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Design & Research Institute, China
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SCIENTIFIC PAPER

UDC 66.022.3:54:66.094.522.091.8

A FORMULATION ADDITIVE FOR SIMULTANEOUSLY IMPROVING FLUE GAS DESULFURIZATION EFFICIENCY AND GYPSUM QUALITY

Article Highlights

- The optimized formulation additive for desulfurization efficiency and gypsum quality were obtained
- The addition of $MnSO_4$ and acrylic acid were found to increase the desulfurization efficiency
- Citric acid contributed to the formation of short and rod-like gypsum crystals
- An desulfurization efficiency of 97.02% and a low gypsum moisture content of 4.32% were obtained

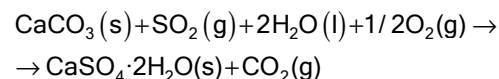
Abstract

The effects of different additives, including $MgSO_4$, $MnSO_4$, adipic acid, citric acid, benzoic acid, succinic acid and acrylic acid on SO_2 removal from a limestone-gypsum wet flue gas desulfurization (FGD) system and gypsum characteristics were investigated. An optimized formulation additive for simultaneously improving flue gas desulfurization efficiency and gypsum quality was found. This formulation additive was successfully applied in an industrial-scale FGD system of a local coal power plant (600 MW, Shanghai Wujing Power Plant, China), and the gypsum quality was improved. The optimized formulation additive was acrylic acid, $MnSO_4$ and citric acid mixed at a ratio of 1:1:2, which can simultaneously achieve an increased desulfurization efficiency of 97.02% and a low gypsum moisture content of 4.32%. Moreover, $MnSO_4$ and acrylic acid were found to be most effective for improving SO_2 removal, and citric acid contributed to gypsum particles with more uniform sizes and regular shapes, as well as good dewatering characteristics.

Keywords: additive, crystal structure, flue gas desulfurization, gypsum, moisture content.

Limestone-gypsum wet flue gas desulfurization (FGD) process is one of the most widely used technologies for SO_2 removal from exhaust flue gas of fossil-fuel power plants [1-3]. Acidic SO_2 can be effectively captured by the limestone slurry scrubbing solution to form gypsum ($CaSO_4 \cdot 2H_2O$), a by-product that can be refined for commercial applications in building construction. A moisture content in gypsum less than

10% is necessary for the consideration of transportation and commercial applications.



With China's increasingly stringent regulations limiting the emission of SO_2 , new emission standards were established since 2014. Improvement of the existing wet FGD process is of urgent necessity. Addition of additives into the limestone slurry is a simple and effective strategy. Inorganic additives like $MgSO_4$ and $MnSO_4$ can effectively improve the SO_2 removal efficiency in wet FGD systems [4,5]. A number of organic acids with buffering capacity including formic acid, adipic acid, maleic acid, acetic acid, acrylic acid, succinic acid and benzoic acid were

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Paper received: 7 July, 2020

Paper revised: 4 May, 2020

Paper accepted: 15 November, 2020

<https://doi.org/10.2298/CICEQ110707042L>

found to enhance the desulfurization process [3,6,7]. However, the application of these additives could undermine the gypsum quality, leading to deteriorated crystal structures and subsequently high moisture contents. For example, acrylic acid has been reported to change the crystal morphology and inhibit calcium sulfate crystallization [8]. Morphology of α -calcium sulfate hemihydrates can be altered by succinic acid in salt solutions [9]. Maleic acid can hinder calcium sulfate formation at high temperature levels [10]. Filtration characteristics of gypsum like particle size distribution (PSD) and crystal structure are important for economic considerations [11–14], since these characteristics are closely related to energy consumption during the filtration process and moisture content of the final gypsum [15]. In this regard, the formation of large and uniform size particles is desired [16]. Therefore, to guarantee good gypsum quality, and to reduce the cost for separation, it is necessary to improve the crystal structure of gypsum.

Recently, citric acid has been reported to trigger a remarkable expansion of metastable lifetime for α -calcium sulfate hemihydrate [17]. Titiz-Sargut *et al.* [15,18] have investigated the influence of citric acid on the morphology of gypsum produced through the reaction between $\text{Ca}(\text{OH})_2$ slurry and H_2SO_4 solution, and they obtained enlarged gypsum crystals with uniform sizes in the presence of 2500 mg L^{-1} citric acid. Rashad *et al.* reported that the presence of citric acid can decrease the crystallinity of gypsum in the process of phosphate acid production [19]. Lanzón and Garcia-Ruiz observed changes in gypsum microstructures when citric acid was used, and they indicated the effectiveness of citric acid in retarding the setting time of gypsum plasters even at a low dosage [20].

Although numerous studies have been conducted on the effect of different additives, most of them focused either on enhancement of SO_2 removal in wet FGD systems, or on the reactive crystallization of pure $\text{Ca}(\text{OH})_2$ and H_2SO_4 solutions for commercial production of gypsum. Little information could be found in the literature regarding crystallization of gypsum as valuable by-product from wet FGD process.

The aim of this study was to investigate the effect of different desulfurization additives on wet FGD performance, and to find out an optimized formulation additive to simultaneously achieve increased SO_2 removal efficiency and desirable gypsum quality.

The effect of single desulfurization additives on wet FGD performance was reported in our previous papers [21,22]. An optimized formulation additive was reported in this paper.

Moisture content, PSD and crystal morphology of the formed gypsum obtained under different scenarios were determined. And the optimized formulation additive obtained from the present study was tested in an industrial-scale wet FGD unit of a power plant.

MATERIALS AND METHODS

Chemicals and materials

N_2 (purity >99.9%), O_2 (purity >99.9%), and SO_2 (purity >99.99%) used in the study were obtained from Shanghai Shenkai Gases Technology Co. Ltd. (China). Limestone supplied by Shanghai Oriental Enviro-Industry Co. Ltd. (China) was grounded to 325 meshes ($44 \mu\text{m}$) before used. Compositions of the limestone were as follows (wt.%): CaCO_3 (95.72%), MgCO_3 (1.70%), SiO_2 (1.68%), Al_2O_3 (0.35%), Fe_2O_3 (0.22%). Other chemicals used were of analytical grade (Sinopharm Chemical Reagent Co. Ltd., Shanghai, China). All solutions were prepared using deionized water.

Experimental setup and procedure

A bubbling reactor is often used in a laboratory to simulate the limestone-gypsum wet FGD process [7,19,23,24]. The experimental setup is illustrated in Figure 1. In the present study, experiments were carried out in an enclosed cylindrical glass reactor (3) with a working volume of 2 L (16.2 cm in diameter, 10.1 cm in height). The reactor was equipped with a thermostatic jacket (4), a mechanical stirring device (5) and a gas inlet (6). Artificial flue gas, which consisted of N_2 , O_2 and $1500 \text{ mg Nm}^{-3} \text{ SO}_2$, was purged into the reactor at a flow rate of $1.05 \text{ N m}^3 \text{ h}^{-1}$. The volume ratio of $\text{N}_2:\text{O}_2$ was adjusted to 94:6 because in actual scenario flue gas usually has a O_2 content of 6%. CaCO_3 slurry was circulated between the bubbling reactor (3) and the slurry circulating tank (7) with a peristaltic pump (9) operated at a flow rate of 270 mL min^{-1} . An air pump was used for oxygen supplement in (7). Reaction temperature in the bubbling reactor was maintained constant at 323 K using a bypass thermostatic water bath (8).

MgSO_4 , MnSO_4 , adipic acid, citric acid, benzoic acid, succinic acid and acrylic acid were investigated in this study. At the beginning of each experiment, 20 g limestone was added into the reactor along with 980 mL deionized water. In the meantime, the desulfurization additive was added into the slurry. Control group labeled as blank was also conducted without any additive. During the bubbling experiment, the off-gas composition and slurry pH were monitored consistently. Initial pH of the prepared slurries ranged from

6.91 to 9.81 depending on the additives employed, and the slurry pH decreased gradually with reaction time. To better compare the effect of different additives on gas desulfurization, all SO₂ removal efficiencies reported in this work were determined at the same pH point of 5.5±0.1, which is the commonly used pH value for industrial scale wet FGD process. When the slurry pH dropped to 4.6±0.1, the bubbling reaction was stopped, and the final slurry obtained was purged with air for 24 h to ensure complete oxidation of SO₃²⁻ to SO₄²⁻ before slurry sample characterization.

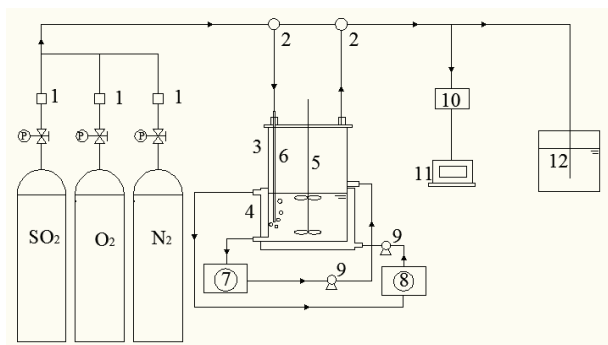


Figure 1. Sketch of bubbling experimental setup used to simulate wet FGD. Arrows show gas and liquid flow directions; 1-rotameter, 2-valve, 3-bubbling reactor, 4-thermostatic jacket, 5-mechanical stirring device, 6-gas inlet, 7-slurry circulating tank, 8-thermostatic water bath, 9-peristaltic pump, 10-flue gas analyzer, 11-computer, 12-NaOH solution.

The SO₂ removal efficiency was calculated according to the following formula:

$$\text{SO}_2 \text{ removal efficiency} = (\text{Inlet concentration of SO}_2 - \text{Outlet concentration of SO}_2) / \text{Inlet concentration of SO}_2$$

Analytical methods

Components of the limestone were determined by an XRF-1800 (Shimadzu, Japan). Gas compositions were analyzed with a Testo 350 gas analyzer, and solution pH was monitored with a BANTE 901b pH meter. The produced gypsums were analyzed for moisture content, particle size distribution (PSD), and crystal morphology. Moisture content was analyzed with the help of a vacuum pump. Specifically, 100 mL of slurry sample was filtered by suction at 0.1 MPa for 10 min and dried at 313 K for 24 h. And moisture content was calculated as the lost weight. The dried samples were stored in a dryer at room temperature before PSD and crystal morphology analysis. Analysis of PSD was conducted by using a LS-POP (VI) laser particle size analyzer, crystal morphology and

structure was observed with a Quanta 250 scanning electron microscope (SEM).

RESULTS AND DISCUSSIONS

Effect of single desulfurization additive

In this section, different additives were added respectively into the limestone slurry at a dosage of 4 mmol L⁻¹. SO₂ removal efficiency of the FGD system and moisture content of the produced gypsum are shown in Figure 2a. Most additives were found to improve the performance of SO₂ removal to different extents except for citric acid. A SO₂ removal efficiency of 90.49% was observed from the blank. Among the 7 additives investigated, acrylic acid and MnSO₄ contributed to the highest desulfurization efficiencies of 97.55 and 96.82%, respectively, followed by adipic acid (95.30%), succinic acid (93.89%), benzoic acid (93.52%), and MgSO₄ (93.29%). Mn²⁺ has been reported to promote the oxidation of sulfite in limestone slurry, and thus the removal efficiency of SO₂ [5]. For MgSO₄ additive, the magnesium sulfite ion pair could facilitate the mass transfer in the slurry and subsequently increased the scrubbing efficiency [4]. Organic acids could prevent fast pH drop caused by SO₂ absorption, facilitating the transport of H⁺ between the gas-liquid and liquid-solid phase [4]. On the other hand, citric acid was found to lower the SO₂ removal efficiency to 88.20%, which might be attributed to the formation of insoluble calcium citrate at pH>5, lowering the concentration of Ca²⁺ in the slurry and consequently decreasing desulfurization performance. Nevertheless, the presence of citric acid significantly decreased the moisture content of gypsum to 1.33%, in comparison to that of 9.76% in the blank. In contrast, MnSO₄ and benzoic acid led to an undesirable high gypsum moisture content of 18.82 and 48.57%, respectively.

Gypsum is a monoclinic crystal. The gypsum crystal is interconnected by SO₄²⁻ tetrahedron and Ca²⁺ to form a double-layer structure. The two-layer structure is connected by H₂O. H₂O are completely cleaved on the (010) crystal plane. Therefore, the gypsum crystal is a plate-like crystal completely parallel to the (010) crystal plane

As indicated in Figure 2b, the addition of both MnSO₄ and acrylic acid led to a relatively wider range of particle size distribution and the formation of many microparticles with diameters smaller than 10 μm, whereas citric acid contributed to the formation of gypsum crystals with diameters mainly within a narrow range of 10–80 μm. Mn²⁺ will be adsorbed on the (110) crystal plane of gypsum crystal. The gypsum

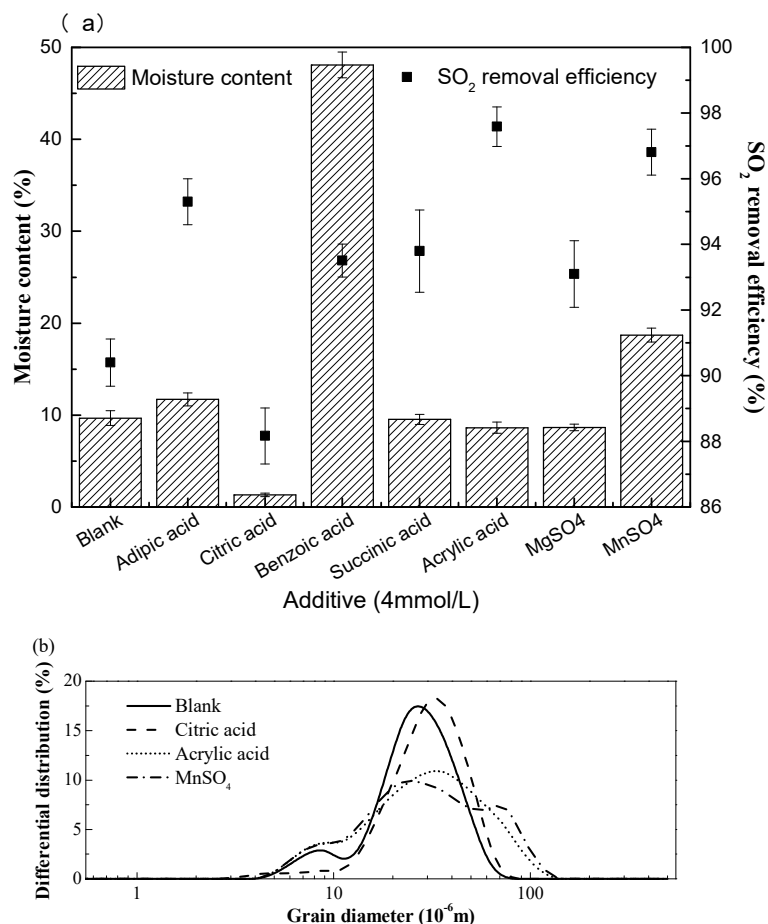


Figure 2. Desulfurization efficiency and gypsum moisture content with different additives (a) and PSD of the produced gypsum crystals (b). Additive concentration = 4 mmol L⁻¹.

crystal growth of *a*-axis was inhibited, and the gypsum crystal was partly a needle-like shape. As a result, the particle sizes of gypsum were more dispersed. S. Figure 1 (Supplementary material, available from the author upon request) displays SEM images of the gypsum samples obtained in the presence of MnSO₄, acrylic acid and citric acid. As indicated, the presence of MnSO₄ and acrylic acid resulted in the formation of a larger amount of elongated, needle-shaped gypsum crystals. Mn²⁺ has been reported to catalyze the oxidation of SO₂ and change the gypsum crystal morphology from a compact plate-like shape to a partly needle-like shape [24,25]. Al-Hamzah *et al.* have indicated the inhibitory effect of poly(acrylic acid) on CaSO₄ crystallization by preferentially attaching to the edges between highly charged and near-neutral faces, controlling the growth of crystallites [8]. Gypsum crystals with irregular shapes and nonuniform sizes will increase the probability of blocking the passage of free water between the gypsum crystals, which often lead to a high moisture content and subsequently high visco-

sity, which is likely to cause equipment blocking. In addition, the bulky volume related with a high moisture content also significantly increases the cost for transportation.

In contrast to MnSO₄ and acrylic acid, the presence of citric acid led to the formation of short and rod-like crystals. This phenomenon could be explained by the existence of citrate, which could be selectively adsorbed onto the (111) crystal plane of the gypsum crystals, and react with Ca²⁺ to form calcium citrates, inhibiting crystal growth on the (110) and (111) crystal planes [14]. The gypsum crystal growth of *c*-axis was inhibited.

Effect of mixed additives

MnSO₄ and acrylic acid exhibited excellent performance in promoting SO₂ removal. Nevertheless, these two additives exerted unfavorable influence on gypsum characteristics. On the other hand, despite the negative impact of citric acid on SO₂ removal, it is beneficial for improving the crystal structure and dewatering properties of gypsum. To simultaneously achieve improved SO₂ removal performance and

compact gypsum crystals with low moisture content, citric acid was mixed respectively with MnSO_4 and acrylic acid at different ratios and applied in the wet FGD system.

Mixture of MnSO_4 and citric acid

MnSO_4 and citric acid were mixed at different mole ratios, and then added to the FGD slurry with a total concentration of 4 mmol L^{-1} . As shown in Figure 3a, the desulfurization efficiency increased with the increase in MnSO_4 proportion, attaining a maximum of 97.18% at a MnSO_4 -to-citric acid ratio of 2:2. Further increasing the proportion of MnSO_4 did not significantly improve the removal efficiency of SO_2 . On the other hand, moisture content of the produced gypsum also increased with the increase in MnSO_4 proportion, but still a relatively low moisture content of 8.66% was obtained when the ratio of MnSO_4 -to-citric acid was 2:2. As shown in Figure 3b, increasing the proportion of citric acid efficiently reduced the amount of gypsum crystals smaller than $10 \mu\text{m}$. But at a ratio

of 2:2, there were still many crystals with diameters smaller than $10 \mu\text{m}$. This phenomenon showed that the inhibition of Mn^{2+} on a-axis gypsum crystal growth was more powerful than citric acid on c-axis. For practical considerations, further improving the dewaterability of gypsum crystals is necessary. Therefore, the kind of additive and the ratio should be adjusted.

Mixture of acrylic acid and citric acid

In this section, acrylic acid was mixed with citric acid and added into the FGD slurry. As indicated in Figure 4a, the desulfurization efficiency dropped from 97.55 to 88.2% with the increase in citric acid proportion, whereas the resulted moisture content in gypsum decreased from 8.68 to 1.33%. The particle size distribution results displayed in Figure 4b were consistent with the moisture content of gypsum crystals obtained. As shown, the increase in citric acid proportion contributed to the formation of larger and more uniformly distributed crystals.

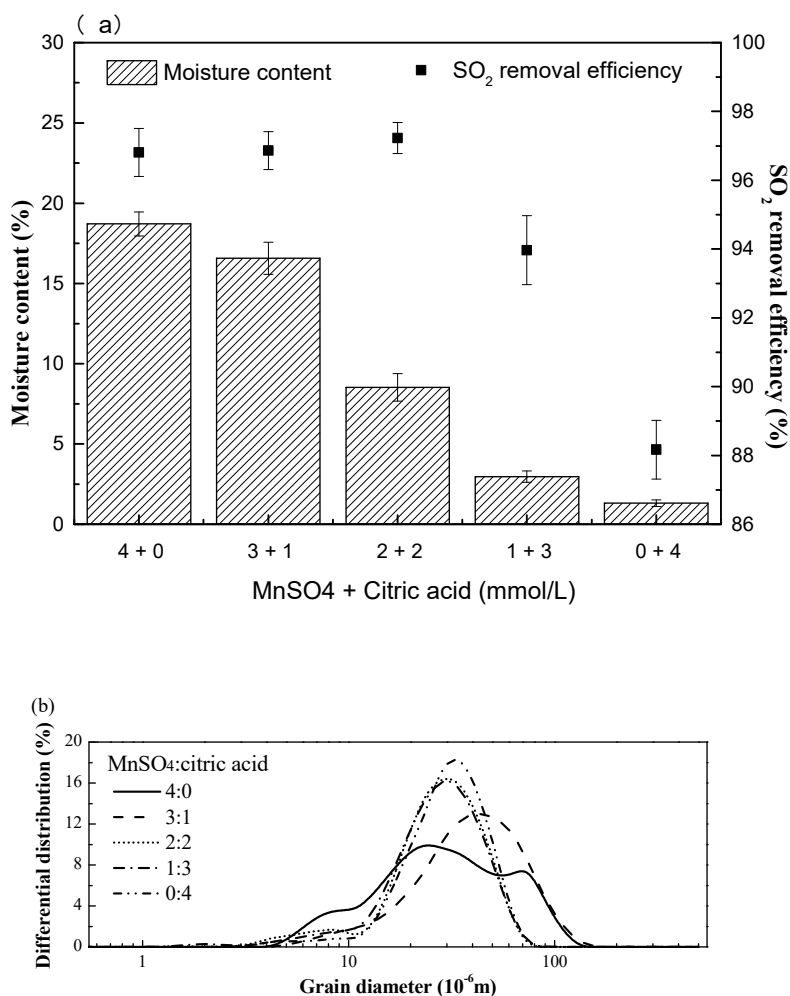


Figure 3. Desulfurization efficiency and gypsum moisture content (a) and PSD of the produced gypsum crystals (b) with the addition of MnSO_4 and citric acid mixed at different ratios. Total additive concentration = 4 mmol L^{-1} .

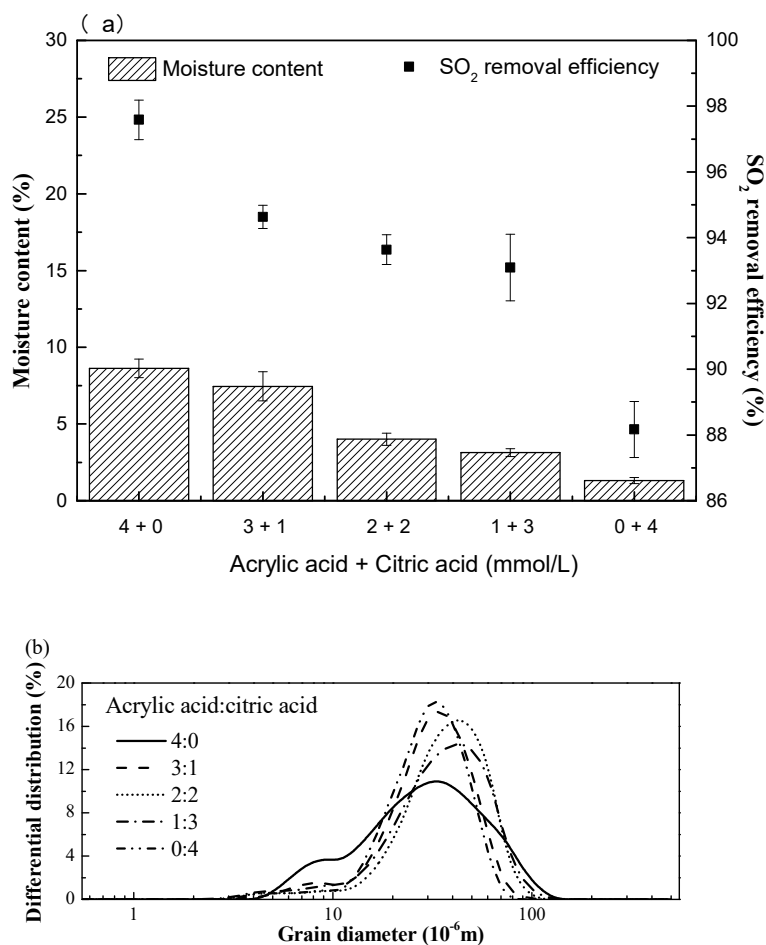


Figure 4. Desulphurization efficiency and gypsum moisture content (a) and PSD of the produced gypsum crystals (b) with the addition of acrylic acid and citric acid mixed at different ratios.

Apparently, neither the mixture of MnSO_4 and citric acid nor the mixture of acrylic acid and citric acid could simultaneously achieve desirable desulfurization efficiency and satisfactory gypsum characteristics. Therefore, in the following experiment, the mixture of acrylic acid, MnSO_4 and citric acid was investigated, with a total additive concentration maintained constant at 4 mmol L^{-1} . The results of SO_2 removal and moisture content at different mix ratios are shown Figure 5a. As shown, high desulfurization efficiencies in the range of 97–98% were obtained at all ratios studied. But the moisture content of gypsum differed significantly, which was detected to be 9.44% at an acrylic acid: MnSO_4 :citric acid ratio of 2:1:1, 14.67% at 1:2:1, and 4.32% at 1:1:2. The increase in citric acid proportion contributed to larger grain diameters of the formed crystals (Figure 5b). Mn^{2+} was adsorbed on the (110) crystal plane of gypsum crystal and citric acid was adsorbed on the (111) crystal plane. It was possible to make gypsum crystals into hexagonal short columnar crystals. When the gypsum crystals became hexagonal short columnar, the passage of

free water between the gypsum crystals was short and unblocked.

Figure 6 displays the SEM images of the gypsum samples. As displayed, thick plate-like crystals were observed in the case of 1:2:1 (Figure 6b), whereas the crystals were column shape for both the cases of 2:1:1 (Figure 6a) and 1:1:2 (Figure 6c). It is noticeable that the crystals in Figure 6c were thicker and shorter than those in Figure 6a. From the point of view of gypsum quality (mainly particle size and moisture content related with filtration properties), the optimized formulation additive was determined to be the mixture of acrylic acid, MnSO_4 and citric acid at a ratio of 1:1:2. In the meantime, the desulfurization efficiency was improved to 97.02%, compared with that of 90.49% obtained from the blank without any additive.

Industrial applications

The optimized formulation additive obtained from lab experiments (acrylic acid, MnSO_4 and citric acid mixed at a ratio of 1:1:2) was applied in an industrial scale wet FGD system of a local coal-power

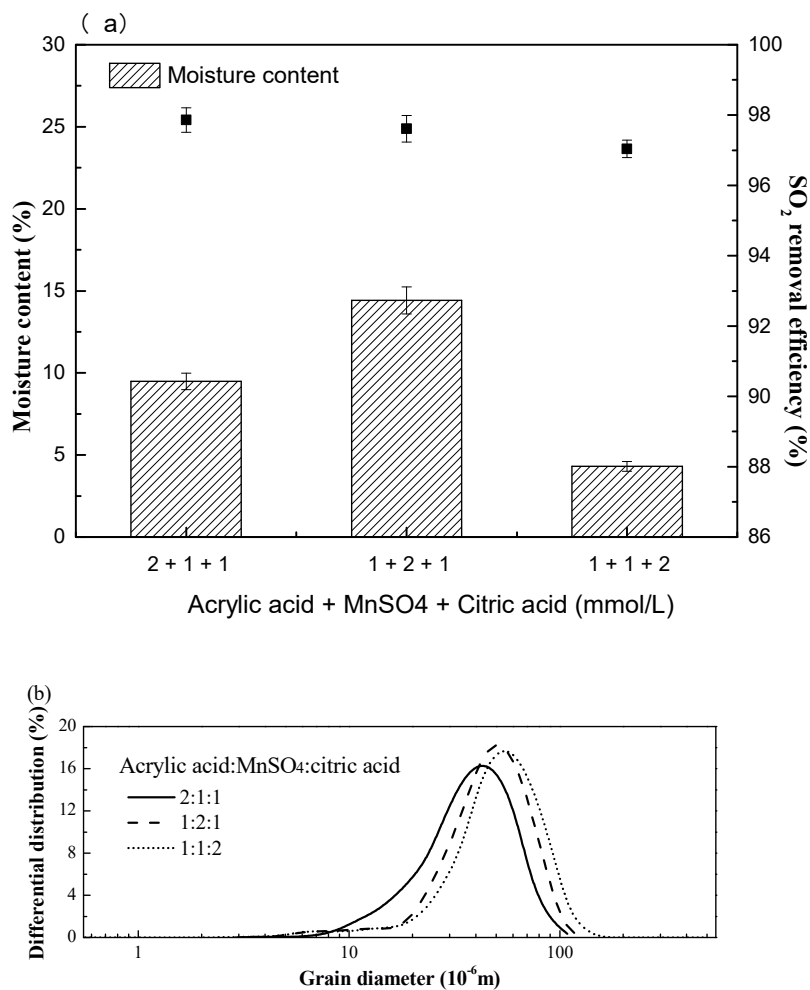


Figure 5. Desulphurization efficiency and gypsum moisture content (a) and PSD of the produced gypsum crystals (b) with the addition of acrylic acid, MnSO_4 and citric acid mixed at different ratios.

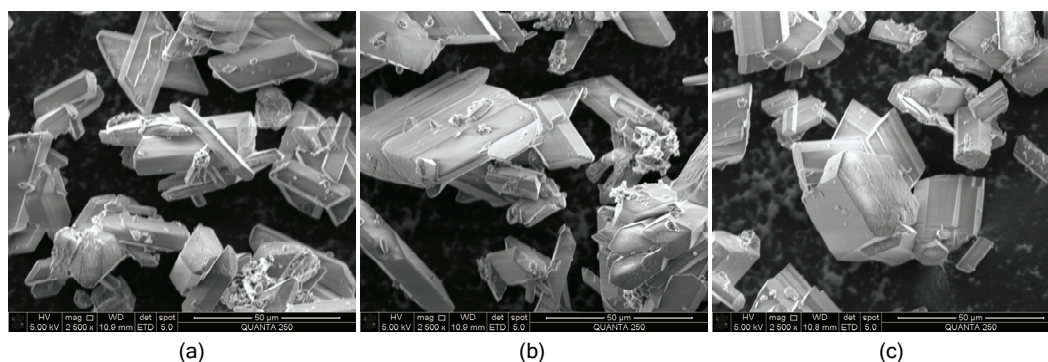


Figure 6. SEM images of gypsum crystals with the addition of acrylic acid, MnSO_4 and citric acid mixed at different ratios, a) 2:1:1, b) 1:2:1, c) 1:1:2.

plant (600 MW, Shanghai Wujing Power Plant, China). Because of the application of polyacrylamide (PAM) as flocculation agent in cooling water system of this plant, the enriched PAM in FGD slurry posed negative effects on the gypsum quality, resulting in the formation of small and sheet shape crystals with a high moisture content ($\geq 20\%$). Figure 7 indicated the

effects of formulation additive on gypsum crystal structure. As shown, one day after the application of formulation additive, the crystals became thicker and plate-like, and the gypsum moisture content decreased significantly to 11.35%. Three days later, short and column shape crystals were obtained, along with a low gypsum moisture content of 3.47% being achieved.

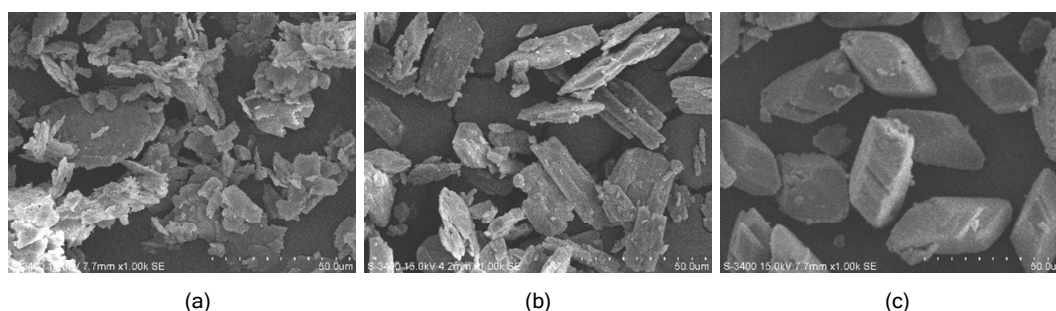


Figure 7. SEM images of gypsum crystals from an industrial scale wet FGD system after the addition of acrylic acid, $MnSO_4$ and citric acid mixed at a ratio of 1:2:1 on: a) day 0, b) day 1 and c) day 3. Total additive concentration = 4 mmol L^{-1} .

CONCLUSIONS

In this study, different additives were investigated in a lab-scale wet FGD reactor, and the conclusions can be drawn as follows:

1) The addition of $MnSO_4$ and acrylic acid were found to increase the desulfurization efficiency by 7.00 and 7.80%, respectively. But $MnSO_4$ gave rise to an undesirably high moisture content of 18.82% in the formed gypsum.

2) Despite the negative effect of citric acid on SO_2 removal, it contributed to the formation of short and rod-like gypsum crystals with a low moisture content.

3) Mixture of acrylic acid, $MnSO_4$ and citric acid at a ratio of 1:1:2 could simultaneously achieve an increased desulfurization efficiency of 97.02% and a desirably low gypsum moisture content of 4.32%. This formulation additive provides an effective and economical alternative to tackle the technical problems related with low SO_2 removal efficiency and poor gypsum quality in wet FGD systems.

Acknowledgement

This work is financially supported by the Key Laboratory of Coal Gasification and Energy Chemical Engineering of Ministry of Education, East China University of Science and Technology and Shanghai Oriental Enviro-Industry Co., Ltd.

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NAUČNI RAD

ADITIV ZA ISTOVREMENO POBOLJŠANJE EFIKASNOSTI DESULFURIZACIJE DIMNOG GASA I KVALITETA GIPSA

Istraženi su efekti različitih aditiva, uključujući $MgSO_4$, $MnSO_4$, adipinsku kiselinu, limunsku kiselinu, benzoevu kiselinu, jantarnu kiselinu i akrilnu kiselinu na uklanjanje SO_2 iz vlažnih sistema krečnjak-gips za odsumporavanja dimnih gasova i karakteristike gipsa. Otkriven je optimizovani aditiv za istovremeno poboljšanje efikasnosti odsumporavanja dimnih gasova i kvaliteta gipsa. Ovaj aditiv uspešno je primenjen u industrijskom sistemu za odsumporavanja u lokalnoj elektrani na uglj (600 MV, Šangajska elektrana Vujiing, Kina), a kvalitet gipsa je poboljšan. Optimizovani aditiv je bio smeša akrilne kiseline, $MnSO_4$ i limunske kiseline u odnosu 1:1:2, kojim se istovremeno može postići povećana efikasnost odsumporavanja od 97,02% i mali sadržaj vlage u gipsu od 4,32%. Štaviše, utvrđeno je da su $MnSO_4$ i akrilna kiselina najefikasniji za poboljšanje uklanjanja SO_2 , dok je limunska kiselina doprinela da se dobiju čestice gipsa ujednačenijih veličina i pravilnog oblika, kao i dobrim karakteristikama uklanjanja vode.

Ključne reči: aditiv, kristalna struktura, odsumporavanje dimnih gasova, gips, sadržaj vlage.