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Comparative Study of Scandium Leaching from Coal Ash with Mineral and Organic Acids

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ABSTRACT

Scandium (Sc) is a critical element with applications in high-performance alloys and energy technologies. Yet, its recovery from secondary sources such as coal ash remains challenging due to low concentrations and strong association with refractory aluminosilicates. This study evaluated the leaching behavior of Sc from coal ash using mineral (HCl, H_2SO_4) and organic (citric, malic, oxalic, tartaric) acids under identical conditions (80 °C, 0.5 M, solid-to-liquid ratio 1:20). Mineral acids achieved the highest extraction yields (82-90%). Still, they exhibited poor selectivity, with extensive co-dissolution of Fe and Al. Among the organic acids, citric acid provided the best balance between recovery and selectivity, yielding up to 68% Sc with Fe and Al dissolution below 26%. A complete factorial design varying citric acid concentration (0.1-1.0 M) and pulp density (25-100 g/L) revealed that both efficiency and selectivity could be tuned, with optimal conditions (0.5 M, 25 g/L) resulting in 64% Sc recovery and selectivity factors Sc/Fe = 6.40, Sc/Al = 3.76.

1. Introduction

Scandium (Sc) is a critical element widely sought after for its application in advanced materials, including solid oxide fuel cells, aluminum-scandium alloys, and phosphor technologies. However, its global supply remains limited and is often tied to by-products of uranium or rare earth element mining [1-3]. In recent years, coal ash has emerged as a promising secondary resource for Sc, particularly in regions with large-scale coal combustion waste. Yet, Sc in coal ash is typically present at low concentrations (10-120 ppm) and is strongly incorporated within refractory aluminosilicate matrices, making its recovery technically challenging [4-6].

Conventional recovery of Sc from coal ash still depends mainly on strong mineral acids-most commonly hydrochloric or sulfuric. Although these lixiviants readily dissolve Sc, they also non-selectively solubilize substantial amounts of matrix elements such as iron and aluminum [7-10]. For instance, high-strength HCl at elevated temperature can co-extract ~66% Al and ~91% Fe together with Sc (and other REEs), producing highly multicomponent liquors [11]. This complexity complicates downstream separation and forces additional purification/precipitation steps, driving up reagent usage and operating costs. Beyond process complexity, the lack of selectivity poses clear environmental and economic risks. Large volumes of acidic waste streams must be neutralized and disposed of, adding treatment expense. Meanwhile, excess Fe and Al in solution interfere with solvent extraction, adsorption, and ion-exchange, lowering both the efficiency and selectivity of Sc recovery [12].

As a result, organic acids have gained attention as alternative leaching agents, offering the dual advantage of moderate leaching strength and metal selectivity via complexation [13-18]. Among them, citric acid has shown particular promise due to its biodegradable, non-toxic nature [19-22]. Previous studies have demonstrated the potential of citric acid and other carboxylic acids for recovering REEs and Sc from a variety of waste streams, including coal ash, bauxite residue, and metallurgical slag [23,24]. For instance, column leaching experiments using citric acid as the lixiviant have shown enhanced REE recovery from coal ash, with selectivity controlled by pH and ligand strength. Electrodialytic remediation with citric acid achieved up to 40% REE recovery

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from coal ash, and the process proved to be energy-efficient and environmentally friendly. Comparative studies have found that citric acid, dl-malic acid, and oxalic acid improve the yield of high-value metals, such as Sc and heavy REEs, mainly due to their strong complexing ability and effectiveness in detaching metal ions from refractory glassy or aluminosilicate frameworks. Other research has highlighted that using organic acids can reduce co-leaching of iron and aluminum compared to mineral acids, simplifying purification and reducing secondary waste. However, systematic kinetic and selectivity data for coal ash under controlled leaching conditions remain limited. Moreover, comparisons between organic and mineral acids under unified operating conditions are rarely explored.

The present study aims to evaluate the leaching behavior of Sc from coal ash using both organic (citric, oxalic, malic, and tartaric) and mineral (HCl and H_2SO_4) acids under identical temperature, concentration, and solid-to-liquid ratio. A particular focus is given to time-dependent extraction, co-dissolution of iron and aluminum, and resulting selectivity indices. Based on these results, citric acid is further optimized via a factorial experimental matrix to explore the trade-off between efficiency and selectivity.

2. Materials and Methods

2.1. Materials

A representative batch of CA was obtained from the ash disposal site of the CHPP-2 power plant in Almaty, Kazakhstan. In total, about 5 kg of CA was collected directly from the dump. Sampling was performed using inert, sterile containers to avoid external contamination.

Analytical-grade organic acids were employed as leaching agents: citric acid ($C_6H_8O_7$), DL-malic acid ($C_4H_6O_5$), oxalic acid ($C_2H_2O_4$), and tartaric acid ($C_4H_6O_6$). Concentrations in the range 0.1-1.0 mol/L were investigated. For comparison, hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) were used as reference mineral acids. All solutions were prepared with distilled water.

2.2. Leaching experiments

Leaching experiments were carried out in a 1 L round-bottom glass reactor equipped with a thermometer. The reactor was charged with 500 mL of 1 M citric acid solution and placed on a magnetic stirrer (IKA RT 5, Germany) to reach the desired temperature. Once the target temperature was achieved, 12.5-50 g of coal ash sample was introduced, corresponding to a solid-to-liquid ratio of 80-200 g/L. Throughout the experiments, the suspension was agitated at 400-600 rpm. At one-hour intervals, aliquots of the leachate were withdrawn using a micropipette and analyzed to determine the concentrations of Fe, Al, and Sc. The leaching efficiency was expressed as the recovery of the target metal (E), calculated according to the following equation:

$$E = \frac{m_1}{m_0} * 100\% \tag{1}$$

Here, m_1 represents the mass of Fe, Al, or Sc in the solution, and m_0 is the mass of Fe, Al, or Sc in the initial solid sample.

Selectivity factors ($S_{Sc/Fe}$ and $S_{Sc/Al}$) were defined as:

$$S_{Sc/Fe} = \frac{E_{Sc}}{E_{Fe}} \tag{2}$$

$$S_{Sc/\mathrm{Al}} = \frac{E_{Sc}}{E_{\mathrm{Al}}} \tag{3}$$

In all leaching experiments, the solid-to-liquid (S/L) ratio was precisely controlled and expressed both as mass concentration (g/L) and as a conventional ratio (solid:liquid). Specifically, coal ash samples were added in the range of 12.5-50 g per 500 mL of lixiviant solution, which corresponded to pulp densities of 25, 50, and 100 g/L (equivalent to S/L ratios of 1:40, 1:20, and 1:10, respectively). This dual representation was adopted to ensure consistency and reproducibility across different reporting standards. The selection of the contact time was based on preliminary kinetic tests conducted over a duration of 0-24 h. The dissolution curves demonstrated that scandium recovery exhibited a rapid increase within the first 3-4 h, followed by a gradual approach to a plateau at approximately 6 h. Beyond this duration, no significant enhancement in Sc extraction was observed. Therefore, 6 h was chosen as the representative contact time for comparative evaluation of mineral and organic acids under fixed temperature and concentration conditions. To ensure statistical reliability, each leaching experiment was performed in triplicate under identical conditions. The reported values of scandium, iron, and aluminum recovery represent the arithmetic mean of three independent runs, with standard deviations calculated to reflect data variability.

2.3. Analytical Techniques

Phase identification of solid samples was performed using X-ray diffraction (XRD) with a DW-27 Mini diffractometer (DFMC, Dandong, China) equipped with a CuKα-radiation source operating at 40 kV and 40 mA. Elemental composition of both solid and liquid samples was analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES, Optima 8300, PerkinElmer, USA). Before analysis, solid samples underwent alkaline fusion followed by acid digestion with concentrated nitric acid in a Tank-Eco microwave digestion system (Sineo, Shanghai, China). Scanning electron microscopy (SEM) was performed using a Quanta 200i 3D microscope (FEI Company, USA).

3. Results and Discussion

3.1. Characterization of initial coal ash sample

The bulk chemical composition of the raw coal ash was (wt.%): SiO_2 – 61.34; Al_2O_3 – 25.72, Fe_2O_3 – 9.51, CaO – 4.02, MgO – 1.59. Minor constituents detected in the sample were as follows (ppb): Sc – 16347, Ce – 55682, Y – 27582, La – 21959.

The XRD pattern of the coal ash sample is shown in Fig. 1. The major crystalline constituents of the sample were determined to be mullite, quartz, and maghemite. These phases are characteristic of coal combustion residues and indicate the high-temperature transformation of aluminosilicate and iron-bearing minerals during firing [25, 26].

SEM observations revealed that the coal ash consists of a heterogeneous mixture of angular particles, glassy fragments, and occasional spherical grains typical of ash morphology (Fig. 2a). At higher magnification (Fig. 2b), the surfaces of individual particles appear rough and porous, with fine crystallites deposited on larger agglomerates. Such morphology is consistent with the mineralogical composition identified by XRD: mullite generally forms elongated and angular grains, and quartz occurs as dense fragments with smooth surfaces. In contrast, iron oxides such as maghemite tend to appear as fine particles covering the glassy matrix.

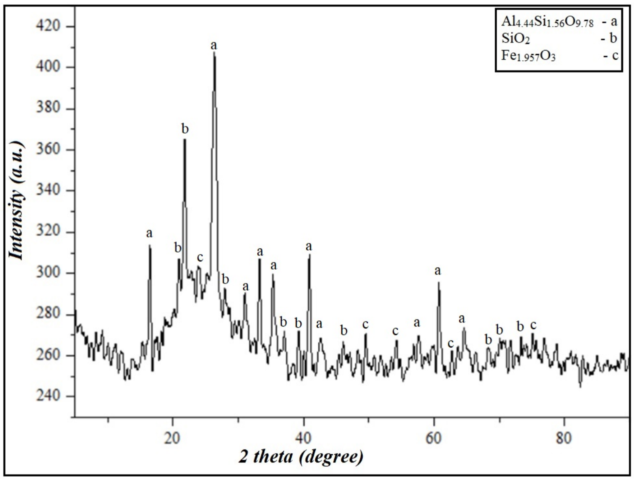

Fig. 1. XRD patterns of coal ash sample.

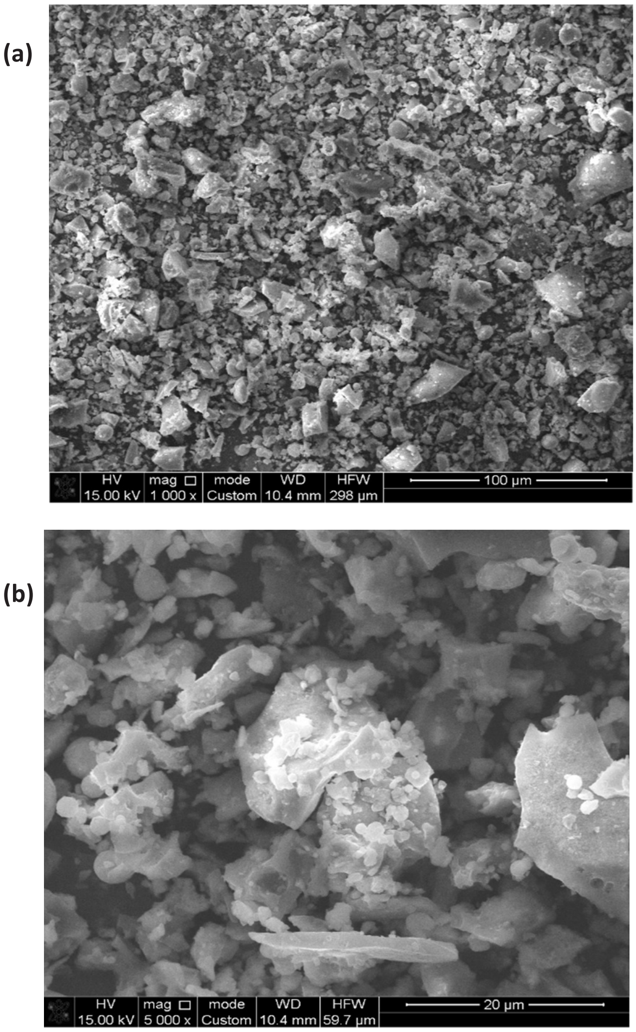

Fig. 2. SEM micrographs of the coal ash sample at 1000× (a) and 5000× (b) magnification.

The cumulative particle size distribution curve of the coal ash sample is illustrated in Fig. 3.

The curve was plotted using a logarithmic scale for particle size to represent the wide range of fractions present better. The sample exhibited a characteristic distribution with D_{10} ≈ approximately 5 µm, D_{50} ≈ approximately 30 µm, and D_{90} ≈ approximately 120 µm. Such values indicate the predominance of fine particles, accompanied by a smaller fraction of coarser grains. The broad distribution reflects both the rapid quenching of molten droplets during combustion and the presence of agglomerated mineral phases.

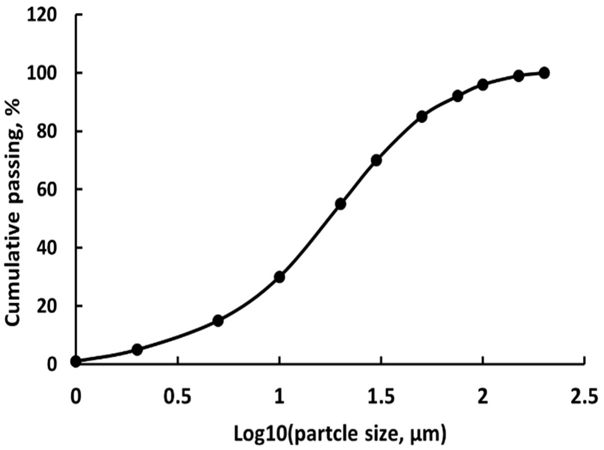

Fig. 3. Cumulative particle size distribution curve for coal ash.

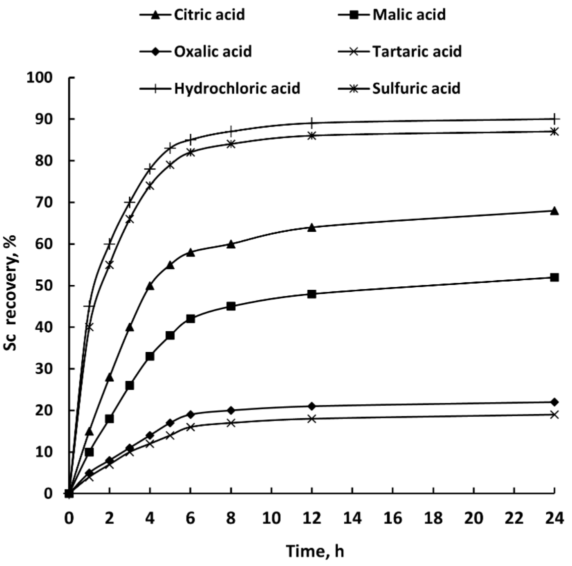

Fig. 4. Effect of acid nature and leaching duration on Sc recovery into solution (0.5 M acid, S:L = 50 g/L, 80 °C).

3.2. Leaching behavior

Four organic acids-citric, malic, oxalic, and tartaric-were selected for investigation, together with hydrochloric and sulfuric acids as mineral references. The organic acids were chosen because they are biodegradable, widely available, and differ significantly in molecular structure and chelating ability [27]. Citric and malic acids contain multiple carboxyl and hydroxyl groups capable of forming stable complexes with scandium. In contrast, oxalic and tartaric acids are known to strongly interact with Fe and Al, which makes their performance less predictable but important to compare [28]. Hydrochloric and sulfuric acids, in turn, were included as benchmarks representing conventional non-selective leaching with high overall efficiency. To ensure comparability across all lixiviants, the main process parameters were fixed at 80 °C, 0.5 M acid concentration, and a solid-to-liquid ratio (S/L) of 1:20 (50 g/L). These conditions were selected as a first approximation: 80 °C accelerates dissolution kinetics without introducing evaporation losses, 0.5 M ensures sufficient proton and ligand availability while avoiding excessive matrix attack, and 1:20 provides measurable concentrations in solution without overloading the liquid phase. Time-dependent tests (0-24 h) were then conducted to determine the kinetics of scandium release and to identify the plateau region for each acid.

Figure 4 shows the dependences of Sc recovery on the acid nature and the duration of leaching.

All acids exhibited rapid initial dissolution within the first 3-4 h, followed by a slower approach to a plateau. Among the organic acids, citric acid demonstrated the highest recovery, reaching ~68% after 24 h, whereas malic acid provided moderate extraction (~52%). Oxalic and tartaric acids were much less effective, with final recoveries of only ~22% and ~19%, respectively. In contrast, the reference mineral acids showed markedly higher efficiencies: hydrochloric acid extracted up to 90% of Sc, and sulfuric acid extracted ~87% after 24 h.

The kinetic profiles (Fig. 4) indicate that Sc dissolution approaches near-plateau values after approximately 6 h of leaching for all acids. Beyond this point, only minor additional extraction was observed, suggesting that 6 h can be considered a representative contact time for comparative analysis. Therefore, this duration was selected for further evaluation of the co-dissolution of Fe and Al, and for calculating the selectivity factors SSc/Fe and SSc/Al to compare the different lixiviants. As can be seen, Sc dissolution plateaued after 6 h, thus this duration was selected for comparative evaluation.

The dissolution behavior of Sc, Fe, and Al in different leaching systems is illustrated in Fig. 5, obtained at 80 °C, 0.5 M acid concentration, and a S/L=50 g/L after 6 h of leaching.

Among the organic acids, citric acid yielded the highest Sc extraction (~58%), while malic acid reached approximately 42%, and oxalic and tartaric acids remained below 20%. In contrast, hydrochloric and sulfuric acids achieved significantly higher Sc recoveries of roughly 85% and 82%, respectively, but were accompanied by extensive co-dissolution of Fe (~80%) and Al (~85%). With the organic acids, Fe and Al dissolution did not exceed 20–26%.

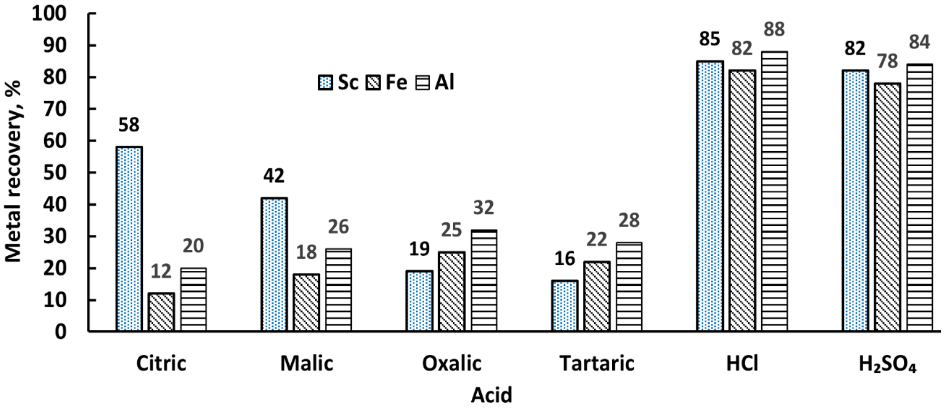

Fig. 5. Dissolution of Sc, Fe, and Al from coal ash in different acids (80 °C, 0.5 M, S:L = 50 g/L, 6 h).

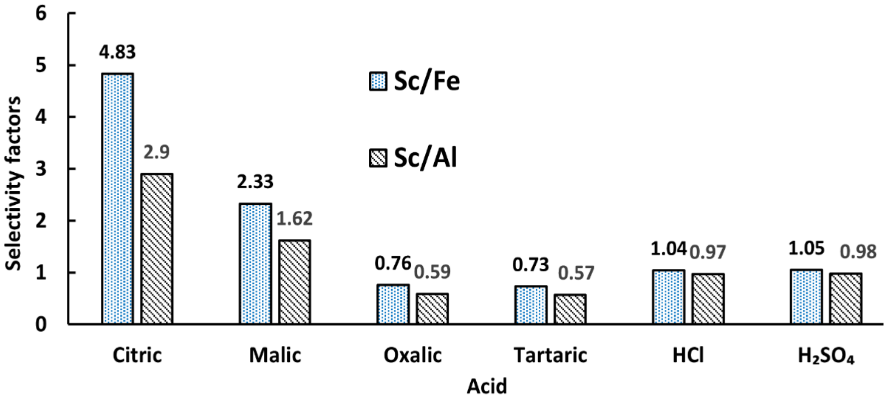

Fig. 6. Selectivity factors Sc/Fe and Sc/Al for coal ash leaching in different acids (80 °C, 0.5 M, S:L = 50 g/L, 6 h).

Based on the dissolution data, selectivity factors were calculated to evaluate the preferential recovery of scandium over matrix elements. The results are summarized in Fig. 6.

Citric acid exhibited the highest selectivity (Sc/Fe ≈ 4.8; Sc/Al ≈ 2.9), clearly outperforming the other organic acids. Malic acid showed moderate selectivity (Sc/Fe ≈ 2.3; Sc/Al ≈ 1.6), whereas oxalic and tartaric acids yielded values below unity, due to the preferential dissolution of matrix elements over Sc. The mineral acids, despite their high absolute Sc recovery, demonstrated selectivity close to one, confirming their non-discriminative nature [29,30]. These results emphasize that citric acid offers the most favorable balance between Sc recovery and suppression of Fe and Al dissolution among the tested lixiviants.

3.3. Factorial optimization of Sc leaching with citric acid

After identifying citric acid as the most promising lixiviant due to its balance between extraction efficiency and selectivity, a focused factorial optimization was conducted to evaluate further the effects of two key process parameters: acid concentration and solid-to-liquid (S/L) ratio. A full 3×3 experimental matrix was implemented, varying citric acid concentration (0.1, 0.5, and 1.0 M) and S/L ratio (25, 50, and 100 g/L), while keeping temperature (80 °C) and contact time (6 h) constant. This specific range of values was selected based on practical considerations, including maintaining moderate reagent consumption, ensuring a manageable pulp density, and maximizing the mobility of scandium within the organic acid medium. Each experiment was evaluated by measuring Sc extraction into the solution, along with the simultaneous dissolution of iron and aluminum, two major interfering elements. From these data, selectivity indicators (Sc/Fe and Sc/Al ratios) were calculated to assess the trade-offs between recovery and purity. The results of this factorial design are summarized in Table 1.

The results presented in Table 1 revealed that Sc extraction improved significantly with increasing acid concentration and decreasing pulp density. The highest scandium recovery of 73% was achieved at an acid concentration of 1.0 M and a solid-to-liquid

Table 1. Experimental matrix for the 3×3 full factorial design using citric acid (80 °C, 6 h)

Run	Citric acid concentration, M	S/L, g/L	Sc extraction, %	Fe extraction, %	Al extraction, %	Sc/Fe	Sc/Al
1	1.0	50	65	16	26	4.06	2.50
2	0.1	50	35	8	14	4.38	2.50
3	0.5	25	64	10	17	6.40	3.76
4	0.1	100	27	11	18	2.45	1.50
5	1.0	25	73	15	24	4.87	3.04
6	0.1	25	39	5	10	7.80	3.90
7	0.5	50	58	12	20	4.83	2.91
8	0.5	100	48	14	23	3.43	2.09
9	1.0	100	53	17	28	3.12	1.89

ratio (S/L) of 25 g/L. In contrast, the lowest yield of 27% was observed under the least favorable condition of 0.1 M and 100 g/L. The experimental parameters also influenced selectivity trends. The Sc/Fe and Sc/Al ratios peaked at 7.80 and 3.90, respectively, under 0.1 M acid and S/L = 25 g/L, showing reduced co-dissolution of iron and aluminum at lower acid strength. However, this came at the cost of overall scandium recovery. Optimal trade-offs between efficiency and selectivity were observed in mid-level conditions (e.g., 0.5 M, S/L = 25 g/L), yielding 64% Sc with Sc/Fe = 6.40 and Sc/Al = 3.76.

3.4. Mechanistic consideration

Scandium leaching from coal ash using organic acids is widely understood to proceed via ligand-promoted dissolution, in which the acid acts both as a proton donor and a chelating agent. Among various ligands, citric acid has been shown to form highly stable complexes with Sc^{3+} due to its three carboxyl groups and one hydroxyl group, resulting in efficient mobilization of Sc even from refractory matrices such as mullite and amorphous aluminosilicates [31]. The general mechanism involves the surface complexation of Sc-bearing phases followed by detachment into solution as soluble citrate complexes. In contrast to strong acids like HCl or H_2SO_4, which indiscriminately dissolve both target and matrix elements, organic acids enable selective extraction, particularly at near-neutral pH, where iron and aluminum tend to precipitate or remain sparingly soluble. The poor dissolution of Fe and Al in our study is consistent with previous work [32]. The slow and progressive nature of Sc dissolution observed in many systems suggests that leaching is controlled not by fast surface protonation, but rather by the gradual deconstruction of complex aluminosilicate frameworks, followed by ligand exchange and the detachment of Sc^{3+}. This is supported by the fact that Sc in coal ash is often associated with glassy, aluminosilicate, or spinel-type phases, which are only partially disrupted under mild organic acid attack [33, 34]. Moreover, previous spectroscopic studies have shown that Sc forms strong inner-sphere complexes with citrate, oxalate, and other polycarboxylate ligands, which can dominate speciation even in the presence of competing ions, such as Fe^{3+} [35, 36].

Compared to citric acid, oxalic and tartaric acids exhibited markedly lower efficiencies for scandium recovery. This can be attributed to two main factors. First, oxalic and tartaric acids strongly interact with matrix elements such as Fe^{3+} and Al^{3+}, leading to preferential dissolution of these species rather than scandium. Their high affinity toward iron and aluminum oxides results in competitive complexation, which reduces the effective mobilization of Sc^{3+} ions. Second, the coordination chemistry of these acids is less favorable for scandium. While citric acid possesses three carboxyl groups and one hydroxyl group capable of forming stable chelating rings with Sc^{3+}, oxalic and tartaric acids have fewer coordination sites and lower stability constants for Sc-ligand complexes. As a result, they cannot efficiently disrupt the refractory aluminosilicate framework and promote scandium solubilization. Consequently, scandium recovery remains limited, and selectivity factors (Sc/Fe, Sc/Al) fall below unity for both acids, highlighting their poor performance relative to citric acid.

4. Conclusions

This study compared the efficiency and selectivity of scandium leaching from coal ash using mineral (HCl, H_2SO_4) and organic (citric, malic, oxalic, tartaric) acids under identical experimental conditions. Mineral acids demonstrated the highest overall scandium recovery (82-90%), but their non-selective behavior resulted in extensive co-dissolution of Fe and Al, complicating downstream separation. In contrast, citric acid provided the most favorable balance between efficiency and selectivity, achieving up to 68% scandium extraction with significantly reduced dissolution of matrix elements. Factorial optimization further demonstrated that scandium recovery and selectivity can be tuned by adjusting the acid concentration and pulp density, with optimal trade-offs achieved at moderate conditions (0.5 M citric acid, S/L = 25 g/L). Mechanistic considerations suggest that citric acid enhances scandium mobilization through the formation of a stable citrate complex, while limiting the solubility of Fe and Al.

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Сравнительное исследование выщелачивания скандия из золы угля минеральными и органическими кислотами

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АННОТАЦИЯ

Скандий (Sc) является стратегически важным элементом, востребованным в производстве высокопрочных сплавов и энергетических технологий, однако его извлечение из вторичных источников, таких как золошлаки, остается затруднительным из-за низких концентраций и прочной связи с упорными алюмосиликатами. В данной работе исследовано поведение скандия при выщелачивании из золошлака с использованием минеральных (HCl, H_2SO_4) и органических (лимонная, яблочная, щавелевая, винная) кислот в идентичных условиях (80 °C, 0,5 M, твердое:жидкое = 1:20). Минеральные кислоты обеспечили наивысшие степени извлечения (82-90%), но показали низкую селективность из-за интенсивного сопутствующего растворения Fe и Al. Среди органических кислот наилучший баланс между эффективностью и селективностью показала лимонная кислота, достигнув до 68% извлечения Sc при растворении Fe и Al менее 26%. Полный факторный эксперимент с варьированием концентрации лимонной кислоты (0,1-1,0 M) и плотности пульпы (25-100 г/л) выявил возможность регулирования как эффективности, так и селективности; в оптимальных условиях (0,5 M, 25 г/л) достигнуто 64% извлечения Sc при коэффициентах селективности Sc/Fe = 6,40 и Sc/Al = 3,76.

Ключевые слова: золошлак, выщелачивание, скандий, лимонная кислота.

Көмір күлінен скандийді минералдық және органикалық қышқылдармен шаймалаудың салыстырмалы зерттеуі

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АҢДАТПА

Скандий (Sc) – жоғары өнімді қорытпалар мен энергия технологияларында қолданылатын маңызды элемент, алайда оны көмір күліндей екіншілік көздерден алу төмен концентрацияға және отқа төзімді алюмосиликаттармен берік байланысына байланысты күрделі болып отыр. Бұл зерттеуде көмір күлінен Sc шаймалау үрдісі минералдық (HCl, H_2SO_4) және органикалық (лимон, алма, қымыздық, шарап) қышқылдар көмегімен бірдей жағдайда (80 °C, 0,5 M, қатты мен сұйықтың қатынасы 1:20) бағаланды. Минералдық қышқылдар ең жоғары экстракция шығымын (82-90%) көрсетті, алайда олардың селективтілігі төмен болып, Fe мен Al-дың кең көлемде қосалқы еріп кетуі байқалды. Органикалық қышқылдардың ішінде лимон қышқылы ең қолайлы нәтиже беріп, шығымдығы 68%-ға дейін жетті, ал Fe мен Al ерігіштігі 26%-дан аспады. Цитрат қышқылының концентрациясын (0,1-1,0 M) және қойыртпақ тығыздығын (25-100 г/л) өзгерте отырып жүргізілген толық факторлық жоспарлау тиімділік пен селективтілікті реттеуге болатынын көрсетті. Оптималды жағдайларда (0,5 M, 25 г/л) 64% Sc қалпына келтірілді, ал селективтілік коэффициенттері Sc/Fe = 6,40 және Sc/Al = 3,76 мәндерін көрсетті.

Түйін сөздер: көмір күлі, шаймалау, скандий, лимон қышқылы.