

Study on the selective chromium leaching behavior and mechanism of steel slag in citric acid media

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Abstract: Taking converter steel slag as the subject of study, this research investigates the effects of different organic acids, concentrations, liquid-to-solid ratios, temperatures, and reaction times on the chromium leaching efficiency, and analyzes its kinetic characteristics and reaction mechanisms. Under conditions of 1 mol·L⁻¹ citric acid concentration, 50°C temperature, a liquid-to-solid ratio of 10:1, and an extraction time of 80 minutes, the leaching efficiency of Cr, Fe, and Ca were 75.68%, 76.67%, and 2.6%, respectively. Analysis using XRD, SEM-EDS, and FTIR revealed that the calcium-containing mineral phases in the steel slag react preferentially with citric acid; the released Ca²⁺ combines with citrate ions to form calcium citrate, while a portion undergoes carbonation to form CaCO₃. The dissolution of the calcium phase weakens the original structural stability of the steel slag, promotes particle fragmentation and the development of a porous structure, and exposes the chromium-bearing mineral phases enclosed within it, thereby providing reaction pathways for Cr and Fe. The Fe–Cr spinel structure is highly stable; citric acid cannot completely break it down, but only causes some of the Cr to be released from the spinel lattice. The dissolved Cr³⁺ forms a complex with the citrate ion, resulting in a stable, water-soluble citrate complex. Fe ions released from the dissolution of iron oxide mineral phases enter the solution by complexing with citrate ions. Kinetic analysis indicates that the reaction follows a contracting-core model and is primarily controlled by interfacial chemical reactions, with an apparent activation energy of 43.42 kJ mol⁻¹.

Keywords: steel slag, chromium, citric acid, selective leaching, kinetics

1. Introduction

In recent years, the continuous rise in domestic crude steel production has directly driven an increase in the output of by-products from steelmaking (Liu et al., 2026; Wang et al., 2026). Among the various types of industrial waste, steel slag ranks first among metallurgical solid wastes in terms of the sheer volume produced (Shen and Forssberg, 2003; Guo et al., 2018), yet its comprehensive utilization rate remains limited. Statistics show that, with the continued expansion of crude steel production capacity, China's annual steel slag output currently remains at a high level of 120 million to 160 million metric tons, and the historical stockpile has exceeded 1 billion metric tons (Liu et al., 2020; Najm et al., 2021; Cai et al., 2023). It is important to note that the various metallic components present in steel slag pose a risk of activation and migration under long-term weathering and leaching (Estokova et al., 2018) which may constitute a potential threat to regional ecological security (Varitis et al., 2017; Alias et al., 2023; Wang et al., 2025). In particular, stainless steel slag generated during the smelting of stainless steel typically has a chromium content ranging from 800 to 10,000 mg/kg (Kim et al., 2015; Gu et al., 2021).

In naturally stockpiled steel slag, trivalent chromium can be oxidized to hexavalent chromium in an oxidizing environment (Zhao et al., 2018; Ramli et al., 2023; Iwama et al., 2026), and is primarily present in stable mineral structures such as spinel and the RO phase (Costa et al., 1997; Zeng et al., 2021); the latter exhibits significantly increased biological toxicity. Hexavalent chromium is highly water-soluble and mobile (Zhitkovich, 2011; Godbole et al., 2025); once it enters the soil or aquatic environment, it is

easily absorbed by crop roots or aquatic organisms, ultimately posing a risk to human health (Proctor et al., 2000; Binnemans et al., 2020; Cao et al., 2024). Therefore, reducing the risk of chromium release into the environment from steel slag has become a technical hurdle that must be overcome before its large-scale utilization can be achieved (Qiu et al., 2023).

There are currently two main technical approaches for the separation and extraction of chromium from steel slag: high-temperature pyrometallurgical methods and liquid-phase hydrometallurgical methods. Firing processes typically impose stringent requirements on reaction temperature, necessitating heating to 1600°C (Kim et al., 2016; Zhao et al., 2020); the resulting high energy consumption (Xu et al., 2023), equipment durability requirements, and capital investment have become the primary bottlenecks limiting their industrial adoption. Wet leaching has given rise to various technical approaches, such as oxidation-assisted leaching and alkali roasting-leaching (Chen and Zhou, 2014; Pikna et al., 2020), which have, to some extent, lowered the operational barriers and improved leaching efficiency; however, these wet leaching methods still face challenges such as insufficient metal selectivity and difficulties in disposing of high-salt waste liquids.

As the concept of green, low-carbon development gains traction, organic acid leaching systems are emerging as a prominent solution in the field of metallurgical separation. Compared with traditional inorganic acid media, organic acids—with their low toxicity, biodegradability, and excellent environmental compatibility—meet the requirements for green resource recovery processes. The molecular structure of citric acid ($C_6H_8O_7$) contains one hydroxyl group (-OH) and three carboxyl groups (-COOH), giving it both moderate acidity and strong chelating ability (Ji et al., 2017). In recent years, the citric acid system has demonstrated excellent performance in research on the resource recovery of spent lithium-ion batteries and electronic waste, enabling the selective recovery of various valuable metals such as lithium, cobalt, and nickel (Nagarajan and Panchatcharam, 2023; Bao et al., 2025). In addition, attempts have been made to introduce hydrogen peroxide (H_2O_2) to establish a synergistic “oxidation-complexation” system to further enhance metal leaching efficiency, and positive progress has been made in research on the extraction of metals such as copper, nickel, and cobalt (Lim et al., 2023; Yang et al., 2024).

However, current research on chromium in steel slag has primarily focused on enhanced techniques such as alkaline roasting, oxidative leaching, and high-temperature reduction, while studies on selective leaching using organic acid media and the mechanisms of mineral phase evolution remain relatively scarce; the underlying reaction mechanisms and conditions still need to be elucidated. Therefore, a citric acid solution was used as the leaching agent. Given that calcium is the primary matrix element in steel slag and its dissolution behavior directly affects acid consumption and process economics, Ca was specifically selected as the reference element in this study. The selective leaching behavior of chromium in steel slag was systematically investigated, and its reaction mechanism was thoroughly examined through a combination of kinetic analysis and microscopic characterization techniques.

2. Materials and methods

2.1. Test materials

The steel slag raw material was sourced from the converter slag of a steel mill in Tangshan (hereinafter referred to as “steel slag”); the original steel slag consisted of irregular lumps. After collection, the steel slag was thoroughly mixed and subdivided to obtain representative experimental samples. Subsequently, the subdivided steel slag was ground using a dry grinder and sieved through a 100-mesh screen. The resulting undersize fraction was thoroughly mixed and used as the experimental sample for subsequent leaching experiments and related characterization analyses.

The ground and screened steel slag test samples were subjected to multi-element chemical analysis, XRD, MLA, particle size distribution analysis, and microstructural characterization. Their chemical composition is shown in Table 1.

As shown in Table 1, steel slag consists primarily of elements such as Ca, Fe, and O, with concentrations of 36.21%, 23.26%, and 23.03%, respectively. In addition to the major elements, the steel slag contains 5.55% Si and 5.01% Al, as well as 2.96% Mg and 1.01% Mn. It also contains small amounts of other elements, including S (0.73%), P (0.49%), Cr (0.27%), and Ti (0.22%).

To determine the mineral and elemental composition of the steel slag, an X-ray diffractometer (XRD) was used to analyze the mineral properties of the steel slag samples. The results are shown in Fig. 1. The MLA image is shown in Fig. 2.

Table 1. Chemical composition of steel slag

Elements	Ca	Fe	O	Si	Al	Mg	Mn	S	P	Cl	Ge	K	Cr	Ti	F	Zn
Content	36.21	23.26	23.03	5.55	5.01	2.96	1.01	0.73	0.49	0.43	0.43	0.34	0.27	0.22	0.02	0.02

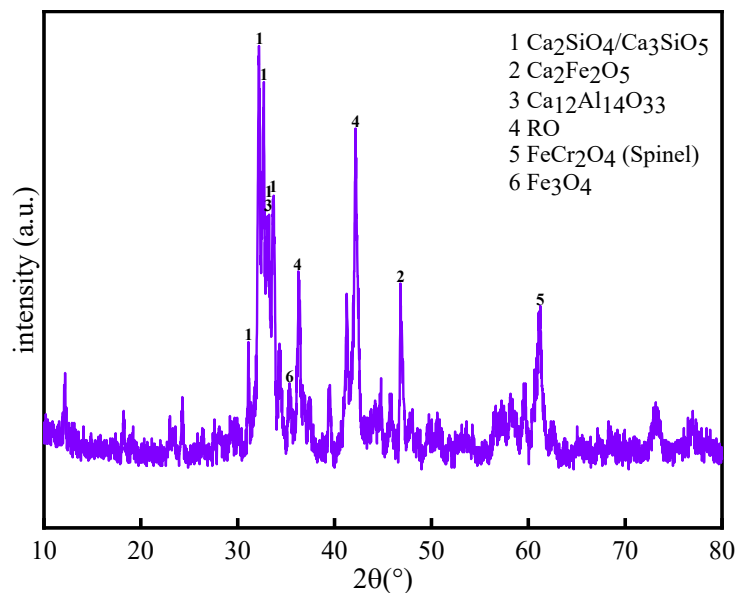


Fig. 1. XRD patterns of steel slag

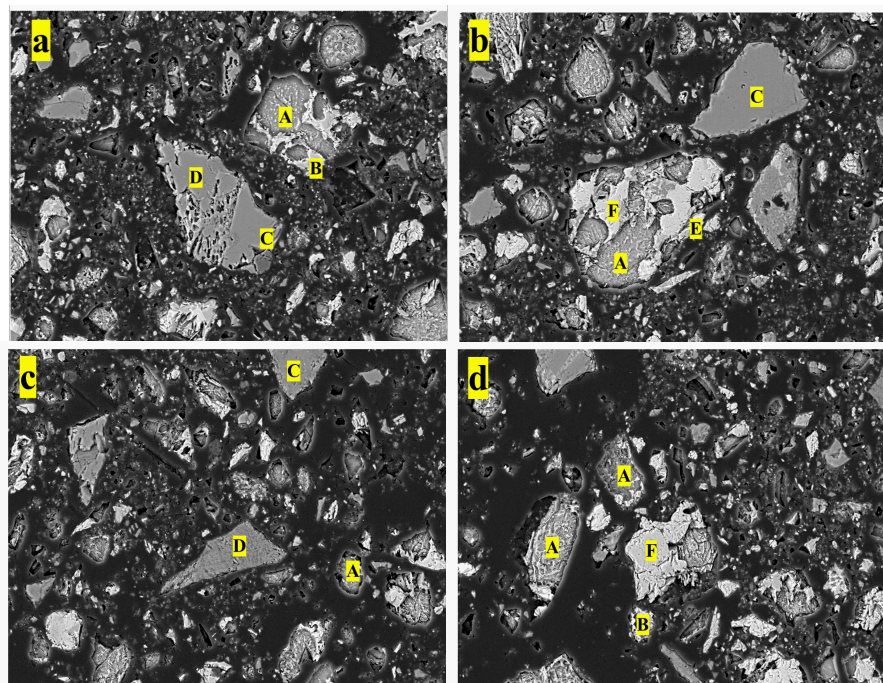


Fig. 2. MLA images of steel slag. (A) dicalcium silicate, tricalcium silicate; (B) calcium ferrite; (C) calcium aluminate; (D) RO phase; (E) spinel; (F) magnetite

As can be seen from the analysis results in Fig. 1 and Fig. 2, steel slag consists primarily of minerals such as dicalcium silicate (Ca_2SiO_4), tricalcium silicate (Ca_3SiO_5), calcium ferrite ($\text{Ca}_2\text{Fe}_2\text{O}_5$), calcium

aluminate ($\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$), the RO phase, the spinel phase (FeCr_2O_4), and magnetite (Fe_3O_4). Since the steel slag used in this study is converter slag, its mineral phases are primarily artificial metallurgical minerals formed through high-temperature melting and subsequent cooling and crystallization during the steelmaking process, rather than natural minerals. Therefore, chromium in steel slag does not typically exist as a separate mineral but, under high-temperature conditions, incorporates into iron-containing oxide phases, forming a symbiotic relationship with iron. Among these, dicalcium silicate, tricalcium silicate, calcium ferrite, and calcium aluminate are the primary mineral phases in steel slag. In addition, it contains the RO phase, which exists as a MgO-FeO-MnO solid solution, as well as iron oxide phases such as spinel and magnetite. In particular, the spinel phase (FeCr_2O_4) and the RO phase serve as important hosts for chromium in steel slag, and chromium and iron exhibit a strong association within the mineral structure. Consequently, during the subsequent citric acid leaching process, the release of chromium is often accompanied by the dissolution of iron-bearing mineral phases, and iron is simultaneously introduced into the leaching system.

A particle size distribution analysis was conducted on the steel slag; the results are shown in Fig. 3.

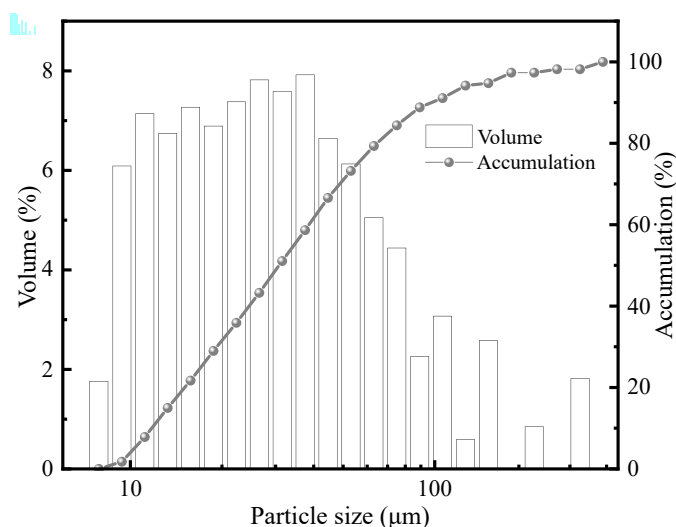
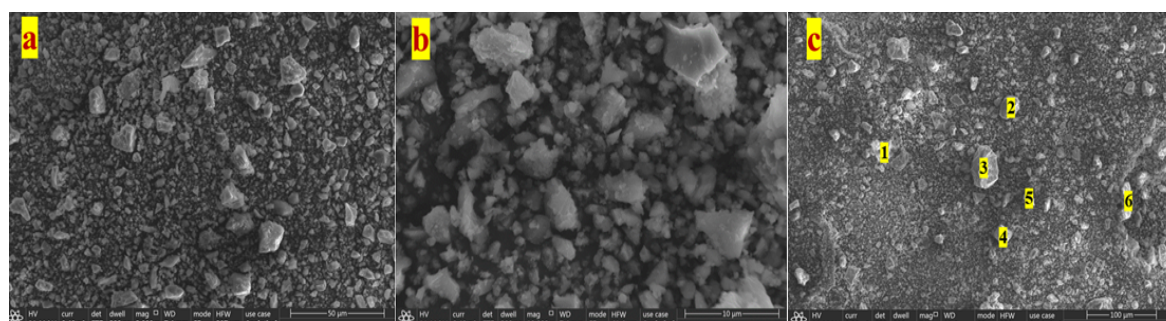


Fig. 3. Particle size distribution of steel slag

As shown in Fig. 3, steel slag particles are primarily distributed below 300 μm , with particles smaller than 100 μm accounting for 92.6% of the total. Calculations show that the volume-weighted average particle size is 38.5 μm , indicating that this steel slag consists mainly of fine-grained particles. The finer particle size facilitates the exposure of mineral phases, providing a favorable particle size basis for subsequent mineral leaching.

To analyze the microstructure and mineral distribution on the surface of the steel slag, the samples were examined using a scanning electron microscope (SEM), and energy-dispersive X-ray spectroscopy (EDS) was employed to study the mineral distribution. The SEM images of the steel slag were observed at different magnifications; the results are shown in Fig. 4. The chemical composition analysis of the steel slag based on EDS is presented in Table 2.



a-SEM image of steel slag (2000 $\times$); b-SEM image of steel slag (10,000 $\times$); c-EDS spot analysis.

Fig. 4. SEM morphology and EDS spot analysis of steel slag

As shown in Fig. 4, the steel slag particles vary in shape, ranging from agglomerates of mixed sizes to densely structured lumps. The surfaces of coarse particles are densely covered with a large number of fine mineral particles, forming distinct heterogeneous coating features; the particle surfaces are uneven with sharp contours. At a magnification of 10,000x, a large accumulation of flocculent material can be observed in the depressions and gaps of the particles; this flocculent material may be a gel formed by the weak hydration of highly reactive mineral phases or adsorbed ultrafine dust.

Table 2. EDS chemical composition analysis of steel slag

Data point	C	O	Mg	Al	Si	Ca	Cr	Fe
Point 1	5.81	26.32	1.71	1.39	7.65	44.92	0.47	11.73
Point 2	1.69	13.58	0.5	0.4	1.63	56.35	0.65	25.2
Point 3	6.91	39.51	1.1	1.51	11.41	37.96	0.1	1.51
Point 4	10.16	30.42	12.14	0.51	0.64	2.74	0.56	42.84
Point 5	4.18	28.19	0.53	1.26	8.63	45.07	0.14	11.99
Point 6	8.9	39.83	1.39	3.77	2.19	27.63	1.23	15.07

As shown in Table 2, Point 1 and Point 5 have relatively high Ca and Si contents and also contain a certain amount of Fe. It is speculated that these consist primarily of iron-doped tricalcium silicate or dicalcium silicate phases, accompanied by a small amount of calcium ferrite. The low Cr content indicates that the degree of Cr solid solution in silicate mineral phases is limited. At Point 2, the Ca and Fe contents are significantly higher, while the Si content is low, indicating that the minerals in this region primarily belong to the Ca-Fe-O system and may be calcium ferrite or calcium-rich iron oxide phases. The distribution of Cr in these iron-rich phases is slightly increased, showing a certain trend toward enrichment. At Point 3, the Si content is highest while the chromium content is lowest, indicating that Cr has difficulty entering the high-purity silicate crystal lattice structure, suggesting that the silicate lattice is not the primary carrier for Cr enrichment. Sample 4 exhibits extremely high Fe and Mg contents and extremely low Si content, and contains a certain amount of chromium; it is therefore presumed to be the RO phase. Chromium may have entered the oxide solid solution (Mg,Fe,Ca)O via isostructural substitution, indicating that chromium tends to coexist with magnesium-iron oxides. At point 6, both Al and Cr contents are relatively high, and the sample exhibits high Fe and low Si characteristics; it is speculated to be an iron aluminate or a chromium-bearing spinel phase. These results suggest that iron-rich aluminum oxide phases may serve as important hosts for chromium.

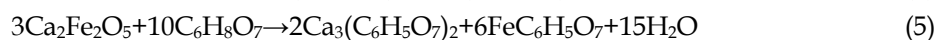
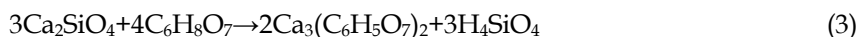
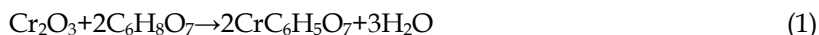
2.2. Test equipment and materials

Instruments: XRF (Panalytical), XRD (Empyrean), SEM-EDS (Scios), laser particle size analyzer (NKT6100-D), MLA (FEI MLA 650), FTIR (Nicolet iS 10), magnetic stirrer (HJ-6B), analytical balance (CP214), Constant-Temperature Forced-Air Oven (3AB), Atomic Absorption Spectrometer (Z-2010).

Reagents: Citric acid, analytical grade (AR); Oxalic acid, analytical grade (AR); Lactic acid, analytical grade (AR); Tartaric acid, analytical grade (AR); Malic acid, analytical grade (AR); Deionized water.

2.3. Test principles and methods

Citric acid was selected as the leaching agent to leach the three elements—Cr, Fe, and Ca. The main reaction equations are as follows:



In the primary reaction (1) in which citric acid solution dissolves chromium, the chromium-rich RO phase is effectively attacked by H^+ ions ionized from citric acid, causing the metal-oxygen bonds in the mineral lattice to break and releasing chromium from the solid-phase surface in the form of Cr^{3+} . At this point, the citrate ions in the solution play a key chelating role, rapidly capturing the free Cr^{3+} to form a

highly stable, water-soluble chromium citrate complex, which promotes the continuous diffusion and dissolution of chromium from the deeper layers of the solid phase into the liquid phase. In reaction (2), because the spinel structure is stable, the citric acid dissolves only a portion of the chromium-iron spinel, and some chromium remains in the unreacted mineral phase. Reaction (3) also proceeds simultaneously. Because the steel slag is strongly alkaline, H^+ is consumed during the leaching process, causing calcium citrate to precipitate. Iron exists primarily in the form of magnetite and calcium ferrite, undergoing chemical reactions (4) and (5). During the citric acid leaching process, the H^+ ions produced by the dissociation of citric acid break the Fe–O bonds in the iron phase, causing the magnetite and calcium ferrite lattices to dissolve and releasing Fe^{2+} and Fe^{3+} into the leaching system. Specifically, the dissolution of magnetite produces Fe^{2+} and Fe^{3+} , while calcium ferrite decomposes under acidic conditions, releasing Fe^{3+} and Ca^{2+} . Subsequently, the released Fe ions form complexes with citrate ions, creating stable iron-citrate complexes that facilitate the migration of iron from the solid phase to the liquid phase. After being released from calcium ferrite, Ca^{2+} readily combines with citrate ions to form an insoluble calcium citrate precipitate, thereby limiting the migration of calcium.

Test Methods: For the leaching experiment, steel slag samples were used that had been thoroughly mixed, subdivided, dry-ground, and sieved through a 100-mesh sieve. The acid solution was diluted with deionized water to the concentration required for the test. Using an analytical balance, 5.00 g of the steel slag sample was accurately weighed, and the corresponding volume of diluted acid solution was added according to a predetermined liquid-to-solid ratio, after which the mixture was placed in a 150 mL conical flask. The constant-temperature water bath was set to the target temperature. Once the temperature reached the set value, the conical flask was placed in the water bath, the magnetic stirrer was turned on, and the stirring speed and reaction temperature were maintained at constant levels. Immediately after leaching was complete, solid-liquid separation was performed via vacuum filtration; the resulting solid was the leaching residue, and the filtrate was the leachate. Collect the leachate and determine the concentrations of Cr, Ca, and Fe; the leaching residue is used for subsequent characterization analyses. The leaching efficiency of each element is calculated using Equation (6).

$$\eta = \frac{CV}{mw} \times 100\% \quad (6)$$

In the equation: C represents the concentrations of Cr, Ca, and Fe in the leachate, in $mg \cdot L^{-1}$; V represents the volume of the leachate, in L; m represents the mass of the steel slag test sample, in g; w represents the mass fractions of Cr, Ca, and Fe in the steel slag test sample, in %.

3. Results and discussion

3.1. Selective leaching of steel slag

Single-factor experiments were conducted to investigate the effects of different acid types, temperatures, liquid-to-solid ratios, concentrations, and times, and the leaching efficiency was calculated to determine the optimal reaction conditions for steel slag leaching.

3.1.1. The effect of organic acid types on chromium leaching efficiency from steel slag

When the organic acid concentration, temperature, liquid-to-solid ratio, and reaction time were $1 \text{ mol} \cdot L^{-1}$, $50 \text{ }^{\circ}C$, $10 \text{ mL} \cdot g^{-1}$, and 80 min, respectively, the effect of the type of organic acid on the chromium leaching efficiency from steel slag was investigated. The experimental results are shown in Fig. 5.

As shown in Fig. 5, the leaching efficiency of Cr is the highest in the citric acid system, reaching 75.68%, while the leaching efficiency of Ca is only 2.6%. The difference between the two is 73.08 percentage points, and the ratio of Cr to Ca leaching efficiency is approximately 29.11. In contrast, the leaching efficiency of Fe was 76.67%, showing a similar trend to that of Cr, indicating that both elements primarily originate from similar spinel mineral structures and are released simultaneously under citrate complexation. Since Ca is primarily present in a highly soluble calcium phase, while Cr and Fe are mainly present in stable oxides or spinel structures, citric acid promotes the migration of transition metals through complexation while simultaneously inhibiting the extensive leaching of Ca, thereby achieving highly selective leaching of Cr. Therefore, citric acid was selected as the acid to be used in subsequent experiments.

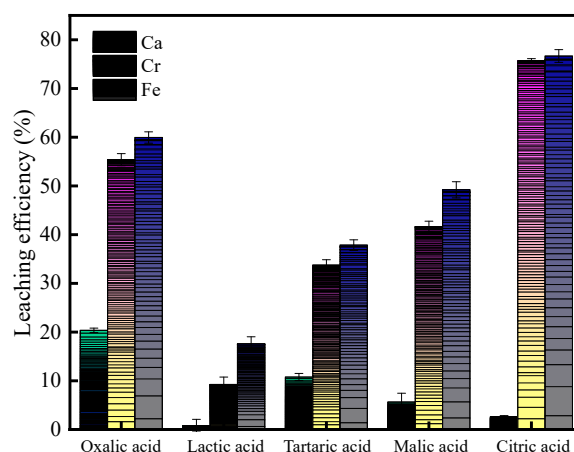


Fig. 5. Effect of organic acid type on leaching efficiency

3.1.2. The effect of temperature on the chromium leaching efficiency from steel slag

When the liquid-to-solid ratio, citric acid concentration, and reaction time were 10 mL $\cdot$ g⁻¹, 1 mol $\cdot$ L⁻¹, and 80 min, respectively, the effect of temperature on the chromium leaching efficiency from steel slag was investigated; the experimental results are shown in Fig. 6.

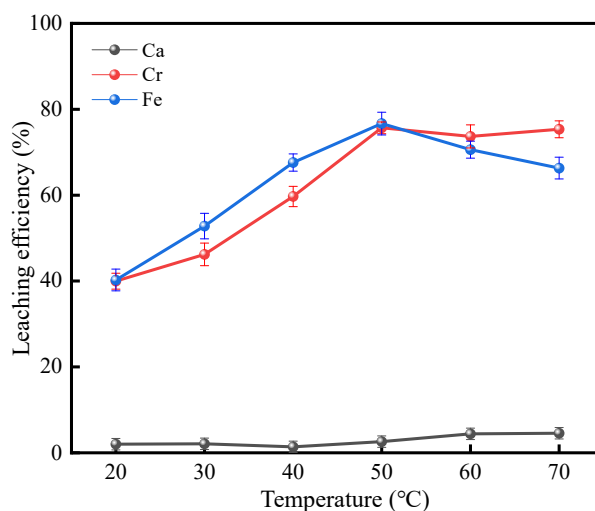


Fig. 6. Effect of temperature on leaching efficiency

As shown in Fig. 6, as the reaction temperature continues to rise, the leaching efficiency of Cr initially increases rapidly and then remains stable; the trend for Fe is essentially consistent with that of Cr, while the leaching efficiency of Ca shows only a slight increase. In the temperature range of 30–50 °C, the leaching efficiency of Cr increased from 46.20% to 75.68%, indicating that higher temperatures promote the leaching process. This phenomenon is primarily attributed to the fact that higher temperatures accelerate the reaction kinetics and increase the rate of molecular diffusion, while also facilitating the complexation reaction between citric acid and Cr³⁺, thereby promoting the leaching of chromium. When the temperature exceeds 50 °C, the Cr leaching efficiency decreases slightly. Meanwhile, the Ca leaching efficiency remains consistently below 5%; therefore, citric acid solution exhibits good selectivity for Cr in steel slag when used as a leaching agent. Consequently, 50 °C is the optimal temperature, at which the Cr leaching efficiency is highest at 75.68%, while the Ca leaching efficiency is 2.6%.

3.1.3. The effect of the liquid-to-solid ratio on chromium leaching efficiency from steel slag

When the temperature, citric acid concentration, and reaction time were 50°C, 1 mol $\cdot$ L⁻¹, and 80 min, respectively, the effect of the liquid-to-solid ratio on the chromium leaching efficiency from steel slag was investigated. The experimental results are shown in Fig. 7.

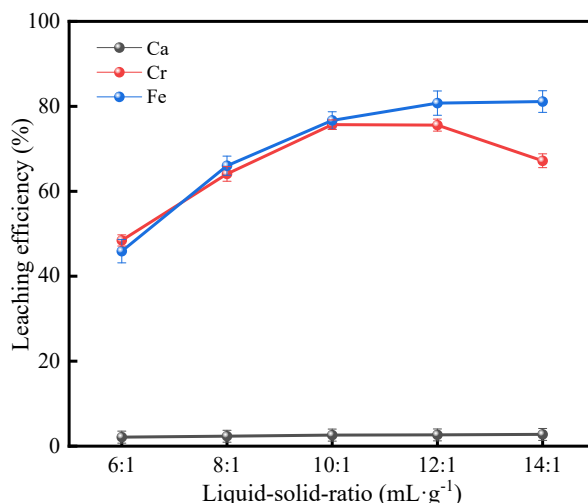


Fig. 7. Effect of the liquid-to-solid ratio on leaching efficiency

As shown in Fig. 7, the leaching efficiency of Cr first increases and then decreases as the liquid-to-solid ratio increases; that of Fe continues to rise, while that of Ca remains relatively stable, showing no significant fluctuations. When the liquid-to-solid ratio was increased to 10 mL·g⁻¹, the leaching efficiency of Cr from the steel slag reached 75.68%, that of Fe was 76.67%, and that of Ca was 2.6%. When the liquid-to-solid ratio was increased to 12 mL·g⁻¹, the Cr leaching efficiency was 75.99%. After increasing the liquid-to-solid ratio from 10 mL·g⁻¹ to 12 mL·g⁻¹, the Cr leaching efficiency increased by only 0.31%, while citric acid dosage increased by 20%, and the Cr leaching efficiency tended to stabilize. Therefore, 10 mL·g⁻¹ was selected as the optimal liquid-to-solid ratio.

3.1.4. Effect of citric acid concentration on the chromium leaching efficiency from steel slag

When the temperature, liquid-to-solid ratio, and time were 50 °C, 10 mL·g⁻¹, and 80 min, respectively, the effect of citric acid concentration on the chromium leaching efficiency from steel slag was investigated. The experimental results are shown in Fig. 8.

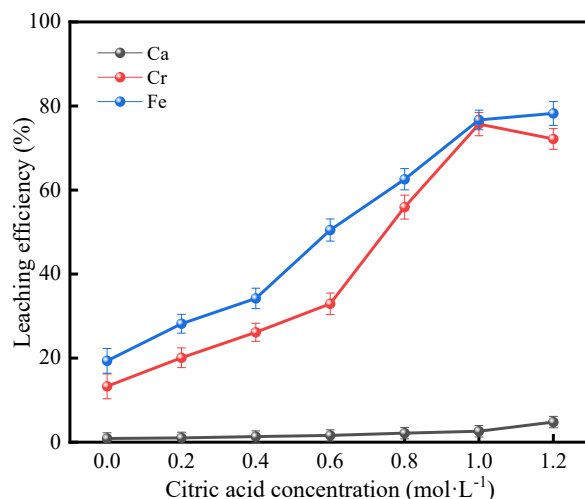


Fig. 8. Effect of citric acid concentration on leaching efficiency

As shown in Fig. 8, the leaching efficiency of Ca remained at an extremely low level—below 5%—throughout the entire concentration range, showing only a very slight increase as the concentration rose. The leaching efficiency of both Cr and Fe showed a significant upward trend as the citric acid concentration increased, with the leaching efficiency of Fe remaining slightly higher than that of Cr throughout the process. Both approached their peak leaching efficiency when the concentration reached 1.0 mol·L⁻¹, and when the concentration was further increased to 1.2 mol·L⁻¹, the leaching efficiency of

Fe rose only slightly, while the leaching efficiency of Cr actually declined slightly. This indicates that $1.0 \text{ mol} \cdot \text{L}^{-1}$ is an optimal concentration that balances leaching efficiency and reagent economy; further increasing the citric acid concentration is unlikely to sustainably enhance leaching performance and may even slightly inhibit the leaching of Cr due to changes in the solution environment.

3.1.5. Effect of time on the chromium leaching efficiency from steel slag

When the temperature, liquid-to-solid ratio, and citric acid concentration were 50°C , $10 \text{ mL} \cdot \text{g}^{-1}$, and $1 \text{ mol} \cdot \text{L}^{-1}$, respectively, the effect of time on the chromium leaching efficiency from steel slag was investigated; the experimental results are shown in Fig. 9.

As shown in Fig. 9, the leaching efficiency of Ca increases very slightly as the reaction time increases. Overall, the leaching efficiency of Cr and Fe showed an upward trend as reaction time increased, between 20 and 80 minutes, the leaching efficiency of both continued to rise, with Cr showing a more significant increase; by 80 minutes, Cr reached a stage peak and approached Fe's leaching efficiency. Between 80 and 100 minutes, the leaching efficiency of both declined slightly; after 100 minutes, Fe's leaching efficiency continued to rise, reaching its highest point at 120 minutes, while Cr's recovery was limited. Considering both the overall leaching efficiency and reaction energy consumption, 80 minutes is the optimal leaching time.

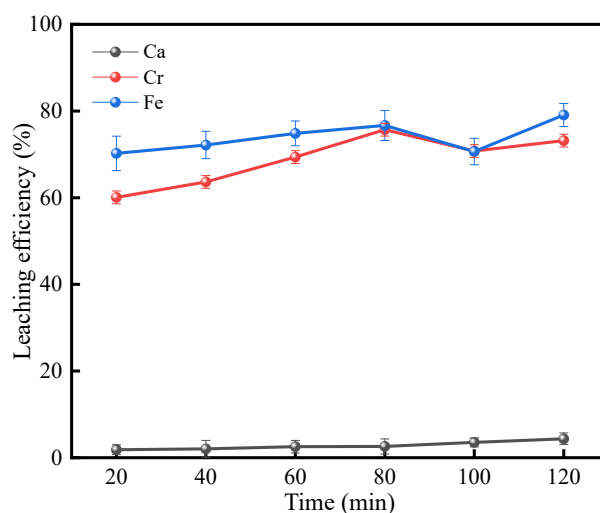


Fig. 9 Effect of time on leaching efficiency

3.2. Analysis of the Leaching Residue After the Reaction

Under the optimized leaching conditions, the leaching residue was subjected to particle size distribution analysis, XRD analysis, microstructural analysis, and infrared analysis.

A particle size distribution analysis was performed on the leaching residue; the results are shown in Fig. 10. A comparison of Fig. 3 and Fig. 10 shows that citric acid leaching results in a significant reduction in the particle size of the steel slag. The particle size of the steel slag experimental sample is primarily distributed in the range of $10\text{--}100 \mu\text{m}$, with a D_{50} of approximately $35 \mu\text{m}$; in contrast, the particle size of leaching residue decreases sharply to the range of $0.5\text{--}5 \mu\text{m}$, with a D_{50} dropping to approximately $1.5 \mu\text{m}$. This order-of-magnitude reduction in particle size indicates that citric acid effectively disrupts the mineral framework of the steel slag, leading to particle disintegration and thereby promoting the dissociation and release of the heavy metal chromium.

To determine the elemental composition of the leaching residue, an X-ray diffractometer (XRD) was used to analyze the mineral properties of the residue; the results are shown in Fig. 11.

A comparison of the phase compositions before and after leaching (Fig. 1 and Fig. 11) shows that the characteristic diffraction peaks of dicalcium silicate and tricalcium silicate originally present in the steel slag completely disappeared after the reaction. This phenomenon confirms that the H^+ ions provided by citric acid disrupt the unstable crystal structure of silicate minerals, and the released Ca^{2+} ions immediately combine with citrate ions to form calcium citrate precipitate. At the same time, the originally ordered siloxane tetrahedral network undergoes depolymerization and reorganization due

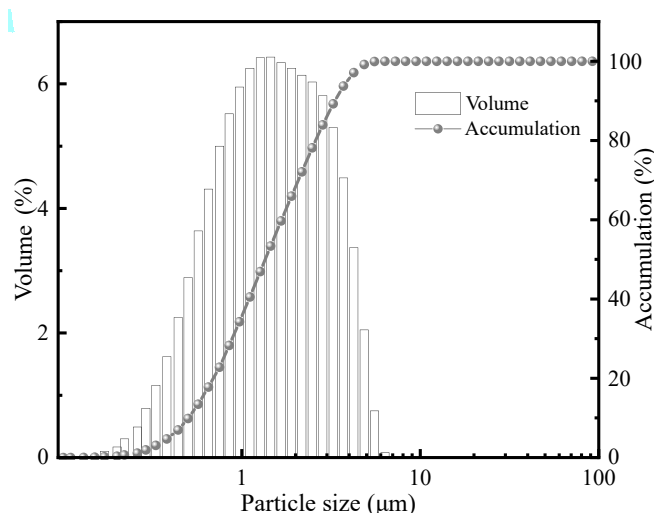


Fig. 10. Particle size distribution of leaching residue

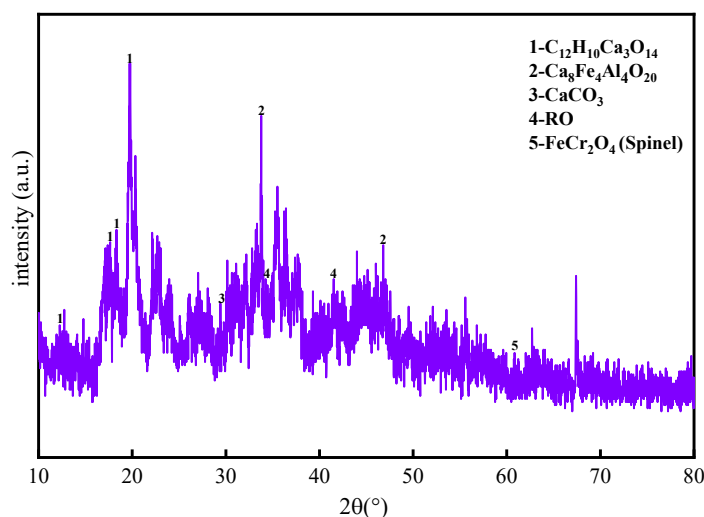


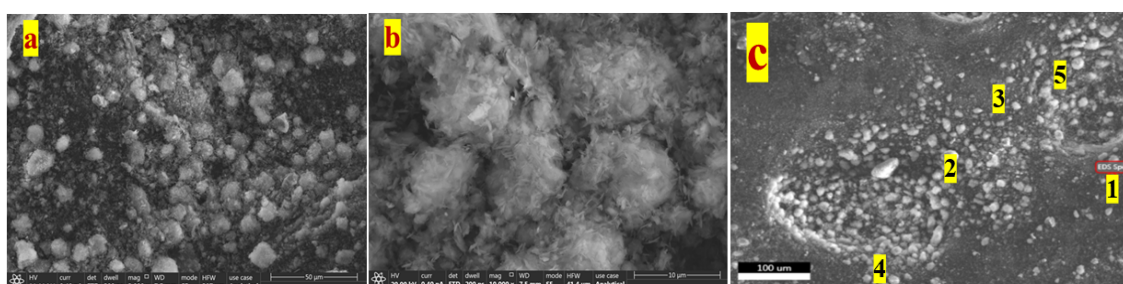
Fig.11. XRD patterns of leaching residue

to its amorphous nature, ultimately evolving into an amorphous siloxane structure. During the leaching process, iron (Fe) enters the solution and combines with citrate ions to form a complex. However, the spinel phase of the chromium-enriched carrier did not dissolve completely; only a portion of it dissolved. The residual characteristic peaks indicate that these dense oxide solid solutions exhibit strong chemical inertness toward weak organic acids. This suggests that the citric acid system not only promotes Cr migration but also regulates Ca behavior through the precipitation of calcium citrate, thereby achieving selective separation of Cr and Ca.

To investigate the differences in morphology and surface composition between steel slag and leaching residue, SEM-EDS techniques were used to analyze the leaching residue. The SEM morphology of the leaching residue is shown in Fig. 12, and the EDS spectral analysis of its chemical composition is presented in Table 3.

As shown in the SEM image in Fig. 12, after citric acid leaching, the surface of the steel slag particles became rough and porous; the originally dense mineral structure was disrupted, and distinct erosion grooves and pores appeared on the surfaces of the larger particles. Numerous fine particles and flocculent matter can be seen tightly adhering to one another.

Based on Table 2, Fig. 11, and Table 3, it can be seen that citric acid leaching significantly altered the speciation of elements and the mineral structure in the steel slag, resulting in the selective migration



a-SEM image of leaching residue (2000×); b-SEM image of leaching residue (10,000×); c-EDS spot analysis.

Fig. 12. SEM morphology and EDS spot analysis of leaching residue

Table 3. EDS chemical composition analysis of leaching residue

Data point	C	O	Mg	Al	Si	Ca	Cr	Fe
Point 1	29.61	51.5	0.94	0.85	11.51	4.86	0.01	0.71
Point 2	7.99	14.99	0	0.09	61.1	14.23	0.23	1.37
Point 3	25.12	52.55	1.62	0.73	0.74	18.03	0.03	1.18
Point 4	22.66	53.57	1.51	0.46	0.57	20.07	0.03	1.13
Point 5	24.17	53.44	1.69	0.59	0.74	17.40	0.03	1.94

of Fe and Cr and the solid-phase enrichment of Ca and Si. Steel slag exhibits distinct multiphase heterogeneous characteristics, with Ca and Fe as the primary elements, accompanied by components such as Si, Mg, Al, and Cr. After leaching, the Fe and Cr contents decreased significantly, consistent with the relatively high leaching efficiency, indicating that most of the Fe and Cr had transferred from the solid phase to the liquid phase. In contrast, Ca remained stably retained in the leaching residue because it combined with citrate ions to form calcium citrate and was partially carbonated to form CaCO_3 . The leaching residue consists primarily of calcium citrate and also contains residual phases such as $\text{Ca}_8\text{Fe}_4\text{Al}_4\text{O}_{20}$, CaCO_3 , the RO phase, and FeCr_2O_4 . Among these, FeCr_2O_4 —due to its high lattice stability—is difficult to completely break down by citric acid and is the primary phase factor limiting the further leaching of Fe and Cr.

To further clarify the changes in the internal phases during the leaching reaction, infrared analysis was performed on the steel slag and leaching residue; the results are shown in Fig. 13 below.

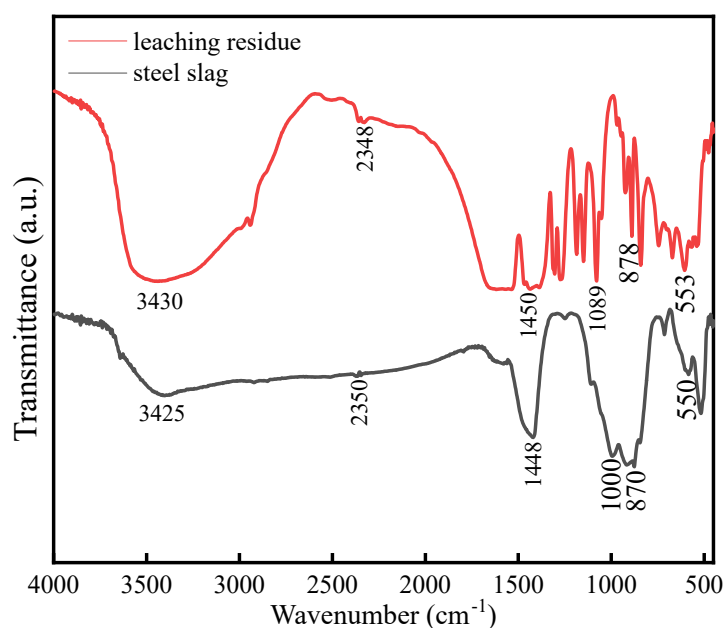


Fig. 13. Comparison of FTIR spectra of steel slag and leaching residue

FTIR results show that Ca is primarily present in the C_2S and C_3S phases of steel slag. In a citric acid system, it readily forms complexes and dissolves, leading to the destruction of calcium-silicon-related structures in the steel slag. Ca is released from the silicate framework and combines with excess citric acid molecules to form calcium citrate precipitate, corresponding to the appearance of the carboxyl stretching vibration peak near 1600 cm^{-1} in the leaching residue. The enhancement of the Si-O-Si vibrational peak at $900\text{--}1100\text{ cm}^{-1}$ further indicates that the original calcium-silicate structure has been disrupted and that Ca has migrated out of the silicate framework. At approximately 1450 cm^{-1} and 870 cm^{-1} , the characteristic peaks of CO_3^{2-} were significantly enhanced, indicating that some of the dissolved Ca underwent carbonation with CO_2 in the air during subsequent processes to form $CaCO_3$ (Yang et al., 2024). This is further corroborated by the characteristic peaks corresponding to calcium carbonate shown in Fig. 11, confirming that only a very small amount of calcium remained in the solution. The simultaneous enhancement of the CO_2 absorption peak in the gas phase at 2348 cm^{-1} indirectly reflects the development of the leach residue's pore structure and its increased ability to adsorb CO_2 , which corresponds to the occurrence of the carbonization process. The decrease in Fe content after leaching indicates that active iron phases, such as magnetite and calcium ferrite, have partially dissolved. However, the Fe-O and Cr-O vibrational peaks remain present, suggesting that the Fe-Cr spinel structure has not been completely destroyed.

In contrast, in the $500\text{--}700\text{ cm}^{-1}$ region, the Fe-O and Cr-O vibrational peaks are still present in the leaching residue, indicating that Cr has not completely entered the solution but remains partially retained in the solid phase. This is because the structurally stable Fe-Cr spinel has not been completely destroyed, and some Fe remains co-residued with Cr in the stable phase. Only a portion of the Cr has been gradually leached out of the original mineral structure and entered the solution; the leaching process is not complete, thus exhibiting the characteristic of "partial leaching and partial retention."

3.3. Analysis of the reaction mechanism

Surface area and pore size analyses were conducted on steel slag and leaching residue; see Fig. 14. As shown in Fig. 14, the citric acid leaching process significantly altered the pore structure characteristics of the steel slag, increasing the material's specific surface area and the degree of pore development. As shown in Fig. 14(a), the adsorption capacity of steel slag is relatively low, and the nitrogen adsorption capacity varies little across the entire range of relative pressure (P/P_0), indicating that its internal pore structure is underdeveloped and gas adsorption sites are limited. In contrast, the N_2 adsorption capacity of the leaching residue increased significantly, with a particularly rapid rise observed in the high relative pressure region ($P/P_0 > 0.8$). This suggests that a large amount of pore structure capable of adsorbing gas has formed in the material after leaching, exhibiting more pronounced mesoporous characteristics. This change may be related to the dissolution of certain easily soluble mineral phases during the citric acid leaching process, which disrupts the original dense mineral

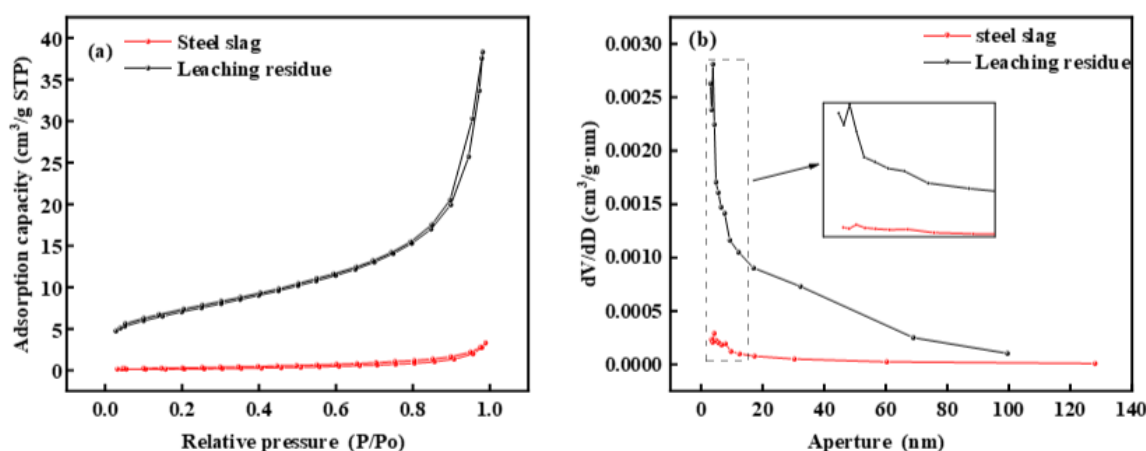


Fig. 14. N_2 adsorption-desorption isotherms (a) and BJH pore size distribution plots (b) for steel slag and leaching residue

structure and contributes to the formation of pore channels and exposed surfaces. As shown in Fig. 14(b), the pore sizes of both steel slag and leaching residue are primarily distributed in the 2–50 nm range; however, the peak of the pore size distribution for the leaching residue is significantly higher than that of the steel slag, and it contributes a greater pore volume in the small-pore size range (approximately 2–20 nm), indicating that the leaching treatment promotes the formation of microporous and mesoporous structures. At the same time, the leaching residue retains a certain pore volume across a wide range of pore sizes, indicating that the dissolution of minerals during the leaching process not only created new pores but also improved the connectivity of the existing pore network. In contrast, the pore size distribution curve for steel slag is generally lower, and its pore structure is relatively simple, consisting mainly of a small number of primary pores.

The total micropore volume, specific surface area, and average pore diameter of steel slag and leaching residue are shown in Table 4.

Table 4. Comparative analysis of pore structure parameters of the steel slag experimental sample and leaching residue

	Total volume of micropores (cm ³ g ⁻¹)	Specific surface area (m ² g ⁻¹)	Average aperture (nm)
Steel slag sample	0.00511	0.87	23.6
Leaching residue	0.0593	25.23	9.4

As shown in Table 4, the specific surface area increased from 0.87 m² g⁻¹ for the steel slag sample to 25.23 m² g⁻¹ for the leaching residue, indicating substantial changes in the overall surface and pore structure of the solid phase. These changes may result from the combined effects of mineral phase dissolution and the formation of secondary products such as calcium citrate. This indicates that the leaching process significantly altered the pore structure of the solid phase and increased the specific surface area. At the same time, the average pore diameter of the leaching residue decreased from 23.6 nm to 9.4 nm, suggesting that the leaching process caused changes in the original mineral structure and promoted the formation of a more abundant microporous and mesoporous structure. This change may be related to the destruction of the original structure and the formation of pore channels caused by the dissolution of mineral phases during the leaching process; at the same time, the formation and deposition of secondary products such as calcium citrate may also have an impact on the surface and pore structure of the leaching residue. Therefore, the increase in the specific surface area of the leaching residue cannot be simply attributed to the dissolution of the original steel slag mineral framework, but should be regarded as the result of solid-phase structural changes caused by the combined effects of mineral phase dissolution and the formation of secondary products.

4. Dynamic analysis

4.1. Leaching kinetic method

To identify the rate-limiting step in the leaching process, the experimental data were fitted using the Shrinking Core Model (SCM) (Chen, 2025), examining three kinetic models: external diffusion control (see Equation 7), chemical reaction control (see Equation 8), and internal diffusion (mixing) control (see Equation 9):

$$\eta = k_d \cdot t \quad (7)$$

$$1 - (1 - \eta)^{1/3} = k_c \cdot t \quad (8)$$

$$\frac{1}{3} \ln(1 - \eta) + (1 - \eta)^{1/3} - 1 = k_m \cdot t \quad (9)$$

Here, η represents the leaching efficiency, t is the reaction time (min), and k is the apparent rate constant under different models. After determining the best-fitting model, the apparent activation energy E_a is calculated using the Arrhenius equation (see Equation 10) based on the rate constants k obtained at different temperatures:

$$\ln(k) = \ln(A) - \frac{E_a}{RT} \quad (10)$$

Here, E_a represents the apparent activation energy (kJ mol^{-1}), R is the gas constant ($8.314 \text{ J mol}^{-1} \cdot \text{K}^{-1}$), T is the thermodynamic temperature (K), and A is the frequency factor.

By comparing the correlation coefficients (R^2) and apparent rate constants from linear fits under different models, it is possible to determine the rate-limiting step of the leaching process under various operating conditions.

4.2. Leaching kinetics

Leaching kinetics experiments were conducted at 20, 30, 40, and 50 °C under conditions of 1 mol L^{-1} citric acid concentration and a liquid-to-solid ratio of 10 mL g^{-1} ; the resulting curves showing the change in Cr leaching efficiency over time are presented in Figure 15a. Based on the Shrinking Core Model (SCM), the experimental data were linearly fitted using diffusion-controlled, interfacial chemical reaction-controlled, and mixed-control models, respectively (Fig. 15b–d).

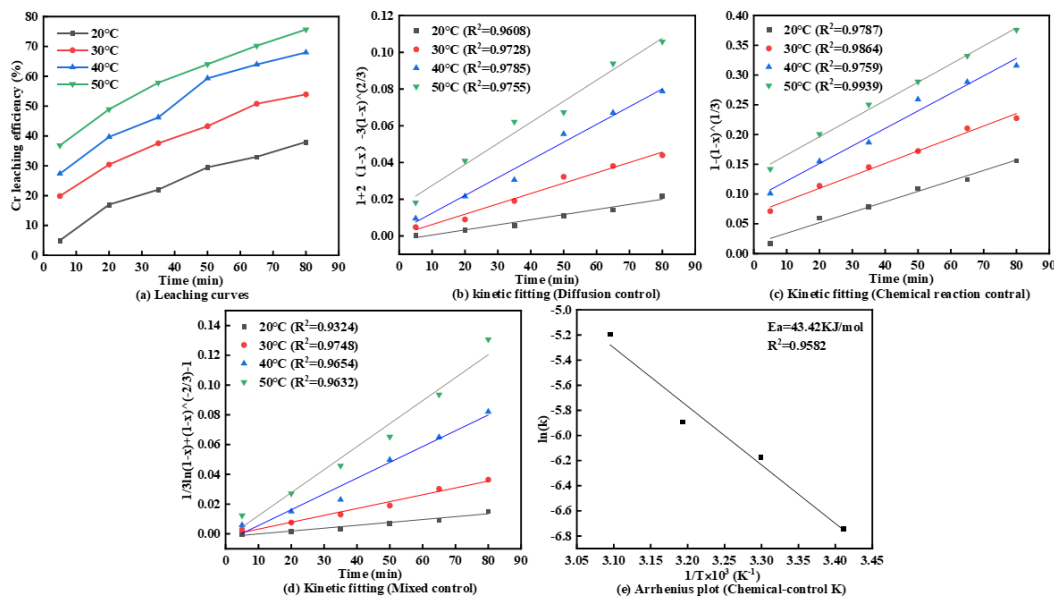


Fig. 15. Kinetics plot for steel slag

The fitting results show that the chemically controlled model achieved higher goodness-of-fit ($R^2 = 0.9759\text{--}0.9939$) at all temperatures, outperforming the diffusion-controlled ($R^2 = 0.9608\text{--}0.9785$) and mixing-controlled ($R^2 = 0.9324\text{--}0.9748$) models. This indicates that, within the time scale and temperature range examined in this study, the Cr leaching process can be approximated as an apparent interfacial reaction-controlled process, suggesting that the leaching efficiency is primarily governed by interfacial reactions between citric acid and chromium-bearing minerals, rather than being limited by liquid film diffusion. Since citric acid can rapidly complex with the eluted Cr^{3+} , reducing the mass transfer resistance of the liquid film, the rate-limiting steps primarily involve the breaking of Cr-O bonds on the mineral surface and interfacial chemical reactions.

To further evaluate the effect of temperature on the leaching efficiency, the apparent rate constants obtained from the chemical reaction rate-control model were used to analyze the relationship between the rate constants and reaction temperature using the Arrhenius equation: Plotting $\ln k$ against $1/T$ (Figure 15(e)) yielded a good linear relationship ($R^2 = 0.9582$). The calculation results show that the apparent activation energy for Cr leaching is $E_a = 43.42 \text{ kJ/mol}$. This value exceeds 40 kJ/mol , falling within the typical range for chemically controlled reactions, which further corroborates that the leaching process is primarily controlled by interfacial chemical reactions, consistent with the results of the kinetic model fitting.

5. Conclusions

- (1) Converter steel slag consists primarily of elements such as Ca, Fe, and O, and contains components such as Si, Mg, Al, and Cr. The main mineral phases include dicalcium silicate (Ca_2SiO_4), tricalcium

silicate (Ca_3SiO_5), calcium ferrite ($\text{Ca}_2\text{Fe}_2\text{O}_5$), calcium aluminate ($\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$), the RO phase, spinel (FeCr_2O_4), and magnetite (Fe_3O_4), among others. In this context, Cr does not exist as an independent mineral but is incorporated into iron-rich oxide phases during the high-temperature smelting and cooling processes in the converter. It is primarily found within the structures of iron-bearing minerals such as the RO phase and Fe-Cr spinel, exhibiting characteristics of co-occurrence with Fe. This provides the mineralogical basis for the subsequent simultaneous leaching of Cr and Fe in the citric acid system.

Under conditions of 1 mol $\cdot$ L⁻¹ citric acid concentration, 80 min elution time, a liquid-to-solid ratio of 10 mL $\cdot$ g⁻¹, and a temperature of 50 °C, the leaching efficiency of Cr, Fe, and Ca were 75.68%, 76.67%, and 2.6%, respectively. Under these conditions, the residual Cr content in the leaching residue was 464.75 mg/kg, which is well below the national risk control limit (1,300 mg/kg) and meets the standards for agricultural land soil (Ministry of ecology and environment & state administration for market regulation, 2018).

- (2) Calcium-containing, readily soluble phases in steel slag—such as dicalcium silicate, tricalcium silicate, and calcium ferrite—react preferentially with citric acid. The released Ca^{2+} combines with citrate ions to form calcium citrate precipitate, and some of this precipitate further reacts with CO_2 in the air through carbonation to form calcium carbonate; The dissolution of the calcium phases disrupts the original dense mineral structure of the steel slag, significantly reducing particle size. The specific surface area of the leached slag increases from 0.87 m² $\cdot$ g⁻¹ in the original slag to 25.23 m² $\cdot$ g⁻¹, and the degree of porosity develops substantially, creating conditions for contact reactions with the internally entrapped chromium phases. Due to the constraints of the stable crystal lattice structure of chromium-iron spinel, only a portion of the chromium is leached; the leached Cr^{3+} forms stable chelates with citrate ions and enters the liquid phase. The Fe-Cr spinel that remains undissolved in the leaching residue is the key factor limiting further improvements in the chromium leaching efficiency.
- (3) The citric acid leaching of chromium (Cr) from steel slag follows the shrinking-core model. Kinetic analysis indicates that the leaching process is primarily controlled by interfacial chemical reactions, and the reaction rate increases with rising citric acid concentration, temperature, and time. Calculations using the Arrhenius equation yielded an apparent activation energy (E_a) for Cr leaching of 43.42 kJ/mol, which falls within the range controlled by chemical reactions. This indicates that the interfacial reaction between citric acid and the mineral phases in the steel slag is the key step controlling the leaching efficiency, while liquid-phase diffusion exerts a relatively weak limiting effect on the overall leaching process. The relatively high activation energy of the reaction indicates that chromium in steel slag is primarily bound in structurally stable iron-bearing mineral phases, such as Fe-Cr spinel, and its release requires processes such as the destruction of the mineral structure, the dissolution of metal ions, and citrate complexation. Therefore, enhancing the chemical reaction between citric acid and the surface of steel slag particles can effectively promote the leaching of chromium and iron, thereby improving the removal efficiency of harmful elements during the resource recovery process of steel slag.

Acknowledgments

Hebei Provincial Science and Technology Program Special Project (Project No.: 25363801D, funded by the Yanzhao Iron and Steel Laboratory), Tangshan Municipal Science and Technology Program Project (Project No.: 25150205E), and Hebei Provincial Natural Science Foundation Key Project (Project No.: E2025209186).

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