

## Industrial Application of Sodium Nitrate Oxidation Treatment Technology for High-Sulphur Bauxite

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### Abstract

With the rapid development of China's alumina industry, its alumina output in 2024 already accounted for 56 % of the world's total. However, as domestic high-quality bauxite resources are increasingly depleted, a large amount of high-sulphur bauxite cannot be directly used in alumina production due to its severe impacts on product quality and equipment corrosion during the Bayer process, becoming a bottleneck restricting the sustainable development of the industry. To address this technical challenge, this paper analyses the oxidation mechanism of sulphur during the digestion of the Bayer process and conducts experimental research on oxidative desulphurization using sodium nitrate (NaNO<sub>3</sub>). After 12 months of continuous operation verification in a 1-Mtpa alumina production line, the results demonstrate that when the sulphur content in bauxite does not exceed 0.5 %, adding sodium nitrate during the bauxite blending process can effectively eliminate the influence of low-valence sulphur, ensuring the iron content in alumina products remains within the first-grade standard. This technology is simple, technically feasible, economically reasonable, and can be promoted in industrial production using high-sulphur bauxite.

**Keywords:** High-sulphur bauxite, Bayer process, Sodium nitrate, Oxidative desulphurization.

### 1. Introduction

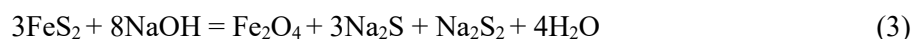
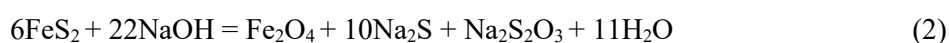
With the rapid development of China's alumina industry, its alumina output in 2024 already accounted for 56 % of the world's total. However, domestic bauxite-based alumina refineries generally face an increasingly acute shortage of high-quality bauxite resources, while simultaneously sitting on massive reserves of high-sulphur, high-carbon bauxite that cannot be directly processed via the Bayer process. According to statistics, China has proven reserves of high-sulphur, high-carbon bauxite amounting to hundreds of millions of tonnes. During high-temperature and high-pressure digestion, sulphur (mainly in low-valence forms) and organic carbon extensively dissolve into the solution, leading to increased viscosity, reduced precipitation efficiency, difficulties in evaporation and salt removal, elevated iron content in aluminium hydroxide products, and diminished whiteness. Additionally, these issues cause severe equipment corrosion and tank leakage problems.

To address the negative impact of sulphur in high-sulphur bauxite on alumina production, flotation desulphurization is applied at the front end of the process, while roasting-based pyrometallurgical desulphurization and wet oxidation desulphurization are primarily employed during alumina production. Other methods, such as electrolytic desulphurization, are also under research [1, 2]. Both flotation desulphurization and roasting desulphurization require the construction of large-scale production units in industrial applications, with investments exceeding hundreds of millions of RMB and high operational costs. Additionally, flotation desulphurization introduces significant amounts of attached water and flotation reagents into the bauxite concentrate, while roasting desulphurization suffers from relatively high energy consumption.

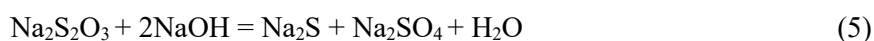
Regarding this technical challenge, this paper analyses the oxidation mechanism of sulphur during the digestion of the Bayer process. Experimental studies were conducted by adding sodium nitrate as an oxidant. After 12 months of industrial application in a 1-Mtpa alumina production line, the results demonstrate that when the sulphur content in bauxite does not exceed 0.5 %, the addition of sodium nitrate during bauxite blending can eliminate the impact of low-valence sulphur, ensuring the iron content in the alumina product remains within the premium-grade range. This technology does not require the construction of large-scale treatment facilities, as sodium nitrate can be directly mixed into the raw materials. It is simple, effective, and suitable for widespread adoption in alumina refineries utilizing high-sulphur bauxite.

## 2. Oxidation Mechanism of Sulphur in Digestion of Bayer Process

The high-sulphur and high-carbon bauxite in China is primarily of the diaspore type, where the sulphur-bearing minerals are mainly pyrite, and the organic carbon consists largely of humic acid and similar compounds. Xiaobin Li, Wankun Chen, Guozhi Lv, et al. [3, 5] conducted a systematic study on the reaction behaviour of pyrite and the behaviour of sulphur in Bayer liquor. Under high-temperature digestion conditions at 260–270 °C, pyrite reacts with caustic soda in sodium aluminate solution, with sulphur primarily entering the solution as S<sup>2-</sup>, while the remainder exists as S<sub>2</sub><sup>2-</sup>, S<sub>2</sub>O<sub>3</sub><sup>2-</sup>, SO<sub>3</sub><sup>2-</sup>, and SO<sub>4</sub><sup>2-</sup>. Most of the organic carbon in the bauxite enters the sodium aluminate solution in the form of high-molecular-chain compounds such as humic acid, which can easily accumulate to high concentrations, darkening the colour of the sodium aluminate solution, sometimes even turning it a dark soy-sauce black. The main reaction between pyrite and sodium hydroxide during the digestion of high-sulphur bauxite is as follows:

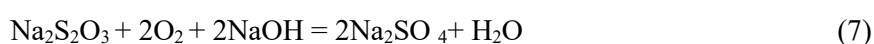
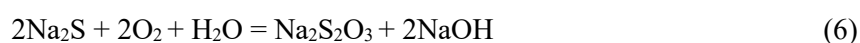


With the increase in temperature, the stability of sodium thiosulphate decreases, leading to its decomposition during the high-temperature digestion process.



Under conditions where no oxygen or other oxidizing agents are added, reactions (1)–(3) dominate, while further oxidation to higher-valence sulphur (SO<sub>4</sub><sup>2-</sup>) becomes considerably more challenging. Reactions (4) and (5) represent the secondary oxidation of intermediate sulphur species generated during the reaction between pyrite and caustic soda, requiring higher temperatures and prolonged durations. Notably, reaction (5) is even more unfavourable than (4).

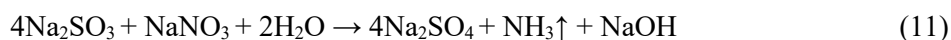
Wenmi Chen et al. [6, 10], conducted laboratory studies on the oxidation of low-valence sulphur and organics in solutions under different temperature conditions, employing atmospheric and high-pressure wet processes with the addition of oxidants such as oxygen, hydrogen peroxide, and metal oxides. All methods achieved favourable oxidation results. In reactions involving oxygen, the primary oxidation process proceeds as follows:



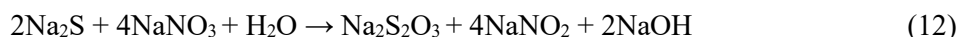


Under the condition of oxygen addition, at atmospheric pressure (95–105 °C), introducing oxygen into the evaporated spent liquor or diluted slurry for several hours can achieve complete oxidation. However, the oxidation intensity for  $\text{S}_2\text{O}_3^{2-}$  and  $\text{SO}_3^{2-}$  is insufficient, preventing sulphur-containing impurities from entering the bauxite residue in the form of double salts. This leads to the accumulation of sulphates, which adversely affect subsequent precipitation and evaporation processes. Additionally, prolonged and large-scale oxygen injection under atmospheric pressure results in excessive oxygen consumption in industrial applications, making it difficult to meet the required residence time. Under the high-pressure digestion conditions of diasporite at 260–270 °C, the introduction of oxygen significantly reduces both the required oxygen dosage and oxidation time, enabling complete oxidation of  $\text{S}^{2-}$  within a short period. Moreover, the oxidation intensity for  $\text{S}_2\text{O}_3^{2-}$  and  $\text{SO}_3^{2-}$  is greatly enhanced. Under high-temperature digestion,  $\text{SO}_4^{2-}$  enters the bauxite residue in the form of double salts. However, achieving the safety and technical requirements for oxygen injection into high-temperature and high-pressure industrial units remains challenging in large-scale production.

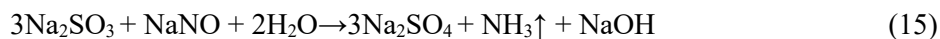
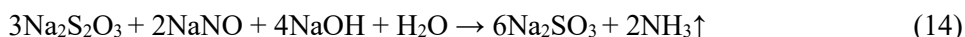
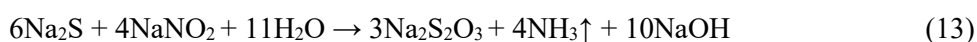
To find a simple and effective method for oxidizing and removing low-valence sulphur in high-temperature Bayer process alumina production, thereby addressing the sulphur-related issues, experimental studies were conducted using sodium nitrate as an additive. The main oxidation reactions involved are as follows:



When the reduction of sodium nitrate is incomplete, the following reaction occurs:



$\text{NO}_2^-$  can also oxidize low-valence sulphur, with the reaction equations as follows:



The composition of sulphur-containing compounds in the solution depends on oxidation conditions, where the concentrations of  $\text{S}^{2-}$ ,  $\text{SO}_3^{2-}$ ,  $\text{S}_2\text{O}_3^{2-}$ , and  $\text{SO}_4^{2-}$  are influenced by factors such as sodium nitrate dosage, reaction temperature, and reaction time. The final oxidation product of  $\text{S}^{2-}$  is  $\text{SO}_4^{2-}$ . During high-temperature digestion, sodium sulphate forms a double salt with sodium silicate residue and is subsequently discharged from the alumina production process via bauxite residue in the solid phase.

### 3. Results and Discussion

#### 3.1 Oxidation Test of Purified Sodium Aluminate Solution

To investigate the oxidation effects of sodium nitrate at different temperatures, purified sodium aluminate green liquor ( $N_k = 250$  g/L,  $S^{2-} = 1.43$  g/L) prepared in the laboratory was treated with added low-valence sulphur compounds and 5 g/L sodium nitrate. The solutions were maintained at 100 °C, 160 °C, 210 °C, and 260 °C for 1 hour, respectively, followed by filtration and analysis of the filtrate composition. The experimental results are shown in Table 1. The concentrations of  $S^{2-}$ ,  $S_2O_3^{2-}$ , and  $SO_3^{2-}$  in the sodium aluminate solution were determined by iodine-sodium thiosulphate titration, while  $SO_4^{2-}$  concentration was measured by gravimetric analysis. The  $NO_3^-$  concentration in the sodium aluminate solution was analysed using ion chromatography.

**Table 1. Effect of temperature on the oxidation efficiency of  $NaNO_3$ , g/L.**

Temperature	$S^{2-}$	$S_2O_3^{2-}$	$SO_3^{2-}$	$SO_4^{2-}$	$NO_3^-$
Green Liquor	1.43	0.82	0.49	0.00	3.65
100 °C	0.75	1.68	0.92	0.04	2.92
160 °C	0.35	2.32	0.91	0.15	2.89
210 °C	0.00	2.46	1.52	0.25	1.82
260 °C	0.00	0.78	2.77	1.60	0.51

From Table 1, it can be observed that the oxidizing capacity of sodium nitrate gradually increases with rising temperature. After maintaining at 100 °C for 1 hour, the  $S^{2-}$  content in the solution decreased from 1.43 to 0.75 g/L. When the temperature was increased to 160 °C, the  $S^{2-}$  content further dropped to 0.35 g/L. After the green liquor was held at 210 °C for 1 hour,  $S^{2-}$  was almost completely oxidized, but a relatively high residual  $NO_3^-$  concentration of 1.82 g/L remained unreacted. Upon reaching 260 °C, the residual  $NO_3^-$  in the solution decreased to 0.51 g/L. As the reaction temperature increases, sodium nitrate can further oxidize  $S^{2-}$  from  $S_2O_3^{2-}$ ,  $SO_3^{2-}$ , and  $SO_4^{2-}$ . This indicates that higher temperatures provide more favourable kinetic conditions for the oxidation of  $S^{2-}$  by sodium nitrate. Therefore, a higher reaction temperature of 260 °C should be selected to achieve better oxidation results.

#### 3.2 Oxidation Test of Industrial Circulating Spent Liquor

To investigate the oxidation effect of sodium nitrate on the circulating spent liquor with complex composition in industrial production, the spent liquor from the refinery (composition shown in Table 2) was treated with a certain amount of sodium nitrate, maintained at 260 °C for a specific duration, and then filtered. The filtrate composition was analysed, with results presented in Tables 3 and 4.

**Table 2. Composition of circulating spent liquor used in test, g/L.**

$Na_2O_K$	$Na_2O_T$	$Al_2O_3$	$S^{2-}$	$S_2O_3^{2-}$	$SO_3^{2-}$	$SO_4^{2-}$
210.15	238.47	114.19	1.30	2.69	1.19	5.87

**Table 3. Effect of  $NaNO_3$  addition amount on sulphur content in circulating spent liquor after 1-hour reaction, g/L.**

No.	Dosage of $NaNO_3$	Calculated as $NO_3^-$	$S^{2-}$	$S_2O_3^{2-}$	$SO_3^{2-}$	$SO_4^{2-}$	$NO_3^-$
1#	3	2.19	0.16	1.88	3.68	6.83	0.86
2#	5	3.65	0.00	1.98	3.80	7.36	1.21

Note: The dosage of sodium nitrate is given in concentration, g/L, the same as below.

**Table 4. Effect of NaNO<sub>3</sub> addition amount on sulphur content in circulating spent liquor after 2-hour reaction, g/L.**

No.	Dosage of NaNO <sub>3</sub>	Calculated as NO <sub>3</sub> <sup>-</sup>	S <sup>2-</sup>	S <sub>2</sub> O <sub>3</sub> <sup>2-</sup>	SO <sub>3</sub> <sup>2-</sup>	SO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>
3#	0.5	0.36	0.69	1.74	5.06	5.76	0.00
4#	1	0.73	0.56	2.21	4.81	5.81	0.00
5#	2	1.46	0.50	2.35	4.86	6.07	0.26
6#	3	2.19	0.03	1.97	4.33	7.34	0.48

From Tables 3 and 4, it can be observed that when the sodium nitrate dosage reaches 3 g/L, the S<sup>2-</sup> in the spent liquor can be essentially removed. Prolonging the reaction time favours the further conversion of lower-valence sulphur to higher-valence sulphur. Meanwhile, as shown in Tables 2 and 3, compared with the prepared pure sodium aluminate solution, under the conditions of a digestion temperature of 260 °C, digestion time of 1 hour, and sodium nitrate dosage of 5 g/L, the industrial complex sodium aluminate solution results in a higher NO<sub>3</sub><sup>-</sup> concentration after oxidation, with a significantly lower reaction rate of sodium nitrate. Additionally, if high-temperature oxidation is to be performed on the spent liquor in actual production, a separate high-temperature and high-pressure system must be constructed. Moreover, the impact of lower-valence sulphur precipitation in the obtained pregnant liquor on the product during digestion has not been pre-emptively controlled. Therefore, the method of separately oxidizing industrial spent liquor is not feasible in production.

### 3.3 Oxidation Test of High-Sulphur Bauxite Digestion in Industrial Production

#### 3.3.1 Test Materials

The plant site utilized high-sulphur bauxite for production, with its chemical composition shown in Table 5. The sulphur content reached 1.50 %, and the XRD pattern of the bauxite (Figure 1) indicated that its main phases included diaspore, muscovite, anatase, pyrite, etc. The test spent liquor composition is presented in Table 6.

**Table 5. Chemical composition of high-sulphur bauxite, %.**

Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	S
74.93	4.31	3.09	5.71	1.50

**Table 6. Composition of circulating spent liquor used in test, g/L.**

Na <sub>2</sub> O <sub>K</sub>	Na <sub>2</sub> O <sub>T</sub>	Al <sub>2</sub> O <sub>3</sub>	S <sup>2-</sup>	S <sub>2</sub> O <sub>3</sub> <sup>2-</sup>	SO <sub>3</sub> <sup>2-</sup>	SO <sub>4</sub> <sup>2-</sup>
226.08	241.52	131.96	0.27	0.63	0.86	0.72

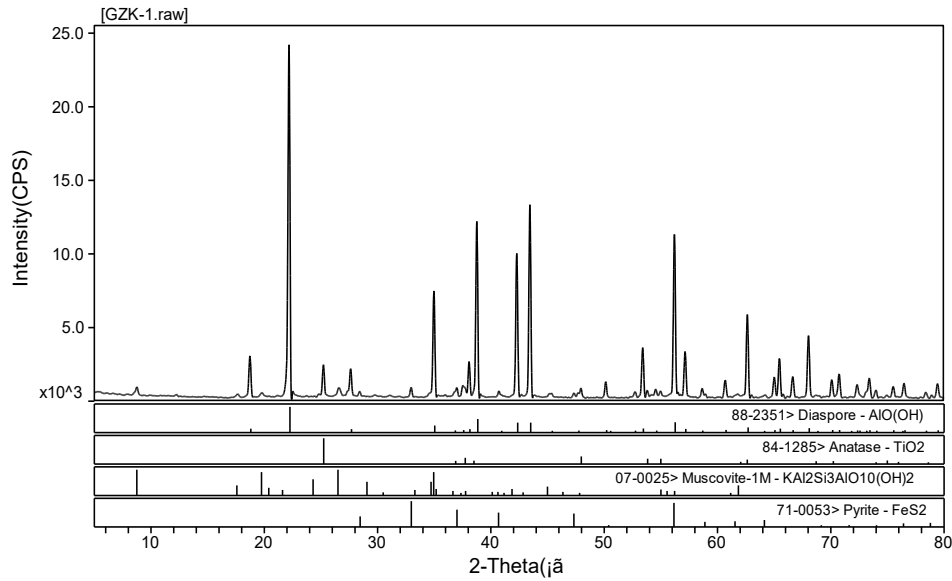


Figure 1. XRD Pattern of High-sulphur Bauxite.

### 3.3.2 Test Method

A mixture of 10 g of high-sulphur bauxite, 1 g of calcium oxide, 50 mL of spent liquor, and a certain amount of  $\text{NaNO}_3$  was added to an autoclave. The digestion was conducted at 260 °C for 1.5 h. After solid-liquid separation, the filtrate was analysed for the contents of  $\text{S}^{2-}$ ,  $\text{S}_2\text{O}_3^{2-}$ ,  $\text{SO}_3^{2-}$ ,  $\text{SO}_4^{2-}$ , and  $\text{NO}_3^-$ .

### 3.3.3 Test Results and Analysis

Under industrial production conditions, with a digestion temperature of 260 °C and a digestion time of 60 minutes, tests were conducted to investigate the effect of different sodium nitrate ( $\text{NaNO}_3$ ) additions on oxidation efficiency. The results are presented in Table 7.

Table 7 Effect of  $\text{NaNO}_3$  addition amount on sulphur content in sodium aluminate solution, g/L.

No.	Dosage of $\text{NaNO}_3$	Calculated as $\text{NO}_3^-$	$\text{S}^{2-}$	$\text{S}_2\text{O}_3^{2-}$	$\text{SO}_3^{2-}$	$\text{SO}_4^{2-}$	$\text{NO}_3^-$	$\text{NO}_3^-$ Consumption
7#	0	0.00	1.36	0.34	1.46	0.78	0.00	0.00
8#	1	0.73	0.79	1.34	0.50	0.84	0.21	0.52
9#	3	2.19	0.66	1.96	1.86	1.08	0.08	2.11
10#	5	3.65	0.12	1.74	2.70	1.24	0.44	3.20
11#	7	5.11	0.12	2.09	2.14	1.42	0.92	4.18

With the increase in sodium nitrate dosage, the concentration of  $\text{S}^{2-}$  in the solution gradually decreases. When 5 g/L of sodium nitrate is added,  $\text{S}^{2-}$  drops to 0.12 g/L, meeting production requirements. The concentration of  $\text{S}_2\text{O}_3^{2-}$  first increases and then decreases, as it is further oxidized to  $\text{SO}_3^{2-}$ . The variation trend of  $\text{SO}_3^{2-}$  concentration with sodium nitrate dosage is similar to that of  $\text{S}_2\text{O}_3^{2-}$ . Meanwhile, the concentration of  $\text{SO}_4^{2-}$  steadily increases. By comparing the  $\text{NO}_3^-$  concentration in the digestion solution, it can be observed that when the sodium nitrate dosage is less than 7 g/L, over 80 % of the added  $\text{NaNO}_3$  is reduced, indicating that most  $\text{NO}_3^-$  participates in the reaction with high utilization efficiency.

### 3.4 Effect of Industrial Continuous Operation Test

Based on laboratory tests, an industrial application test was conducted at a 1-Mtpa refinery. The digestion unit operated at 260–265 °C with a residence time of 50–60 minutes. The sodium nitrate dosage was adjusted according to the S<sup>2-</sup> content in the pregnant liquor to maintain the S<sup>2-</sup> concentration no higher than 0.15 g/L. The 12-month continuous operation monitoring results are as follows.

#### 3.4.1 Sulphur Content in Bauxite Fed into the Grinding Mill Process

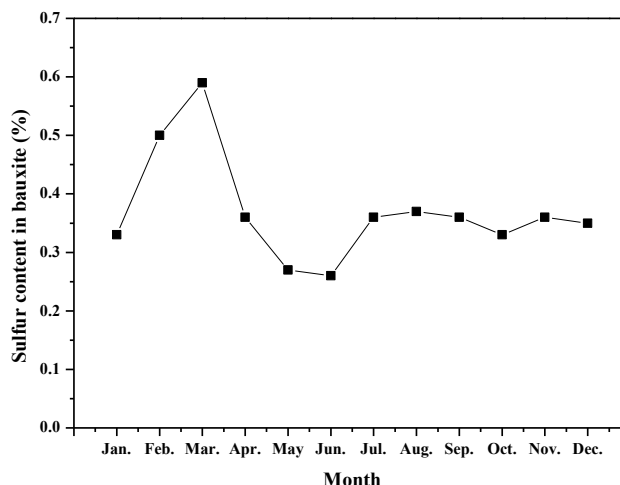


Figure 2. Monthly average curve of sulphur content in bauxite fed into the grinding process.

The monthly average sulphur content in the production-grade bauxite ranges from a minimum of 0.33 % to a maximum of 0.59 %, with an annual average of 0.37 %.

#### 3.4.2 Status of S<sup>2-</sup> in Pregnant Liquor

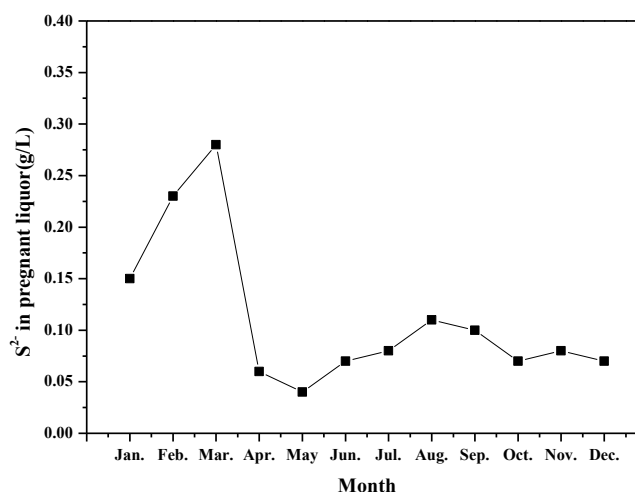


Figure 3. Monthly average curve of S<sup>2-</sup> in pregnant liquor.

In production, the monthly average sulphur content (S<sup>2-</sup>) in the pregnant liquor ranged from a minimum of 0.05 g/L to a maximum of 0.28 g/L, with an annual average of 0.11 g/L. When the

bauxite sulphur content exceeded 0.5 %, the level surpassed the empirically established safety threshold of 0.15 g/L for production.

### 3.4.3 Fe<sub>2</sub>O<sub>3</sub> Content in Product

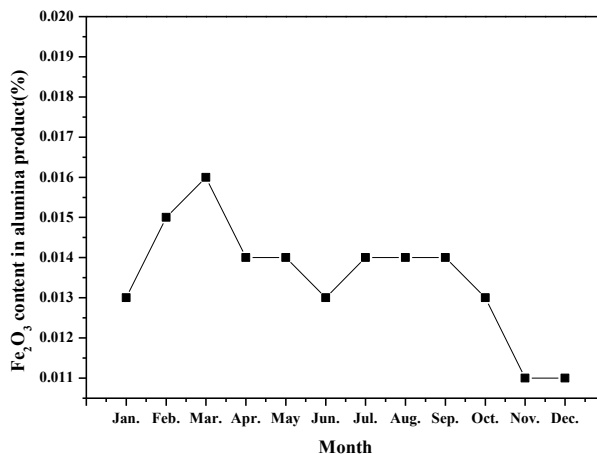


Figure 4. Monthly average curve of Fe<sub>2</sub>O<sub>3</sub> content in product.

In production, the monthly average Fe<sub>2</sub>O<sub>3</sub> content in alumina products ranges from a minimum of 0.011 % to a maximum of 0.016 %, with an annual average of 0.0135 %. When the sulphur content in the bauxite is below 0.4 %, the addition of sodium nitrate can eliminate the impact of low-valent sulphur on product quality. However, when the bauxite's sulphur content reaches 0.5 % or higher, although the iron content in the product remains within the first-grade standard, it shows an upward trend. In this case, simply increasing the dosage of sodium nitrate becomes difficult to control and leads to a significant rise in consumption, resulting in excessively high costs.

### 3.4.4 Changes of Organics in Digestion Solution

This study investigated the oxidation of humic acid-like macromolecular organics during high-temperature digestion with sodium nitrate addition through industrial tests. The test group for digestion received sodium nitrate addition while the control group did not. The comparative absorbance curves over 40 days are shown in Figure 5.

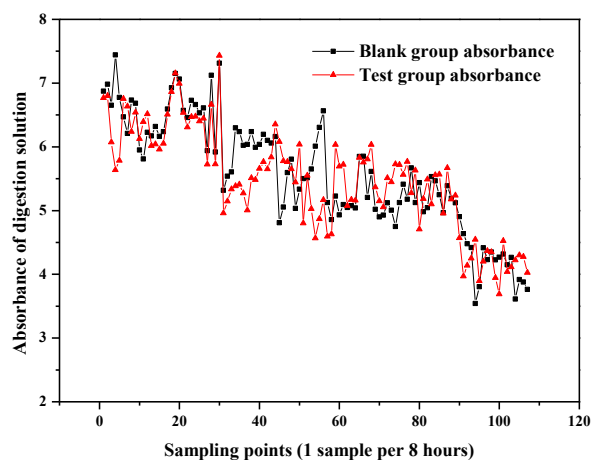


Figure 5. Comparison curve of absorbance in digestion solution.

In the digestion solution, the weak oxidation effect on organics is difficult to distinguish by analysing the organic content in the solution, but it can be characterized by detecting changes in

absorbance. As shown in the figure above, under industrial testing conditions without excess sodium nitrate addition, the absorbance trend curves of the test group and the blank group largely overlap, indicating a minimal oxidation effect. The statistical average shows only a 1.3 % reduction, demonstrating that organics are hardly oxidized by sodium nitrate under actual industrial production conditions. To enhance the oxidation of organics, a significant excess of sodium nitrate would be required. However, excessive  $\text{NO}_3^-$  would enter the bauxite residue in the form of double salts, increasing losses and raising costs. Therefore, alternative methods for organic removal must be explored.

#### 4. Conclusions and Recommendations

The use of high-sulphur bauxite for alumina production can be effectively and simply controlled by adding sodium nitrate during the bauxite blending process to mitigate the adverse effects of sulphur. During the oxidation process,  $\text{S}^{2-}$  in the slurry is first oxidized to  $\text{S}_2\text{O}_3^{2-}$ , then further oxidized to  $\text{SO}_3^{2-}$  and  $\text{SO}_4^{2-}$ . Increasing the dosage of sodium nitrate, raising the reaction temperature, and prolonging the reaction time help enhance the oxidation degree of sulphur and improve the oxidation effects. The oxidation of  $\text{S}^{2-}$  in the solution reduces the formation of sodium hydroxythioferrate, thereby decreasing the dissolved iron content in the solution. Additionally, NaOH is generated as a byproduct in the oxidation reaction involving  $\text{NaNO}_3$ , which can partially compensate for soda consumption. According to industrial test results, when the sulphur content in bauxite does not exceed 0.5 %, the adverse effects of sulphur on alumina production can be effectively controlled by adding sodium nitrate to oxidize low-valence sulphur. This method proves to be a simple and effective solution for alumina refineries that process bauxite containing minor high-sulphur bauxite or intentionally blend small amounts of high-sulphur bauxite to compensate for raw material shortages. With this technology, the  $\text{S}^{2-}$  concentration in pregnant liquor can be maintained within safe limits, the iron oxide content in final products meets quality standards, and the additional cost remains acceptable. This technique is recommended for promotion and application by domestic high-temperature Bayer refineries utilizing high-sulphur bauxite.

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