Effects of Foaming and Antifoaming Agents on the Performance of a Wet Flue Gas Desulfurization Pilot Plant

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Foaming is a common phenomenon in industrial processes, including wet flue gas desulfurization (FGD) plants. A systemic investigation of the influence of two foaming agents, sodium dodecyl sulphate (SDS) and egg white albumin (protein), and two commercial antifoams on a wet FGD pilot plant operation has been carried out. Foaming caused by 0.03 g SDS/(L slurry) reduced the desulfurization degree from 84 to 74% and the solids and limestone concentrations of the slurry from 58 to 48 g/(L slurry) and from 1.4 to 1.0 g/(L slurry), respectively. These effects were attributed to the foaming transferring small particles to the foam layer present on top of the slurry in the holding tank. The addition of 0.03 g antifoams/(L slurry) to SDS foam eliminated the foam, but the desulfurization degree remained low. Potential mechanisms for the observed behavior are analyzed. © 2014 American Institute of Chemical Engineers *AIChE J*, 60: 2382–2388, 2014
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Introduction

Foaming within industrial processes may provide both benefits and operational problems. Benefits include an increased surface area for absorption,[1] potential separation of minerals by flotation,[2] and protection of melts/beer wort from the surrounding atmosphere.[3] Operational problems in industrial equipment, such as waste water treatment, distillation towers, and wet flue gas desulfurization (FGD) plants, include incorrect process monitoring (density and liquid level readings) and slurry overflow. Several foaming episodes have also occurred at Danish[4] and international wet FGD plants.[5]

The individual bubbles in the gas–liquid matrix, constituting the foam layer, may either be polyhedral or spherical depending on the liquid content.[6] Foaming can be induced by surfactants, which lower the surface tension and help stabilizing the foam by the Gibbs-Maragoni effect, or by polymers/macromolecules, which form a viscoelastic network that enhances the mechanical stability of the lamella and slow down thinning of the lamella (increased viscosity and steric interactions).[7] The term 'froth' is used to describe foaming in the presence of particles, which may play an important role for bubble stability.[8]

In wet FGD plants, flue gases, containing SO_2 and other acidic gases, are brought into contact with an alkaline limestone slurry. The SO_2 removal efficiency depends on mass transfer through both the gas and the liquid film.[9] To obtain a saleable gypsum ($CaSO_4{\cdot}2H_2O$) product, most installed FGD capacity utilizes the wet FGD forced oxidation system.[10] The overall chemical reaction in this case is

$$CaCO_3(s) + SO_2(g) + 2H_2O(l) + {}^1/_2O_2(g) \rightarrow CaSO_4 \cdot 2H_2O(s) + CO_2(g) \quad (1)$$

Foaming in wet FGD plants can interfere with process monitoring equipment (liquid level and slurry density readings) and cause gypsum scaling, high solids concentrations (because of an artificial low density signal), carryover of slurry into the duct work and the booster fan, and potentially cavitation of recycle pumps.[5] When discovered, foaming can, in principle, be controlled by addition of antifoam, but the required dosage and the effectiveness may vary considerably from one incident to the next. A few observations of foaming in wet FGD pilot plants, including a falling film column[11] and a jet bubbling reactor (JBR),[1] have been reported in the literature. Due to an enhanced contact surface area between the flue gas and the slurry, a significantly increased desulfurization degree was observed in the JBR experiments (from 80 to $>99\%$).[1] A systematic investigation of the influence of particles, electrolytes, and buffers on weak foaming in a wet FGD pilot plant has also been reported.[12] Effects of three antifoams on foams generated by three foaming agents in a small Bikerman lab-scale setup, at conditions of relevance for wet FGD plants, were previously investigated.[4] The present work considers the influence of powerful foaming agents and antifoaming agents on a wet FGD pilot plant operation. In particular, the desulfurization degree and the concentration of residual limestone in the slurry have been studied.

Strategy of Investigation

This investigation of wet FGD operation during foaming and antifoaming is based on a series of pilot-scale

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experiments. The SO_2 concentration (1000 ± 30 ppmv) simulates the combustion of coals containing 1.2% sulfur, 10% ash, and 10% moisture at $\lambda = 1.2$ (air-fuel equivalence ratio). The SO_2 (g) and HCl (g) absorbed from the flue gas will be converted into gypsum and Cl^- in the slurry, respectively.

The latter is simulated by addition of $CaCl_2$ [5 g Cl^-/(L slurry)] to the wet FGD slurry, representative of typical cocombustion with biomass. Gypsum [100 or 150 g/(L slurry)] was added to the holding tank before each experiment to simulate the gypsum concentration at commercial wet FGD conditions. Limestone (Faxe Foderkalk) has been used as reactant, except for one experiment in which NaOH was used to test the interaction between foaming and a solid-free (liquid) reactant. Sodium dodecyl sulphate (SDS) and egg white albumin (protein) are chosen as foaming agents. SDS is a powerful classical surfactant and a common foaming agent in the foaming literature.[13,14] SDS is not present at typical wet FGD operational conditions but is used in this investigation to represent a strong and persistent foaming agent. Organic contaminations and impurities present in wet FGD plants can introduce such powerful foaming agents, which makes SDS foaming an important part of this study. In addition, SDS has been used in many other foaming studies, and therefore, the SDS results of this work may also have comparative relevance in other fields. Protein-induced foaming has been experienced within various industrial processes, for example, in waste treatment plants[15,16] and fermentation processes.[17] In wet FGD systems, the slurry provides a very suitable environment (temperatures in the range of 40–60°C) for bacterial growth and degradation to generate compounds, which might potentially induce foaming operational problem in wet FGD plants.[5,18] For example, in Denmark, large volumes of slurry are stored during maintenance of wet FGD plants, giving time for bacterial growth. This often causes foaming, overflow to ductwork and flooding of the surrounding area, when the slurry is returned to the wet FGD holding tank. High bacterial/biological activity has been detected in those slurries by a test kit.[4] Therefore, protein is chosen in this work as a foaming agent. The commercial antifoams used in this work, Nalco FM-37 (oil-based, Nalco) and Foamtrol 2290 (silicon-oil, General Electric (GE) Power and Water) have been reported to be useful for international wet FGD plants.[5] Antifoam can be used to both prevent foam formation and break existing foam.

Experimental Setup

The wet FGD pilot plant used in this work, shown in Figure 1, simulates a single cocurrent channel in a commercial packed wet FGD tower. The setup consists of a natural gas burner, a falling film column (absorber), a holding tank, a feed tank, a product tank, and a recycle pump. SO_2 is added to the flue gas from the gas burner. The absorber is a 7-m vertical tube made of polyvinyl chloride (PVC) with multiple sampling sites (gas phase concentrations and pH of slurry). In the absorber, flue gas contacts with an alkaline slurry and SO_2 is removed. The 110-L holding tank is made of PVC and contains 30-L slurry. The holding tank is equipped with four baffles, one stirrer in the center, and air injection. The temperature in the holding tank is kept at 40–45°C and the pH value in the holding tank is kept constant (5.4) by addition of 7.3 wt% limestone slurry based on a pH regulation.

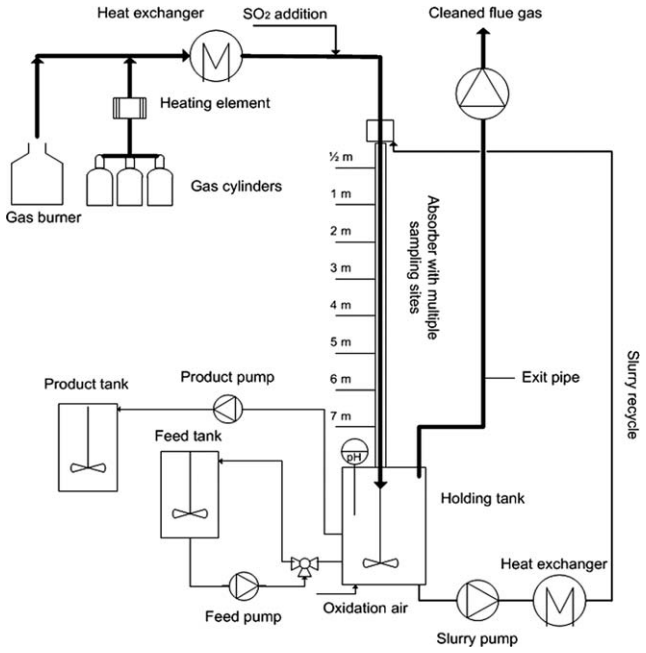

Figure 1. Schematic diagram of wet FGD pilot plant used in this study (after Hansen et al., 2011).[19]

Slurry and foam samples can be withdrawn from the holding tank. Slurry from the holding tank is recycled to the top of the absorber by a peristaltic pump.

Experimental Procedure

An overview of the experiments performed is shown in Table 1. The conditions of the experiments have been chosen to simulate the process conditions in wet FGD plants. Each experiment starts with 2 h desulfurization of a 1000 ± 30 ppmv SO_2 flue gas stream in the absence of foaming. During the initial 2 h "base case" (marked as index "a"), the SO_2 concentration and pH values in the absorber were measured and slurry samples were withdrawn from the holding tank. Limestone input is controlled by pH in the holding tank, whereas the product pump maintains a fixed liquid level during the initial 2 h "base case." The startup slurry is made from demineralized water, gypsum (99%, Sigma-Aldrich), and $CaCl_2$ (technical purity, Macco Organique). Due to sedimentation, less gypsum, about 60 or 90 g/(L slurry), than initially added [100 or 150 g/(L slurry)] is obtained from the slurry sampled from the holding tank. During the initial 2 h base case operation, the limestone input rate, the limestone concentration in the system, and the desulfurization degree will become stable. The next part of the experiment, marked as index "b," is initiated by addition of foaming or antifoaming agents through the absorber pH sampling port at 5 m. Foaming agents include technical grade SDS (Riedel-de Haeen AG) and grade II egg white albumin (Sigma-Aldrich) as well as two commercial antifoams: Nalco FM-37 (oil-based, Nalco) and Foamtrol 2290 (silicon-oil, GE Power and Water). No slurry is removed during this part of the experiment, thereby ensuring a fixed level of foaming agent in the pilot plant. The change in slurry volume, due to the feed flow, is limited to 2–3 L slurry, because of the short experimental duration.

Table 1. Overview of the Experimental Conditions in the Pilot Plant, Solids, and Limestone Concentrations in the Slurry and Desulfurization Degree at 7 m and Exit (A Represents Base Case; B Represents Foaming; C Represents Antifoam Addition).

Exp.	pH in Holding Tank (−)	Foaming Agent [g/(L slurry)]		Duration (h)	Solids Concentration [g/(L slurry)]	Limestone Concentration [g/(L slurry)]	Foam Height (10^{-2} m)	η at 7m (%)	η at Exit (%)
1A	5.38 ± 0.03	SDS	0	2	57 ± 0.9	1.4 ± 0.2	0	70	84
1B	5.43 ± 0.07	SDS	0.03	1	48 ± 1.4	1.0 ± 0.1	10	64	74
2A	5.38 ± 0.03	SDS	0	2.5	60 ± 0.6	1.3 ± 0.1	0	70	84
2B	5.44 ± 0.07	SDS	0.06	2	49 ± 0.2	1.0 ± 0.1	29	62	73
2B[a]	5.44 ± 0.07	SDS	0.06	2	67 g/foam L	–	–	–	–
3A	5.49 ± 0.06	SDS	0	2	90 ± 4.4	1.9 ± 0.3	0	72	86
3B	5.58 ± 0.11	SDS	0.03	2	86 ± 0.6	1.7 ± 0.2	15	64	78
4A[b]	8.77 ± 0.68	SDS	0	0.5	[c]	0	0	86	93
4B[b]	7.81 ± 0.1	SDS	0.03	0.5	[c]	0	10	85	93
5A	5.39 ± 0.02	SDS	0	2	62	1.13	0	76	90
5B1	5.41 ± 0.03	SDS	0.03	120[d]	56	1.07	20	66	79
5B2	5.40 ± 0.02	SDS	0.03	120[d]	62	1.85	9	67	81
5B3	5.39 ± 0.02	SDS	0.03	120[d]	71	1.40	6	68	82
5B4	5.39 ± 0.01	SDS	0.03	120[d]	83	1.91	0	75	88
6A	5.38 ± 0.01	Egg white albumin	0	2	67 ± 0.2	1.4 ± 0.1	0	72	84
6B1	5.37 ± 0.01	Egg white albumin	0.33	2	54 ± 0.8	2.1 ± 0.1	16	65	75
6B2	5.38 ± 0.01	Egg white albumin	0.33	2	54 ± 0.8	2.6 ± 0.5	10	71	84
7A	5.389 ± 0.02	–	0	2	61 ± 1.0	1.5 ± 0.1	0	71	84
7C[e]	5.389 ± 0.01	–	0.03	1.5	68 ± 0.5	1.9 ± 0.1	0	70	83
8A	5.389 ± 0.01	SDS	0	2	65 ± 0.1	1.5 ± 0.2	0	71	84
8B	5.389 ± 0.02	SDS	0.03	1.5	65 ± 0.5	1.2 ± 0.1	10	64	75
8C[e]	5.389 ± 0.01	SDS[a]	0.03	2	58 ± 1.0	1.4 ± 0.1	0	63	76
8C[e,f]	5.389 ± 0.01	SDS[a]	0.03	2	176	3.7	0	–	–
9A	5.4	SDS	0	2	64 ± 0.9	1.0 ± 0.1	0	71	84
9B	5.4	SDS	0.03	1.5	55 ± 0.3	0.9	6	64	76
9C[g]	5.4	SDS[b]	0.03	1	58 ± 1.5	1.5	0	63	76

[a]Sample from foam layer.
[b]NaOH was used as reactant.
[c]Only Na_2SO_4 (highly soluble) formed because NaOH was used as reactant.
[d]7 out of the 120 h were desulfurization operation time.
[e]0.03 g/(L slurry) Nalco FM-37.
[f]Sample withdrawn from the surface of the liquid phase.
[g]0.03 g/(L slurry) Foamtrol 2290.

Analyses performed include: the SO_2 concentration before the absorber and at selected points along the absorber, the pH profile in the absorber, and foam and liquid heights in the holding tank. Slurry samples from the holding tank are withdrawn to measure the solids and limestone concentration by drying and thermo-gravimetric analysis. For selected experiments, samples from the foam layer have been withdrawn to determine the content of solids and limestone. Particle-size distribution (PSD) analyses have been performed by laser diffraction (Malvern Mastersizer S longbed), using ethanol as background solution. All PSD analyses are the average of five individual and sequential measurements. Scanning electron microscopy (SEM) pictures of selected solid samples were furthermore performed at the Technical University of Denmark, Center for Electron Nanoscopy (DTU-CEN) using a Field Emission Inc (FEI) Inspect S SEM. Previous pilot plant investigations[9] have shown a swift stabilization (<0.2 h) of the desulfurization degree with a standard deviation of the SO_2 measurement around 1%. An uncertainty of this magnitude was also found for the experiments of this work (not shown in all figures).

Results and Discussion

The influence of SDS and protein foaming on the desulfurization degree, pH profile in the absorber, and the solids and limestone concentrations of the slurry will now be discussed. Subsequently, the effects of two commercial antifoams are considered. Details of experimental conditions can be found in Table 1.

Effects of SDS foaming on wet FGD operations

SDS is a powerful foaming agent, capable of generating a rather persistent foam layer that enables an investigation of interactions between foam and the desulfurization degree. Low SDS concentrations [0.03 and 0.06 g/(L slurry)] are able to generate sustainable foam. Figure 2 shows the desulfurization degree of three base case experiments (Exp. 1a, 2a, and 3a) and three SDS experiments (Exp. 1b, 2b, and 3b)

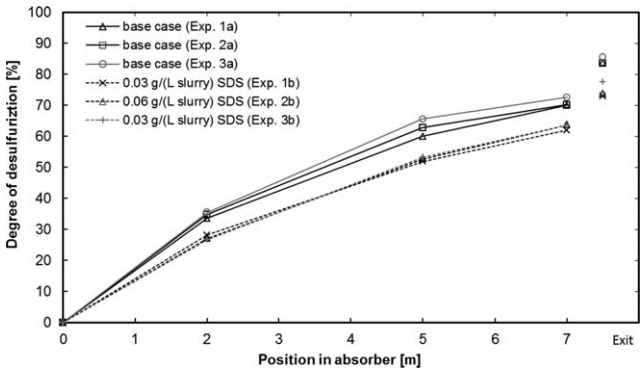

Figure 2. Desulfurization degree for base case experiments and SDS foam experiments.

Exp. 1a, 1b, 2a, and 2b all have a low gypsum concentration [48–60 g/(L slurry)], whereas Exp. 3a and 3b both have a high gypsum concentration [86–90 g/(L slurry)]. Further details are provided in Table 1.

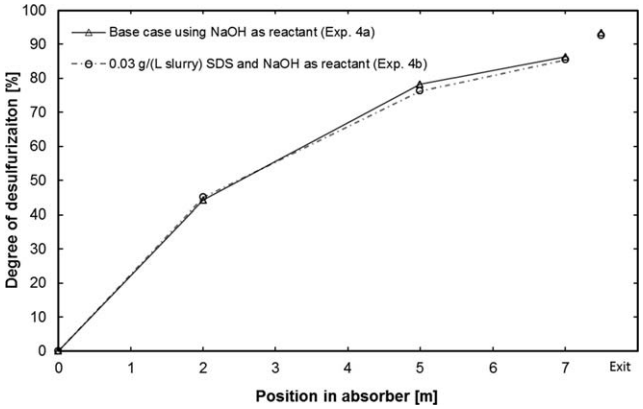

Figure 3. The influence of SDS [0.03 g/(L slurry)] foam on the desulfurization degree when using 1M NaOH as reactant instead of limestone.

Experimental conditions are provided in Table 1.

with two SDS foaming concentrations [0.03 g/(L slurry) and 0.06 g/(L slurry)]. During SDS foaming, the overall desulfurization degree decreases from 84.3 ± 1.0 to $74.7 \pm 2.5\%$, with a limited dependence on foam height and SDS concentrations (as illustrated in Figure 2).

Figure 3 shows that the degree of desulfurization in the absorber, while using NaOH (1 M) as reactant (i.e. a liquid reactant instead of particulate limestone), is unaffected by SDS foaming. This suggests that transfer of limestone particles to the foam layer reduces the amount of limestone available in the slurry and the SO_2 capture in the absorber is reduced (lower desulfurization degrees were obtained during foaming for all limestone experiments). The presence of a foam layer may reduce the gas residence time in the holding tank and thereby potentially the overall desulfurization degree: no such effect was, however, seen in Exp. 4 where NaOH (1 M) was used as reactant. Slightly lower gypsum concentrations [5–10 g/(L slurry) lower] were observed in the slurry during foaming at all gypsum concentrations investigated [from 57 to 90 g/(L slurry), Table 1]. Due to a required replacement of a pH controller and repair of a feed valve, better control of limestone input (base case) was obtained after the first two experiments (Exps. 3 and 5) with some variations seen in the base case limestone concentration [from 1.0 to 1.9 g/(L slurry)]. In Exp. 2, the gypsum concentration decreased from 60 g/(L slurry) before addition of SDS to 49 g/(L slurry) 1.5 h after the onset of foaming,

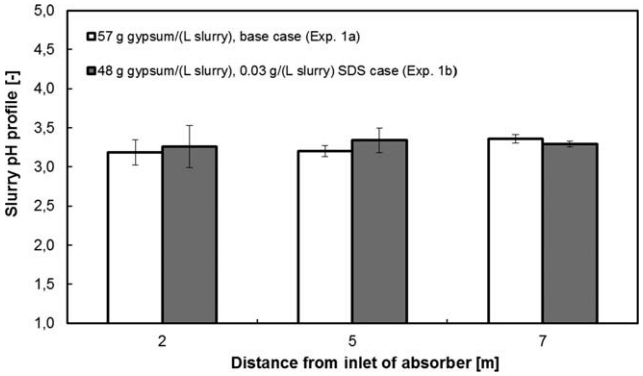

Figure 4. pH profile in the absorber (error bars indicate maximum/minimum value measured).

Experimental conditions are provided in Table 1.

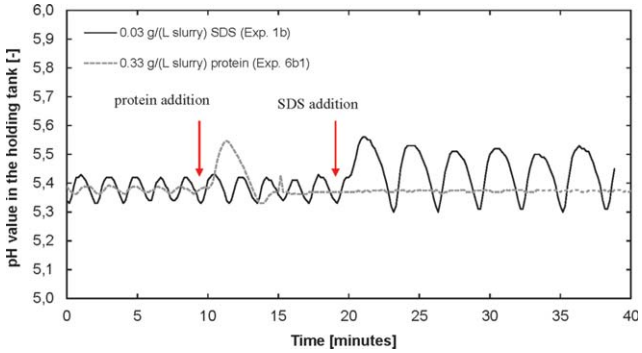

Figure 5. pH value in the holding tank when using SDS and protein (Exp.1b and 6b1).

Experimental conditions are provided in Table 1. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

meaning that 543 g or 27% of the gypsum in the slurry (initially present + formed during the 1.5 h) should have been transferred to the foam layer. However, a foam sample, withdrawn from Exp. 2, indicated an even higher gypsum concentration in the foam layer [67.9 g/(L slurry) or in total 1110 g in the 16.3 L foam]. This was likely due to a compression of the foam during sampling and thereby an increase in volumetric concentration.

The pH value in the absorber is influenced by the SO_2 absorption, the availability of limestone, and the rate of limestone dissolution. The absorber pH does not change significantly during foaming (Figure 4), whereas the holding tank pH shows higher fluctuations after the onset of foaming (Figure 5), likely due to the entrainment of small limestone particles by the foam layer. It can be seen in Figure 5 that before SDS addition, pH in the holding tank is kept at 5.38 ± 0.03, while the onset of SDS foaming changes pH in the holding tank to 5.44 ± 0.07. The higher pH fluctuations in the holding tank cause unstable limestone input, resulting in the lower desulfurization degree.

For further verification that small particles were transferred into the foam layer, PSDs are shown in Figure 6, and SEM pictures recorded for solids sampled from slurry and foam layer are presented in Figure 7. For comparison, PSD's of pure limestone and gypsum are also shown. The data shows that smaller particles are transferred to the foam layer. As shown in Figure 7, pure limestone (upper left) and the

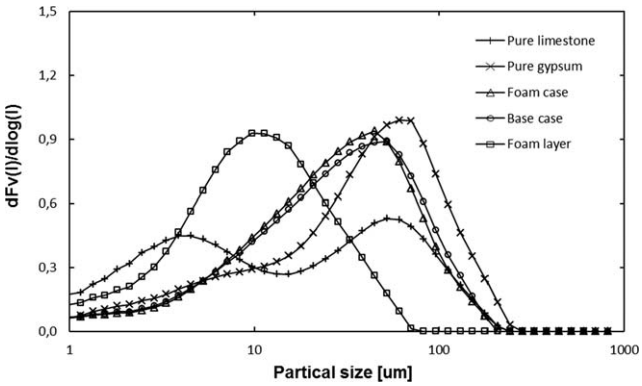

Figure 6. PSDs of solid samples from wet FGD slurry and foam layer.

PSDs of pure limestone and pure gypsum are also shown.

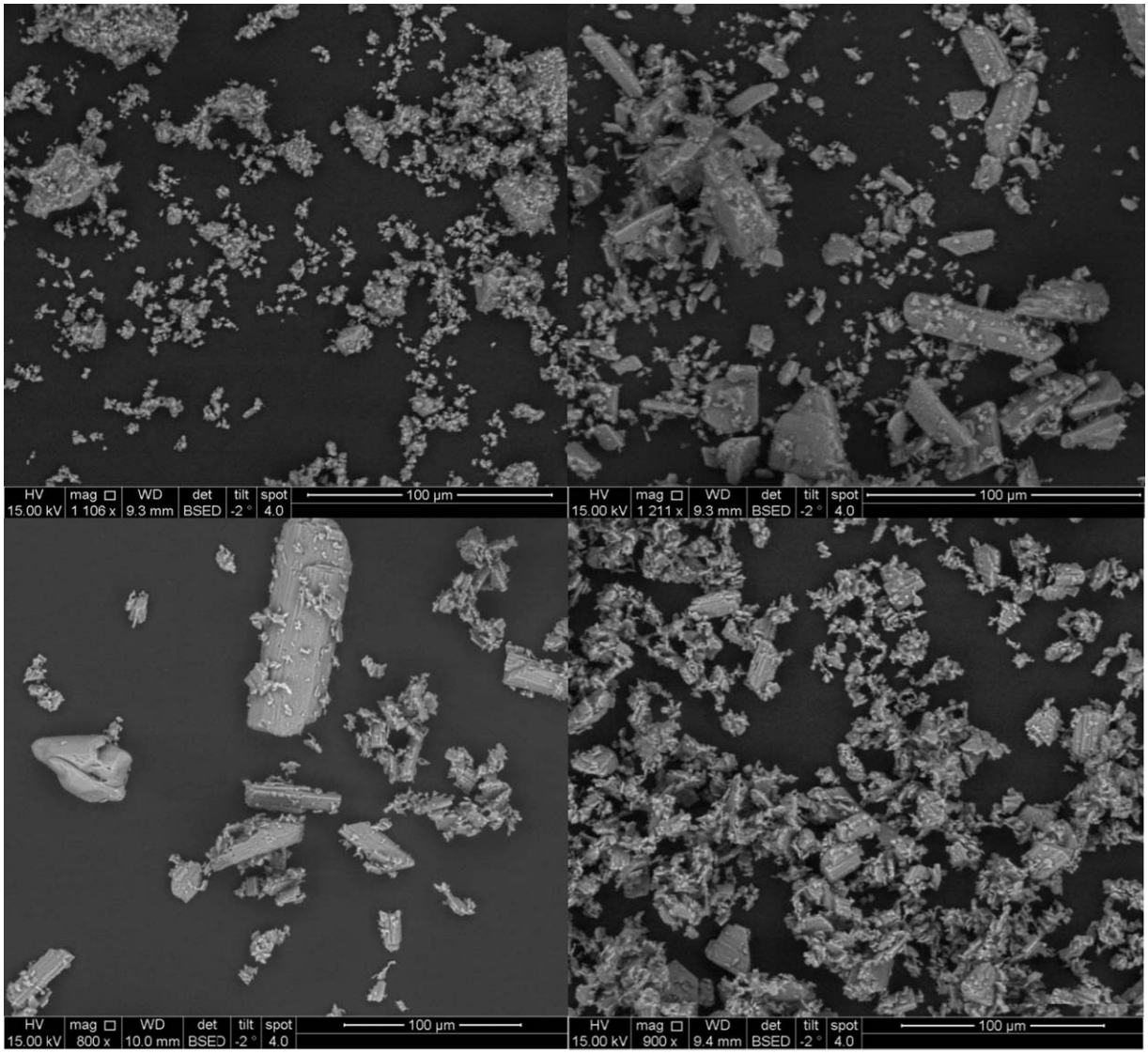

Figure 7. SEM pictures of slurry and raw materials [upper left pure limestone (Faxe Foderkalk); upper right pure gypsum (Sigma-Aldrich); lower left base case slurry; lower right foam layer slurry].

solid particles in the SDS foam layer (lower right) mainly consist of small particles, while pure gypsum (upper right) and solid particles of base case slurry (lower left) consist of larger particles. With time, the SDS foam will disappear and the entrained particles should be returned to the slurry, thereby restoring the conditions and system performance back toward what was seen for the initial base case. Figure 8 shows the development of desulfurization degree as a function of time after the onset of SDS foaming. During the first 47.5 h (6 h desulfurization and 41.5 h standby), the desulfurization degree remains significantly lower than the base case value (from 78 to 82% vs. 89%), but a slow return is subsequently seen [88% after 120 h (7 h desulfurization and 113 h standby)]. However, after 120 h, the presence of a very low SDS foam layer is still able to affect the desulfurization degree.

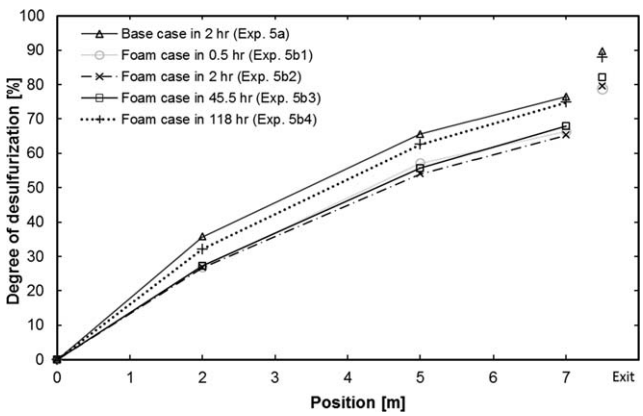

Figure 8. Long term base case and SDS foam experiments [0.03 g/(L slurry)].

For the SDS foam experiments 1 h of desulfurization was performed each day, the remaining 23 h the slurry was stored at 40–45°C. Experimental conditions are provided in Table 1.

Effects of protein foam on wet FGD operations

Protein, as a natural polymer, has been intensively studied in the literature.[20–22] Compared to SDS, protein is a weak foaming agent, but the effects on wet FGD performance can

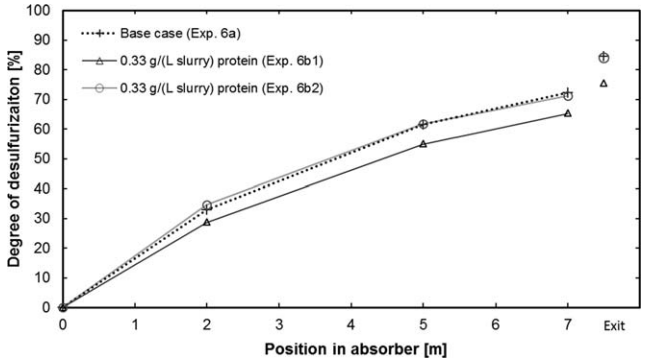

Figure 9. Influence of protein 0.33 g protein /(L slurry) on the desulfurization degree.

Experimental conditions are provided in Table 1. Data for Exp. 6b1 was measured after 2.5 h; Data for Exp. 6b2 was measured after 3.5 h.

still be important. Moreover, protein is a highly suspected compound to cause foaming operational problems in wet FGD plants, where electrolyte concentrations and pH value may affect its foaming behavior.[4,18] Because of its lower foaming potential, a high concentration of protein is selected [0.33 g/(L slurry)]. In Figure 9, the influence of protein foaming [0.33 g/(L slurry)] on the desulfurization degree is shown. The first hour after addition of protein, the total desulfurization degree decreases from 84 to 75%. Meanwhile the solids concentration decreased from 67 ± 0.2 to 54 ± 0.8 g/(L slurry). However, the foaming declines and the desulfurization degree returns to normal after approximately 2 h. The changes observed may be caused by protein precipitation or by the compaction of hydrophilic parts of the protein molecule causing weaker repulsion between surfaces and thereby a lower foam stability.[21] The pH value in the holding tank, shown in Figure 5, reveals that pH is only slightly affected by protein addition/foaming.

Antifoam effects on wet FGD operations

The effects of two commercial antifoams (Nalco FM-37 and Foamtrol 2290) were investigated. An initial screening test showed a concentration level of about 0.03 g antifoam/(L slurry) war sufficient to destroy the foam. This concentration was used throughout the investigations. The results of 0.03 g Nalco FM-37 antifoam/(L slurry), in the absence of a foaming agent, are shown in Figure 10. In the experiment,

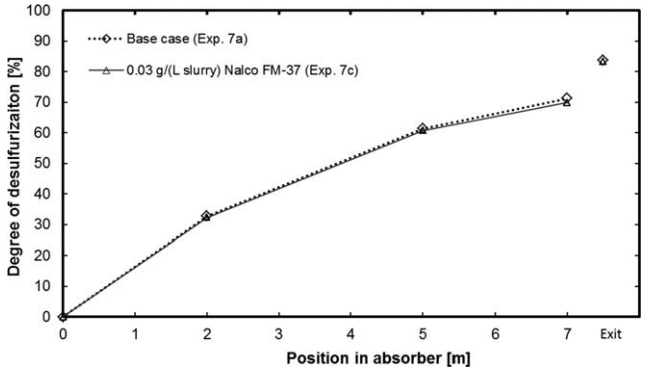

Figure 10. Effects of 0.03 g Nalco FM-37 antifoam /(L slurry) on the desulfurization degree.

Experimental conditions are provided in Table 1.

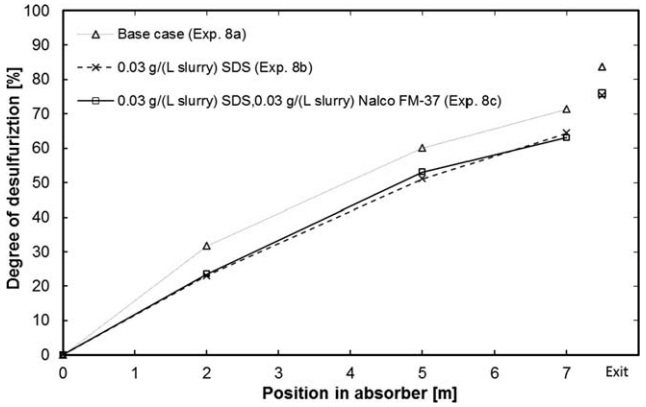

Figure 11. Effects of SDS [0.03 g/(L slurry)] and antifoam [0.03 g/(L slurry) Nalco FM-37] on the desulfurization degree.

The antifoam war added after 1.5 h foam case operation. Experimental conditions are provided in Table 1.

there is no influence on the desulfurization degree, but the solids concentration increases from 61 to 68 g/(L slurry), mainly due to desulfurization [estimated to form 5.5 g/(L slurry)].

When 0.03 g/(L slurry) antifoam (Nalco FM-37 or Foamtrol 2290) is added to a foaming SDS solution, shown in Figures 11 and 12, the foam immediately disappears, but individual scattered bubbles still appear. The solids concentration increases from 55 to 58 g/(L slurry), corresponding to the 3.4 g/(L slurry) estimated to be formed by the desulfurization. However, the desulfurization degree remains unchanged. In Exp. 8 [0.03 g Nalco FM-37/(L slurry)], the solids concentration at the holding tank slurry surface [176 g/(L slurry)] was three times higher than the solids concentration in the bulk holding tank slurry [58 ± 1.0 g/(L slurry)]. Moreover, the limestone concentration at the slurry surface [3.7 g/(L slurry)] was much higher than the limestone concentration in the bulk [1.4 ± 0.1 g/(L slurry)]. Although the antifoams used were able to destroy the foam immediately, they were unable to prevent the accumulation of small limestone particles at the slurry surface and thereby an associated lower bulk slurry limestone concentration and desulfurization degree. As seen in Figures 11 and 12, the two antifoams

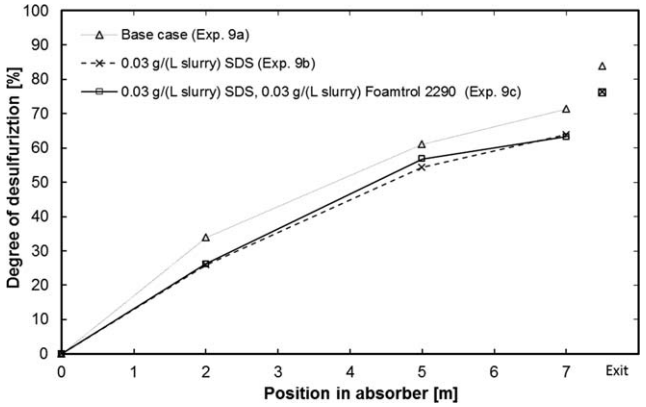

Figure 12. Effects of 0.03 g SDS/(L slurry) and 0.03 g Foamtrol 2290 antifoam/(L slurry) on the desulfurization degree.

The antifoam war added after 1 h foam case operation. Experimental conditions are provided in Table 1.

were equally effective with respect to the desulfurization degree.

Conclusions

The influence of two foaming agents on the desulfurization degree, absorber pH, solid particles, and limestone concentrations has been systemically investigated in a wet FGD pilot plant. SDS foaming is able to carry fine particles to the foam layer and decrease the limestone concentration in the slurry, thereby decreasing the desulfurization degree from 84.3 ± 1.0 to $74.7 \pm 2.5\%$, with a limited influence of foam height. Foaming induced by protein, lowers the desulfurization degree from 84 to 75%, but only for about 1–2 h because of its weak foaming ability. In the presence of protein foam, the solids concentration decreased. The addition of 0.03 g/(L slurry) of a commercial antifoam (Nalco FM-37 or Foamtrol 2290) eliminated the foam, but the desulfurization degree remained low because of small particles remaining on the top of the slurry. From a practical point of view, the results suggest that foam can directly lower the desulfurization degree and antifoams destroy the foam layer, but not necessarily help restoring the desulfurization degree.

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