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Mass transfer in the absorption of SO_2 and NO_x using aqueous euchlorine scrubbing solution

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Abstract

Attempts have been made to generate euchlorine gas by chlorate-chloride process and to utilize it further to clean up SO_2 and NO_x from the flue gas in a lab scale bubbling reactor. Preliminary experiments were carried out to determine the gas and liquid phase mass transfer coefficients and their correlation equations have been established. Simultaneous removal of SO_2 and NO_x from the simulated flue gas using aqueous euchlorine scrubbing solution has been investigated. Euchlorine oxidized NO into NO_2 completely and the later subsequently absorbed into the scrubbing solution in the form of nitrate. SO_2 removal efficiency around 100% and NO_x removal efficiency around 72% were achieved under optimal conditions. Mass balance has been confirmed by analyzing the sulfate, nitrate, euchlorine and chloride ion using ion chromatograph/auto-titrator and comparing it with their corresponding calculated values.

Key words: mass transfer; sulfur dioxide; nitric oxide; bubbling reactor; euchlorine

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Introduction

The combustion of fossil fuels leads to the emission of sulfur oxides (SO_x) and nitrogen oxides (NO_x). Besides, sulphuric acid industry and roasting of sulfide ore in metallurgy release substantial amount of sulfur oxides into atmosphere. Volcanic eruption is yet another natural source of SO_2 emission. The emission of SO_2 and NO_x has been a major environmental concern because of their hazardous effects on human health and the ecosystems. SO_2 is the most pervasive air pollutant and is the main cause of acid rain. NO_x are particularly responsible for atmospheric ozone depletion, smog and visibility problems. Due to the stringent regulations in the recent years, an efficient technology for the abatement of SO_2 and NO_x emissions from both stationary and mobile sources is thus highly desirable.

Recently, considerable attention has been focused on the simultaneous removal of SO_2 and NO_x in a single reactor considering the capital investment, operation cost and space for equipment (Adewuyi *et al*., 1999; Harriott *et al*., 1993; Lee *et al*., 2005). Flue gas desulphurization (FGD) is the most widely used process which can remove SO_2 efficiently. If minor adjustment in wet FGD process may work for combined removal of SO_2 and NO_x, it may prove a compact and cost effective technology for the future. However, NO can not be as easily absorbed as SO_2.

Technologies for NO_x removal include combustion control and post-combustion treatment. Combustion control aims at reducing the NO_x formation during combustion of fossil fuel. Post-combustion methods include selective non-catalytic reduction (SNCR), selective catalytic reduction (SCR) and scrubbing etc. Among these technologies, scrubbing methods are economically competitive and have advantage of controlling other acid gases and particulates at the same time (Yang *et al*., 1996). In general, additives are added into scrubbing system first to convert relatively inert NO into NO_2 which can be subsequently removed by alkaline absorbents. Aqueous solutions of numerous oxidative absorbents such as hydrogen peroxide (Baveja *et al*., 1979), per acid (Littlejohn and Chang, 1990), organic tertiary hydro peroxides (Perlmutter *et al*., 1993), sodium chlorite (Sada *et al*., 1978; Brogen *et al*., 1998; Chu *et al*., 2001; Lee *et al*., 2005), $KMnO_4$ (Brogen *et al*., 1997; Chu *et al*., 1998) and chlorine dioxide (Jin *et al*., 2006; Deshwal *et al*., 2008) have been investigated to determine their efficiency in the removal of NO_x. Sodium chlorite has proved the most efficient oxidant among them. However, the drawback with sodium chlorite is that it has good oxidizing ability at lower pH while the absorbing capability is good only at higher pH. Therefore, pH is a crucial parameter to oxidize NO into NO_2 and to absorb NO_2 thereafter. In addition, sodium chlorite is relatively unstable and quite expensive chemical.

In recent years, chlorine-dioxide has attracted considerable attention due to its wide applications in the fields

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of bleaching, oxidation, disinfection, and gas absorption. It can clean up both NO_x and SO_2 simultaneously in a wide range of pH (Jin *et al*., 2006; Deshwal *et al*., 2008). Although chlorine-dioxide can be produced from acid solutions of either sodium chlorite (Deshwal *et al*., 2004a, Deshwal and Lee, 2005a, White *et al*., 1942) or sodium chlorate (Ni and Wang, 1997; Burke *et al*., 1993, Deshwal and Lee, 2004b, Deshwal and Lee, 2005b), yet sodium chlorate is better cost effective raw material for chlorine-dioxide generation. In fact, it is very difficult to generate 100% pure chlorine-dioxide. The chlorate-chloride process gives highest yield of ClO_2 at the lowest cost among all other commercial processes. Although this process is extremely simple to operate, responds immediately but theoretically, it gives euchlorine, i.e., a mixture of chlorine dioxide and chlorine in the molar ratio of 2:1. The general stoichiometry of chlorate-chloride process may be expressed as Reaction (1) (Deshwal and Lee, 2004b):

$$4H^+ + 2ClO_3^- + 2Cl^- \longrightarrow 2ClO_2 + Cl_2 + 2H_2O \qquad (1)$$

The standard oxidation potential of chlorine-dioxide and chlorine in the solution phase is –1.188 and –1.396 V, respectively (Dean, 1999), which implies that both have potential to oxidize NO into NO_2. Hence euchlorine, generated from chlorate-chloride process, can be utilized directly to oxidize NO. It will not only reduce the cost but also solve the problem of pH adjustment. Therefore, with this view, aqueous euchlorine solution has been chosen for the combined removal of NO_x and SO_2 from the flue gas.

1 Materials and methods

The experimental system is divided into two parts: euchlorine generation unit and flue gas treatment unit. A schematic diagram of the experimental system is shown in Fig.1.

1.1 Euchlorine generation unit

This unit is composed of a reactor which is well stirred and sealed vessel having total volume of 2.5 L. Concentrated sodium chloride solution (1–2 mol/L) was continuously injected into reactor at a suitable flow rate (0.5–1.5 mL/min) by a syringe pump (Model-200, KDS Scientific Inc., USA). The reactor was filled with 1.5 L solution of sodium chlorate (0.2–0.4 mol/L) in a relatively concentrated sulfuric acid (11–12 mol/L). Continuous stirring was provided by a mechanical agitator. Temperature of the reaction vessel was controlled within (45 ± 0.1)°C by water thermostat (WBC-1506D, JEIO TECH, Korea). Nitrogen gas was purged through the reaction mixture using a bubbling device at a flow rate of 2 L/min. Euchlorine thus carried by N_2 gas was further introduced into bubbling reactor.

1.2 Flue gas treatment unit

Flue gas cleansing unit composed of simulated flue gas supply system, bubbling reactor, pH control system, euchlorine absorber, data acquisition system, and sampling cum analysis system. The bubbling reactor is a well stirred and sealed vessel (ID-15 cm, Height-45 cm) having internal volume of 8 L. The simulated flue gas was obtained by controlled mixing of SO_2, NO, and N_2 using mass flow controllers (MFC). Air was introduced into reactor using air pump to maintain the dissolved O_2 concentrations about 30% to 40% of the saturated O_2 concentrations. Continuous stirring was provided by a mechanical agitator (4 blades, disc turbine type impeller) with a speed of 250 r/min. Temperature of the reaction vessel was controlled within (45 ± 0.1)°C, a typical scrubbing temperature. The

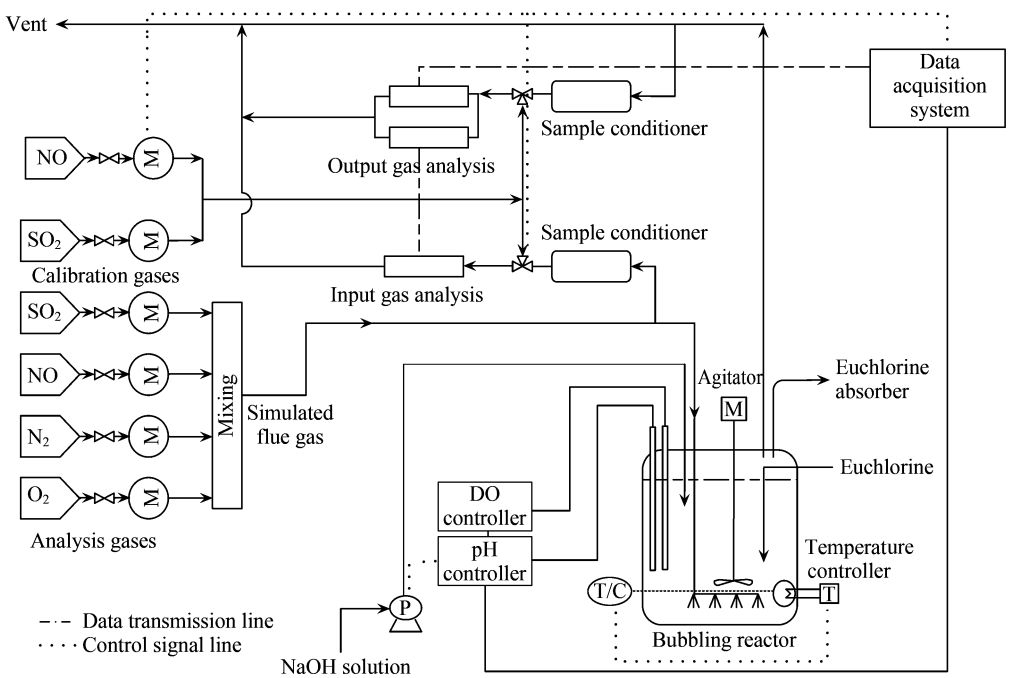

Fig. 1 Schematic diagram of the bubbling reactor.

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pH of reaction solution was controlled by using an auto-pH control system (KFC-MK-250, Korea) by continuous addition of NaOH (0.2 mol/L) solution with peristalsis pump (Cole-Palmer Co., USA). The euchlorine absorber (2 L vessel) consisted of ca. 2% carbonate buffered potassium iodide solution (1.5 L). Samples from reactor and absorber were analyzed by either ion chromatograph (IC) or by iodometric titration using auto-titrator (Metrohm-Swiss). The inlet and outlet gas concentrations were analyzed after removing its moisture in the sample conditioner by the SO_2 analyzer (Model-Ultramat 23, IR type, Siemens, Germany), NO analyzer (Model-42C, Chemiluminescent type, Thermo Environmental Instruments Inc., USA), and DO meter (835A, Thermo Orion, USA).

1.3 Materials

Standard gases included N_2 (99%), SO_2 span gas (99%), and NO span gas (99.9%). N_2 and SO_2 were the products of Anjeon Gas Co., Korea and NO was the product of Mathieson Co., New Zealand, Sodium chlorate (98%, Junsei Chemical Co., Ltd., Japan), sodium chloride (99.5%, Aldrich Chem. Co., Inc., USA), sulfuric acid (98%, PFP, Osaka, Japan), potassium iodide (99.5%, Samchun Pure Chem. Co., Ltd., Korea), and sodium thiosulfate (99%, Shinyo Pure Chem. Co., Ltd., Japan) used in the present study were the analytical grade reagents.

2 Results and discussions

2.1 Mass transfer characteristics

A number of preliminary experiments were carried out to assess the physical characteristics, particularly the gas phase and liquid phase mass transfer coefficients. The product of gas phase mass transfer coefficients k_g (mol/10^5(cm^2·s·Pa)) and interfacial area per unit volume a (cm^{-1}) were measured by absorbing sulfur dioxide from SO_2/N_2 mixture into NaOH solutions (0.2 mol/L). Since in relatively concentrated NaOH solution, the dissolved SO_2 reacted instantaneously and irreversibly with the liquid phase reactant at the gas-liquid interface, as a result, the liquid phase mass transfer resistance is considered to be negligible. The values of $k_g \times a$ were calculated from the absorption rate (r_{SO_2}, mol/(cm^3·s)) of SO_2 for SO_2/NaOH system as follows (Lancia *et al.*, 1997):

$$r_{SO_2} = k_g \times a \times \Delta P_{SO_2} \tag{2}$$

where, ΔP_{SO_2} (1×10^5 Pa) is the gas-liquid driving force for absorption, and can be evaluated by:

$$\Delta P_{SO_2} = P_{(SO_2)}{}_{av} - H_{SO_2} \times C_{(SO_2)aq} \tag{3}$$

where, H_{SO_2} (1×10^5 (Pa·L)/mol) is the Henry's constant for SO_2, $C_{(SO_2)aq}$ (mol/L) is concentration of SO_2 in liquid bulk, and $P_{(SO_2)av}$ (1×10^5 Pa) is the logarithmic average of inlet $P_{(SO_2)in}$ (1×10^5 Pa). Outlet SO_2 partial pressure $P_{(SO_2)out}$ (1×10^5 Pa) and can be calculated as:

$$P_{(SO_2)av} = \frac{P_{(SO_2)in} - P_{(SO_2)out}}{\ln(P_{(SO_2)in}/P_{(SO_2)out})} \tag{4}$$

Because SO_2 is absorbed completely in 0.2 mol/L NaOH solution, the product $H_{SO_2} \times C_{(SO_2)aq}$ can be ignored, and Eq. (2) can be rewritten as:

$$r_{SO_2} = k_g \times a \times P_{(SO_2)av} \tag{5}$$

The experimental absorption rate of sulfur dioxide r'_{SO_2}, can be calculated as follows:

$$r'_{SO_2} = Q_L \times C_{SO_3^{2-}} / V_R \tag{6}$$

where, Q_L (L/min) is the liquid flow rate, $C_{SO_3^{2-}}$ (mol/L) is the concentration of sulfite ions in the reactor, and V_R (cm^3) is the reactor volume.

Now comparing Eqs. (5) and (6), can get:

$$k_g \times a = \frac{Q_L \times C_{SO_3^{2-}}}{V_R \times P_{(SO_2)av}} \tag{7}$$

Experiments were carried out at different SO_2 gas flow rates and input SO_2 concentrations and the values of $k_g \times a$ were determined from $P_{(SO_2)in}$, $P_{(SO_2)out}$, and the concentration of sulfite in the reaction vessel. The concentrations of sulfite were determined by iodometric titration of the reaction sample against standard sodium thiosulfate solution.

The $k_g \times a$ can be correlated to gas flow rate (Q_g, L/min), partial pressure of SO_2 ($P_{(SO_2)av}$), and agitation speed (N, r/min) as follows:

$$k_g \times a = k_1 \times Q_g^{k_2} \times P_{(SO_2)av}^{k_3} \times N^{k_4} \tag{8}$$

The agitation speed was varied from 60 to 360 r/min, and absorption rate of SO_2 was found independent of the agitation speed. Thus neglecting the term N^{k_4} and taking logarithm on both sides, Eq. (9) can be get:

$$\log(k_g \times a) = \log k_1 + k_2 \log Q_g + k_3 \log P_{(SO_2)av} \tag{9}$$

The values of k_1, k_2, and k_3 have been calculated by plotting the graph $\log(k_g \times a)$ vs. $\log Q_g$ or $\log P_{(SO_2)av}$ and the correlation equation was obtained as:

$$k_g \times a = 3.80231 \times 10^{-6} \times Q_g^{1.26} \times p_{(SO_2)av}^{0.2811} \tag{10}$$

Further, the data for liquid phase mass transfer coefficients k_L (cm/s), were obtained by absorbing O_2 from air into distilled water with a low concentration of O_2. The O_2 in the feed water was removed by blowing N_2 into the reactor. The gas phase mass transfer resistance was assumed to be negligible because O_2 has a low solubility in water and the O_2 concentration in atmospheric air is as high as 20 percentage of volume. The dissolution rate of O_2 in the distilled and degassed water can be expressed as Eq. (11):

$$\frac{dC}{dt} = k_L \times a \times (C_S - C) \tag{11}$$

Integrating the Eq. (11), can get:

$$\ln(C_s - C_t) = k_L \times a \times t + \ln(C_S - C_0) \tag{12}$$

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where, C_S (mg/L) is the saturated dissolved O_2 concentration, C_0 (mg/L) and C_t (mg/L) are the dissolved O_2 concentration in the initial stage and after time t respectively. Experiments were carried out at different O_2 gas flow rates and agitation speeds and the values of $k_L \times a$ were obtained from Eq. (12).

The $k_L \times a$ in O_2/H_2O system can be correlated to Q_g and N as follows:

$$k_L \times a = k_1 \times N^{k_2} \times Q_g^{k_3} \tag{13}$$

Taking logarithm on both sides, we get:

$$\log(k_L \times a) = \log k_1 + k_2 \log N + k_3 \log Q_g \tag{14}$$

where, the values of k_1, k_2, and k_3 have been calculated by plotting the graph $\log(k_L \times a)$ vs. $\log Q_g$ or $\log N$ and the correlation equation was obtained as:

$$k_L \times a = 6.395 \times 10^{-4} \times N^{0.452} \times Q_g^{0.3198} \tag{15}$$

2.2 Gas film control and physical absorption

According to the two film theory (Fig. 2), the rate of straight mass transfer of a gas (R) in water (only via absorption) can be expressed as Eq. (16) (Levenspiel, 1999):

$$R = \left(\frac{1}{k_g a} + \frac{H}{k_L a}\right)^{-1}(P_{av} - H \times C_{aq}) \tag{16}$$

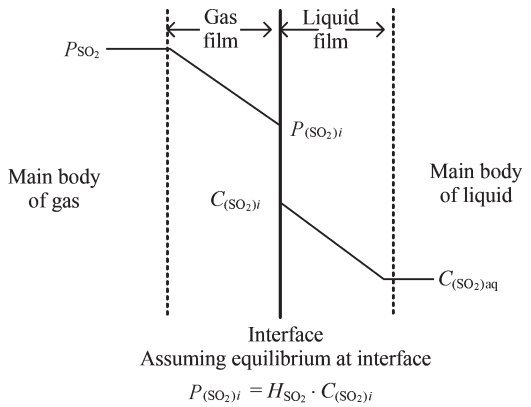

Fig. 2 Absorption of sulfur dioxide as visualized by the two film theory.

where, H (1×10^5 (Pa·L)/mol) is the Henry's constant for gas and all other terms have the usual significance.

The absorption rate of SO_2 or NO are plotted against absorption driving force, i.e., logarithmic mean of SO_2 or NO partial pressure (Fig. 3), where a couple of straight lines are reported which represent the upper and lower limits for the gas absorption rate. The upper line describes conditions of gas film control (no liquid-side resistance); however, the lower line is the representative of conditions in which only physical absorption occurs. The rate of mass transfer of a gas increases when reaction occurs within the liquid film and it can be expressed as:

$$R' = \left(\frac{1}{k_g \times a} + \frac{H}{\Phi \times k_L \times a}\right)^{-1}(P_{av} - H \times C_{aq}) \tag{17}$$

where, R' is the rate of mass transfer of a gas when reaction occurs within the liquid film and Φ is the enhancement factor due to chemical reaction of gas with the aqueous euchlorine solution. The absorption rate of a gas in the aqueous euchlorine scrubbing solution (R_A) was calculated experimentally as follows:

$$R_A = Q_A \times (C_{A(in)} - C_{A(out)}) \times 10^{-6} \times \frac{1}{22.4} \times \frac{1}{V_R} \tag{18}$$

where, Q_A (L/s) is gas flow rate, $C_{A(in)}$ (ppmv) and $C_{A(out)}$ (ppmv) are input and output concentration of gas respectively. The liquid-side resistance is quite significant in the absorption of NO as is apparent from the Fig. 3b, whereas Fig. 3a shows that the absorption rate of SO_2 is mainly controlled by the gas-side resistance.

In case of SO_2, almost no liquid-phase mass transfer resistance has been noticed if there is sufficient euchlorine in the scrubbing solution. Thus, the absorption rate of SO_2 is mainly controlled by gas film resistance. On the other hand, the solubility of NO in water (1.25×10^{-3} mol/10^5 (Pa·L)) is about 560 times lesser than that of SO_2 (7.03×10^{-1} mol/10^5 (Pa·L)) at 50°C. Moreover, the dissolved NO is first oxidized into NO_2 by euchlorine, and later is absorbed by the scrubbing solution. Therefore, liquid-phase mass transfer resistance mainly controls the absorption of NO. Although the physical absorption of NO occurs but its absolute value is smaller than that of gas film control.

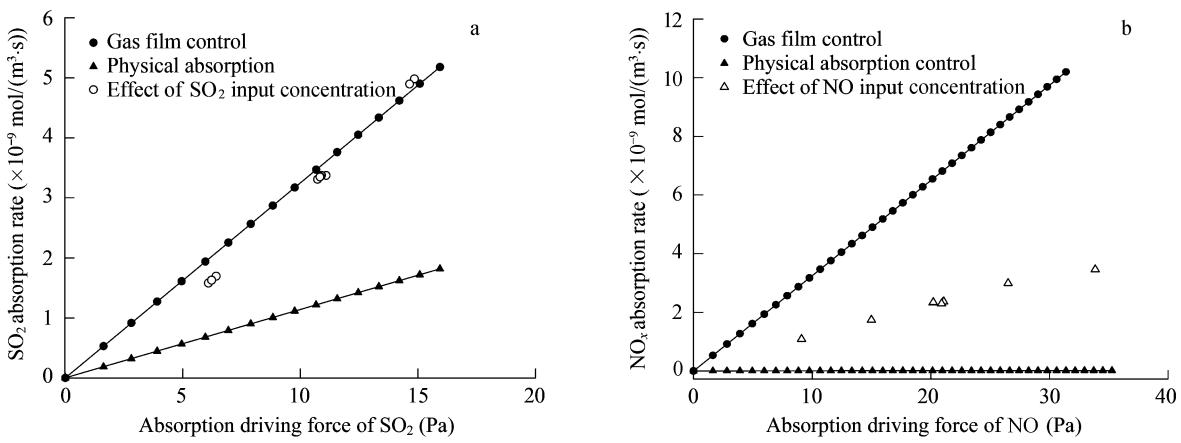

Fig. 3 Gas film control and physical absorption in the euchlorine-NO-NaOH system (a) and euchlorine-SO_2-NaOH system (b).

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Φ can be calculated by $\Phi = R'/R$. Enhancement factors of SO_2 and NO_x increased as evident from Fig. 4. SO_2 being more soluble, thus a small increase in enhancement factor occurred. However, NO and NO_2 being less soluble, are difficult to absorb and have the main resistance in the liquid phase; thus enhancement factor increased significantly by their chemical reaction with the aqueous euchlorine scrubbing solution.

2.3 Removal of SO_2 and NO_x from flue gas by euchlorine solution

Simultaneous removal of SO_2 and NO was studied at 45°C, pH 3.5. The input SO_2 and NO concentrations were 500 and 350 ppmv respectively, and were introduced by passing euchlorine gas into the scrubbing solution. The SO_2 and NO_x removal efficiencies at different euchlorine feeding rates are presented in Fig. 5.

Euchlorine firstly cleaned up the more reactive and soluble SO_2 gas and thereafter the surplus euchlorine oxidized NO gas. At the feeding rate of 0.825 mmol/min, all of the euchlorine was utilized in SO_2 absorption and NO_x removal was found negligible. However, at higher euchlorine feeding rates, it was observed that euchlorine cleaned up both SO_2 and NO quite efficiently. Euchlorine oxidized NO into NO_2 completely. NO_2 absorption efficiency increased with the increasing euchlorine feeding rates. A consistent and reproducible SO_2 and NO_x absorption efficiencies around 100% and 72% were observed at euchlorine feeding rates of 3.045 mmol/min. Furthermore, euchlorine proved a remarkably efficient oxidant as well as absorbent in a wide pH range of 3.5 to 8 as is evident from Fig. 6. In addition, the oxidizing as well as absorption ability of euchlorine was not affected by pH, thus making it a superior oxidative absorbent compared to sodium chlorite.

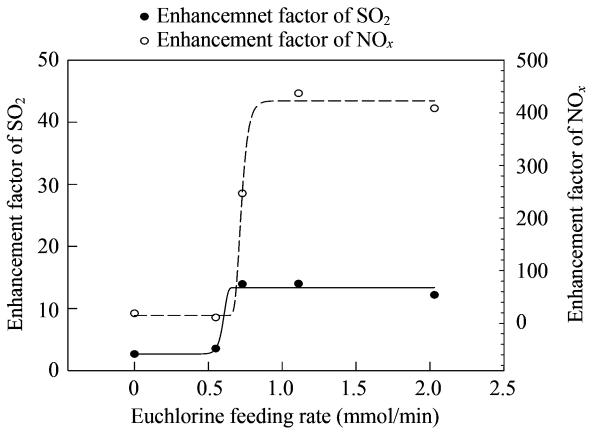

Fig. 4 Enhancement factor of SO_2 and NO_x at various euchlorine feeding rates.

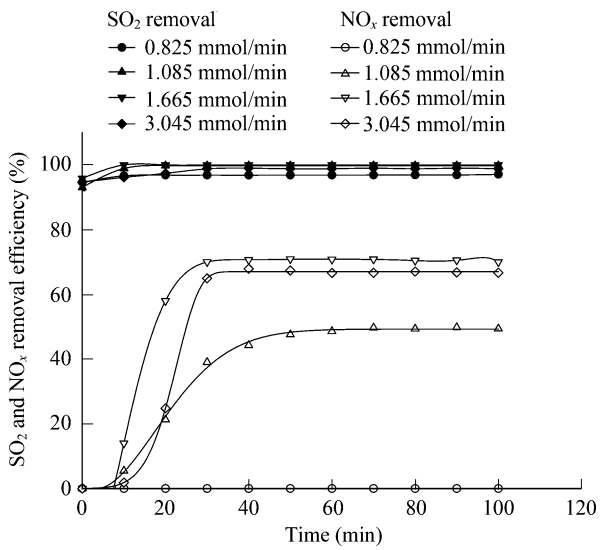

Fig. 5 Simultaneous removal of SO_2 and NO_x with time at various euchlorine feeding rates. Conditions: temperature 45°C; pH 3.5; input NO concentration 350 ppmv; input SO_2 concentration 500 ppmv.

Flue gases mainly contain NO and NO_2 but the major component of NO_x is NO (ca. 90%). Euchlorine is a mixture of chlorine-dioxide and chlorine at the molar ratio of 2:1, where both the constituents have the ability to oxidize NO. Chlorine-dioxide, a major constituent of euchlorine is believed to clean up NO and SO_2 via following reactions (Jin *et al.*, 2006):

$$5SO_2 + 2ClO_2 + 6H_2O \longrightarrow 5H_2SO_4 + 2HCl \quad (19)$$

$$5NO + 2ClO_2 + H_2O \longrightarrow 5NO_2 + 2HCl \quad \text{(oxidation)} \quad (20)$$

$$5NO_2 + ClO_2 + 3H_2O \longrightarrow 5HNO_3 + HCl \quad \text{(absorption)} \quad (21)$$

The overall reaction for the NO_x removal can be written as Reaction (22) (Jin *et al.*, 2006; Deshwal *et al.*, 2008):

$$5NO + 3ClO_2 + 4H_2O \longrightarrow 5HNO_3 + 3HCl \quad (22)$$

Chlorine gas, another constituent of euchlorine is also a strong oxidant. It is already reported that chlorine is capable of oxidizing NO into NO_2 and nitrate (Yang *et al.*, 1996). The stoichiometry of reaction between chlorine with NO can be expressed as:

$$NO + Cl_2 + H_2O \longrightarrow NO_2 + 2HCl \quad (23)$$

$$2NO + 3Cl_2 + 4H_2O \longrightarrow 2HNO_3 + 6HCl \quad (24)$$

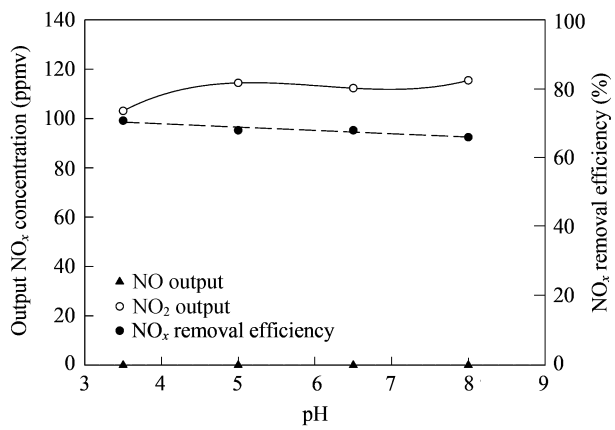

Fig. 6 NO_x removal efficiency and output concentrations of NO and NO_2 at various pH. Conditions: temperature 45°C; input SO_2 concentration 250 ppmv; input NO concentration 350 ppmv; euchlorine feeding rate 1.66 mmol/min.

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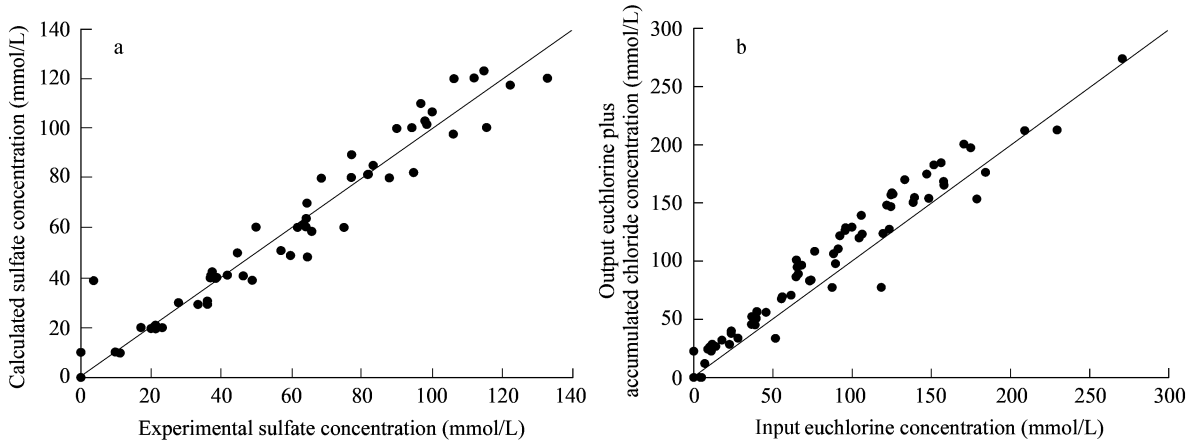

Fig. 7 Comparison between experimental and calculated sulfate concentrations (a) and of input euchlorine and output euchlorine plus accumulated chloride concentrations (b).

The presence of sulfate, nitrate and chloride in the samples of the bubbling reactor confirmed the mechanism proposed above.

2.4 Mass balance for removal of SO_2 and NO_x

The formation of sulfate, nitrate and chloride ions as suggested above in the Reactions (19)–(24) is confirmed by analyzing the sample from bubbling reactor using ion chromatograph. The samples from euchlorine absorber were quantitatively analyzed iodometrically using autotitrator to examine the euchlorine evolved unreacted. The relation between calculated and experimental sulfate is illustrated in Fig. 7a. The mass balance for euchlorine is demonstrated in Fig. 7b. Both figures have demonstrated a fair mass balance.

3 Conclusions

The present study deals with the simultaneous removal of SO_2 and NO_x using aqueous euchlorine scrubbing solution in the lab-scale bubbling reactor. Mass transfer characteristics have been examined critically by determining the gas and liquid phase mass transfer coefficients. Removal efficiency around 100% and 72% has been achieved for SO_2 and NO_x respectively at a typical scrubbing temperature of 45°C. The byproducts of the reaction in the combined removal of SO_2 and NO_x using euchlorine solution are sulphate, nitrate and chloride which are not hazardous materials, thus causing no secondary pollution. The mass balance for sulfate, nitrate and chloride ions has been confirmed. The oxidizing as well as absorption ability of euchlorine was found unaffected by pH, thus making it a superior oxidative absorbent compared to sodium chlorite.

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