

Dissolution behavior of Caldag lateritic nickel ore subjected to a sequential organic acid leaching method

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Abstract: The dissolution behavior of Caldag lateritic nickel ore subjected to a sequential organic acid leaching method was investigated. The effects of the type of organic acid, acid concentration, leaching time, and leaching temperature on the lateritic nickel ore were examined. Organic acids were used individually prior to sequential leaching. Citric acid was more effective than the other two acids for the selective leaching of nickel and cobalt. An increase in the citric acid concentration negligibly affected the dissolution of the metals, whereas temperature exhibited a strong beneficial effect. Oxalic acid was determined to be the most appropriate organic acid for the second leaching step. After 8 h (4 h + 4 h) of leaching with organic acids (0.5 M citric + 0.5 M oxalic) in sequence at 90°C, 89.63% Ni, 82.89% Co, and 69.63% Fe were leached from the lateritic nickel ore. A sequential citric + oxalic acid leaching method could represent a viable alternative for the dissolution of metals from lateritic nickel ore.

Keywords: laterite; leaching; citric acid; oxalic acid; dissolution

1. Introduction

Lateritic nickel ore is an important natural mineral resource that contains vital amounts of valuable metals such as nickel and cobalt. These metals are extensively utilized in modern industrial and metallurgical applications. The depletion of high-grade ore deposits and an increase in demand from metallurgists for strategic applications have encouraged researchers to focus on low-grade lateritic nickel ore and nickel-containing waste as new sources of nickel and cobalt.

Conventional techniques such as pyrometallurgical and hydrometallurgical methods have been applied to the recovery of nickel from lateritic nickel ores, and less attention has been paid to the recovery of cobalt than to that of nickel. In fact, the price of cobalt has significantly increased over the past several years. Recently, new technologies have been proposed for nickel and cobalt extraction from lateritic nickel ores, such as sulfate atmospheric leaching, hydrochloric acid leaching processes, bioleaching of oxide ores,

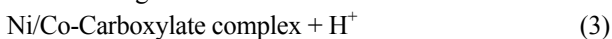
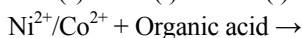
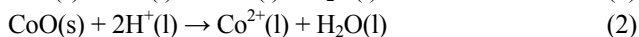
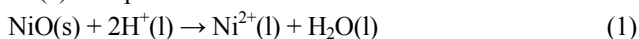
and direct nickel processing [1].

As an alternative to pyrometallurgical and hydrometallurgical processes, bio-hydrometallurgy is potentially an economical and environmentally friendly process. The bioleaching of lateritic nickel ore appears to offer a new technology with a lower cost, lower energy demand, and safer operation compared with conventional hydrometallurgical techniques. In this process, the dissolution of nickel and cobalt from lateritic nickel ore occurs with a range of organic acids/metabolites produced by different heterotrophic bacteria. In recent years, substantial advancements have been realized in the field of nickel extraction from low-grade lateritic nickel ores using different heterotrophic fungal species that produce organic acids such as oxalic, citric, and acetic acids, which facilitate the complexation of metals [2–8]. Acidophilic, iron-oxidizing, and/or sulfur-oxidizing chemolithotrophic microorganisms have been intensively studied [9]. Current bio-oxidation and bioleaching investigations and operations utilizing these organisms are focused on the processing of gold and copper [10–11]. The use of a mixed culture of *Acidithiobacillus ferrooxidans*,

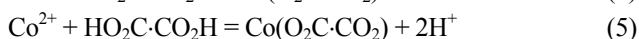
Acidithiobacillus caldus, and *Leptosprilium ferrooxidans* for the bacterial leaching of lateritic nickel ore has been studied [12]. However, bio-hydrometallurgical processing has been less successful with limonite ores for commercial applications because of their high-grade iron content in the form of goethite. Extensive effort has been devoted to dissolving iron oxides using organic acids [13–14].

Different research groups have investigated the leaching of lateritic nickel ores with organic acids and have implemented optimization studies using similar organic acids [15–18]. Several technical problems (i.e., excessive leaching time and low metal recovery) specific to the use of pure organic acids must be resolved before industrial applications can be implemented; however, selective leaching with organic acids might be an effective method to extract valuable metals from laterite ores [16]. Oxalic, citric, and acetic acids are organic reducing agents that reduce some high-valence metal ions, e.g., Mn(IV) to Mn(II) [19]. The reactions between nickel/cobalt oxides and an organic acid are represented as follows:

(a) Complexation/chelation.



(b) Precipitation.



The leaching efficiency of metals from a lateritic nickel ore is dependent on several factors, including the type and concentration of the organic acids, leaching time, and temperature. Hence, optimization of these operating parameters is always required so that the best leaching efficiency can be achieved. In this study, instead of using a metabolic organic acid produced by microbes, analytically pure organic acids were used. The main objective of this study was to investigate the dissolution behavior of Caldag lateritic nickel ore when subjected to a sequential organic acid leaching method.

2. Experimental

2.1. Sample preparation

The lateritic nickel ore was provided by the Caldag pilot plant located in the Western part of Turkey. The raw lateritic nickel ore was crushed and ground with a jaw crusher and ball mill, respectively. The ground ore was subjected to coning and quartering and classified into five different size fractions using standard sieves. The ore fractions were

weighed and stored in plastic bags for further studies. All leaching experiments were conducted using the 100% –0.053-mm particle size fraction.

2.2. Material characterization

Initially, the specific gravity of a ground ore sample was measured with a Quantachrome multipycnometer (Quantachrome Instruments, Florida, USA) using helium gas. The surface area was determined using the Blaine test method. The chemical composition of the ground ore sample was analyzed using X-ray fluorescence (XRF) spectrometry (Philips PW-2404). The percentages of nickel, cobalt, and iron in the ore were analyzed by atomic absorption spectroscopy (AAS) (Thermo Scientific 3300). The mineralogical composition of the lateritic nickel ore was determined by X-ray diffraction (XRD) on a Bruker DX8 Advance equipped with a Cu-K α ($\lambda = 0.154$ nm) radiation source and calibrated with a silicon standard for alignment of the $2\theta = 10^\circ$ – 70° radiation generated at 30 mA and 40 kV. Mineralogical identification was performed using the International Centre for the Diffraction Data ICDD PDF-4/Minerals software. A surface morphological study was conducted by scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) (Jeol JSM-5600 LV). The pH value and oxidation reduction potential (ORP) of the leach slurry were measured using a dual-input Hach 40d pH meter. All chemicals used in this study were of analytical grade.

2.3. Experimental procedure

The first-step leaching experiments were conducted in a 250-mL Pyrex water-jacketed reactor equipped with an overhead mechanical stirrer. The reactor was equipped with a glass reflux condenser to prevent evaporation. A circulating water bath was electrically heated and adjusted to the desired temperature using a thermostat. Then 100 mL of organic acid and the required amount of ore sample were transferred into the reactor, and the leach slurry was properly stirred. At the end of each leaching experiment, the mechanical stirrer and temperature-controlled water bath were switched off; the pH value and the ORP of the slurry were then measured. Thereafter, the slurry was filtered using Whatman 589/2 filter paper and washed with deionized water several times. In a volumetric flask, 500 mL of supernatant was collected and subjected to AAS analysis to determine its nickel, cobalt, and iron concentrations. Subsequently, the solid residue was dried in an oven at 100°C for 2 h; the dried residue was subsequently stored in a desiccator. In all leaching experiments, the solid-to-liquid ratio and

stirring speed were maintained at 100 g/L and 500 r/min, respectively.

In the second-step leaching experiments, the stored solid residues from the first leaching step were weighed and leached with a different organic acid using the same procedure and setup previously described. At the end of the second leaching step, the slurry was filtered and the residue was washed with deionized water and placed in an oven for 2 h at 100°C. The dried residue was weighed and digested in hot aqua regia until the residue turned white. The supernatant and digested residues were analyzed by AAS to enable a metallurgical mass balance calculation. Most of the leaching experiments were performed three times to assess reproducibility. The leaching efficiencies of the metals were calculated according to the following formula:

$$E = (C_f/C_i) \times 100\% \quad (6)$$

where E is the leaching efficiency, C_f is the metal content in the leach liquor (wt%), and C_i is the original metal content in the ore (wt%).

3. Results and discussion

3.1. Materials characterization

The specific gravity of the 100% –0.053-mm sample was 2.805 g/cm³, and its surface area was 5158 cm²/g. XRF analysis was performed to determine all of the metal constituents present in the lateritic nickel ore as shown in Table 1. 1 g of ore was transferred to 40 mL of aqua regia (3:1 (v/v), HCl:HNO₃) in a 250-mL beaker covered with a watch glass, and the mixture was heated on a hot plate. After the digestion of the ore, the slurry was filtered using a Büchner funnel. The white residue was washed with deionized water several times. The solution was kept in a clean volumetric flask that had been rinsed with deionized water. The solution volume was then increased to 500 mL by adding deionized water. This solution was subsequently analyzed by AAS to determine its nickel, cobalt, and iron concentrations; the results are summarized in Table 2. Fig. 1 depicts the XRD pattern of the lateritic nickel ore. The XRD analysis revealed the presence of quartz, kaolinite, and calcite as the major phases. Minor mineral peaks were attributed to goethite, hematite, albite, lizardite, asbolane (nickel- and cobalt-rich phases), and serpentine. Figs. 2 and 3 depict the results of an SEM morphological investigation of the lateritic nickel ore. SEM–EDX examination revealed that the hematite/goethite-like mineral particles contained nickel and cobalt, as shown in Table 3. A small amount of the nickel and cobalt were associated with clay-like mineral particles that are the main silicate-bearing phase, as shown in Table 4.

The hematite/goethite-like mineral particles contained approximately 80wt% Fe with 4.2wt% Ni and 0.62wt% Co. The clay-like mineral particles consisted of 48wt% Si, 0.35wt% Mg, 0.31wt% Al, 0.17wt% Ca, 0.29wt% Ni, and 0.06wt% Co. These XRD and SEM–EDX results are consistent with the findings of previous researchers [20–21].

Table 1. Chemical composition of the lateritic nickel ore, as determined by XRF

Mineral	Content / wt%
MgO	4.5860
Al ₂ O ₃	2.5386
SiO ₂	45.1178
SO ₃	0.1035
K ₂ O	0.3030
CaO	3.4110
TiO ₂	0.0930
Cr ₂ O ₃	0.8610
MnO	0.3748
Fe ₂ O ₃	31.6869
Co ₂ O ₃	0.0902
NiO	1.4482
LOI*	9.3860

Note: *Loss on ignition.

Table 2. Elemental composition of the lateritic nickel ore, as determined by AAS

Sample No.	Ni content / wt%	Co content / wt%	Fe content / wt%
1	1.0805	0.0490	22.161
2	1.0794	0.0494	21.032
3	1.0840	0.0496	22.556
Average	1.0813	0.0493	21.916

3.2. Effects of acid type and time on dissolution

Figs. 4(a)–4(c) describe the leaching efficiencies of nickel, cobalt, and iron for different organic acids at 1, 2, 4, and 6 h and at a concentration of 0.5 M. A constant leaching temperature of 90°C was maintained. Fig. 4(a) demonstrates that the iron, nickel, and cobalt leaching efficiencies increased concomitantly to more than 40% in the first 1 h and that the nickel and cobalt leaching efficiencies subsequently decreased to 20% as the leaching time increased. This result might be because of the adsorption of the nickel cations by colloidal silica and the precipitation of nickel and cobalt as oxalates [22]. However, the iron leaching efficiency slightly increased, to 51.25% within 2 h, and then reached a plateau. These results confirm that oxalic acid exhibited a consider-

able reverse selectivity for iron over nickel and cobalt.

In contrast, citric acid exhibited a strong beneficial effect on the cobalt leaching efficiency (Fig. 4(b)). The cobalt leaching efficiency increased to 60.21% within 1 h and then

remained relatively constant. The nickel and iron leaching efficiencies gradually increased as the leaching time increased. At 6 h, leaching efficiencies of 48.19% for Ni and 20.80% for Fe were achieved.

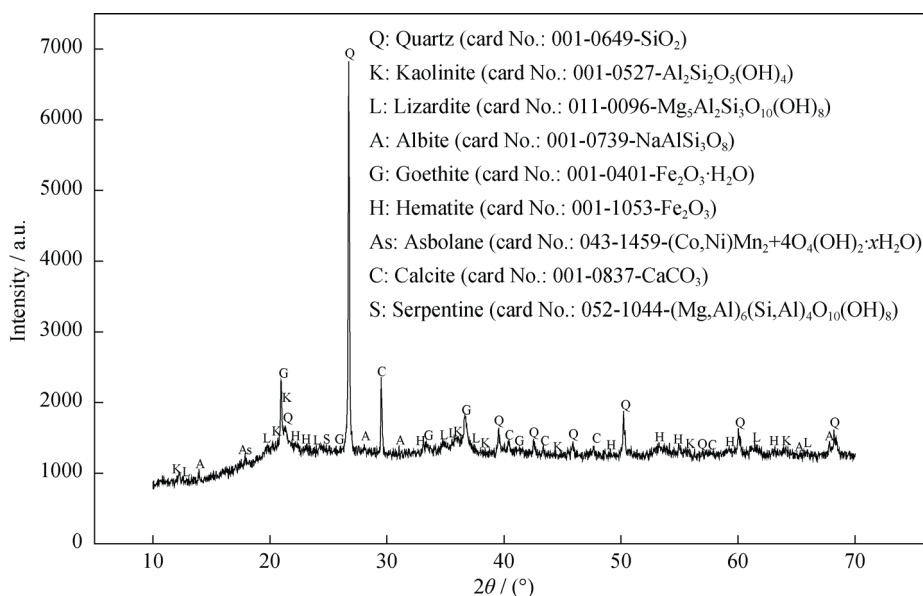


Fig. 1. XRD pattern of the lateritic nickel ore.

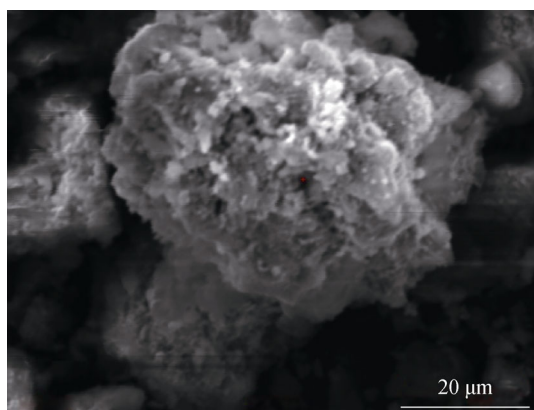


Fig. 2. SEM image of the hematite/goethite-like mineral particles.

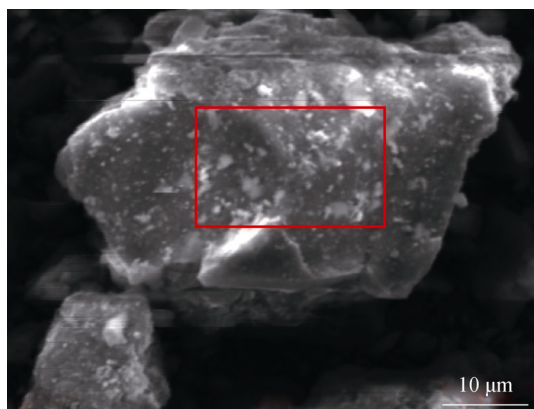


Fig. 3. SEM image of the clay-like mineral particles.

Table 3. EDX elemental analysis results for the hematite/goethite-like mineral particles

Element	Line	Intensity / a.u.	Content / wt%
O	Ka	45.75	2.912
Mg	Ka	11.46	0.555
Al	Ka	19.01	0.711
Si	Ka	292.61	8.814
Ca	Ka	48.89	1.078
Fe	Ka	1885.32	80.920
Co	Ka	12.23	0.619
Ni	Ka	66.31	4.272

Table 4. EDX elemental analysis results for the clay-like mineral particles

Element	Line	Intensity / a.u.	Content / wt%
O	Ka	786.85	45.033
Mg	Ka	21.51	0.354
Al	Ka	22.38	0.314
Si	Ka	3734.93	48.037
Ca	Ka	9.93	0.170
Fe	Ka	58.31	1.813
Co	Ka	1.65	0.060
Ni	Ka	7.04	0.293

Acetic acid exhibited very low (<15%) leaching efficiencies for the metals (Fig. 4(c)) because of the acetic acid dis-

sociation constant ($pK_a = 4.76$). The pK_a values of the organic acids tested decrease in the order citric acid > acetic acid > oxalic acid. The pK_a values of the three acids tested indicate that oxalic acid is a stronger acid than the other two acids [17].

Figs. 5(a)–5(c) display the pH value and ORP changes of the leach slurry as a function of time. These figures indicate that the pH value of the leach slurry with oxalic acid was

lower than that of the two other organic acids because of the oxalic acid dissociation constant ($pK_{a1} = 1.27$; $pK_{a2} = 4.28$), which is the lowest among the dissociation constants of the three organic acids tested. Additionally, the pH value of the slurry increased with increasing leaching time, whereas the ORP decreased, implying that the ionic activities were lower at higher pH levels. These lower ionic activities were attributed to the lower recoveries at these pH levels.

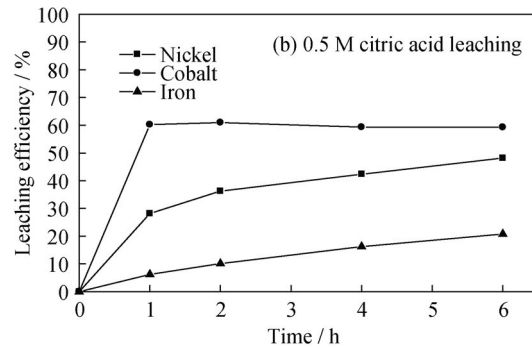
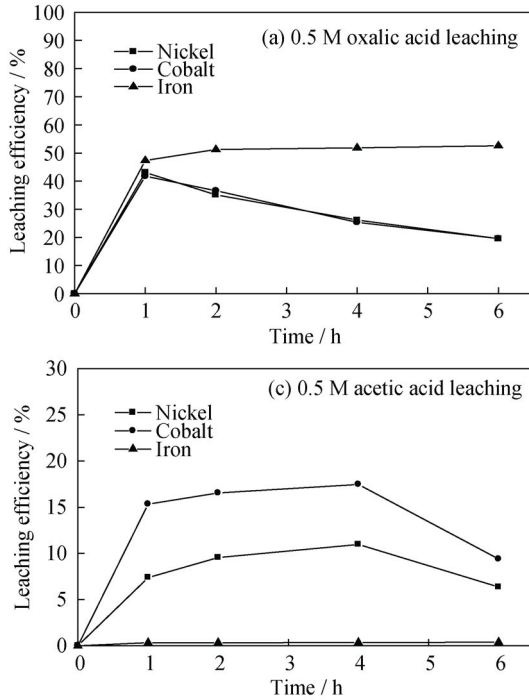


Fig. 4. Effects of organic acid type and leaching time on dissolution: (a) 0.5 M oxalic acid leaching; (b) 0.5 M citric acid leaching; (c) 0.5 M acetic acid leaching.

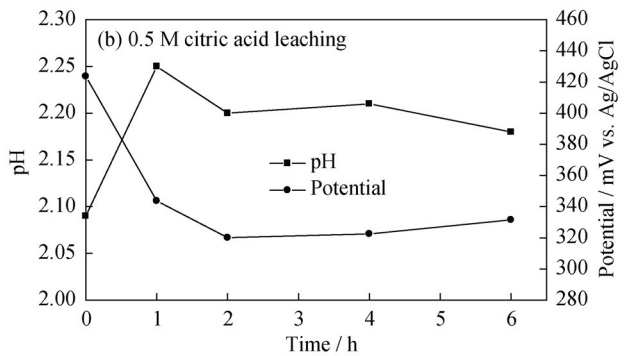
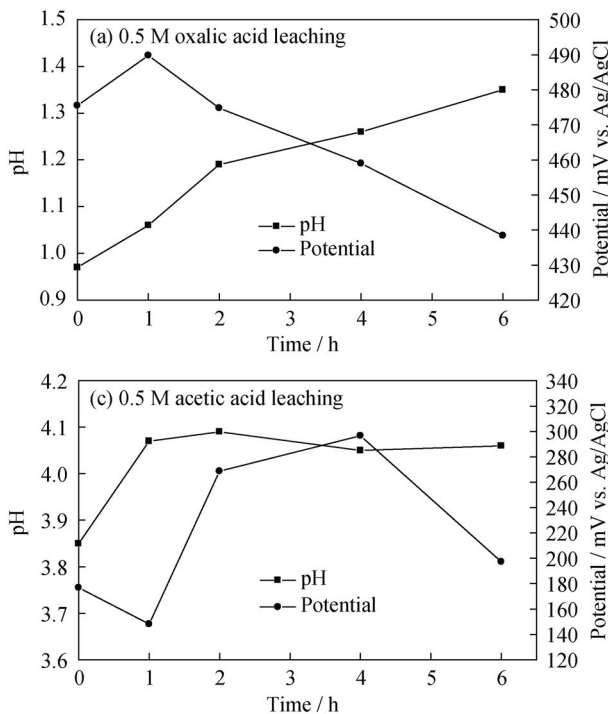


Fig. 5. Effects of pH value and ORP changes on dissolution: (a) 0.5 M oxalic acid leaching; (b) 0.5 M citric acid leaching; (c) 0.5 M acetic acid leaching.

Citric acid appeared to be the most appropriate organic acid for the extraction of nickel and cobalt from the lateritic nickel ore. Briefly, the leaching efficiencies of nickel and cobalt decreased in the following order: citric > oxalic > acetic acid. Therefore, the leaching efficiencies of the metals could be enhanced by increasing the citric acid concentration.

3.3. Effect of leaching temperature on dissolution

Fig. 6 illustrates the leaching efficiencies of nickel, cobalt, and iron in 0.5 M citric acid at different leaching temperatures for 1 h. The figure indicates that the leaching efficiency of nickel, cobalt, and iron increased with increasing temperature. Between a leaching temperature of 50 and 90°C, the nickel and cobalt leaching efficiencies significantly increased. More than 60% Co and 25% Ni were achieved at 90°C. Studies have shown that the dissolution of lateritic nickel ore is significantly affected by an increase in the leaching temperature under atmospheric conditions [23–25]. Lower energy consumption makes leaching at ambient temperature and atmospheric pressure more practical. However, citric acid leaching of lateritic nickel ores at atmospheric pressure requires a high temperature for the dissolution of metals.

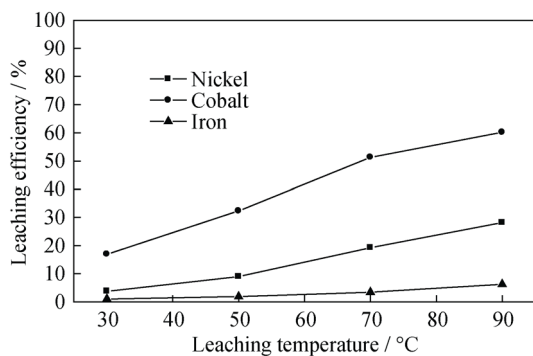


Fig. 6. Effect of leaching temperature on dissolution: 0.5 M citric acid, 1 h leaching time, and 100 g/L solid-to-liquid ratio.

3.4. Effect of the citric acid concentration on dissolution

Fig. 7 shows the effect of the citric acid concentration on the nickel, cobalt, and iron leaching efficiencies at 90°C for 1 h. The figure indicates that the leaching efficiencies of nickel, cobalt, and iron slightly increased with increasing leaching agent above a citric acid concentration of 0.5 M. At a citric acid concentration of 1.25 M, leaching efficiencies of 70.36% for Co and 34.02% for Ni were achieved. The selectivity of nickel and cobalt for iron remained almost constant when the concentration of the leaching agent was increased. This result suggests that the leaching of nickel

and cobalt cannot readily be made more efficient solely by increasing the concentration of citric acid.

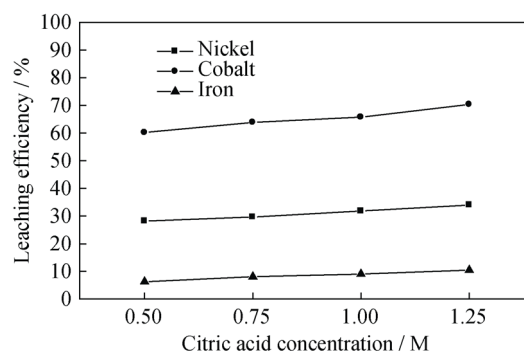


Fig. 7. Effect of citric acid concentration on dissolution: 1 h leaching time, 90°C temperature, and 100 g/L solid-to-liquid ratio.

3.5. Effect of oxalic acid addition on dissolution

To enhance the leaching efficiencies of the metals, the addition of oxalic acid was tested. Leaching tests were conducted at 90°C for 1 h at a citric acid concentration of 1.25 M. The amount of oxalic acid added ranged from 0.2 to 3.0 g. Fig. 8 shows the effect of the addition of oxalic acid on the dissolution of the metals. The figure reveals that the addition of oxalic acid significantly decreased the leaching efficiency of both nickel and cobalt by approximately 20% in the leach solution. This decrease could possibly be due to the precipitation of nickel and cobalt in oxalate form. However, the leaching efficiency of iron slightly increased from 10% to 25% at 3.0 g of added oxalic acid. Therefore, the addition of oxalic acid during the dissolution of lateritic nickel ore in a citric acid solution was not beneficial for nickel extraction.

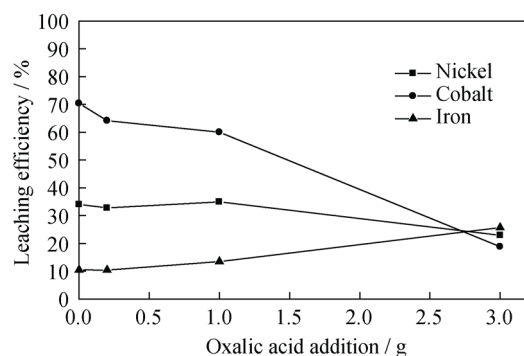


Fig. 8. Effect of oxalic acid addition on dissolution: 1.25 M citric acid, 1 h leaching time, 90°C temperature, and 100 g/L solid-to-liquid ratio.

3.6. Effects of second-step leaching on dissolution

After the optimal parameters for the first organic acid leaching step were determined, second-step leaching ex-

periments were performed to enhance the total leaching efficiencies of the metals. In the second leaching step, the residues from the first leaching step were used as the starting material. The leaching experiments were conducted at 90°C using 0.5 M oxalic and citric acids in sequence. Figs. 9(a) and 9(b) show the effects of the second leaching step on dissolution. These figures reveal that oxalic acid was more effective than citric acid in the second step. The iron leaching efficiency significantly increased with increasing leaching time. The nickel leaching efficiency was positively affected by using oxalic acid for up to 4 h in the second step. This improvement could be due to the destruction of Si–O, Al–O, and Al–Si–O bonds by citric acid during the first leaching step [14]. The leaching efficiencies of metals when

sequential (oxalic + citric) acid leaching was employed were very low (Fig. 9(b)). This result might be due to the insolubility of nickel and cobalt oxalate precipitates in citric acid solution.

Fig. 10 exhibits the effects of sequential leaching on dissolution. The leaching with two organic acids in sequence (i.e., citric + oxalic) clearly increased the total leaching efficiency of nickel, cobalt, and iron from the lateritic nickel ore. However, selectivity among the metals decreased. After 8 h (4 h + 4 h) of leaching with organic acids (0.5 M citric + 0.5 M oxalic) in sequence at 90°C, 89.63% Ni, 82.89% Co, and 69.63% Fe were leached from the lateritic nickel ore. A summary of the experimental procedure for sequential leaching is shown in Fig. 11.

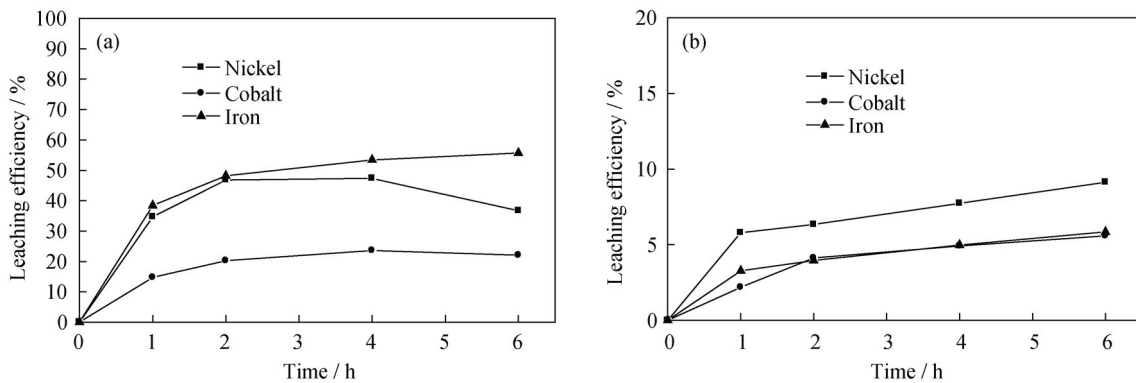


Fig. 9. Effects of second leaching step on dissolution: (a) first-step leaching with 0.5 M citric acid and second-step leaching with 0.5 M oxalic acid; (b) first-step leaching with 0.5 M oxalic acid and second-step leaching with 0.5 M citric acid.

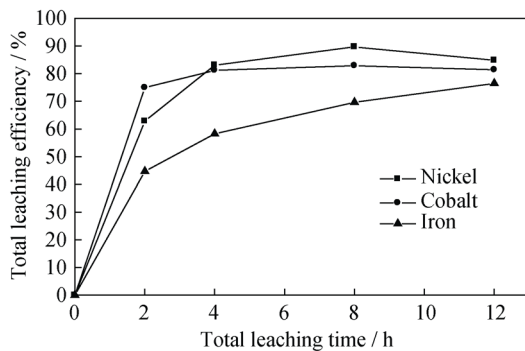


Fig. 10. Effects of sequential organic acid leaching on dissolution: first-step leaching with 0.5 M citric acid and second-step leaching with 0.5 M oxalic acid.

3.7. Comparison of this study with previous studies

Numerous studies on the extraction of nickel and cobalt from lateritic nickel ores using inorganic and organic acids under atmospheric and high-pressure conditions have been reported (Table 5). Inorganic acid leaching at atmospheric

pressure is one of the two common hydrometallurgical processes used during the extraction of nickel and cobalt from lateritic nickel ore on an industrial scale. Although the high-pressure inorganic acid leaching process offers some advantages throughout the leaching process, it also has some disadvantages, such as expensive construction costs and high required temperatures and pressures. The use of strong inorganic acids generates significant corrosion problems for leaching equipment. High acid consumption and low metal selectivity are other drawbacks to the use of inorganic acids.

In contrast, organic acids can be produced from secondary sources or wastes such as sawdust, molasses, and corncobs. Hence, the leaching efficiency of nickel and cobalt can be increased by the addition of organic acids produced from chemically and biologically treated secondary sources/wastes. Table 5 summarizes and compares the present and previous inorganic and organic acid leaching results for lateritic nickel ores. In previous studies, heap leaching (HEAP), high-pressure acid leaching (HPAL), and atmospheric-pressure acid leaching (AL) of Caldag lateritic

nickel ore with H_2SO_4 yielded approximately 79%, 94%, and 90.2% Ni dissolution, respectively. In the present study, sequential leaching at 90°C for 8 h with 0.5 M (citric + oxalic) acids achieved 89.6% Ni and 82.9% Co dissolution. Compared with the results of previous studies, sequential organic acid leaching provided the highest simultaneous ex-

tractions of nickel and cobalt. The sequential organic acid leaching method decreased the leaching time as well as the acid consumption. As a replacement for other conventional leaching techniques, the sequential organic acid leaching method can be used as an alternative for the dissolution of metals from lateritic nickel ore.

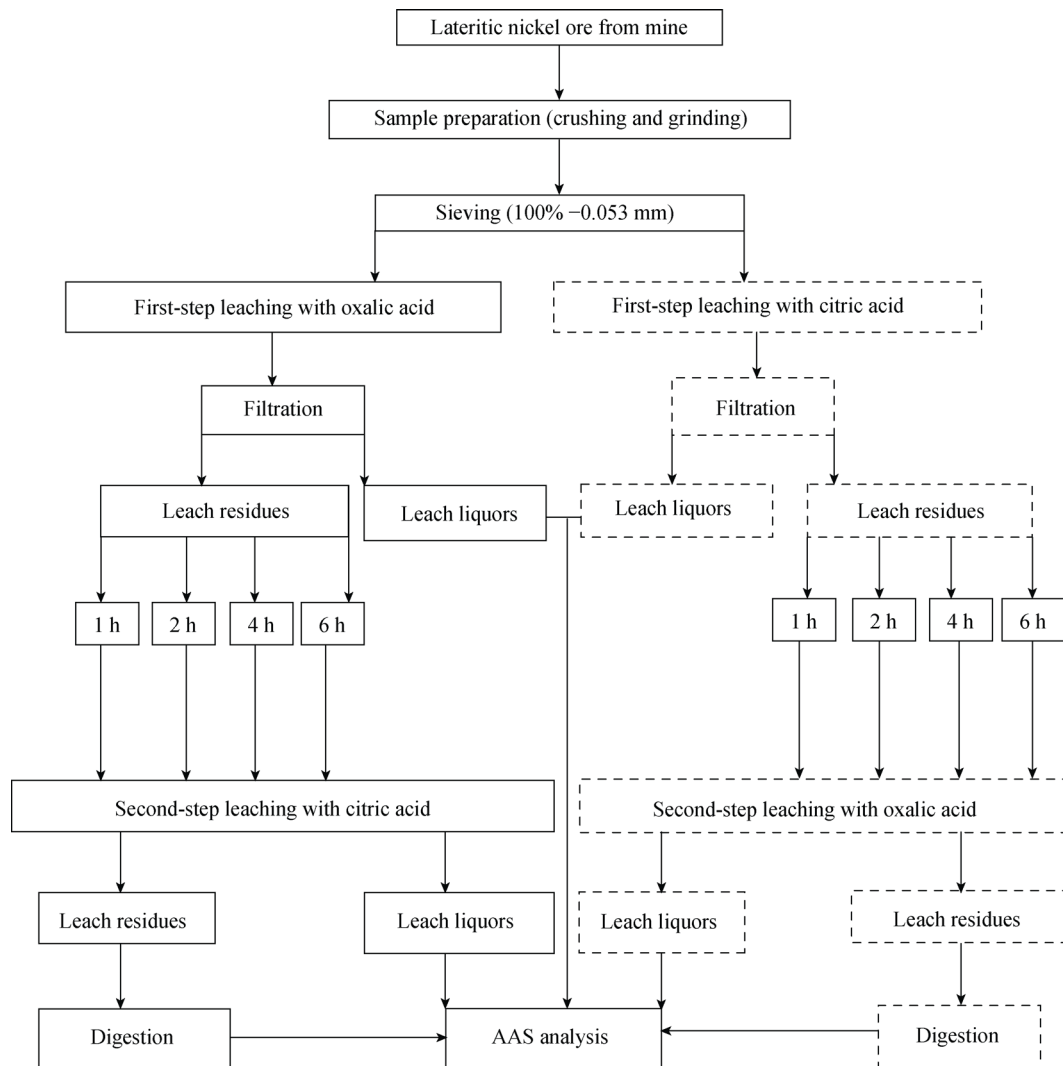


Fig. 11. Experimental procedure for sequential leaching.

4. Conclusions

The leaching of metals from Caldag lateritic nickel ore was investigated using a sequential organic acid leaching method. A sequential leaching method using citric + oxalic acids was observed to provide the best results when applied to lateritic nickel ore under the tested conditions.

The following results were observed: (1) Oxalic acid exhibited considerable reverse selectivity in the first step. (2)

Citric acid was determined to be the most appropriate individual organic acid for the extraction of nickel and cobalt from lateritic nickel ore. (3) An increase in the citric acid concentration negligibly affected the leaching efficiencies of the metals, whereas an increase in the leaching temperature had a strong beneficial effect. (4) Oxalic acid was more effective than citric acid for second-step leaching. (5) After 8 h (4 + 4 h) of leaching with organic acids (0.5 M citric + 0.5 M oxalic) in sequence at 90°C , 89.63% Ni, 82.89% Co, and 69.63% Fe were leached from the lateritic nickel ore.

Table 5. Comparison of the results of the present study with those of previous studies

Acid type	Locations of laterite deposits	Leaching type	Acid type	Leaching time	Temperature / °C	Particle size / mm	Solid to liquid ratio / (g·L ⁻¹)	Stirring speed / (r·min ⁻¹)	Dissolution / %	Activation energy / (kJ·mol ⁻¹)	Ref.
Organic acids	Saprolite ore, New Caledonia	Atmospheric	**0.6 M citric acid	10 d	30	-0.053	100	—	Ni: ~77	—	[3–4]
	Kastoria laterite ore, Greece	Atmospheric	**0.5 M citric acid	24 h	60	-0.106	100	—	Ni: 34, Fe: 33.5	—	[15]
	Limonite type laterite, Indonesia	Atmospheric	**30 g/L citric acid with 10 g/L ammonium bifluoride	2 h	Ambient	-0.074	100	23	Co: 84.5	—	[16]
	Sukinda Valley, India	Atmospheric	**1 M oxalic acid	40 h	32	-0.50 + 0.106	100	—	Ni: ~23	—	[17]
	Sukinda Valley, India	Atmospheric	**0.15 M oxalic acid	3 h	80	-0.075 + 0.053	20	—	Ni: 63.6, Co: 44.33	Ni: 74, Co: 84.37	[18]
	Caldag, Turkey	Sequential atmospheric	** First step with 0.5 M citric, second step with 0.5 M oxalic	8 h	90	-0.053	100	500	Ni: 89.63, Co: 82.89, Fe: 69.63	—	Present study
Inorganic acids	Caldag, Turkey	Heap atmospheric	*528 kg/t H ₂ SO ₄	548 d	Ambient	-25.00	—	—	Ni: 79.4	—	[20]
	Caldag, Turkey	High pressure	*325 kg/t H ₂ SO ₄	1 h	250	-1.00	—	—	Ni: 94.1	—	[21]
	Adatepe, Turkey	Atmospheric	**60% H ₂ SO ₄	2 h	95	-0.435	333	—	Ni: 99.2	Ni: 30.36	[24]
	Gordes, Turkey	Atmospheric	**5 N H ₂ SO ₄	24 h	95	-0.074	200	—	Ni: 93.1	—	[26]
	Karacam, Turkey	Atmospheric	**2 M H ₂ SO ₄	4 h	80	-0.038	200	200	Ni: 100	Ni: 68.66	[27]
Caldag, Turkey	Atmospheric	**200 g/L H ₂ SO ₄	8 h	80	-0.038	100	600	Ni: 90.2, Co: 96.8	Ni: 47.34	[28]	

Note: * acid consumption; ** acid concentration.

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