



Wet oxidation and absorption procedure for NO_x removal

Jiaorong Yan, Feixiang Zhou, Ying Zhou, Xianghao Wu, Qjulian Zhu, Huayan Liu, Hanfeng Lu*

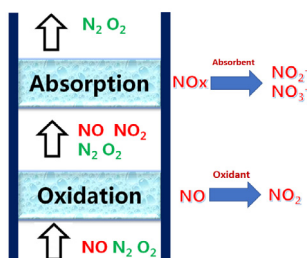
Innovation team of air pollution control, Institute of Catalytic Reaction Engineering, College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310014, China



HIGHLIGHTS

- A methodology is presented to remove NO_x with low oxidation degree, low temperature and high vapor content at room temperature.
- The method is considered potential low-cost proposal and have advantage of simple equipment and low operating temperature.

GRAPHICAL ABSTRACT



An interesting and universal method to remove NO_x with low oxidation degree, low temperature and high vapor content by liquid oxidation and liquid absorption at room temperature.

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ABSTRACT

The removal of NO_x with low oxidation degree, low temperature, and high vapor content from small coal-fired boiler emission through selective catalytic reduction is difficult. This study investigated liquid oxidation and absorption to remove NO_x systematically. Thermodynamic calculation showed that the equilibrium constants of the NO oxidation and NO_x absorption reaction are high; thus, NO_x treatment by liquid oxidation and absorption is feasible. Four oxidants (NaClO₂, NaClO, H₂O₂, and KMnO₄) and four absorbents (Ca(OH)₂, CaCO₃, NaOH, and Na₂CO₃) were used to study the reaction mechanism of the oxidation and absorption processes. Results show that NaClO₂ solution is the best oxidant, and the optimum pH value of the oxidation reaction is 5–6. The oxidation degree reaches 100% when the concentration of the NaClO₂ solution is 1.0%, and the oxidation degree can still reach approximately 50% when the concentration is 0.1%. Ca(OH)₂ slurry has the best absorption performance among the four kinds of absorbents. The NO_x removal efficiency of Ca(OH)₂ slurry can reach approximately 70% when the oxidation degree is 50%. The removal efficiency is up to 80% when the oxidation degree is 100%.

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* Correspondence to: Zhejiang University of Technology, 18# Chaowang Road, Hangzhou 310014, China.
E-mail address: luhf@zjut.edu.cn (H. Lu).

0. Introduction

NO_x ($\text{NO} + \text{NO}_2$) and SO_2 , as the major gas-phase pollutants emitted from coal-fired boiler, raise significant environmental problems, such as acid rain and smog (Adewuyi et al., 1999). NO_x causes ozone damage and photochemical smog. Thus, numerous denitrification (de- NO_x) and desulfurization (de- SO_2) technologies have been investigated to abate NO_x and SO_2 emissions. Currently, flue gas desulfurization (FGD) technology is the most effective and widely used method for SO_2 control; however, the de- NO_x technology remains to be studied (Wang and Zhong, 2016).

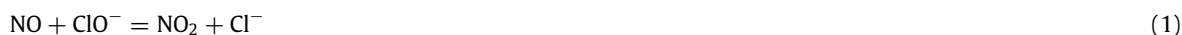
For NO_x control, selective catalytic reduction (SCR) (Liu and Ihl Woo, 2006), selective non-catalytic reduction (Bae et al., 2006), wet absorption (Joshi et al., 1985), adsorption (Mok et al., 2003), and electron beam irradiation (Person and Ham, 1988) processes have been developed; among these processes, SCR is considered the best available technology (Radojevic, 1998). SCR has been widely used for its high de- NO_x efficiency, but problems, such as corrosion of NH_3 , catalyst toxicity, secondary pollution, and high operation temperature (approximately 300 °C), are inevitable (Muzio et al., 2002). Thus, SCR is unsuitable in certain cases, such as low-temperature outlet gas from small coal-fired boiler. Wet absorption is a potential low-cost alternative to its simple equipment and particularly low operating temperature, reaching even below the dew point, which is suitable for low-temperature de- NO_x after the FGD process. However, NO comprises more than 90% of NO_x in the flue gas from coal-fired boiler, and the low solubility of NO in aqueous solution appreciably increases the liquid-phase resistance to mass transfer (Walker et al., 1937). Moreover, oxidants are used to first convert the relatively water-insoluble NO to reactive NO_2 , which can be further removed by absorbents to increase the overall removal efficiency of NO_x .

In absorption-based methods, knowledge on the process variables and their influence on oxidation and absorption capacities is required to maximize NO_x removal efficiency by pre-decided oxidants and absorbents. This study aims to establish test data for the design and operation of the de- NO_x absorption system.

1. Thermodynamic calculation for NO oxidation and absorption

NO oxidants can be divided into liquid- and gas-based oxidants by reaction place. Gas-based oxidants include ozone (Lin et al., 2016) and chlorine dioxide (Hoigné and Bader, 1994). The drawbacks of gas-based oxidants (O_3 and ClO_2) include leaking and high prices; however, liquid oxidants can avoid these problems. Commonly used liquid oxidants include sodium hypochlorite (Chen et al., 2005), sodium chlorite (Chien et al., 2003; Deshwal et al., 2008; Guo et al., 2013), hydrogen peroxide (Thomas and Vanderschuren, 1996; Liémans and Thomas, 2013; Bhanarkar et al., 2014), and potassium permanganate (Brogren et al., 1997; Chu et al., 1998, 2001).

NO in the flue gas is oxidized to NO_2 by the following reactions (Deshwal et al., 2008).



The thermodynamic calculation of Eqs. (1)–(4) is as follows. **Table S1** (Supporting information) displays the thermodynamic data of related substances (Dean, 1979).

$$\Delta_r H_m(T) = \Delta_r H_m^\theta(298.15 \text{ K}) + \int_{298.15 \text{ K}}^T \Delta_r C_{p,m} dT \quad (5)$$

$$\begin{aligned} \Delta_r G_m(T) &= \Delta_r H_m(T) - T \Delta_r S_m(T) = \Delta_r H_m^\theta(298.15 \text{ K}) - T \Delta_r S_m^\theta(298.15 \text{ K}) \\ &+ \int_{298.15 \text{ K}}^T \Delta_r C_{p,m} dT - T \int_{298.15 \text{ K}}^T \frac{\Delta_r C_{p,m}}{T} dT. \end{aligned} \quad (6)$$

The last two items can be ignored in approximate calculation, that is,

$$\Delta_r G_m(T) = \Delta_r H_m^\theta(298.15 \text{ K}) - T \Delta_r S_m^\theta(298.15 \text{ K}) \quad (7)$$

$$\ln K(T) = -\frac{\Delta_r G_m(T)}{RT} \quad (8)$$

where $\Delta_r H_m^\theta$ (kJ mol^{-1}) is the standard enthalpy change, $\Delta_r G_m^\theta$ (kJ mol^{-1}) is the standard Gibbs free energy change, $\Delta_r S_m^\theta$ ($\text{kJ mol}^{-1} \text{ K}^{-1}$) is the standard entropy change, $\Delta_r C_{p,m}$ ($\text{J K}^{-1} \text{ mol}^{-1}$) is the molar heat capacity at constant pressure, R is the gas constant, and K is the equilibrium constant.

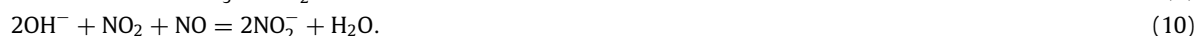
The enthalpy change, Gibbs free energy change, and equilibrium constant at 298.15 K of reactions (1)–(4) were calculated according to Eqs. (5), (7), and (8), respectively. **Table 1a** summarizes the results.

In **Table 1a**, the standard enthalpy change of reactions (1)–(4) is negative, indicating that the four reactions are exothermic reactions, and increasing reaction temperature is unfavorable to product formation. The equilibrium constant of the four reactions is large, thereby indicating that these reactions show a trend.

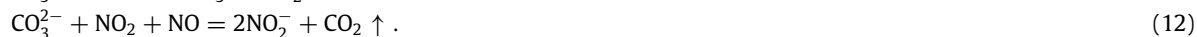
Table 1
Enthalpy change, Gibbs free energy change and equilibrium constant of Reactions at 298.15 K.

	Reaction	$\Delta_r H_m(T)$ (kJ mol ⁻¹)	$\Delta_r G_m(T)$ (kJ mol ⁻¹)	Equilibrium constant (K)
(a)	(1)	-118.17	-130.80	8.2498E+22
	(2)	-216.96	-221.10	5.4601E+38
	(3)	-152.85	-139.34	2.5862E+24
	(4)	-276.84	-287.45	2.2971E+50
(b)	(9)	-201.80	-166.20	1.3145E+29
	(10)	-158.47	-124.90	7.6371E+21
	(11)	-92.32	-110.11	1.9558E+19
	(12)	-48.99	-68.81	1.1362E+12

Alkaline absorption, which is one of the earliest methods for NO_x treatment is simple and consumes minimal energy. NaOH/Ca(OH)₂ can react with NO₂ in NO_x, but not with NO. In general, a certain amount of NO is added into NO_x to adjust the ratio of NO and NO₂; thus, it can be absorbed by lye. NaOH/Ca(OH)₂ and NO₂ may react as follows (Kameoka and Pigford, 1977).



Meanwhile, Na₂CO₃/CaCO₃ and NO₂ may react as follows.



The thermodynamic calculation is expressed in Eqs. (9)–(12). **Table S1** lists the thermodynamic data of related substances (Dean, 1979). The enthalpy change, Gibbs free energy change, and equilibrium constant at 298.15 K of reactions (9)–(12) were calculated according to Eqs. (5), (7), and (8), respectively. **Table 1b** presents the results.

In **Table 1b**, the standard enthalpy change of reactions (9)–(12) is negative, thereby indicating that the four reactions are exothermic. The equilibrium constant of reaction (9) is larger than reaction (10), thus suggesting that the trend of reaction (9) is superior. Moreover, the reaction between lye and NO₂ is reaction (9). The reaction mechanism of NO₂ and alkali is as follows:



The overall reaction obtained by combining the above equations is written as reaction (9). The equilibrium constant of reaction (11) is larger than reaction (12), suggesting that the trend of reaction (11) is superior. The mechanism of the reaction of carbonate with NO₂ in the solution is similar to the reaction of the alkali solution, as shown below.



The overall reaction obtained by combining the above equations is written as reaction (11).

2. Experiments and methods

2.1. Experimental setup

Fig. S1 illustrates the experimental setup used for NO_x removal. The setup is composed of simulated flue gas supply, oxidation absorption, and data analysis systems. The simulated flue gas was obtained by controlled mixing of bottled nitrogen, oxygen, and NO gas using individual mass flow controllers. The reactor was a bubble column of 32 mm inner diameter and 360 mm height. The standard inlet gas composition is as follows: 500 ppm NO, 3.0% O₂, and N₂ balance at a total flow rate of 1.0 L/min. The detailed experimental conditions for the present study are listed in **Table S2**.

2.2. Materials

Standard gases included N₂ (99.99%), O₂ (99.99%), and NO (1.00% balanced with N₂). All gases are products of Jingong Gas Co. Ltd., China. Sodium chlorite (80%; Aladdin, China), sodium hypochlorite (100%; Aladdin, China), potassium permanganate (99.5%; Aladdin, China), hydrogen peroxide (30%; Yonghua Chemical Technology Co., Ltd, China), citric acid (99.5%; Aladdin, China), calcium carbonate (99%; Aladdin, China), calcium hydroxide (95%; Lianhua New Material Technology Co. Ltd., China), sodium carbonate (99.8%; Taicang Meida Reagent Co. Ltd., China), and sodium hydroxide (96%; Xilong Science Co. Ltd., China) were the analytical grade reagents used in the present study.

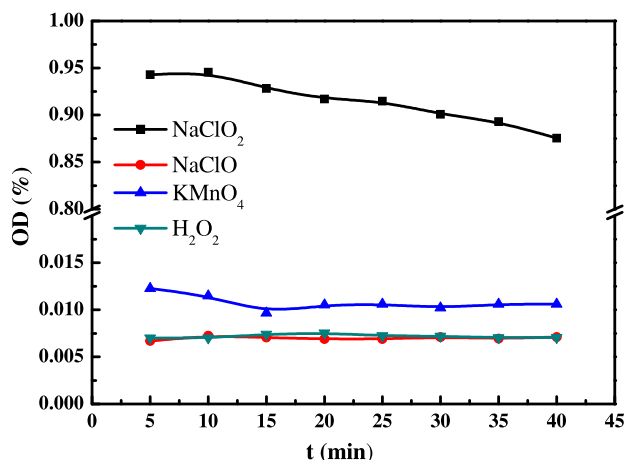


Fig. 1. NO oxidation by different oxidants. Condition: input NO 500 ppm; O₂ content 3.0%; gas flow rate 1.0 L/min; temp. 25 °C.

2.3. Analysis of gases

The inlet and outlet flue gas concentrations were analyzed by flue gas analyzer Testo350 (Model-XL, Testo SE & Co. KGaA, Germany). The oxidant activity was measured by the oxidation degree (%) of NO_x after oxidation reaction; the oxidation degree is calculated by using Eq. (17).

$$OD = \frac{[NO_2]_{out}}{[NO_x]_{out}} \times 100\%. \quad (17)$$

The total NO_x removal efficiency (η , %) is calculated as follows.

$$\eta = \frac{[NO_x]_{in} - [NO_x]_{out}}{[NO_x]_{in}} \times 100\% \quad (18)$$

where $[NO_x]_{in}$ is the inlet concentration of NO_x, $[NO_2]_{out}$ is the outlet concentration of NO₂, and $[NO_x]_{out}$ is the outlet concentration of NO_x.

3. Results and discussion

3.1. Effect of oxidants on NO oxidation and NO_x removal

3.1.1. Screening of oxidants

In this study, NO oxidation reaction was tested by different oxidants, such as NaClO₂, NaClO, H₂O₂, and KMnO₄ solution. Fig. 1 depicts the results of NO oxidation reaction by different oxidants with the same effective oxygen concentration. Effective oxygen concentration refers to the mass fraction of oxygen involved in the redox reactions. The effective oxygen concentrations of oxidants were fixed at 0.17% (w/w); thus, the concentrations of oxidants were 0.50% (w/w), 0.82% (w/w), 0.37% (w/w), and 0.87% (w/w). The initial pH values of NaClO₂, NaClO, and KMnO₄ solutions were adjusted to 5–6 with 0.1% citric acid solution. Furthermore, 0.1% NaOH was added to the H₂O₂ solution, as required, to adjust the pH to 10 because H₂O₂ is oxidative in alkaline conditions.

The results showed that the NaClO₂ solution has better oxidation performance than NaClO, KMnO₄, and H₂O₂. Possibly because the equilibrium constant of NaClO and H₂O₂ that react with NO is smaller than NaClO₂. NO is directly oxidized to NO₃⁻ by KMnO₄ without producing NO₂, as reaction (4); thus, the oxidation degree is low. Thus, the optimum oxidant NaClO₂ solution was used for the subsequent experiment.

3.1.2. Effects of the different pH of NaClO₂ solutions on NO_x oxidation degree and removal efficiency

A set of experiments was made with 0.5% NaClO₂ aqueous solutions at pH values of 4–5, 5–6, 7–8, and 9–10 to determine the effect of pH on NO oxidation and NO_x removal. The original pH of 0.5% NaClO₂ was 7–8. The initial pH values were adjusted with 0.1% citric acid and 0.1% NaOH solution.

In Fig. 2a, the oxidation degree is lower at pH 9–10 than pH 7–8. The result suggests that sodium chlorite has the poor oxidative capability in alkaline medium. The oxidation degree increases before 25 min at pH 7–8; however, the oxidation degree was maintained at approximately 100% when at pH 4–5 and 5–6. This phenomenon indicates that the oxidation reaction of the NaClO₂ solution with NO can be promoted by acid, and the oxidation rate can be accelerated.

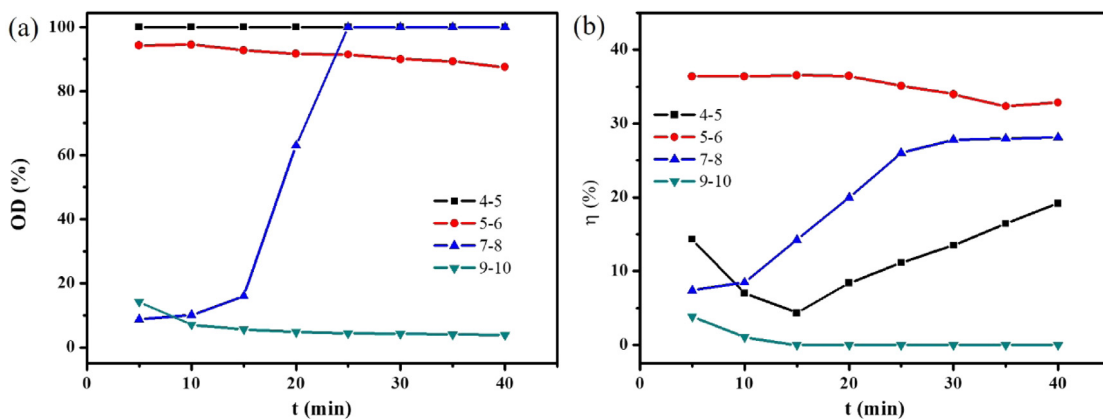


Fig. 2. Effects of different pH of NaClO₂ solutions. (a) on NO oxidation degree; (b) on NO_x removal efficiency. Condition: NaClO₂ concn. 0.5%; NaClO₂ quantity 100 g; input NO 500 ppm; O₂ content 3.0%; gas flow rate 1.0 L/min; temp. 25 °C.

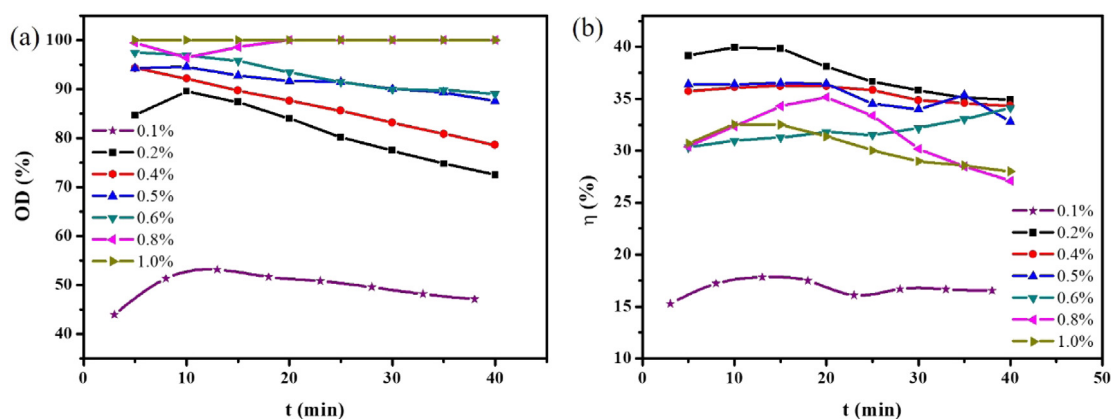


Fig. 3. Effects of different concentration of NaClO₂ solutions. (a) On NO oxidation degree; (b) on NO_x removal efficiency. Condition: NaClO₂ quantity 100 g; input NO 500 ppm; O₂ content 3.0%; gas flow rate 1.0 L/min; pH 5–6; temp. 25 °C.

In Fig. 2b, the NO_x removal efficiency of the NaClO₂ solution is lower at pH 9–10 than at pH 7–8, thereby implying that the NaClO₂ solution at alkaline pH is detrimental to the formation of nitrate from NO_x. The NO_x removal of the NaClO₂ solution at pH 4–5 is less efficient than at pH 7–8, suggesting that a robust acid solution is not conducive to NO_x removal. The NO_x removal efficiency of the NaClO₂ solution at pH 5–6 remains stable, thus indicating that the pH 5–6 is suitable.

In summary, the optimal pH value of the NaClO₂ solution was selected as 5–6 considering experimental results and industrial application.

3.1.3. Effect of the concentration of NaClO₂ solution on NO_x oxidation degree and removal efficiency

The NO oxidation by different concentration of the NaClO₂ solution was investigated and compared in Fig. 3a. The NO oxidation to NO₂ depends on the amount of the NaClO₂ solution used. The oxidation degree can reach 100% when the concentration of the NaClO₂ solution is 1.0%, and the oxidation degree is approximately 50% when the concentration is 0.1%. The oxidation degree decreased with decreasing concentration, indicating that the effective oxidant content in the solution also decreases while the solution concentration decreases; thus, the oxidation degree is reduced. The oxidation degree of NO_x constantly decreases when the concentration of the NaClO₂ solution is 0.4%–0.6%. However, the oxidation degree increases in time and then decreases when the concentration is 0.1% and 0.2%; this effect may be attributed to a new oxidant produced at low concentration. The oxidation degree increases while the new oxidant is generated and then decreases while the new oxidant is consumed. The results in Fig. 3a suggest that the oxidation degree can be regulated by varying the concentration of the NaClO₂ solution.

Fig. 3b presents the NO_x removal efficiency of the various concentrations of NaClO₂ solution. The NO_x removal efficiency is less than 20% when the concentration is 0.1%. The maximum value of NO_x removal efficiency is 40%, which is attained when the concentration is 0.2%. In addition, the removal efficiency decreases with increasing NaClO₂ concentration; this result is unexpected. The possible reason is that when NaClO₂ concentration increases, the oxidation degree increases, and the NO₂

Table 2
Parameters of four industrial absorbent.

No.	Absorbent	Concentration (% w/w)	pH	Quantity (g)
1	Ca(OH) ₂	15	9–10	100
2	CaCO ₃	15	5–6	100
3	NaOH	1	13–14	100
4	Na ₂ CO ₃	5	10–11	100

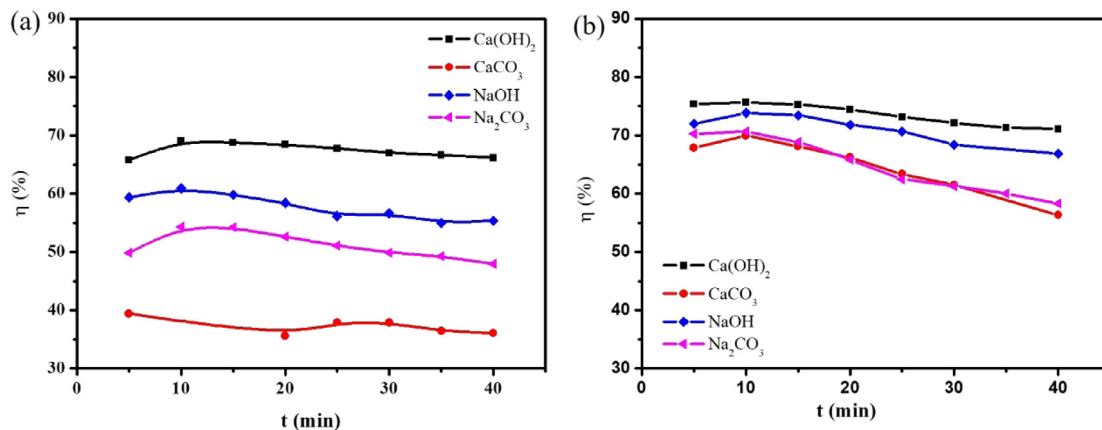


Fig. 4. NO_x removal of different absorbents. (a) When the oxidation degree is about 50%; (b) when the oxidation degree is 75%–90%. Condition: NaClO₂ conc. 0.1%, 0.2%; NaClO₂ quantity 100 g; input NO 500 ppm; O₂ content 3.0%; gas flow rate 1.0 L/min; temp. 25 °C.

content in NO_x increases. However, the NO₂ removal performance of NaClO₂ solution is limited. Therefore, the remaining NO₂ content in NO_x increases with increasing of NaClO₂ concentration, and the removal efficiency decreases with increasing of NaClO₂ concentration.

The results presented above indicate that NO_x oxidation degree can be regulated by adjusting the concentration of the NaClO₂ solution. The absorption properties of the various absorbents were further studied using 0.1%, 0.2%, and 0.6% as the concentrations of oxidants.

3.2. Comparison of the removal performance of the absorbents

The simulated flue gas was first treated with a NaClO₂ solution to produce NO₂, as described above, and then the modified flue gas (NO₂-rich flue gas) was directed to the absorber. Four absorbents, such as Ca(OH)₂, CaCO₃, NaOH, and Na₂CO₃, are investigated. Table 2 lists the parameters of the absorbents involved, which are derived from industrial data.

Fig. S2 depicts the variation in NO_x removal efficiency with the different absorbents at different oxidation degrees. Ca(OH)₂ is the best in the four absorbents because of the good reaction thermodynamics between Ca(OH)₂ and NO₂ and the high concentration of Ca(OH)₂ slurry. The removal efficiency of various absorbents varied significantly at low oxidation degree, as illustrated in Fig. S2, whereas the difference in NO_x removal efficiency decreases when the oxidation degree is below 70%. In this study, the removal efficiency of Ca(OH)₂ slurry can reach up to 75% when the oxidation degree reaches 80%.

Several experiments were performed at NaClO₂ concentrations of 0.1% and 0.2%, as depicted in Fig. 4a and b, respectively, to confirm the removal efficiency of the four kinds of absorbents.

The inlet NO_x oxidation degree of the absorption system is approximately 50%, which agrees well with the results obtained in Fig. 3a, when the concentration of the NaClO₂ solution is 0.1%. The removal efficiencies of the four absorbents are stable when the concentration is 0.1%, as shown in Fig. 4a because the NO oxidation is relatively stable and NO_x oxidation degree is nearly unchanged; thus, the removal efficiency is relatively stable. The NO_x removal efficiency of Ca(OH)₂ when the inlet NO_x oxidation degree is approximately 50% is best, followed by NaOH, and then Na₂CO₃ and CaCO₃, which nearly coincides with the thermodynamic results. However, the removal efficiency of absorbents decreases in time when the concentration is 0.2%, as displayed in Fig. 4b. In the first 10 min, the removal efficiency of all absorbents is 65%–75%; the removal efficiency of the Na₂CO₃ solution and CaCO₃ slurry eventually decreases rapidly, and the removal efficiency of Ca(OH)₂ is approximately 76%.

Among the four absorbents, the NO_x removal efficiency of Ca(OH)₂ slurry is the highest, followed by NaOH solution. Ca(OH)₂ slurry is selected as the absorbent of the system.

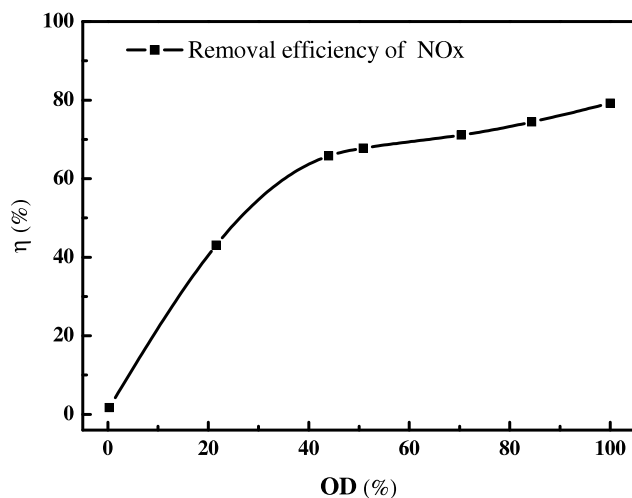


Fig. 5. Effect of NO_x oxidation degree on NO_x removal efficiency of Ca(OH)₂. Condition: NaClO₂ conc. 0.05%, 0.1%, 0.2%; NaClO₂ quantity 100 g; input NO 500 ppm; O₂ content 3.0%; gas flow rate 1.0 L/min; temp. 25 °C.

3.3. NO_x removal of Ca(OH)₂ at different NO_x oxidation degrees

According to the experiment above, Ca(OH)₂ slurry was selected as the best NO_x absorbent. The NaClO₂ solution was used as the oxidant, and then the de-NO_x performance of calcium hydroxide at different oxidation degrees was studied. NO was oxidized by NaClO₂ solution at pH 5–6 with various concentrations of 0.05%, 0.1%, 0.2%, 0.6%, and 1.0% prior to reaction with 15% CaCO₃ slurry.

Fig. 5 illustrates the NO_x removal efficiency of Ca(OH)₂ at different NO_x oxidation degrees. Furthermore, the removal efficiency is 0 when the oxidation degree is 0, thereby indicating that Ca(OH)₂ does not react with NO. The removal efficiency increases with the increase in the oxidation degree because additional NO₂ is absorbed by Ca(OH)₂ slurry. The removal rate increases rapidly when the oxidation degree is 0–50%; however, the removal rate increases slowly when the oxidation degree is more than 50%, indicating that the increase in the oxidation degree has a minimal effect on the removal efficiency when the oxidation degree is higher than 50%. The removal efficiency of Ca(OH)₂ slurry can reach approximately 70% when the oxidation degree is 50%, and the removal efficiency is approximately 80% when the degree oxidation is 100%.

4. Conclusion

(1) Thermodynamic calculation showed that the oxidation reaction of NO and absorption reaction of NO_x were exothermic at room temperature, and the equilibrium constants of the reactions were large.

(2) Among the four oxidants (NaClO₂, NaClO, H₂O₂, and KMnO₄ solutions) with the same effective oxygen concentration, the NO oxidation and NO_x removal of NaClO₂ is the best during the oxidation process. The best pH value of the NaClO₂ solution is 5–6. The oxidation degree can reach 100% when the concentration of the NaClO₂ solution is 1.0%, and a 50% oxidation degree can still be attained when the concentration of NaClO₂ solution is 0.1%.

(3) In the four kinds of absorbents (15% Ca(OH)₂ slurry, 1% NaOH solution, 15% CaCO₃ slurry, and 5% Na₂CO₃ solution), Ca(OH)₂ slurry has the best absorption performance during the wet absorption process. The removal efficiency of NO_x in Ca(OH)₂ slurry can reach approximately 70% when the oxidation degree of NO_x is 50%. The removal efficiency is up to 80% when the oxidation degree is 100%.

(4) A system with a low concentration of the NaClO₂ solution as oxidant and 15% Ca(OH)₂ slurry as absorbent was proposed based on the aforementioned experimental results. The NO inlet concentration is 500 ppm, the flue gas rate is 1.0 L/min, and the NO_x in the simulated exhaust gas can be reduced to less than 100 ppm when the molar fraction of O₂ is 3.0%.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found online at <https://doi.org/10.1016/j.eti.2018.03.006>.

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