

Study of a photocatalytic oxidation and wet absorption combined process for removal of nitrogen oxides

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Abstract

A two-stage process incorporating photocatalytic oxidation (PCO) and wet absorption was developed in this study for the treatment of nitrogen oxides (NO_x). At first, nitric oxide was converted to nitrogen dioxide by PCO. Then the nitrogen oxides were absorbed effectively in a wet scrubber by using sulfite solution as an absorbent. The experimental results showed that the removal efficiency of NO_x by this combined process could reach 75%, when the inlet concentration of nitrogen oxides was 240 ppm (200 ppm NO). It was also observed that NO_x removal efficiency had a maximum value when the absorbent concentration (Na₂SO₃) was 2 wt%. The addition of Na₂S₂O₃ could effectively retard the excessive oxidation of sulfite by oxygen. 0.5 wt.% Na₂S₂O₃ was considered the suitable inhibitor concentration. The main products of this combined process were nitrite and nitrate.

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1. Introduction

The emission of NO_x into the atmosphere is a great environmental concern because of its detrimental effects (e.g., acid rain, photochemical smog and greenhouse effects) on mankind and various ecosystems. The components of NO_x emissions from fuel combustion are mainly NO as well as small quantities of NO₂. Various processes, including combustion modifications, dry processes and wet processes have been developed to remove NO from flue gases [1–9].

Traditional wet processes need lower investment and operation costs compared to other flue gases treatment methods. NO₂ can be effectively absorbed in some aqueous solutions such as sulfite, while NO cannot be removed by wet processes because of its low solubility and reactivity in aqueous solutions [10–13]. There is particular interest in sulfite because it is produced by the traditional desulfurization scrubbers and reacts with NO₂ through a free radical mechanism. Thus, NO oxidation is a crucial step for the aqueous absorption of NO_x.

The slow oxidation rate of NO in air can be improved by the addition of a strong oxidizing agent such as ozone (O₃) [14], chlorine dioxide (ClO₂) [15], hydrogen peroxide (H₂O₂) [16] or chlorine (Cl₂) [17]. However, the addition of a strong oxidizing agent will increase the operation cost and the potential leakage of the oxidizing agent will bring new environmental problem. Thus, there is a need to develop an environmentally friendly and cost-effective oxidation method for the oxidation of NO.

Photocatalytic oxidation (PCO) may be a promising approach to convert NO to NO₂. Heterogeneous photocatalysis has been shown to oxidize a wide range of contaminants in recent years [18,19]. Also, a lot of studies have been done with concerning the PCO of NO_x in the ambient atmosphere (either outdoor or indoor air), using TiO₂ as a photocatalyst [20–23]. Furthermore, Devahasdin et al. [24] reported the transient behavior and reaction kinetics on the PCO of NO and discussed the feasibility for the treatment of NO_x from flue gases. Recently, Barman and Philip [25] reported an integrated system for the treatment of nitrogen oxides from flue gases, using PCO or ozone oxidation as a gas phase oxidation section coupled with a biofilter system.

In this study, the removal of NO_x by a two-stage process incorporating photocatalytic oxidation (PCO) and wet absorption was investigated. TiO₂ nano-particles coating on woven glass fabric was used as the photocatalyst and sodium sulfite was

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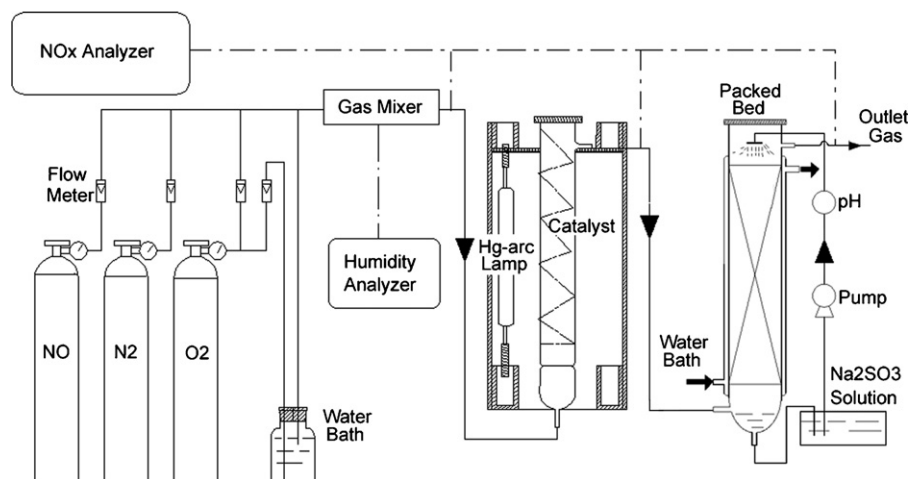


Fig. 1. Diagram of the combined process for nitrogen oxides treatment.

used as the absorbent. The performance of the combined process was evaluated under a simulated flue gases. The relationship between photocatalytic oxidation and removal efficiency was discussed. The ions concentrations in the aqueous absorbent were determined to investigate the reaction pathway and final products. Finally, an attempt to inhibit sulfite excessive oxidation by oxygen was carried out.

2. Experimental

2.1. Preparation of TiO_2 catalysts

Degussa P25 (Degussa Co. Ltd., Germany) was used as the photocatalyst. Immobilization was carried out by the dip-coating method. Five grams of TiO_2 was first mixed with 100 mL deionized water to prepare slurry. Then the woven glass fabric (4 cm × 80 cm, pretreatment: 500 °C, 1 h) was dip-coated with the TiO_2 slurry. The loaded fabric was dried at 100 °C for 1 h in an oven. This procedure was repeated three times, and the final immobilized catalyst was then dried at 100 °C for 24 h. The typical catalyst loading achieved by this approach was $>1.0 \text{ mg cm}^{-2}$.

2.2. Experimental set up

The diagram of the experimental set up is shown in Fig. 1, which included the gas supply, photocatalytic oxidation reactor, wet scrubber system and analytical system.

The NO (Jingong Gas, China, 10,000 ppm), N_2 and O_2 were mixed to obtain the desired concentration of NO_x (200 ppm NO, 240 ppm NO_x). The immobilized catalyst was set into a Pyrex reactor with “Z” type. The Pyrex reactor (volume: 340 mL; length: 30 cm) was irradiated with one Hg-arc lamp (500 W, Philips; the maximum light intensity: 365 nm; distance to reactor: 6 cm) outside. The Pyrex reactor and light source were set in a hollow chamber that was coated with tinfoil. The temperature of the reactor and the lamp were lowered by a fan. The reaction temperature in the reactor was held at 80 ± 5 °C, heated by the irradiation of the Hg-arc lamp. NO, NO_2 and O_2 were

measured by a Kane International Limited Model KM-9106 flue gas analyzer. The relative humidity was determined by a relative humidity analyzer (Testo Co. Ltd., Model 605-H1).

The aqueous absorption section consisted of a packed-bed column scrubber unit made from Pyrex, and a solution-recycle system. The packed tower was a 60 cm long and 4 cm diameter column with a 50 cm section packed with 4 mm glass bleedings. The packed tower was linked with a vessel which served as the scrubbing solution reservoir. A peristaltic circulating pump (Baoding Longer Precision Pump Co. Ltd., BT01-100) was used to pump the absorbent from the reservoir. The recirculation rate of the absorption solution was 3 L h^{-1} . NO_2^- and NO_3^- in the absorption solution were studied by an ion chromatogram (Dionex 500), SO_3^{2-} and $\text{S}_2\text{O}_3^{2-}$ were determined by classical chemical analysis [13].

The NO oxidation efficiency and the NO_x removal efficiency are evaluated according to Eqs. (1) and (2).

$$\text{Oxidation efficiency} = \left(\frac{[\text{NO}]_{\text{inlet,PCO}} - [\text{NO}]_{\text{outlet,PCO}}}{[\text{NO}]_{\text{inlet,PCO}}} \right) \times 100 \quad (1)$$

$$\text{Removal efficiency} = \left(\frac{[\text{NO}_x]_{\text{inlet}} - [\text{NO}_x]_{\text{outlet}}}{[\text{NO}_x]_{\text{inlet}}} \right) \times 100 \quad (2)$$

3. Result and discussion

3.1. PCO of nitrogen oxides

The PCO reaction was started after the gas concentration was constant at the gas inlet and the gas outlet. Fig. 2 shows the PCO behavior of nitrogen oxides in 10 h irradiation. It indicates that the reaction approached a steady state after 60 min of irradiation. It was found that the nitrogen mass equilibrium was reached at the steady state: $\text{NO}_{\text{out}} + \text{NO}_{2,\text{out}} = \text{NO}_{\text{in}} + \text{NO}_{2,\text{in}}$, which indicated that the main product of PCO was nitrogen dioxide and

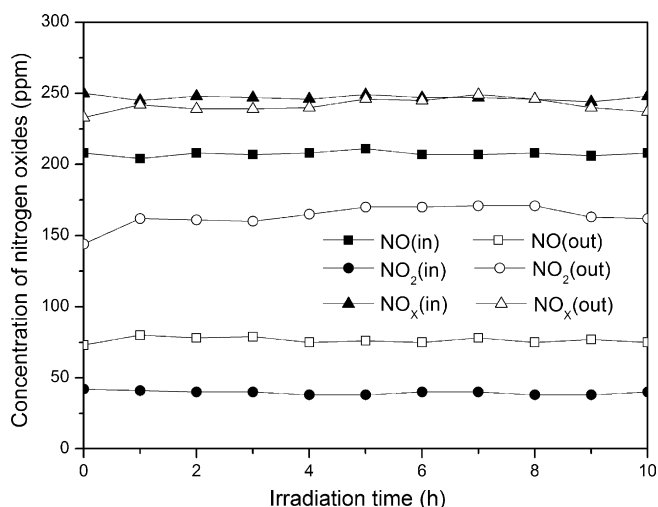


Fig. 2. Photocatalytic oxidation behavior of NO_x in 10 h of irradiation (relative humidity, 70.0%; reaction temperature, 80 ± 5 °C; space time, 10 s).

that no other products were produced during this process. The oxidation efficiency of PCO was 63.9% at the steady state. The PCO of NO consisted of a series of oxidation steps by OH[•] and O₂⁻: NO → HNO₂ → NO₂ → HNO₃. If adequate HNO₃ was adsorbed on the catalyst surface, the oxidation of NO terminated at the step of NO₂ [24,26].

The deactivation test of the photocatalyst was divided into five separated reaction parts and the duration of each part was 10 h, shown in Fig. 3. In the first part, the oxidation efficiency had an initial decline and then reached a steady state. This phenomenon might be attributed to the high initial rate of adsorption plus the reaction of NO. In the subsequent parts, the oxidation efficiency showed little decline in the test time. It was due to the slow accumulation of nitrate species on the catalyst surface which might have deactivated the catalyst. This slow accumulation rate might be the result of the high reaction temperature (80 ± 5 °C) in the experiment. All these results proved that PCO was an effective and promising method for the oxidation of NO.

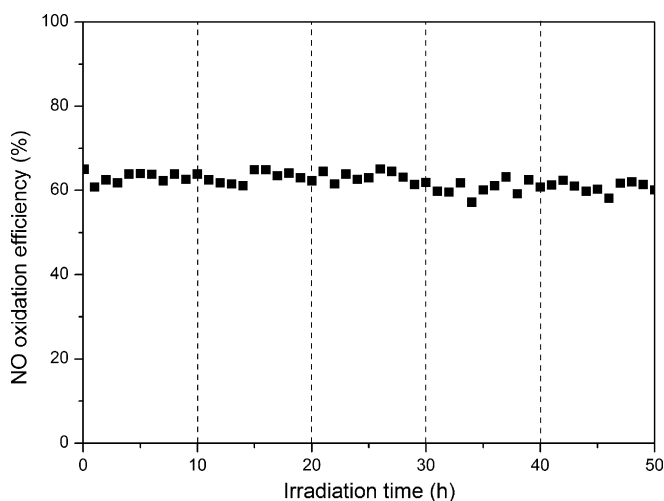
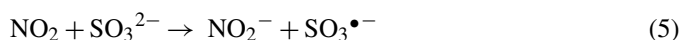
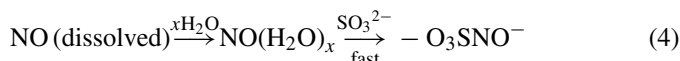


Fig. 3. Photocatalyst deactivation test in 50 h of irradiation (relative humidity, 70–75%; reaction temperature, 80 ± 5 °C; inlet NO concentration, 200 ppm; space time, 10 s).

3.2. Removal of nitrogen oxides by absorption

After the PCO of NO_x, the modified exhaust gases were directed to an absorber containing the absorbent. At the inlet of the absorber, the NO_x concentration was maintained at 240 ppm (NO, ca. 80 ppm; NO₂, ca. 160 ppm). Removal efficiencies of NO, NO₂, and NO_x were calculated with flue gases after the PCO section as the gas source.

The absorbent in the absorber was sodium sulfite (Na₂SO₃), and the reaction between NO, NO₂ and the absorbent is as follows [13,27,28]:



As mentioned in Section 1, the absorption of NO was greatly restrained because of its low solubility. The relationship between the fraction of NO/NO_x and NO_x removal efficiency was investigated and the results are shown in Fig. 4. As observed, the NO_x removal efficiency increased quickly with the increase of NO₂ content in the exhaust gases. Without the oxidation step, the NO_x removal efficiency by sodium sulfite was less than 10%. After the PCO, the highest removal efficiency reached 72.6%. Obviously, NO should be converted into NO₂ for effective removal.

Fig. 5 shows the variations of SO₃²⁻ concentration, NO₂⁻ concentration, NO₃⁻ concentration, pH value and NO_x removal efficiency with time when 2 wt% Na₂SO₃ was used as absorbent. Fig. 5a indicates that the NO_x removal efficiency had little decline during first 60 min of absorption. After that, the declining rate was accelerated. After 120 min of reaction, the NO_x

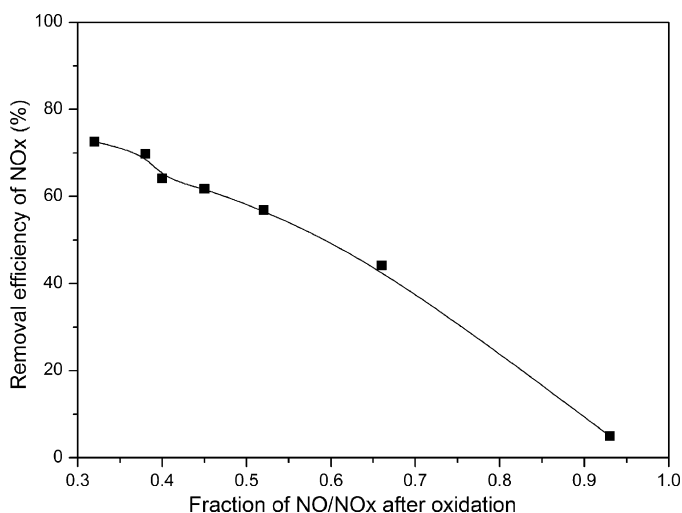


Fig. 4. Relations between oxidation state and removal efficiency (sodium sulfite concentration, 2 wt%).

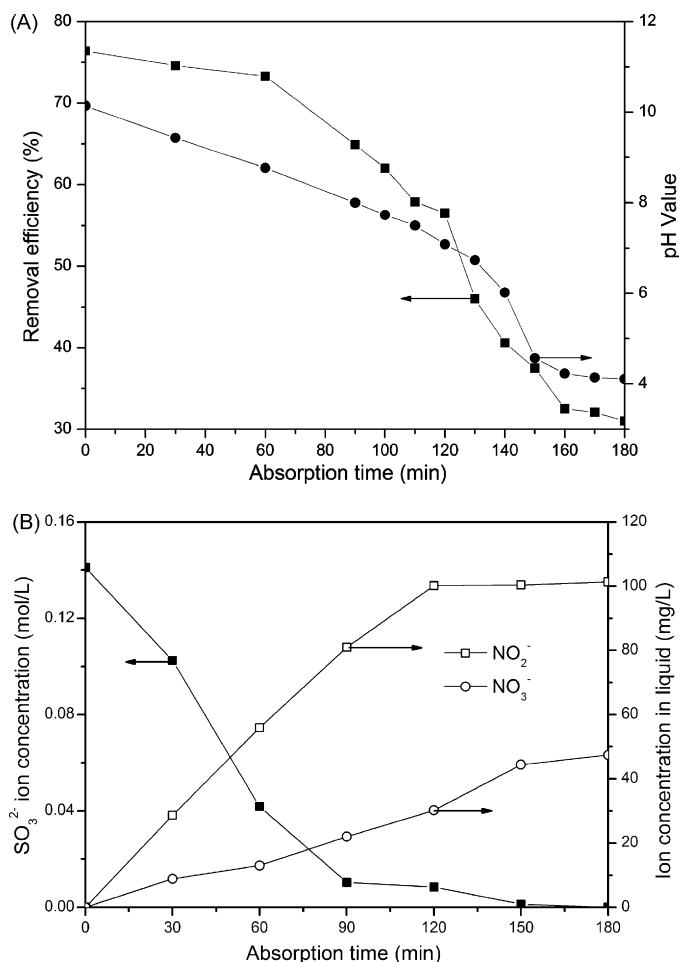


Fig. 5. The variations of SO_3^{2-} , NO_2^- , NO_3^- , pH value and NO_x removal efficiency in 3 h of absorption (absorbent, 2 wt% Na_2SO_3).

removal efficiency declined quickly from 60% to 30% in 20 min. It also showed that the pH value declined with the absorption time, and that the pH value had dramatic decline when pH value decreased below 7.0. SO_3^{2-} concentration variation in the liquid phase which was given in Fig. 5b verified this phenomenon. Sulfite ions in the liquid phase were consumed very fast in the first 90 min of absorption. After 90 min of absorption, the concentration of sulfite ions in liquid phase was very low and the rate of consumption began to decrease. The depletion of the absorbent took place after 150 min absorption.

It was found that the sulfite ion oxidation rate has a linear relation to oxygen concentration [27]. It was also reported that NO_2 reacted with sulfite through a free radical chain reaction mechanism, that one mole of NO_2 absorbed could lead to the oxidation of 4 or more moles of sulfite in the presence of oxygen [11,27,29]. The high concentration of oxygen was favored for the PCO of NO [24,26]. However, this would lead to an excessive consumption of the absorbent which has no positive effect on the removal of NO_x . The dramatic decline of removal efficiency around pH 7.0 was attributed to the formation of HSO_3^- in the liquid phase. The pK value for the deprotonation of bisulfite is 7.19 (Eq. (9)). In general, free radicals react with SO_3^{2-} more

rapidly than with HSO_3^- [10]



Nitrite and nitrate were analyzed to verify the main products of the absorption reaction. Fig. 5b shows that nitrite ions in the liquid phase increased rapidly with the absorption time, while the content of nitrite ions augmented little after 120 min of absorption. However, the nitrate ion concentration in the liquid phase continued to increase with the absorption time. These results indicated that Eq. (5) was the key reaction in the absorption and that nitrite ions were the main product of absorption. With the decline of pH value, the reaction rates of Eqs. (7) and (8) were accelerated. The depletion of the absorbent and the formation of HNO_3 inhibited the production of nitrite ions.

Fig. 6 shows the influences of sodium sulfite concentrations on the NO_x removal efficiency, ranging from 0.5 wt% to 20 wt% (0.04–1.59 M). It indicates that removal efficiency of nitrogen dioxide reached its highest value (near 100%) when the absorbent concentration was higher than 2 wt%. Meanwhile, the removal efficiency of NO declined after the absorbent concentration was higher than 2 wt%. This result proved that the increase of the liquid-phase reaction rate has little contribution to NO absorption because of its low solubility. It was also suggested that the salting-out effect would be considered when the sulfite concentration was higher than 0.2 M [11]. Therefore, it was considered that the increased absorbent concentration would bring the salt-out effect which would decrease the solubility of gases in the aqueous solution. Hence, NO absorption efficiency declined after the concentration of sodium sulfite was higher than 2 wt%. As for NO_2 , the salting-out effect could be neglected because of its high solubility.

From the study by Shen and Rochelle [13], thiosulfate ($\text{S}_2\text{O}_3^{2-}$) was a strong inhibitor of sulfite oxidation. It was suggested that thiosulfate could act as a free radical scavenger and thus was able to terminate the sulfite oxidation through the following routes:

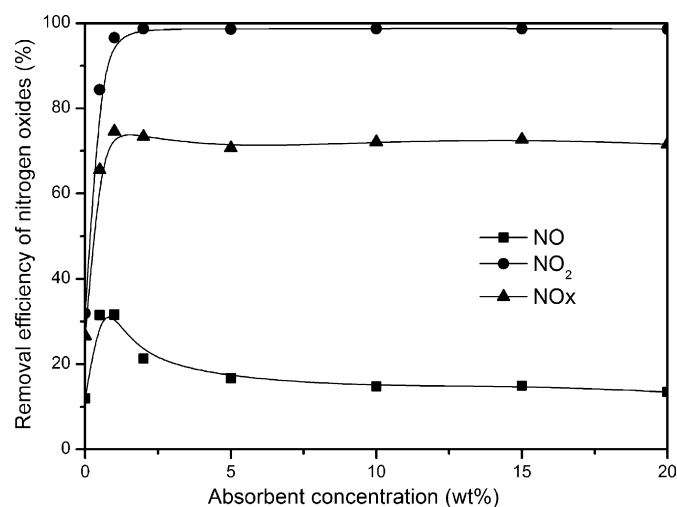
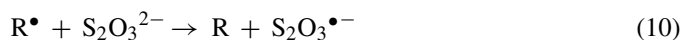


Fig. 6. The influences of absorbent concentration on NO_x removal efficiency.

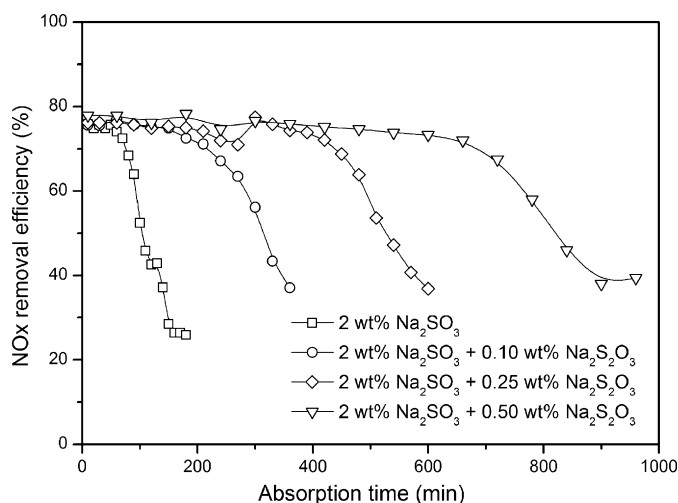
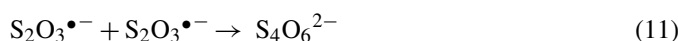


Fig. 7. The effects of inhibitor concentration on NO_x removal efficiency.



The removal efficiency of nitrogen oxides using sodium thiosulfate as an additive was shown in Fig. 7. It indicated that the effective absorption time was prolonged with the augment of the thiosulfate concentration. Unfortunately, high addition concentration of thiosulfate would increase the operation cost of the combined process. Thus 0.5 wt% addition concentration of thiosulfate could be a suitable value.

Fig. 8 shows that the NO_x removal efficiency and the pH value had little decline after 180 min of absorption. Furthermore, the pH value had a slight initial increase and then decreased with time. Fig. 9 shows that the consumption rate of sulfite ion decreased greatly compared to the absorption without the addition of Na₂S₂O₃. As seen, the consumption of S₂O₃²⁻ ions was also very low in 3 h absorption. This proves that the addition of Na₂S₂O₃ inhibited the excessive oxidation of SO₃²⁻ by oxygen and retained the removal efficiency of NO_x effectively.

Fig. 9 shows the variation of NO₂⁻ concentration and NO₃⁻ concentration in the liquid phase together. The increase rate

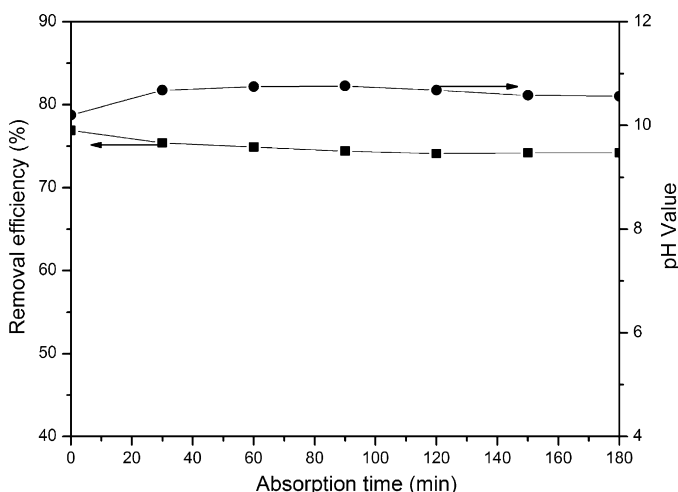


Fig. 8. The variations of pH value and NO_x removal efficiency in 3 h of absorption (absorbent, 2 wt% Na₂SO₃ + 0.5 wt% Na₂S₂O₃).

of nitrite ion concentration increased quickly with the absorption time, while the nitrate ion concentration in the liquid phase showed a little increase during the continuing reaction. This phenomenon confirmed that the formation of nitrate ion was slow under high pH value. It verified that the nitrite ion was the main product of this two-stage process.

In general, with this two-stage process, the nitrogen oxides could be removed effectively. Also, the combined process offered the following advantages: (1) the environmentally friendly and economical gas-phase oxidation; (2) no extra reactants such as NH₃ or O₃ required. However, a number of salts in the absorption solution, such as nitrite, nitrate and nitrogen-sulfur compounds were formed [30]. Thus, appropriate treatment methods might be necessary for the exhausted solutions that contain high levels of sulfate, nitrite, nitrate, nitrogen-sulfur compounds and so on. Fortunately, the treatments of nitrite, nitrate and nitrogen-sulfur compounds in the aqueous solution have been studied and many methods could be selected, such as aqueous oxidation [32], liquid-phase catalysis [31], evaporation (or distillation), ion exchange, biological denitrification, and so on [32,33]. Furthermore, the best treatment method of nitrite and nitrate was to have them removed from the scrubbing liquor as alkaline-metal salt precipitates for industrial reuse.

Overall, this combined process only required one additional unit (PCO section) in traditional wet scrubbers for the NO_x removal, and the by-products of wet desulfurization (sulfite salt) could be used as the absorbent for the absorption of NO₂. Therefore, it would be a promising method to control NO_x emissions from flue gases.

4. Conclusion

A combined process that incorporated the PCO and wet absorption to remove nitrogen oxides from flue gases was developed. The reaction mechanism and the effects of operation conditions were then carried out. The following conclusion can be drawn:

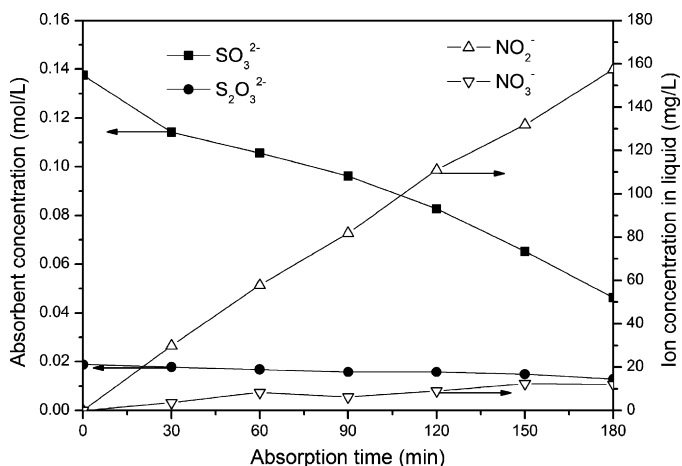


Fig. 9. The variations of SO₃²⁻, S₂O₃²⁻, NO₂⁻, and NO₃⁻ concentration in 3 h absorption (absorbent, 2 wt% Na₂SO₃ + 0.5 wt% Na₂S₂O₃).

- (1) The combined process proposed in this study was effective for the removal of NO_x. The removal efficiency of NO_x for the combined process could reach around 75%, when the inlet concentration of nitrogen oxides was 240 ppm (200 ppm NO).
- (2) At the steady state, the main product of PCO was nitrogen dioxide and no other products were formed during this process. The observed photocatalyst deactivation was not evident after 50 h of reaction because of the low accumulation rate of nitrate species on the catalyst surface. It proved that PCO was an effective and promising method for the oxidation of NO.
- (3) For the removal of NO_x, the considered absorbent concentration (Na₂SO₃) was 2 wt%. The removal efficiency of NO declined after the absorbent concentration was higher than 2 wt%. This decline was attributed to the salt-out effect brought by the increased absorbent concentration.
- (4) Oxygen needed for the PCO of NO would lead to an excessive consumption of the absorbent, since the rate of depletion of the absorbent was much faster than that expected by reaction stoichiometry. The addition of Na₂S₂O₃ could retard the excessive oxidation of sulfite by oxygen, and 0.5 wt.% Na₂S₂O₃ was considered a suitable inhibitor concentration.

Acknowledgements

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