

Article

Intensified Roasting at Low-Temperature and Alkaline Leaching to Efficiently Remove Harmful Elements for Green Utilization of Secondary Aluminum Dross

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Abstract

Secondary aluminum dross (SAD) from the aluminum industry is a hazardous residue that restricts sustainable aluminum production. Residual aluminum nitride (AlN) and insoluble fluorides after conventional pretreatment are major barriers to the safe utilization of SAD. In this study, a low-temperature intensified roasting–alkaline leaching process was developed to remove harmful elements by using reaction heat and waste heat to expose enveloped AlN and fluoride phases, form soluble sodium-bearing compounds, and intensify AlN oxidation. Without additives, roasting at 750 °C for 3 h converted 91.65% of AlN, leaving 1.37% AlN in the roasted SAD. With Na₂O₂ as an oxygen donor in situ, NaF as a mineralizer to reduce roasting temperature, and sodium-bearing species as reactants for soluble NaAlO₂/Na₂SiO₃ formation, the AlN conversion increased up to 97.01% under 5% NaF and 2.5% Na₂O₂ at 750 °C for 3 h. Afterwards, higher temperature, longer duration, lower roasted SAD dosage, and higher caustic soda concentration all improved fluorine removal in the subsequent alkaline leaching. Under 100 g/L Na₂O, 20 g/L roasted SAD, and 100 °C, fluorine and chlorine removal efficiencies reached 92.52% and 99.47%, respectively. The final high-alumina residue contained 74% Al₂O₃, mainly in the forms of α -Al₂O₃, NaAl₁₁O₁₇, and MgAl₂O₄, with only 0.19% F and 0.03% Cl, making it suitable for the preparation of various alumina-bearing materials and alumina production.

Keywords: secondary aluminum dross; removal; roasting; leaching; fluorides; AlN

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1. Introduction

Secondary aluminum dross (SAD), primarily derived from aluminum electrolysis, processing, and recycling [1–3], is classified as hazardous waste and constitutes a major impediment to the sustainable and green development of the aluminum industry [4–7]. The reactive aluminum-bearing species in SAD (e.g., Al, AlN, and Al₄C₃) readily generate flammable, explosive, and toxic gases (such as H₂, NH₃, and CH₄) in aqueous solution and moist air [6,8,9]. Meanwhile, the soluble NaF and NaCl pose a severe risk of soil and groundwater contamination, and a large amount of insoluble fluorides also gives rise to environmental risk. Consequently, the critical challenge for the comprehensive and safe utilization of SAD lies in the economic removal of harmful elements.

Owing to the quick conversion of Al into alumina, many papers have focused on denitrification by converting the reactive AlN into alumina or aluminum hydroxide

through pyrometallurgy and hydrometallurgy [3,5,8,10–17]. Previous denitrification studies largely concentrated on oxidative roasting and additive-assisted activation at high temperature, such as directly roasting at more than 1100 °C, which transformed AlN to α -Al₂O₃ without additives, while low temperature led to 93.8% transformation efficiency [18]. Meanwhile, additives (e.g., Na₂CO₃, CaO, Na₃AlF₆, NaF, cryolite) accelerated the conversion [1,19–21]. However, the enveloped corundum layer (25–45 nm) and the mutual enveloped AlN within the inert corundum/spinel/ β -Al₂O₃ generally leave some residual AlN (1.5–2%) after roasting, posing significant environmental risk. Furthermore, pyrometallurgical treatment often generates some NO under temperature/particle-size heterogeneity [12], even though 99.7% of nitrogen is transformed into N₂ at 1400 °C [13]. Meanwhile, the wet denitrification process has often been reported in water, alkaline and acidic solution without the safe utilization of H₂ from Al and Si [22]. As about 1–3% of AlN remains in SAD after water washing in practice, a dilute solution with less than 20 g/L NaOH is generally defined as a catalyst to promote the hydrolysis of AlN in SAD [11,23]. Although high AlN removal efficiency was achieved, the uncontrolled reaction accompanied by a sustainable exothermal reaction led to danger due to uncontrolled gases. The residual AlN and the irritative NH₃ remaining in the residue notably limit the application of SAD [23]. Therefore, the efficient transformation of enveloped AlN is a key problem.

In contrast to the considerable research on denitrification, the removal of fluorine and chlorine from SAD has often been neglected [7,15,24]. High-temperature pyrometallurgical treatment often volatilizes NaCl, whereas fluorides are difficult to volatilize because of the stability of CaF₂, Na₃AlF₆ and AlF₃. Furthermore, formation of calcium-bearing compounds was believed to efficiently solidify fluorine by adding CaO, Ca(OH)₂ and CaCO₃. In contrast, NaCl is readily removed in hydrometallurgy while fluorine removal is significantly constrained (typically <50%) because of the insoluble CaF₂, MgF₂, Na₃AlF₆ and AlF₃ [9,23,25,26]. Water leaching/multi-stage process [27], sodium aluminate solution [28,29], and concentrated NaOH solution [25] have been adopted. Tang et al. [30,31] reported that sodium aluminate solutions achieved approximately 55% removal efficiency of fluorine in the concentrated sodium aluminate solution (Bayer liquor). Overall, depending on the occurring forms of fluorides, the halide problem generally appears as “Cl easy, F hard” in traditional hydrometallurgical processes. To efficiently remove fluorides, the combined pyro–hydro processes (e.g., water leaching plus roasting) and alternative strategies have been proposed. F and Cl removal efficiency of 87.68% and 99.02% via water leaching at 60 °C following 10 min of ball milling were both achieved [32]. Meanwhile, 97.5% F removal efficiency (reducing residual F content to 0.2%) was achieved by combining water leaching at 80 °C with subsequent roasting at more than 950 °C. The low removal efficiency of fluorides is mainly attributed to the insoluble fluorides and enveloped fluorides encapsulated by the inert alumina [33,34]. Therefore, destroying the inert layer of alumina enveloped on fluorides and AlN is a key process to efficiently remove AlN and fluorides in safe utilization of SAD. An intensified roasting at low-temperature alkaline leaching is then provided to efficiently remove harmful elements through utilizing the waste heat and reaction heat in roasting, dissolving the encapsulating layer during leaching.

Herein, this study first investigates the effects of additives on the conversion of AlN at 750 °C. The influences of leaching temperature, duration, liquid-to-solid ratio, and alkali concentration on fluorine removal are then systematically examined. The phase distribution of the leaching residues is also analyzed to elucidate the underlying mechanisms. A high-alumina residue characterized by low fluorine and zero chlorine content is successfully obtained. The results provide a novel approach to efficiently remove harmful elements in SAD, ultimately facilitating its safe and comprehensive utilization.

2. Experimental

2.1. Material

2.1.1. Secondary Aluminum Dross

SAD was sourced from industrial aluminum electrolysis and processing. As shown in Figure 1, the sample contained inert compounds, reactive substances, and soluble salts. The reactive aluminum-bearing phases consisted predominantly of metallic Al and AlN, whereas the inert aluminum-bearing compounds included MgAl₂O₄ (magnesium–aluminum spinel), α -Al₂O₃ (corundum), and NaAl₁₁O₁₇ (β -Al₂O₃). Fluorine and chlorine were mainly present as CaF₂ and NaCl, respectively. Minor Si and SiO₂ were also detected. Semi-quantitative XRD analysis performed using MDI Jade 9 (Table 1) indicated that α -Al₂O₃ (57.6 wt%) and AlN (16.4 wt%) were the major crystalline phases.

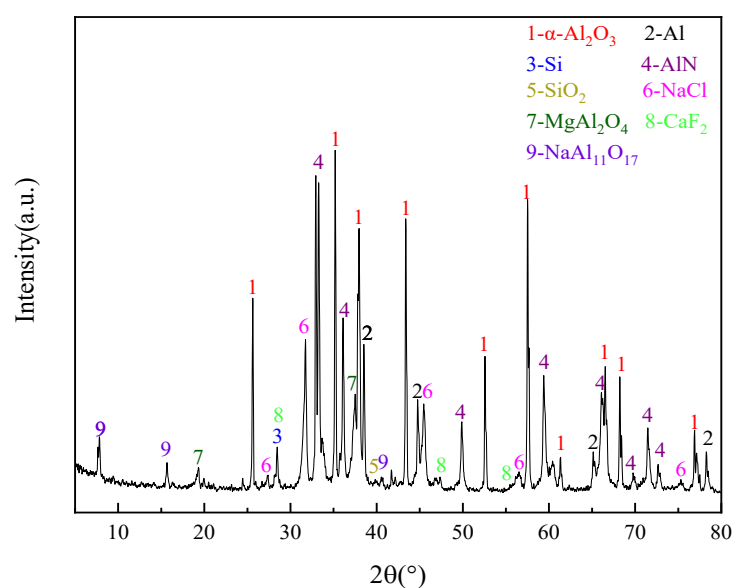


Figure 1. XRD pattern of SAD.

Table 1. Semi-quantitative XRD phase analysis results of SAD (wt%).

	α -Al ₂ O ₃	AlN	Al	MgAl ₂ O ₄	CaF ₂	NaCl	SiO ₂
%	57.6	16.4	6.6	4.9	3.1	3.0	1.3

The particle size distribution (PSD) curve of SAD is shown in Table 2 and Figure 2. SAD was characterized by a wide and heterogeneous size distribution in Figure 2. The predominant fraction occurred between 1 and 100 μ m, yielding a median diameter (d₅₀) of 21.0 μ m.

Table 2. Particle size distribution of SAD.

Sample	d (0.1)/ μ m	d (0.5)/ μ m	d (0.9)/ μ m
Raw SAD	3.43	21.0	85.34

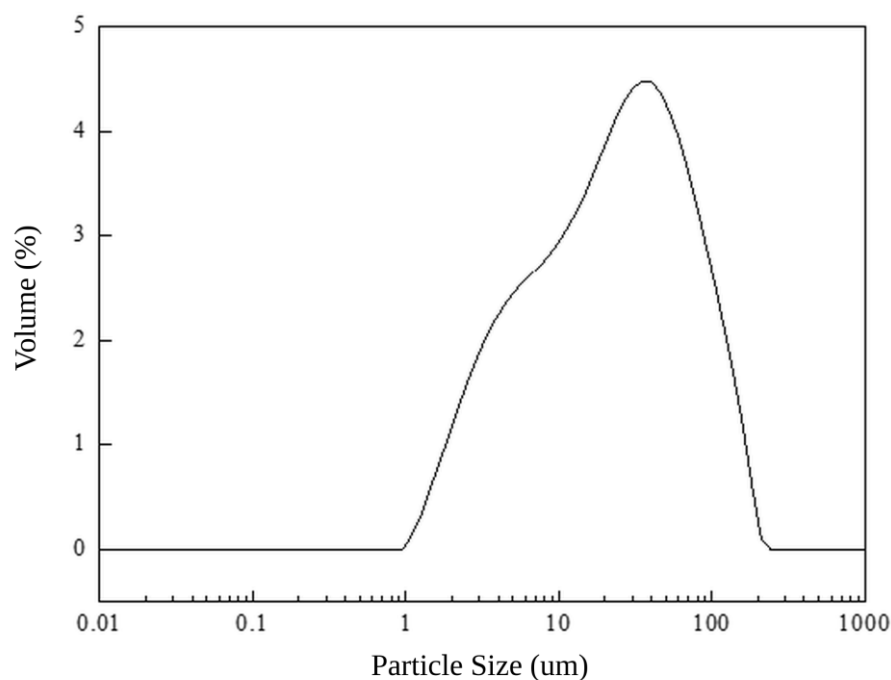


Figure 2. Particle size distribution (PSD) of SAD.

The SEM images and EDS maps of the SAD are shown in Figure 3. Alumina (Al_2O_3), aluminum nitride (AlN), and metallic aluminum (Al) were intergrown and enveloped within one another. Specifically, NaCl existed as large discrete particles, while sodium (calcium, magnesium) fluorides were dispersed within the aluminum-bearing compounds. The distribution of aluminum and oxygen was highly uniform and dense.

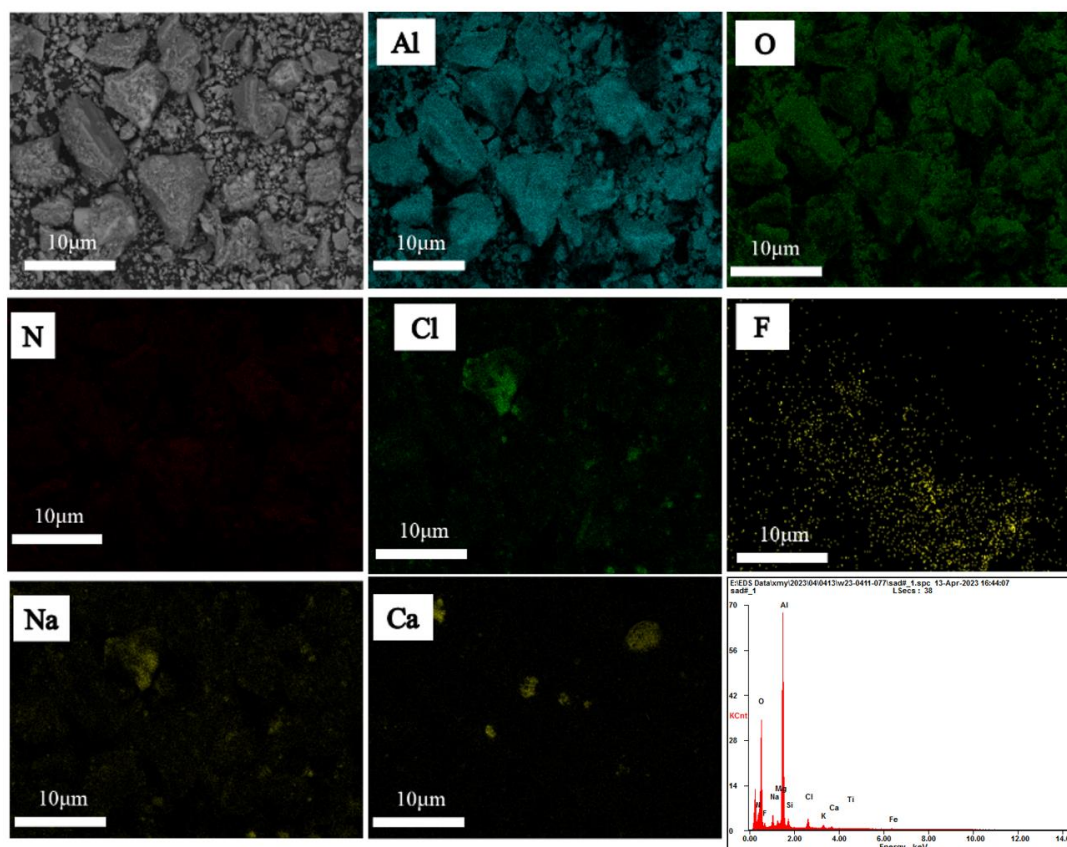


Figure 3. SEM image and EDS mapping of SAD.

In addition, aluminum hydroxide and sodium hydroxide were used to prepare sodium aluminate solution according to the composition of Bayer liquor. The spent liquor had a high molar ratio (α_k) of caustic soda (Na_2O) to alumina (Al_2O_3) in solution.

2.1.2. Experiment Procedure

For each roasting test, 4.00 g of dried SAD was thoroughly mixed with the required additives for 4 h. Additive dosages were calculated as mass percentages relative to the dry SAD mass as Figure 4 shown. For example, 5% NaF and 2.5% Na_2O_2 corresponded to 0.200 g NaF and 0.100 g Na_2O_2 for 4.00 g of SAD. The mixture was placed in a 100 mL corundum crucible, introduced into a muffle furnace, and roasted at the desired temperature for the specified duration under air without additional atmosphere control. After furnace cooling, the clinker was weighed and transferred into 200 mL of sodium aluminate solution for dechlorination and defluorination. Leaching was conducted in a 250 mL thermostatic glass reactor equipped with a reflux condenser and a mechanical stirrer at 400 rpm. The reactor was covered during leaching to minimize evaporation and CO_2 absorption, and the alkaline filtrate environment was controlled by the initial Na_2O concentration and α_k (molar ratio of Na_2O to Al_2O_3) of the sodium aluminate solution. After leaching, the slurry was vacuum filtered. The filter cake was washed with boiling water and dried at 105 °C for 2 h, and the filtrate was sealed immediately for the analysis of F and Cl.

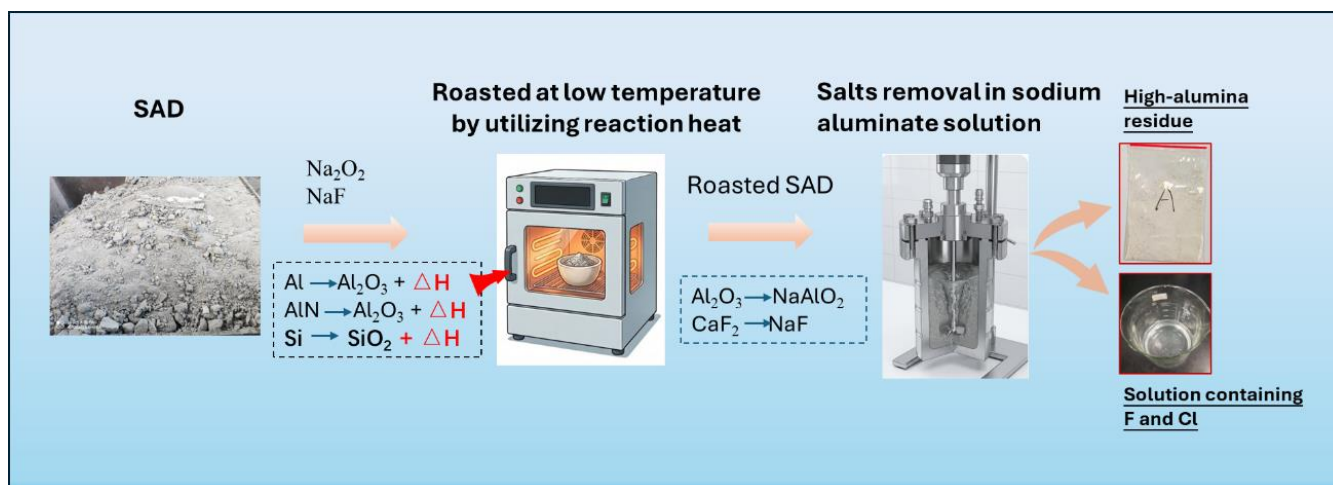


Figure 4. Schematic diagram of the experimental procedure.

2.1.3. Characterizations and Methods

X-ray diffraction (XRD) patterns were obtained using a Rigaku TTR-III diffractometer (Rigaku Corporation, Tokyo, Japan) with $\text{Cu K}\alpha$ radiation at a scanning rate of $10^\circ \text{ min}^{-1}$; the tube voltage and current were 40 kV and 250 mA, respectively. XRD phase identification and semi-quantitative analysis were conducted using MDI Jade 9. The morphology of SAD, roasted products, and residues was characterized by scanning electron microscopy (SEM; JSM-6360LV, JEOL, Tokyo, Japan) at an accelerating voltage of 20 kV. Particle size distribution (PSD) was measured using a laser particle analyzer (MS 2000, Malvern Instruments, Malvern, UK) after sample was dispersed in water with 90 W ultrasonic wave.

Fluorine and chlorine concentrations in the filtrates were determined by ion chromatography after appropriate dilution and calibration with fluoride and chloride standards. Before analysis, the filtrates were filtered through 0.22 μm membranes and diluted with

pure water to ensure that the analyte concentrations were within the calibration range. Duplicate analytical determinations were performed for each filtrate sample to reduce analytical fluctuation. Selected residue compositions were verified by chemical analysis/ICP-based elemental analysis after digestion. The Na₂O and Al₂O₃ concentrations of sodium aluminate solutions were determined by titration. Na₂O and Al₂O₃ concentration of sodium aluminate solution was determined by titration.

AlN content in the raw and roasted SAD samples was calculated by NH₃ volume in the leaching process. The NH₃ volume was then determined from the (NH₄)₂SO₄–H₂SO₄ solution using an NH₃ gas-sensing electrode (PNH3–1, Leici Instrument Co., Ltd., Shanghai, China) [30]. To account for the mass change during roasting, the AlN conversion efficiency was calculated based on both the sample mass and the AlN mass fraction, according to Equation:

$$\eta_{\text{AlN}}(\%) = \frac{w_{\text{AlN},0} - w_{\text{AlN},t}}{w_{\text{AlN},0}} \times 100\%$$

where $w_{\text{AlN},0}$ and $w_{\text{AlN},t}$ are the AlN contents of the raw SAD and the roasted SAD after roasting for t hours, respectively

F (or Cl) removal efficiency was calculated on basis of equation, The F (or Cl) removal efficiency (η_{F} or η_{Cl}) was calculated from the dissolved amount in the leachate relative to the initial element content in SAD.

$$\eta_1(\text{F/Cl}) = \frac{C_1 V_1}{\alpha_1 M} \times 100\%$$

where C_1 and V_1 are concentration (g/L) of fluorine or chlorine in a V volume (mL), respectively. α represents mass percent in SAD, and M is mass of SAD.

The main quantitative trends are reported as average values from duplicate analytical determinations. The duplicate analyses were used to reduce analytical fluctuation, while broader process uncertainty caused by feed heterogeneity and scale-up operation will be further evaluated in pilot-scale experiments [6,8,9,15–17,35].

3. Results and Discussions

3.1. Transformation of AlN and Al in SAD During Roasting at Low Temperature

To inhibit volatilization of halides and evaluate the chemical transformation of the reactive species, Al and AlN reactions during the roasting process at low temperature were carried out. Meanwhile, the effect of additives on AlN transformation was carefully discussed.

3.1.1. Effect of Additives on Al and AlN Reactions in Thermodynamics During Roasting

Both Al and AlN can be converted into alumina spontaneously in air through the reactions listed in Table 3. The standard reaction Gibbs free energy ($\Delta G_{\text{r}\theta}$) for each reaction was calculated using HSC Chemistry 6.0, and the corresponding temperature-dependent curves were plotted using OriginPro (2025 10.2). The calculated $\Delta G_{\text{r}\theta}$ values at 750 °C are also listed in Table 3 to provide a direct quantitative comparison with Figure 5. The strongly negative $\Delta G_{\text{r}\theta}$ for Al oxidation and the negative $\Delta G_{\text{r}\theta}$ value for AlN oxidation (Equations (2)–(6)) indicate the thermodynamic feasibility of these transformations. Among the possible gaseous products, N₂ is expected to be favored, although nitrogen oxides (NO, NO₂, N₂O, and N₂O₅) are also thermodynamically possible under specific oxygen potentials.

Table 3. Possible chemical reactions of Al and AlN during roasting.

No.	Reaction Equations	ΔG_r^θ at 750 °C (kJ mol ⁻¹)
(1)	$\text{Al} + 3/4\text{O}_2 = 1/2\text{Al}_2\text{O}_3$	-744
(2)	$\text{AlN} + 3/4\text{O}_2 = 1/2\text{Al}_2\text{O}_3 + 1/2\text{N}_2$	-428
(3)	$\text{AlN} + \text{O}_2 = 1/2\text{Al}_2\text{O}_3 + 1/2\text{N}_2\text{O}$	-352
(4)	$\text{AlN} + 5/4\text{O}_2 = 1/2\text{Al}_2\text{O}_3 + \text{NO}$	-318
(5)	$\text{AlN} + 7/4\text{O}_2 = 1/2\text{Al}_2\text{O}_3 + \text{NO}_2$	-270
(6)	$\text{AlN} + 2\text{O}_2 = 1/2\text{Al}_2\text{O}_3 + 1/2\text{N}_2\text{O}_5$	-214
(7)	$\text{AlN} + 1/2\text{Na}_2\text{O}_2 = \text{NaAlO}_2 + 1/2\text{N}_2$	-556
(8)	$\text{AlN} + 1/2\text{Na}_2\text{O}_2 + \text{O}_2 = \text{NaAlO}_2 + \text{NO}$	-462

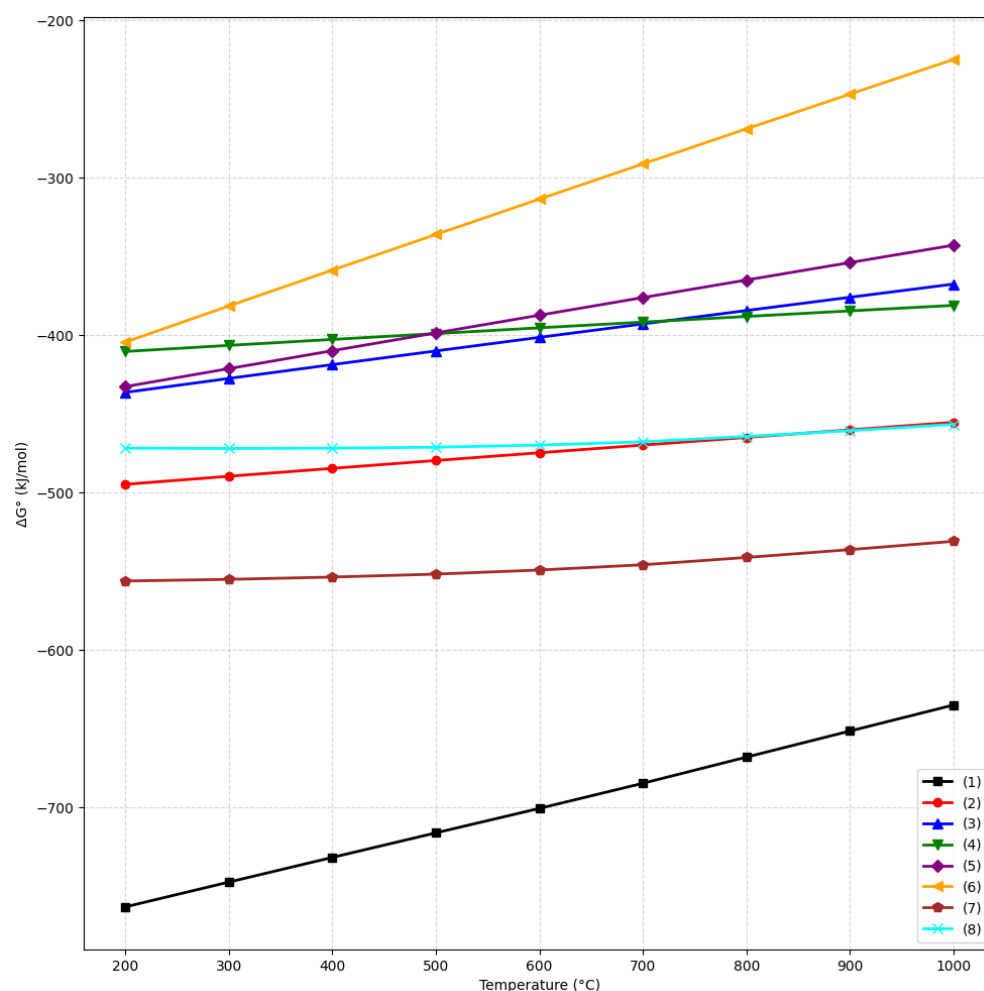


Figure 5. Relationship between reaction Gibbs free energy change (ΔG^θ) and temperature for Reactions (1)–(8).

The thermodynamic results also indicate that the additive-induced reactions of Al and AlN are spontaneous, as evidenced by the negative ΔG_r^θ values. In particular, the reaction between AlN and Na_2O_2 (Equation (7)) has a more negative ΔG_r^θ than the conventional oxidation pathways in Equations (2)–(4), indicating a stronger thermodynamic driving force for NaAlO_2 formation. Equation (8) is also thermodynamically spontaneous. These results support the use of Na_2O_2 -NaF additives to promote the conversion of nitrogen-bearing phases into Al_2O_3 and NaAlO_2 at low temperature.

3.1.2. SAD Traditionally Roasted in the Absence of Additives

SAD was roasted directly at 550 °C, 600 °C, 650 °C, 700 °C, and 750 °C for 3 h, as shown in Figure 6. Elevating roasting temperature remarkably reduced AlN content in SAD. AlN conversion efficiency was 84.63% at 550 °C, while elevating temperature raised conversion efficiency. The conversion efficiency reached its maximum of 91.65% with 1.37% AlN remaining at 750 °C. These results indicate that the complete transformation of AlN is extremely difficult through direct roasting alone [11] when volatilization of chloride and fluoride are inhibited at low temperature.

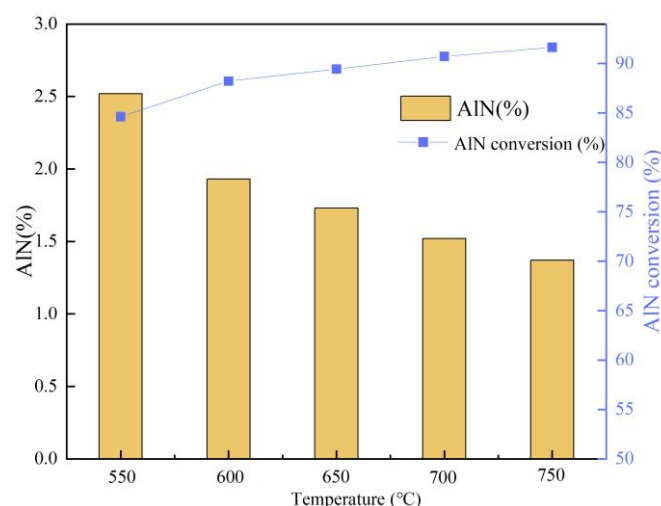


Figure 6. Effect of roasting temperature on the AlN conversion in SAD. Experimental conditions: $t = 3$ h.

XRD patterns of roasted SAD are shown in Figure 7. After roasting at 750 °C for 3 h, the good characteristic peaks were assigned to α - Al_2O_3 , while the peaks for Al and Si were all disappeared, proving complete transformation of Al and Si during roasting. Meanwhile, the intensity of AlN peaks decreased remarkably, except for MgAl_2O_4 , $\text{NaAl}_{11}\text{O}_{17}$, and CaF_2 . These observations confirm that a large portion of the reactive species are oxidized during roasting. Because the reactive AlN were enveloped by the fresh alumina (corundum), O_2 from air has extremely difficulty reacting with AlN through diffusion; some AlN, thereby, always remained in the roasted SAD, as shown in Figure 7. Even though temperature was elevated up to 1200 °C, more than 1% AlN remained in SAD in practice. Furthermore, the distinct peaks of NaCl and CaF_2 in the roasted SAD suggest that the volatilization of NaCl is difficult at 750 °C.

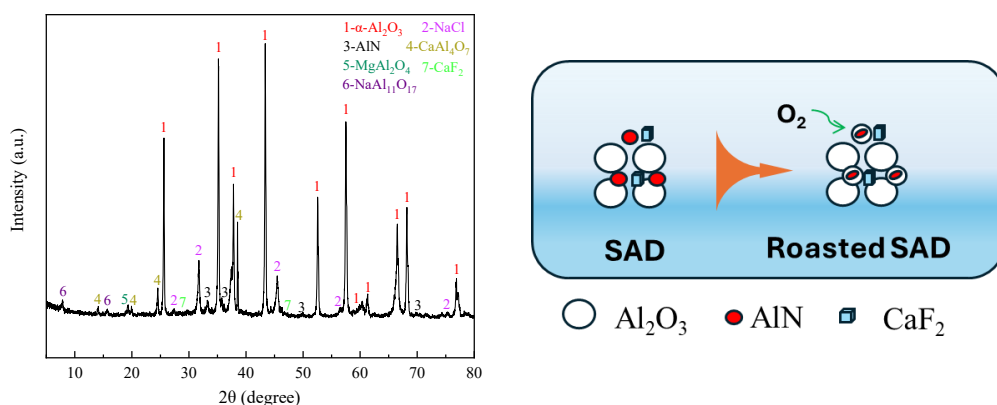


Figure 7. XRD pattern of SAD after direct roasting at 750 °C for 3 h (left) and schematic diagram of AlN remaining in the roasted SAD (right).

3.2. Enhanced AlN Conversion via Intensified Roasting

During the roasting process, the formation of Al_2O_3 on the surface of Al and AlN particles tends to encapsulate the internal AlN, thereby inhibiting further oxidation. Although further increasing the roasting temperature ($>1200\text{ }^\circ\text{C}$) can promote AlN oxidation, it is accompanied by substantial halide volatilization. To accelerate oxidation and enhance the AlN conversion at low temperature to inhibit volatilization of halides, in addition to roasting at low temperature, Na_2O_2 was introduced as a powerful oxidant to enrich O_2 in situ for intensified oxidation on basis of results in Figure 5 (as shown in Figure 8). Additionally, NaF was added as a mineralizer to lower the reaction temperature and further promote the conversion of AlN [20]. Meanwhile, sodium-bearing species also promoted formation of soluble NaAlO_2 and Na_2SiO_3 to destroy the enveloped AlN and fluorides, and efficient removal of harmful elements was possibly achieved. In addition, sodium aluminate solution was used because of reactive compounds converting into sodium aluminate solution in NaOH solution, while $\text{Al}(\text{OH})_3$ was readily precipitated from the saturated sodium aluminate solution, and sodium aluminate solution was then recycled without wastewater. Therefore, the present Na_2O_2 –NaF combination was selected to couple strong oxidation at $750\text{ }^\circ\text{C}$. In this work, the role of NaF is discussed in terms of roasting activation and possible modification of fluoride occurrence, rather than as direct evidence of net fluorine removal. The formation of NaAlO_2 from the Na-bearing Na_2O_2 may benefit further oxidation and fluorine removal. The intensified roasting–alkaline leaching approach is shown in Figure 8.

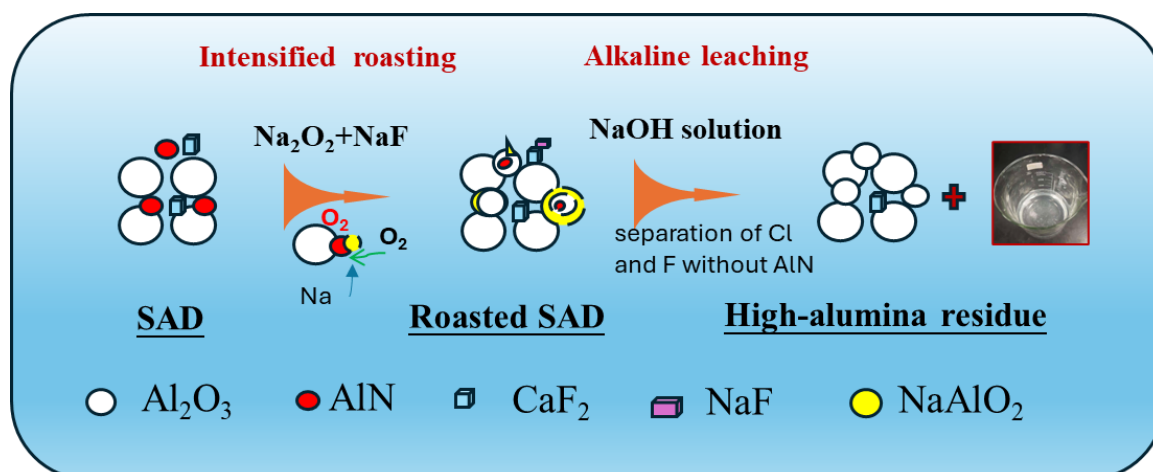


Figure 8. Schematic diagram of harmful element removal by adding additives roasted at low temperature.

The enriched O_2 from Na_2O_2 and air both raised the oxidation of AlN, and NaF reduced roasting temperature, both leading to high conversion of AlN. Meanwhile, sodium from Na_2O_2 and NaF favored the formation of the soluble NaAlO_2 , benefiting the destruction of the enveloped AlN and fluorides. All raised the removal efficiency of the harmful elements.

3.2.1. Effect of Oxidization Dosage

Na_2O_2 is a sodium-bearing oxidant. Its decomposition supplies O_2 in situ, which promotes the oxidation of AlN and Al during roasting. The effect of Na_2O_2 dosage on AlN conversion is shown in Figure 9.

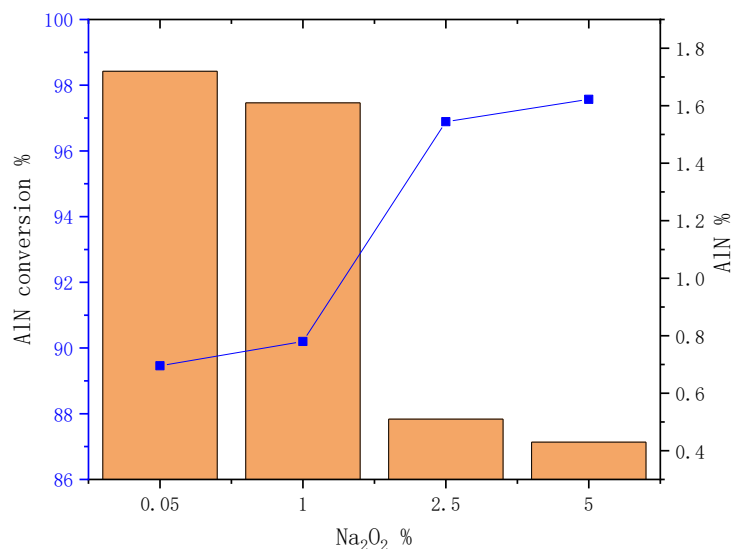


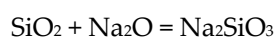
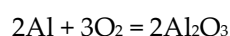
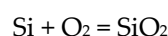
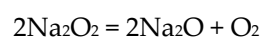
Figure 9. Effect of Na₂O₂ dosage on AlN conversion in SAD. Experimental conditions: SAD + 5% NaF + 2.5% Na₂O₂; T = 750 °C; t = 3 h.

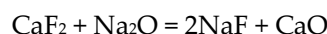
From Figure 9, adding Na₂O₂ raised AlN conversion efficiency, which is in good agreement with the results in Figure 5. Meanwhile, the 96.89% AlN conversion efficiency was more than the 91.65% at 750 °C in Figure 6. However, adding 5% Na₂O₂ slightly increased AlN conversion compared with the results from adding 2.5% Na₂O₂. Therefore, 2.5% Na₂O₂ was adopted in the following experiments. In addition, the masses of chlorine and fluorine in the roasted SAD were nearly equal to those in SAD, proving the stability of halides at low roasting temperature (NaCl melting point 801 °C, NaF melting point 996 °C).

3.2.2. Effect of Roasting Temperature

The variation in AlN conversion efficiency as a function of roasting temperature is shown in Figure 10a. As the roasting temperature increased, the AlN conversion efficiency increased continuously. At the same temperature, the additive-assisted roasting shown in Figure 10a was more effective than the direct roasting shown in Figure 6 because Na₂O₂ supplied active oxygen in situ, and sodium-bearing phases promoted the formation of soluble NaAlO₂/Na₂SiO₃, which weakened the encapsulating alumina layer. The conversion efficiency reached 97.01% at 750 °C, which was higher than the 91.65% obtained by direct roasting (Figure 6). The higher AlN conversion is expected to reduce the risk of NH₃ release during subsequent aqueous treatment.

The XRD patterns of the roasted products are presented in Figure 10b. In addition to α-Al₂O₃, NaAl₁₁O₁₇, CaF₂, and MgAl₂O₄, new compounds such as NaF, Na₂SiO₃, NaAlO₂, and SiO₂ were all observed, while the characteristic peaks of Al and Si both disappeared. The following reactions are proposed to interpret the observed phase evolution during roasting:





Furthermore, both the number and intensity of AlN diffraction peaks decreased significantly. When the roasting temperature exceeded 650 °C, no obvious AlN diffraction peaks were detected. Meanwhile, the diffraction peaks of Al_2O_3 were intensified with increasing temperature, suggesting that more conversion of Al and AlN to Al_2O_3 occurred. This behavior is consistent with the high oxygen availability generated by Na_2O_2 decomposition. The results in Figures 6 and 7 suggest that additives remarkably raised the oxidation of AlN in Figure 10. Formation of the soluble NaF, Na_2SiO_3 and NaAlO_2 at low roasting temperature then benefits the destruction of the alumina layer enveloping AlN and fluorides, and accordingly raises the removal efficiency of fluorides in the leaching process compared to the uncovered fluorides from the dissolution mentioned above in Figure 8 and reactions. However, minor AlN still remained because some particles were tightly enveloped within the SAD matrix [16].

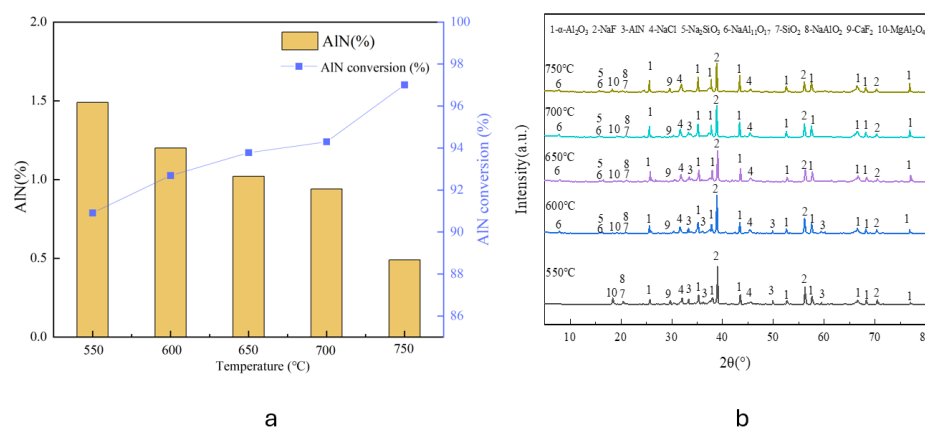


Figure 10. (a) Effect of roasting temperature on the AlN conversion in SAD; (b) XRD patterns of roasted products at different temperatures. Experimental conditions: SAD + 5% NaF + 2.5% Na_2O_2 ; $t = 3$ h.

3.2.3. Effect of Roasting Duration

The variation in the AlN conversion efficiency as a function of roasting duration is shown in Figure 11. The AlN conversion efficiency in the SAD reached approximately 90% at 750 °C for 0.5 h, while the conversion efficiency reached up to 96.95% for 2 h. Further extension of the roasting time slightly raised the conversion efficiency at 3 h. This fact is mainly due to the minor residual AlN encapsulated by $\alpha\text{-Al}_2\text{O}_3$ rather than NaAlO_2 , without enough Na-bearing species.

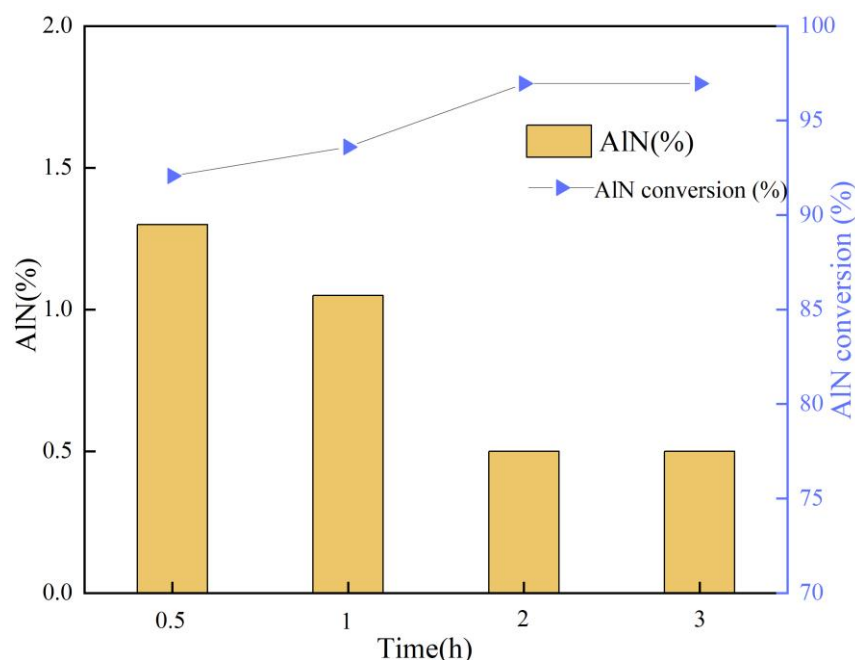


Figure 11. Effect of roasting duration on the AlN conversion in SAD. Experimental conditions: SAD + 5% NaF + 2.5% Na₂O₂; T = 750 °C.

Therefore, 100% AlN in SAD was difficult to convert in the traditional roasting process without additives. Adding Na₂O₂ and NaF remarkably increased AlN conversion and eliminated detectable Al and Si through enriched O₂ in situ from Na₂O₂ and the formation of NaAlO₂ and Na₂SiO₃. These soluble Na-bearing phases (NaAlO₂, Na₂SiO₃ and NaF) may also contribute to subsequent fluoride leaching.

3.3. Enhanced Removal of Fluorine and Chlorine from SAD via Intensified Roasting

Although AlN was efficient with additives at 750 °C with approximately 97% of AlN conversion, high removal efficiency of the fluorides is urgently required. In the presence of NaAlO₂, Na₂SiO₂ and NaF, salts containing chlorine and fluorine were then efficiently removed. To further investigate the effect of intensified roasting on chlorine and fluorine removal, the SAD and SAD roasted at 750 °C were then utilized for dechlorination and defluorination in an alkaline solution. The results are displayed in Table 4.

Table 4. Dechlorination and defluorination of SAD in alkaline solution after roasting at 750 °C without additives.

Roasting Conditions		Reaction Conditions	Fluorine Removal Efficiency (%)	Chlorine Removal Efficiency (%)
SAD	/	Na ₂ O 50g/L, α _k 3.0; 25 °C, 3 h; 20g/LSAD	54.42	85.40
Roasting SAD	750 °C, 3 h	Na ₂ O 50g/L, α _k 3.0; 100 °C, 3 h; 20g/LSAD	72.14	99.90

Removal efficiencies of chlorine and fluorine in the dilute sodium aluminate solution were 85.40% and 54.42%, respectively. Meanwhile, the fluorine removal efficiency increased to 72% and the chlorine removal efficiency reached nearly 100% in an alkaline solution at 100 °C after roasting at 750 °C. The circumstances suggest the roasting process remarkably raises the removal of salt in SAD. However, owing to the insoluble fluorides and enveloped fluorides in SAD, the fluorine content in the residue remained above 0.5%, still classifying it as high-fluorine residue with high environmental risks. Therefore,

intensified roasting processes to raise the conversion efficiency of AlN and the soluble NaAlO₂ covering AlN and fluorides is required.

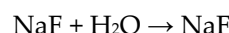
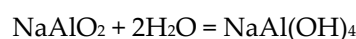
In addition, as the removal efficiency of chlorine was nearly 100%, the effect of the intensified roasting process on the removal efficiency of fluorine was then investigated in detail.

3.3.1. Effect of Leaching Temperature

The roasted SAD produced at 750 °C for 3 h with Na₂O₂ and NaF was used to investigate fluorine removal and the phase evolution of the high-alumina residue after leaching.

The effect of leaching temperature on fluorine removal from the roasted SAD is shown in Figure 12a. The fluorine removal efficiency increased from 76.06% at 50 °C to 83.16% at 100 °C. Compared with the results in Table 4, intensified roasting markedly enhanced fluorine removal. This improvement is attributed to the disruption of enveloped AlN/fluoride associations, dissolution of soluble NaAlO₂ and Na₂SiO₃, and partial conversion or exposure of fluoride-bearing phases during roasting (Figure 8).

XRD patterns at different leaching temperatures are shown in Figure 12b. The inert α -Al₂O₃, NaAl₁₁O₁₇ and MgAl₂O₄ remained in the residue, whereas NaAlO₂, Na₂SiO₃ and NaF all disappeared because of their ready dissolution in the dilute sodium aluminate solution, as shown in the following equations.



where s represents solid, and l stands for liquid.

Furthermore, the characteristic peaks of CaF₂ weakened significantly. This phase evolution suggests that intensified roasting partially altered the occurrence of fluoride-bearing phases and exposed part of the previously less accessible fluorides to alkaline leaching, as shown in Figure 8. Increasing temperature also accelerated mass transfer between the solid and liquid phases, thereby facilitating the dissolution of fluorine. Therefore, elevating temperatures were favorable for fluorine removal from the roasted SAD.

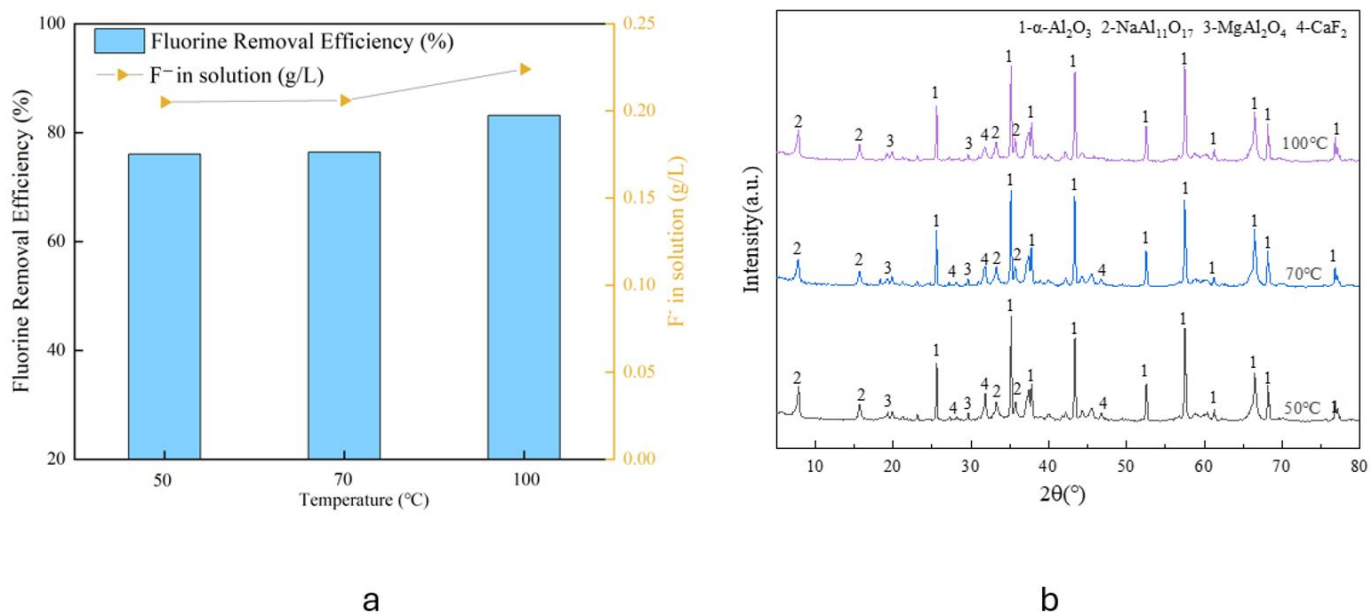


Figure 12. (a) Effect of leaching temperature on the fluorine removal from the roasted SAD; (b) XRD patterns of the leaching residues at different temperatures. Experimental conditions: sodium aluminate solution with $\text{Na}_2\text{O} = 30 \text{ g/L}$, $\alpha\text{k} = 3.0$; $t = 3 \text{ h}$; $\text{S/L} = 20 \text{ g/L}$.

3.3.2. Effect of Leaching Duration

Leaching duration is related to production cost. The effect of duration on fluorine removal is shown in Figure 13. Because soluble Na-bearing compounds dissolved rapidly, extending the leaching duration had only a minor effect on fluorine removal efficiency. The fluorine removal efficiency was 81.8% after 1 h and increased by only approximately 2% after 4 h, with the fluorine ion concentration in solution reaching 0.234 g/L.

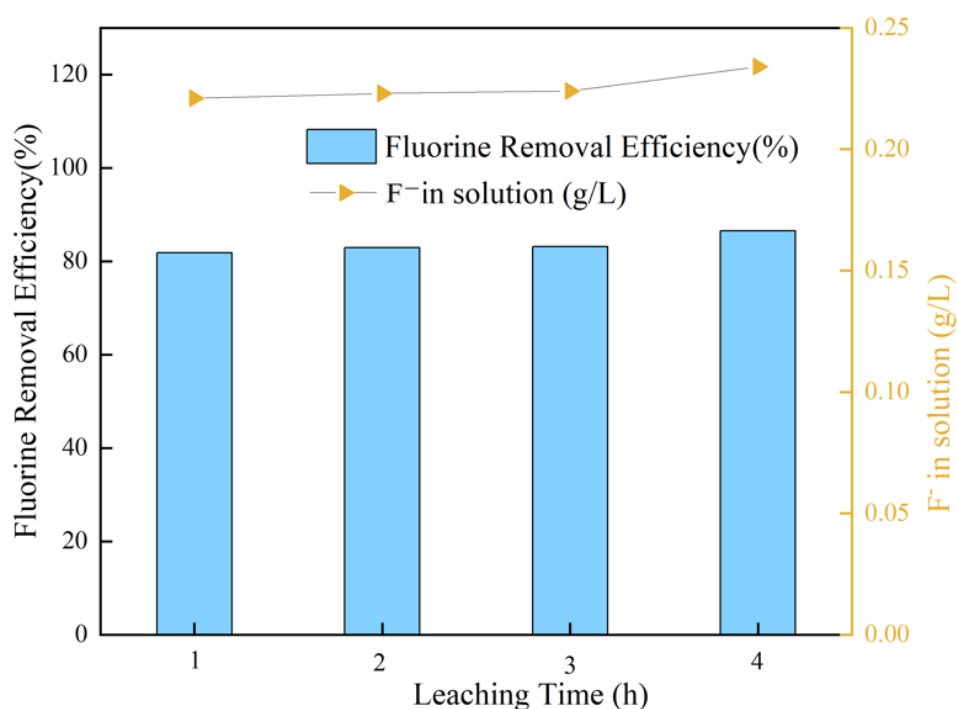


Figure 13. Effect of leaching duration on the fluorine removal from the roasted SAD. Experimental conditions: sodium aluminate solution with $\text{Na}_2\text{O} = 30 \text{ g/L}$, $\alpha\text{k} = 3.0$; $T = 100 \text{ }^\circ\text{C}$; $\text{S/L} = 20 \text{ g/L}$.

3.3.3. Effect of Liquid-to-Solid Ratio

The liquid-to-solid ratio determines the fluorine concentration of leachate and fluorine removal efficiency. From Figure 14a, increasing the dosage of the roasted SAD slightly reduced the removal efficiency of fluorine. The fluorine removal efficiency decreased from 83.15% at 20 g/L to 79.53% at 200 g/L, respectively. In contrast, raising the dosage of the roasted SAD notably raised fluorine concentration. Fluorine concentration was 2.2 g/L with the addition of 200g/L roasted SAD. Concentrated fluorine-bearing solutions will benefit the recovery of value-added fluorides.

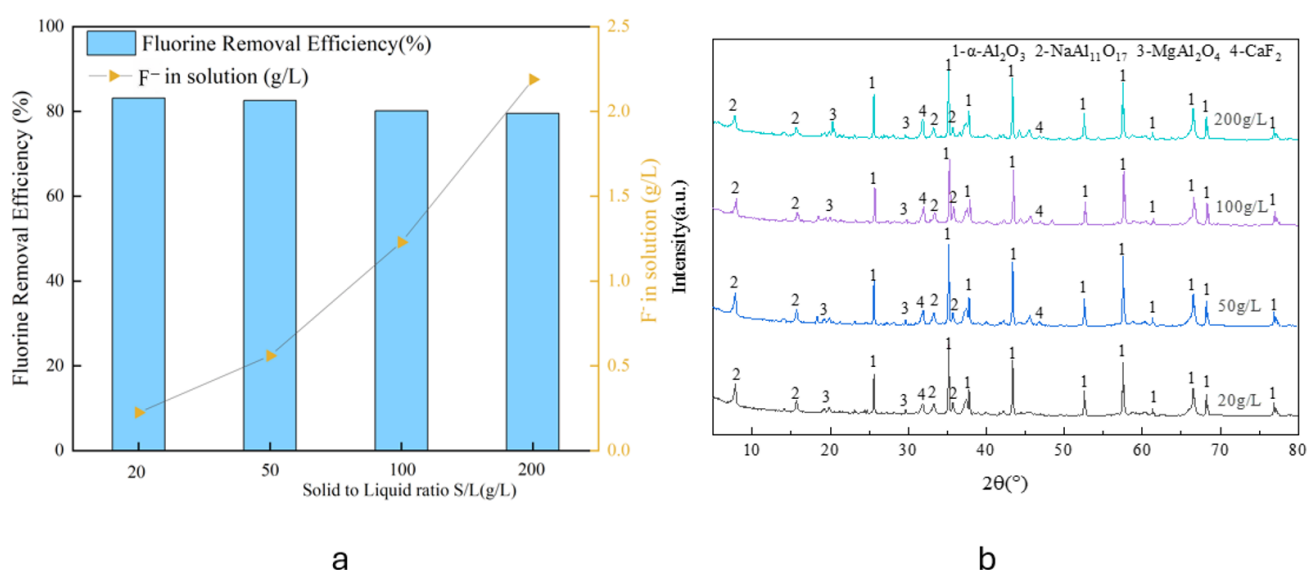
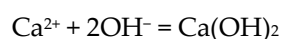
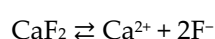


Figure 14. (a) Effect of liquid-to-solid ratio on the fluorine removal from the roasted SAD; (b) XRD patterns of the leaching residues at different liquid-to-solid ratios. Experimental conditions: sodium aluminate solution with Na₂O = 30 g/L, αk = 3.0; T = 100 °C; t = 3 h.

The corresponding XRD patterns of the residues in Figure 14b show progressively strengthened CaF₂ diffraction peaks with increasing roasted SAD, consistent with a large fraction of fluoride remaining in the residue. This inhibition is attributed to the increased viscosity of the sodium aluminate solution from high alumina concentration and fluorine concentration, thereby reducing fluoride solubility, and limiting the transfer of fluorine into the sodium aluminate solution.

3.3.4. Effect of Caustic Soda Concentration

Caustic soda concentration affects the dissolution equilibrium of fluoride-bearing phases, as rewritten by the following reactions:



Furthermore, raising caustic solution also improves the stability of the sodium aluminate solution with high molar ratio of αk, benefiting the removal of fluorides.

Figure 15a shows that increasing the Na₂O concentration from 10 to 100 g/L raised the fluorine removal efficiency from 82.64% to 92.52%, while the fluoride concentration in solution increased from 0.223 to 0.25 g/L. These results indicate that intensified roasting increased the fraction of fluorine that could be transferred into the dilute sodium aluminate solution, although the present data do not directly prove a specific CaF₂-to-NaF pathway.

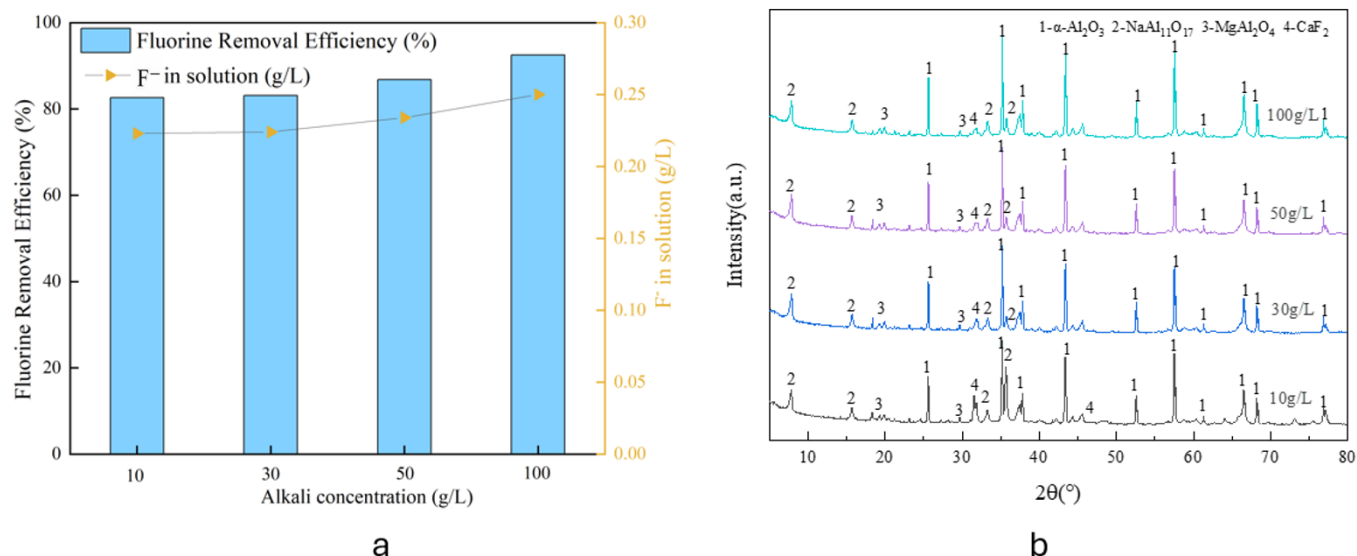


Figure 15. (a) Effect of alkali concentration (Na₂O) on the fluorine removal from the roasted SAD; (b) XRD patterns of the leaching residues at different alkali concentrations. Experimental conditions: T = 100 °C; t = 3 h; S/L = 20 g/L.

XRD patterns of the residues in Figure 15b confirm that the inert α -Al₂O₃, NaAl₁₁O₁₇ and MgAl₂O₄ remained in the residue. The residual CaF₂ peak intensities weakened systematically with increasing Na₂O concentration, indicating improved dissolution and/or liberation of fluoride-bearing phases in the sodium aluminate solution.

Therefore, different from NaOH leaching and roasted SAD at high temperature without additives, 83.16% and 92.52% removal efficiency of fluorine was achieved in the dilute sodium aluminate solution and concentrated solution at 100 °C, leading to minor fluorine in the residue without reactive species. Harmful elements were all efficiently removed. Moreover, a high concentration of fluorine was enriched in the sodium aluminate solution.

3.4. High-Alumina Residue After Intensified Roasting–Leaching Process

The chemical composition of the high-alumina residue after the leaching process is presented in Table 5. The chlorine content in the residue was 0.03%, representing a removal efficiency of 99.47%, while the fluorine content remained at 0.19%. A total of 74% Al₂O₃ remained in residue with 12.3 mass ratio of alumina to silica, characterized by a high grade of alumina-bearing materials to produce alumina. In addition, 0.27% of sodium was detected in the residue, which is mainly attributed to the formation of desilication products.

Table 5. Composition and contents of the leaching residue (wt%).

Element	Al	O	Na	Si	K	Mg	Fe	Ca	F	Cl
Content (wt%)	39.22	26.32	0.27	2.81	1.29	1.28	0.75	1.01	0.19	0.03

Figures 16 and 17 present the XRD pattern and SEM images of the low fluorine in the high-alumina residue, respectively. The high-alumina residue consisted of the inert α -Al₂O₃, NaAl₁₁O₁₇ and MgAl₂O₄. The weak peaks assigned to CaF₂ were also found, and its intensity was much less than that in SAD in Figure 1. The high-alumina residue then acts as raw material in alumina production, with more than 14.4 million tons in 2025 [36]. Furthermore, the residue is also used as raw material for producing refractories [37], cements [38], ceramics [39] and water purifiers [27] without halide volatilization.

SEM analysis reveals that the residue possesses an irregular morphology, primarily characterized by large, coarse particles. The observed high specific surface area suggests significant potential for adsorbent applications [28,29]. Detailed investigations into the further high-value utilization of this alumina-rich residue will be carried out in the future.

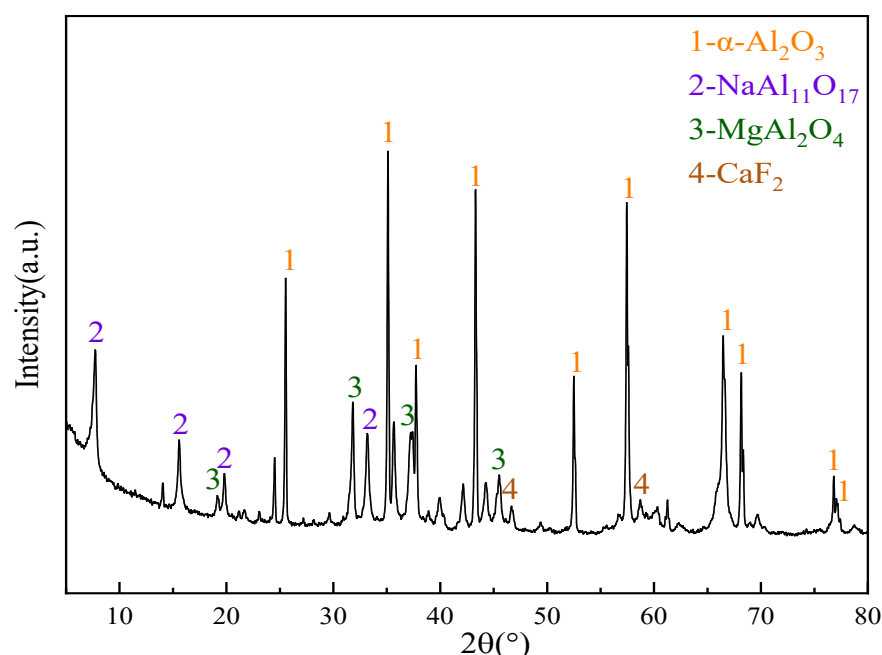


Figure 16. XRD pattern of the residue after roasting-leaching process.

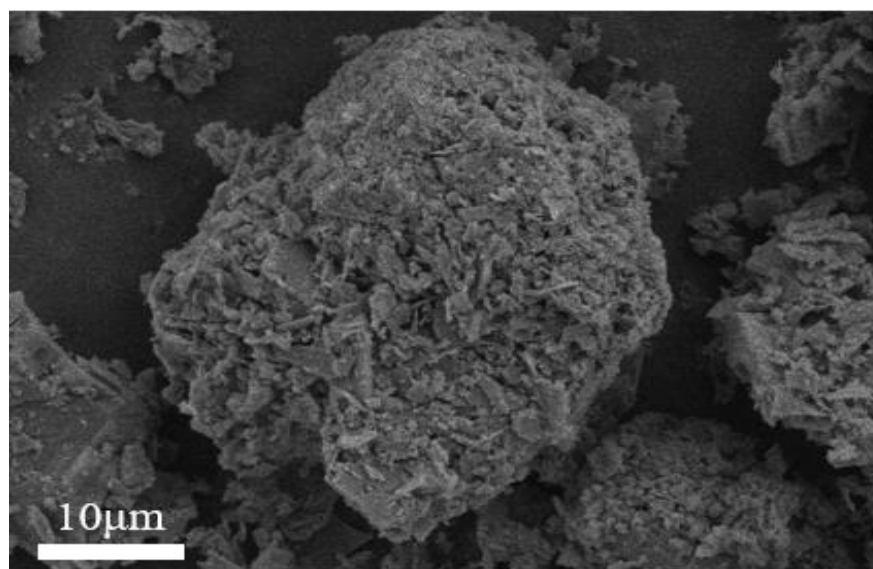


Figure 17. SEM image of high-alumina residue.

3.5. Safe Utilization of SAD Through Efficient Removal of Harmful Elements

Different from the preparation of notable value-added alumina-bearing products (nano-alumina, water purifier, absorbent, ceramics etc.) through the complex long pyrometallurgy-hydrometallurgy process, value-added high-alumina residue was produced by the following intensified roasting-leaching in the sodium aluminate solution without halide volatilization, residual AlN and NH₃, high content of fluorine in products and expensive utilization of irritant and explosive gases (Figure 18).

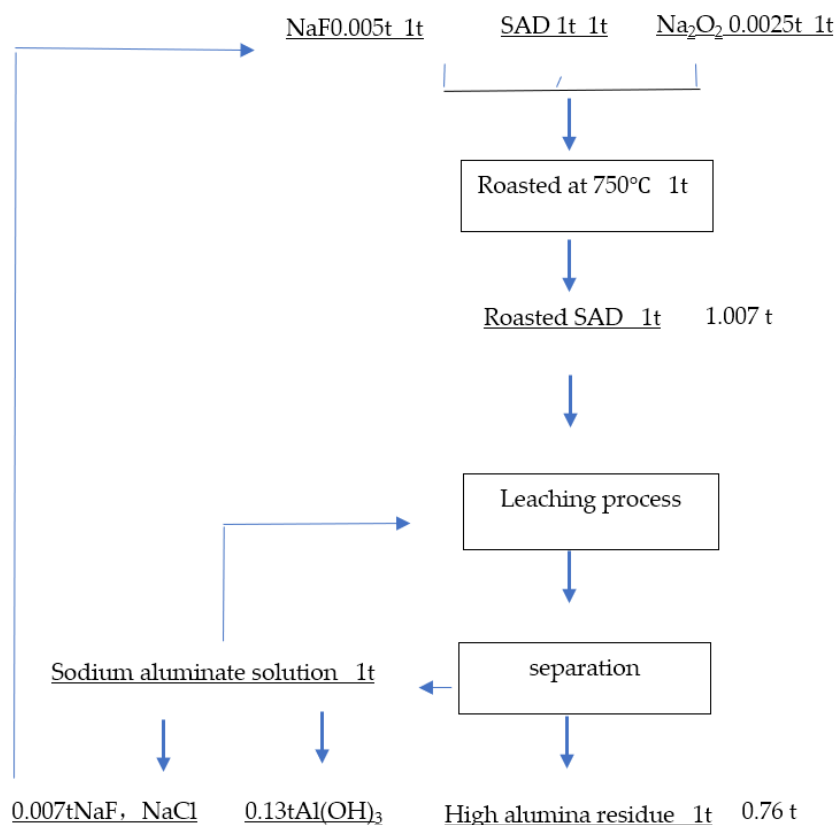


Figure 18. Intensified roasting–leaching process to utilize SAD.

Partial NaF was used as additives, while spent sodium aluminate solution returned to the leaching process, both contributing to the green process.

Compared with the traditional route to remove harmful elements, the novel approach provides promising application due to diverse advantages, as shown in Table 6. Without the adverse effects of chlorine, fluorine and AlN, the high-alumina residue can be used as a raw material for alumina production, and its potential applications in refractories, cement, ceramics, and water-purification materials are retained. Ultrafine alumina trihydrate (gibbsite) can be precipitated from dilute sodium aluminate solution, while NaCl and NaF can be recovered from concentrated sodium aluminate solution. The sodium aluminate solution can then be recycled, reducing wastewater generation.

In addition, the high-alumina residue has economic potential because of its high alumina content and high alumina-to-silica mass ratio. It may be used as a raw material for alumina, refractories, cement, ceramics, calcium aluminate, and sodium aluminate production, provided that product-specific quality requirements are satisfied.

The recovery of $\text{Al}(\text{OH})_3$, NaCl, and NaF would further improve process economics. However, the product-recovery route and cost–benefit balance should be verified in pilot-scale tests.

Pilot-scale experiments and a detailed techno-economic analysis will be carried out in future work to evaluate industrial applicability.

Table 6. Comparison between Na_2O_2 –NaF roasting–leaching process and representative conventional SAD treatment routes.

Route	Typical Conditions	AlN-Related Performance	F/Cl Removal Performance	Main Limitation	Reference
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Direct oxidative roasting.	>1100 °C without additives; direct roasting at 750 °C in this work.	Most AlN can be converted at high temperature; 91.65% AlN conversion efficiency in this work.	F/Cl separation was not the main target.	High energy demand; halide volatilization at high temperature; residual AlN remained at low temperature. [15–18]; repeated experiments in this work.
Additive-assisted roasting with CaO/Na ₂ CO ₃ /cryolite/NaF.	Typically 750–1400 °C with mineralizers or Ca/Na-bearing additives.	Improved denitrification and phase transformation of AlN and Al.	Cl separation by volatilization, F remained in roasted SAD.	Fluorine immobilized as CaF ₂ ; high additive dosage or high temperature. [15–17,20,21].
Water/alkaline leaching.	Ambient temperature to 100 °C in water, NaOH, or sodium aluminate solution.	AlN hydrolysis, producing NH ₃ and H ₂ .	Efficient NaCl removal, inefficient F removal from the insoluble CaF ₂ , MgF ₂ , Na ₃ AlF ₆ , and AlF ₃ .	Substantial gas released from Al/AlN hydrolysis; high content of F in residue after defluorination. [6,8,9,11,25,30,31]
Combined mechanical/leaching or pyrohydro routes.	Ball milling–water leaching, water leaching–roasting, roasting–leaching.	Efficiency of AlN, Al and Cl.	F removal efficiency up to 87.68–97.5%; Cl removal up to 99.02%.	Additional pretreatment, high roasting temperature and high content of F in residue. [32–34].
This work: Na ₂ O ₂ –NaF intensified roasting–sodium aluminate leaching.	750 °C roasting with additives leaching in solution with 100 g/L Na ₂ O.	97.01% AlN conversion in roasting process, 100% removal efficiency of AlN, Al and Cl.	92.52% F removal; residue contained 0.19% F and 0.03% Cl.	Additive recycling, low temperature roasting, off-gas/dust control, halides recovery from solution and high-alumina residue. This work.

Because the process operated at 750 °C, below the melting points of NaCl (801 °C) and NaF (996 °C), halide volatilization is efficiently restrained. Meanwhile, the high conversion of AlN significantly benefited the leaching process. Waste heat, reaction heat and low-temperature roasting all contributed to the green process. Overall, the proposed process shows promise as a safe, cleaner, and potentially economical route for the comprehensive utilization of SAD, although pilot-scale validation and techno-economic analysis are still required.

4. Conclusions

Increasing the roasting temperature promoted AlN conversion, reaching 91.65% conversion at 750 °C for 3 h without additives. With Na₂O₂ supplying O₂ in situ and NaF acting as a mineralizer, 5% NaF and 2.5% Na₂O₂ increased the AlN conversion efficiency to 97.01% at 750 °C for 3 h. The formation of NaF, Na₂SiO₃, and NaAlO₂ then contributed to the conversion of AlN and Al and improved the subsequent leaching behavior by uncovering the enveloped AlN and fluorides.

Roasting markedly enhanced chlorine and fluorine removal. Higher leaching temperature, longer duration, lower roasted SAD dosage, and higher caustic soda concentration all improved fluorine removal. Under 100 g/L Na₂O, 20 g/L roasted SAD, and 100 °C, fluorine and chlorine removal efficiencies reached 92.52% and 99.47%, respectively.

The efficient removal of harmful elements was attributed to intensified oxidation, the disruption of enveloped AlN/fluoride associations, and the integration of low-temperature roasting with alkaline leaching. The final residue contained 74% Al₂O₃ mainly as α-Al₂O₃, NaAl₁₁O₁₇, and MgAl₂O₄, with only 0.19% F and 0.03% Cl. The residue, Al(OH)₃, NaCl, and NaF may be recovered as value-added products, while NaF and the sodium

aluminate solution can be recycled to reduce halide volatilization and wastewater discharge.

The proposed low-temperature intensified roasting process detoxifies SAD and enables efficient fluorine/chlorine separation through a relatively simple recycling-oriented route. This provides a feasible approach for the safe and high-value utilization of SAD.

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