

Article

Oxidation Study and Mechanism Analysis of Desulfurization Ash in Dense-Phase Tower

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Abstract: Dense-phase-tower desulfurization technology is an emerging semi-dry flue-gas desulfurization ash process, i.e., the flue gas is allowed to enter the desulfurization tower from the bottom up and, at the same time, is sprayed with a desulfurizing agent that undergoes an acid–base reaction with the flue gas in the ascent process. The calcium sulfite and calcium sulfate produced by the reaction and the part of the desulfurization agent that is not involved in the reaction will enter the subsequent dust removal system, and what is retained is the by-product desulfurization ash. This desulfurization ash contains a large amount of calcium sulfite, which leads to its unstable nature; it is easily oxidized and expands in volume, and, if used in the field of building materials, it will lead to cracking and other problems, so it is difficult to effectively use it. In order to solve this problem, XRF, XRD, and iodometric and other analytical methods were used to determine the specific composition of desulfurization ash, and the muffle furnace and vertical tube furnace were used to study the thermal oxidative modification of calcium sulfite in desulfurization ash, to investigate the effects of the oxygen content, reaction temperature, medium flow rate, and chloride content on the oxidation of calcium sulfite, and to analyze the thermodynamics in the high-temperature oxidation reaction. The results showed that the oxidation rate of calcium sulfite increased with higher reaction temperatures. Increased oxygen content promoted the oxidation rate, particularly at low oxygen levels. The oxidation rate of calcium sulfite correlated positively with the medium flow rate until a rate of 75 mL·min^{−1} was reached. At a reaction temperature of 420 °C and a gas flow rate of 85 mL·min^{−1}, the oxidation conversion efficiency exceeded 89%. Chloride content significantly reduced the oxidation rate of calcium sulfite, although this inhibition weakened at temperatures above 500 °C. Kinetic analysis suggested that the oxidation reaction of calcium sulfite predominantly occurred below 500 °C. These findings have both theoretical and practical implications for the thermal oxidation treatment and disposal of desulfurization ash.

Keywords: dense-phase tower; desulfurization ash; anaerobic fermentation; calcium sulfite; thermal oxidation



Citation: Lu, G.; Li, H.; Ma, H.; Leng, T. Oxidation Study and Mechanism Analysis of Desulfurization Ash in Dense-Phase Tower. *Processes* **2024**, *12*, 1008. <https://doi.org/10.3390/pr12051008>

Academic Editor: Antoni Sanchez

Received: 15 April 2024

Revised: 11 May 2024

Accepted: 12 May 2024

Published: 15 May 2024



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1. Introduction

Dense-phase-tower semi-dry desulfurization technology is a type of semi-dry flue-gas desulfurization [1,2]. This technology uses a powdered desulfurization agent to provide moisture. Sulfurous flue gas is sprayed into the bottom of the desulfurization tower. As the flue gas rises, the sulfur dioxide is absorbed and, at the same time, there is evaporation of water. Finally, the desulfurized ash was obtained in the dust removal system and then recycled to the treatment system and reused in the desulfurization tower. By continuously monitoring the effective CaO content in the circulating desulfurizing agent, the ash discharge time and ash discharge amount are determined. The reacted desulfurization ash is discharged as a desulfurization by-product from the discharge device at the bottom of the

dust collector to the by-product bin. This process has extensive applications in the iron and steel industries due to the demand for ultra-low emission standards. This technology is widely used for treating flue gases in metallurgical sintering and pelletizing, and in hot blast furnaces, rolling mill heating furnaces, and other metallurgical furnaces and kilns. It not only ensures ultra-low emissions but also has the advantages of stable operation, low cost, and no wastewater discharge [3]. But the technology does produce a significant amount of by-product desulfurization ash, which presents challenges for direct utilization. Consequently, a considerable accumulation of desulfurization ash will consume substantial land resources and pose environmental risks. The proper treatment and disposal of desulfurization ash have become a major hindrance to the widespread adoption of semi-dry desulfurization technology.

The semi-dry desulfurization ash mainly consists of three components: CaSO_3 , CaSO_4 , and Ca(OH)_2 [4]. Additionally, it contains relatively significant amounts of Al_2O_3 and SiO_2 , along with smaller quantities of MgO , Fe_2O_3 , Na_2O , and other elements. Some heavy metals, including Pb and Cr, may also be present [5]. According to Kang et al. [5], calcium sulfite and calcium sulfate are found to be important components of desulfurization ash. Calcium sulfite is found in twice the quantity of calcium sulfate, making up around 20–40% of the ash. The remaining components include less than 15% calcium carbonate and 20% to 50% calcium hydroxide. Thermogravimetric analysis of the desulfurized ash [6–8] revealed that a significant oxidation of calcium sulfite occurs at temperatures ranging from 400 °C to 560 °C, leading to a substantial exothermic reaction in this temperature range. In an inert atmosphere, calcium sulfite only decomposes at a higher temperature of 800 °C, indicating its high thermal stability [9].

Several studies [10–12] have investigated the use of desulfurized ash as a cement retarder, subject to certain proportion limits. Fu et al. [13] discovered that the cement retarding effect of desulfurization ash primarily comes from calcium sulfate, while calcium sulfite has minimal impact [14,15]. However, the oxidation of calcium sulfite can lead to the formation of crystalline calcium sulfate, resulting in micro-expansion in building materials and potentially affecting their safety performance. Consequently, it becomes essential to restrict the amount of desulfurization ash added [7,16,17]. To address this issue, it is crucial to oxidize calcium sulfite, which is inherently unstable, into calcium sulfate. This allows for secure and steady use in construction materials. This study employed desulfurization ash from the semi-dry desulfurization process of dense-phase towers as experimental material to investigate the factors influencing its oxidation conversion rate under high-temperature conditions. It also explored the optimal process conditions for the oxidation treatment of desulfurization ash in dense-phase towers, taking into account economic considerations, and it provides a foundation for the resourceful utilization of desulfurization ash in subsequent applications.

2. Materials and Methods

2.1. Materials and Instruments

The desulfurization ash used in this study was obtained from the semi-dry desulfurization process of the dense-phase tower at a steel company. The ash was a greyish-white powder with a strong alkaline nature. The flue-gas desulfurization (FGD) ash mainly comprised CaSO_3 , $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, CaCO_3 , Ca(OH)_2 , and CaCl_2 , with mass fractions of $22.53 \pm 10\%$, $40.00 \pm 10\%$, $15.00 \pm 5\%$, $10.00 \pm 5\%$, and $10.00 \pm 5\%$, respectively. After XRF testing, the results of the analysis of its main elements are shown in Table 1. Through testing, the particle size distribution of desulfurization ash was determined, as shown in Table 2.

Table 1. Results of desulfurization ash element analysis %.

Element	CaO	SO ₃	CO ₂	Cl	K ₂ O	MgO	N	Fe ₂ O ₃	SiO ₂	Na ₂ O	F	Al ₂ O ₃
Desulfurization ash	51.9	27.75	15.54	3.7	1.79	1.07	0.83	0.69	0.54	0.52	0.33	0.39

Table 2. The particle size distribution of desulfurization ash.

Particle Size Range (μm)	<3.02	<5.03	<7.07	<11.79	<27.64
Proportion (%)	27.40	49.82	66.14	86.52	99.77

The measurements were performed according to the Methods of Chemical Analysis of Gypsum (GB/T 5484-2012) [18], indicating that it was a high-calcium and high-sulfur desulfurization ash. The desulfurization ash was first pre-treated by drying at 105 °C for 4 h. The agents used in this study are shown in Table 3.

Table 3. Experimental agents used in this study.

Experimental Methods	Agents	Specifications	Experimental Methods	Agents	Specifications
The iodometric method	Iodine	AR	Barium sulphate method	Potassium iodate	AR
	Potassium iodide	AR		Hydrochloric acid	AR
	Sodium hyposulfide	AR		Silver nitrate	AR
	Sodium carbonate	AR		Ashless filter paper	-
	Soluble starch	AR		Barium chloride	AR
	Sodium acetate	AR	Sucrose method High temperature oxidation test	Nitric acid	AR
	Acetic acid	AR		Sucrose	AR
	Phosphoric acid	AR		Calcium chloride	AR

Since the main experimental reagents were used in the quantitative determination of the composition of desulfurization ash, analytically pure reagents were generally used in order to reduce the error of composition determination. The experimental equipment mainly included a muffle furnace, vertical tube furnace, drying furnace, electronic balance, X-ray diffractometer, laser particle size analyzer, scanning electron microscope, thermogravimetric analyzer, and air pump.

2.2. Methodology

The high temperature oxidation test is divided into a dynamic oxidation test and a static oxidation test.

The dynamic oxidation experimental equipment included a gas distribution system and a tube furnace control system, in which the gas distribution system included oxygen, nitrogen, and a gas flowmeter to control the composition and rate of the gas entering the vertical tube furnace. In each group of experiments, 5 g of dried desulfurization ash was placed in a vertical tube furnace. The heating rate of the furnace was set to 10 °C/min, and the reaction conditions were determined by varying the heating temperature, the heating time, and the rates of the nitrogen and oxygen passage. The nitrogen–oxygen ratio, which is the ratio of the rates of nitrogen and oxygen fluxes, was set at 3:2, 3.5:1.5, 4:1, and 4.5:0.5, while maintaining a gas flow rate of 250 mL·min^{−1}. The roasting was conducted at two different temperatures, 400 °C and 500 °C, with a reaction time of 30 min each. The purpose was to investigate the impact of oxygen content on the oxidation of calcium sulfite. Furthermore, the study aimed to examine the influence of medium flow rate on the oxidation of calcium sulfite. For this purpose, the gas flow rates were varied at 25, 50, 75, 100, 125, and 250 mL·min^{−1}, while keeping the reaction temperature constant at 450 °C and the reaction time at 30 min. A temperature of 450 °C was chosen because above 450 °C, the rate of oxidation of calcium sulfite decreases significantly with increasing temperature.

The static oxidation experiment employed a muffle furnace as the experimental device. To explore the impact of temperature on the oxidation of calcium sulfite, seven groups of 5 g of desulfurization ash were added to different muffle furnaces, and the reaction temperatures were set at 350 °C, 380 °C, 400 °C, 420 °C, 450 °C, 500 °C, and 550 °C, with a reaction time of 30 min for each temperature. To explore the impact of chloride content on the oxidation of calcium sulfite, different proportions of CaCl₂ (0%, 4%, 10%, and 16%) were added to the desulfurization ash. The mixture was homogenized and then placed

into the muffle furnace. The subsequent reaction temperatures were set at 350 °C, 400 °C, 450 °C, 500 °C, and 550 °C with a reaction time of 30 min for each temperature. In total, there were 20 sets of experiments. Throughout the experiment, the calcium sulfite content before and after the reaction was carefully measured and calculated. This analysis aimed to investigate the conversion mechanism of the corresponding calcium sulfite under the specified conditions.

Chemical composition tests include the iodometric method, barium sulphate method, and sucrose method.

The iodometric method allows the sulfite in the desulfurization ash to undergo a redox reaction with a quantitative amount of iodine in an acidic solution, and the excess iodine is titrated with a standard solution of sodium thiosulphate to determine the amount of calcium sulfate in the desulfurization ash.

The barium sulphate method uses concentrated hydrochloric acid to dissolve the sulphate ions from calcium sulfate to react with barium chloride to generate barium sulphate with very low solubility, and the content of calcium sulfate in desulfurization ash is calculated by measuring the weight of the generated barium sulphate.

The sucrose method uses sucrose and the calcium oxide in the desulfurization ash in water to produce water-soluble calcium sucrose. Then, hydrochloric acid is used to neutralize the reaction, and, through the titration, the volume of hydrochloric acid is used to calculate the content of active calcium oxide in the desulfurization ash.

The desulfurization ash was also tested by XRF, thermogravimetric analysis, and particle size analysis.

The technical flowchart of this study is shown in Figure 1.

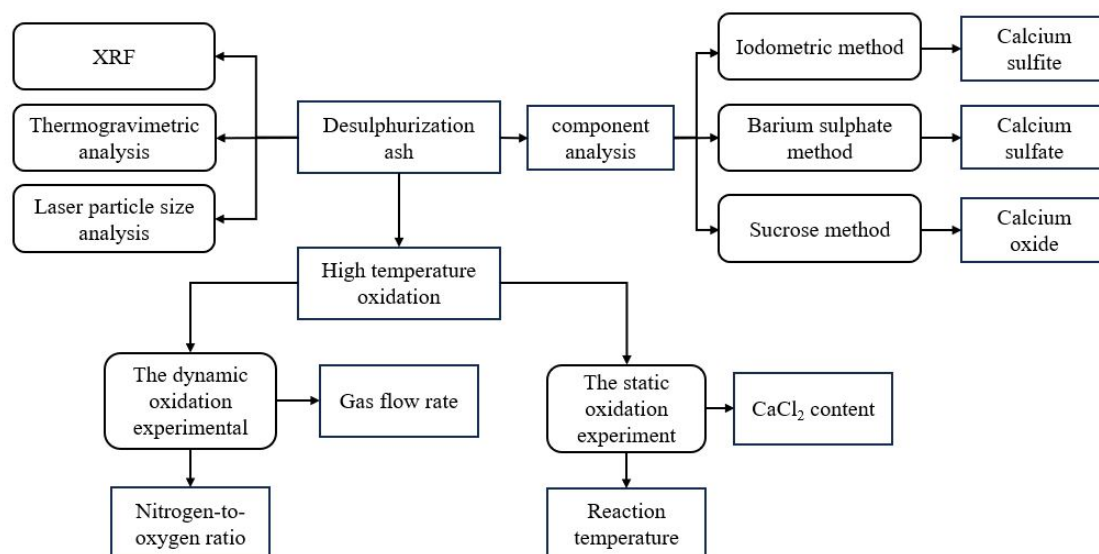


Figure 1. The technical flowchart of this study.

3. Results and Discussion

3.1. Effect of Nitrogen-to-Oxygen Ratio on the Oxidation of CaSO_3

The nitrogen–oxygen ratio is the ratio of nitrogen to oxygen, by volume, in the gas fed to the vertical tube furnace. As can be seen from Figure 2, from the overall law of the curve, it is observed that under different atmospheric nitrogen–oxygen ratios, with the increase in nitrogen–oxygen ratio, the conversion rate of calcium sulfite decreased, and this decrease and the ratio of nitrogen–oxygen ratios basically show a linear relationship.

As shown in Figure 2a, at a temperature of 500 °C, the nitrogen–oxygen ratio was 4.5:0.5, and the conversion rate of the calcium sulfite system after 30 min of reaction was greater than 85%. When the nitrogen–oxygen ratio was reduced to 3.5:1.5, the conversion rate of calcium sulfite increased to approximately 93%. Thus, under the same reaction time and temperature, an increase in oxygen concentration resulted in an 8% increase in

conversion rate. At a temperature of 400 °C, the increase in oxygen concentration had a greater influence on the conversion rate, resulting in a more pronounced elevation in the curve. As shown in Figure 2b, the oxidation rate of calcium sulfite for 30 min at 400 °C was approximately 16% when the nitrogen–oxygen ratio was 4.5:0.5. Upon reduction of the nitrogen–oxygen ratio to 3.5:1.5, the calcium sulfite conversion rate increased to approximately 85%. The conversion rate of calcium sulfite oxidation of calcium sulfate was increased by approximately 69%. However, as the oxygen concentration continued to increase, that is, the nitrogen-to-oxygen ratio was less than 3.5:1.5, the conversion rate decreased as the oxygen concentration increased.

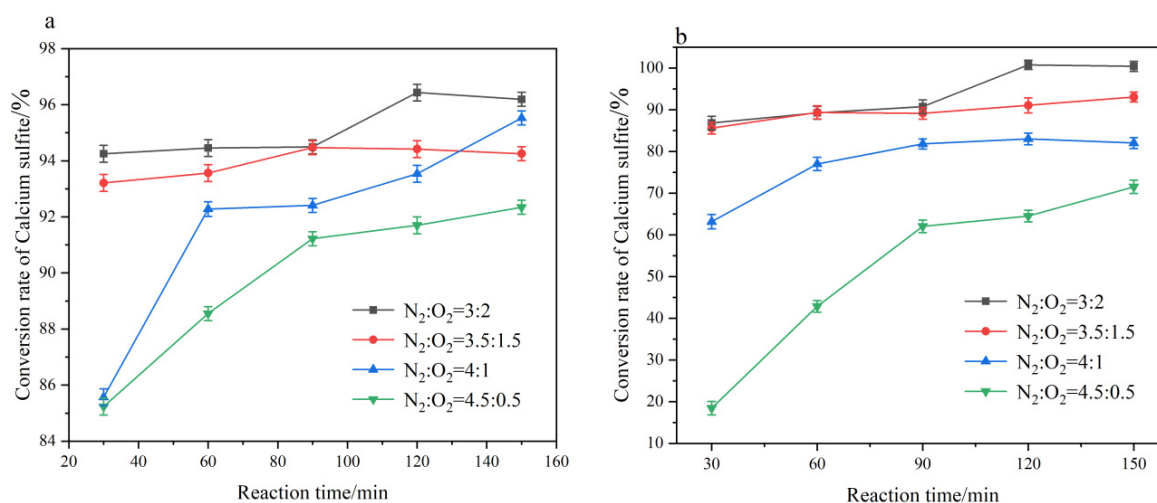


Figure 2. Change in conversion rate at 500 °C (a) and 400 °C (b) with different nitrogen–oxygen ratios.

A comparison of the corresponding points on the left and right graphs in Figure 2 reveals that the conversion curves at 500 °C are above the curve at 400 °C. This demonstrates that an increase in temperature can lead to an increase in conversion rate in real time. When the nitrogen–oxygen ratio was less than 3.5:1.5, i.e., when the oxygen concentration was high, the increase of 400 °C to 500 °C led to an increase of approximately 8% in the conversion rate. Conversely, when the nitrogen–oxygen ratio was greater than 4:1, i.e., when the oxygen concentration was low, the effect of temperature on the conversion rate was highly significant. At a nitrogen–oxygen ratio of 4.5:0.5, the lowest conversion rate at 500 °C was 85%, while the lowest conversion rate at 400 °C was 17%, and the highest was 71%. These experiments amply demonstrated that at lower oxygen concentrations, an increase in temperature was required to compensate for the lack of an oxidizing atmosphere for the oxidation of calcium sulfite.

3.2. Effect of Temperature on the Oxidation of CaSO₃

The effect of reaction temperature on CaSO₃ oxidation is illustrated in Figure 3. It is evident from the graph that an increase in temperature led to a significant rise in the conversion rate, suggesting that the CaSO₃ oxidation reaction adhered to the Arrhenius equation, where higher temperatures accelerate the reaction rate. Below 350 °C, CaSO₃ exhibited excellent thermal stability and did not undergo any reaction, maintaining its chemical composition unchanged. As the temperature rose to 380–400 °C, the conversion rate increased rapidly from 0 to 80%, indicating an evident enhancement within this temperature range. At 400–500 °C, the curve exhibits a significantly low slope, indicating a noticeable slowdown in the conversion rate. This observation implies that the conversion reaction had progressed to its later stage, and the active components had been largely consumed, leading to exhaustion. Consequently, CaSO₃ is essentially oxidized at temperatures of 550 °C and above.

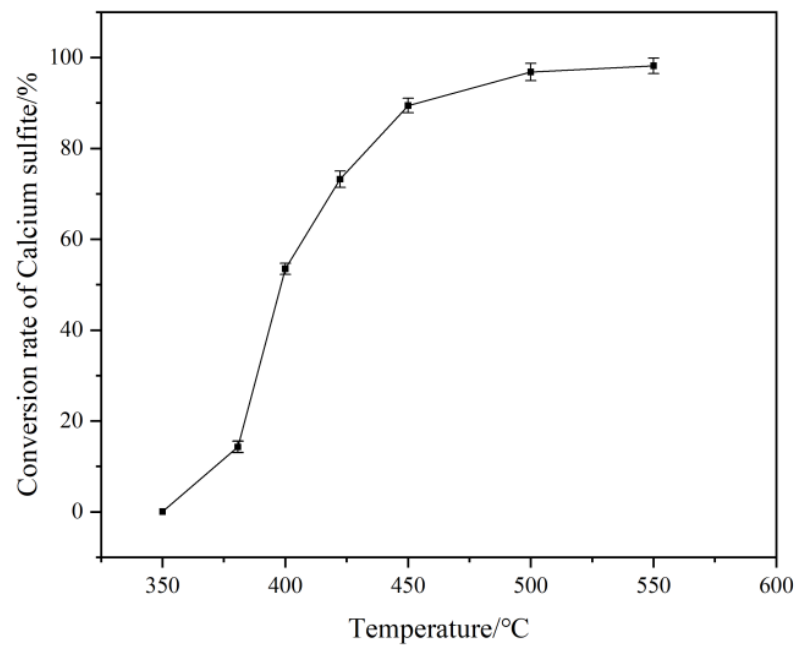


Figure 3. Effect of temperature on the oxidation of CaSO_3 .

3.3. Effect of Gas Flow Rate on the Oxidation of CaSO_3

As depicted in Figure 4, the relationship curve between the medium flow rate and CaSO_3 conversion rate demonstrates a pattern of first increasing and then decreasing. From the graph, it is evident that when the gas flow rate was below $75 \text{ mL} \cdot \text{min}^{-1}$, the CaSO_3 oxidation rate increased as the gas flow rate rose, and this increase could range from 70% to 90%, representing a 20% increment. This could be attributed to the fluidization of the sample with an increase in gas velocity. As the gas particles became more spaced apart, the gaps between them enlarged, leading to better mixing of the gas–solid reaction. The enhanced mixing promoted the oxidation reaction of calcium sulfite, resulting in the observed increase in the conversion rate.

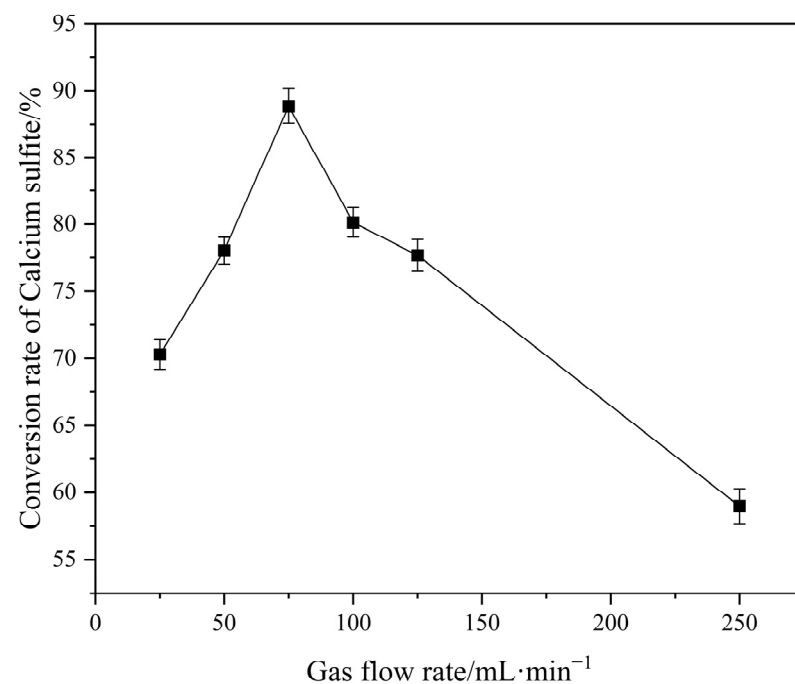


Figure 4. Effect of gas flow rate on the oxidation of CaSO_3 .

At a gas flow rate of $75 \text{ mL} \cdot \text{min}^{-1}$, the reaction conversion rate reached its peak value, approximately 90%. However, as shown in the figure, when the gas flow rate increased from $75 \text{ mL} \cdot \text{min}^{-1}$ to $250 \text{ mL} \cdot \text{min}^{-1}$, the conversion rate gradually decreased from 90% to 60%. This might have been related to the gas velocity, which determines the amount of gas supplied per unit time. With the increase in gas velocity, the gas–solid contact time became shorter. As a consequence, the final oxidation atmosphere may not have contained enough oxygen, and the reduced gas contact time led to insufficient progress of the reaction, thereby reducing the oxidation efficiency.

In summary, an optimal gas flow rate of $75 \text{ mL} \cdot \text{min}^{-1}$ resulted in the highest conversion rate due to the proper balance between gas–solid contact time and the availability of oxygen for the oxidation reaction. However, beyond this optimal point, the increase in gas velocity caused a decline in conversion rate due to inadequate gas–solid interaction and a limited supply of oxygen for the reaction. The reason for this may be that if the oxygen atmosphere enters and leaves the tube furnace at a high flow rate, its contact time with the solid desulfurized ash is reduced, thus reducing the reaction time between oxygen and calcium sulfite, which is not favorable to the oxidation process.

3.4. Effect of CaCl_2 Content on the Oxidation of CaSO_3

After analyzing various desulfurization ash samples, it was established that the primary chloride component present was CaCl_2 . To better understand the influence of chloride ions on the oxidation of CaSO_3 , this experiment was designed to investigate the impact of chloride by mixing different concentrations of calcium chloride.

As evident from Figure 5, the conversion rates of desulfurization ash samples with varying ratios of added CaCl_2 were lower compared to those of calcium sulfite samples. This observation indicates that chloride has an inhibitory effect on the oxidation of CaSO_3 . Furthermore, as the content of CaCl_2 increased, the inhibition of CaSO_3 oxidation became more pronounced. Meanwhile, the change curve of calcium sulfite conversion rate for CaCl_2 content below 10% indicates that a reduction in chloride content of less than 10% led to a decrease in calcium sulfite conversion rate of up to 15%. This finding provides valuable guidance for effectively controlling chlorine content with a balance between production costs and the utilization of by-products.

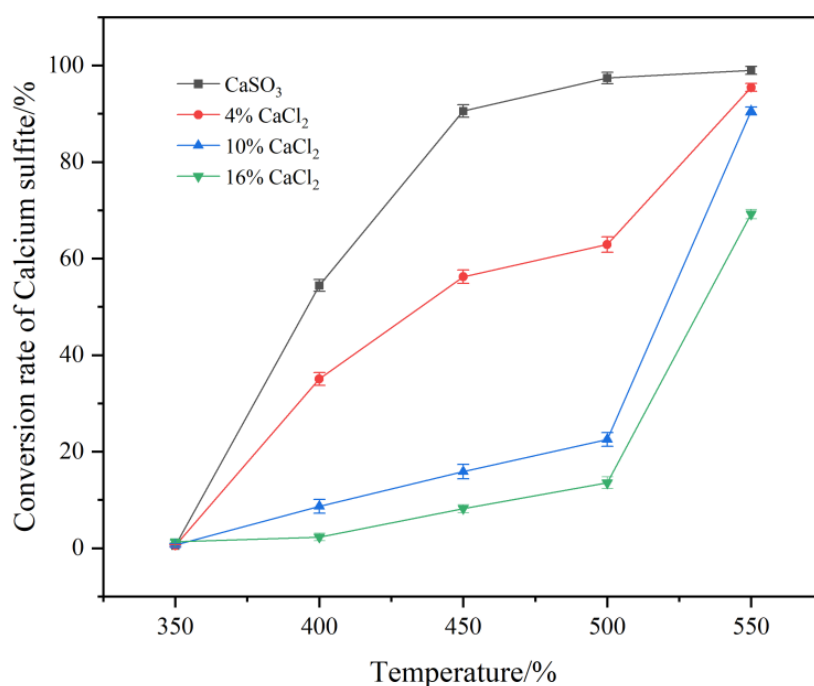


Figure 5. Effect of CaCl_2 content on CaSO_3 oxidation.

From the figure, it can also be observed that in the two sets of experiments with CaCl_2 content of 10% and 16%, the conversion rate of calcium sulfite was significantly lower, and the change in the conversion rate with temperature was relatively slow between 300 °C and 500 °C, followed by a sharp increase when the temperature exceeded 500 °C. This might have been due to the fact that 500 °C was an interface temperature, beyond which CaCl_2 began to decompose. This observation provides valuable guidance for companies dealing with desulfurization ash containing more than 10% CaCl_2 . As shown in the figure, when the reaction temperature reached 550 °C, chloride had minimal effect on the conversion rate of calcium sulfite. This finding serves as a crucial guide for the design of the desulfurization ash oxidation process, and the temperature of 550 °C can be considered a significant interface temperature.

3.5. Thermodynamic Analysis of Desulfurization Ash Oxidation

Using the second law of thermodynamics, the direction and limit of a chemical reaction can be explained based on the principle of entropy increase. However, for complex reactions involving multiple components, especially those with various phase reactions like water, gas, and solid, determining the outcome solely through equilibrium considerations becomes challenging. In such cases, employing the Gibbs free energy function can provide valuable insights into the direction and extent of the reaction by comparing changes in the function.

The reaction equations involved in this experiment are listed below. By calculating the Gibbs free energies at different temperatures using the HSC 6.0 software, it was found that only Equations (1), (3) and (7) proceeded spontaneously below 600 °C.



Specifically, the results of Gibbs free energy calculations, as depicted in Figure 6, reveal that the reaction of calcium sulfite and oxygen ($\Delta G^0 < 0$) represents an easily favored oxidation reaction. Conversely, the corresponding decomposition of calcium sulfite into calcium oxide and sulfur dioxide does not occur readily. Equation (3) indicates that sulfite readily produces calcium sulfate and calcium sulfide. However, Equations (4) and (7) depict decomposition reactions of calcium sulfate and reactions of calcium sulfite with iron salts, respectively, both exhibiting $\Delta G^0 > 0$ which indicates that these reactions do not proceed easily. In Equation (5), $\Delta G^0 > 0$ below 500 °C and $\Delta G^0 < 0$ at 550~600 °C, suggesting that the decomposition reaction of $\text{Ca}(\text{OH})_2$ is possible above 550 °C. When ΔG^0 is less than $-40 \text{ kJ}\cdot\text{mol}^{-1}$, the reaction limit is considered deep, while ΔG^0 greater than $-40 \text{ kJ}\cdot\text{mol}^{-1}$ implies a shallow reaction limit. Consequently, the most probable reactions are CaSO_3 oxidation, followed by CaSO_3 decomposition into CaO and SO_2 . The reaction between CaSO_3 and Fe_2O_3 may be weak, and the decomposition reaction of $\text{Ca}(\text{OH})_2$ is possible above 550 °C.

In order to study the oxidation mechanism of CaSO_3 , analytically pure CaSO_3 was raised to 800 °C in air at four different heating rates, and the TG and DSC data plots of the reaction were obtained after treatment, as shown in Figure 7.

As can be seen from Figure 7, CaSO_3 oxidation in air was divided into the following stages. In the first stage, calcium sulfite carried 0.5 water of crystallization at this stage of decomposition, weight loss rates was about 9%. The second stage was the oxidation of calcium sulfite to calcium sulfate; the sample mass increased, mainly from the oxygen

obtained. At 550 °C, CaSO_3 was basically completely oxidized, the sample mass no longer changed.

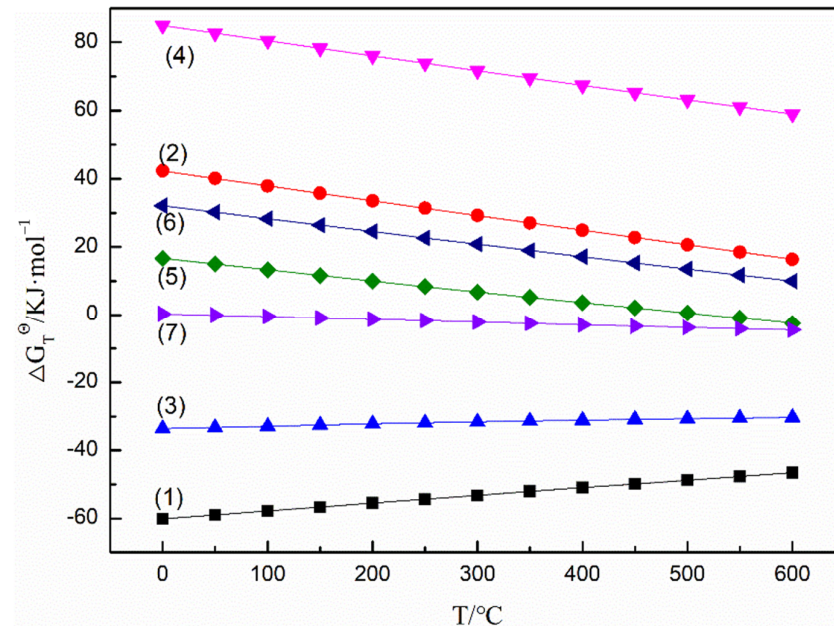


Figure 6. G–T plots for reactions (1) to (7).

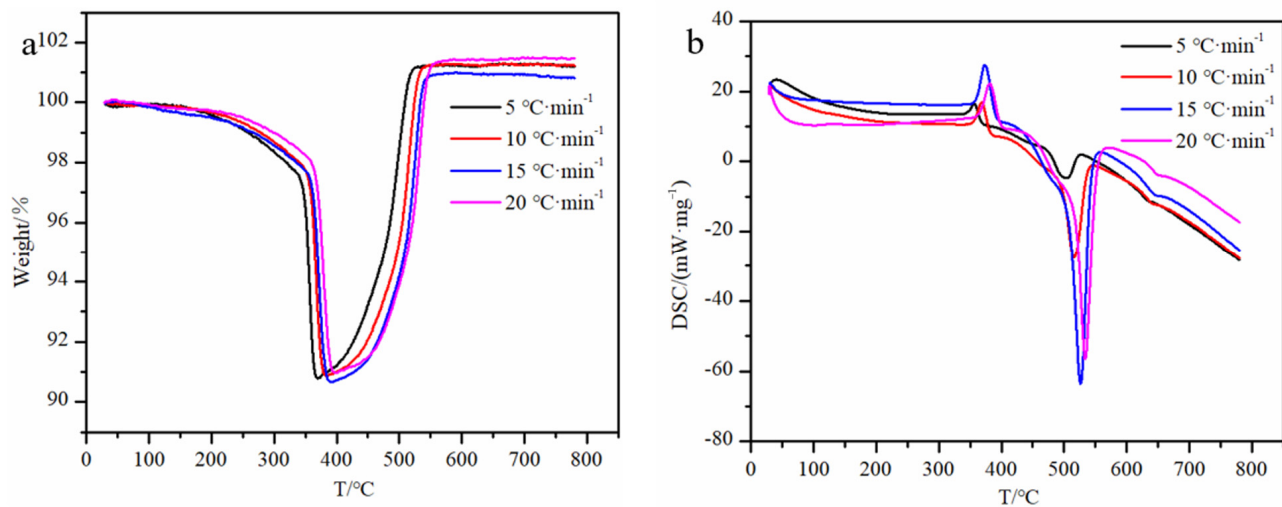


Figure 7. TG and DSC plots of CaSO_3 at different warming rates. (a) TG curve of CaSO_3 oxidation and (b) DSC curve of CaSO_3 oxidation.

4. Conclusions

- (1) The conversion of CaSO_3 in dense-phase tower desulfurization ash was achieved through heating with oxygen-containing flue gas flowing in the main body of the equipment. The efficiency of this conversion process depended on the extent of gas contact, which was more uniform when the flow rate of gas was sufficient. Additionally, the conversion efficiency was influenced by the gradient change in the reactor's reaction flow rate velocity cross-section. Research findings indicate that at a reaction temperature of 420 °C and a gas flow rate of 85 mL·min^{−1}, the conversion efficiency exceeded 89%.
- (2) The oxidation rate of CaSO_3 in desulfurization ash was influenced by the oxygen content, and the conversion rate increased with higher oxygen content. However, the effect of oxygen content had an upper limit and did not show an unlimited impact.

Moreover, at low oxygen content, increasing the reaction temperature significantly enhanced the oxidation conversion rate of CaSO_3 .

- (3) The presence of chloride hindered the oxidation reaction of CaSO_3 , with a particularly pronounced inhibition effect when its percentage exceeded 10%. However, the inhibition effect diminished rapidly at reaction temperatures above 500 °C.
- (4) Through thermodynamic analysis, it was determined that the oxidation temperature of the desulfurization ash should be set below 550 °C during the actual industrial oxidation process, thus preventing the decomposition of calcium sulfate and calcium hydroxide.

Author Contributions: Investigation, H.L.; Writing—original draft, H.L.; Writing—review & editing, H.M. and T.L.; Supervision, H.M.; Project administration, G.L.; Funding acquisition, G.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: Authors Gang Lu and Tingshuang Leng were employed by the company Beijing Beike Environmental Engineering Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest. The Beijing Beike Environmental Engineering Co., Ltd. had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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