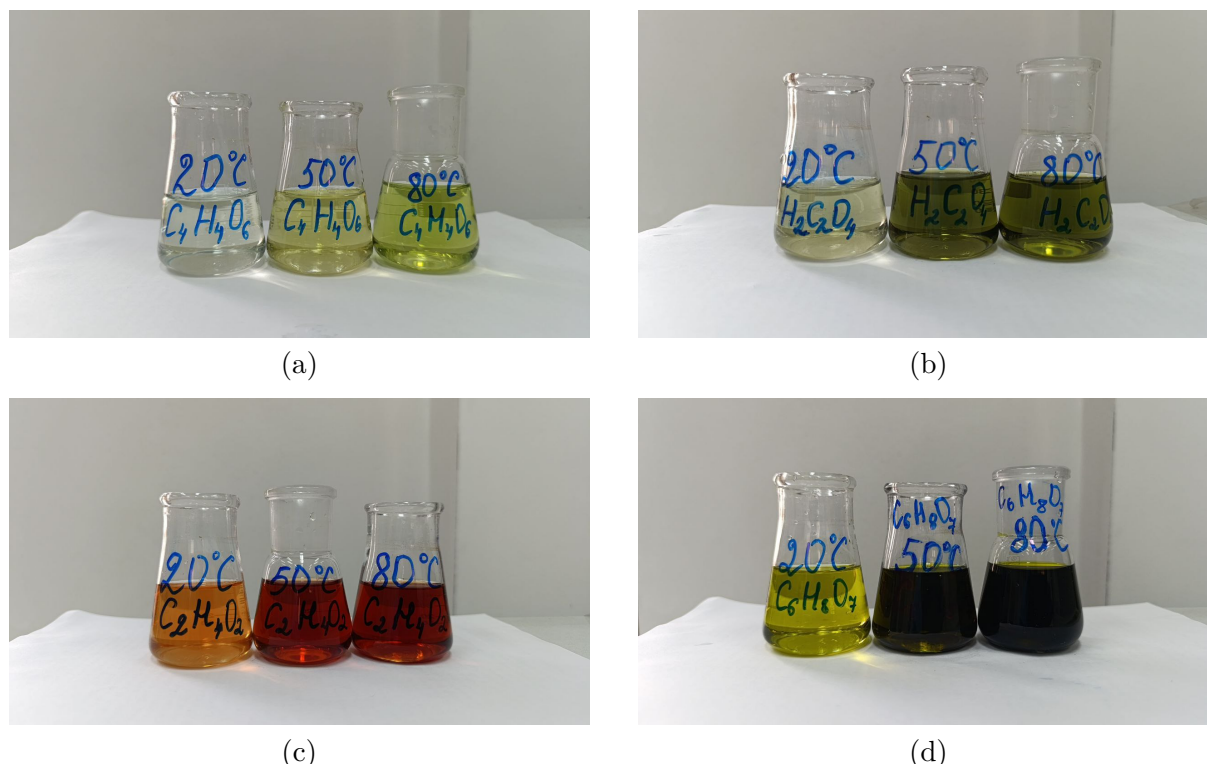


Figure 2 Visual appearance of pregnant leach solutions obtained using (a) tartaric, (b) oxalic, (c) acetic, and (d) citric acid at 20, 50, and 80 °C (left to right in each panel). The coloration generally becomes more intense at higher temperatures, particularly in the citric acid solutions

The visual appearance of the obtained leach liquors (Figure 2) further supports the differences observed in the efficiency of nickel extraction among the investigated organic acids. Significant variations in color intensity were observed depending on the acid type and leaching

temperature. The solutions obtained after citric acid leaching exhibited the darkest coloration, particularly at 50°C and 80°C, suggesting enhanced dissolution of metal-bearing phases and the formation of stable soluble metal–citrate complexes. Acetic acid solutions showed an intermediate reddish-brown color, whereas oxalic and tartaric acid solutions remained comparatively pale, reflecting their lower dissolution efficiency. The progressive increase in color intensity with temperature suggests that higher temperatures promote the transition-metal species from the solid phase into the solution. Although the solution color cannot be directly correlated with nickel concentration alone, it provides qualitative evidence of increased dissolution of iron- and nickel-containing phases. The pronounced darkening observed in the citric acid solutions is consistent with its superior leaching performance and strong complexation ability toward dissolved metal ions. This qualitative observation agrees well with the quantitative nickel extraction data presented in Figures 1, 3, and 4.

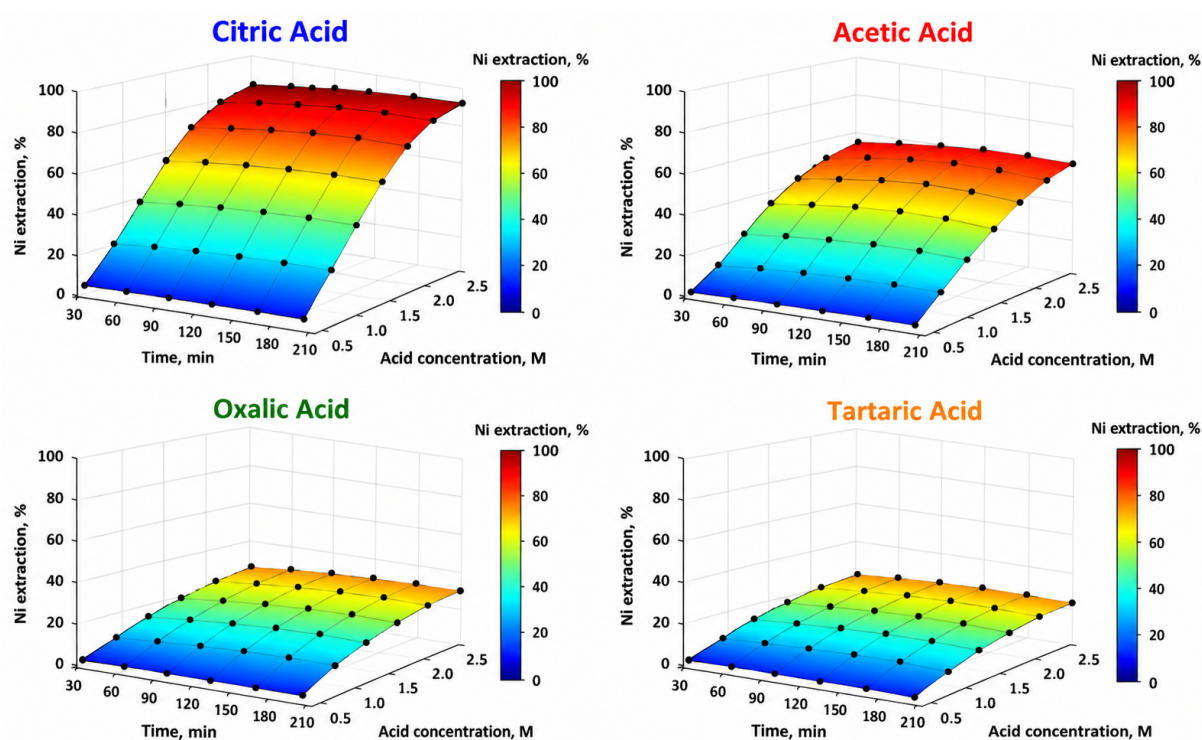


Figure 3 Surface plots showing the combined effects of acid concentration and leaching time on nickel extraction at 80°C using (a) citric acid, (b) acetic acid, (c) oxalic acid, and (d) tartaric acid

Figure 3 shows the combined effect of organic acid concentration and leaching time at 80°C. Nickel extraction increased rapidly during the initial leaching stage and then gradually approached a plateau. This behavior was particularly evident for the citric acid. At 2.0 M citric acid, nickel extraction increased from 33.52% after 30 min to 68.50% after 120 min and reached 78.82% after 180 min. Further extension of the leaching time to 210 min increased nickel extraction to 79.30%. A similar trend was observed for acetic acid, where extraction increased from 18.95% after 30 min to 57.72% after 180 min and then only marginally to 58.60% at 210 min. The same plateau tendency was observed for oxalic and tartaric acids, although at considerably lower extraction levels.

The limited increase in nickel extraction after 180 min indicates that the leaching process becomes progressively slower at longer reaction times. The occurrence of this plateau behavior suggests a gradual transition from an initially reaction-controlled regime to a diffusion-influenced dissolution stage. This behavior may be associated with the partial depletion of readily accessible nickel-bearing sites, accumulation of less reactive residual phases, and increased diffusion resistance through the altered surface layer formed during leaching. Therefore, 180 min was

selected as the optimal leaching duration. Extending the process to 210 min did not provide a meaningful improvement in nickel recovery: the additional increase was only 0.48, 0.88, 0.68, and 0.35 percentage points for citric, acetic, oxalic, and tartaric acids, respectively. From both kinetic and practical viewpoints, 180 min is a more reasonable processing time.

Figure 4 shows the combined effect of leaching time and temperature at an organic acid concentration of 2.0 M. Temperature had a pronounced positive effect on nickel extraction for all the studied acids. At 180 min, increasing the temperature from 20°C to 80°C enhanced nickel extraction from 28.64% to 78.82% for citric acid, from 16.83% to 57.72% for acetic acid, from 11.25% to 36.52% for oxalic acid, and from 7.94% to 24.95% for tartaric acid. The strong temperature dependence indicates that thermal activation facilitates both mineral decomposition and soluble nickel-organic complexes.

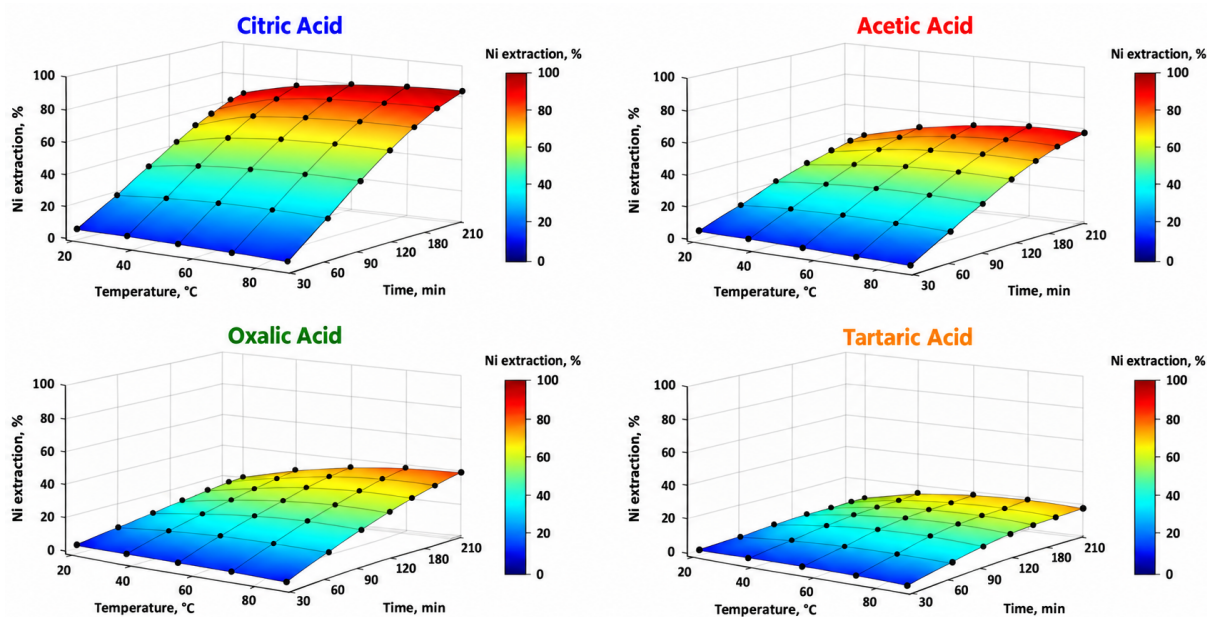


Figure 4 Surface plots showing the combined effects of temperature and leaching time on nickel extraction at an acid concentration of 2.0 M using (a) citric acid, (b) acetic acid, (c) oxalic acid, and (d) tartaric acid

The effect of temperature was most pronounced for citric and acetic acids, suggesting that these lixiviants are more effective in promoting nickel dissolution when sufficient thermal energy is provided. Increasing the temperature likely enhances proton mobility, accelerates the breakdown of nickel-bearing silicate or iron oxide-associated phases, increases diffusion rates in the liquid phase, and promotes complexation reactions between dissolved Ni^{2+} and organic ligands. Nevertheless, the extraction increase from 60°C to 80°C was lower than that observed from 20°C to 60°C, indicating a gradual approach toward a kinetic plateau at higher temperatures. Based on the obtained results, 80°C was selected as the most favorable temperature among the investigated range.

Overall, the experimental results identified 2.0 M citric acid, 80°C, and 180 min as practical leaching conditions. Under these conditions, nickel extraction reached 78.82%, compared with 57.72%, 36.52%, and 24.95% for acetic, oxalic, and tartaric acids, respectively. Although increasing the acid concentration to 2.5 M increased nickel extraction to 82.60%, the additional improvement was limited compared with the associated increase in reagent concentration. Therefore, 2.0 M citric acid, 80°C, and 180 min were selected as the practical operating conditions. The superior performance of citric acid results from the combined effects of proton-assisted mineral dissolution and stabilization of dissolved Ni^{2+} through multidentate citrate complexation. In contrast, the lower efficiencies of oxalic and tartaric acids may be associated with limited nickel

solubilization, sparingly soluble complex formation, and insufficient nickel-bearing matrix. Although the efficiency of nickel extraction is the primary indicator of leaching performance, the practical applicability of an organic lixiviant also depends on its selectivity toward the target metal. The extraction efficiencies of Ni, Fe, and Mg, together with the corresponding Ni/Fe and Ni/Mg selectivity ratios under the conditions providing the maximum nickel extraction, are summarized in Table 2.

As shown in Table 2, all investigated organic acids dissolved nickel considerably more readily than iron, demonstrating pronounced selectivity toward the iron-rich matrix. Citric acid provided the highest nickel extraction (82.6%) while dissolving only 4.3% of iron, resulting in the highest Ni/Fe selectivity ratio (19.21).

Acetic acid also showed high Ni/Fe selectivity (11.81), whereas oxalic acid exhibited substantially lower selectivity owing to its relatively higher iron dissolution (13.5%). Although tartaric acid extracted only 26.1% of nickel, its very low iron dissolution (2.1%) resulted in a relatively high Ni/Fe selectivity ratio (12.43). In contrast to iron, magnesium dissolution was considerably higher for all investigated acids, indicating that Mg-bearing phases are more susceptible to organic-acid attack than the iron-rich fraction. The Ni/Mg selectivity ratios remained close to unity (0.81–1.18), showing that magnesium dissolution accompanies nickel extraction to a much greater extent than iron dissolution. These results confirm that the investigated organic acids exhibit pronounced selectivity toward nickel over iron, but not toward magnesium. Under the optimum citric-acid conditions, iron dissolution was restricted to only 4.3%, demonstrating that efficient nickel extraction can be achieved while strongly suppressing the co-dissolution of iron. From a practical and metallurgical standpoint, this selective leaching behaviour is more significant than nickel recovery alone.

Table 2 Comparative extraction efficiencies and selectivity ratios for Ni, Fe, and Mg obtained using different organic acids at 2.5 M, 80°C, and 180 min

Organic acid	Ni extraction (%)	Fe extraction (%)	Mg extraction (%)	Ni/Fe selectivity ratio	Ni/Mg selectivity ratio
Citric	82.6	4.3	74.5	19.21	1.11
Acetic	61.8	5.2	52.3	11.81	1.18
Oxalic	39.2	13.5	48.6	2.90	0.81
Tartaric	26.1	2.1	22.4	12.43	1.17

The high Ni/Fe selectivity observed in the present study also distinguishes this process from most previously reported organic-acid leaching systems. Earlier investigations predominantly focused on maximizing nickel extraction, whereas the selective suppression of iron dissolution received comparatively less attention (Astuti et al., 2016; McDonald & Whittington, 2008). For example, (Kursunoglu & Kaya, 2015) demonstrated that sequential citric–oxalic acid leaching enhanced nickel dissolution from lateritic ore; however, the process was primarily evaluated in terms of nickel recovery rather than Ni/Fe selectivity. In contrast, the present process achieved high nickel recovery while maintaining iron dissolution remarkably low (2.1%–13.5%) across all investigated systems. Under the conditions providing the maximum nickel extraction with citric acid, iron dissolution was restricted to only 4.3%, demonstrating that efficient nickel extraction can be achieved while limiting the co-dissolution of iron. Hernández et al. (2007) investigated the percolation leaching of Cuban nickel tailings (0.34% Ni, 0.08% Co, 44.2% Fe) using tartaric and oxalic acids at ambient temperature.

The authors reported that a mixture of 0.15 M tartaric acid and 0.05 M oxalic acid extracted approximately 70% Ni and 80% Co after 5 days of leaching, while iron extraction remained limited to 30%. This Ni/Fe selectivity pattern is qualitatively consistent with the present findings, although the extraction efficiency achieved in the present study using citric acid (82.6% Ni) is substantially higher, likely due to the prior magnetic beneficiation step that enriched the nickel-

bearing fraction. Notably, Hernández et al. (2007) demonstrated that magnetic separation of the leaching residues yielded a magnetic product containing >50% Fe, confirming the potential for valorization of the solid residue—a strategy that aligns with the integrated flowsheet proposed in the present study for producing both a nickel-rich solution and a high-grade iron concentrate.

The limited iron dissolution is technologically important because excessive iron transfer into the leach solution complicates downstream purification and may promote the formation of iron-bearing precipitates during subsequent pH adjustment. Under citric acid leaching, only 4.3% of iron was dissolved, while 82.6% of nickel was extracted. This behavior confirms that nickel can be preferentially removed from the iron-rich magnetic fraction and reduces the downstream iron removal potential reagent demand.

3.2 Kinetic Analysis of the Leaching Process

To identify the rate-controlling mechanism of nickel dissolution, the leaching data obtained in citric, acetic, oxalic, and tartaric acid solutions were fitted using the Drozdov–Rotinyan, Prout–Tompkins, shrinking-core, and film-diffusion models. These models represent mixed reaction–diffusion control, progressive exposure of reactive sites, surface-reaction control, and external mass-transfer control. Therefore, their comparative application is more informative than the use of a single model for a heterogeneous oxide–silicate material (Baigenzhenov et al., 2024). Nickel dissolution in organic acid media may involve several simultaneous steps. Proton attack promotes metal–oxygen bonds in nickel-bearing domains, while carboxylate ligands stabilize the released Ni^{2+} ions in solution. The overall rate may also be affected by reactant and dissolved products across the liquid boundary layer and through progressively altered regions of the particles. Accordingly, the model selection was based on both the determination coefficients and the consistency of the resulting kinetic interpretation with the experimentally observed extraction and selectivity trends.

The Drozdov–Rotinyan model provided the highest and most consistent agreement with the experimental data (Figure 5a). The corresponding R^2 values were 0.9818, 0.9737, 0.9812, and 0.9979 for citric, acetic, oxalic, and tartaric acids, respectively, giving an average R^2 of 0.9837 (Table 3). Its applicability across all four acid systems indicates that a coupled reaction–diffusion regime rather than by a single elementary rate-controlling step. The initial stage is associated with relatively rapid attack on accessible nickel-bearing regions, whereas the subsequent decrease in extraction rate may reflect the depletion of readily reactive sites and increased transport resistance within the altered particle regions. Nevertheless, the different extraction efficiencies of the organic acids demonstrate that ligand chemistry remains important. Citric acid gave the highest nickel extraction because its proton-donating and multidentate complexing properties favor both mineral attack and dissolved Ni^{2+} . Acetic acid exhibited lower complexing capacity, whereas the comparatively poor performance of oxalic acid may be associated with the formation of sparingly soluble nickel oxalate species. Hussaini et al. (2024) similarly attributed the reduced efficiency of oxalic acid during the dissolution of a mixed nickel–cobalt hydroxide precipitate to the precipitation of metal oxalates. Their reported activation energy for nickel dissolution in citric acid was only 3.1 kJ mol^{-1} , considerably lower than the value obtained in the present study, reflecting the greater reactivity of a hydroxide precipitate relative to the iron-rich oxide–silicate material investigated here. The Prout–Tompkins model also produced relatively high correlations, with R^2 values ranging from 0.9104 to 0.9924 and an average value of 0.9561 (Figure 5b and Table 3).

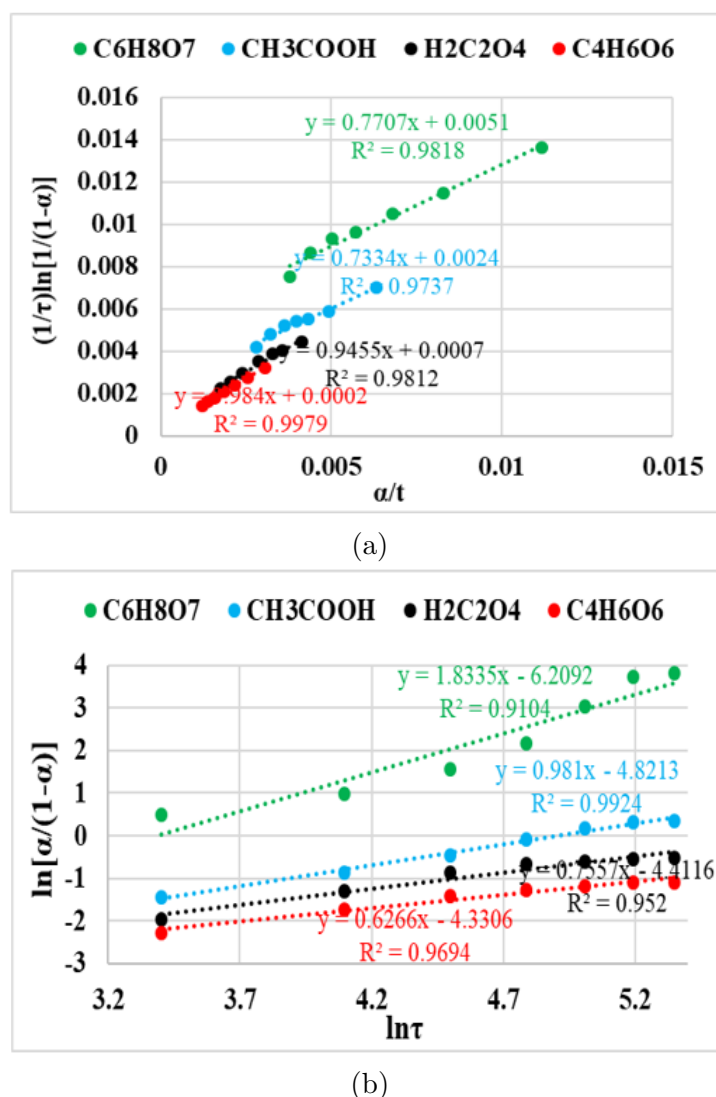


Figure 5 Kinetic fitting of nickel leaching data in different organic acid solutions using (a) the Drozdov–Rotinyan and (b) the Prout–Tompkins models

Table 3 Comparison of correlation coefficients (R^2) for different kinetic models describing nickel leaching in organic acid solutions

Model	Citric	Acetic	Oxalic	Tartaric	Average R^2
Drozdov–Rotinyan	0.9818	0.9737	0.9812	0.9979	0.9837
Prout–Tompkins	0.9104	0.9924	0.9520	0.9694	0.9561
Shrinking core	0.9024	0.9392	0.8325	0.8810	0.8888
Film diffusion	0.9661	0.9664	0.8557	0.9660	0.9386

Its applicability suggests that the progressive exposure of new reactive sites may contribute to nickel dissolution. However, its performance among the four acids indicates that surface activation or structural disintegration is not the sole controlling mechanism. Yamini et al. (2025) reported that citric–ascorbic acid leaching of nickel furnace slag involved proton-promoted matrix dissolution and reductive conversion of Fe^{3+} to Fe^{2+} . Extensive dissolution of the iron-bearing matrix enhanced nickel and cobalt release. In contrast, the present results show that substantial nickel extraction can be achieved with limited iron dissolution, indicating that complete destruction of the iron-rich matrix is not required. The shrinking-core model had the lowest overall agreement with the experimental data (Figure 6a). The R^2 values ranged from 0.8325 to 0.9392, with an average of 0.8888. This relatively poor fit indicates that nickel dissolution

does not occur through a uniform inward-moving reaction front. Such behavior is consistent with a heterogeneous distribution of nickel-bearing regions within the magnetic fraction, where dissolution may occur simultaneously at multiple accessible sites rather than exclusively at a sharply defined interface.

The film diffusion model produced R^2 values of 0.9661, 0.9664, 0.8557, and 0.9660 for citric, acetic, oxalic, and tartaric acids, respectively, with an average value of 0.9386 (Figure 6b and Table 3). These relatively high correlations indicate that external mass transfer contributes to the observed kinetics. However, the poorer overall agreement compared with the Drozdov–Rotinyan model shows that boundary-layer diffusion is not the sole rate-controlling step. The particularly low fit for oxalic acid may reflect additional complexation–precipitation phenomena not represented by a simple diffusion equation.

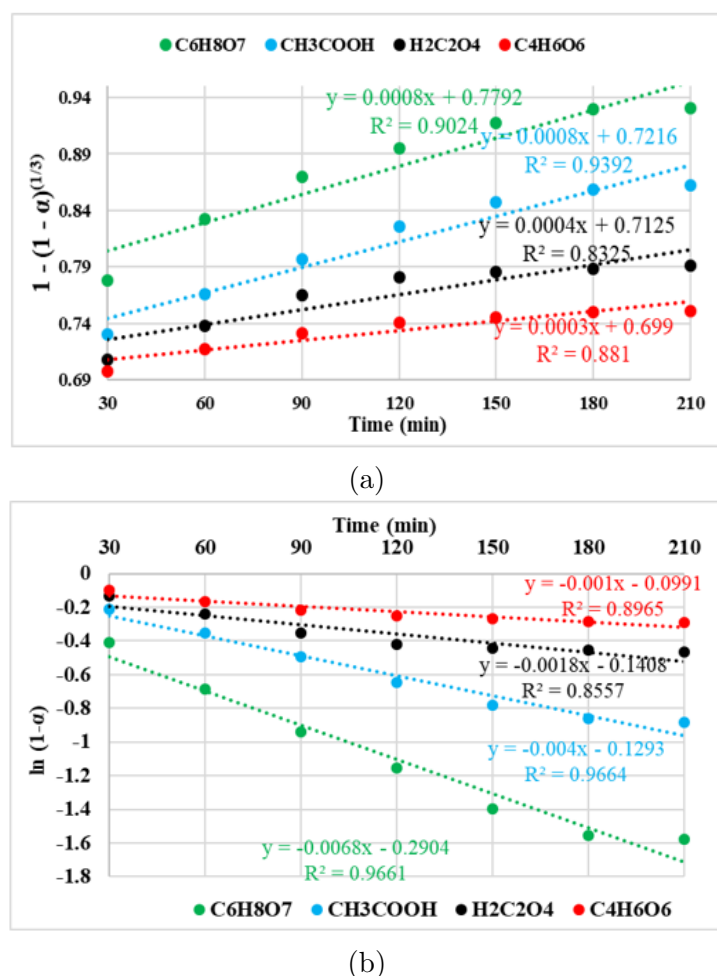


Figure 6 Kinetic fitting of nickel leaching data in different organic acid solutions using (a) shrinking-core and (b) film-diffusion models

The average R^2 values decreased in the following order: Drozdov–Rotinyan (0.9837) > Prout–Tompkins (0.9561) > film diffusion (0.9386) > shrinking core (0.8888). The Drozdov–Rotinyan equation is the most appropriate empirical description of nickel dissolution under the investigated conditions. However, a high determination coefficient alone does not constitute definitive proof of a particular elementary mechanism. Rather, the comparison indicates that the experimental behavior is most consistent with a mixed regime involving interfacial dissolution and diffusion-related retardation. The strong dependence of nickel extraction on the type and concentration of organic acid. Hseu et al. (2017) reported that nickel release from serpentine soils increased with decreasing pH and that organic acids produced a stronger dissolution response than inorganic acids because of ligand-promoted reactions. They also observed the effects of ionic strength on nickel release, indicating that solution chemistry and mass-transfer phenomena can

jointly affect the dissolution rate. These observations are consistent with the mixed reaction–diffusion behavior identified in this study. Senanayake et al. (2011) similarly demonstrated that the dissolution of nickel- and iron-bearing oxide materials may shift from proton-assisted surface reaction during the initial stage to diffusion through a progressively altered porous layer at longer reaction times. Thus, the kinetic regime can evolve during leaching and should not necessarily be represented by a single idealized limiting step.

The apparent activation energy was determined for citric acid because it provided the highest nickel extraction. The Arrhenius relationship constructed using the Drozdov–Rotinyan model as the apparent rate constants k was linear over the investigated temperature range, with $R^2 = 0.9794$ (Figure 7). The calculated apparent activation energy was $21.03 \text{ kJ mol}^{-1}$. This value lies near the lower boundary of the range commonly associated with mixed kinetic control and is close to values generally attributed to diffusion-influenced dissolution. Therefore, diffusion-related resistance makes a substantial contribution, although the interfacial reaction cannot be excluded.

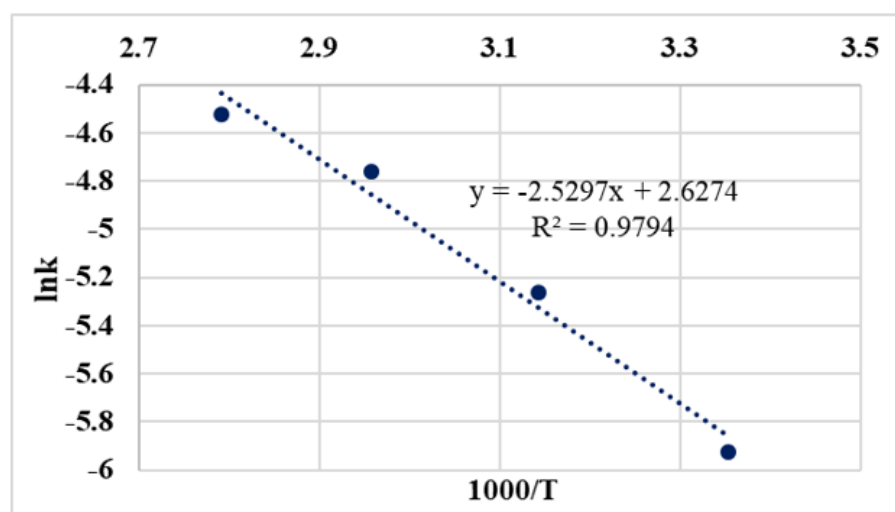


Figure 7 Arrhenius relationship between $\ln k$ and $1000/T$ for nickel leaching in citric acid

The obtained activation energy is within the range reported for diffusion-assisted leaching of nickel-bearing oxide and silicate materials (Mamyrbayeva et al., 2024). Astuti et al. (2016) reported an apparent activation energy of approximately 12 kJ mol^{-1} for citric acid leaching of saprolite, whereas Hussaini et al. (2024) obtained 3.1 kJ mol^{-1} for a more readily soluble mixed hydroxide precipitate. The higher value obtained in this study reflects the more refractory nature of the iron-rich oxide–silicate feedstock. Kim et al. (2022) reported an apparent activation energy of 19.7 kJ mol^{-1} for reductive dissolution of synthetic magnetite in oxalic acid, which is close to the value of $21.03 \text{ kJ mol}^{-1}$ obtained here. Lee et al. (2006) found that the activation energy for the oxalic-acid dissolution of iron oxides varied substantially with the mineral composition and temperature, ranging from diffusion-controlled values to considerably higher values associated with the chemical reaction. These comparisons demonstrate that the apparent activation energy is strongly dependent on mineralogy and reaction conditions, but they also support a significant transport contribution in this system. From an engineering perspective, the mixed reaction–diffusion regime indicates that process intensification should consider both interfacial reaction and mass transfer. Increasing the temperature or acid concentration may accelerate the mineral attack and complex formation, whereas decreasing the particle size and improving the solid–liquid contact may reduce the diffusion limitations. Nevertheless, the present kinetic results were obtained in batch experiments at a laboratory scale; therefore, the individual effects of particle size, pulp density, and agitation should be quantified before selecting a reactor configuration or extrapolating the rate parameters to a larger-scale operation.

Overall, the consistently superior fit of the Drozdov–Rotinyan model and the apparent activation energy of $21.03 \text{ kJ mol}^{-1}$ indicate mixed reaction–diffusion control. The process involves

proton-assisted attack on nickel-bearing regions, stabilization of dissolved Ni^{2+} by organic ligands, and diffusion-related retardation as leaching progresses. This interpretation explains the rapid initial extraction followed by a gradual approach to a plateau, which is consistent with the limited dissolution of iron. Consequently, citric acid provides the most effective combination of nickel extraction, Ni/Fe selectivity, and kinetic performance among the investigated lixiviants.

4. Conclusions

Organic acids enabled selective nickel leaching from the magnetic fraction of chrysotile asbestos beneficiation waste while limiting iron dissolution. Citric acid provided the highest nickel extraction of 82.6%, followed by acetic (61.8%), oxalic (39.2%), and tartaric (26.1%) acids. Iron extraction remained within 2.1%–13.5%, and a maximum Ni/Fe selectivity ratio of 19.21 was achieved using citric acid. Increasing the acid concentration, temperature, and leaching time enhanced nickel extraction, although the improvement was limited above 2.0 M and beyond 180 min. The Drozdov–Rotinyan model provided the best overall kinetic fit, with an average R^2 of 0.9837. The apparent activation energy of 21.03 kJ mol^{−1} indicated mixed reaction–diffusion control with a substantial contribution from diffusion-related resistance. The kinetic behavior is consistent with proton-assisted dissolution of nickel-bearing regions, stabilization of dissolved Ni^{2+} by organic ligands, and increasing transport resistance as leaching progresses. Overall, citric acid offered the most favorable combination of nickel extraction and Ni/Fe selectivity. Further studies should address acid regeneration, nickel recovery from the pregnant solution, residue characterization, and validation at higher pulp density and larger scale.

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Author Contributions

Rustam Sharipov: Investigation, data collection, formal analysis, writing – original draft, funding acquisition. **Galymzhan Maldybayev:** Conceptualization, methodology, supervision, writing – review and editing. **Assel Dagubayeva:** Investigation, data analysis, visualization, writing – review and editing. **Mohd Dzul Hakim Wirzal:** Methodology, formal analysis, writing – review and editing. **Setyo Budi Kurniawan:** Data analysis, validation, writing – review and editing. **Zaher Mundher Yaseen:** Formal analysis, validation, writing – review and editing. **Omirsarik Baigenzhenov:** Conceptualization, methodology, supervision, writing – review and editing. **Ahmad Hosseini-Bandegharai:** Validation, writing – review and editing. All authors reviewed and approved the final manuscript.

Conflict of Interest

No potential conflict of interest was reported by the authors.

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