

Technical Report

**R&D of a New Process
to Produce Alumina
from Secondary Aluminum Dross**

December 2023

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1. Introduction

About 2 million tons of aluminum slag are generated during the process of primary aluminum smelting, fabrication and processing, and waste aluminum recycling in China.

The main components in the aluminum slag are metallic aluminum, alumina, aluminum nitride, fluoride salts, chloride salts, as well as the elements such as iron, silicon, magnesium, and calcium. The secondary aluminum dross is generated by separation of metallic aluminum from aluminum slag and is toxic and harmful, which pollutes the environment and damages people's health.

Several countries, such as China, have defined secondary aluminum dross as hazardous solid waste (China, National hazardous waste catalogue code HW48: 321-024-48321-024-48). Its hazardous characteristics are reactivity and toxicity. Its reactivity refers to the harmful substance aluminum nitride (AlN), while the toxicity refers to soluble fluorides such as NaF and KF.

Extensive researches have been conducted on the resource utilization process of the secondary aluminum dross both domestically and internationally, including the preparation of refractory materials, building and road materials, water cleaning agents, and steelmaking refining agents, etc.

However, the resource utilization rate of secondary aluminum dross is still

relatively low due to such factors as complex composition, technical limitations, small market capacity and low product value. The main component of secondary aluminum dross is alumina, with a content range of about 65-80%, which is a high-quality resource for producing alumina. The best technical solution for resource utilization of the secondary aluminum dross is to develop a new process to produce alumina from the dross by using a clean production technology to remove all the toxic and harmful components in the dross based on the systematic study of the composition and reaction behavior of all the substances in the dross.

In this report a new process has been developed, in which two stages are carried out: first one is a roasting stage to change all the aluminum element including Al in AlN into sodium aluminate and all fluorides into stable CaF_2 ; the second one is an alumina production stage to change sodium aluminate into alumina or alumina chemicals.

This new process can be used for large scale treatment of secondary aluminum dross and the industrial tests have been conducted for alumina and chemical hydrate production from the secondary aluminum dross in China.

2. Characteristics of secondary aluminum dross

The aluminum slag is in the form of agglomerated slag, which contains a significant amount of aluminum metal. Due to the high value of metallic aluminum, heat treatment or cold treatment processes are generally used to recover the 10% to 30% of metallic aluminum contained in the aluminum slag. The secondary aluminum dross is accounting for approximately 70% to 90% of the aluminum slag, which the metal aluminum content remained is generally less than 5%.

It can be seen that the secondary aluminum dross produced by primary aluminum industry is grayish white in color, while the secondary aluminum alloy dross and aluminum recycling dross are mainly grayish black in color, which is because aluminum alloy dross and aluminum recycling dross contain more chloride refining agents and alloying elements. In addition the colors of primary aluminum dross and aluminum alloy dross from different sources also vary due to differences in composition and processing technology.



Figure 2-1 Aluminum slag and secondary aluminum dross from different sources (a) Primary Aluminum Slag from Henan (b) Primary Aluminum Dross from Henan (c) Primary Aluminum Dross from Guangxi (d) Aluminum Alloy Dross from Jiangsu (e) Aluminum Alloy Dross from Hebei (f) Aluminum Recycled Dross from Jiangsu

2.1 Chemical composition of secondary aluminum dross

The chemical composition analysis has been systematically conducted for the aluminum slag samples from the different workplaces such as aluminum smelters, alloy manufacturers and fabrication plants and recycling centers for waste aluminum in China.

The nitrogen content in aluminum slag and secondary aluminum dross was determined by distillation neutralization titration method, and X-ray fluorescence analysis (XRF) was used to analyze chemical composition of the samples.

The aluminum metal content in secondary aluminum dross accounts for about 40%, indicating that secondary aluminum dross has high recycling value.

The chemical composition of secondary aluminum dross from different sources is shown in Table 2-1. In addition, there are significant differences

in the trace element content in the secondary aluminum dross from different sources. The samples from primary aluminum smelters contains high fluoride salts and almost no alloying elements due to direct contact with aluminum electrolyte. The samples from aluminum alloy and recycling plants have high chloride content due to the refining agents added during operations, and contain a certain amount of alloy elements such as Cu, Zn, Mn, Ni, etc.

Table 2-1 Chemical composition of secondary aluminum dross from different sources

Content (%)	Primary aluminum dross 1	Primary aluminum dross 2	Aluminum alloy dross 1	Aluminum alloy dross 2	Aluminum recycled dross 1	Aluminum recycled dross 2
Al	39.29	40.17	38.14	39.43	36.53	37.92
O	38.59	37.52	40.33	39.14	38.12	38.9
N	6.60	5.96	6.39	3.67	3.21	5.63
Si	2.33	2.38	2.18	4.69	3.57	4.04
Fe	1.85	0.34	0.97	0.52	0.98	1.03
Ca	0.97	1.17	1.34	1.19	1.48	1.06
Cl	0.83	0.47	2.13	4.64	3.83	5.03
Total F	4.70	5.97	2.51	0.69	2.4	1.03
Soluble F(mg·L ⁻¹)	157.6	243.5	91.5	12.8	13.24	33.5
Na	1.49	3.26	1.48	0.41	2.47	1.63
K	0.47	0.35	0.34	0.34	0.61	0.5
Mg	3.29	0.79	2.25	3.75	4.38	2.15
Ti	0.11	—	0.29	0.64	0.29	0.13
Zn	—	—	0.13	0.11	0.19	0.24
Mn	—	0.01	0.28	—	0.17	0.11
Ni	—	—	0.012	0.003	0.013	0.009
Cu	0.07	—	0.31	0.52	0.99	0.47

2.2 Mineral composition of secondary aluminum dross

The mineral composition tests have been carried out by X ray diffraction. The mineral composition of secondary aluminum dross samples from the different sources are shown in Figure 2-2. The aluminum components in the secondary aluminum dross are mainly composed of alumina, aluminum nitride, metallic aluminum, and magnesium aluminate spinel. Iron, silicon, and magnesium elements mainly exist in the minerals of hematite, quartz, and magnesium aluminate spinel. In addition, sodium, potassium, and calcium elements mainly exist in the fluoride salts and chloride salts.

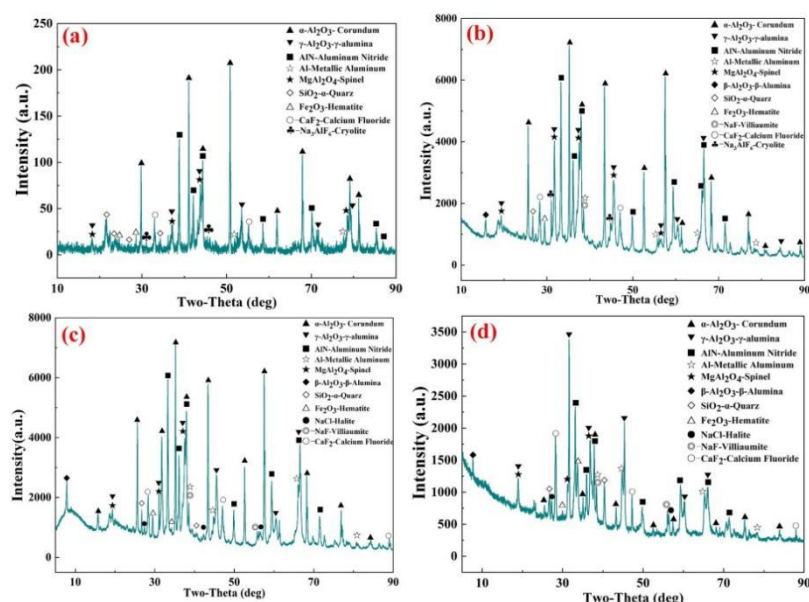


Figure 2-2 Minerals in secondary aluminum dross from different sources (a) (b)

Primary aluminum dross (c) Aluminum alloy dross (d) Aluminum recycled dross

The alumina minerals are recognized as α -Al₂O₃、 γ -Al₂O₃ and β -Al₂O₃ formed in the reaction of molten aluminum with oxygen in the air, while the aluminum nitride is mostly generated by the reaction of molten aluminum with nitrogen in the air at high temperatures during the casting and refining processes. The minerals of hematite, quartz, and magnesium

aluminate spinel are mainly formed by the oxidation of impurities Fe, Si, and Mg elements in the molten aluminum. Chloride salts and fluoride salts are fluxes and refining agents are added during the melting and refining processes for improving heat transfer performance and fluidity of metals, in addition preventing aluminum oxidation by covering the surface of molten aluminum. Another source of fluoride salts is the electrolyte from the primary aluminum smelting process.

The chemical composition of the secondary aluminum dross is complex, various minerals are tightly embedded, making it difficult to separate from each other and to recover.

The study task should be focused on that the valuable components in the secondary aluminum dross such as alumina are economically recovered, while the harmful components such as aluminum nitride and fluorides are efficiently removed.

3. R&D of a new process to produce alumina from secondary aluminum dross

3.1 The new process flow scheme

The aluminum element, which weighted approximately 40% in the secondary aluminum dross is the most valuable part for recycling, mainly is in the composition of α -Al₂O₃, γ -Al₂O₃, AlN, MgAl₂O₄, with a small content of metallic aluminum. The toxic and harmful components mainly include aluminum nitride, soluble fluoride salts and chloride salts. It is difficult to make resource utilization for secondary aluminum dross since the valuable and toxic elements coexist and their reactions interfere with each other.

This project is based on the study of the occurrence status of chemical and mineral compositions in the secondary aluminum dross, as well as the study on the reaction behavior of the entire component with the additives.

A new process has been developed for large scale resource utilization of the secondary aluminum dross. Not only detoxification of secondary aluminum dross is achieved, but also valuable alumina is efficiently extracted to be returned into aluminum production chain.

To extract alumina from α -Al₂O₃, γ -Al₂O₃, AlN and MgAl₂O₄, the best routine is alkaline roasting, in which AlN will react with oxygen to be eliminated. The calcium additive will react with fluorides to form stable and harmless CaF₂ at the roasting temperatures. Based on the mentioned

above a new process flow scheme for alumina extraction from secondary aluminum dross has been developed and is shown in Figure 3-1.

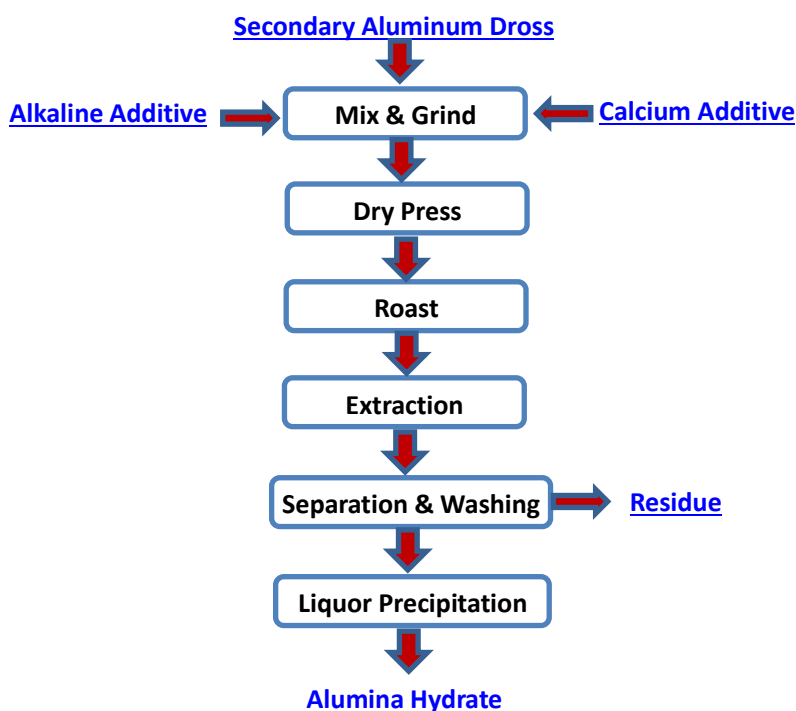


Fig.3-1 Flow Scheme of the New Process

3.2 Experimental methods

The Fig.3-2 shows all the experimental methods used for the new process development including the optimum operation parameters tests.

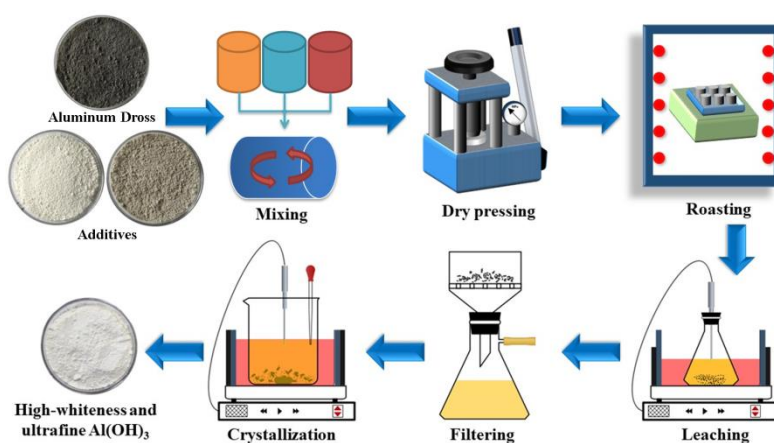


Figure 3-2 Experimental Methods

Firstly, a certain amount of secondary aluminum dross is accurately

weighed. A certain amount of additives are accurately weighed according to the theoretical formula calculations and added to the vibration grinding and thoroughly mixed evenly with the secondary aluminum dross.

The mixed and ground materials is dry extruded under a pressure of 10-20 MPa, and heated to a predetermined temperature, then roasted for a definite time.

After being crushed and finely grounded, the roasted clinker is leached out in a caustic liquor. After the reaction is completed, solid-liquid separation is carried out, the sodium aluminate liquor can be used to produce alumina or chemicals.

3.3 Effect of different additives on alumina extraction

The goals of this process are to oxidize aluminum nitride in the secondary aluminum dross into nitrogen, volatilize chloride salts into flue gas, and solidify soluble fluoride salts into harmless CaF_2 by the additives. At the same time, the components containing aluminum elements are reacted with alkaline additives to form soluble sodium aluminate, which is recycled back into the alumina production chain. Other impurity elements such as silicon, magnesium, iron, and the rare alloy elements such as copper, manganese, and nickel are insoluble in the alkaline liquor and separated into the residues.

The alumina extraction coefficient by alkaline additive and the impurity removal coefficient by calcium additive should be defined as following

formula for an accurate and efficient roasting recipe, which can be determined by a series of tests.

$$n(\text{N}/\text{AF}) = \frac{n(\text{Na})}{n(\text{Al}) + n(\text{Fe})} \quad (3-1)$$

$$n(\text{C}/\text{SF}) = \frac{n(\text{Ca})}{2n(\text{Si}) + 0.5n(\text{F})} \quad (3-2)$$

3.3.1 The effect of alkaline additives for alumina extraction

Figure 3-3 (a) shows the mineral composition of roasting clinker with different contents of alkaline additives added. It can be found that the main minerals in the roasting clinker are sodium aluminate, periclase, dicalcium silicate, sodium ferrite, spinel, cristobalite, and calcium fluoride.

When $n(\text{N}/\text{AF})$ increases from 0.95 to 1.15, the diffraction peaks of magnesium aluminate spinel and cristobalite in the clinker continue to decrease, while the diffraction peaks of periclase and sodium aluminum silicate significantly increase. This indicates that alumina in magnesium aluminate spinel can be combined with alkaline additives and the cristobalite reacts with alkaline to form sodium aluminum silicate.

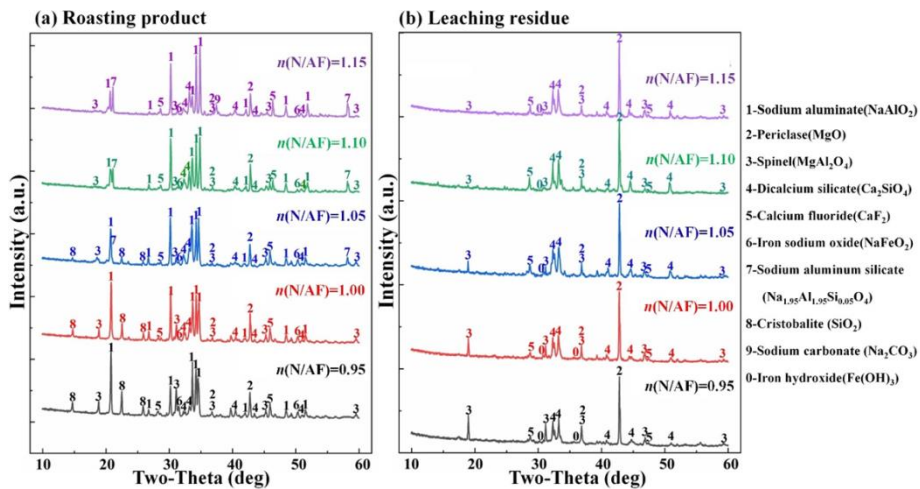


Figure 3-3 Mineral phases of (a) calcined clinker and (b) leaching residue under

different $n(N/AF)$

It can be seen in Figure 3-3 (b) that the main minerals in the leaching residue are periclase, dicalcium silicate, iron hydroxide and calcium fluoride. This shows that sodium aluminate in the roasted clinker has been leached out into the caustic liquor containing sodium aluminate. At the same time, the impurities such as magnesium, silicon, and iron are separated into the residue.

Figure 3-4 shows the standard leaching rates of alumina and sodium oxide in the roasted clinker with different amount of alkaline additive addition. Both standard leaching rates of alumina and sodium oxide increase first and then decrease with increase of the alkaline additive. The leaching rates reach their maximum value at $n(N/AF)=1.1$, which is 93.92% and 96.39%, respectively.

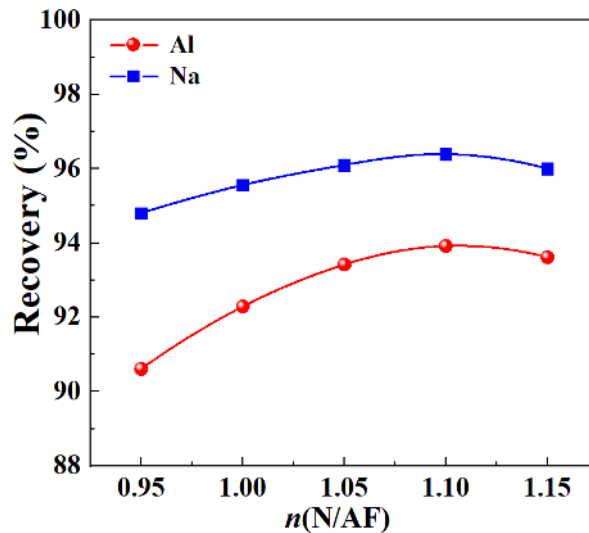


Figure 3-4 Standard leaching rates of aluminum and sodium from calcined clinker under different $n(N/AF)$

When the alkaline addition content is too low, the aluminum element in alumina, aluminum nitride, and magnesium aluminum spinel fails to fully

react to form sodium aluminate, resulting in poor leaching performance of roasted clinker. When the alkaline addition content becomes too high, the alkaline loss will be increased at the roasting temperatures, and the side reactions in the leaching process will be intensified as well, resulting in the loss of alumina and alkaline.

3.3.2 The effect of calcium additives on impurity removal

Figure 3-5 shows the standard leaching rates of alumina and sodium oxide in the roasted clinker with different calcium additive addition. Both the standard leaching rates of alumina and sodium oxide increase first and then decrease with the increase of calcium addition. The standard leaching rates reach their maximum value at $n(C/SF)=1.0$, which is 94.53% and 97.21% respectively.

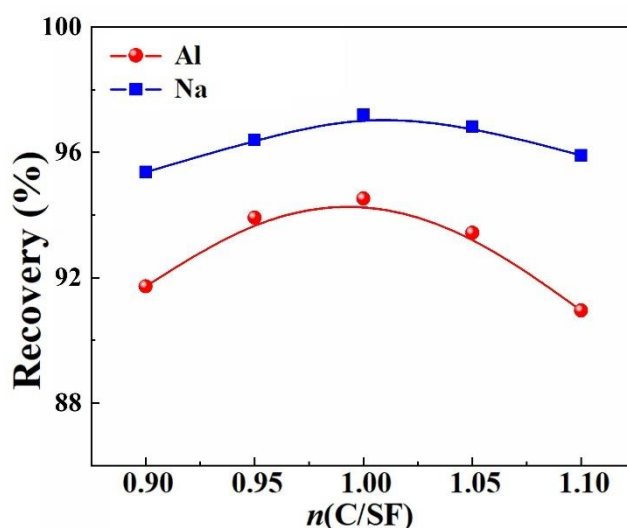


Figure 3-5 Standard leaching rates of aluminum and sodium from calcined clinker at different $n(C/SF)$

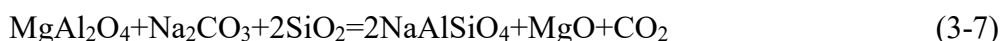
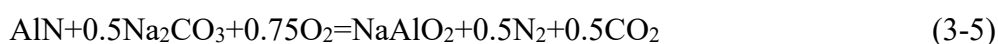
When the calcium addition content is too low, it is not sufficient to completely combine with silica in the dross to form dicalcium silicate,

resulting in forming some sodium aluminosilicate and loss of alumina and alkaline. When the calcium addition content is too high, there is a lot of free calcium oxide present in the roasted clinker, which is prone to side reactions during the leaching, resulting in the loss of alumina as well (Eq. 3-3, Eq. 3-4).



During the roasting process, the harmful aluminum nitride in secondary aluminum dross is oxidized into non-toxic nitrogen and the chloride salts volatilize into the flue gas, while the fluoride salts is reacted with calcium additive into harmless and stable calcium fluoride. Almost all the aluminum elements react with alkaline additive to form soluble sodium aluminate, which can be recycled back into the alumina production. Other impurity elements such as magnesium, silicon, iron, and such rare alloy elements as copper, manganese and nickel will enter the leaching residue as the insoluble substances to be separated.

The reactions possibly happened during the roasting process are shown below:



8)



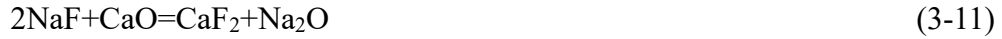
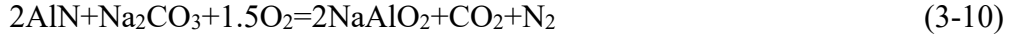


Figure 3-6 (a) shows the results of the removal rates of chlorine and nitrogen elements by roasting at different roasting temperatures.

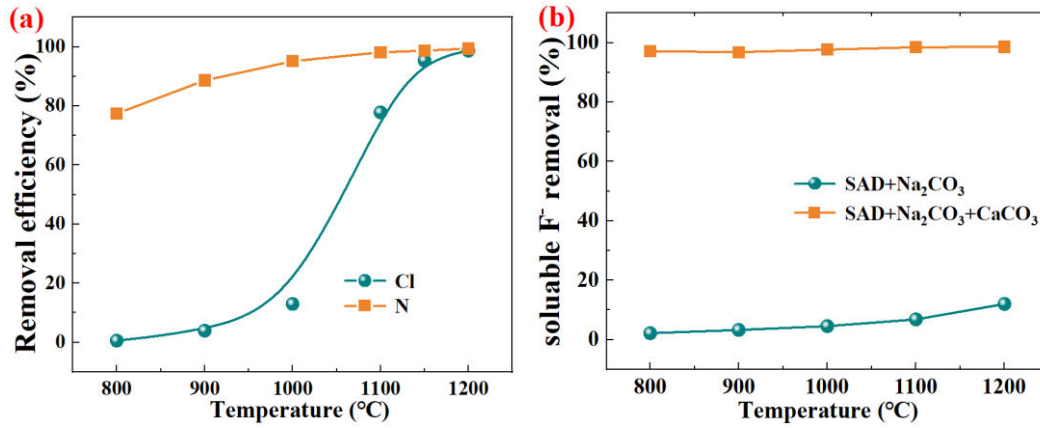


Figure 3-6 Removal of (a) chlorine, nitrogen, and (b) soluble fluorides from roasted clinker at different temperatures

It can be seen that when the temperature reaches chloride melting point ($> 800\text{ }^{\circ}\text{C}$) chloride salts begin to slowly evaporate. The volatilization rate significantly increases at above $1000\text{ }^{\circ}\text{C}$, and the chloride removal rate reaches 95.38% at $1150\text{ }^{\circ}\text{C}$. Most aluminum nitride can react with alkaline additive below $800\text{ }^{\circ}\text{C}$, and the removal rate can reach 98.68% at $1150\text{ }^{\circ}\text{C}$. Figure 3-6 (b) compares the removal rate of soluble fluorides in roasted clinker before and after the addition of calcium additive. It can be seen that as the roasting temperature increases to $1200\text{ }^{\circ}\text{C}$, the removal rate of soluble fluorides slightly increases without addition of calcium additive. This indicates that although there is a small amount of fluoride volatilized when the temperature exceeds $1000\text{ }^{\circ}\text{C}$, most of it still remains in the

roasted clinker. After adding calcium additive, the removal rate of soluble fluorides under different roasting temperatures was greater than 96%, and the removal rate reached 98.57% at 1150 °C. This is because sodium fluoride reacts with calcium additive and alumina during the roasting process to produce sodium aluminate and insoluble calcium fluoride, which is consistent with thermodynamic analysis.

3.4 The effect of roasting temperature and time changes

The standard leaching rates of alumina and sodium oxide of the roasted clinker for the same composition at the different roasting temperatures from 1000 °C to 1200 °C are shown in Figure 3-7.

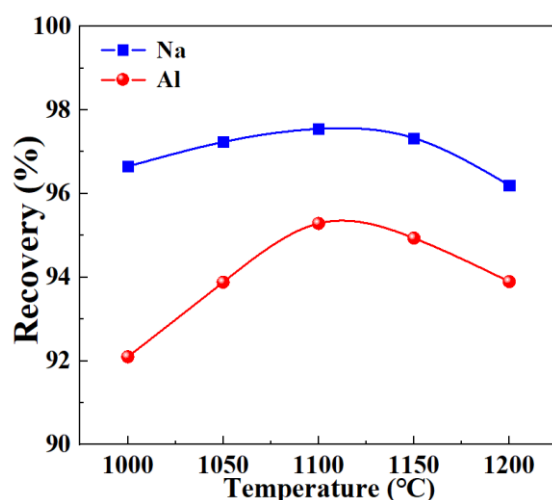


Figure 3-7 The leaching performance of the clinker at different roasting temperatures

When the roasting temperature increases, both the standard leaching rates of alumina and sodium oxide increase first and then decrease, reaching their maximum values of 95.47% and 97.78%, respectively at about 1100 °C. This is because the materials mainly rely on solid-solid reactions during the roasting process. When the roasting temperature is too low, there

is less liquid phase on the material surfaces and the reaction rate will be very slow. And the obtained clinker has high porosity, low strength and poor leaching performance. When the roasting temperature goes up too high, a large amount of liquid phase appears on the clinker surfaces causing too much alkaline loss, promoting the hardness of the roasted clinker and making the leaching performance of the clinker worse. Therefore, the appropriate roasting temperature is necessary for the best quality roasted clinker to achieve suitable strength and porosity and the best leaching performance.

Figure 3-8 shows the effect of roasting time on the leaching performance of the roasted clinker.

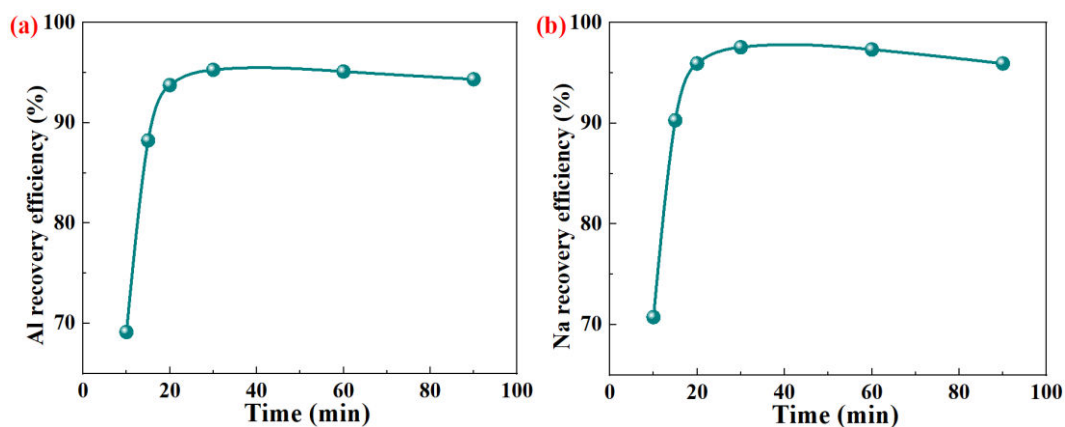


Figure 3-8 Effect of different roasting times on the leaching performance of the clinker

Both the standard leaching rates of aluminum and sodium in the clinker first rapidly increase and then slowly decrease with the prolongation of roasting time. The reaction is almost completed in 20 minutes, and both the standard leaching rates reach their maximum values of 95.29% and 97.55% in 30 minutes, respectively. Moderately extending the roasting time is

beneficial for the reaction to proceed, but excessive roasting time can also lead to more liquid phase on the clinker surfaces, reducing the porosity and deteriorating its leaching performance.

3.5 The effect of forming pressure molding before roasting

The comparative roasting tests on the same composition mixed materials were conducted by using two methods:

- ① Dry extrusion molding under a pressure of 10-20 MPa;
- ② Direct roasting without pressure molding.

The test results indicate that leaching rates of the clinker by the pressure molding is only slightly higher than that of the sample without the pressure molding, which means the satisfactory leaching performance can be achieved for both the dry extrusion molding sample and the sample without pressure molding before roasting.

3.6 Optimal process operation parameters

Based on the study mentioned above a resource utilization technology for the secondary aluminum dross has been developed. The optimal process operation parameters are:

- ① The best recipe coefficients: $n(\text{N/AF})$ is 1.1, $n(\text{C/SF})$ is 1.0;
- ② The optimum roasting temperature range is 1100-1120°C;
- ③ The best roasting time is 30 minutes.

Under the operation conditions mentioned above the standard alumina leaching rate will be $\geq 95\%$ and the standard sodium oxide leaching rate

will be $\geq 97\%$.

4. Industrial tests of the new process

4.1 Composition of secondary aluminum dross in the industrial tests

The appearance and composition of the secondary aluminum dross from different batches used in the industrial tests are shown in Fig4-1 and Table 4-1.



Figure 4-1 Secondary aluminum dross warehouse and its appearance for industrial tests

Table 4-1 Chemical composition of secondary aluminum dross from different batches

Batches	Al ₂ O ₃ %	SiO ₂ %	CaO%	MgO%	Fe ₂ O ₃ %
1	70.4	4.79	3.00	6.43	0.48
2	60.8	6.81	2.70	6.55	0.55
3	58.4	7.32	6.61	5.57	1.32
4	69.2	6.27	4.21	9.65	1.07
5	77.6	4.59	5.41	7.72	0.9
6	80.6	2.68	4.51	5.57	0.87
7	79.6	3.04	0.60	3.22	0.69
8	80.6	2.68	4.51	5.57	0.87
9	76.8	4.69	1.20	9.22	0.76
10	76.4	7.01	6.33	8.78	0.87
11	84.8	3.53	5.27	8.53	0.73
12	88.4	3.00	2.81	11.04	0.38
13	76.8	4.69	1.20	9.22	0.76
14	80.0	14.49	4.92	7.53	1.67
15	84.8	3.53	5.27	8.53	0.73
16	88.4	3.00	2.81	11.04	0.38
17	80.0	6.00	4.92	7.53	1.67
Average	77.27	5.18	3.90	7.75	0.86

4.2 Industrial tests of the new process

The industrial tests of the new process has been conducted in a Chinese plant to treat secondary aluminum dross for alumina and the chemicals.

4.2.1 Industrial roasting tests for the secondary aluminum dross

The industrial test site is located in a factory area of the company.

A rotary kiln shown in Fig. 4-2 was used for the industrial roasting operation of the mixture of the secondary aluminum dross with additives.

It was a success to obtain about 2000 t of roasting clinker from the rotary kiln, the chemical and mineral compositions of which corresponded to expectation. .



Figure 4-2 Rotary Kiln and Main Control Office Building

The major process technical parameters such as the additive addition, roasting temperature, residence time and adjusts the process parameters in real-time were provided based on the composition of the secondary aluminum dross. The accurate weighting, grinding and mixing of various materials, accurate control of kiln temperature and residence time, fast response and feedback of sampling analysis and detection were ensured in entire industrial test process as shown in Fig 4-3 and 4-4.



Figure 4-3 Central Control Room and Raw Material Storage



Figure 4-4 Secondary aluminum dross discharge and ball mill for mixing and grinding

The temperature of the rotary kiln is controlled in the range of 1150°C - 1250°C to ensure that the secondary aluminum dross fully reacts with additives for expected quality of the roasted clinker. The residence time of the roasting charge in the high-temperature zone is controlled for 20 minutes. Such test data as temperature changes in the rotary kiln, natural gas consumption and raw material input were recorded throughout the entire test process. The appearance and composition of the roasted clinker are shown in Fig. 4-5, Fig 4-6 and Table 4-2.



Figure 4-5 Appearance of the roasted clinker in the industrial tests

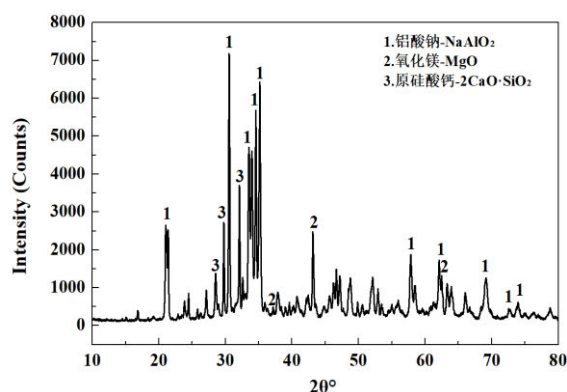


Figure 4-6 Mineral composition of the roasted clinker

The mineral composition results shown in Fig.4-6 indicate that the roasted clinker is mainly composed of sodium aluminate, calcium orthosilicate, and magnesium oxide etc. The chemical composition of the roasted clinker in the industrial tests is shown in Table 4-2.

Table 4-2 Chemical composition of the roasted clinkers

Batches	Al ₂ O ₃ %	Na ₂ O%
1	50	35
2	44.9	31.7
3	50.25	35.37
4	50.94	35.88
5	48.57	34.21
Average	48.93	34.43

4.2.2 Leaching and filtration tests of the roasted clinker

The leaching and filtration tests were conducted in the laboratory of the plant.

The roasted clinker was leached by alkaline liquor of different caustic and

alumina concentrations. And the leached slurry was filtered and separated into sodium aluminate liquor and the residue.

The sodium aluminate liquor could be precipitated by seed addition to produce high white alumina hydrate and other chemicals.

The roasted clinker was ground and leached by alkaline solutions from a Chinese refinery. The appearance of the solutions and the leaching slurry are shown in Fig 4-8 and Fig 4-9.

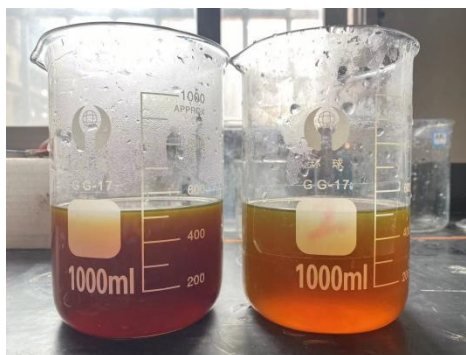


Figure 4-8 Alkaline solutions

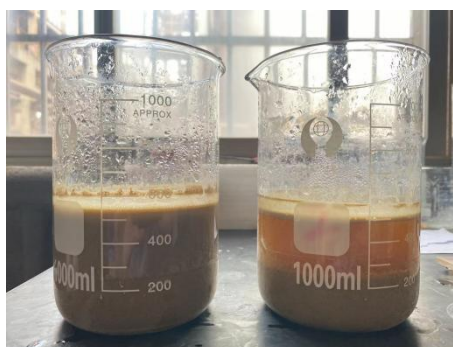


Figure 4-9 Leaching slurry for separation

The components in the residue obtained from the leaching and filtration tests are shown in Fig.4-10.

High leaching rates of alumina and sodium oxide were achieved to >90% with a weight loss of 75-80% of solid by using the optimum leaching

condition of 85 °C and the leaching solution with the modest caustic concentrations developed in the lab tests in order to further combine with normal Bayer process for alumina production.

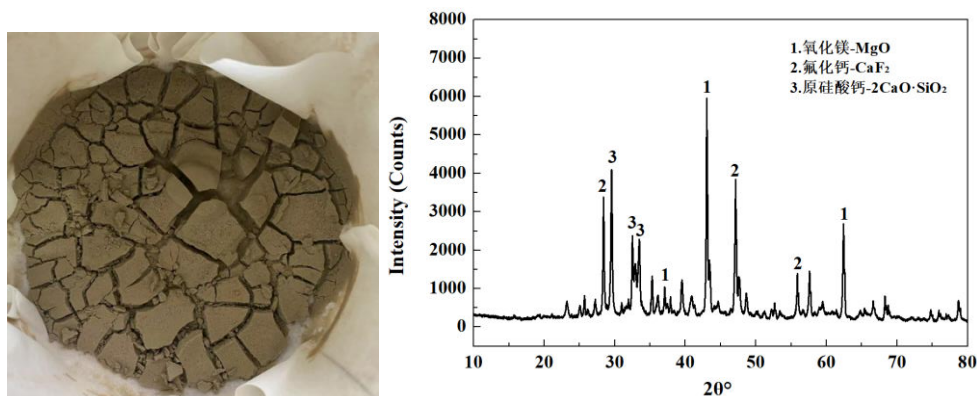


Figure 4-10 Appearance and XRD of the Residue after Leaching

The test results further prove the feasibility of the new process. The XRD results of the leached residue show that the residue is mainly composed of magnesium oxide, calcium fluoride and calcium orthosilicate, indicating that alumina and sodium oxide in the roasted clinker have been efficiently extracted, and all the other impurities have been separated into the residue.

5. Conclusions

(1) A new process to produce alumina from the secondary aluminum dross has been developed in China. It is a large scale treatment technology for the secondary aluminum dross harmful to environment and human health.

(2) This new process has two stage operations:

① Roasting operation of the secondary aluminum dross with alkaline and calcium additives for the roasted clinker, the major component of which is soluble sodium aluminate without any harm substances.

② In the leaching and filtration operation of the roasted clinker sodium aluminate is separated from small amount of residue containing the harmless substances with Magnesium, Silicon and Iron etc.. The sodium aluminate can be used for alumina or chemical production.

(3) The optimal process parameters for roasting process are: $n(N/AF)=1.1$, $n(C/SF)=1.0$, roasting temperature 1100-1120 °C, roasting time 30 min. The roasting clinker under the optimum roasting conditions has the best leaching behavior: the alumina leaching rate $\geq 90\%$ and sodium oxide leaching rate $\geq 95\%$ can be obtained by the normal Bayer solution leaching.

(4) The industrial tests have been conducted in a rotary kiln of a Chinese plant and more than 2000 tons of roasted clinker containing about 900 tons of alumina have been produced. It is proved by the industrial tests that the secondary aluminum dross can be efficiently treated for alumina

production on a large scale and its resource utilization will be feasible and achieve the social and economic benefits.

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