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A NOVEL METHOD FOR ALUMINA PREPARATION BY ELECTROTRANSFORMATION $AlCl_3$ SOLUTION UNDER NH_4Cl-CATALYZED AND CO_2-INJECTED CONDITIONS

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Abstract

As a refractory industrial solid waste, high-alumina fly ash is discharged into nature in large quantities, causing serious environmental harm. The extraction of valuable components, such as aluminum, from high-alumina fly ash has become a research hotspot at home and abroad. The shortcomings of traditional alumina extraction from high-alumina fly ash include long technological processes, large amounts of waste acid and alkali, and serious equipment corrosion. A novel method for alumina preparation by electrotransformation $AlCl_3$ solution(EAS) under NH_4Cl catalysis with injected CO_2 has been proposed by Northeastern University. The process has advantages, such as short flow and no waste acid discharge. In this study, the influence of initial NH_4Cl concentration in the cathodic chamber on solution pH, phase and morphology of electrotransformation products, phase, D(50) and D(90) of roasting products, and Al recovery rate were examined. Simultaneously, EAS was examined and compared with and without injected CO_2, as well as EAS under NH_4Cl catalysis with and without injected CO_2. The results showed that, when the solution in the cathodic chamber was NH_4Cl with CO_2 injection, with electrotransformation time extension, pH increased slowly and the morphology of electrotransformation products were all rough and fluffy block structures. Increasing initial NH_4Cl concentration effectively improved the product crystallization states, roasting products D(50) and D(90), and Al recovery rate.

Keywords: *$AlCl_3$ solution; Alumina; High-alumina fly ash; Electrotransformation products*

1. Introduction

In recent years, with the rapid development of economy and science and technology, the environmental issues have become the world focus of attention [1]. Fly ash is a refractory industrial solid waste produced in large amounts by burning coal in high temperature coal-fired power plants. Although quite a bit of fly ash is widely used for low-value applications in the production of bricks, cement, concrete, ceramic products, building materials, and road bases [2-7], a large portion of fly ash is still discharged arbitrarily into the environment. The discharge of this industrial waste not only occupies a large amount of potential agricultural land [8] and endangers human health, but it also brings a serious of environmental burden, causing local river congestion, water pollution, PM2.5 rise, and other problems [9-11]. The annual emission of fly ash in the world is as high as 500 million tons [12], with the annual emission of fly ash in China as high as 50 million tons [13], which is approximately 10% of the world fly ash annual emission. Meanwhile, it can be considered to be the world's fifth largest raw material resource [14]. According to investigation, the alumina content in Inner Mongolia's high-alumina fly ash is as high as 40–50 wt% [15-16], which is much higher than the alumina content in ordinary fly ash (20–30 wt%) and similar to China's middle and low-grade bauxite (50–60 wt%) [17-24], indicating that high-alumina fly ash is a promising resource for alumina production [25]. Extracting aluminum (Al) and other valuable elements from high-alumina fly ash offers a precious opportunity to create high value-added products from this refractory industrial solid waste [26-28]. The chemical composition (wt%) of fly ash in a power plant in Inner Mongolia shows that Al_2O_3 and SiO_2 are the main chemical components of high-alumina fly ash, accounting for nearly 91 wt% (Table 1 [29]). There are also small amounts of other components, such as Fe_2O_3, CaO, MgO, and unburned components. China has rich reserves of bauxite, with conserved reserves of 2.288 billion tons, but commercially available bauxite (A/S > 7) accounts for only 33.05% (246.7 million tons) of

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reserves. Also, the Al industry, which has a significant impact on the country's economic development, is facing a situation of resource depletion. With commercially available bauxite resources continuously diminishing and alumina demand increasing, high-alumina fly ash has become a potential ideal substitute for bauxite in alumina production [30]. Reasonable and effective recycling of high-alumina fly ash paves the way for its efficient and economical utilization, which is of great significance for sustainable development. Therefore, valuable component extraction, such as Al, from high-alumina fly ash has become a research hotspot at home and abroad.

The traditional methods that have proven effective in extracting alumina from high-alumina fly ash are acid and alkali methods. The acid method has become the primary choice for traditional alumina extraction from high-alumina fly ash, having the advantages of low energy consumption and low slag production. However, there are serious equipment corrosion and high cost disadvantages. Furthermore, the problems of water and acid recycling have limited this method's development. The alkali method for treating high-alumina fly ash is a complicated process with a series of procedures, including sintering, silica-alumina separation, and purification [31]. This method has the disadvantages of high energy consumption, instability, much industrial solid waste and low extraction rate. Therefore, it is necessary to develop a new technology for extracting alumina from high-alumina fly ash to solve the traditional methods shortcomings, utilize this large resource, and reduce the burden on the environment.

In view of the current gloomy situation, such as bauxite resource shortages and serious environmental problems, and inspired by the chlor-alkali industry, Northeastern University has proposed a new method for preparing alumina by electrotransformation $AlCl_3$ solution (EAS) under NH_4Cl catalysis and CO_2 injected into a cathodic chamber. The technological process for extracting alumina from high-alumina fly ash is shown in Figure 1. Compared with traditional extraction processes, this new method has the advantages of short process flow and no waste acid. Compared with direct EAS, EAS with injected CO_2, and EAS with NH_4Cl catalysis with and without injected CO_2 in cathodic chamber, this method increased the Al recovery rate, increased of EAS product crystallinity, and the D(50) and D(90) of roasting products greater. The current study investigated the influence of initial NH_4Cl concentration on solution pH, phase and morphology of EAS products, phase, D(50), and D(90) of roasting products, and Al recovery rate. Simultaneously, EAS with and without injected CO_2 and with and without NH_4Cl catalysis were compared.

Table 1. *The chemical composition of fly ash in a power plant in Inner Mongolia (wt%)*

Elements	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	Others
Contents	53.62	37.35	2.32	1.36	1.23	4.12

2. Experimental

2.1. Experimental material

Experiments were carried out in a controlled system and raw materials all pure chemical reagents. The main chemical reagents included aluminum chloride hexahydrate ($AlCl_3·H_2O$, 98.0 – 100.5 wt%) and ammonium chloride (NH_4Cl, Sinopharm Chemical Reagent Co., Ltd., Shanghai, China, ≥99.5 wt%).

2.2. Experimental characterization

The experimental instruments and equipment used in the process of experimental characterization were as follows. The pH and temperature of solutions during electrotransformation were monitored using a precision acidity meter (Precise pH meter; PHSJ–3F, Shanghai Electronic Scientific Instrument Co., Ltd., Shanghai, China). Products were analyzed by X-ray diffraction (XRD; D8 Advance, Bruker Corp., Billerica, MA, USA; 40 kV, 40 mA, 2θ = 10° – 90°, Cu-Kα X-ray source) to identify crystalline phases. The morphologies of product surfaces sputtered with Au were examined via scanning electron microscopy (SEM; SU-8010 Carl Zeiss AG, Oberkochen, Germany). A large particle size instrument (BT-801; Dandong Baxter Instrument Co. Ltd., Dandong, China) was used to examine the products D(50) and D(90). Prodigy Plus full spectrum direct Reading

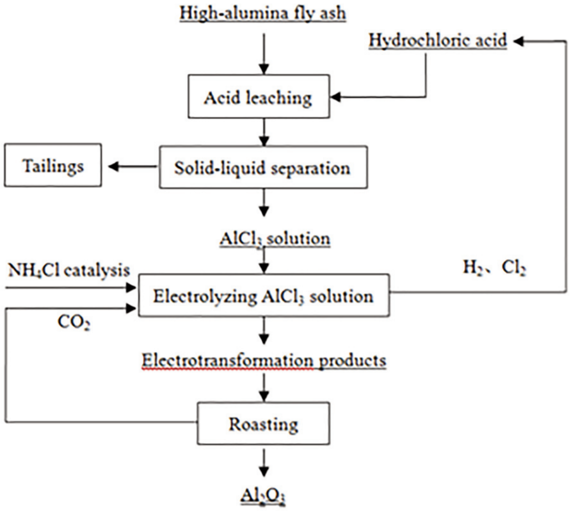

Figure 1. *Technological process of extracting alumina from fly ash by electrotransformation*

Emission Spectrometer (ICP; Teledyne Leeman Labs, Hudson, NH, USA) was used to examine Al concentrations in solution.

2.3. *Experimental process*

The main experimental conditions and specific experimental process in this study were described in this section. The main experimental conditions were a 2 A current, 2 cm electrode spacing and 3 h electrotransformation time. The experimental process was as follows: 100 g/L of $AlCl_3$ solution and 20, 40, 60, 80, and 100 g/L of NH_4Cl solution were prepared. Then, 250 mL of an $AlCl_3$ solution was poured them into the anodic chamber and 250 mL of NH_4Cl solution or $AlCl_3$ was poured them into the cathodic chamber. CO_2 gas, if desired, was injected into the cathodic chamber. The power supply was then turned on and adjusted to start electrotransformation experiments. The solution pH was recorded during the electrotransformation process. After an experiment was finished, the solids in the cathodic chamber were poured out, filtered, dried, and roasted. A schematic diagram of the EAS process is shown in Figure 2.

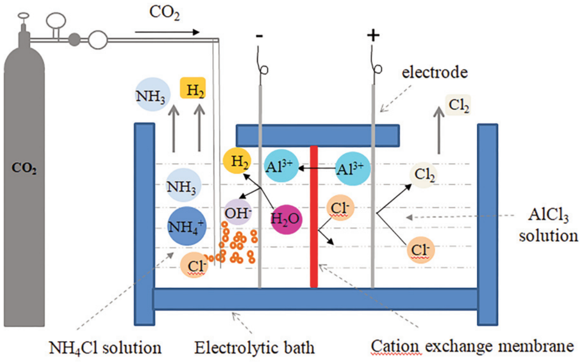

Figure 2. *Schematic diagram of electrotransformation $AlCl_3$ solution process*

3. Results and discussion

The results obtained during the EAS process, as well as the characteristics of electrotransformation products and roasting products are summarized in Figure 3 –8 below and discussed in this section.

Observations of solution pH in EAS under different experimental conditions indicated that, with extended electrotransformation time, the solution pH increased (Fig. 3). When the solution in the cathodic chamber was NH_4Cl, the solution pH in the electrotransformation process was all higher than that when the solution in the cathodic chamber was $AlCl_3$. When the solution was NH_4Cl without injected CO_2 in the cathodic chamber, solution pH fluctuated greatly during the process, with solution pH as high as 8.53 by experiment's end (Fig. 3 (1)). This was mainly because viscous substances produced in the process under this condition floated on the liquid surface and around the cation exchange membrane, resulting an uneven solid and liquid distribution, hindering ion combination and leading to a greater pH fluctuation. When the solution was NH_4Cl with injected CO_2, the solution pH slowly increased and all values ≤ 7.25 (Fig. 3 (2)). At the same time, the solution pH was seen not to change significantly with increased initial NH_4Cl concentration in the same processing time. This was mainly because CO_2 injection made the solids and liquids more evenly distributed, which increased the ion binding chance in solution and reduced solution pH fluctuation. At the same time, $(NH_4)_2CO_3$ and H_2CO_3 produced in this process acted as a solution buffer.

XRD of electrotransformation products under different experimental conditions showed that, when the solution in the cathodic chamber was NH_4Cl and its concentration greater than or equal to 60 g/L, electrotransformation products had high crystallinity and the product $Al(OH)_3$ (Fig. 4). When the solution in

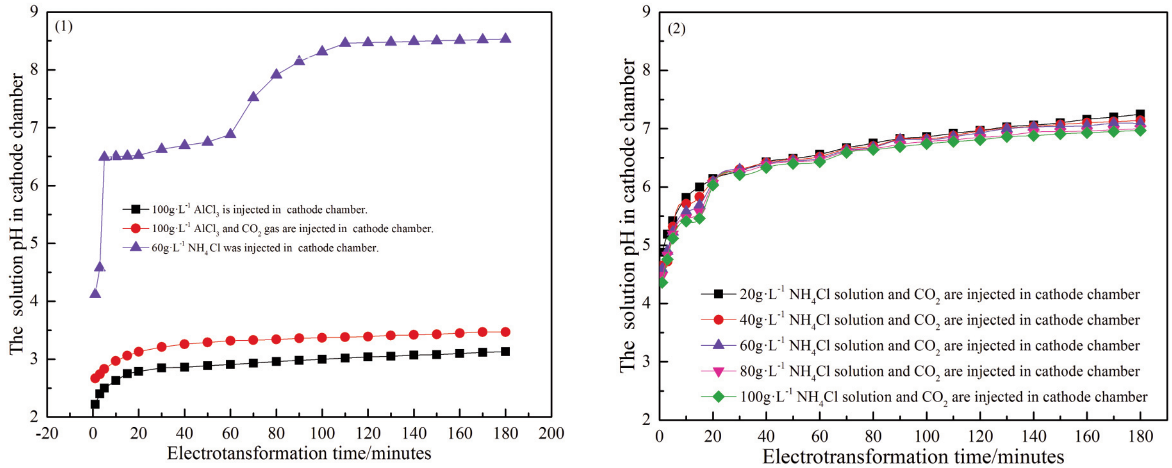

Figure 3. *Solution pH under different electrotransformation conditions*

cathodic chamber was $AlCl_3$ and the initial NH_4Cl concentration in the cathodic chamber ≤ 60 g/L, electrotransformation products were amorphous substances, with low crystallinity. With increased initial NH_4Cl concentration with injected CO_2, the crystallinity of electrotransformation products tended to increase (Fig. 4 (2)). In other words, increasing the initial NH_4Cl concentration under CO_2 conditions was beneficial for obtaining $Al(OH)_3$ precipitation with better crystallinity.

SEM of electrotransformation products prepared under different experimental conditions showed that, when the cathodic chamber solution was $AlCl_3$, electrotransformation products had sheet structures (Fig. 5 (1)). When the cathodic chamber solution was NH_4Cl, the products were blocky and spherical structures with surface micropores. When the solution was NH_4Cl with injected CO_2, the products showed rough and fluffy blocky surface structures ((Fig. 5 (2)). Thus, both NH_4Cl and CO_2 was seen to change electrotransformation product morphologies.

XRD of roasting products under different experimental conditions showed that the products were all alumina and the diffraction peaks were all in the shape of steamed buns (Fig. 6). At the same time, when the cathodic chamber solution was NH_4Cl solution, the number of diffraction peaks of the products was seen to decrease, and the peak strength of the diffraction peak decreased. In addition, the products diffraction peaks moved to the right, compared with those when the solution was $AlCl_3$ (Fig. 6 (1)). When the solution was NH_4Cl with injected CO_2, the diffraction peak strength of the products did not change with increased initial NH_4Cl concentration (Fig. 6 (2)). In other words, the initial NH_4Cl concentration had no clear influence on the XRD of roasting products.

The D(50) and D(90) of roasting products under different experimental conditions showed that products resulting when the cathodic chamber solution contained NH_4Cl were greater than those prepared when the solution $AlCl_3$ (Fig. 7). This was mainly because viscous $Al(OH)_3$ obtained by electrotransformation with NH_4Cl easily accumulated and the product also easily solidified and formed massive structures of large particle size and hardness in the drying process. D(50) and D(90) of the products increased with increased initial NH_4Cl concentration with injected CO_2. The initial NH_4Cl concentration effectively improved the D(50) and D(90) of roasting products, which was mainly because, with increased initial cathodic chamber NH_4Cl concentration, more viscous $Al(OH)_3$ was produced, the easier it was to accumulate, leading to increased particle size.

The Al recovery rate under different experimental conditions showed that the Al recovery rates when the cathodic chamber solution was NH_4Cl were all higher than when prepared with $AlCl_3$ solution (Fig. 8). This was mainly because NH_4Cl did not participate in oxidation and reduction reactions in the electrotransformation process when the cathodic chamber solution was NH_4Cl, which was considered as a hydroxide transfer medium; In addition, when electrolyte pH reached the Al^{3+} precipitation range, Al^{3+} combined with OH^- in the cathodic chamber to precipitate $Al(OH)_3$, accompanied by NH_3 generation. This made solution pH rapidly increase, thus increasing precipitation in cathodic chamber and increasing the Al recovery rate. At the same time, when the cathodic chamber solution was NH_4Cl, injected CO_2 was observed to make Al recovery rate greatly improve. With increased the initial NH_4Cl concentration, the Al recovery rate was observed to increase with injected CO_2. NH_4Cl and CO_2 were concluded to increase Al recovery rate, which was mainly because with increased initial NH_4Cl concentration, more NH^{4+} was in solution. Thus, NH_3 was easier to produce, and pH more easily increased, such that the amount of precipitation in the cathodic chamber increased, and Al recovery rate increased.

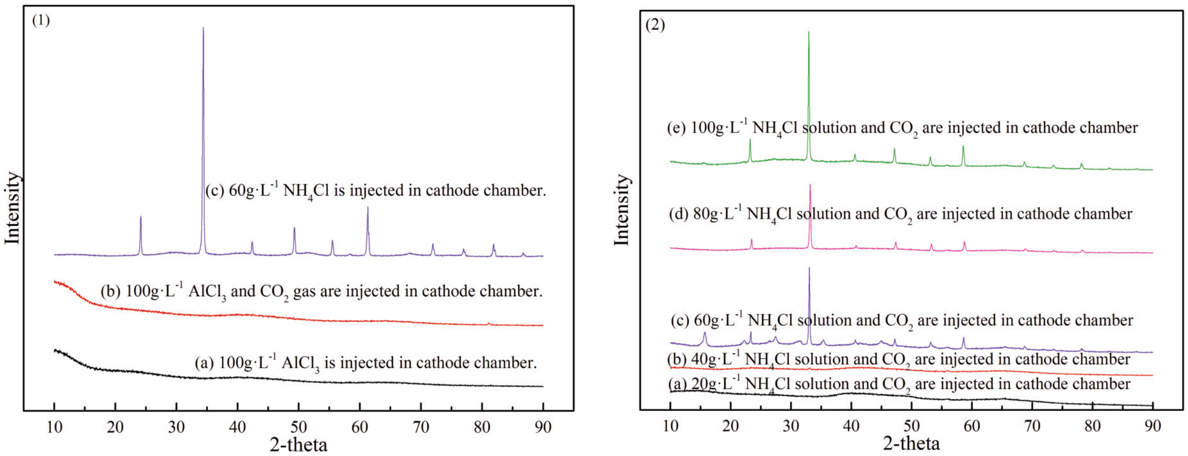

Figure 4. *XRD of electrotransformation products prepared under different conditions*

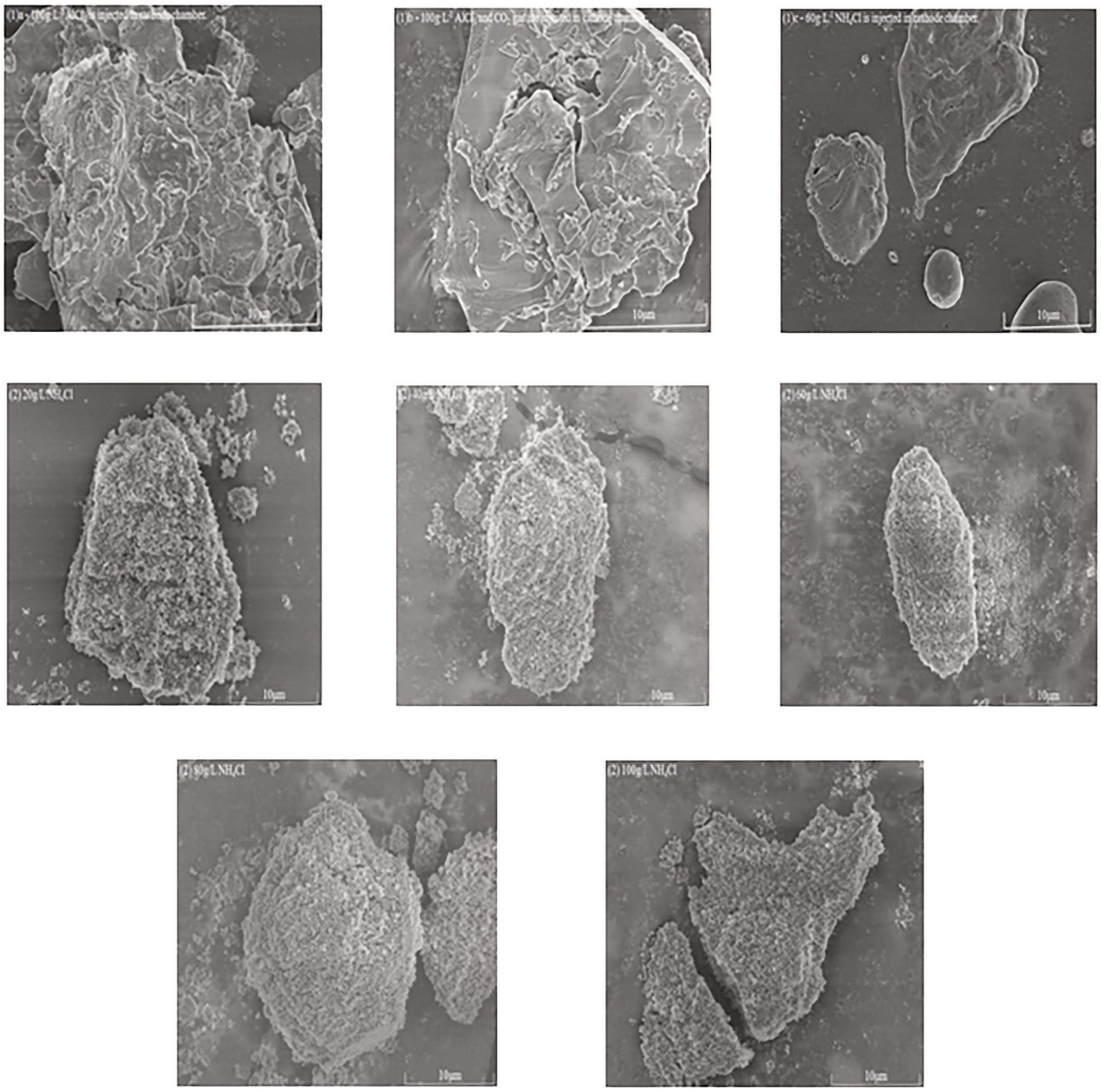

Figure 5. *SEM of electrotransformation products prepared under different conditions*

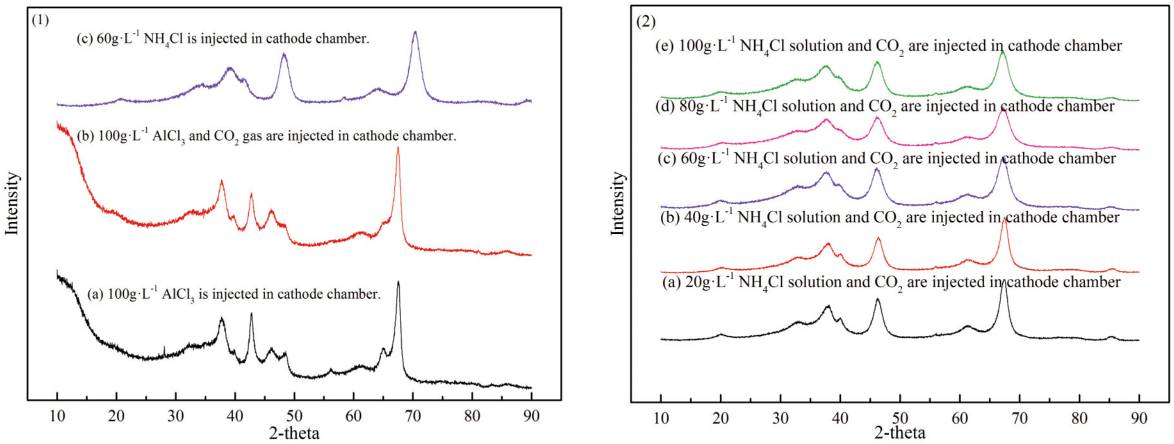

Figure 6. *XRD of roasting products prepared under different conditions*

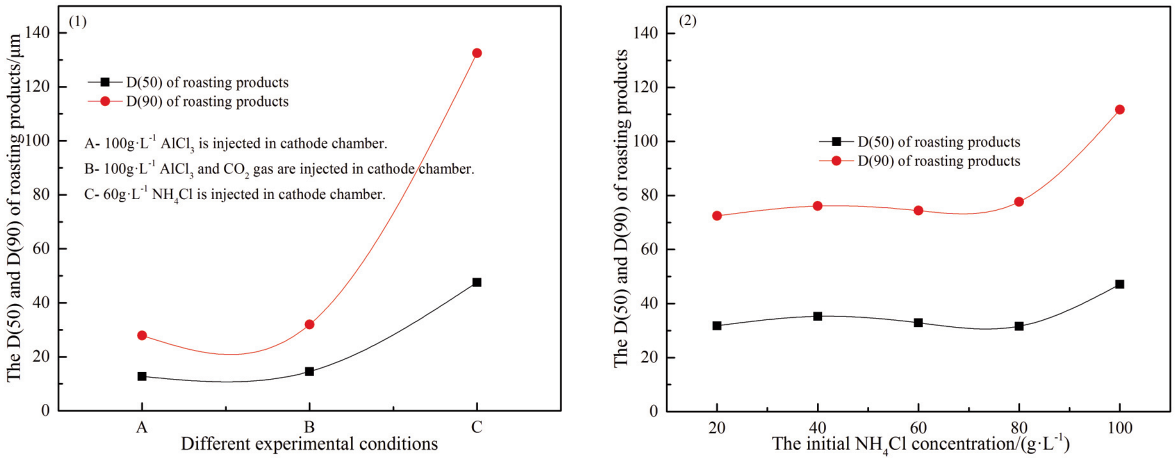

Figure 7. *D(50) and D(90) of roasting products prepared under different conditions*

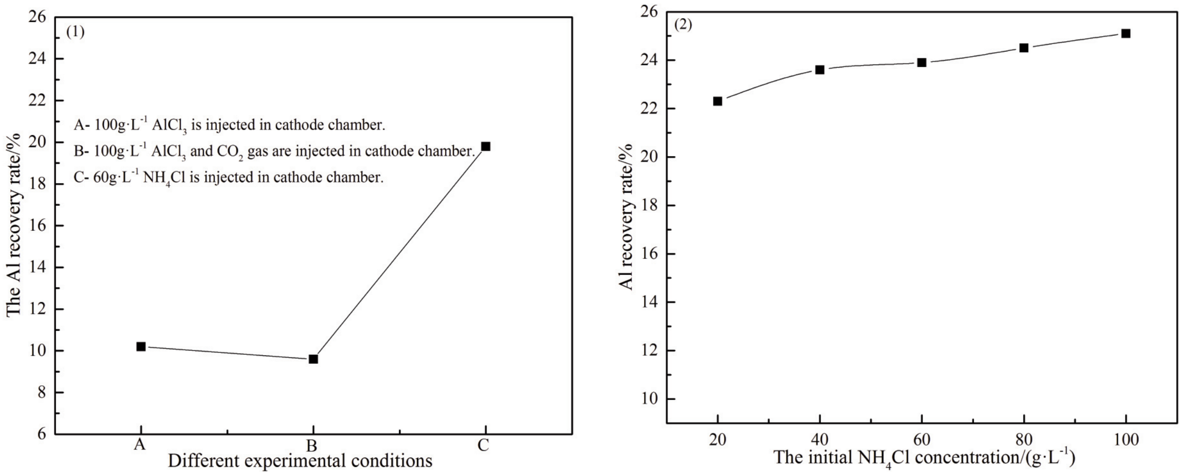

Figure 8. *Al recovery rate under different experimental conditions*

4. Conclusions

In this study, a theoretical direction was taken by observing the influence of initial NH_4Cl concentration with injected CO_2 on the electrotransformation process. The results were compared with those prepared under other experimental conditions. The following conclusions were drawn.

(1) When the cathodic chamber solution was NH_4Cl with injected CO_2, an extended electrotransformation time led to slow, pH increase and all values decreased with increased initial NH_4Cl concentration.

(2) When the cathodic chamber solution was NH_4Cl with injected CO_2, electrotransformation product morphology were all rough and fluffy block structures. In addition, initial NH_4Cl concentrations with injected CO_2 had no clear influence on these morphologies and the XRD of roasting products.

(3) Under injected CO_2 conditions, increased initial NH_4Cl concentration effectively improved the crystallization state of electrotransformation products, the D(50) and D(90) of roasting products, and Al recovery rate. When the initial cathodic chamber NH_4Cl concentration was ≥60 g/L, the crystallization state of electrotransformation products, D(50) and D(90) of roasting products, and Al recovery rate were all better than those when the cathodic chamber solution was $AlCl_3$.

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Author contributions

Conception and design of study: Xiu-Xiu Han and

Ting-An Zhang. Acquisition of data: Xiu-Xiu Han and Yan-Mei Liu. Analysis and/or interpretation of data: Xiu-Xiu Han, Li-Xin Yang, Ying-Feng Lu and Run-Ting Wang. Drafting the manuscript: Xiu-Xiu Han. Critical revision: Xiu-Xiu Han.

Statement of the manuscript

This manuscript is our original unpublished work, it has not been submitted to any other journal for reviews and present submission to Journal of Mining and Metallurgy, Section B: Metallurgy is not a duplicate sent to another publication.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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NOVA METODA ZA DOBIJANJE GLINICE ELEKTROTRANSFORMACIJOM $AlCl_3$ RASTVORA U PRISUSTVU NH_4Cl KAO KATALIZATORA I CO_2

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Apstrakt

Kao refraktorni industrijski čvrsti otpad, pepeo sa visokim sadržajem glinice ispušta se u prirodu u velikim količinama i izaziva veliku štetu po životnu sredinu. Ekstrakcija vrednih komponenti, kao što je aluminijum, postala je centar istraživanja u zemlji i inostranstvu. Nedostaci tradicionalne ekstrakcije aluminijuma iz refraktornog elektrofilterskog pepela podrazumevaju duge tehnološke procese, velike količine otpadnih kiselina i baza, kao i ozbiljnu koroziju opreme. Severoistočni univerzitet je predložio novi metod za dobijanje glinice elektrotransformacijom $AlCl_3$ rastvora (EAS) u prisustvu NH_4Cl kao katalizatora i CO_2. Prednost ovog postupka su kratak protok i neispuštanje otpadnih kiselina. U ovom radu je ispitan uticaj početne koncentracije NH_4Cl u katodnoj komori na pH rastvora, fazu i morfologiju produkata elektrotransformacije, faze D(50) i D(90) kao produkte pečenja i brzinu izvlačenja Al. Istovremeno je EAS postupak ispitan i upoređen sa i bez dodatog CO_2, kao i EAS u prisustvu NH_4Cl kao katalizatora sa i bez CO_2. Rezultati su pokazali da, kada je rastvor u katodnoj komori bio NH_4Cl sa dodatim CO_2, sa produženjem vremena elektrotransformacije, pH se polako povećavao i morfologija proizvoda elektrotransformacije je bila gruba i pahuljasta blok struktura. Povećanje početne koncentracije NH_4Cl je poboljšalo stanje kristalizacije proizvoda, kao i produkte prženja D(50) i D(90) i brzinu dobijanja Al.

Ključne reči: *$AlCl_3$ rastvor; Glinica; Leteći pepeo sa visokim sadržajem aluminjuma; Proizvodi elektrotransformacije*