



Experimental and Modeling Studies of SO₃ Homogeneous Formation in the Post-Flame Region

Haiping Xiao¹, Cong Qi^{1*}, Qiyong Cheng¹, Chaozong Dou¹, Xiang Ning^{1,2}, Yu Ru¹

¹ Key Laboratory of Condition Monitoring and Control for Power Plant Equipment Ministry of Education, North China Electric Power University, Beijing 102206, China

² Datang Environment Industry Group Co., Ltd., Beijing 100097, China

ABSTRACT

SO₃ exists in the atmosphere in the form of sulfuric acid aerosol, heavily polluting the environment and decreasing the safety of boiler equipment. This study focuses on the homogeneous formation mechanism of SO₃ in the post-flame region. We conducted experiments and simulations to investigate the influence of various combustion parameters on SO₃ generation. The formation of SO₃ was affected by factors such as temperature and the concentrations of O₂, SO₂, NO, and H₂O. With a rise in temperature, the SO₃ concentration initially increased but then decreased, reaching its maximum at about 1000°C, which indicates that SO₂ can promote the formation of SO₃ over a certain temperature range. An increase in the O₂ concentration promoted the formation of O and OH radicals, which enhanced the generation of SO₃ from SO₂. The presence of NO resulted in direct and indirect interactions between NO_x and SO_x species for different reaction sets, potentially enhancing SO₃ generation. With an increase in the H₂O concentration, SO₃ formation initially increased rapidly before plateauing. ROP (rate of production) and sensitivity analyses suggest that adding H₂O will produce O and OH, which strongly influence SO₃ formation. Furthermore, the sensitivity analysis indicated that radicals and the direct reaction between SO₂ and NO₂ play significant roles in SO₃ formation.

Keywords: Sulfuric acid aerosol; Chemical kinetics; Mechanism analysis; ROP analysis; Sensitivity analysis.

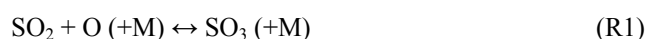
INTRODUCTION

SO₂ is the atmospheric pollutant emitted from the combustion of fossil fuel. The main source of SO₂ is the combustion of coal (Kato *et al.*, 2016). A small amount of SO₂ will subsequently be oxidized to SO₃. SO₃ causes serious damage to the equipment and environment of the boiler tail. Kagawa (2014) deemed that the growth of coal combustion has led to greater amounts of S emitted to the atmosphere. At temperatures above 200°C, as long as there is about 8% moisture in the flue gas, 99% of SO₃ will be converted into sulfuric acid vapor. SO₃ has always been in the atmosphere in the form of sulfuric acid aerosol (the diameter is generally 0.4–1.2 μm) (Hardman *et al.*, 1988). When the flue gas temperature is lower than the acid dew point, low temperature corrosion may occur on the air preheater and low temperature economizer. SO₃ can react with NH₃, generating NH₄HSO₄ which will obstruct the air preheater. Furthermore, SO₃ also reduces the rate of

mercury removal from flue gas (Moser, 2007).

Mist particles are emitted into the atmosphere, which gives smoke its blue color, and the minimum concentration of SO₃ causing this phenomenon is related to atmospheric conditions. In general, if the content of SO₃ in flue gas is above 10 ppm, smoke opacity will be significant. When the SO₃ concentration is 5 ppm, plume opacity can be as high as 20%. When the SO₃ concentration is more than 10 ppm, smoke will be visibly blue, increasing the H₂SO₄ aerosol concentration near the power plant (Srivastava *et al.*, 2004). A U.S. EPA report showed that SO₃ and H₂SO₄ aerosols may be associated with a series of adverse health effects, including respiratory tract irritation and difficulty breathing (Walsh *et al.*, 2006).

The concentration of SO₃ in flue gas is related to SO₂ concentration, the excess air coefficient, flue gas temperature, fly ash composition, and other factors (Lou, 2008). The conversion rate of SO₂ to SO₃ is about 0–2%. Kio (2001) determined that oxygen atoms in the high temperature combustion zone of a furnace react with SO₂ to form SO₃:



The formation of SO₃ was shown by Fleig *et al.* (2009) to be the primary SO₃ formation pathway at 1000–1400°C:

* Corresponding author.

Tel.: 18810789291

E-mail address: ncepuqc@163.com



At 700–1000°C, SO₃ formation occurs via HOSO₂:



It has been confirmed that (R2) and (R3) are insignificant above 1000°C, because of the low stability of HOSO₂ (Alzueta *et al.*, 2001). According to Han (2015), SO₃ formation is mainly controlled by thermodynamic equilibrium at high temperatures, and by reaction kinetics (Monckert *et al.*, 2008) at low temperatures.

During combustion, the S element in the fuel is partially converted to SO₂, and if oxygen is excessive, some SO₂ will be further oxidized to SO₃ (Lee *et al.*, 1967). Previous studies have shown that the primary condition for SO₃ production is excess O₂. If O₂ concentrations are below 3%, SO₃ production is sensitive to the O₂ concentration. But above this concentration, an increase in O₂ concentration has little effect on the formation of SO₃. Combustion and flue gas cooling cover a large range of temperatures. At high temperatures (above 1300°C), formation of SO₃ can reach equilibrium within 2 s, while at low temperatures (i.e., 300–700°C), equilibration may take days to years. Temperature directly affects homogeneous SO₃ formation by changing the reaction rate and the equilibrium point of the reaction. The oxidation rate declines with the increase of temperature. It can be concluded SO₃ is mainly formed at intermediate temperatures of about 1000°C, given the residence time of flue gas in each temperature section. Glarborg *et al.* (1996) studied the reaction characteristics of SO₂ and found that peak SO₃ formation occurred around 950°C. On the other hand, Bayless *et al.* (2000) found that at low temperatures if free radicals (O, H, etc.) are abundant, a certain amount of SO₃ can also be obtained to maintain the

performance of ESP. In addition, Merryman (1979) studied staged combustion, assuming that temperature and O atom concentration were the main factors affecting formation of SO₃, and suggested that temperature affects oxidation of SO₂ by changing the concentration of O atoms. Therefore, temperature not only affects SO₃ formation by changing the reaction equilibrium (the rate of SO₃ decomposition and formation), but also via important indirect effects.

Currently, most research is focused on the generation mechanism of SO₂ and NO_x, ignoring SO₃ generation. In this paper, homogeneous formation of SO₃ and its influencing factors were studied by experiments and simulations, and the homogeneous formation mechanism of SO₃ in post-flame region was clarified.

EXPERIMENTAL AND MODELING

Introduction of Experimental Equipment

The experimental device is shown in Fig. 1. The setup consists of three major sections: burner, reactor, and sample collector. The inlet gas composition is SO₂, N₂, O₂, CO₂, NO and H₂O. Gas is mixed into flue gas analyzer (testo 350). After the gas content is measured, the first three-way valve is adjusted to make N₂ and CO₂ enter the ultrasonic atomizer to carry H₂O. Then adjust the second three-way valve to make all gases enter the reactor, which is heated by a tubular resistance furnace (SK-2.5-13TS). Exhaust gas then passes through a serpentine condenser tube. Using a digital display thermostatic water bath (HH-1), the condenser temperature is maintained at 80°C. The SO₃ concentration is measured by the U.S. EPA method 8A. The ideal one-dimensional flow reactor model is adopted, with a residence time of 4 s and a pressure of 1 atm.

The quartz tube reactor is 1065 mm long, with an outer diameter of 28 mm and an inner diameter of 24.4 mm. The total flow of the reaction gas is 3 L min⁻¹, and the reaction gas composition is shown in Table 1.

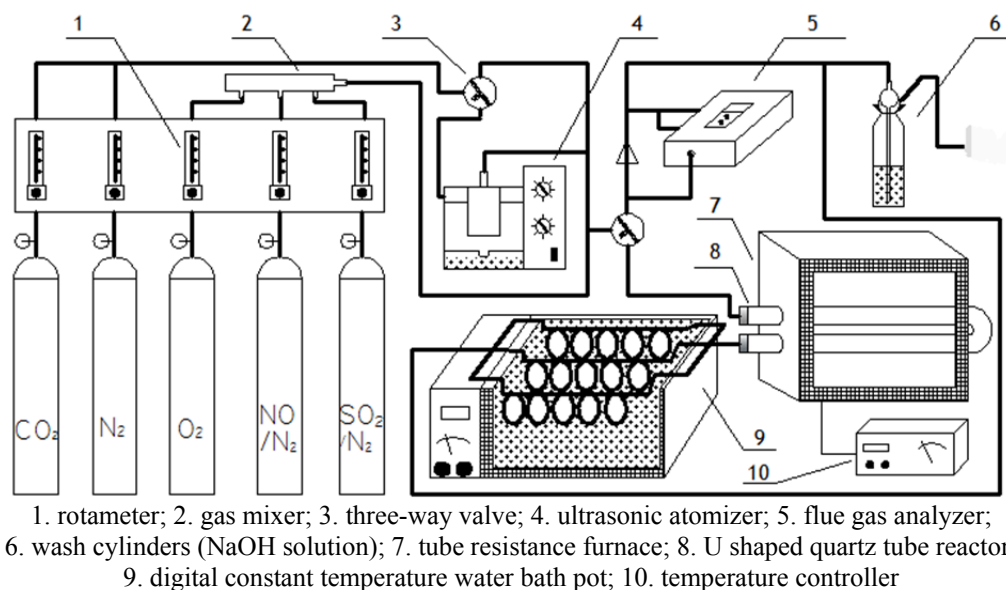


Fig. 1. Schematic diagram of experimental apparatus.

Simulation Methods and Models

A detailed gas-phase kinetic model was utilized to discuss the measured SO₃ concentrations. The calculations were carried out by CHEMKIN-IV software. This combined model considers C, H, O, N, and S, include 694 reactions. But heterogeneous chemistry is not included in the mechanism. And the interactions between sulfur and chlorine are not included. The mechanism includes the oxidation of hydrocarbons based on the work of Glarborg *et al.* (1998, 2003) and Alzueta *et al.* (2008). The nitrogen chemistry and the interactions between hydrocarbons and nitrogen species are based on the work of Glarborg *et al.* (1998) and Dagaut *et al.* (2008). The sulfur chemistry in the mechanism is described in the work by Alzueta *et al.* (2001) and Giménez-López *et al.* (2011). Moreover, the reactions of sulfur-containing elements are more comprehensive in this model than previously. And the direct interactions between SO_x and NO_x species were considered in this model.

RESULTS AND DISCUSSION

Gas-phase coal combustion experiments were performed to obtain speciation data of SO_x under a variety of operating conditions. Sampling temperature ranged from 400°C to 1300°C within the reactor. Other factors included O₂, SO₂, NO, and H₂O concentrations.

Effect of Temperature on SO₃ Formation

The experimental conditions were 550 ppm NO, 2000 ppm SO₂, 2.22% H₂O, and 5% O₂ (Fig. 2).

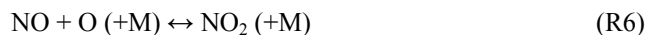
As shown in Fig. 2, the experimental results and simulated data indicated that the concentration of SO₃ first increased, but then decreased with increasing temperature. The maximum SO₃ concentration was around 1000°C. At 400–1000°C, the concentration of SO₃ increases with an increase in temperature, and the concentration of SO₃ decreased with an increase in temperature when the

temperature is higher than 1100°C. When the temperature was below 700°C, the SO₃ concentration was very near or below the limit of detection. Wang (2015) also observed a similar trend in SO₃ concentration with temperature. This model is modified to ensure that the modeling results in the middle temperature region (800–1200°C) are consistent with the experimental results, but the discrepancy in the low temperature region (400–700°C) cannot be completely eliminated.

Through ROP analysis, the main reactions of SO₃ were (R1)–(R4).



When the resistance furnace wall temperature is between 600°C and 800°C, the base reaction for SO₃ formation is (R4). The wall temperature of the resistance furnace increases the concentration of activated molecules in the reaction gas, and decomposition of H and OH with H₂O increases. According to the literature (Mantashyana, 2014), O radicals are mainly produced by (R5) and decomposition of O₂. The concentration of H radical increased in this temperature range, which promoted the formation of O radical. The concentration of NO₂ also was increased by (R6), and the rate of formation of (R4) increased as well. Higher concentrations of NO₂ result in (R4) playing a decisive role in the generation of SO₃.



As temperature increases, the total formation rate of SO₃ increases rapidly. Fleig (2013) measured the formation of SO₃ by introducing different gas components into a one dimensional furnace. Results showed that NO promoted SO₃ formation at low temperatures.

Table 1. Gas composition in experiments.

Test	Inlet gas composition					
	N ₂ (%)	CO ₂ (%)	SO ₂ (ppmv)	NO (ppmv)	O ₂ (%)	H ₂ O (%)
1	77.525	15	2000	550	5	2.22
2	81.525	15	2000	550	1	2.22
3	79.525	15	2000	550	3	2.22
4	75.525	15	2000	550	7	2.22
5	73.525	15	2000	550	9	2.22
6	77.675	15	500	550	5	2.22
7	77.625	15	1000	550	5	2.22
8	77.575	15	1500	550	5	2.22
9	77.475	15	2500	550	5	2.22
10	77.425	15	3000	550	5	2.22
11	77.48	15	2000	0	5	2.22
12	77.4546	15	2000	254	5	2.22
13	77.3814	15	2000	986	5	2.22
14	79.645	15	2000	550	5	0
15	67.445	15	2000	550	5	12.2
16	61.545	15	2000	550	5	18.1
17	46.445	15	2000	550	5	33.2

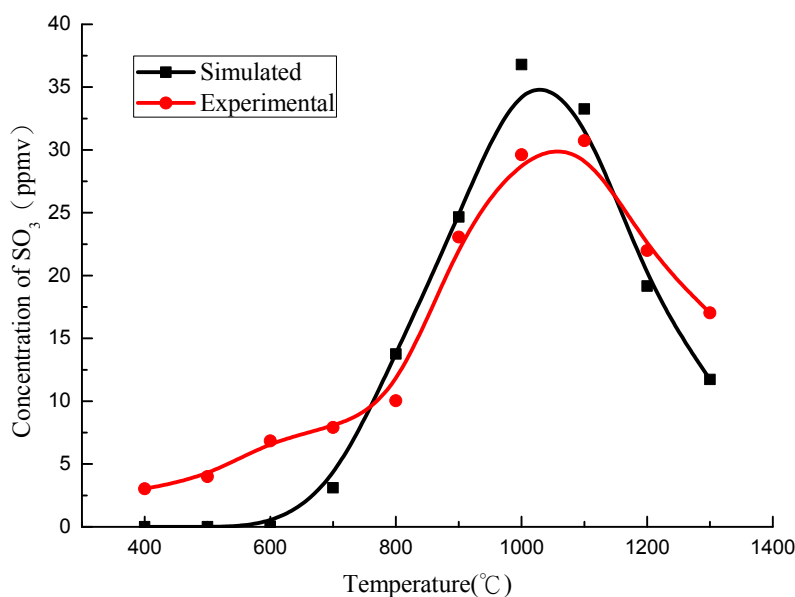


Fig. 2. Relationship between SO₃ concentration and temperature.

When the resistance furnace wall temperature is between 800°C and 1600°C, the basic reactions for SO₃ formation are (R3) and (R4). With an increase in temperature, H and OH production is enhanced by H₂O decomposition. An increase in the concentration of H radicals promotes formation of O radicals, enhancing (R7) and leading to an increase in OH concentration. A combination of H and OH will promote (R2), which will facilitate (R3).



The concentration of OH radical is higher than NO₂, so the effect of reaction (R3) is greater than that of (R4).

When the resistance furnace wall temperature is 1100°C, as the temperature continues to rise, the reaction rate of (R6) is accelerated and the concentration of NO₂ begins to decline. N₂ reacts with O₂ to generate large amounts of NO, and higher concentrations of NO inhibit (R4). Above 1100°C, the free radical HOSO₂ begins to decompose in the reaction gas.

Fleig *et al.* (2009) pointed out that formation of SO₃ in a power plant boiler mainly occurs between 900 and 1300°C, which is approximately consistent with the average temperature range of SO₃ generation.

Effect of O₂ on SO₃ Formation

The O₂ concentration of the combustible mixture was varied from 1% and 9% in simulations and experiments. Other inlet gas content remained unchanged.

Fig. 3 shows the outlet concentration of SO₃ derived for different concentrations of O₂ in the inlet gas. The SO₂ concentration was 2000 ppm in all experiments. SO₂ formation was favored by high O₂ concentrations, as well as by temperature. The concentration of SO₃ increased with increasing O₂, but the degree of influence gradually decreased. Duan (2015) also demonstrated that oxygen enrichment enhances SO₃ formation. When the concentration

of O₂ is less than 7%, the increase in O₂ concentration promotes the formation of SO₃. On the contrary, the influence on the formation of SO₃ is reduced. As shown in (R5), with increasing O₂ concentration, a higher concentration of O and OH radicals is expected, which contributes to higher SO₃ formation. Due to the fixed content of SO₂, when O₂ concentration is low, increasing the oxygen content can rapidly increase SO₃ formation. However, the O₂ that can participate in the reaction is affected by SO₂ concentration and temperature, so the effect of oxygen content on the SO₃ production rate gradually decreases.

ROP analysis was performed on reactions (R1)–(R4). When the concentration of O₂ increases, both (R3) and (R4) play a major role in the formation of SO₃. An increase in O₂ concentration promoted (R5) to produce more free radical O and OH, and then promoted (R6), such that (R3) plays a decisive role in the formation of SO₃.

Effect of SO₂ on SO₃ Formation

The experimental and modeling conditions were 550 ppm NO, 2.22% H₂O, 5% O₂, and a residence time of 4 s, SO₂ concentration ranged 500–3000 ppm. Fig. 4 shows the experimental and simulated SO₃ concentrations for different inlet SO₂ concentrations.

As shown in Fig. 4, both the experimental and modeling results show that the concentration of SO₃ increased with an increase in SO₂. A similar trend was observed in a study (Belo *et al.*, 2014) investigating gas-phase conversion of SO₂ to SO₃ in a simulated oxy-combustion environment, which was explained by the dependency of the reaction order on SO₂ concentration.

The simulation process analyzed by ROP shows that the most important reactions for SO₃ formation are (R3) and (R4) with increasing SO₂ concentrations. Increasing the concentration of SO₂ promotes reaction (R2), thus promoting (R3).

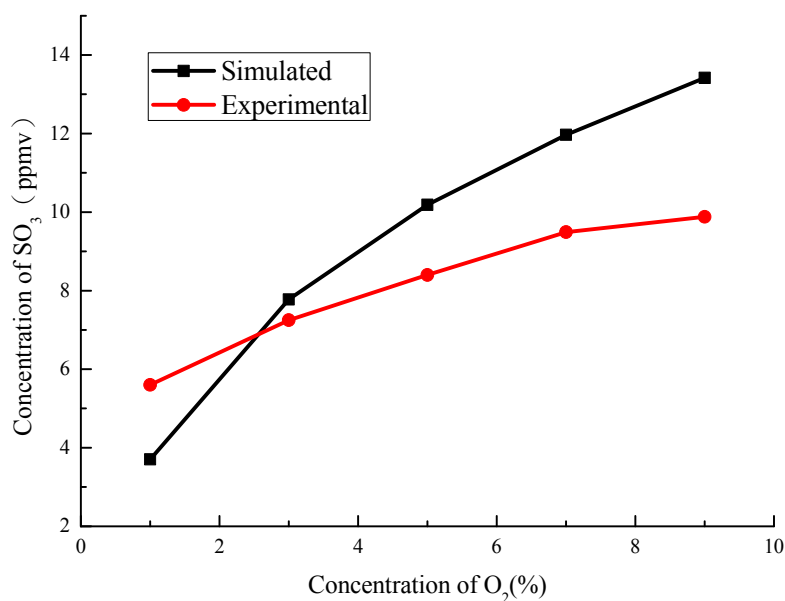


Fig. 3. The relationship between SO₃ concentration and O₂ concentration.

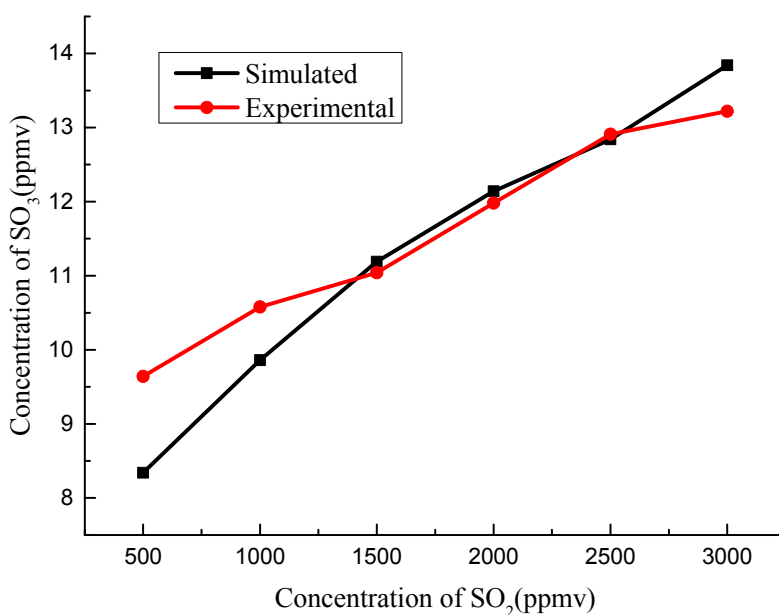


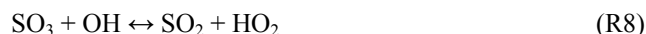
Fig. 4. SO₃ formation versus SO₂ concentration.

Effect of NO Concentration

NO concentrations between 0 and 1000 ppm were analyzed in simulations and experiments to investigate the effect of NO on the final SO₃ concentration (Fig. 5).

NO is known to affect the amount of SO₃ produced in this system. Increasing NO concentration, an increase in SO₃ concentration was observed for both experiments and simulations. When the NO concentration is below 600 ppmv, there are some differences between experimental data and simulated data. However, for NO concentration above 600 ppmv, the experimental results are basically consistent with simulated results. In a previous investigation, Fleig *et al.* (2011) showed that small amounts of NO can result in increased SO₃ generation during combustion.

ROP analyses showed that the following reactions were important to SO₃ formation: (R1), (R3), (R4), and (R8). (R3) and (R4) were determined to be most important for SO₃ generation.



The NO inlet concentration influenced the SO₃ outlet concentration, because NO acts on the radical pool through (R6) and (R9).



(R9) increases OH production, which can promote (R2)

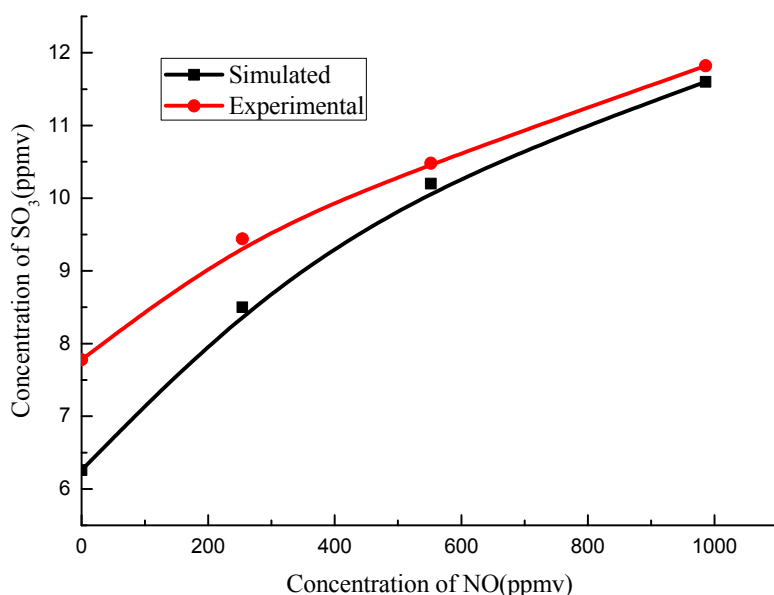


Fig. 5. Relationship between the concentrations of SO₃ and NO.

and (R3), and results in higher production rates of SO₃ from SO₂ via HOSO₂. However, as (R6) indicates, when NO is converted into NO₂, O is consumed. This reaction is in competition with (R1). But the most important direct interaction between NO_x and SO_x species is (R4), although (R6) consumes O and affects SO₃ generation, the SO₂ generated by (R6) will eventually generate SO₃ through (R4). Thus, the production of SO₃ increases with an increased NO concentration. A computational parametric study conducted by Choudhury *et al.* (2016) also concluded that the direct interaction between SO_x and NO_x mostly occurred via (R4), while indirect interactions changed the radical pool.

Effect of H₂O Concentration

In addition to obtaining the SO₃ content in coal combustion flue gas under different combustion parameters, the influence of H₂O was investigated through kinetic simulations and experiments (Fig. 6). There is a discrepancy between the experimental and modeling results. As far as the experimental results are concerned, the generation of SO₃ is very low when there is no H₂O. When the inlet gas contains 2.22% H₂O, the amount of SO₃ generation initially increases rapidly. Then with the increase of H₂O content, the generation of SO₃ remains constant. However, the simulation results demonstrate that the concentration of SO₃ increases gradually with the increase of H₂O content to 18.1%, and then remains unchanged. The reason for the discrepancy may be that the H₂O added in the experiment may not be vaporized in time. Anyway, the trend of the experimental and modeling results is consistent. H₂O has a positive effect on the formation of SO₃.

When the inlet gas contains H₂O, H and OH free radical concentrations will be increased via H₂O decomposition through (R10) (Wine *et al.*, 1984). The free radical H thereby participates in the reaction with O₂. In addition, the free radical O will react with H₂O, which can promote formation

of OH. Therefore, O and OH free radical concentrations increase through these reactions. The increase in O and OH results in an increase in the SO₃ formation by shifting (R1) to the right. In addition, because of the increase in OH, (R2) is promoted and SO₃ is mainly formed via HOSO₂.



Most previous studies are on the effect of H₂O on SO₃ formation, but their conclusions are different. There is still no consistent conclusion about the effect of H₂O on SO₃ formation. Belo (2014) suggested that the conversion of SO₂ to SO₃ is independent of water content. He found that increasing the moisture concentration from 3% to 9% did not have a significant effect on the conversion of SO₂ to SO₃. Wang (2015) found that the outlet SO₃ concentration decreased drastically with the injection of steam, which indicated steam could inhibit SO₃ formation. Fleig *et al.* (2013) concluded that an increase in H₂O concentration clearly increased SO₃ formation, as evidenced by experimental measurements and model predictions.

SENSITIVITY ANALYSIS

In the current research, experimental results and simulation data of others are similar, with the exception of the effect of H₂O concentration. To study the effect of inlet H₂O concentration on SO₃ generation, a sensitivity analysis was carried out.

Sensitivity analyses were performed on the model to determine the rate dominating reactions for SO₃ formation. The sensitivity function in the CHEMKIN-IV software was used for this purpose and the data was analyzed based on normalized coefficients. Different sets of reactions were observed to play a role in SO₃ formation for this mechanism. The reactions (R2), (R11), (R12), (R3), (R4), (R1) and (R-6) were found to influence SO₃ formation.

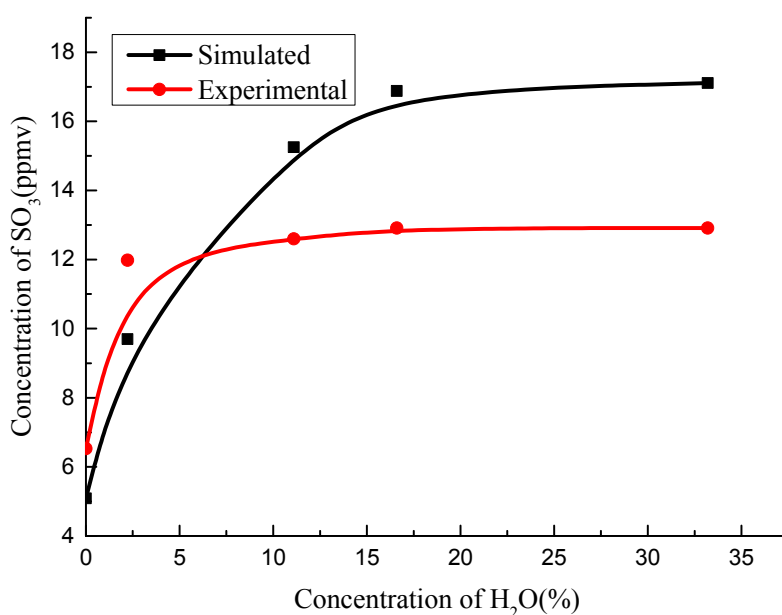


Fig. 6. Relationship between SO₃ and H₂O concentrations.

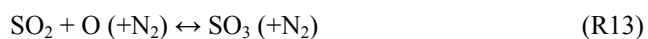
The sensitivity coefficients of the most sensitive reactions of input gas containing H₂O are shown in Fig. 7. The X-scale is the logarithmic sensitivity coefficient, which can be calculated as “ $\partial \ln m / \partial \ln k$ ”. Where m is SO₃ concentration in flue gas and k is the pre-exponential factor of the Arrhenius formula.



Positive sensitivities towards SO₃ generation were found for (R2), (R11), (R12), (R3), (R4), and (R1). Evolution of OH radicals and O₂ through these reactions facilitates SO₃ generation via (R1)–(R3). (R4) had a strong positive effect on SO₃ generation by the direct interaction between SO₂ and NO₂. (R2) was found to affect SO₃ formation to the greatest extent. Since the inlet gas contained water, a large amount of OH and O free radicals were generated by (R10), (R5), and (R7). So that, (R2), (R11), (R12), and (R1) were encouraged, enhancing the formation of SO₃.

A negative influence was observed for (R-6) due to an increase in O free radical concentration. The sensitivity analysis showed that S formation is highly sensitive to reactions involving H, OH, and O radicals.

On the other hand, when the input gas did not contain H₂O, the main reactions that affect SO₃ generation were (R11), (R1), (R-6), (R4), and (R13). The sensitivity coefficients of the most sensitive reactions of input gas without H₂O are shown in Fig. 8.



Obviously, (R11) was found to affect the SO₃ formation to the greatest extent, instead of (R2). In this case, (R11), (R1), (R4), and (R13) had a positive effect on SO₃ formation.

On the contrary, (R6) still had a negative effect on SO₃ formation. Compared with adding water, fewer reactions affect SO₃ generation. Moreover, the negative effect of (R6) is greater than when water is added. The reason for this is that without water, relatively small amounts of OH and O form.

The simulation results therefore show that the direct reaction between SO₂ and NO₂ plays an important role in the formation of SO₃, whether or not H₂O is in the input gas.

SO₃ gas generation experiments and chemical kinetic simulation results for the transformation of SO₂ to SO₃ are summarized in Fig. 9. Changing different factors influence processes (a), (b), and (c), affecting transformation of SO₂ to SO₃.

CONCLUSIONS

In this comprehensive study, sulfur chemistry, particularly SO₃ generation in the post-flame region, was investigated by both modeling and experimental methods. In particular, the influence of flue gas conditions (temperature, O₂, SO₂, NO, and H₂O) on the formation of SO₃ was studied.

Modeling temperature ranged from 400°C to 1300°C. With an increase in temperature, the concentration of SO₃ increased initially and then decreased, achieving its maximum value at around 1000°C. Additionally, the SO₃ concentration increased with the SO₂ concentration, and the outlet SO₃ concentration increased with the NO and O₂ concentrations. H₂O also contributed to the formation of SO₃.

To further study the effect of the influential factors on SO₃ in flue gas, an improved kinetic mechanism was built based on previous research. The direct interactions between SO_x and NO_x species were addressed in this model, and it was found that significant SO₃ formation occurs due to the direct interaction of SO₂, NO, and NO₂. ROP analyses revealed that reactions involving O, OH, and H dominated SO₃ generation.

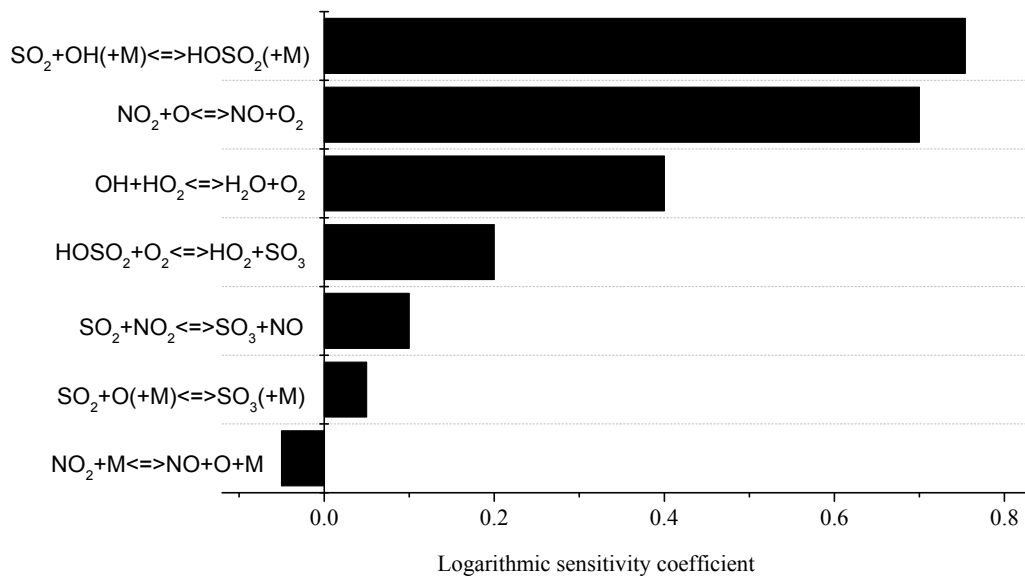


Fig. 7. Sensitivity coefficients of the most sensitive reactions of inlet gas containing H₂O.

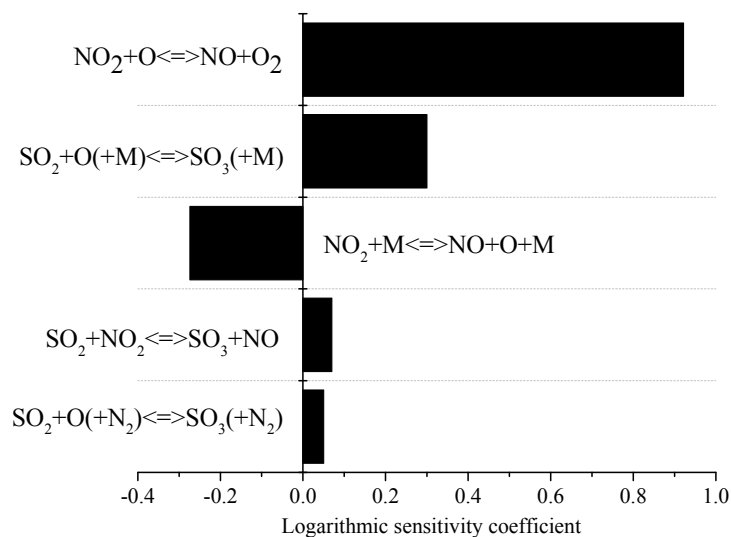


Fig. 8. Sensitivity coefficients of the most sensitive reactions of inlet gas without H₂O.

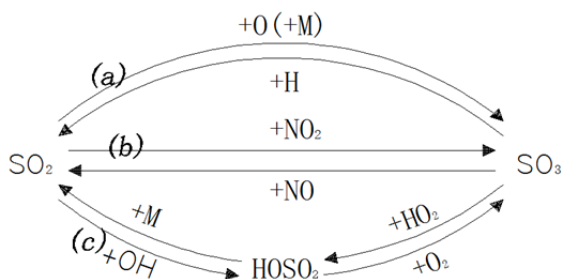


Fig. 9. Schematic of SO₃ generation.

Sensitivity analysis was carried out for two cases: inlet gas with and without H₂O. The direct reaction between SO₂ and NO₂ played an important role in the formation of SO₃. Moreover, when the inlet gas contained H₂O, the subsequently generated OH and O significantly affected

the formation of SO₃.

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