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Photochemical Oxidation Removal of NO and SO₂ from Simulated Flue Gas of Coal-Fired Power Plants by Wet Scrubbing Using UV/H₂O₂ Advanced Oxidation Process

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ABSTRACT: Photochemical oxidation removal of NO and SO₂ from simulated flue gas of coal-fired power plants by wet scrubbing using UV/H₂O₂ advanced oxidation process was studied in a semicontinuous and small-scale ultraviolet (UV)-bubble column reactor with different conditions, including UV, H₂O₂ concentration, solution initial pH value, solution temperature, and liquid layer height. The results show that under all conditions studied, the SO₂ is removed completely. The UV has an obvious impact on NO removal efficiency. With the increase of H₂O₂ concentration or liquid layer height, the NO removal efficiency greatly increases at first, and then the growth rate of NO removal efficiency gradually become smaller. The NO removal process is enhanced by the solution initial pH value, but it is inhibited by the solution temperature. The gaseous and liquid reaction products are determined using ion chromatography and gas analyzer, respectively. The removal path of NO and SO₂ are also preliminarily discussed.

1. INTRODUCTION

During the coal burning process, the noxious gases, SO₂ and NO_x, are released. They can form acid rain and photochemical smog, finally causing great harm to human health and environment.^{1,2} Although wet limestone-gypsum flue gas desulfurization (WFGD-Ca) and ammonia selective catalytic reduction (SCR-NH₃) processes have been developed and applied at large scale for flue gas purification, neither of them alone can achieve the simultaneous removal of SO₂ and NO_x. The combined process of WFGD-Ca and SCR-NH₃ can simultaneously remove SO₂ and NO_x, but the large and complex systems and the high capital and operating costs limit their utilization in the developing world.^{3,4} So developing more effective flue gas purification technologies is always one of the main research interests in the field of flue gas purification.

Advanced oxidation processes (AOP) can produce ·OH free radicals to simultaneously oxidize and remove multiple pollutants from flue gas.^{5–12} So many interesting advanced oxidation processes, mainly including plasma oxidation,^{5,6} photochemical oxidation,^{7,8} sonochemical oxidation,^{9,10} and Fenton oxidation^{11,12} have been developed and used for removing various gaseous pollutants, such as SO₂, NO_x, trace elements, H₂S, and volatile organic compounds (VOCs) in recent years. However, so far, none of them can successfully substitute for the combined process of WFGD-Ca and SCR-NH₃ in the short term due to the current challenges in costs or technical implementation. Therefore, developing more effective advanced oxidation flue gas purification technologies (AOFGPT) is still a main research subject in recent years.

UV/H₂O₂ AOP can produce ·OH free radicals by photolysis of H₂O₂ to oxidize and remove various pollutants, and it has strong oxidation, simple and secure process, low energy consumption, and no secondary pollution.¹³ So the UV/H₂O₂ AOP has been widely studied and applied for the degradation and discoloration of organic pollutants in the water treatment field for the past 20 years.¹³ Recently, some results^{14–19} show that the

UV/H₂O₂ AOP can also be used for effectively purifying the multiple pollutants from flue gas. Cooper et al.¹⁴ first used a semidry UV/H₂O₂ AOP to oxidize and remove NO from simulated flue gas by radiating UV and spraying H₂O₂ in flue. The results show that the NO removal efficiency is markedly increased compared with the single H₂O₂ oxidation, but this semidry UV/H₂O₂ AOP is not further developed because of the large self-decomposition consumption of H₂O₂ in high temperature flue. Ma et al.¹⁵ used a wet UV/H₂O₂ AOP to remove NO_x and SO₂ from simulated flue gas by using a UV lamp to radiate the surface of H₂O₂-containing bubble column reactor. The results show that the NO_x and SO₂ achieved high removal efficiencies and especially the utilization rate of H₂O₂ was improved significantly compared with the semidry UV/H₂O₂ AOP. Recently, Liu et al.^{16–19} developed a new and more applicable wet UV/H₂O₂ AOP to remove NO and SO₂ from simulated flue gas by setting a UV lamp into a bubble column reactor. The several main process parameters, such as the gas flow, the initial concentration of NO, the initial concentration of SO₂, the partial pressure of O₂, and the UV lamp power, were studied preliminarily. The results showed that the removal process was markedly affected by the gas flow, the initial concentration of NO, the initial concentration of SO₂, and the UV lamp power. However, so far, the potential effects of some factors, such as UV, H₂O₂ concentration, solution initial pH value, solution temperature, and liquid layer height on the removal efficiencies of NO and SO₂ have not been studied yet. The gaseous and liquid reaction products have also not been determined. The objective of this work is to study the effects of some factors, such as UV, H₂O₂ concentration, solution initial pH value, solution temperature, and liquid layer height, on NO

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Table 1. Constant Conditions Summary for Wet Removal of NO and SO₂ with UV/H₂O₂ AOP

mixed flue gas compositions	SO ₂	NO	O ₂	UV lamp power	gas flow
N ₂ , SO ₂ , NO, O ₂	1000 ppm	400 ppm	6.0%	36 W	500 mL/min

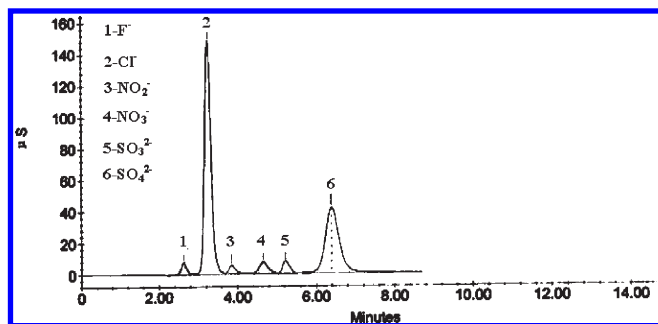
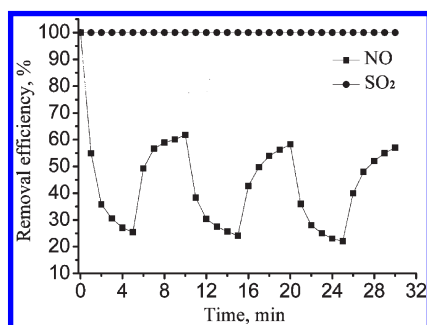


Figure 1. The standard chromatography profile of ions measured.

Figure 2. Effects of UV on removal efficiency in the UV/H₂O₂ AOP system. Conditions: H₂O₂, 1.5 mol/L; solution initial pH, 3.2; solution temperature, 298 K; liquid layer height, 15.9 cm.

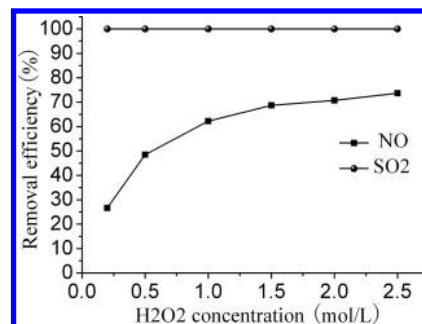
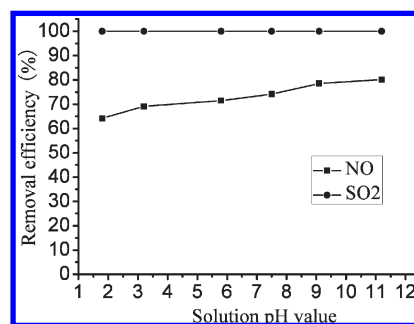
and SO₂ removal efficiencies, and to determine the gaseous and liquid reaction products. The results may provide some scientific basis for the development and application of this new technology.

2. EXPERIMENTAL SECTION

2.1. Experimental Procedures. Experiments were conducted in a bubble column reactor. The details of the experimental system can be found elsewhere.¹⁸ Solutions with different concentrations of H₂O₂ were prepared with 30% H₂O₂ solution (Shanghai Chemical Reagent Co., AR) and deionized water. The pH of the solutions was adjusted to the required values through addition of HCl (0.5 mol/L) or NaOH (0.5 mol/L) solutions (Shanghai Chemical Reagent Co., AR). The volume of the solutions used for each experiment was 600 mL, and the pH was 3.2, except for the experiments of changing pH.

Four kinds of gases, N₂, O₂, SO₂, and NO (Nanjing Specialty Gas Production Plants, high-purity gases), were used to make the simulated flue gas. The compositions and flow rates of the simulated flue gas and the concentrations of the pollutants in the simulated flue gas were regulated by the rotameters, and the inlet concentrations of pollutants were measured using the gas analyzer (MRU-VARIO PLUS, Germany) through the gas bypass.

The UV lamp power (PL-L36W, produced by Philips, wavelength of 253.7 nm) was kept at 36 W. The temperature of the reactor was controlled by a mercury thermometer and a constant

Figure 3. Removal efficiencies of NO and SO₂ under different H₂O₂ concentrations. Conditions: solution initial pH, 3.2; solution temperature, 298 K; liquid layer height, 15.9 cm.Figure 4. Removal efficiencies of NO and SO₂ under different initial pH values. Conditions: H₂O₂, 2.0 mol/L; solution temperature, 298 K; liquid layer height, 15.9 cm.

temperature water bath (DCW-1015, ± 0.1 °C, Ningbo Jiangnan Instrument Factory) with a cooling circulating pump. Each experimental run was 20 min and the outlet concentrations of the pollutants per minute were measured through the gas analyzer.

For each experiment, the solution was collected after the experiment ended. The ion products in the sample solutions were analyzed using ion chromatography (792 Basic IC, Metrohm in Switzerland). Ion chromatography has an anion exchange column of anion dual 2, an automatic regeneration suppression system made of H₂O and 60 mmol H₂SO₄, and an eluent made of 1.0 mmol/L Na₂CO₃ and 1.5 mmol/L NaHCO₃. The ion chromatography conditions were set at a flow rate of 0.80 mL/min, an injection volume of 25 μ L, and a column temperature of 303 K.

The gas compositions and constant conditions used in the experiments are summarized in Table 1. The other conditions are listed under the titles of Figures 1–9, respectively.

2.2. Data Processing. The average concentration within 20 min 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 is the removal efficiency; C_{in} is the inlet concentration; C_{out} is the outlet concentration.

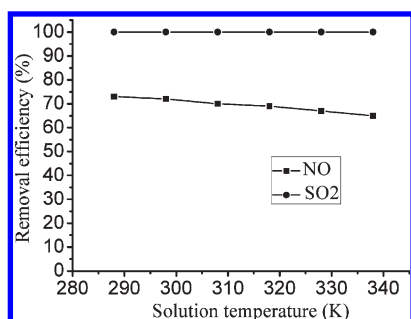


Figure 5. Removal efficiencies of NO and SO₂ under different solution temperatures. **Conditions:** H₂O₂, 2.0 mol/L; solution initial pH, 3.2; liquid layer height, 15.9 cm.

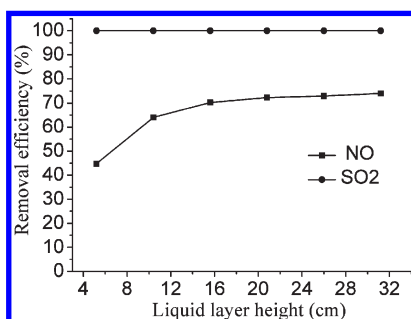


Figure 6. Removal efficiencies of NO and SO₂ under different liquid layer heights. **Conditions:** H₂O₂, 2.0 mol/L; solution initial pH, 3.2; solution temperature, 298 K.

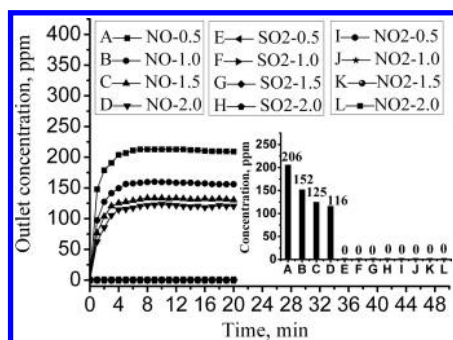


Figure 7. Removal efficiencies of NO, NO₂, and SO₂ under different H₂O₂ initial concentrations. **Conditions:** initial pH, 3.2; solution temperature, 298 K; liquid layer height, 15.9 cm.

3. RESULTS AND DISCUSSIONS

3.1. Effects of UV. The effects of UV on the NO and SO₂ removal efficiencies were studied. As shown in Figure 2, with the turning on (wave trough) and turning off (wave peak) of the UV lamp, the SO₂ removal efficiency keeps constant, and it has complete removal, but the NO removal efficiency has a great change. It can be seen that the removal efficiency of NO with UV radiation (turning on UV lamp) was much higher than that without UV radiation (turning off UV lamp), and the change trend of the NO removal efficiency shows good reproducibility.

The results show that there is a significant cooperative effect between UV and H₂O₂, and the cooperative mechanism of UV and H₂O₂ may be explained by the following main reasons. On the one hand, when there is no UV radiation (turning off the UV

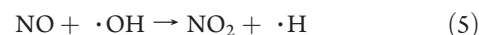
lamp), NO can only be removed by single oxidation of H₂O₂ according to the following reactions:^{20–23}



On the other hand, when UV is added (turning on the UV lamp), a lot of $\cdot\text{OH}$ free radicals are produced by photolysis of H₂O₂ according to the following reaction:^{24–27}

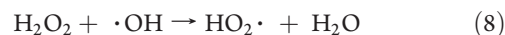


As the $\cdot\text{OH}$ free radical has extremely strong oxidation (the reaction rate constant of $\cdot\text{OH}$ and NO reaches $5.5 \times 10^{14} \text{ mol} \cdot (\text{L} \cdot \text{s})^{-1}$ at 298 K),²⁸ when UV and H₂O₂ simultaneously exist, the NO also can be effectively removed by oxidation of $\cdot\text{OH}$ free radical according to the following reactions.^{6–10,28}



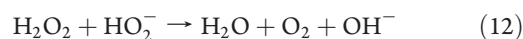
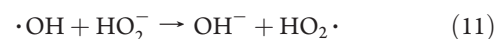
3.2. Effects of H₂O₂ Concentration. The effects of H₂O₂ concentration on NO and SO₂ removal efficiencies were studied and the results are shown in Figure 3. It can be seen that the SO₂ reaches 100% removal efficiency under different H₂O₂ concentrations. However, when the H₂O₂ concentration increases from 0 to 2.0 mol/L, the NO removal efficiency has a great increase, increasing from 0 to 70.8%. Then the further increase of H₂O₂ concentration from 2.0 to 2.5 mol/L only causes a small increase in the NO removal efficiency.

The effect of H₂O₂ concentration on NO removal efficiency is generally explained by the following mechanisms. On the one hand, it has been known that H₂O₂ plays a key role in the photochemical reaction because it can release $\cdot\text{OH}$ free radicals under UV radiation.^{24–26} When the H₂O₂ concentration is low, the increase of H₂O₂ concentration may further enhance reactions 4–7, finally increasing the NO removal efficiency.^{29,30} On the other hand, the further increase of the H₂O₂ concentration also may cause several side reactions 8 and 9, in solutions, leading to the great self-loss in $\cdot\text{OH}$ free radicals.^{24,25}



Therefore, the further great increase of H₂O₂ concentration only causes a small impact on the NO removal efficiency.

3.3. Effects of Initial pH Values. The effects of the solution initial pH value on NO and SO₂ efficiencies were studied, and the results are shown in Figure 4. It can be seen that SO₂ achieved 100% removal under different solution initial pH values. The NO removal efficiency increases with the increase of solution initial pH. The effects of solution initial pH on NO removal efficiency can be explained by the following several reasons. On the one hand, in alkaline media, several side reactions, such as the following reactions 10–12, can occur.^{24,25,29,30}



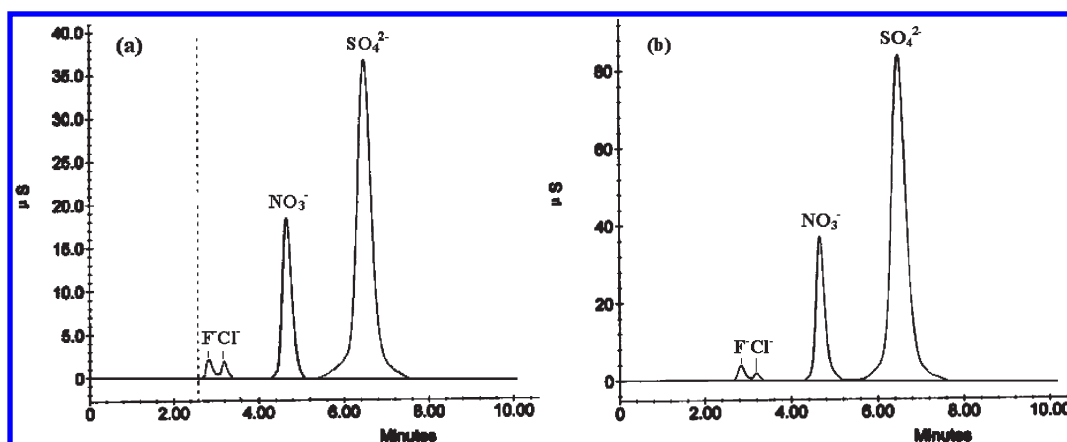


Figure 8. Ion chromatograms of ions in solution at 20 min (a) and 40 min (b). **Conditions:** H₂O₂, 2.0 mol/L; initial pH, 3.2; solution temperature, 298 K; liquid layer height, 15.9 cm.

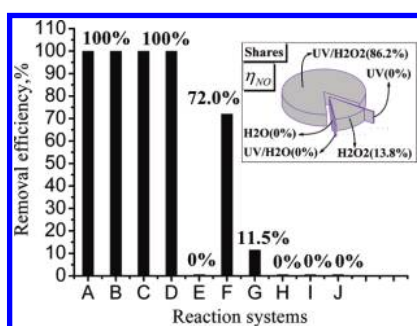


Figure 9. Removal efficiencies of NO and SO₂ in different reaction systems. **Conditions:** Solution initial pH, 3.2; solution temperature, 298 K; liquid layer height, 15.9 cm; H₂O₂ concentration, 2.0 mol/L. **Reaction systems:** A, SO₂–UV/H₂O₂ AOP; B, SO₂–H₂O₂; C, SO₂–UV/H₂O; D, SO₂–H₂O; E, SO₂–UV; F, NO–UV/H₂O₂ AOP; G, NO–H₂O₂; H, NO–UV/H₂O; I, NO–H₂O; and J, NO–UV.

First, from the side reactions 10–12, we can see that the increase of solution initial pH will increase the yield of HO₂⁻ by enhancing the hydrolysis reaction of H₂O₂. HO₂⁻ is an extremely effective scavenger of ·OH free radicals, and it can consume ·OH free radicals, finally decreasing the effective utilization rate of ·OH free radicals.^{24,25,29,30} Second, it is reported that H₂O₂ solution will have a stronger oxidation ability at lower solution pH.^{22,29} So the increase of solution initial pH will diminish the oxidation ability of H₂O₂ to NO. Furthermore, the HO₂⁻ also can enhance self-decomposition of H₂O₂ to produce H₂O and O₂, finally reducing the effective utilization rate of H₂O₂.^{15,29,30} Hence, the increase of solution initial pH will be detrimental to the removal of NO and the effective utilization of H₂O₂.

On the other hand, with the increase of solution initial pH, OH⁻ can cause an acid–base neutralization reaction.³¹ The consumption of neutralization absorption of OH⁻ to H⁺ which produced by eqs 2, 4, 6, and 7 can enhance the shift of absorption reactions to the right, finally increasing the gas–liquid reaction absorption rate. So the increase of solution initial pH is beneficial for the removal of NO. In summary, although the removal process is simultaneously controlled by these positive and negative factors, here the latter positive factors may play a leading role.

3.4. Effects of Solution Temperatures. The change of solution temperature has little effect on SO₂ removal efficiency, but it

has a slight negative impact on NO removal efficiency. As shown in Figure 5, the SO₂ still has complete removal under all solution temperatures. However, when the solution temperature increases from 288 to 348 K, the NO removal efficiency almost linearly decreases from 71.0% to 64.2%. The effect of solution temperature on the NO removal efficiency can be explained by the following two reasons. On the one hand, the increase of solution temperature can increase the chemical reaction rate, finally increasing the NO removal efficiency.^{29,30} On the other hand, with the increase of solution temperature, the solubility of NO in H₂O₂ solution decreases, finally reducing the NO removal efficiency.¹⁵ The two reasons jointly dominate the removal process, but the effect of the latter may be larger than that of the former. So the NO removal efficiency decreases with the increase of solution temperature.

Furthermore, it also could be seen that the solution temperature only had a small impact on the NO removal efficiency as a whole. It is well-known that as the activation energies of free radical reactions are very low, and even the reaction activation energies between free radicals are close to 0, according to Arrhenius law, the change of solution temperature only has a small effect on the photochemical reactions.^{29,30}

3.5. Effects of Liquid Layer Height. As shown in Figure 6, under different liquid layer heights, the SO₂ still has complete removal, but the NO removal efficiency changes under different liquid layer heights. When the liquid layer height increases from 5.3 to 15.9 cm, the NO removal efficiency increases from 42.0% to 70.4%, but with a further increase of the liquid layer height from 15.9 cm to 31.8 cm, the NO removal efficiency has only a slight increase.

There are two main reasons to explain the results. On the one hand, with the increase of the liquid layer height, the residence time of pollutants in reactor increases; in other words, the pollutants will have more adequate reaction time to be oxidized and removed in reactor, being beneficial for increasing the NO removal efficiency. On the other hand, as the reaction rate between ·OH and NO is very fast,²⁸ the removal process of NO using UV/H₂O₂ AOP may be a fast reaction. When the residence time or the reaction time becomes enough, the control step of gas–liquid mass transfer–reaction process may transfer from chemical reaction control to mass transfer control. At this time, enhancing the mass transfer process may become more effective than further lengthening the residence time.³²

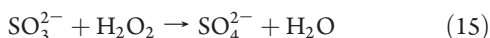
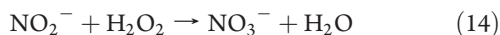
3.6. Determination of Reaction Products. The experiments on determination of gaseous and liquid reaction products were carried out using ion chromatography and a gas analyzer, respectively, and the results are shown in Figures 7 and 8.

As shown in Figures 7 and 8, the potential and noxious gaseous byproduct, NO_2 , is not found, and the SO_2 also achieves complete removal under all experimental conditions. Many results^{3,15,22,23} show that compared with that of NO , it is much easier for the removal of NO_2 by wet scrubbing in solutions because of the higher solubility of NO_2 . Hence, as the intermediate product, the NO_2 may have been absorbed and removed in the reactor again before it got away from the reactor according to reactions 6 and 13.^{22,23}



Furthermore, compared with the NO of low solubility, the high solubility and the hydrolysis reactions of SO_2 in solutions make it become more advantageous to enter into the liquid phase reaction zone to react with $\cdot\text{OH}$ free radicals and H_2O_2 ,^{15,18,32} so the wet scrubbing of SO_2 is usually much easier than that of the NO under the same conditions.

In addition, as shown in Figure 9, NO_3^- and SO_4^{2-} are the main ion products in the H_2O_2 solutions. The potential and noxious liquid byproduct, NO_2^- and SO_3^{2-} , are not found in the H_2O_2 solutions. The absence of NO_2^- and SO_3^{2-} may attribute to the instability of NO_2^- and SO_3^{2-} in oxidation solutions. The potential intermediate products, NO_2^- and SO_3^{2-} , may be quickly oxidized into more stable NO_3^- and SO_4^{2-} in UV/ H_2O_2 AOP system^{20–23} according to reactions 7, 14, and 15.



Furthermore, it can be seen that F^- and Cl^- in solutions also can be determined. We cautiously believe that F^- and Cl^- may derive from the UV decomposition products of sealing ring made of poly(tetrafluoroethylene) ($\text{CF}_3(\text{CF}_2\text{CF}_2)_n\text{CF}_3$) and pipelines made of polyvinyl chloride (CH_2-CHCl) in the reactor we used, but due to traces, they may have little impact on our results.

Finally, in order to avoid secondary pollution or achieve recovery and utilization of ion products in solutions, it is very necessary that post-treatments of ion products should be considered in the removal process of NO and SO_2 by wet scrubbing using UV/ H_2O_2 AOP. At present, some existing post-treatment methods,^{33–36} such as multistage flash distillation, multi-effect boiling, reverse osmosis, biological denitrification, and the liquid-phase catalytic method, have been developed and applied at full scale for the removal of soluble ions in solutions. These existing post-treatment methods may potentially be used for disposing of the nitric acid and sulfuric acid solutions from the removal process of NO and SO_2 by wet scrubbing using UV/ H_2O_2 AOP, so the removal process will not cause secondary pollution, and even the potential risk and liability of pollution problem may be advantageously converted into assets by improving and using these existing post-treatment processes.

3.7. Reaction Mechanism. On the basis of the known results,^{24–27,29,30} there are three main removal pathways of pollutants using UV/ H_2O_2 AOP, mainly including the excitation removal of UV, the oxidation removal of H_2O_2 , and the oxidation removal of $\cdot\text{OH}$ free radicals. Usually, the oxidation removal of $\cdot\text{OH}$ free radicals plays a leading role among them.

As shown in Figure 9, the NO only achieves a removal efficiency of 11.5% in the single H_2O_2 system. When the single UV system, the single H_2O system, and the single UV/ H_2O system are used alone to remove the NO , respectively, there is almost no NO removed, but using the UV/ H_2O_2 AOP system achieves an NO removal efficiency of 72.0%. The results show that H_2O has almost no absorption ability of NO , and the UV decomposition removal of NO also fails to occur in the removal process of NO . The oxidation removal of $\cdot\text{OH}$ free radicals plays a leading role (it has a share of 86.2%) and the oxidation removal of H_2O_2 only plays a secondary role (it only holds a share of 13.8%) in the removal of NO by wet scrubbing using UV/ H_2O_2 AOP. In addition, the analysis results of ion chromatography also show that NO and SO_2 were mainly removed by oxidation, and the NO_3^- and the SO_4^{2-} are the final ion products in the H_2O_2 solutions. In summary, on the basis of our results, although several other side reactions can also possibly occur in the solutions, the main removal path of NO and SO_2 can be briefly illustrated by eqs 2–7 and 16–18.^{9,10,21,28}



Finally, more research remains necessary due to the complexity of the removal mechanism of NO and SO_2 by wet scrubbing using UV/ H_2O_2 AOP.

4. CONCLUSIONS

The photochemical oxidation removal process of NO and SO_2 from simulated flue gas of coal-fired power plants by wet scrubbing using the UV/ H_2O_2 advanced oxidation process was studied in a semicontinuous and small-scale ultraviolet (UV)-bubble column reactor. Some useful conclusions are obtained as follows:

- (1) Under all experimental conditions, the SO_2 was completely removed. With the turning on and the turning off of the UV lamp, the NO removal efficiency has a great change, indicating that there is a significant cooperative effect between UV and H_2O_2 .
- (2) With the increase of H_2O_2 concentration and liquid layer height, the NO removal efficiency greatly increased at first, and then the growth rate of NO removal efficiency gradually became smaller.
- (3) The NO removal process was enhanced observably by the solution initial pH value, but it was inhibited slightly by the solution temperature. The NO and the SO_2 were mainly removed by oxidation, and the NO_3^- and the SO_4^{2-} were the final ion products in remaining solutions.
- (4) The potential and noxious gaseous byproduct, NO_2 , is not found in exhaust. The potential and noxious liquid byproduct, NO_2^- and SO_3^{2-} , are also not found in the H_2O_2 solutions. Furthermore, some existing post-treatment methods may potentially be used for disposing of the nitric acid and sulfuric acid solutions, so the removal process of NO and SO_2 by wet scrubbing using UV/ H_2O_2 AOP will not cause secondary pollution.
- (5) In order to know the SO_2 reaction characteristics in depth, the SO_2 absorption process in UV/ H_2O_2 AOP system should be further studied alone by modifying the

optimized design of the bubble column and optimizing the operating conditions. Furthermore, more research works remains to be carried out due to the complexity of the removal mechanism of NO and SO₂ by wet scrubbing using UV/H₂O₂ AOP.

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