# Causes and Control Technology of Slurry Overflow in an Ammonia Desulfurization Tower

Xiaodong Chen,* Xiaoqing Shang, Zhiye Cheng, Zhe Liu, and Xufei Chen

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**ABSTRACT:** In the flue gas ammonia desulfurization process of the coal chemical industry, ammonium sulfate slurry in the desulfurization tower often foams and overflows, which wastes resources and pollutes the environment. The solution to this problem remains largely unknown. This paper aims to reveal the causes of foaming by analyzing foam composition, ammonia desulfurization process raw material source, and characteristics of the flue gas source of the coal chemical industries. It is seen that the organic carboxylate ammonium salt surfactant in the slurry was the main cause of ammonium sulfate slurry foaming. Moreover, due to ammonium sulfate crystals and ash in foam forming a skeleton to support the foaming structure, the foam was not easy to break. More importantly, an appropriate defoaming agent was screened and optimized by an ammonia desulfurization tower simulated device in the laboratory. The YLZ-3 compound defoaming agent, with the optimal defoaming efficiency, was obtained by combining a polyether siloxane copolymer, *n*-octyl alcohol, fumed silica, and deionized water. It had a good temperature stability and little influence on the ammonium sulfate slurry drying time. However, defoaming agent addition could affect the ammonium sulfate crystal form. The foam overflowing could be controlled by spraying the defoaming agent from the top of the tower. Thus, the problem of bubbling overflow of the ammonia desulfurization tower could be resolved very well.
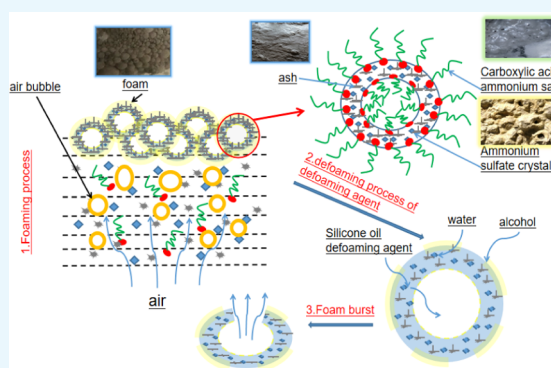
## 1. INTRODUCTION

To reduce environmental pollution, organic waste gas that is generated in the production of the coal chemical industry is generally burned in a self-built electric field for power generation. However, flue gas produced by power plants contains sulfur dioxide $(SO_2)$,[1] which causes serious secondary environmental pollution. Therefore, manufacturers use flue gas desulfurization technology to cope with $SO_2$.

The ammonia desulfurization process is relatively advanced among desulfurization processes. This process has the advantages of simple operation, easy adjustment, strong practicability, strong controllability, lower material consumption during desulfurization,[2−4] and usage of the separated ammonium sulfate as a fertilizer.[5] However, during the ammonia desulfurization process, the slurry in the desulfurization tower is often accompanied by bubbling. If too much bubbling foam is generated and cannot break in time, a large amount of the material overflows from the overflow port, which can waste significant resources and cause serious environmental pollution. Less research exists on bubbling formation in the ammonia desulfurization tower. Therefore, bubbling overflow must be addressed urgently because of the safety and environmental problems.

In this work, we reported the results of the study on the causes of slurry foaming and overflow in the flue gas ammonia

desulfurization tower in a coal chemical industry plant. On the self-built laboratory desulfurization tower simulation device, a novel appropriate defoaming agent was screened and optimized. It had a good temperature stability and little influence on the ammonium sulfate slurry drying time. The foaming height and foam overflowing could be controlled by spraying the defoaming agent from the top of the tower. Thus, the problem of bubbling overflow of the ammonia desulfurization tower, a thorny environmental problem for businesses, could be resolved very well. The result of this research also provided a new technical method for the normal operation of flue gas desulfurization units in other fields.

## 2. MATERIALS AND METHODS

**2.1. Experimental Materials.** Ammonia desulfurization foam slurry (Figure 1a,b) used in the experiment was sampled from the overflow port of a desulfurization tower at Shaanxi
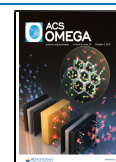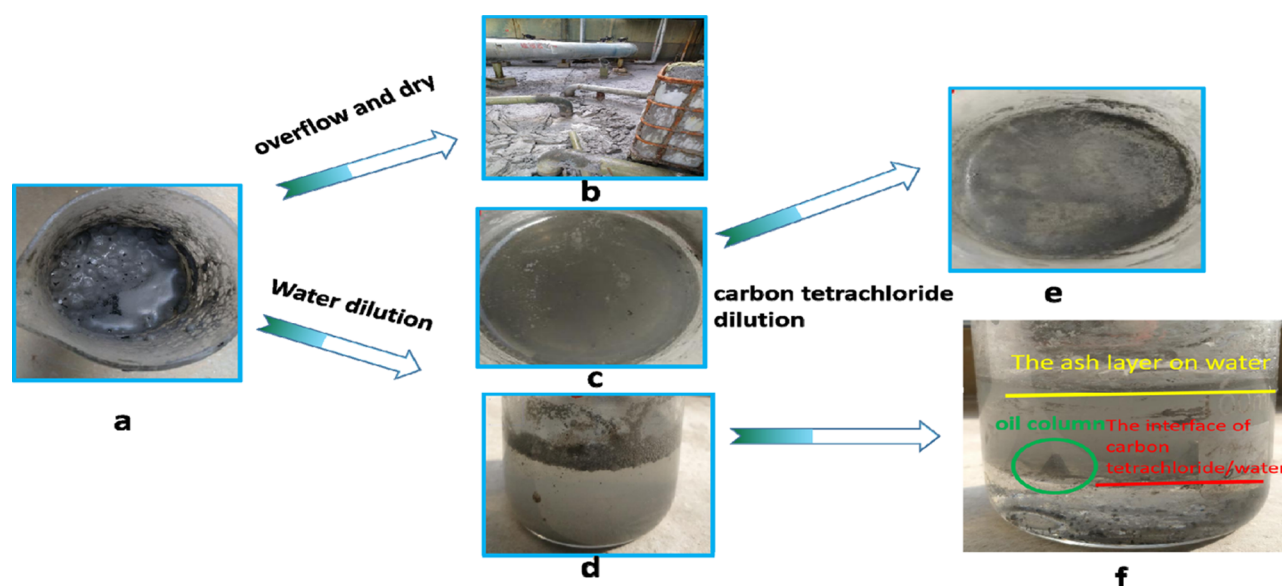
**Figure 1.** Status of the foam overflow of the ammonium sulfate slurry in the factory and water/oil solubility, (a) ammonium sulfate slurry from the overflow port of the desulfurization tower, (b) foam overflows the desulfurization tower to the ground, (c) oil film on the liquid surface after water addition to the foam, (d) observation of oil beads on the liquid surface after water addition to the foam, (e) disappearance of the oil film on the liquid surface after carbon tetrachloride addition, and (f) formation of hollow oil column beads at the carbon tetrachloride/water interface.
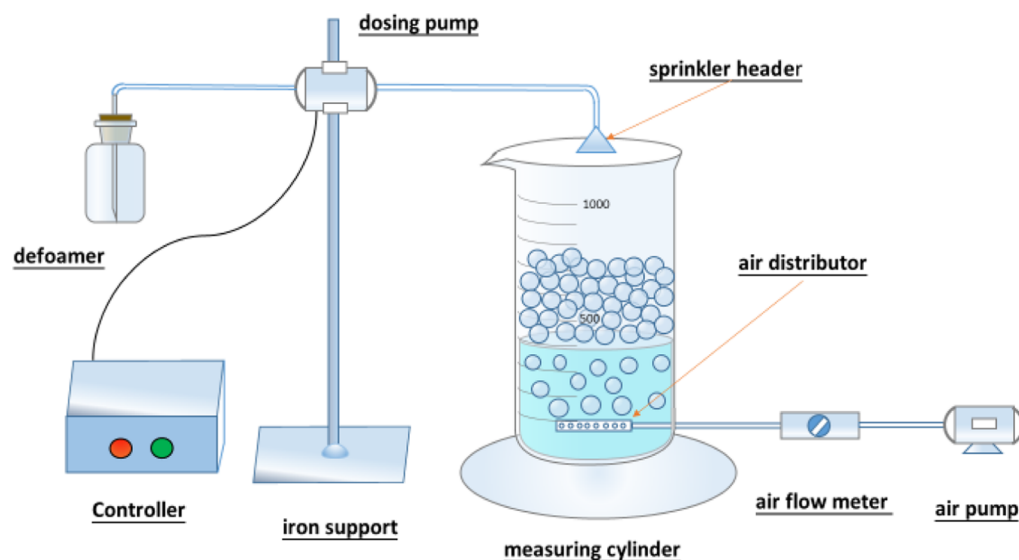


**Figure 2.** Laboratory simulation device for the ammonia desulfurization tower.

Future Energy Chemical Co. Ltd. in Yulin, Shaanxi, China. Non-silicon defoaming agent (BAPE), polyether defoaming agent (GPES polyether), tributyl phosphate (TDP), and polyether siloxane (PDMS) copolymer are commercially available industrial products. *N*-Octyl alcohol, anhydrous ethanol, and carbon tetrachloride were pure reagents for analysis.

**2.2. Experimental Methods.** *2.2.1. Formation Causes Analysis of Foaming.* The 100 g of foam slurry was sampled from the desulfurization tower overflow port, which was diluted with deionized water to 500 g and left to stand for 2 h. Then, the some oily substance floated on the surface of solution. The upper oily substance was extracted with carbon tetrachloride, filtered with quantitative filter paper, and placed with the filter paper in a vacuum drying oven at 100 °C to dry to a constant mass. Infrared spectrum analysis was conducted

on the dried residue by using a Bruker EQUINX55 FT-IR spectrometer in the mid-IR range (4000−500 cm$^{-1}$) with 2 cm$^{-1}$ resolution using KBr pellets. The quantitative overflow foam slurry was placed in an evaporating dish and dried in a vacuum drying oven at 120 °C to constant mass. The morphological and composition analyses of the dried solid dried substances were carried out by scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) analysis performed using a Sigma 300 environmental scanning electron microscope operating at an accelerating voltage of 10 kV.

*2.2.2. Optimization of Defoaming Agent and Choice of Dosing Method at Room Temperature.* A laboratory-simulated ammonia desulfurization tower device was built to simulate the working principle of an ammonia desulfurization tower, as shown in Figure 2. The 300 g of ammonium sulfate
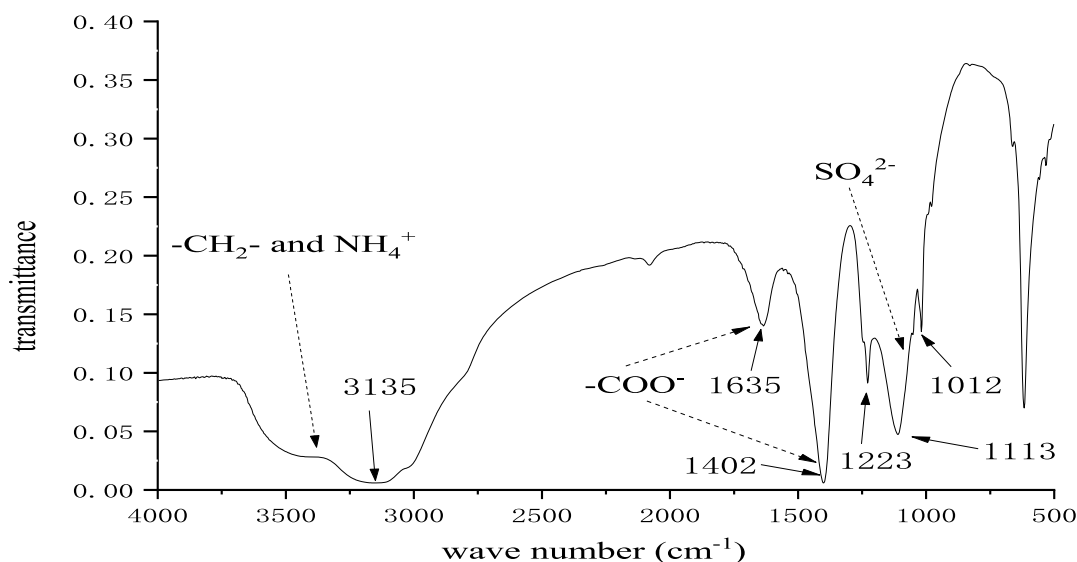
**Figure 3.** Infrared spectra of the oily matter in the carbon tetrachloride solution after extraction.

slurry in the desulfurization tower was placed into a 1000 mL measuring cylinder. A small aeration pump (220−240 V, 3.0 W, air flow 3.0 L/min) was used to inflate the gas to the slurry at a constant flow rate from the bottom of the measuring cylinder. Foam growth was observed and the maximum height that was achieved by foam generation was recorded.

The experimental defoaming agent samples were added quantitatively to 300 g of the ammonium sulfate slurry and inflated in the same manner and at a constant air flow rate. The foam production process was observed and the maximum foam height was recorded.

An industrial production process must select a reasonable and practical approach to defoaming agent addition for foam elimination and to provide a more conducive approach to industrial application. The first method was the addition of the defoaming agent directly into the ammonium sulfate slurry, with even stirring and mixing, and then to conduct aeration bubbling experiments. The second method was to dilute the defoaming agent 10 times with an appropriate amount of the solvent. When the foam grew to a maximum in the foaming experiment, the diluent of the defoaming agent was sprayed with a spray device until the foam collapsed. Comparing the defoaming effect and the amount of the defoaming agent of the two defoaming methods to provide the better selection of a defoaming agent and addition method.

*2.2.3. Influence of Operating Temperature of Desulfurizer on Defoaming.* An optimum dose of the defoaming agent was added at room temperature to the ammonium sulfate slurry, which was heated in a constant temperature water bath (between 60 and 70 °C) to simulate the defoaming test at the desulfurization tower operating temperature. The concentration gradient of the defoaming agent addition at 70 °C was obtained by defoaming using the same steps at room temperature. The foam height ($H_n$) and defoaming time ($T_n$) were recorded. By comparing the experimental data at room temperature and at 70 °C, the influence of the operating temperature of the desulfurization tower on the defoaming effect was revealed. The optimal dosing concentration at 70 °C was established.

*2.2.4. Effect of Defoaming Agent on Crystallization of Ammonium Sulfate.* Four 200 mL beakers were numbered 1,

2, 3, and 4. Saturated ammonium sulfate solution (70 °C) was prepared with reagent ammonium sulfate in beaker 1. A mixture of a saturated ammonium sulfate solution with an optimum concentration of the defoaming agent at 70 °C was prepared in beaker 2. A mixture of the ammonium sulfate slurry and the optimum concentration of defoaming agent at 70 °C was prepared in beaker 3. A mass of 20 g of the ammonium sulfate slurry was placed in beaker 4. Then, 20 g of each of the above four substances placed in glass evaporating dishes were dried at 100 °C in a vacuum drying box at the same time, recording the change in dishes and total mass of the remaining material every 10 min, until the mass no longer decreased. After three repetitions, the average value was calculated for comparative analysis, to establish the influence of the defoaming agent on the crystallization of ammonium sulfate.

## 3. RESULTS AND DISCUSSION

**3.1. Formation Cause Analysis of Foaming.** The desulfurization tower slurry foam was diluted with deionized water and observed after standing. The foam was divided into two layers. The upper layer was oil-formed bubbles and the lower layer was multicomponent aqueous solution (Figure 1c,d). After carbon tetrachloride was added, the solution divided into three layers. The original oil-formed bubbles that were floating on the water surface dissolved into a carbon tetrachloride phase in the lower layer, while leaving only a small amount of ash in the upper layer (Figure 1e). It was interesting to note that a tower-shaped hollow oil-like bubble column formed at the interface between the aqueous phase and carbon tetrachloride (Figure 1f). These phenomena could prove that the layer formation might have resulted because of the presence of a surfactant with a high surface tension in the oil. Therefore, the extracted liquid was filtered by a filter paper to remove mechanical impurities and dry in a vacuum drying oven at 100 °C. Then, its composition was analyzed by infrared spectroscopy.

Infrared spectrum analysis shows that there was organic carboxylate ammonium salt surfactants in the ammonium sulfate slurry-like soap substances. As shown in Figure 3, a wide strong absorption peak existed between 3300 and 2900

$cm^{-1}$ in the infrared spectra, which was the characteristic absorption peaks of the alkyl group and $NH_4^+$. A weak and strong absorption peak existed at 1641 and 1402 $cm^{-1}$, respectively, both of which were characteristic absorption peaks of $-COO^-$ in the carboxylic ammonium salt. Three characteristic absorption peaks of $SO_4^{2-}$ existed at 1223, 1113, and 1012 $cm^{-1}$. The soap surfactant was the main cause of foaming during aerated oxidation. This result was consistent with the fact that the ammonia water used in the industry desulfurization tower was recovered from the preceding process, and the ammonia water contained organic acid ammonium salt.

The foam slurry was taken quantitatively in the evaporating dish and dried in a vacuum drying oven at 120 °C to constant mass. During the foam drying, the gas escaped and formed a gray-white to light-yellow porous solid substance with a considerable volume. The dried solid substance was sampled for morphological and composition analyses using IE350 scanning electron microscope and an EDS energy spectrum analyzer. SEM analysis of the light-yellow porous solid showed that it was crystalline.

On the basis of the EDS element analysis (Table 1), if the sulfide in the flue gas was oxidized completely to ammonium

**Table 1. EDS Analysis of the Ammonium Sulfate Slurry Which Contains (Yes) and Not Contains the Defoamer (No) after Drying**

| element | mass % | | atom % | |
|---|---|---|---|---|
| | yes | no | yes | no |
| C K | 22.49 | 2.11 | 37.90 | 3.21 |
| N K | 5.79 | 22.82 | 8.36 | 29.81 |
| O K | 16.66 | 47.38 | 21.07 | 54.19 |
| F K | 1.56 | 0 | 1.66 | 0 |
| Na K | 0.68 | 0 | 0.60 | 0 |
| Mg K | 0.37 | 0 | 0.30 | 0 |
| Si K | 5.03 | 0 | 3.62 | 0 |
| S K | 38.97 | 19.56 | 24.60 | 11.16 |
| Zr L | 8.46 | 8.13 | 1.88 | 1.63 |
| total quantity | 100.00 | 100 | 100.00 | 100 |

sulfate, and the nitrogen oxide was oxidized completely to ammonium nitrate, it was calculated that the slurry would consist of ammonium sulfate (mass ratio content was ~43.24%), ammonium nitrate (~48%), organic ammonium salt soap (~5%), and a small amount of ash powder.

The processes of foaming and deforming are shown in Figure 4. In the ammonia desulfurization process, air needs to be pumped from the bottom of the tower to oxidize sulfur dioxide, which will produce bubbles in the slurry. As the bubble rises, the ash, ammonium sulfate, and ammonium carboxylate in the slurry can enter the bubble film forming a stable structure, leading to the bubble not being easy to break. If too much bubbling foam is generated, a large amount of the material overflows from the overflow port. It can result in resource wastage and environmental pollution. In order to solve this problem, it is necessary to add a defoaming agent to the slurry to control the generation of foam.

**3.2. Defoaming Mechanism of Defoaming Agent in the Desulfurization Tower.** A defoaming agent generally has two properties, one is the anti-foaming property that inhibits solution foaming and the other is rapid breaking of the formed foam.[6] Therefore, when a defoaming agent is selected, we should choose two kinds of strong properties of the defoaming agent.

The defoaming agent mechanism can be summarized as follows.[7-9] (I) Electrolyte in the defoaming agent disintegrates the surfactant double electrical layer and can induce the liquid film to drain for making the foam lose its stability. (II) The defoaming agent viscosity can prevent liquid loss between liquid films and prevent bubble coalescence. (III) The liquid film surface of the defoaming agent has a high electrodynamic layer potential, which can inhibit bubble coalescence. (IV) The defoaming agent can destroy the membrane elasticity, that is, defoaming agent addition will diffuse to the liquid interface, which makes it difficult for the surfactant with bubble stabilization to restore the membrane elasticity. (V) Hydrophobic solid particle addition is for bubble breakage. The addition of low-molecular alcohol substances allows for full mixing with the solution of the liquid film, which reduces the effective concentration, solubilizes the vacuolizing surfactants, and the local surface tension of the foam can be reduced.
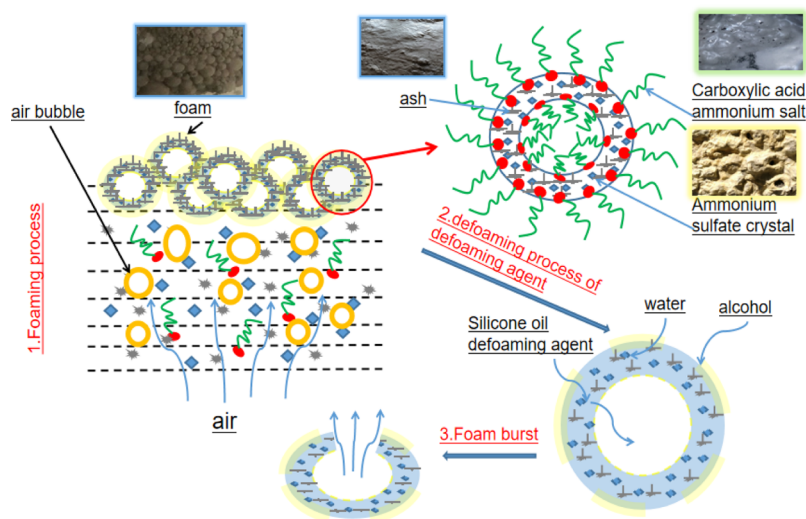


**Figure 4.** Processes of foaming and deforming of the defoaming agent and foam burst in the desulfurization tower.
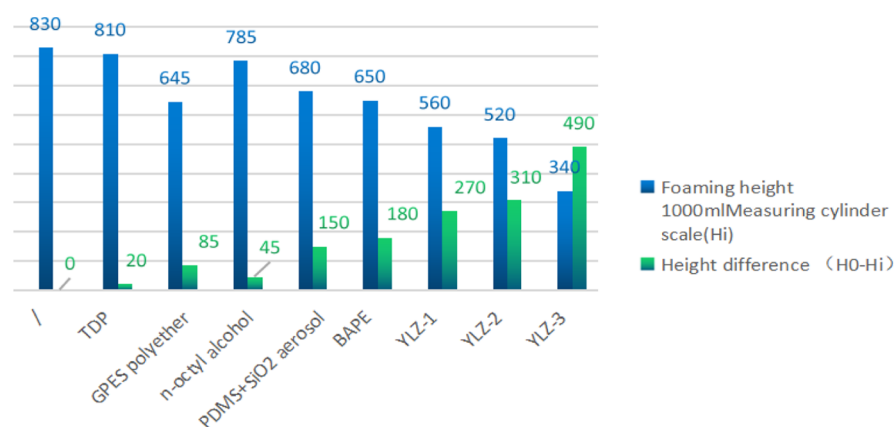
**Figure 5.** Type of the defoamer screened by a desulfurized tower ammonium sulfate slurry defoaming simulator at room temperature (the amount of the defoamer added was 1% of the slurry mass).
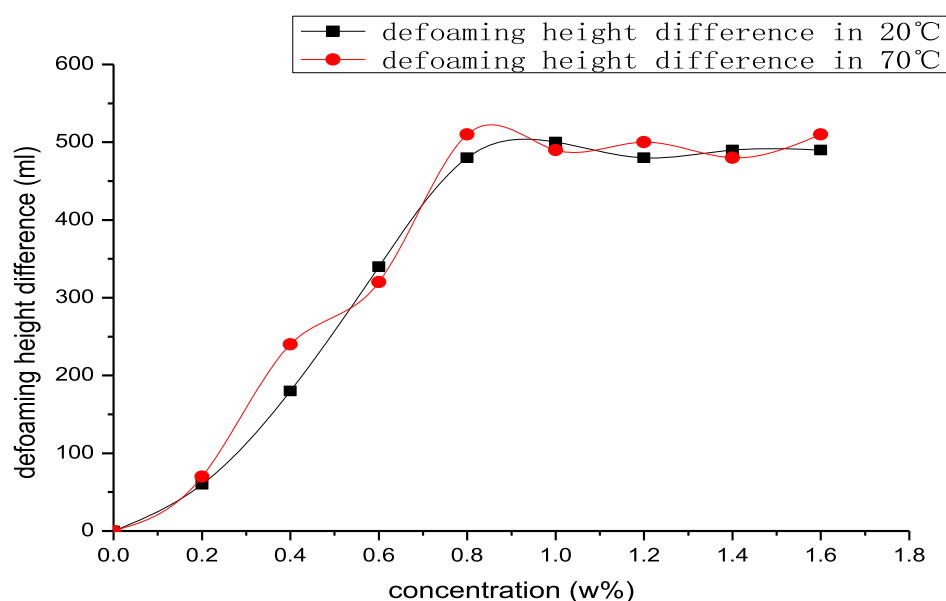


**Figure 6.** Comparison graph of the defoaming effect of the amount of the defoaming agent added at 70 and 20 °C simulated desulfurization tower.

In this work, one or more of the above-mentioned defoaming mechanisms were used to establish the best defoaming scheme and prepare an efficient defoaming agent. Obviously, the foaming phenomenon of the ammonium sulfate slurry in the ammonia desulfurization tower was like that in coating production. The foaming and defoaming method of coating was used as a reference. Because of the difference in the environment and defoaming mechanism in the ammonia desulfurization process, the corresponding market defoaming agents were selected for screening or compounding, and the formula was optimized. The ammonium sulfate slurry defoaming agent was prepared with water, butanol, and silicone oil as the main components. The ammonium carboxylate salt was dissolved with butanol, the ammonium sulfate crystal was dissolved with water, and the double electric layer of the foam was destroyed with a silicone oil defoaming agent (Figure 4). Altogether, the foam of ammonium sulfate slurry can be effectively eliminated.

**3.3. Defoaming Agent Optimization.** *3.3.1. Defoaming Agent Screening.* At room temperature, 300 g of the ammonium sulfate slurry in the desulfurization tower was placed into a 1000 mL measuring cylinder, and the liquid level

was recorded at 280 mL on the measuring cylinder. To simulate the working principle of the desulfurization tower, a small electric charging pump was used to inflate the slurry at a constant flow rate from the bottom of the measuring cylinder, the foam growth process was observed, and the maximum height ($H_0$) that was reached by the foam production was recorded. The defoaming agent was added quantitatively, and the maximum height [$H_i$ ($i = 1-8$)] that was generated by the foam was recorded. The defoaming agent effect was given by the defoaming height difference ($H_0 - H_i$).

To screen suitable defoaming agents, an organosilicone defoaming agent, non-silicon defoaming agent, polyether defoaming agent, and TDP were purchased in the market, and we prepared the compound defoaming agents. For example, YlZ-1 was composed of a PDMS copolymer, ethanol, and deionized water. YlZ-2 was composed of PDMS copolymer, *n*-octyl alcohol, and deionized water. YLZ-3 was composed of the PDMS copolymer, *n*-octyl alcohol, fumed silica, and deionized water.

A comparison with the experimental data is shown in Figure 5. When the amount of the defoaming agent added was 1% of the slurry mass, polydimethylsiloxane (PDMS) and polyoxy-
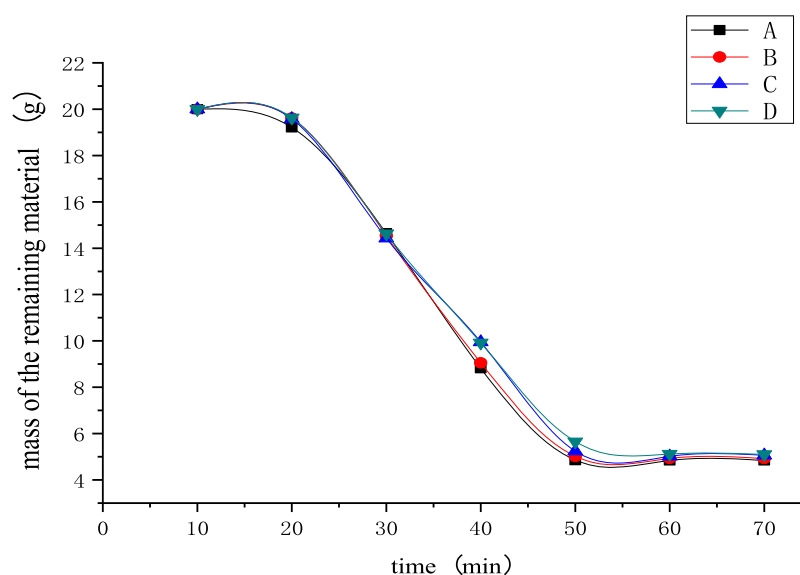
**Figure 7.** Effect of the defoamer on ammonium sulfate crystallization by the crystallization experiment at 100 °C in a vacuum drying oven (A) saturated ammonium sulfate solution (70 °C), (B) mixture of the 0.8% YlZ-3 defoamer was added to the saturated solution of ammonium sulfate, (C) mixture of the 0.8% YlZ-3 defoamer was added to ammonium sulfate slurry, and (D) ammonium sulfate original slurry.

ethylene amine ether (BAPE) that contained fumed silica exhibited a better defoaming effect than TDP, GPES polyether, and n-octyl alcohol alone. However, the foam height remained high when each defoaming agent was used alone, which cannot satisfy the requirements for defoaming in industrial production. According to the desulfurization tower process, the characteristics of the ammonium sulfate slurry and several defoaming mechanisms, theYlZ-3 defoaming agent, which was composed of the PDMS copolymer, n-octyl alcohol, fumed silica, and deionized water, exhibited the best defoaming effect and antifoaming effect, and increased the defoaming height difference to 490 mL. Its foam level was only 340 mL high, which was only 50 mL higher than the original slurry height 280 mL, and controlled effectively the foam height.

The above data show that this polyether-modified organosilicone has the advantages of polyether and organosilicone. The siloxane segment is the lipophilic group and the polyether segment is the hydrophilic group. In the polyether chain segment, the polypropylene oxide chain can provide hydrophilicity, foam breaking properties, and permeability, which reduces the surface tension. The addition of fumed silica can increase the spreading property of the defoaming agent on the bubble, n-octyl alcohol can increase the solubility, so a synergistic role is obtained, which improves the anti-foaming ability of the defoaming agent.

*3.3.2. Influence of Operating Temperature of Desulfurization Tower on Defoaming Property.* The production temperature of the desulfurization tower was between 60 and 70 °C, but laboratory evaluation of the performance of defoaming agents was carried out at room temperature; therefore 70 and 20 °C (room temperature) were selected in the simulated defoaming comparison experiment. The YlZ-3 defoaming agent was added to the ammonium sulfate slurry at different concentrations, and foaming experiments using the same method were performed. The best addition amount was selected depending on the maximum difference in the defoaming height.

According to the experimental data in Figure 6, the defoaming property of the YLZ-3 defoaming agent at room temperature 20 and 70 °C are not significantly different, which indicates that it has a good temperature stability. The defoaming agent effect improves with an increase in the amount of the defoaming agent. When the slurry mass ratio exceeds 0.8%, the growth is flat, and the effect of an increase in the amount of the defoaming agent is small. Therefore, the optimal addition amount of the defoaming agent in the desulfurization tower is a slurry mass ratio of 0.8%.

The YlZ-3 defoaming agent was diluted 10 times with deionized water and loaded into a spray bottle to simulate a spray device at the top of the desulfurization tower. When the foam grew to a maximum during the foaming experiment, the fog-like defoaming agent diluent was sprayed, and the defoaming agent property was studied. The experimental process showed that when the foam increased to a maximum, a small amount of the diluted defoaming agent was sprayed, and the foam dropped rapidly. However, when the spraying stopped, the foaming continued with continuous ventilation at the bottom, and the foaming height was lower than the previous time.

After repeated bubbling, defoaming agent spraying, and foam dropping, when the total defoaming agent concentration was up to 1%, the height difference of the defoaming agent tended to be stable at 480 mL.

In another way, the defoaming agent, diluted 10 times with deionized water, was mixed well with the slurry, and then air was pumped at the bottom, which was used to simulate the way of dosing at the bottom of the tower. A comparison of the antifoaming effects of the two dosing methods showed that the top spraying method was intermittent dosing, which controlled the foam height and prevented the foam from overflowing the overflow port. This method consumed a lower defoaming dose than the bottom method, which was more economical and effective.

**3.4. Influence of Defoaming Agent on Crystallization of Ammonium Sulfate.** To eliminate the influence of the defoaming agent on the subsequent ammonium sulfate crystallization in industrial application,[10] it is necessary to simulate the industrial process and investigate whether the

defoaming agent affects the crystallization time of the paste after a defoaming agent addition of 0.8%.

The four materials 20 g each were accurately weighed [(A) saturated ammonium sulfate solution (at 70 °C), (B) the mixture of 0.8% YlZ-3 defoaming agent was added to the saturated solution of ammonium sulfate, (C) the mixture of 0.8% YlZ-3 defoaming agent was added to the ammonium sulfate slurry, and (D) ammonium sulfate original slurry]. The four materials were placed in the evaporating dish with the corresponding number and in a high-temperature (100 °C) vacuum drying oven for the crystallization experiment. Every 10 min, the change in the total mass of the weighing surface dish and the remaining materials were recorded until the mass was stable. After three repetitions, an average value was taken for comparative analysis to determine the influence of the defoaming agent on the crystallization of ammonium sulfate.

The industrial crystallization of ammonium sulfate was simulated by using a vacuum drying chamber. With drying, water in the material continued to evaporate, and the four samples reached a constant mass within ∼50 min. In addition, the overall variation trend of the four samples is basically the same. Therefore, at the same temperature and vacuum drying time, regardless of whether the reagent was ammonium sulfate or ammonium sulfate slurry, the time difference in water evaporation in the solution with and without defoaming agent was small (Figure 7). Maybe because the amount of the added defoaming agent was relatively small, which has little influence on the ammonium sulfate slurry drying time.

The SEM diagram (Figure 8a,b) of the ammonium sulfate slurry crystal before and after defoaming agent addition
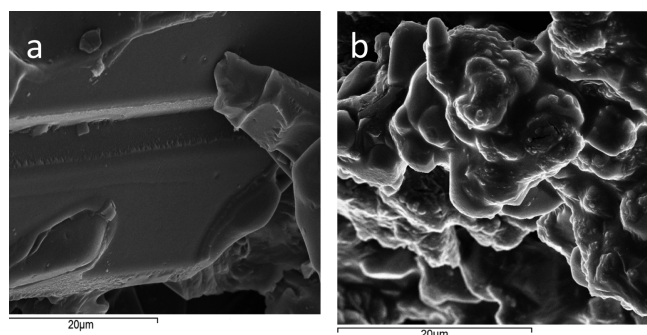


**Figure 8.** SEM comparison of ammonium sulfate slurry crystal no defoamer addition (a) and after defoamer addition (b).

showed that the ammonium sulfate crystal without defoaming agent presents a regular crystal type, whereas the ammonium sulfate crystal changes after defoaming agent addition. The energy spectrum analysis of the desulfurized tower ammonium sulfate slurry after drying and defoaming agent addition (Table 1) show that the high C content and some elemental F and Si are present because of the defoaming agent contribution. Therefore, the defoaming agent can affect the crystallization of ammonium sulfate, and the mechanism of its influence needs further work.

## 4. CONCLUSIONS

During ammonia desulfurization processes, foaming overflow often occurs in the ammonium sulfate slurry of the desulfurization tower. The overflow wastes resources and results in industrial environmental pollution. In this study, the main cause of foaming of the ammonium sulfate slurry was

found. In the ammonia desulfurization process, pumped air from the bottom of the tower to oxidize sulfur dioxide will produce bubbles in the slurry. As the bubble rises, the ash, ammonium sulfate, and the organic carboxylate ammonium salt in the slurry can enter the bubble film, forming a stable structure, leading to the bubble not being easy to break. If too much bubbling foam is generated, a large amount of the material is carried by the foam overflow from the overflow port. The appropriate defoaming agent was screened and optimized by the laboratory simulation ammonia desulfurization tower device. The YlZ-3 compound defoaming agent that was prepared from the PDMS copolymer, *n*-octyl alcohol, fumed silica, and deionized water had the highest defoaming efficiency and a good temperature stability. The foam height could be controlled by spraying the defoaming agent from the top of the tower, and the defoaming dose was less than that of the dosing method at the bottom of the tower, with a higher economy and more obvious effect. Although defoaming agent addition affects the ammonium sulfate crystal form, it has little effect on the crystallization time. Therefore, defoaming agent addition provides a feasible method to control the foam overflow of the desulfurization tower, which can solve the problem of resource wastage and environmental pollution that is caused by the bubbling overflow of the desulfurization tower in the industrial ammonia method.

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**Notes**

The authors declare no competing financial interest.

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