



Simultaneous removal of NO and SO₂ from coal-fired flue gas by UV/H₂O₂ advanced oxidation process

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ARTICLE INFO

Article history:

Received 18 May 2010

Received in revised form 1 July 2010

Accepted 1 July 2010

Keywords:

UV/H₂O₂

NO

SO₂

AOP

Reaction pathways

ABSTRACT

Simultaneous removal of NO and SO₂ from coal-fired flue gas by UV/H₂O₂ advanced oxidation process (AOP) was studied in an ultraviolet (UV)-bubble column reactor. Effects of UV lamp power and H₂O₂ concentration on NO and SO₂ removal efficiencies were studied. The results showed that under all conditions, SO₂ achieved 100% removal; between UV and H₂O₂ had a significant cooperative effect, and the cooperative factor reached 6.0; The NO removal was enhanced with the increase of H₂O₂ concentration or UV lamp power; The ion products in solution were analyzed with ion chromatography (IC), and the material balances for NO and SO₂ were calculated. The reaction pathways of removal of NO and SO₂ by wet scrubbing with UV/H₂O₂ were also preliminarily discussed.

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1. Introduction

During coal burning process, a large number of pollutants, including SO₂, NO_x, trace elements and volatile organic compounds (VOCs), are released. These pollutions have brought great harm to human health and environment [1,2]. Although wet limestone–gypsum flue gas desulfurization and selective catalytic reduction processes have achieved large-scale industrial application for flue gas treatment in coal-fired power plants, neither of them can achieve alone the integration removal of multiple pollutants [3,4]. The wet limestone–gypsum flue gas desulfurization and selective catalytic reduction combination technology can simultaneously remove SO₂ and NO_x, but the large and complex systems and the high capital and operating costs limit its utilization in the developing world [3,4].

Because of low capital and operating costs and simultaneous removal of multiple pollutants, integration removal technology has a good prospect of development and application [5,6]. Several integration flue gas treatment technologies, such as plasma removal [7], adsorbent adsorption removal [8], oxidant oxidation removal [1,2,4,6,9], complex adsorption removal [3,5,10], have been developed in the last several decades. However, due to high costs or technical problems, these integration technologies cannot still completely substitute for the wet limestone–gypsum flue gas desulfurization and the selective catalytic reduction combination

technology [11]. Therefore, developing a new integration flue gas treatment technology has become one of the major development trends in the coal-fired flue gas control field.

Advanced oxidation process can produce free radicals with strong oxidation, such as •OH, •O and HO₂•, to remove pollutants by oxidation reaction [11]. Some advanced oxidation processes, such as plasma removal [7], photocatalytic oxidation [11], and sonochemistry oxidation [12,13], have been developed to achieve integration removal of multiple pollutants from flue gas. However, due to high costs or technical problems, they cannot still achieve the goal of large-scale industrial application during a short time [14]. Because of strong oxidation ability and environmentally friendly characteristics, the UV/H₂O₂ advanced oxidation process has a wide range of studies in the water treatment field [15–17]. In the gas purification field, Cooper et al. [18,19] used •OH free radicals produced by UV decomposition of H₂O₂ in flue to remove NO. The results showed that in the range of 423–723 K high temperatures, NO achieved 65% removal. Jeong [20] used plasma combined with UV lamp to remove Hg⁰ and NO from gas. The results showed that UV irradiation effectively improved the oxidation efficiency of Hg⁰ and NO by producing more •OH free radicals. However, this kind of semi-dry UV/H₂O₂ removal process [18–20] has little potential of further development and application due to a very low utilization of H₂O₂ and a lack of effective cooling measures for ultraviolet lamp in high temperature flue.

According to the deficiencies of this kind of semi-dry UV/H₂O₂ removal process [18–20], a kind of wet UV/H₂O₂ removal process with a small laboratory scale was developed by installing a UV lamp in a bubble column reactor, which has some advantages in

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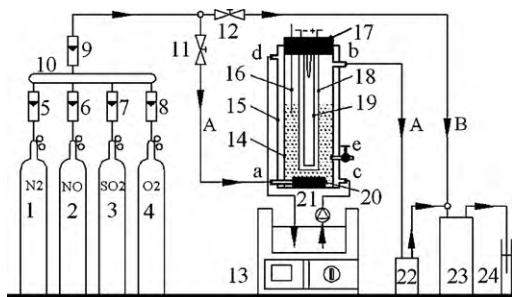


Fig. 1. Sketch of experimental system: (1–4) – N₂, NO, SO₂, O₂ gas cylinders; (5–9) – rotameters; (10) – buffer tank; (11, 12) – valves; (13) – constant temperature water bath; (14) – bubble column reactor; (15) – jacket heat exchanger; (16) – mercury thermometer; (17) – rubber plug; (18) – quartz casing; (19) – UV lamp; (20) – gas distributor; (21) – cooling circulating pump; (22) – gas dryer; (23) – gas analyzer; (24) – tail gas absorber; (A) – gas primary road; (B) – gas bypass; (a) – gas inlet; (b) – gas outlet; (c) – cooling water inlet; (d) – cooling water outlet; (e) – sampling outlet.

high utilization of H₂O₂ and effective cooling of UV lamp due to mild reaction conditions of wet removal process. However, as two major pollutants from coal-fired flue gas, wet removal of SO₂ and NO with UV/H₂O₂ is not still studied so far. In this paper, a preliminary study on effects of H₂O₂ concentration and UV lamp power on NO and SO₂ removal efficiencies, and the reaction pathways of removal of NO and SO₂ by wet scrubbing with UV/H₂O₂ was done in a semi-continuous ultraviolet (UV)-bubble column reactor. The results will be able to provide some theoretical guidance for development and application of removal of NO and SO₂ by wet scrubbing with UV/H₂O₂.

2. Experimental section

2.1. Experimental apparatus

Experimental system is shown in Fig. 1. The gases are supplied by cylinders (1–4) filled with different gases. There are some rotameters (5–9) in the tubings of connecting the cylinders (1–4) for the adjustment of gas composition and flow. The gases from cylinders (1–4) are mixed in buffer tank (10). The flow direction of mixed gas is controlled through switching two valves (11, 12). The inlet concentrations of mixed gas are measured by the gas bypass (B) and the experiments are conducted by the gas primary road (A).

The body of photochemical reactor is constructed by a PMMA bubble column reactor (14) (height, 40.0 cm; inside diameter, 8.0 cm), and the top is covered with a rubber plug (17). A sand chip gas distributor (20) (outside diameter 7.0 cm; high 3.0 cm; average pore size 15.0–40.0 μm) located at the bottom of bubble column reactor (14) is used to obtain a uniform distribution of gas. The UV lamp (19) is set inside a quartz casing (18) (outside diameter, 4.0 cm) which is fixed to the rubber plug (17). In order to keep a suitable reaction temperature, a jacket heat exchanger (15) is set outside the bubble column reactor (14) and the temperature is adjusted by a constant temperature water bath (13) (DCW-1015, ±0.1 °C, Ningbo Jiangnan Instrument Factory) with a cooling circulating pump (21). Solution is added into or moved out the bubble column reactor (14) by opening the rubber plug (17) and its temperature during experiment is measured by a mercury thermometer (16). There are five small inlets and outlets, including inlet (a) and outlet (b) of mixed gas, inlet (c) and outlet (d) of cooling water and sampling outlet (e) of solution, on the body of photochemical reactor.

The gas dryer (22), containing anhydrous calcium chloride (Granular, Shanghai Chemical Reagent Co., AR), is used to dry the mixed gas scrubbed to ensure the measure accuracy and the safety of electrochemical probes in gas analyzer (23). A gas analyzer (23) (MRU-VARIO PLUS, Germany) with the ability for simultaneous and

continuous measurements of NO, NO₂, SO₂, O₂, CO, H₂S, H₂ and CO₂ from gas is used to measure the inlet and outlet concentrations of pollutants. The residual pollutants in mixed gas are further scrubbed by a tail gas absorber (24), containing 800 mL mixed solution of (0.05 mol/L) KMnO₄ and (0.1 mol/L) NaOH (both of them are from Shanghai Chemical Reagent Co., AR), to avoid environmental pollution.

2.2. Experimental procedures

Firstly, H₂O₂ solution was prepared with 30% H₂O₂ solution (Shanghai Chemical Reagent Co., AR) and deionized water according to the required H₂O₂ concentrations, and the volume of solution used for each experiment was 600 mL. The initial pH values of solution were adjusted through HCl (0.5 mol/L) and NaOH (0.5 mol/L) solutions (Shanghai Chemical Reagent Co., AR) and were measured by an acidimeter (PHB-3, Shanghai leici instrument Co., China), and the solution prepared was added into bubble column reactor (14) by opening the rubber plug (17). After closing the rubber plug (17), the solution temperatures were kept at 298 K for all experiments by regulating the constant temperature water bath (13).

Secondly, four kinds of gases, N₂, O₂, SO₂ and NO (high-purity gases, Nanjing Specialty Gas Production Plants), were used to make mixed gas. The compositions, concentrations and gas flows of pollutants from mixed gas were regulated by the rotameters (5–9). After opening the valve (12) and closing the valve (11), the inlet concentrations of pollutants from mixed gas were measured using the gas analyzer (23) through the gas bypass (B).

Furthermore, when the solution temperature was steady, the experiment was started. After closing the valve (12) and opening the valve (11), the mixed gas firstly flowed into the bubble column reactor (14) through the gas primary road (A) to clear away the remaining air in the bubble column reactor (14). When the UV lamp (19) was turned on, the pollutants from mixed gas were scrubbed in the bubble column reactor (14), and the outlet concentrations of pollutants with time were also recorded simultaneously (each experimental run was 20 min, recording once per minute) through the gas analyzer (23). In order to study reaction pathways of removal of NO and SO₂ by wet scrubbing with UV/H₂O₂, the sample solutions were obtained by the sampling outlet (e) and deposited in brown sampling jugs to be analyzed. The UV lamp powers were changed by replacing and using three sets of UV lamps with different powers. Here three sets of UV lamps (PL-L18W and PL-L36W, produced by Philips; HL-L72W, produced by Haining Light Factory) were employed, and all of them are the same model (L-L) and wavelength (253.7 nm).

Finally, after being scrubbed, the mixed gas with moisture was dried by the gas dryer (22). The remaining pollutants from mixed gas were further scrubbed by the tail gas absorber (24). Turning off UV lamp and closing cylinder valves, the experiment was finished. The ion products in the sample solutions were analyzed with ion chromatography (792 Basic IC, Metrohm in Switzerland).

Chromatographic conditions: Anion dual 2 anion column, eluent (1.0 mmol/L Na₂CO₃ + 1.5 mmol/L NaHCO₃), flowrate (0.80 mL/min), injection volume (25 μl), column temperature (303 K), and automatic regeneration suppression system (H₂O and 60 mmol H₂SO₄). The qualitative and quantitative results of ion products in solution were obtained according to the retention time and the peak area, respectively.

The constant conditions are summarized in Table 1. The other conditions are listed under the titles of Figs. 2–5, respectively.

2.3. Data process

Each experimental run was 20 min, and a concentration value per minute was recorded. The average concentration within 20 min

Table 1
Constant conditions summary for removal of NO and SO₂ with UV/H₂O₂.

| Gas composition | Solution composition | Solution volume | Gas flow | Temperature | pH | O ₂ |
|---|--|-----------------|------------|-------------|-----|----------------|
| N ₂ , NO, SO ₂ , O ₂ | H ₂ O ₂ solution | 600 mL | 500 mL/min | 298 K | 3.2 | 6.0% |

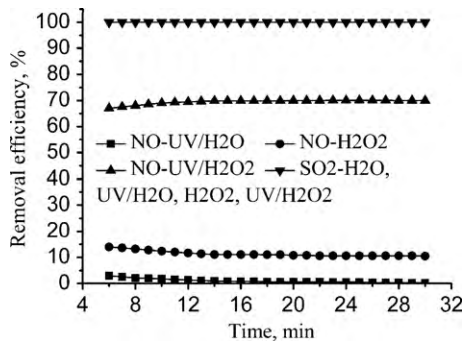


Fig. 2. Removal efficiencies of NO and SO₂ under different reaction systems. Conditions: UV lamp power, 36 W; NO concentration, 407 ppm; SO₂ concentration, 978 ppm; H₂O₂ concentration, 2.0 mol/L.

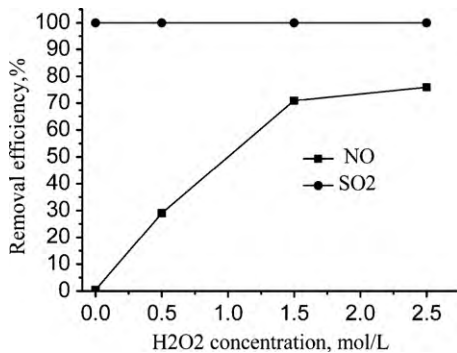


Fig. 3. Removal efficiencies of NO and SO₂ under different H₂O₂ concentrations. Conditions: UV lamp power, 36 W; NO concentration, 419 ppm; SO₂ concentration, 948 ppm.

was used as the outlet concentration C_{out} , and the removal efficiency η was calculated by the following Eq. (1):

$$\eta = \frac{C_{in} - C_{out}}{C_{in}} \times 100\% \quad (1)$$

where η , removal efficiency; C_{in} , inlet concentration; and C_{out} , outlet concentration.

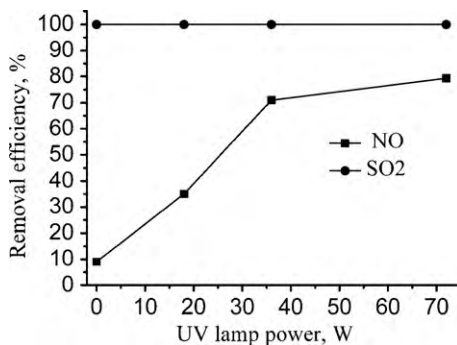


Fig. 4. Removal efficiencies of NO and SO₂ under different UV lamp powers. Conditions: H₂O₂ concentration, 2.0 mol/L; NO concentration, 414 ppm; SO₂ concentration, 1013 ppm.

3. Results and discussions

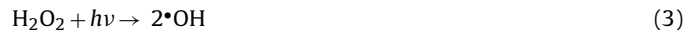
3.1. Contrast study on NO and SO₂ removal efficiencies in different systems

The experiments in different reaction systems were carried out and the results are shown in Fig. 2. As shown in Fig. 2, SO₂ achieved 100% removal. The NO could only achieve 11.5% removal in H₂O₂ solution, and even little NO was removed in UV/H₂O. However, when both of UV and H₂O₂ simultaneously existed, NO achieved 72.0% removal. The results showed that between UV and H₂O₂ had a significant cooperative effect. The cooperative factor ε could be calculated by the following Eq. (2):

$$\varepsilon = \frac{\eta_{UV/H_2O_2}}{\eta_{UV} + \eta_{H_2O_2}} \quad (2)$$

where ε , cooperative factor; η_{UV/H_2O_2} , removal efficiency of UV/H₂O₂; η_{UV} , removal efficiency of UV; and $\eta_{H_2O_2}$, removal efficiency of H₂O₂.

The results showed that the cooperative factor ε was 6.0, and oxidation removal of $\cdot OH$ free radicals was a leading reaction pathway in removal of NO and SO₂ with UV/H₂O₂. The cooperative mechanism between UV and H₂O₂ can be explained by the following reaction [21–23]:



The $\cdot OH$ free radicals have an extremely strong oxidation ability for removal of NO and SO₂ [12,13], leading to a sharp increase in removal efficiencies. In order to study the roles of H₂O₂ and UV in removal of NO and SO₂ by wet scrubbing with UV/H₂O₂ in depth, the effects of H₂O₂ concentration and UV lamp power on the NO and SO₂ removal efficiencies were further studied in the next two sections.

3.2. Effects of H₂O₂ concentration

Many research results [15,16,24] showed that the H₂O₂ concentration had a significant impact on the removal efficiency of pollutant with UV/H₂O₂. So here some experiments were con-

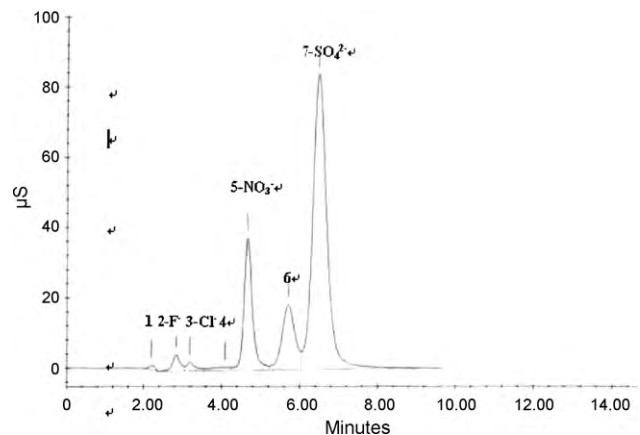
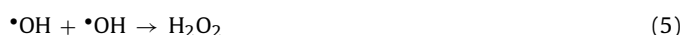


Fig. 5. Ion chromatogram analysis of ions in solution at 40 min. Conditions: H₂O₂, 2.0 mol/L; UV lamp power, 36 W; NO concentration, 472 ppm (632 mg/m³); 408 ppm (1166 mg/m³); gas flow, 800 mL/min; t_r , 40 min.

ducted with different H₂O₂ concentrations and the results are shown in Fig. 3.

It could be seen that SO₂ achieved 100% removal. However, little NO was removed when H₂O₂ concentration was zero, suggesting the UV/H₂O had almost no removal ability for NO. When H₂O₂ concentration increased from 0 to 1.5 mol/L, the NO removal efficiency had a sharp increase, increasing from 0 to 69.5%. Then further increase of H₂O₂ concentration only caused a slight increase in NO removal efficiency. It has been known that as a releasing agent of •OH free radicals, H₂O₂ plays a key role in photochemical reaction. The effect of H₂O₂ concentration on the NO removal efficiency can be explained by the following reasons. On the one hand, when the H₂O₂ concentration is appropriate, it may cause a reaction such as Eq. (3) in the solution [22,23]. Therefore, within a certain range, the increase in H₂O₂ concentration can improve the yield of •OH free radicals, and then increase the NO removal efficiency; on the other hand, when exceeding a certain value, further increase of H₂O₂ concentration may cause several side reactions in the solution [15,16]:



So the H₂O₂ is also the etchant of •OH free radicals besides the releasing agent. The oxidation abilities of HO₂• free radicals (1.60 eV) and H₂O₂ (1.77 eV) produced by side reactions (4) and (5) are much smaller than that of •OH free radicals (2.80 eV) [15,24]. Therefore, further increase of H₂O₂ concentration only has a slight impact on the NO removal efficiency at this time.

3.3. Effect of UV lamp power

The experiments with different UV lamp powers were carried out, and the results are shown in Fig. 4. Also SO₂ achieved 100% removal under different UV lamp powers. The NO removal efficiency was only 10.8% when without UV light. The NO removal efficiency revealed an obvious increase, increasing from 10.8 to 72.5% when the UV lamp power increased from 0W to 36W. However, further increasing the UV lamp power from 36 to 72W, the growth rate of NO removal efficiency gradually stabilized.

There are two main reasons for explaining the results as below. On the one hand, under UV light irradiation, H₂O₂ can release •OH free radicals by Eq. (3) reaction [15,23,24]. Due to extremely high redox potential (2.80 eV, next to 2.87 eV of fluorine) [15,16], the •OH free radicals have extremely strong oxidation ability to remove NO by oxidation reaction [12,13]. Therefore, compared with the reaction system without UV light, the addition of UV light can greatly enhance NO removal.

Furthermore, the Beer–Lambert law holds that the photochemical reaction yield is proportional to the UV irradiation intensity (UV irradiation intensity is also proportional to UV lamp power), which means that increasing UV lamp power can improve the energy density per unit in solution, producing more effective photons, and finally generate more •OH free radicals [15,16,24]. Therefore, the NO removal efficiency increased with the increase of UV lamp power; on the other hand, when the UV lamp power exceeds a certain value, several side reactions, such as Eqs. (4) and (5), may be also caused in solution, leading to a great loss of •OH free radicals [15,16,24]. So further increase of UV lamp power only has a little impact on NO removal efficiency at this time.

3.4. Analysis of ion products in solution

The experiments for analysis of ion products in solution were carried out, and the results are shown in Fig. 5 and Table 2. As shown

Table 2
Material balance summary for NO and SO₂.

| Category | Run 1 | Run 2 | Run 3 |
|---|-------|-------|--------|
| Time <i>t</i> , min | 20 | 40 | 60 |
| Gas flow <i>Q</i> , mL/min | 800 | 800 | 800 |
| Solution volume <i>V_L</i> , mL | 600 | 600 | 600 |
| <i>C_{in}</i> (NO), ppm | 472 | 472 | 472 |
| <i>C_{out}</i> (NO), ppm | 178 | 172 | 173 |
| NO removal efficiency, % | 62.2 | 63.6 | 63.3 |
| <i>C_{in}</i> (SO ₂), ppm | 408 | 408 | 408 |
| <i>C_{out}</i> (SO ₂), ppm | 0 | 0 | 0 |
| SO ₂ removal efficiency, % | 100 | 100 | 100 |
| <i>C</i> (NO ₂ ⁻) actual value, mg/L | 0 | 0 | 0 |
| <i>C</i> (SO ₃ ²⁻) actual value, mg/L | 0 | 0 | 0 |
| <i>C</i> (NO ₃ ⁻) actual value, mg/L | 21.31 | 41.82 | 64.28 |
| <i>C</i> (NO ₃ ⁻) calculation value, mg/L | 21.67 | 44.29 | 66.21 |
| <i>C</i> (NO ₃ ⁻) error, mg/L | 0.36 | 2.47 | 1.93 |
| <i>C</i> (SO ₄ ²⁻) actual value, mg/L | 43.15 | 91.89 | 138.85 |
| <i>C</i> (SO ₄ ²⁻) calculation value, mg/L | 46.63 | 93.26 | 139.89 |
| <i>C</i> (SO ₄ ²⁻) error, mg/L | 3.48 | 1.37 | 1.04 |

C(x): abbreviation of concentration.

in Fig. 5, the ion chromatography (IC) analysis results showed that NO and SO₂ were removed by oxidation reaction.

Due to instabilities of NO₂⁻ and SO₃²⁻ in H₂O₂ solution [34,35], NO₂⁻ and SO₃²⁻ were not found, and NO₃⁻ and SO₄²⁻ were the major ion products in solution. Furthermore, a small quantity of F⁻, Cl⁻ and several non-identified ions in solution were also found. We believe that they may derive from the oxidative decomposition products or UV excitation decomposition products of reactor wall (PMMA), sealing ring (poly(tetrafluoroethylene), CF₃(CF₂CF₂)_{*n*}CF₃) or the residue impurities from pipelines (polyvinyl chloride, CH₂–CHCl). But due to trace, we believe that they may only have a very slight impact on our analysis results.

In order to further verify the reaction pathways for removal of NO and SO₂ with UV/H₂O₂, based on the IC analysis results, the material balances for NO and SO₂ were calculated. The related conditions and calculation results are shown in Table 1. Based on the mass conservation of nitrogen element from NO or sulphur element from SO₂, the calculation values of NO₃⁻, NO₂⁻, SO₃²⁻ or SO₄²⁻ in solution can be calculated by the following Eq. (6):

$$C_{\text{cal}} = \frac{\eta \cdot C_{\text{in}} \cdot Q \cdot t \cdot M_1}{M_2 \cdot V_L} \quad (6)$$

where *C_{cal}*, calculation concentration of ions in solution, mg/L; *η*, removal efficiency of pollutants, %; *C_{in}*, inlet concentration of pollutants, mg/m³; *Q*, gas flow, mL/min; *t*, reaction time, min; *M₁*, molar mass of ions in solution, g/mol; *M₂*, molar mass of pollutants, g/mol; and *V_L*, solution volume, L.

As shown in Table 2, the low valence nitrogen element (+2) in NO and the low valence sulphur element (+4) in SO₂ almost completely transformed into the high valence nitrogen element (+5) in NO₃⁻ and the high sulphur element (+6) in SO₄²⁻, respectively. Compared with the actual values, the calculation values only had relatively small errors.

3.5. Reaction pathways

Based on known results [25–28], there were three major reaction pathways confirmed for removal of pollutants using UV/H₂O₂, including excitation removal of UV, oxidation removal of H₂O₂, and oxidation removal of •OH free radicals, and the oxidation removal of •OH free radicals usually played a leading role among them.

As shown in Fig. 2, SO₂ also achieved 100% removal even in different reaction systems. NO only achieved 11.5% removal in H₂O₂ solution, and even little NO was removed in UV/H₂O, but UV/H₂O₂ achieved 72.0% NO removal. The results showed that excitation removal of UV for NO failed to occur. The oxidation removal of

·OH free radicals played a leading role, and oxidation removal of H₂O₂ only played a secondary role in removal of NO and SO₂ by wet scrubbing using UV/H₂O₂. The results of ion chromatography analysis and material balance calculation also showed that NO and SO₂ were removed by oxidation reaction.

In summary, on the basis of our results, although several other side reactions can possibly occur in solution, the major reaction pathways for removal of NO and SO₂ by wet scrubbing using UV/H₂O₂ can be preliminarily concluded as follows:

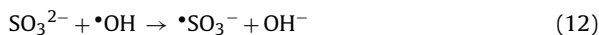
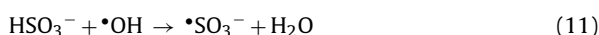
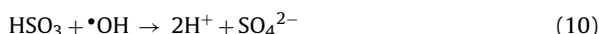
(i) At first, the ·OH free radicals are produced by UV excitation decomposition of H₂O₂, such as Eq. (3), it is an initiation step for the whole free radical chain reactions [21–23,29–31].

(ii) Reaction pathways for removal of SO₂:

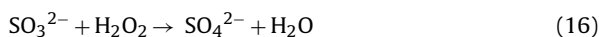
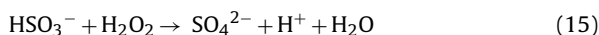
(1) The hydrolysis reactions of SO₂ in water [32,33]:



(2) The oxidation removal of ·OH free radicals [12,13,36–41]:



(3) The oxidation removal of H₂O₂ [33,34]:

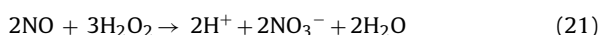


(iii) Reaction pathways for removal of NO:

(1) The oxidation removal of ·OH free radicals [12,13,39–43]:



(2) The oxidation removal of H₂O₂ [34,35]:



(iv) When H₂O₂ concentration or UV lamp power exceeds a great value, some side reactions may be also caused, such as Eqs. (4) and (5) [15,16,24].

4. Conclusions

Under all conditions, SO₂ could achieve 100% removal; between UV and H₂O₂ had a significant cooperative effect, and the cooperative factor reached 6.0. With the increase of H₂O₂ concentration or UV lamp power, the NO removal efficiency increased at first, and then the growth rate of NO removal efficiency gradually stabilized; the results of IC analysis and material balance calculation showed that NO and SO₂ were removed by oxidation. The oxidation removal of ·OH free radicals played a leading role, and oxidation removal of H₂O₂ only played a secondary role in removal of NO and SO₂ by wet scrubbing using UV/H₂O₂. The major reaction pathways for removal of NO and SO₂ by wet scrubbing using UV/H₂O₂ were also preliminarily concluded.

Acknowledgements

This study is supported by the International Cooperation Projects of the National Natural Science Foundation of China (No. 50721140649). The authors are grateful for the test support of Analysis and Testing Center of SEU.

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