

기계적 활성화처리한 블랙드로스의 염산 침출

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Hydrochloric Acid Leaching Behavior of Mechanically Activated Black Dross

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요 약

블랙드로스에 함유된 알루미나를 회수하기 위해 불밀처리가 염산침출에 미치는 영향을 조사하였다. 불밀처리 시간과 회전속도는 알루미나 침출에 큰 영향을 미치지 않았다. 최적의 불밀처리(1시간, 700 rpm)에서 알루미나 침출은 침출시간과 온도에 영향을 받았다. 본 논문의 실험조건에서 산화마그네슘은 모두 용해되었으며, 칼륨, 철, 실리콘과 타이타늄 산화물은 일부만 용해되었다. 알루미나는 80% 정도 침출되었지만 상기 산화물이 미량 용해되므로 순수한 알루미나용액을 회수하기 위해서는 분리공정의 도입이 필요하다.

주제어 : 알루미나, 블랙드로스, 염산침출, 기계적 활성화

Abstract

Effect of ball milling treatment on the hydrochloric acid leaching performance of black dross was investigated to recover alumina. Ball milling time and speed showed limited effect on the leaching behavior of the alumina in the mechanically dross. Under the optimum mechanical activation condition (for 1h at 700 rpm), the leaching of alumina in hydrochloric acid solution was significantly affected by leaching time and reaction temperature. MgO was completely dissolved in most of the leaching conditions, while a small amount of Ca, Fe, Si and Ti oxides was dissolved. Although 80% of alumina was dissolved, the dissolved minor components such as Ca, Fe, Mg, Si and Ti oxides should be separated to recover pure alumina solution.

Key words : Alumina, Black dross, HCl leaching, Mechanical activation

1. Introduction

Aluminum metal has specific properties such as high

hardness, high abrasion resistance, and bio-inertness^{1,2}.

Owing to the increasing demand for aluminum metal, the recycling of aluminum from waste products and

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scraps becomes very important. In the recycling of aluminum scraps, primary and secondary aluminum melting are practiced. During the melting of aluminum scraps, large amounts of aluminum dross/salt are generated, which are classified as toxic industrial wastes. Disposal of aluminum dross in the landfills causes serious ground water and atmosphere pollution. Therefore, aluminum dross should be pretreated by proper methods before discharging.

Based on metal contents, the aluminum dross is divided into three main types such as white dross (15-70% Al), black dross (12-18% Al) and salt cake (3-5% Al)³. The increase in annual production of dross and their economic impacts have urged the development of proper recycling technology and the utilization of dross⁴. Both pyrometallurgical and hydrometallurgical processes are commonly employed to recover aluminum and other components from the dross. The pyrometallurgical route is more effective for the recovery of Al metals from white dross, while hydrometallurgical route is suited to recover some components from black dross.

In the hydrometallurgical process, alumina and other oxides are dissolved from black dross by employing either acid or alkaline solutions. Alkaline leaching offers selective leaching of alumina over other oxides from black dross but the leaching percentage of alumina is very low^{2,5}. Moreover, the high dissolution of silica from the dross in alkaline solution makes it necessary to remove the dissolved silica for the production of pure alumina from the leaching solution. The leaching efficiency of alumina from black dross is enhanced in acid leaching processes. Moreover, silica remains substantially insoluble in these acid solutions and thus obviates the removal of the dissolved silica from the leaching solution^{1,6}. Recently, we reported experimental results on the combined treatment of black dross, which consisted of ball milling treatment followed by alkaline leaching⁷. The alumina leaching solution with purity of 99% was obtained in this method but leaching percentage of alumina by alkaline solution under the optimum condition was lower than 40%. Although sulfuric acid solutions gave high leaching percentage of alumina and less corrosion, the main disadvantage of H₂SO₄ leaching systems

is low solubility of aluminum sulfate in concentrated sulfuric acid⁶. Among several inorganic acid solutions, hydrochloric acid solution is adequate for the dissolution of alumina from the black dross with high content of silica in terms of low solubility of silica and the possibility of selective crystallization of alumina products⁸. In HCl leaching, the alumina dissolution is influenced by the adsorption of chlorides which depends on the operation conditions such as solution pH and reaction temperature^{9,10}. The increase of surface area or change in the crystalline structure of black dross through mechanical activation might enhance alumina dissolution by HCl solution. In fact, little information has been reported on the leaching of the oxides from mechanically activated black dross using hydrochloric acid solution. In order to compare the leaching behavior of mechanically activated black dross between NaOH and HCl solution, ball milling treatment followed by HCl leaching was investigated in this work. For this purpose, the effect of ball milling time and speed on the leaching behavior of the oxides was investigated. In hydrochloric acid leaching, acid concentration, leaching time and temperature, and pulp density was varied to obtain an optimum leaching condition.

2. Materials and Methods

2.1. Materials and pretreatment of black dross

The black dross employed in this work was provided by a company in Korea. This black dross contained the oxides of Al, Ca, Fe, Mg, Si and Ti together with some salts like KCl and NaCl. Since these salts result in the production of some poisonous gases and consume the lixivants during the subsequent leaching step, these salts should be recovered from the black dross⁴. For this purpose, the black dross was treated by hot water under the optimum conditions obtained from previous work; temperature of 90°C, pulp density of 100 g/L and leaching time of 2 h¹¹. Twice leaching with water at above-mentioned conditions led to the complete removal of NaCl and KCl from the black dross⁷. The chemical composition of the black dross after water treatment was analyzed by an X-ray fluorescence spectrometer

Table 1. The chemical composition of black dross after water leaching⁷⁾

Element	Al	Ca	Fe	Mg	Si	Ti
Wt%	40.5	3.6	8.3	2.9	15.1	12.0

(SEA1200VX ID 1147) and is shown in Table 1.

A vertical planetary ball mill (Fritsch Pulverisette 7 Bead Mill) with a rotation speed up to 800 rpm was employed in the ball milling treatment of the black dross after water treatment. Mechanically activated samples were prepared as follows: 8 g of the black dross was added into a vessel containing 40 g agate balls (a ball of 6 mm in diameter) with the weight ratio of ball/black dross of 5:1 and then milled for 1, 3, 5, 7 and 10 h at the milling speed of 400 rpm. In the case of effect of milling speed, the samples were milled at 250, 400, 550 and 700 rpm for 1h at the ball to powder weight ratio of 5:1.

2.2. Hydrochloric acid leaching

Mechanically activated black dross was employed as a feed for the hydrochloric acid leaching experiments. Pure HCl solution (Daejung Co., 35%) was dissolved in doubly distilled water to prepare leaching solutions. Leaching experiments of unactivated and mechanically activated black dross were conducted by taking 100 mL of HCl with desired acidity in a 250 mL of three-neck round bottom flask with a magnetic stirrer bar in a heating mantle. In all leaching experiments, the weight ratio of the black dross to leachant (pulp density) was fixed at 20 g/L except the effect of pulp density and the slurries were stirred at 200 rpm. After the required reaction period, the slurries were filtered using vacuum filtration. The concentration of metals in leaching solutions was measured by inductively coupled plasma optical emission spectrometers (ICP-OES, Spectro Arcos).

3. Results

3.1. Effect of mechanical activation

In ball milling treatment, an increase in milling time and speed leads to a reduction in the particle size and

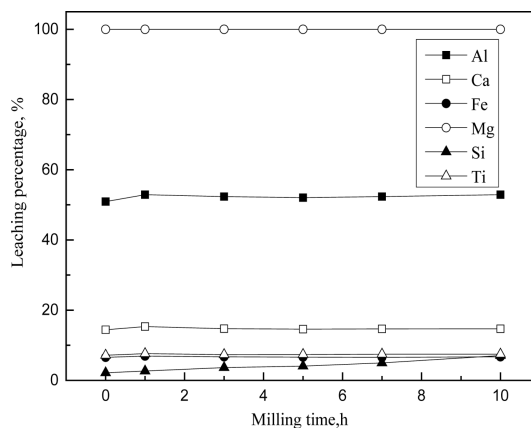


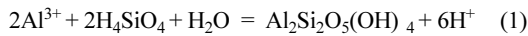
Fig. 1. Effect of milling time on the leaching of the oxides from the mechanically activated black dross in 3 M HCl solution. (Leaching condition: leaching time, 2 h; temperature, 50°C; stirring speed, 200 rpm; pulp density, 20 g/L).

a change in the morphology of the black dross³⁾. In order to investigate the effect of ball milling time on leaching behavior of the oxides in black dross, ball milling time was varied from 1 h to 10 h and then the mechanically activated black dross was leached using 3 M HCl solution at 50°C for 2h at a stirring speed of 200 rpm. The leaching percentage of the oxides from the unactivated and mechanically activated black dross is shown in Fig. 1. The comparison of the XRD patterns between the unactivated and mechanically activated black dross indicates that no new crystalline phase was produced by ball milling but the diffraction peak of alumina was broadened and its intensity increased after mechanical activation⁷⁾. This might be the reason why ball milling has a positive effect of the leaching of alumina from the black dross (see Fig. 1).

According to the Fig. 1, the leaching percentage of all the oxides from the mechanically activated black dross was slightly higher than that from the unactivated black dross. The leaching percentage of alumina increased to 53% after 1h and then was constant with the further increase of milling time. In general, crystalline size of a sample has a relation with its leaching kinetics. Therefore, it might be said that a decrease in crystalline size of the mechanically activated dross would promote

the leaching reaction rate of the dross. The fact that the leaching percentage of mechanically activated dross was almost constant after 1 h of milling time agreed well with the X-ray results that there was no change in phases of the oxides with milling time⁷⁾.

During the leaching of the black dross, the dissolved silica present as H_4SiO_4 in the leaching solution might react with the dissolved alumina to form precipitate as aluminosilicate¹³⁻¹⁶⁾, which is represented in Eq. (1). In this case, there should be no change in the concentration of dissolved alumina.



As milling time increased to 10 h, the leaching percentage of silica increased slowly from 2 to 7%, while that of Ca, Fe, Mg, and Ti oxides was nearly constant. When the leaching kinetics of silica from the black dross is slow in HCl solution, the leaching reaction would be promoted as the crystalline size of the mechanically activated dross decreases with the increase of ball milling time. Although ball milling time did not lead to a much difference in the leaching behavior of the oxides, milling time significantly affected the filterability of the slurries. The filterability of the slurries after leaching was accelerated from 120 min to 20 min as milling time increased from zero to 10h. Therefore, mechanical activation of the black dross for 1h was selected for further experiments on the basis of the easy filterability and low leaching percentage of silica.

In order to investigate the effect of milling speed, the milling speed increased from 250 to 700 rpm for 1h and then this mechanically activated black dross was leached using 3 M HCl solution at a pulp density of 20 g/L, 50°C for 2h. The effect of milling speed on the leaching efficiency of the mechanically activated black dross is illustrated in Fig. 2. There was little change in the leaching percentage of all oxides, indicating that the milling speed in our experimental range did not have any remarkable effect on the morphology of the mechanically activated dross. The constancy in the leaching percentage of the oxides at high milling speed might be ascribed to the formation of large clusters which hinders the mass transfer of HCl into smaller

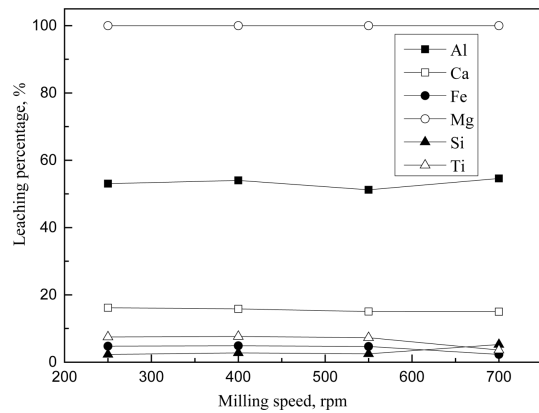


Fig. 2. Effect of milling speed on the leaching of the oxides from the mechanically activated black dross in 3 M HCl solution. (Leaching condition: leaching time, 2 h; temperature, 50°C; stirring speed, 200 rpm; pulp density, 20 g/L).

nanoparticles of the oxides⁷⁾. However, the solid/liquid separation of the slurries was improved as milling speed increased. Therefore, milling speed of 700 rpm was selected as the optimum speed in further experiments.

3.2. Effect of pulp density

Effect of pulp density on the leaching of the oxides from the mechanically activated black dross was investigated in the range of 20 to 100 g/L. In these experiments,

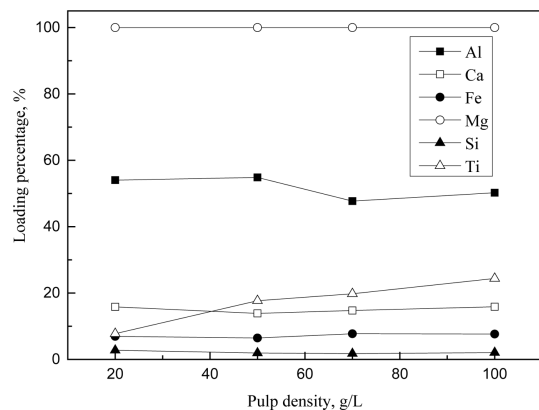


Fig. 3. Effect of pulp density on the leaching of the oxides from the black dross mechanically activated at 700 rpm for 1 h. (Leaching condition: HCl concentration, 3 M; leaching time, 2 h; temperature, 50°C; stirring speed, 200 rpm; pulp density, 20-100 g/L).

the black dross mechanically activated for 1 h at 700 rpm was leached in 3 M HCl solution for 2 h at 50°C. Fig. 3 shows the variations in the leaching percentage of the oxides with pulp density. The leaching percentage of alumina decreased slightly with the increase in pulp density from 20 to 100 g/L, while an opposite trend was observed for TiO₂ at the same condition. The leaching percentage of Ca, Mg, Fe and Si oxides was almost constant at any pulp density. The decrease in the leaching percentage of alumina with pulp density might be related to the formation of aluminosilicate gel¹⁶. As pulp density increased from 20 to 100 g/L, the mass of dissolved silica increased but the amount of free HCl would be decreased. At these conditions, aluminosilicate gel is easily formed. Moreover, the formation of the aluminosilicate gel at high pulp density also made the separation of the slurries difficult.

The large difference in leaching percentage of CaO (15%) and MgO (100%) from the black dross might be related to the reaction of dissolved metals and silica to form precipitates. In the presences of dissolved Ca ions, aqueous silica ions can form precipitates of poly-metal-silicates (see Eq. (2))¹⁷ and these precipitates are dissolved in sodium chloride solutions¹⁸. By contrast, Mg-Si precipitates were easily dissolved in acidic medium¹⁹ and thus most of MgO from black dross was leached in the HCl solution. The low dissolution percentage of iron oxides from the black dross by HCl solution might also be due to the reaction of dissolved iron with dissolved silica to form ferric silicates as represented in Eq. (3)²⁰. In the leaching of TiO₂ in acidic solutions, the stability of TiO²⁺ significantly depend on the TiO²⁺ concentration and temperature. Namely, TiO²⁺ can be easily hydrolyzed to form titanium precipitates under low concentration of TiO²⁺ and high temperature²¹. At low pulp density, the dissolved TiO²⁺ forms precipitates through the hydrolysis reaction and thus the leaching percentage of titanium oxide decreased with a decrease in pulp density.

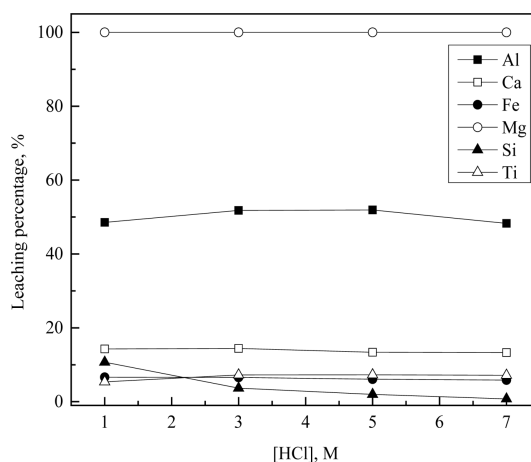
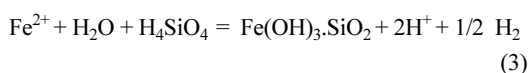
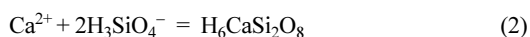


Fig. 4. Effect of HCl concentration on the leaching of the oxides from the black dross mechanically activated at 700 rpm for 1 h. (Leaching condition: HCl concentration, 1-7 M; leaching time, 2 h; temperature, 50°C; stirring speed, 200 rpm; pulp density, 20 g/L)

3.3. Effect of HCl concentration

In order to investigate the effect of HCl concentration on the leaching of the oxides from the black dross mechanically activated for 1 h at 700 rpm, a series of leaching experiments were performed by varying HCl concentration from 1 to 7 M for 2 h at 50°C. In these experiments, the pulp density was kept at 20 g/L. The leaching percentage of alumina slightly increased to 52% with an increase in HCl concentration up to 3 M and then slowly decreased with the further increase of HCl concentration (see Fig. 4). It has been reported that the dissolved alumina in the solution blocks the diffusion path of hydrogen ion and thus reduces the dissolution rate of alumina at high HCl concentration^{1,22}. Our results agree well with these reported results. However, there was no change in the leaching percentage of the oxides of Ca, Fe, Mg, and Ti in our HCl concentration ranges.

Under strong acidic conditions, silica form precipitates through the polymerization of monomeric silica and this polymerization reaction becomes faster with the increase of acid concentration²³. This might be the reason why the leaching percentage of silica decreased as HCl concentration increased from 1 to 7 M. In general, the removal of dissolved silica from the leaching solution

is more difficult than that of other oxides dissolved in acid solutions²⁴). Therefore, 3 M HCl solutions were selected for further experiments in terms of low leaching percentage of silica.

3.4. Effect of leaching temperature

The effect of temperature on leaching behavior of the oxides from the black dross mechanically activated for 1 h at 700 rpm was investigated in the range from 30 to 100°C. In these experiments, the other leaching parameters were fixed at 3 M HCl, pulp density of 20 g/L and leaching time of 2 h. The leaching percentage of alumina steadily rose from 50 to 63% with the increase of temperature from 30 to 100°C (see Fig. 5). MgO was completely leached in the temperature ranges, while there was little difference in the leaching percentage of other oxides. In general, the leaching reaction is favored as the reaction temperature increases. Among oxides in the black dross, the leaching behavior of alumina agreed well with this general trend. The indifference to the reaction temperature in the leaching percentage of other oxides except MgO might be ascribed to the formation of precipitates.

Considering leaching efficiency and energy consumption, leaching at 90°C was suggested as an optimum condition for selective leaching of alumina from the

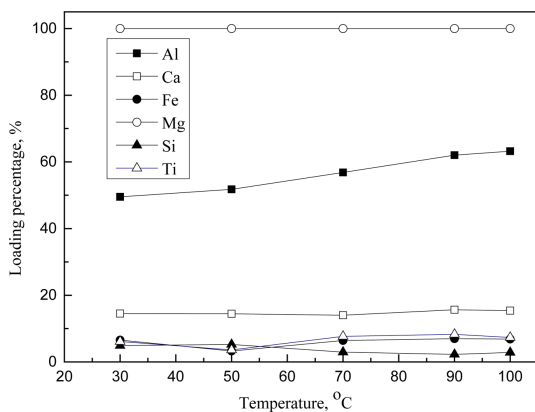


Fig. 5. Effect of leaching temperature on the leaching of the oxides from the black dross mechanically activated at 700 rpm for 1 h. (Leaching condition: HCl concentration, 3 M; leaching time, 2 h; temperature, 30-100°C; stirring speed, 200 rpm; pulp density, 20 g/L).

mechanically activated black dross.

3.5. Effect of leaching time

In order to investigate the effect of reaction time on the leaching behavior of the oxides from the black dross, the black dross mechanically activated for 1 h at 700 rpm was leached by varying time from 2 to 10 h. In these experiments, the other leaching parameters were fixed at 3 M HCl, pulp density of 20 g/L and 90°C. Fig. 6 shows the variations in the leaching percentage of the oxides with leaching time. Except alumina, leaching time did not affect the leaching behavior of the other oxides in the black dross. Most of MgO and less than 20% of Ca, Fe, Si and Ti oxides were leached in the leaching time range. The leaching percentage of alumina increased from 62 to 80% with increase of leaching time from 2 to 4 h and then slowly decreased with the further increase of leaching time. The leaching behavior of alumina with prolonged reaction time might be related to the formation of aluminosilicate¹³⁻¹⁶. Moreover, the presence of Ca and Mg cations in leaching solution might act as catalyst to promote the precipitation of aluminosilicate²⁵). This might explain why the leaching percentage of alumina decreased with increase of leaching time. Thus, the optimum leaching time for the mech-

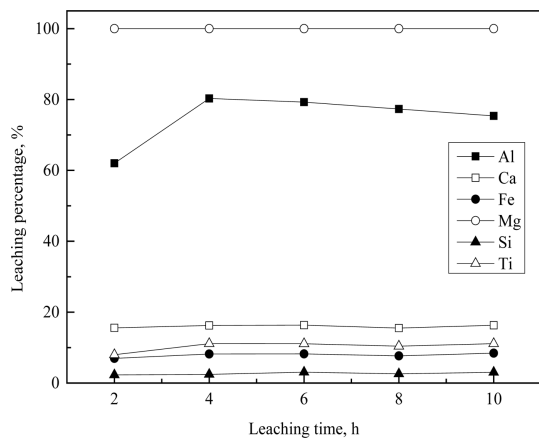


Fig. 6. Effect of leaching time on the leaching of the oxides from the black dross mechanically activated at 700 rpm for 1 h. (Leaching condition: HCl concentration, 3 M; leaching time, 2-10 h; temperature, 90°C; stirring speed, 200 rpm; pulp density, 20 g/L).

anically activated black dross was found to be 4 h at a reaction temperature of 90°C. Under the optimum condition, the leaching percentage of alumina by HCl in the present work was two times higher than that by NaOH solution obtained in previous work⁷⁾. However, other oxides were dissolved in HCl solution and thus separation steps are necessary to recover pure alumina from the HCl leaching solution.

4. Conclusions

In order to recover alumina from black dross, a combined process consisting of ball milling treatment and HCl leaching was investigated. Ball milling time and speed did not show a significant effect on the leaching behavior of the oxides in black dross but the filterability was improved. The optimum condition for ball milling treatment of the black dross was found to be milling time of 1 h at milling speed of 700 rpm. In HCl leaching, MgO was completely dissolved in most of the leaching conditions. The leaching percentage of alumina from mechanically activated black dross was enhanced at higher temperature, while pulp density and acid concentration did not show significant effect. At the optimum leaching condition (3 M HCl, 20 g/L pulp density, 90°C and 4 h), 80% of alumina was dissolved together with less than 20% of Ca, Fe, Si and Ti oxides from the mechanically dross. Subsequent separation step of the leaching solution should be employed to recover pure alumina solution.

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