

## Removal of NO from flue gas by aqueous chlorine-dioxide scrubbing solution in a lab-scale bubbling reactor

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### Abstract

The present study attempts to clean up nitric oxide from the simulated flue gas using aqueous chlorine-dioxide solution in the bubbling reactor. Chlorine-dioxide is generated by chloride–chlorate process. Experiments are carried out to examine the effect of various operating variables like input NO concentration, presence of SO<sub>2</sub>, pH of the solution and NaCl feeding rate on the NO<sub>x</sub> removal efficiency at 45 °C. Complete oxidation of nitric oxide into nitrogen dioxide occurred on passing sufficient ClO<sub>2</sub> gas into the scrubbing solution. NO is finally converted into nitrate and ClO<sub>2</sub> is reduced into chloride ions. A plausible reaction mechanism concerning NO<sub>x</sub> removal by ClO<sub>2</sub> is suggested. DeNO<sub>x</sub> efficiency increased slightly with the increasing input NO concentration. The presence of SO<sub>2</sub> improved the NO<sub>2</sub> absorption but pH of solution showed marginal effect on NO<sub>2</sub> absorption. NO<sub>x</sub> removal mechanism changed when medium of solution changed from acidic to alkaline. A constant NO<sub>x</sub> removal efficiency of about 60% has been achieved in the wide pH range of 3–11 under optimized conditions.

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**Keywords:** Flue gas; Bubbling reactor; Chlorine-dioxide; NO<sub>x</sub> removal; Mechanism

### 1. Introduction

#### 1.1. Chlorine-dioxide: a new generation oxidant

Chlorine-dioxide is a novel neutral oxy-chlorine species which is powerful oxidant and disinfectant. In the recent years, it has attracted significant commercial attention not only due to environmental concern but also due to its wide applications in the fields of bleaching, oxidation and disinfection. It is commercially used in textile, paper, fat and pulp bleaching, waste water treatment, water purification, removal of iron, manganese, H<sub>2</sub>S and phenols from industrial wastes, medical treatment, sanitation, food processing, fumigation, deodorization and aquaculture, etc. The chlorine-dioxide-based bleaching process, so-called ECF technology [1], has become quite popular in the production of bleached chemical pulps. Chlorine-dioxide

is a very powerful biocide and rapidly inactivates most of microorganisms over a wide pH range. It helps in eliminating taste and odor problems from drinking waters.

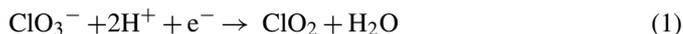
Though chlorine-dioxide is a well known strong oxidant capable of oxidizing NO into NO<sub>2</sub> yet removal of NO<sub>x</sub> from the flue gas using ClO<sub>2</sub> has, to the best of our knowledge, never been reported in the literature. Therefore, treatment of industrial waste gases by aqueous chlorine-dioxide solution is the major objective of the present study.

Chlorine-dioxide is produced from acid solutions of either sodium chlorite [2–4] or sodium chlorate [5–10]. Most of the small and medium scale generators use sodium chlorite as the precursor material. Other applications where large quantities of chlorine-dioxide are needed, utilize sodium chlorate. Though the conditions for the production of ClO<sub>2</sub> from sodium chlorite can be controlled better than from sodium chlorate but chlorite is more expensive and unstable chemical. So from industrial point of view, sodium chlorate is the most suitable raw material for ClO<sub>2</sub> generation.

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Chlorine-dioxide can be formed by reduction of chlorate with a wide variety of organic and inorganic reducing agents in a relatively concentrated acid solution as follows:



The optimum reaction conditions, by-products and the economics of the process help in deciding the suitable reducing agent. In all chlorate based processes, chloride ion plays a crucial role. No chlorine-dioxide is formed if chloride ion is not present in the reaction medium. With this view, we selected chloride–chlorate process to generate  $\text{ClO}_2$  gas for further use in removing NO from the flue gas. The general stoichiometry of chloride–chlorate process [9,10] can be expressed as:

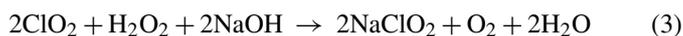


Theoretically, it gives euchlorine, which is a mixture of chlorine-dioxide and chlorine in molar ratio of 2:1. This process is extremely simple to operate, responds immediately and gives highest yield of  $\text{ClO}_2$  at the lowest cost among all other commercial processes.

### 1.2. Removal of NO

Though the flue gas from stationary sources such as power plants, incinerators and boilers mainly contains NO and  $\text{NO}_2$  but the major component of  $\text{NO}_x$  is NO (ca. 90%). The inert nature of NO has posed a persistent problem in the industry. Technologies for  $\text{NO}_x$  removal include combustion control and post-combustion treatment. Combustion control aims at reducing the  $\text{NO}_x$  formation during combustion of fossil fuel. Post-combustion methods include selective non-catalytic reduction (SNCR), selective catalytic reduction (SCR), thermal  $\text{DeNO}_x$  and scrubbing, etc. Among these technologies, scrubbing methods are economically most competitive and have advantage of controlling other acid gases and particulates at the same time [11].

In general, additives are added into scrubbing system to first convert relatively inert NO into  $\text{NO}_2$  which can be removed by alkaline absorbents. Aqueous solutions of numerous inorganic and organic absorbents have been investigated to determine their effectiveness in  $\text{NO}_x$  removal [11–31]. Sodium chlorite [16–24] has proved the most efficient oxidant among various NO absorption agents. However the problem with sodium chlorite is that it operates best under slightly acidic conditions. It has good oxidizing ability at lower pH but good absorbing ability at higher pH. So pH is a crucial parameter to oxidize NO into  $\text{NO}_2$  and absorb  $\text{NO}_2$  thereafter. Further, it is quite expensive chemical and it is itself produced by reduction of chlorine-dioxide as follows:



Keeping all these facts in mind, it seems reasonable to use  $\text{ClO}_2$  directly to oxidize NO. It will not only reduce the cost but also solve the problem of pH adjustment. Therefore the purpose of this study is to clean up NO from flue gas using aqueous chlorine-dioxide solution.

## 2. Experimental

The experimental system composed of two parts namely chlorine-dioxide generation unit and flue gas cleansing unit.

### 2.1. Chlorine-dioxide generation unit

$\text{ClO}_2$  generation unit consisted of a reactor which is well stirred sealed vessel having total volume of 2.5 L. Concentrated sodium chloride solution (2 M) was continuously injected into reactor at a suitable flow rate by syringe pump. The reactor was filled with 1.5 L solution of sodium chlorate (0.4 M) in concentrated sulfuric acid (~12N). Continuous stirring was provided by a mechanical agitator. Temperature of the reaction vessel was controlled within  $45 \pm 0.1$  °C by water thermostat. The reactor was wrapped with an aluminium foil to avoid any photo-dissociation of  $\text{ClO}_2$ . Analytical grade reagents and distilled de-ionized water were used throughout. Nitrogen gas was purged through the reaction mixture using a bubbling device at a flow rate of 2 L/min.  $\text{ClO}_2$  carried by  $\text{N}_2$  gas was further introduced into bubbling reactor.

### 2.2. Flue gas cleansing unit

Flue gas cleansing unit included simulated flue gas supply system, bubbling reactor, pH control system,  $\text{ClO}_2$  absorber, data acquisition system, and sampling cum analysis system. The simulated flue gas was obtained by controlled mixing of NO,  $\text{N}_2$  and  $\text{O}_2$  using mass flow controllers (MFC). The bubbling reactor is made up of acrylic material. The inner diameter and height of the reactor are 15 and 45 cm, respectively. Continuous stirring was provided by mechanical agitator with a speed of 250 rpm. The temperature of the bubbling reactor was controlled within  $45 \pm 0.1$  °C, the most common operating temperature for wet scrubbing methods. The pH of reaction solution was controlled by using an auto-pH control system by continuous addition of NaOH (0.2 M) solution with the help of peristalsis pump. The  $\text{ClO}_2$  absorber (2 L vessel) consisted of ca. 2% carbonate buffered potassium iodide solution (1.5 L). Samples from reactor and absorber were analyzed using Dionex ion chromatograph (IC) or titrated iodometrically using auto-titrator (Metrohm-Swiss). The potentiometric titration system included a 670 titroprocessor, 730 sample changer, 665 dosimat and platinum electrode. The inlet and outlet  $\text{NO}_x$  concentra-

Table 1  
The operating conditions of bubbling reactor and chlorine-dioxide generator

Bubbling reactor		Chlorine-dioxide generator	
Reactor volume	5 L	Reactor volume	2.5 L
Temperature	45 °C	Temperature	45 °C
pH	3–12 (3.5)	$\text{N}_2$ flow rate	2 L/min
Input $\text{SO}_2$ conc.	0–1800 ppm	NaCl conc.	1–2 M
Input NO conc.	150–1180 ppm	$\text{H}_2\text{SO}_4$ conc.	10–12N
$\text{ClO}_2$ flow rate	2 L/min	$\text{NaClO}_3$ conc.	0.2–0.4 M
Flue gas flow rate	45 L/min	NaCl feeding rate	0.5–1.5 mL/min
Forced air flow rate	4 L/min		
GSD	4–12 cm (6 cm)		

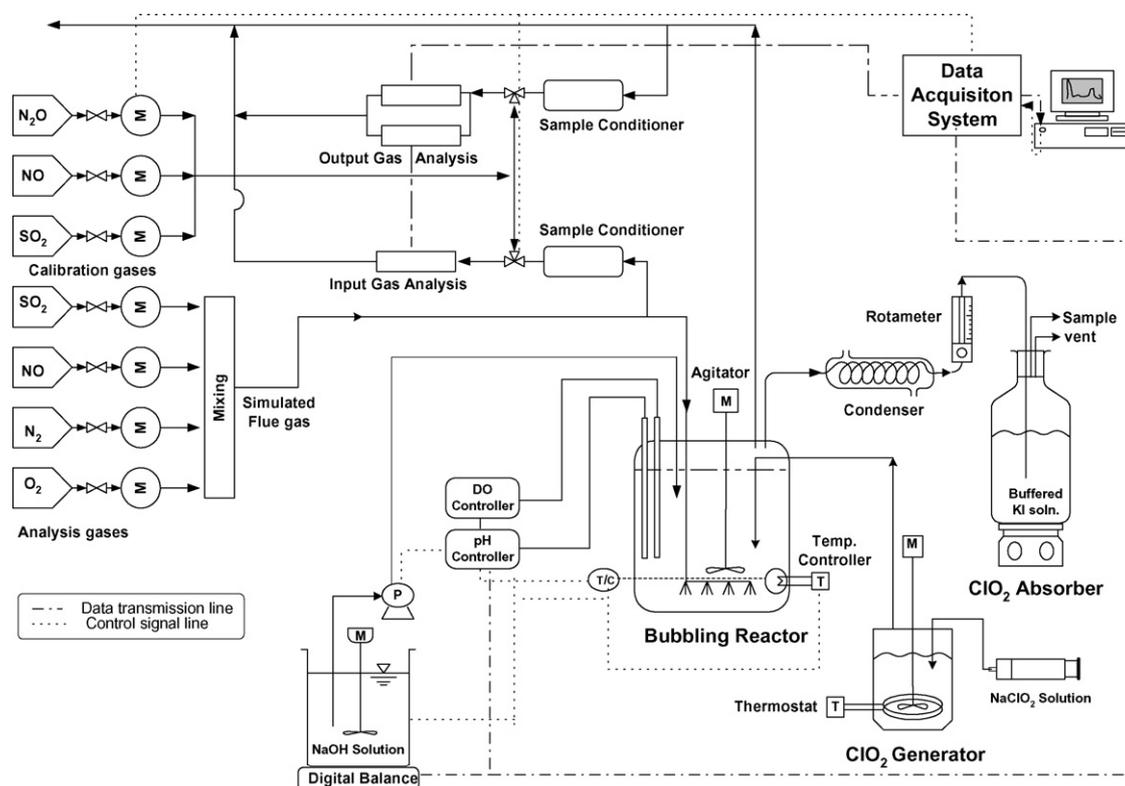


Fig. 1. A schematic diagram of bubbling reactor for the removal of NO from the flue gas.

tions were analyzed using the  $\text{NO}_x$  analyzer (Chemiluminiscent type, Model: 42C, Thermo Environmental Instruments, USA) after removing the moisture in the sample conditioner. The  $\text{O}_2$  concentrations were analyzed with help of dissolved oxygen (DO) meter. The overall specifications of the experimental setup are given in Table 1. A schematic diagram of the experimental system is shown in Fig. 1.

### 3. Results and discussion

Chlorine-dioxide has been extensively used for oxidation, disinfection and bleaching, but no reference is cited on using aqueous  $\text{ClO}_2$  solution in the removal of NO. There is no doubt about the oxidizing capability of  $\text{ClO}_2$ . Standard oxidation potential of chlorine-dioxide in gas and solution phase is  $-0.95$  and  $-1.27$  V, respectively [32]. If it can oxidize NO into  $\text{NO}_2$ , then it may prove useful for removal of NO from flue gas. With this view, experiments for the removal of NO have been conducted using aqueous chlorine-dioxide solution. Effect of various parameters, viz., input NO concentration, presence of  $\text{SO}_2$ , NaCl feeding rate and pH of solution has been studied in a lab-scale bubbling reactor at  $45^\circ\text{C}$ .

#### 3.1. Removal of nitric oxide from simulated flue gas

Experiments were carried out at pH of 3.5 and input NO concentration of 500 ppm in absence of  $\text{SO}_2$  at  $45^\circ\text{C}$  to investigate the  $\text{NO}_x$  removal using aqueous chlorine-dioxide scrubbing solution. Fig. 2 displays the variation of pH, NO and  $\text{NO}_2$  outlet

concentration with the passage of time.  $\text{NO}_x$  removal efficiency and ion concentration are plotted versus time in Fig. 3. It was observed that  $\text{ClO}_2$  can oxidize NO into  $\text{NO}_2$  completely and a consistent and reproducible  $\text{NO}_2$  absorption efficiency of about 60% is achieved.

In the acidic medium, a plausible mechanism for the removal of  $\text{NO}_x$  by aqueous chlorine-dioxide solution is as follows:

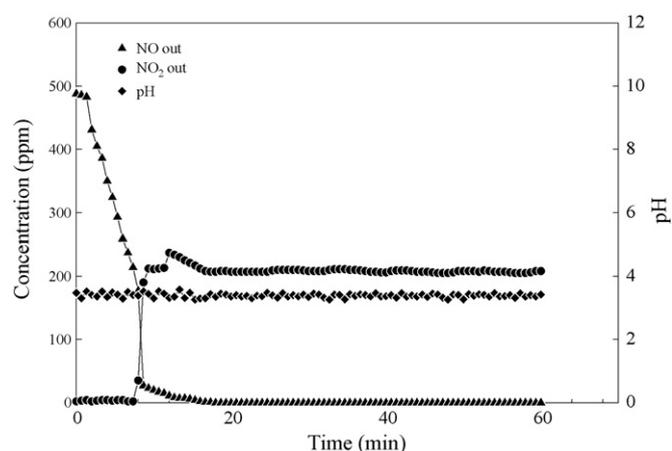


Fig. 2. Variation in the outlet concentration of NO,  $\text{NO}_2$  and pH with the passage of time.

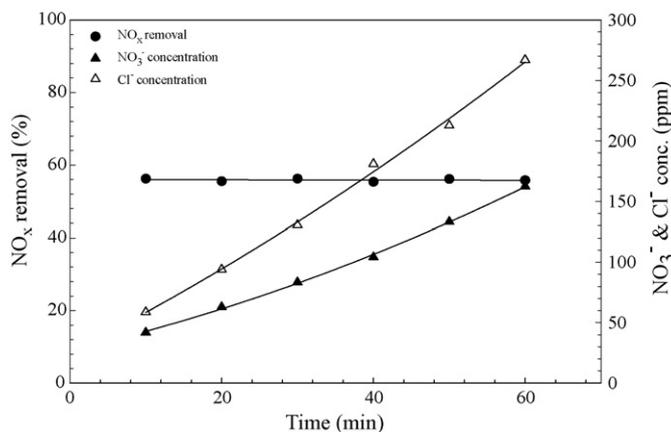
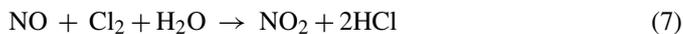


Fig. 3. NO<sub>x</sub> removal and ion concentration with passage of time (pH 3.5, input NO = 500 ppm,  $T = 45^{\circ}\text{C}$ ).

The overall reaction for NO<sub>x</sub> removal may be written as:



Chlorine gas produced along with chlorine-dioxide as suggested in Eq. (2) is also a strong oxidant. Yang et al. [11] reported that chlorine is capable of oxidizing NO into NO<sub>2</sub> and nitrate. Stoichiometry of reaction of chlorine with NO can be expressed as:



Formation of nitrate and chloride as suggested in the above mechanism was confirmed by analyzing the sample from bubbling reactor using Dionex ion chromatograph. The variation of ion concentration with time is presented in Fig. 3.

### 3.2. NO<sub>x</sub> removal in presence/absence of sulfur dioxide

NO<sub>x</sub> removal was also studied at input NO concentration of 350 ppm, pH of 3.5 in absence as well as in presence of SO<sub>2</sub>. Fig. 4 displays the variation of pH, NO and NO<sub>2</sub> outlet concentration with the passage of time. SO<sub>2</sub> and NO<sub>x</sub> removal

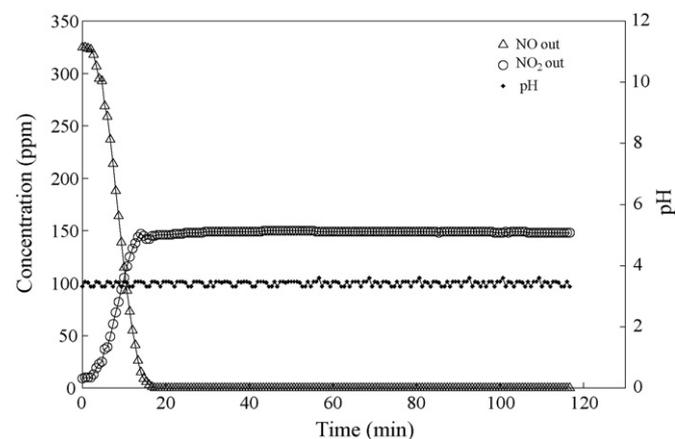


Fig. 4. Variation of NO and NO<sub>2</sub> concentration with time (input NO = 350 ppm, input SO<sub>2</sub> = 500 ppm, pH 3.5,  $T = 45^{\circ}\text{C}$ ).

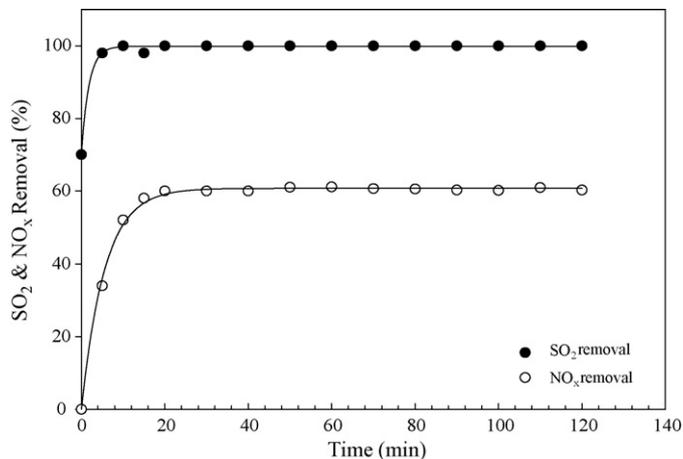


Fig. 5. Simultaneous removal SO<sub>2</sub> and NO<sub>x</sub> with time (input NO = 350 ppm, input SO<sub>2</sub> = 500 ppm, pH 3.5,  $T = 45^{\circ}\text{C}$ ).

efficiencies are presented in Fig. 5. It is observed that ClO<sub>2</sub> can clean up both SO<sub>2</sub> and NO quite efficiently. The maximum DeSO<sub>x</sub> and DeNO<sub>x</sub> efficiency obtained at an optimum NaCl (2 M) feeding rate of 0.5 mL/min are about 100 and 60%, respectively. In the acidic medium, SO<sub>2</sub> removal by chlorine-dioxide appears to take place by the following mechanism:



### 3.3. Effect of SO<sub>2</sub> concentration on NO<sub>x</sub> removal

Experiments were carried out at pH of 3.5 and input NO concentration of 350 ppm to investigate the effect of input SO<sub>2</sub> concentration on NO<sub>x</sub> removal efficiency. Fig. 6 shows that NO<sub>x</sub> removal increased from 51 to 58% when input SO<sub>2</sub> concentration was increased from 200 to 700 ppm. SO<sub>2</sub> is moderately soluble in aqueous solution and undergoes hydrolysis to form bisulfite (HSO<sub>3</sub><sup>-</sup>), sulfite (SO<sub>3</sub><sup>2-</sup>) and disulfite (S<sub>2</sub>O<sub>5</sub><sup>2-</sup>) ions [20]. The equilibrium concentrations of all these species depend upon partial pressure of SO<sub>2</sub> and pH of the solution. These S(IV) species are believed to catalyze the NO<sub>2</sub> absorption [33,34].

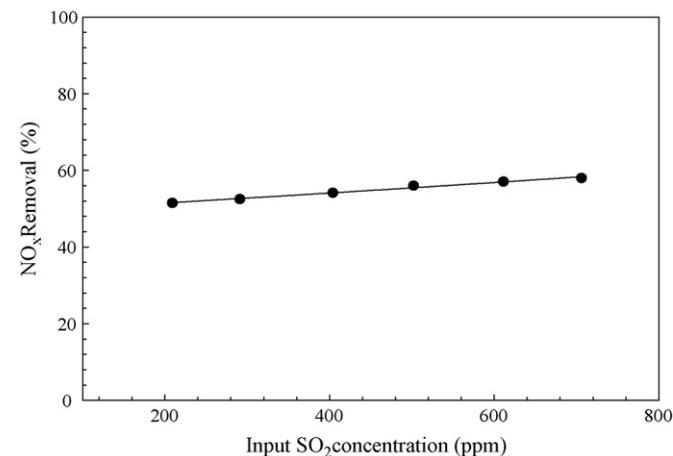


Fig. 6. Effect of input SO<sub>2</sub> concentration on NO<sub>x</sub> removal efficiency (pH 3.5,  $T = 45^{\circ}\text{C}$ , input NO = 350 ppm).

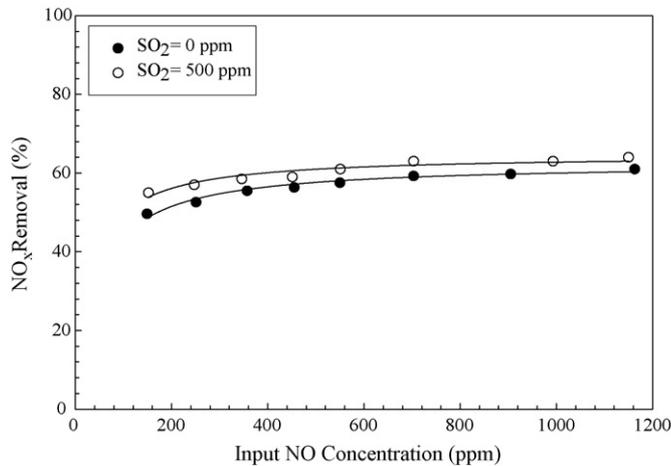


Fig. 7. Effect of input NO concentration on NO<sub>x</sub> removal efficiency in presence and absence of SO<sub>2</sub> ( $T = 45\text{ }^{\circ}\text{C}$ , pH 3.5).

### 3.4. Effect of input no concentration on NO<sub>x</sub> removal efficiency

Effect of input NO concentration on the NO<sub>x</sub> removal has been investigated at pH of 3.5. In absence of SO<sub>2</sub> gas, it is found that NO<sub>x</sub> removal efficiency increased from 50 to 61% when NO input concentration is increased from 150 to 1150 ppm. In presence of SO<sub>2</sub> (500 ppm), NO<sub>x</sub> removal efficiency is found better than in absence of it as can be seen from Fig. 7. This slight increase in NO<sub>x</sub> removal in presence of SO<sub>2</sub> may be due to reaction of S(IV) species with nitrogen dioxide as predicted by earlier researchers [33,34].

### 3.5. Effect of NaCl feeding rate

We also performed some experiments to investigate the effect of NaCl feeding rate on NO<sub>x</sub> removal efficiency at pH of 3.5, input NO concentration of 500 ppm in absence of SO<sub>2</sub> at 45 °C. It is apparent from Fig. 8 that NO<sub>x</sub> removal increases sharply with increasing NaCl (2 M) feeding rate up to 0.4 mL/min and thereafter it attains a steady state on achieving ~60% DeNO<sub>x</sub> efficiency. NO oxidation remained 100% complete throughout.

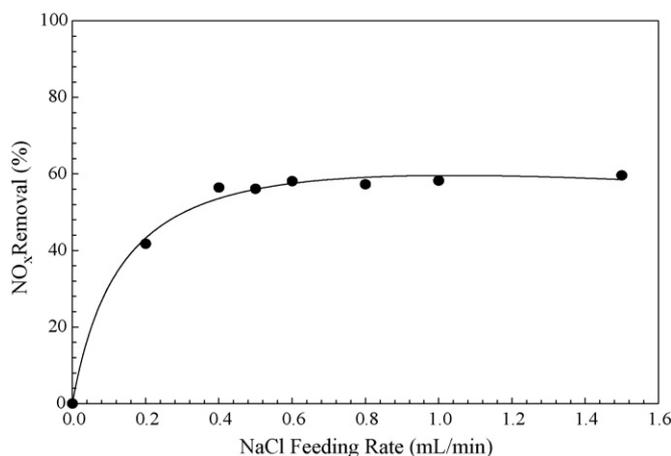


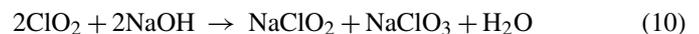
Fig. 8. Effect of NaCl feeding rate on NO<sub>x</sub> removal in absence of SO<sub>2</sub> (pH 3.5, input NO = 500 ppm).

The increasing NaCl feeding rate into ClO<sub>2</sub> generator increased the rate of ClO<sub>2</sub> generation (Eq. (2)), which subsequently facilitated the NO oxidation as well as NO<sub>2</sub> absorption. The excess chlorine-dioxide in the scrubbing solution did not improve the NO<sub>2</sub> absorption rate. Thus the optimum concentration and feeding rate of sodium chloride solution can be easily decided on the basis of input NO concentration.

### 3.6. Effect of pH

In the present study, pH of reaction medium was varied from 3 to 11 at constant input NO concentration of 500 ppm at 45 °C and its effect on NO<sub>x</sub> removal, output NO and NO<sub>2</sub> concentration is reported in Fig. 9. At constant NaCl (2 M) feeding rate of 0.5 mL/min, there occurred no change in the NO<sub>x</sub> removal efficiency when pH is increased from 3 to 6 but thereafter removal efficiency decreased in the pH range of 6–8 and then increased up to pH of 9 and attained a steady state. The sudden decrease in NO<sub>x</sub> removal in the pH range of 6–8 and then increase in NO<sub>x</sub> removal in pH > 8 may be attributed to the different mechanisms involved in acidic and alkaline media.

In acidic medium (pH < 7), NO<sub>x</sub> removal mechanism is controlled by ClO<sub>2</sub> (as proposed in Eqs. (4)–(6)). When pH reaches ≥ 7, the disproportionation [3,35] of chlorine-dioxide slowly starts and speed up with increasing pH. It leads to formation of chlorite and chlorate as follows:



The 50% of ClO<sub>2</sub> (from Eq. (10)) is thus converted into sodium chlorite, which is again a strong oxidant and absorbent for NO and NO<sub>2</sub>, respectively. In alkaline medium (pH > 7) NO<sub>x</sub> removal mechanism thus changed and is now controlled by NaClO<sub>2</sub> as follows:

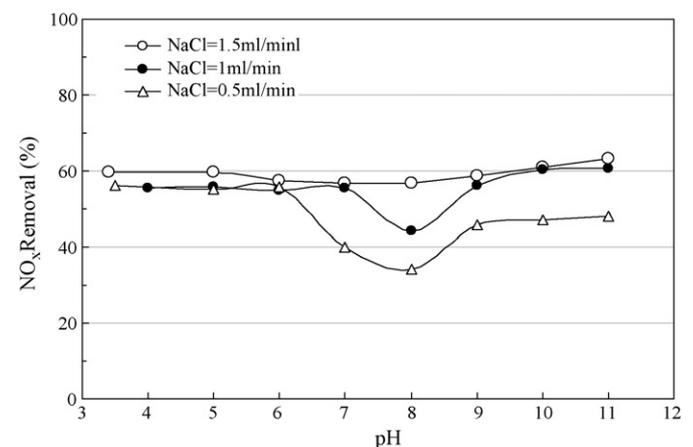
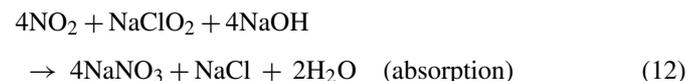
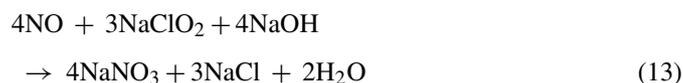


Fig. 9. Effect of pH on NO<sub>x</sub> removal at different NaCl feeding rates (input NO = 500 ppm,  $T = 45\text{ }^{\circ}\text{C}$ ).

The overall reaction can be written as:



The above mechanism has been discussed in detail by several workers [16,18–20,22–24].

The decrease in  $\text{NO}_x$  removal efficiency at  $\text{pH}$  of  $\geq 7$  is believed due to insufficient chlorine-dioxide and chlorite in reaction solution. To confirm that  $\text{NO}_x$  removal mechanism changes in the alkaline medium, the  $\text{NaCl}$  feeding rate was enhanced so that more chlorite is formed by disproportionation of unused dissolved  $\text{ClO}_2$ . It was found that  $\text{NO}_x$  removal attained a steady state at higher  $\text{NaCl}$  feeding rate due to formation of sufficient chlorite. At  $\text{NaCl}$  (2 M) feeding rate of 1.5 mL/min, constant  $\text{NO}_x$  removal efficiency of around 60% was achieved in the wide  $\text{pH}$  range of 3–11.

#### 4. Conclusions

Chlorine-dioxide has proved the most promising among various additives used so far in removal of  $\text{NO}_x$ . It has several advantages over sodium chlorite, which is supposed to be the best additive so far. The merits of chlorine-dioxide can be summarized as:

- It exhibited 100%  $\text{NO}$  oxidation and  $\sim 60\%$   $\text{NO}_x$  removal efficiency which is comparable with that observed using other additives.
- Comparing the capital cost, chlorine-dioxide is much cheaper than sodium chlorite as the later is produced itself by reduction of chlorine-dioxide. Hence it is more appropriate to utilize  $\text{ClO}_2$  instead of first converting it into chlorite and then using it for  $\text{NO}_x$  removal.
- When  $\text{NaClO}_2$  is used for  $\text{NO}_x$  removal,  $\text{pH}$  of the scrubbing solution needs a tight control. The  $\text{pH}$  should be low enough to provide  $\text{NaClO}_2$  high oxidizing ability and high enough to allow absorption of  $\text{NO}_2$  thereafter. However in case of  $\text{ClO}_2$ , there is no need of such  $\text{pH}$  adjustment. It is as effective at low  $\text{pH}$  as at high  $\text{pH}$ .
- Sodium chlorite has high oxidizing power at low  $\text{pH}$  but simultaneously it decomposes at lower  $\text{pH}$  producing chlorine-dioxide which is another secondary pollutant. However if  $\text{ClO}_2$  is supplied from outside then it is easy to control the supply of  $\text{ClO}_2$  to avoid its excessive emission into atmosphere.
- Operation of the system is comparatively easier.
- It is also easy to handle the waste water by simply adjusting the  $\text{pH}$  because the major components in the effluent are  $\text{HCl}$  and  $\text{HNO}_3$ .

Based upon the above merits, it would be reasonable to conclude that chlorine-dioxide is a promising additive for  $\text{NO}_x$  removal due to wide  $\text{pH}$  range, cost effectiveness and easy operation.

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