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Removal of iron from acidic aluminum chloride solutions by solvent extraction using D2EHPA

D2EHPA kullanılarak asidik alüminyum klorür çözeltilerinden demirin solvent ekstraksiyonu ile uzaklaştırılması

Authors: Mücevher TURAN¹, Muhammed Kürşat AYDEMİR², Mehmet Deniz TURAN³, Murat ERDEMOĞLU⁴

ORCID¹: 0000-0002-5779-4847

ORCID²: 0000-0003-0846-4355

ORCID³: 0000-0002-2136-1425

ORCID⁴: 0000-0003-2922-7965

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Removal of Iron from Acidic Aluminum Chloride Solutions by Solvent Extraction Using D2EHPA

Highlights

- ❖ Iron is a significant contaminant in solution in aluminum leaching.
- ❖ D2EHPA was found to be a selective solvent for iron removal in chlorinated solutions.
- ❖ The amount of iron removed decreased as the concentrations of iron, aluminum, potassium, and sodium in the solution increased
- ❖ With the two-stage solvent extraction, there was 96% Fe removal and 2% Al loss.

Graphical Abstract

In this study, the removal of iron from acidic aluminum chloride solutions by solvent extraction method using di(2-ethylhexyl)phosphoric acid (D2EHPA) was investigated (Figure).

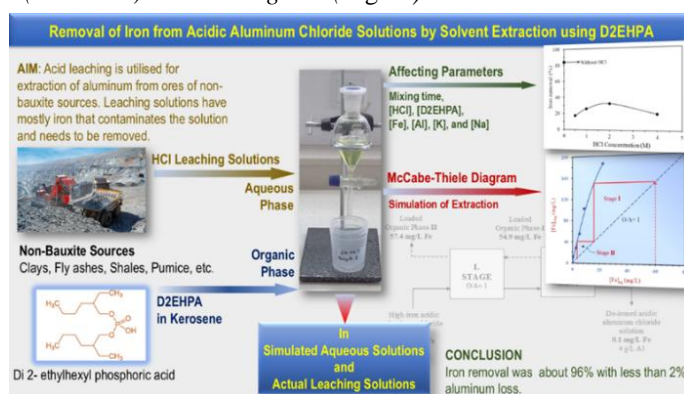


Figure. Graphical diagram of the studies on the iron removal via solvent extraction

Aim

The study aims to remove iron from acidic aluminum chloride solutions to improve the purity of alumina produced from aluminum silicate clays by acid leaching method.

Design & Methodology

Iron was removed from acidic aluminum chloride solutions via solvent extraction using D2EHPA in kerosene. Experiments were conducted with both simulated and actual leach solutions obtained from the process in which an aluminum silicate clay ore was leached with HCl. The study examined the effects of time, phase ratio (O/A), and concentrations of HCl, D2EHPA, Fe, Al, K and Na.

Originality

This study considered both simulated and actual leaching solutions and iron removal from acidic aluminium chloride solutions was achieved for the first time by solvent extraction method.

Findings

The results showed that iron extraction improved with higher concentrations of HCl and D2EHPA, although excess chloride reduced efficiency. According to the McCabe–Thiele diagram, two extraction stages are required for over 91% removal.

Conclusion

In conditions that can be considered optimal, a 30-minute mixing process, a 20% D2EHPA concentration, and a 1:1 O/A ratio for iron removal resulted in a 95.58% success rate.

Declaration of Ethical Standards

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

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Araştırma Makalesi / Research Article

Mücevher TURAN¹, Muhammed Kürşat AYDEMİR^{1*}, Mehmet Deniz TURAN², Murat ERDEMOĞLU¹

¹Faculty of Engineering, Department of Mining Engineering, Inonu University, Türkiye

²Faculty of Engineering, Department of Metallurgical and Materials Engineering, Fırat University, Türkiye

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ABSTRACT

This study presents a method for purifying acidic aluminum chloride solutions, specifically targeting the removal of iron (Fe) impurities to enhance the quality of alumina produced from aluminum silicate clay. The research employed solvent extraction using di(2-ethylhexyl)phosphoric acid (D2EHPA) dissolved in kerosene as the extracting agent. The investigation was conducted using both simulated solutions and actual leach solutions derived from hydrochloric acid processing of clay ore. Key experimental parameters included reaction time, organic-to-aqueous phase ratio (O/A), and the concentrations of HCl, D2EHPA, iron, aluminum, potassium, and sodium. The results demonstrated that the efficiency of iron extraction improved significantly with increased concentrations of both HCl and the D2EHPA extractant. However, the presence of excessive chloride ions was found to have a negative impact on this efficiency. Analysis using a McCabe-Thiele diagram indicated that a high iron removal rate exceeding 91% necessitates a two-stage extraction process. The study identified optimal conditions for this purification: a mixing time of 30 minutes, a D2EHPA concentration of 20%, and an equal organic-to-aqueous phase ratio (O/A = 1:1). Under these precise conditions, the process achieved a remarkable iron removal efficiency of 95.58%, demonstrating its high effectiveness for this specific application.

Keywords: Acidic aluminum chloride solutions, Iron removal, Solvent extraction, D2EHPA

D2EHPA Kullanılarak Asidik Alüminyum Klorür Çözeltilerinden Demirin Solvent Ekstraksiyonu İle Uzaklaştırılması

ÖZ

Bu çalışma, bir alüminyum silikat kil cevherinden elde edilen alüminanın saflığını artırmak amacıyla, asidik alüminyum klorür çözeltilerindeki demir (Fe) gibi safsızlıkların uzaklaştırılması için bir yöntem sunmaktadır. Araştırmada, kerosen içinde çözülmüş di(2-etilheksil)fosforik asidin (D2EHPA) çözücü olarak kullanıldığı bir solvent ekstraksiyon yöntemi uygulanmıştır. Çalışma, hem yapay çözeltiler hem de kil cevherinin hidroklorik asit ile liç ile edilen gerçek liç çözeltileri kullanılarak yürütülmüştür. Anahtar deneysel parametreler arasında reaksiyon süresi, organik-ağır faz oranı (O/A) ve HCl, D2EHPA, demir, alüminyum, potasyum ve sodyum derişimleri yer almaktadır. Sonuçlar, demir uzaklaştırma verimliliğinin hem HCl hem de D2EHPA'nın derişiminin artmasıyla önemli ölçüde iyileştiğini göstermiştir. Bununla birlikte, aşırı klorür iyonu varlığının bu verimlilik üzerinde olumsuz bir etkisi olduğu tespit edilmiştir. McCabe-Thiele diyagramı kullanılarak yapılan analiz, %91'in üzerinde yüksek bir demir uzaklaştırma oranı elde etmek için iki aşamalı bir ekstraksiyon sürecinin gerekli olduğunu işaret etmiştir. Çalışma, bu saflaştırma işlemi için optimal koşulları belirlemiştir: 30 dakikalık bir karıştırma süresi, %20'lik bir D2EHPA konsantrasyonu ve eşit organik-ağır faz oranı (O/A = 1:1). Bu kesin koşullar altında, proses, bu spesifik uygulamaya için yüksek etkinliğini gösteren %95,58'lik kayda değer bir demir uzaklaştırma verimliliğine ulaşmıştır.

Anahtar Kelimeler: Asidik alüminyum klorür çözeltileri, Demir Uzaklaştırma, Çözücü ekstraksiyonu, D2EHPA

1. INTRODUCTION

Aluminum is widely used due to its light weight and alloy-forming ability, becoming increasingly important with technological advances. Today, it's the second most used metal after steel. Although over 270 aluminum minerals exist 40% being aluminum silicates [1], aluminum production mainly relies on bauxite. The Bayer process extracts alumina (Al₂O₃) from bauxite using caustic soda, followed by the Hall-Héroult process to produce pure aluminum via electrolysis.

Alternative sources to bauxite for alumina production have been studied, including coal shales, alunite, nepheline syenite, kyanite, and fly ash from power plants [2-8]. High-alumina clay minerals like kaolinite, pyrophyllite, illite, and muscovite, as well as acidic pumice rock, have also been studied [9-15]. Alumina is typically extracted from these sources using acid leaching, often preceded by thermal [11] or mechanical activation [9, 10, 15] to improve aluminum recovery.

Previous studies have studied impurity removal from acidic leach solutions using solvent extraction, focusing

*Sorumlu Yazar (Corresponding Author)
e-mail : aydemir.kursat@outlook.com

on the effects of various organic solvents. Zhou et al. [16] used tributyl phosphate and 2-octanol to separate Fe^{3+} and Al^{3+} from fly ash leaching solutions. Yi et al. [17] studied Fe^{3+} removal from chloride solutions using a mixed solvent of TBP, 2-octanol, and kerosene. Zhang et al. [18] targeted iron removal from red mud with Aliquat 336, while Sokolov et al. [19] examined iron extraction from bauxite leachates using neutral oxygenated extractants. On the other hand, there are various iron extraction methods that have found their place in industrial applications, such as precipitation methods in the form of hematite, jarosite, and goethite. However, the choice of these methods generally depends on the solution pH, the form in which the iron is present, and the iron concentration in the solution. In this study, the fact that all the iron was in the Fe^{3+} form, the acidic leach solution, and its low concentration suggested that solvent extraction could be used. Nevertheless, the particular challenge of extracting iron by means of solvent extraction from acidic leach solutions of aluminum silicate minerals – a matrix characterized by very high aluminum concentrations – continues to be unaddressed in the extant literature.

The present study utilized the extractive potency of di(2-ethylhexyl)phosphoric acid (D2EHPA), dissolved in kerosene, to selectively target and remove iron from acidic aluminum chloride solutions. The research systematically probed the influence of critical variables on extraction efficiency and selectivity. The parameters that were considered included the concentrations of HCl and D2EHPA, the mixing time, the organic-to-aqueous phase ratio, and the potentially interfering ionic levels of aluminum, iron, potassium, and sodium in the aqueous matrix.

2. MATERIAL and METHOD

2.1. Material

All synthetic solutions for solvent extraction studies were prepared with analytical-grade reagents in accordance to simulate the composition of the actual pregnant leach solution. The aqueous phase was constituted by the dissolving of FeCl_3 in deionized water as the iron source, and $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, KCl , and NaCl to provide aluminum, potassium, and sodium ions, respectively. Hydrochloric acid was used to prepare the acidic solutions. The organic phase consisted of di(2-ethylhexyl)phosphoric acid (D2EHPA) dissolved in kerosene, which served as the extractant for the removal of the iron.

2.2. Method

Iron removal was carried out using both simulated and actual leach solutions based on acidic aluminum chloride media. The simulated solutions were prepared to match the iron and aluminum concentrations of actual leach solutions, which were obtained by leaching pyrophyllite-rich clay (23.6% Al_2O_3 , 0.25% Fe_2O_3 , etc.) in 2–4 M HCl, as described by Erdemoğlu et al. [10]. Table 1 presents iron and aluminum levels in these actual leach solutions at varying HCl concentrations. Although the HCl

concentration of actual leach solutions (ALS) represents a certain value, experimental studies were also conducted under conditions where HCl is not present.

Table 1. Iron and aluminum concentrations of the actual leach solutions

Concentration (g/L)	HCl concentration used for the ore leaching (M)		
	2 (ALS-2)	3 (ALS-3)	4 (ALS-4)
Fe	0.057	0.058	0.064
Al	4.065	4.085	4.295

To understand the mechanism of iron removal by D2EHPA from acidic aluminum chloride solutions, simulated aqueous solutions (SAS) were prepared to mimic the actual leach solutions (ALS) before conducting tests. Two simulated aqueous solutions were prepared with HCl concentrations of 2, 3, and 4 M, each containing varying levels of iron, aluminum, sodium, and potassium (Table 2). SAS-1 was based on average element concentrations in actual leach solutions, while SAS-2 assumed complete dissolution of all elements from the pyrophyllite ore.

Table 2. Content of the simulated aqueous solutions prepared using 2, 3 and 4M HCl

Solution	Concentration, g/L			
	Al^{3+}	Fe^{3+}	K^+	Na^+
SAS-1	4.0	0.06	0.67	0.215
SAS-2	6.245	0.0875	0.67	0.215

Iron extraction was performed using D2EHPA in kerosene, mixed with the aqueous phase at a 1:1 volume ratio. The two phases were combined in a separatory funnel, sealed, and stirred at 600-rpm at room temperature. After mixing, the solution was allowed to settle for 1 hour. Separated phases were collected, and Fe concentrations in the aqueous phase were measured using AAS (Perkin-Elmer A-Analyst 400). Each sample was analyzed at least three times, and the percent iron removal was calculated by comparing initial and final Fe levels (Equation 1). Aluminum content was also analyzed to assess D2EHPA's selectivity.

$$\text{Iron Removal (\%)} = [(W - W_1) / W] \times 100 \quad (1)$$

Where W is the initial iron content (mg) in the aqueous phase, and W_1 is the remaining iron content (mg) after solvent extraction.

The McCabe-Thiele diagram was constructed using data from studies at various organic/aqueous phase ratios. Iron concentrations in both phases were calculated using the equations (2) and (3):

$$W_0 - W_1 = OW \quad (2)$$

$$\text{Iron concentration in the organic phase (g/L)} = \frac{OW}{OV} \times 100 \quad (3)$$

Where, W_0 = initial iron in the aqueous phase (mg), W_1 = remaining iron in the aqueous phase after removal (mg), OW = amount of iron transferred to the organic phase (mg), SV = volume of aqueous solution (mL), OV = volume of organic solution (mL)

3. RESULTS AND DISCUSSIONS

3.1. Effects of the Parameters

3.1.1. HCl concentration

To investigate the effect of HCl concentration, it varied while keeping other variables constant. Iron removal decreased significantly from 83.2% without acid to 17.3% at 0.5 M acid concentration. At 1 M, iron removal increased slightly, but it decreased again at 4 M acid concentration (Figure 1). It is assumed that D2EHPA primarily retains iron in an acid-free medium [20].

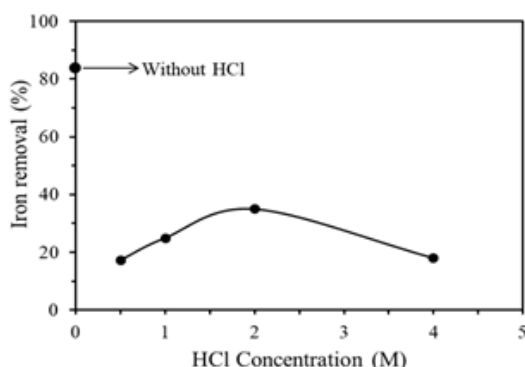


Figure 1. Effect of HCl concentration on the iron removal (Al^{3+} : 4 g/L, Fe^{3+} : 0,06 g/L; D2EHPA in kerosene 20% by volume; mixing time: 2 min.; settling time: 1 h; O/A ratio: 1/1)

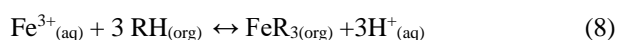
As HCl concentration increases, the concentrations of H^+ and Cl^- ions rise, leading to the formation of various complexes between Fe^{3+} and Cl^- ions [21]. These complexes are shown in reactions 4-7. Biswas and Begum [20] reported that at 0.003 M chloride concentration, Fe^{3+} ions exist. With increasing HCl or Cl^- concentration, complexes like $FeCl_2^+$, $FeCl_2^{2+}$, $FeCl_3$, and $FeCl_4^-$ are formed. $FeCl_2^+$ and $FeCl_3$ are found around 1 M Cl^- , and $FeCl_4^-$ appears at HCl concentrations of 3 M and higher [18-20]. At pH values where iron ions and positively charged iron chloride complexes are present, D2EHPA can extract these species, but $FeCl_3$ and $FeCl_4^-$ cannot be extracted at higher pH.



In solvent extraction studies without HCl, the initial pH was between 3.1 and 3.6. During the experiments, pH measurements of the water ranged from 5.5 to 6.5. The slight pH decreases upon adding D2EHPA is attributed to its weakly acidic nature. In experiments without HCl, the presence of $AlCl_3$ and $FeCl_3$ salts caused the pH to drop from 3.1–3.6 to nearly 2 by the end of the experiment. This decrease is likely due to the hydrolysis of metal salts in water and the extraction process, where cations bind to the organic phase, removing hydronium ions (H_3O^+) from D2EHPA.

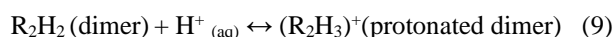
At low concentrations of HCl, species such as Fe^{3+} , $FeCl_2^+$, and $FeCl_2^{2+}$ are extracted by D2EHPA via cation exchange.

The loading reaction is as follows:

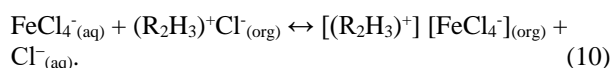


Where, RH is D2EHPA molecule; FeR_3 : is the complex formed by D2EHPA with Fe^{3+} .

D2EHPA is a weak organic acid, often represented in its dimeric form as R_2H_2 . Its protonation in an acidic medium like HCl can be thought of as the reverse of its dissociation [22]. The simplified protonation reaction in HCl solution is:



At high HCl concentrations, the $FeCl_4^-$ anion becomes dominant and then anion-pair extraction occurs with the protonated D2EHPA cation ($(R_2H_3)^+$). The reaction is:



As shown in Figure 1, the highest iron removal occurred without HCl. However, since the aim of this study was to remove iron from acidic aluminum chloride solutions, a 2 M HCl concentration was chosen for further experiments to investigate other parameters.

Reactions 4-10 are collectively shown schematically in Figure 2.

3.1.2. Mixing time and D2EHPA concentration

In experiments investigating the effect of mixing time and D2EHPA concentration, HCl concentration was kept constant at 2 M. It is assumed that increasing HCl concentration negatively affects the functional groups of D2EHPA. When D2EHPA was 5% by volume, iron removal significantly increased at 15 minutes of mixing time, but even at 60 minutes, only about 32% of iron was removed (Figure 3). Mixing time is a key parameter in solvent extraction [23], as it improves extraction kinetics by dispersing the organic phase into small droplets within the aqueous phase, increasing the retention of ferrous

ions and/or positively charged ferric chloride complexes by D2EHPA [24].

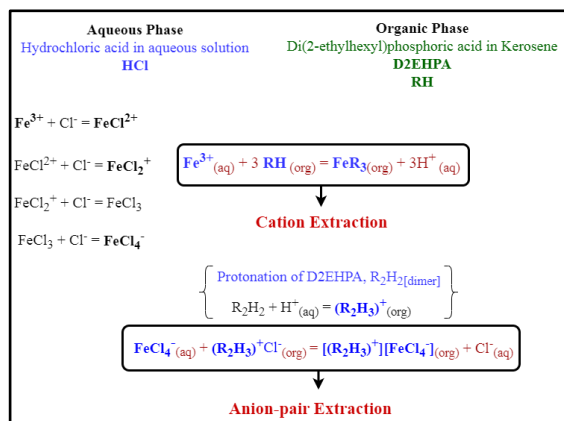


Figure 2. Schematic illustration of the iron extraction mechanisms of the D2EHPA

However, no significant improvement in iron removal was observed with longer mixing times. To address the possibility of insufficient D2EHPA, additional extraction experiments were conducted with varying D2EHPA concentrations. The amount of metal extracted depends on the active extractant concentration in the solution [25].

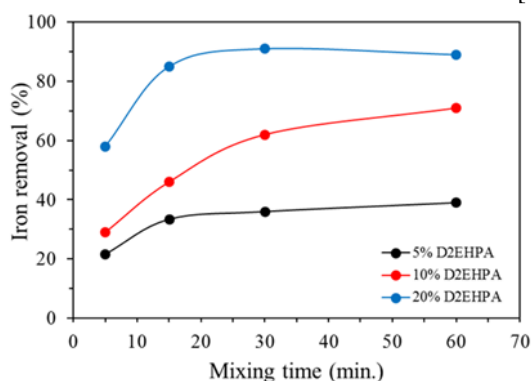


Figure 3. Effect of mixing time on iron removal at different D2EHPA concentrations (4 g/L Al^{3+} , 0.06 g/L Fe^{3+} , 2 M HCl, O/A ratio: 1/1)

In experiments with 10% D2EHPA by volume, iron removal increased with longer mixing times, reaching approximately 71% after 60 minutes. In Figure 3, when 20% D2EHPA was used, 58% of iron was removed after 5 minutes, increasing to 91% after 30 minutes, with no significant change at longer mixing times. Studies have shown that iron removal increases with higher concentrations of D2EHPA [21, 25].

3.1.3. Organics to aqueous phase ratio

In experiments with 20% D2EHPA, the O/A ratio was varied while keeping the total volume constant. Figure 4 shows that as the O/A ratio increases, iron removal also increases, reaching the highest value of 99% at a 5/1 ratio. The phase ratio influences the extraction process by affecting phase distribution and droplet size [26]. However, high O/A ratios can lead to increased solvent consumption and higher costs. A more reasonable O/A ratio of 1, yielding 91% iron removal, was considered more appropriate.

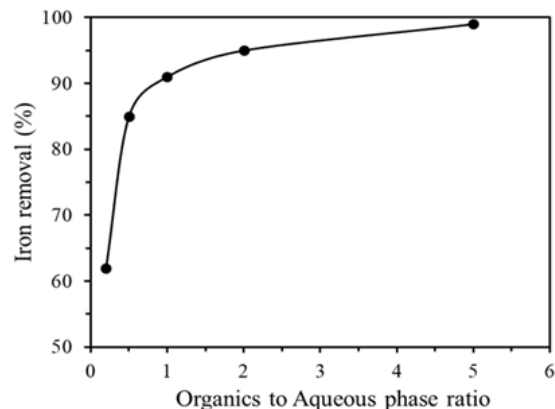


Figure 4. Effect of organics to aqueous phase ratio on iron removal (4 g/L Al^{3+} , 0.06 g/L Fe^{3+} , 2 M HCl, 20% D2EHPA by volume, mixing time: 30 min)

3.1.4. Effects of iron, aluminum, sodium and potassium concentrations

At an iron concentration of 0.06 g/L, 91% iron removal was achieved, but this decreased to approximately 75% at 0.1 g/L (Figure 5). Metal ion concentration is a key parameter that directly affects metal removal in solvent extraction. As shown in equation (11), with constant organic concentration, increasing metal concentration in the aqueous phase decreases the free organic concentration. If other conditions remain the same, increasing metal concentration in the aqueous phase increases metal concentration in the organic phase, but the free organic concentration and removal efficiency decrease [27].

$$(\text{HA})\text{F} = (\text{HA})\text{T} - (\text{M.nA}) \quad (11)$$

Where, (HA)F is free organic concentration, (HA)T is total organic concentration, (M.nA) is metal concentration transferred to the organic phase.

The effect of aluminum, sodium, and potassium concentrations on iron removal was investigated in solutions containing 0.06 g/L iron (Figure 5). When examining the effects of K^+ and Na^+ concentrations, the solution contained 4 g/L Al^{3+} . It was found that iron removal did not change significantly in the presence of aluminum, suggesting that D2EHPA is highly selective for iron removal [28]. The small variations in iron removal with changes in Al concentration are likely due to the increased chloride ion concentration introduced into the aqueous medium with higher aluminum concentrations.

Like Fe, Al has a 3+ valence. However, its extraction behavior with D2EHPA is not the same. Al^{3+} undergoes much stronger hydration than Fe^{3+} and exists in the aqueous phase as $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$. When pH increases, Al^{3+} rapidly converts to $\text{Al}(\text{OH})_x$ species, which have difficulty being extracted with D2EHPA. Fe^{3+} forms iron-chloride complexes that can be extracted with Cl^- ions. The stability constants of Fe^{3+} complexes with D2EHPA are higher than with Al^{3+} . In other words, Fe^{3+}

more easily diffuses into the organic phase under the same conditions. Al^{3+} , with its higher hydration energy, is difficult to dissociate from the aqueous phase. Fe^{3+} has a lower hydration energy and is easier to extract with Cl^- . If the appropriate pH range is not met for Al^{3+} , extraction will not begin and therefore remains unloaded.

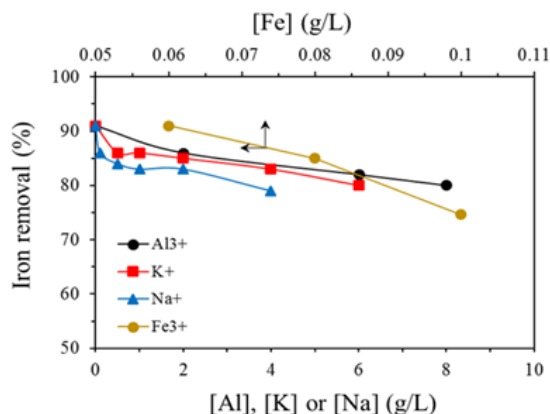


Figure 5. Effect of initial Fe^{3+} , Al^{3+} , K^+ , Na^+ concentration on the iron removal (2 M HCl, 20% D2EHPA by volume, Mixing time: 30 min)

Figure 5 also shows that iron removal decreases with increasing concentrations of sodium and potassium. While iron removal was 91% in the absence of sodium and potassium, it decreased to 80% with 4 g/L Na^+ and 6 g/L K^+ . Since different amounts of AlCl_3 , NaCl , and KCl salts were used to adjust the concentrations of aluminum, sodium, and potassium, the chlorine concentration in the solution also increased. This led to the formation of various chlorinated complex ion species, such as FeCl_2^+ , FeCl_2^+ , FeCl_3 , and FeCl_4^- , with iron ions [18, 20]. It has been reported that the presence of anionic species in the aqueous phase can negatively affect metal extraction [27]. If the stability of the metal complex in the aqueous phase is higher than the stability of the metal-organic complex in the organic phase, the metal will not transfer to the organic phase and will not be extracted [27].

3.2. Iron Removal from Simulated and Actual Solutions

As the concentration of HCl used to prepare the simulated aqueous solution increased, iron removal decreased significantly (Figure 6). For both simulated aqueous solutions prepared in 2 M HCl, iron removal was around 80%, while for solutions prepared in 4 M HCl, iron removal dropped to less than 10%. When comparing the two simulated solutions, iron removal was 86% in the SAS-1 solution, which had a relatively lower metal concentration. Iron removal similarly decreased as the HCl concentration used to leach the pyrophyllite ore increased. In the actual pregnant leach solutions, iron removal was calculated as 95.58% for ALS-2, 90.91% for ALS-3, and 69.69% for ALS-4 (Figure 6).

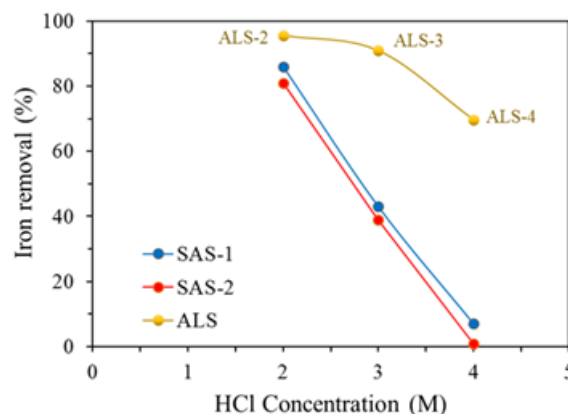


Figure 6. Effect of HCl concentration on the iron removal from the simulated aqueous solutions and the actual pregnant leach solutions

As observed in both simulated aqueous solutions and actual pregnant leach solutions, iron removal decreases significantly with increasing HCl concentration. This is due to the increase in H^+ and Cl^- ions in the medium as HCl concentration increases, and the Fe^{3+} and Cl^- complexes formed cannot be retained by D2EHPA. At 2, 3, and 4 M HCl concentrations, the dominant form of Fe^{3+} ions in the aqueous phase is FeCl_3 , but FeCl_4^- complexes start to form from 3 M HCl [18, 20, 29]. The decrease in iron removal at 3 and 4 M HCl may be due to the inability of these chlorine complexes to be retained by D2EHPA. Iron removal also decreased with increasing iron concentration in both simulated aqueous and actual pregnant leach solutions. These differences are attributed to the varying initial iron concentrations. For SAS-1, the initial iron concentration was 0.06 g/L, and the amount of iron removed was 0.0516 g/L, while for SAS-2, the initial iron concentration was 0.0875 g/L, and the amount of iron removed was 0.0709 g/L.

The initial iron concentrations in the actual leach solutions were 0.0575 g/L, 0.0582 g/L, and 0.0649 g/L for ALS-2, ALS-3, and ALS-4, respectively, and iron removal decreased with the increasing concentration of metal ions. It has been reported that increasing the concentration of various metal ions in the aqueous phase increases the amount of metal transferred to the organic phase, which leads to a decrease in metal removal due to solvent deficiency [27]. Accordingly, depending on the loading capacity of D2EHPA, more iron can be removed. The reason why the iron removal calculated for ALS-2 (95.58%) was higher than that of both artificial solutions (86% for SAS-1 and 81% for SAS-2) is believed to be due to the consumption of chloride salts of the cations, which increased the initial Cl^- concentration in the solution far more than in the actual pregnant leach solutions. This means that the chloride ion concentration in the simulated aqueous solutions was much higher compared to the actual pregnant leach solutions. This high concentration of chlorine ions, along with increasing acid concentration, leads to the formation of negatively charged iron-chloride complexes at higher concentrations, which negatively affect iron removal.

Aluminum loss in the actual leach solution obtained at 2 M HCl, which provided the highest iron removal, was investigated and the results are presented in Table 3. It was found that there was no significant aluminum loss from SAS-1 and ALS-2. The aluminum loss from SAS-2, which has higher initial iron and aluminum concentrations, was calculated to be 1.73%. These results indicate that iron is selectively removed from an acidic solution containing both aluminum and iron with less than 2% aluminum loss.

Table 3. Comparison of iron and aluminum removal from simulated and actual solutions (2 M HCl)

Solution	Initial Iron (g/L)	Initial Aluminum (g/L)	Removal (%)	
			Iron	Aluminum
SAS-1	0.06	4.0	86	0
SAS-2	0.0875	6.245	81	1.73
ALS-2	0.0575	4.065	95.58	0

3.3. Determination of Loading Capacity and Number of Extraction Stages

Loading capacity experiments were performed to determine the amount of iron removed from the leaching solution using a continuous countercurrent solvent extraction process. The process used 30 mL of solution with 20% D2EHPA, a 1:1 O/A ratio, 600 rpm mixing, and a 30-minute contact time. After phase separation, the organic phase was mixed with fresh solution until the iron concentration no longer changed. The results showed that 0.16 g/L of iron was removed in three steps with 20% D2EHPA (Figure 7).

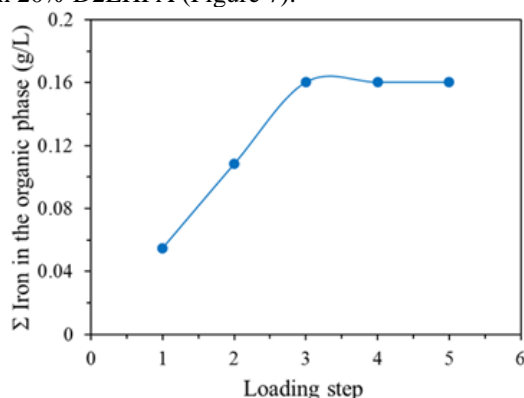


Figure 7. Iron loading capacity of D2EHPA (0.0575 g/L Fe³⁺, 20% D2EHPA by volume, Mixing time: 30 min, Mixing speed: 600 rpm)

Solvent extraction experiments were conducted at different organic-to-aqueous (O/A) phase ratios (1/5, 1/2, 1/1, 2/1, 5/1) using 20% D2EHPA in a 30 mL total volume. The experiments were performed at room temperature, with a 600-rpm mixing speed and a 30-minute contact time. The iron removal data is presented in the McCabe-Thiele diagram shown in Figure 8.

The amount of iron loaded into the organic phase decreased as the O/A ratio increased. At an O/A ratio of 1/5, the iron load was 0.1856 g/L, but this dropped to 0.1019 g/L at a 1/2 ratio and further decreased to 0.0119

g/L at a 5/1 ratio. Interestingly, the amount of iron in the aqueous phase did not increase as expected with the higher O/A ratios; instead, it decreased. This suggests that iron species may have been trapped in the organic phase rather than binding to D2EHPA. Similar findings were reported by Uysal [30] and Serin [31].

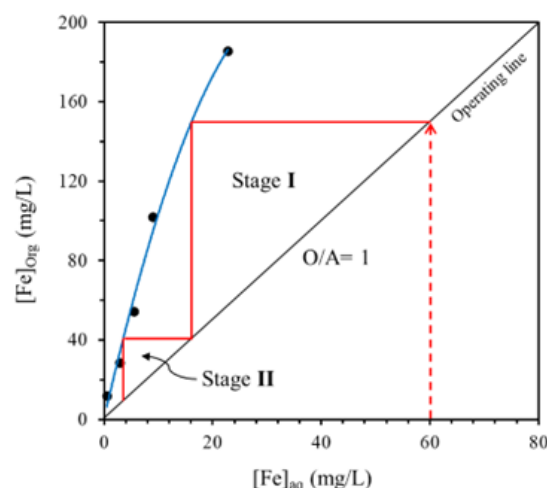


Figure 8. McCabe - Thiele diagram for iron removal from acidic aluminum chloride solutions

The McCabe-Thiele diagram indicates that iron can be removed from acidic alumina chloride solution in two stages using 20% D2EHPA at a 1:1 O/A ratio. A continuous countercurrent process, shown in Figure 9, demonstrates that iron loading to the organic phase occurs in two steps. The majority of iron transitions to the organic phase in the first stage, and the iron concentration in the aluminum-rich, iron-free solution reaches 0.1 mg/L.

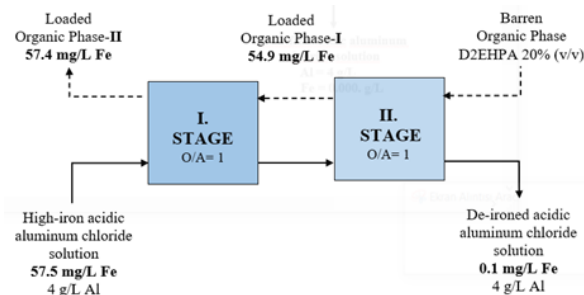


Figure 9. Two-stage continuous counter flow extraction of the ALS-2 with D2EHPA

4. CONCLUSIONS

Based on the comprehensive solvent extraction studies utilizing D2EHPA for the purification of acidic aluminum chloride solutions, the following key conclusions were drawn:

1. Optimal Parameters: A highly efficient and selective iron removal process was successfully demonstrated under the following optimized conditions: an HCl concentration of 2 M, a D2EHPA concentration of 20% (v/v), a mixing time

of 30 minutes, and an organic-to-aqueous phase ratio of 1:1. Crucially, McCabe-Thiele examination determined that only two counter-current extraction stages are required to achieve high removal rates, underscoring the method's practical feasibility for industrial application.

2. **Selectivity and Interference:** The process exhibited strong selectivity for iron over aluminum, as variations in initial aluminum concentration had a negligible impact on extraction efficiency. This is a significant finding for the target application of alumina purification. In contrast, the study revealed that the presence of competing cations, specifically potassium (K^+) and sodium (Na^+), had a deleterious effect on iron removal, likely due to their interaction with the extractant. Furthermore, extraction efficiency was observed to decrease with higher initial iron concentrations, suggesting potential saturation of the organic phase.
3. **Process Efficacy:** Most suggestively, the application of this two-stage methodology resulted in the highly effective removal of 95.58% of iron from the acidic solution. This high efficiency was achieved while concurrently ensuring negligible co-extraction and loss of aluminum, confirming the exceptional selectivity and overall viability of the D2EHPA-based process for this separation.

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DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Mücevher Turan: Writing – review & editing, Writing – original draft, Investigation, Methodology, Formal analysis, Data curation, Conceptualization.

Muhammed Kürşat Aydemir: Writing – review & editing, Writing – original draft, Data curation, Conceptualization.

Mehmet Deniz Turan: Writing – review & editing, Writing – original draft, Supervision, Methodology, Formal analysis, Data curation, Validation, Conceptualization.

Murat Erdemoğlu: Writing – review & editing, Writing – original draft, Methodology, Conceptualization, Supervision, Resources, Project administration, Funding acquisition.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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