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≫ 연구논문 ≪

침출에 의한 알루미늄 드로스의 처리 및 유용성분의 회수

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Treatment and Recovery of Valuable Materials from Aluminum Dross by Leaching

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요 약

알루미늄 재활용공정에서 화이트드로스와 블랙드로스가 발생한다. 블랙드로스에 함유된 유용 성분을 회수할 수 있는 공정은 아직까지 개발되지 않았다. 습식공정은 알루미늄 드로스의 처리에 적합하다. 블랙드로스에 함유된 염은 물로 용해시켜 회수할 수 있다. 염회수 후 잔사는 알칼리용액이나 산용액으로 침출한다. 알칼리용액에서 침출속도는 산성용액에서의 속도보다 낮지만, 중간생성물이 알루미나를 함유한 제품을 생산하기에 더욱 적합하다. 알루미늄 드로스의 처리 및 유용성분의 회수를 위한 방향에 대해 고찰했다.

주제어 : 알루미늄 드로스, 산, 알칼리, 침출

Abstract

White and black dross are resulted from the recycling of aluminum. There are no established processes to recover valuable materials from black dross. Hydrometallurgical processes seem to be suitable for the treatment of aluminum dross. The salts in the black dross are recovered by dissolving with water. The residues are treated by either alkaline or acid leaching. Although the leaching rate of alumina by NaOH is lower than that by acid, its intermediates are more suitable to the production of alumina-based materials. The future direction for the treatment and recovery of valuable materials from aluminum dross is discussed.

Key words : aluminum dross, acid, alkaline, leaching

1. Introduction

Treatment and recycling of hazardous wastes attract much attention. When some hazardous wastes are dumped in landfill sites, toxic components might be dissolved into ground water which causes serious environmental problems[1]. The increasing demand for valuable materials and environmental standard enforcement have

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forced the development of suitable treatment methods for industrial wastes.

Aluminum dross represents a residue resulted from primary and secondary aluminum recycling. According to metal contents, aluminum dross can be classified as white and black dross. White dross contains 40% and black dross contains 10% of aluminum[2)]. White dross (wet) is produced from the primary melting operation. It has a high metal content and is compact in largely clotted lumps and blocks. White dross may contain from 20 to 70% recoverable metallic aluminum. Black dross is resulted from secondary recycling process, containing about 12 - 18% recoverable aluminum. Compared to white dross, black dross contains lower metal content and higher amount of oxides and salts. According to the physical and chemical characterization of black dross, it contains metallic aluminum, aluminum nitride, alumina, sodium chloride, potassium chloride and silica with 50% of the dross being a mixture of sodium chloride and potassium chloride[3-8)]. In general, black dross has a granular texture.

The dross consists of a mixture of free Al metal and nonmetallic substances such as aluminum oxide, nitride, and carbide, salts, metal oxides and other elements like magnesium, silicon. The overall chemistry depends on the alloying elements present in the molten Al and the metallurgical practices[2)]. The content of aluminum metal in the dross may be from 5 to 70%. Generally, aluminum metal which is considered to be the most valuable component is recovered from the solidified dross by first fracturing the dross by mechanical milling and then screening out the aluminum. After this step, fractured dross is heated in the presence of additional salt flux to free metallic aluminum[6)]. The composition of aluminum dross depends on the recycling conditions owing to the various alloying elements added during recycling (see Table 1).

According to the evaluation of the economics, the treatment of black dross to recover aluminum compounds is plausible. When the aluminum metal content is higher than 10% in black dross, the revenues from the recovered aluminum and salt are sufficient to pay back the operating and capital cost of the recycling process[9)]. Wastes from this process (non-metallic products (NMP) and salts) are usually landfilled or disposed of without any treatment, causing many environmental damages. The non-metallic products which are the residues after water leaching of the black dross might be the raw materials for the production of concrete blocks[10,11)].

Table 1. Chemical composition of aluminum dross

Compound/element	Percent (w/w)
Al_2O_3	64.8
CaO	0.93
SiO_2	4.0
Fe_2O_3	1.5
MgO	3.2
Na_2O	2.75
K_2O	0.51
Cl	3.9
C	1.25
S	0.22

2. Treatment of White Dross

White dross has higher metal content and is produced from primary and secondary aluminum smelters. White dross may contain 15 to 70% recoverable metallic aluminum and thus is reprocessed to recover aluminum. Less than half of the white dross is recycled and over half of the white dross dust residues are landfilled by the dross reclamation industries[12,13)]. N.S. Ahmad et al.[14)] evaluated the influence of sodium hydroxide on the surface area and pore size of aluminum dross. The treatment of white dross consists of three steps, namely, leaching with sodium hydroxide, precipitation and calcination. The characteristics of aluminum dross during the leaching with NaOH was evaluated on the basis of NaOH concentration, reaction time and temperature. As reaction time, temperature and NaOH concentration increase, the surface area of the aluminum dross decreases, which has a negative effect on the leaching efficiency. Thus, the reactivity of the aluminum dross is better at 25°C and the dissolved aluminum was precipitated by adding hydrogen peroxide at room temperature. After filtering and washing with distilled water, the filtrates were calcined to obtain alumina at 600°C for 3 hours.

3. Treatment of Black Dross

Black dross consists of a heterogeneous mixture of aluminum oxides, metallic aluminum and chlorides and fluorides of potassium, calcium and sodium. It is chemically active due to the presence of various aluminum carbides and nitrides. The total aluminum content in black dross is about five percent by mass and there is no commercially viable method for the recovery of aluminum metal. Hydrometallurgical and pyrometallurgical processes have been proposed for the treatment of black dross. Pyrometallurgical processes employ some reducing agents at relatively high temperature and thus generally face the problems of high energy consumption[15-17]. On the other hand, hydrometallurgical processes employ either alkaline or acidic leaching to dissolve the recoverable components at relatively low temperatures.

3.1. Recovery of salt from black dross

The salts employed during melting are water-soluble salts like KCl and NaCl. Therefore, black dross contains a small amount of metallic aluminum together with lots of the salt-flux mixtures[18]. Recovery of these salts from black dross might be economically advantageous and reduce the amount of toxic wastes. Many methods have been proposed to solve the problem of salt cake disposal. Traditionally, the salts are recovered by water leaching. Treatment of the black dross with water at low and high temperatures was proposed to dissolve the salts[19,20]. Peng Li has also proposed a new method of employing CO_2-saturated water to optimize the recovery of salts from dross[21]. In an integrated process, most of the salts were dissolved from the black dross by water at 90°C for 1 h[22]. Dash et al.[23] reported that two step water leaching of black dross for 1 h at 80°C led to complete recovery of the salt. This sodium and potassium chloride thus dissolved can be transformed into hydroxides by electrolysis. A method was proposed to recover salts and hydrated aluminum oxide from aluminum dross by water leaching. The dross was leached with water at 250°C [24]. Peng et al.[21] compared the leaching of black dross between pure water and CO_2-saturated water as a function of reaction time and pulp density. In the case of leaching with CO_2-saturated water, 95% of the NaCl and KCl present in the salt was dissolved at a pulp density of 1/20 for 3 hours at room temperature.

Thermal treatment was employed to remove salts from black dross at the temperature range between 1150°C and 1200°C[25]. This work indicates that the combined effect of high temperature and reduced pressure has a favorable effect on the removal percent of the salts from the dross as long as the reaction temperature is higher than 1200 °C.

3.2. Sulfuric acid leaching

Sulfuric acid leaching of the residue after water treatment has been tried to produce aluminum sulfate[26-29]. Other processes are proposed to recover alumina by leaching with sulfuric acid followed by heat treatment of the aluminum hydroxide. Dash et al. reported that sulfuric acid leaching at 90°C after water washing of black dross led to 84% recovery of η-alumina[22]. This alumina prepared at 900°C can be the raw material for the manufacture of catalysts. The reactions occurring during the leaching can be represented as follows

$$Al_2O_3 + 3H_2SO_4 = Al_2(SO_4)_3 + 3H_2O \quad (1)$$

$$2Al + 3H_2SO_4 = Al_2(SO_4)_3 + 3H_2 \quad (2)$$

$$2Al + 6H_2SO_4 = Al_2(SO_4)_3 + 3SO_2 + 6H_2O \quad (3)$$

A γ-Al_2O_3 was recovered from the washed dross free of chloride salt by sulfuric acid leaching at 70°C for 1 h[30]. The dissolved Al was precipitated as $Al(OH)_3$ by adding NaOH solution and this aluminum hydroxide power was then calcined at 600°C to obtain γ-Al_2O_3. B. R. Dash[31] reported that the salt in black dross can be easily dissolved in sulfuric acid solution at 90°C for 1 hour and the amount of salt in black dross affects its leaching behavior. Prior dissolution of salt by water can improve the leaching efficiency of alumina[32].

After recovering metallic aluminum from the black dross, the residues were treated by leaching in sulfuric acid solution at 200°C for 2 hours followed by hydro-chlorination to obtain aluminum chloride[33,34]. First, sulfuric acid leaching at 100°C for 5 hours resulted in 50% leaching of Al. By using hydro-chlorination method in the second step, aluminum dross residues can be changed into aluminum chloride. Such processes could lead to high yields of Al compounds from aluminum dross residues as aluminum chloride or alumina.

Fusion treatment of the dross with Na_2CO_3 led to sodium aluminate liquid from which high purity alumina ($Al_2O_3 > 99.5\%$) was prepared by precipitation and calcination[35]. Another process was proposed to recover active α-Al_2O_3 with high purity from the black dross[36]. First, the dross was sintered at high temperature after mixing with soda and then the mixtures were dissolved in sulfuric acid solution. After removing the impurities by EDTA, NH_4HCO_3 was added to the solution to precipitate the aluminum. These precipitates were calcined at 1000°C to recover pure alumina. It has been reported that a process consisted of acid leaching at 90°C-purification-precipitation-calcination could recover alumina with purity higher than 99%[37].

3.3. NaOH leaching

Alkaline leaching of black dross followed by crystallization or precipitation results in $Al(OH)_3$, which is considered to be economical and environment-friendly process[38,39]. In general, leaching by bases has the follow-

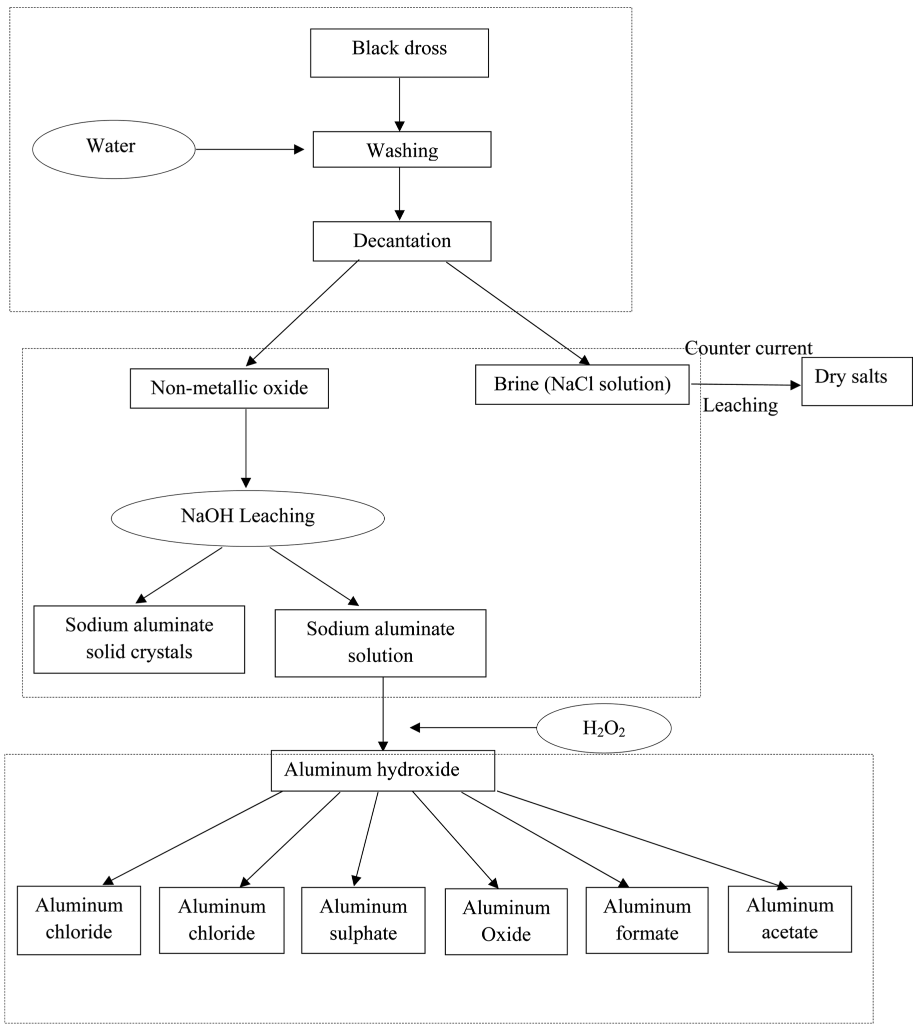

Fig. 1. Overall process flowchart for the recycling of black dross.

ing advantages: negligible corrosion problems, suitability for ores containing carbonate gangue, more selectivity because iron oxides would not be leached.

About 57% of aluminum was dissolved by high-pressure alkaline leaching of the residues after water treatment, using 260 g/L of NaOH at 240°C for 100 min and about 57% of aluminum was obtained[40]. Another process similar to Bayer process was proposed to recover $Al(OH)_3$ from black dross[41]. After alkaline leaching under the conditions: 10% NaOH solution at ambient temperature for 2 hours in molar ratio $Na_2O : Al_2O_3 = 2$ and precipitation by CO_2, the obtained $Al(OH)_3$ was used as an additive material for waste water cleaning ($Al_2(OH)_5Cl$)[42]. A process was proposed to recover $Al(OH)_3$ from the black dross by 10% NaOH solution leaching followed by hydrolysis of the resulting solution[39]. It was reported that about 70% of aluminum was dissolved during the NaOH leaching. The chemical reactions occurring during the treatment of black dross with NaOH can be represented as[42]

$$2Al + 2NaOH + 2H_2O = 2NaAlO_2 + 3H_2 \quad (4)$$

$$Al_2O_3 + 2NaOH = 2NaAlO_2 + H_2O \quad (5)$$

From the sodium aluminate solution obtained by alkaline leaching of the dross, aluminum hydroxide was precipitated by using different precipitating agents such as H_2O_2, CO_2, NH_4HCO_3, $(NH_4)_2CO_3$, and $(NH_4)Al(SO_4)_2$)[43]. A γ-Al_2O_3 with 95% purity was obtained by calcining the $Al(OH)_3$ at 600°C which was precipitated by H_2O_2.

A comparison on the leaching of black dross with caustic soda was made between under atmospheric pressure and high pressure[44]. Although strong leaching conditions were applied, the maximum recovery of aluminum from black dross by atmospheric pressure leaching (50%) is lower than that (73%) reached by high pressure leaching. Hydroxides and oxides with high purity, such as Al_2O_3, MgO and $MgAl_2O_4$ together with non-metallic products (MNP) were recovered from black dross by leaching with NaOH[45,46]. First, the black dross was dissolved in NaOH solution at 98°C for 8 hours and $Al(OH)_3$ of 99.7% purity was precipitated from the caustic solution by lowering its pH. Detailed experimental conditions to obtain Al_2O_3 with a higher surface area of 55 $m^2 \cdot g^{-1}$ were reported by leaching the black dross in 10% NaOH solution at 96°C for 2 hours[47]. In these experiments, $NH_4Al(SO_4)_2 \cdot H_2O$ was employed as a precipitating agent at solution pH of 10.5. $Al(OH)_3$ can be prepared from the sodium aluminate solution by using several methods. This aluminum hydroxide thus recovered can be the starting materials for the manufacture of aluminum chloride, aluminum nitrate, aluminum sulfate, aluminum oxide, aluminum formate and aluminum acetate (see Fig. 1).

3.4. Comparison between acid and alkaline leaching

Acid and alkaline leaching of black dross have advantages and disadvantages. Table 2 lists a comparison on the leaching of black dross between H_2SO_4 and NaOH solution. In the case of H_2SO_4 leaching, the leaching rate of aluminum is generally very high. The leaching percentage of aluminum by NaOH solution is lower than that by H_2SO_4 solution. However, the intermediates obtained after the alkaline leaching seem to be more appropriate to the production of alumina based materials and non-metallic products from black dross. Optimum conditions for the treatment of specific black dross should be obtained.

4. Conclusions

Aluminum dross is classified as white and black dross. At present, there are no established processes to recover valuable metals from the black dross. In this review, the pretreatment of black dross to recover salts and subsequent leaching of the residues to recover aluminum components are discussed. The salts in the black dross can be successfully recovered as salts by dissolving with water at atmospheric pressure or as hydroxide forms by electrolysis of the resulting solution. Alumina can be obtained from black dross by hydrothermal process consisting of acid/alkaline leaching, purification, precipitation and calcination. The leaching percentage of aluminum by H_2SO_4 solution is higher than that by NaOH. However, the product obtained after alkaline

Table 2. Comparison among leaching of H_2SO_4 and NaOH from black dross

Reagent	Leaching condition		% Leaching	Treatment of solution	Product	Ref.
H_2SO_4	Concentration Temp. Time Stirring speed	10% H_2SO_4 70°C 1 hour 450 rpm	Al: 84%	Addition of ammonia → calcination	η-alumina	[22]
	Concentration Temp. Time Stirring speed	2M H_2SO_4 70°C 1 hour 450 rpm	Al: 93.2%	Addition of alkaline → calcination	γ-alumina	[30]
	Concentration Temp. Time Solid concentration	30% H_2SO_4 90°C 1 hour 10%	Al: 85%		Aluminum	[31]
	Concentration Temp. Time solid/ liquid	10% H_2SO_4 100°C 5 hours 0.111	Both aluminum and nitrogen 88.5%		Aluminum, Nitrogen	[32]
	Concentration Time	98% H_2SO_4 2 h	Al_2O_3: 99.5%	Precipitation by NH_4HCO_3 → calcination	Alumina	[35]
	Concentration Temp. Time	0.9 M H_2SO_4 85°C 2.5 hours	Al_2O_3: 99%	Precipitation by NH_4HCO_3 → calcination	α-alumina	[36]
	Concentration Temp. Time Solid/ liquid	15 wt% H_2SO_4 90°C 5 hours 1/12	Al_2O_3: 99.28%	Precipitation by NH_4HCO_3 → calcination	Alumina	[37]
NaOH	Concentration Temp. Time Stirring speed	10% NaOH Room temp. 2 hours	Al: 71%	Precipitation by CO_2	$Al(OH)_3$	[39]
	Concentration Time Molar ratio Na_2O: Al_2O_3	10% NaOH 2 hours 2	Al_2O_3 80%	Precipitation by CO_2	$Al(OH)_3$	[41]
	Concentration Temp. Time Solid/ liquid	200 g/L NaOH 100°C 200 min 0.3	Al_2O_3: 95%	Precipitation by H_2O_2 → calcination	γ-Al_2O_3	[43]
NaOH	Concentration Temp. Time Stirring speed	30% NaOH 180 °C 100 min	Al: 73%	Precipitation → calcination	α-Al_2O_3	[44]
	Concentration Temp. Time	NaOH 98.8°C 8 hours	Al_2O_3:56%		$Al(OH)_3$	[45]
	Concentration Temp. Time S: L ratio	10% NaOH 96°C 120 min 1: 100	Al: 42%	Precipitation by $NH_4Al(SO_4)_2$	Alumina	[46]

leaching seems to be more appropriate to the production of alumina based materials and non-metallic products from black dross.

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