J Mater Cycles Waste Manag (2016) 18:618–624
DOI 10.1007/s10163-016-0526-8

SPECIAL FEATURE: ORIGINAL ARTICLE

Recent researches on Thermal Treatment and Emission Control (9th i-CIPEC)

Study of nitrogen oxide absorption in the calcium sulfite slurry

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Received: 4 February 2016 / Accepted: 16 June 2016 / Published online: 11 July 2016

Abstract Experiments were conducted using a bubbling reactor to investigate nitrogen oxide absorption in the calcium sulfite slurry. The effects of $CaSO_3$ concentration, NO_2/NO mole ratio and O_2 concentrations on NO_2 and SO_2 absorption efficiencies were investigated. Five types of additives, including $MgSO_4$, Na_2SO_4, $FeSO_4$, $MgSO_4$/Na_2SO_4 and $FeSO_4$/Na_2SO_4, had been evaluated for enhancing NO_2 absorption in $CaSO_3$ slurry. Results showed that $CaSO_3$ concentration had significant impact on NO_2 and SO_2 absorption efficiencies, and the highest absorption efficiencies of SO_2 and NO_2 could reach about 99.5 and 75.0 %, respectively. Furthermore, the NO_2 absorption was closely related to the NO_2/NO mole ratio, and the existence of NO_2 in flue gas may promote NO absorption. The presence of O_2 in simulated flue gas was disadvantage for NO_x removal because it can oxidize sulfite to sulfate. It was worth pointing out that $FeSO_4$/Na_2SO_4 was the best additive among those investigated additives, as the NO_2 removal efficiency was significantly increased from 74.8 to 95.0 %. IC and in situ FTIR results suggest that the main products were NO_3^- and NO_2^- in liquid phase and N_2O, N_2O_5 and HNO_3 in gas phase during the $CaSO_3$ absorption process.

Keywords Nitrogen oxides · Absorption · $CaSO_3$ slurry · Products · Additives

Introduction

Nitrogen oxides (NO_x) generated from fuel combustion and waste incineration in stationary sources are precursors of acid rain, photochemical smogs and fine aerosols, have received unprecedented public attention [1]. In China, a series of NO_x emission standards for different industries, which are regarded as the strictest standards in history, have been successively issued by the Chinese government to limit NO_x emissions. The emission limit value of NO_x for waste incineration has changed from 400 to 250 mg m^{-3} [2]. Therefore, an urgent issue of air quality improvement is NO_x emission control. Currently, various removal technologies for control of NO_x have been widely used, including combustion modification technology, selective catalytic reduction (SCR) method, selective non-catalytic reduction (SNCR) method and wet absorption process [3]. However, most of these single-function unit operations suffer high operating costs. Emerging cost-effective technologies for multi-pollutants control are necessary. One promising candidate is the chemical absorption process for the co-capture of pollutants.

For the chemical absorption deNO_x process, sufficient NO_2 is the precondition. Accordingly, numerous aqueous oxidants such as $KMnO_4$, H_2O_2, Fe(II)-EDTA and $NaClO_2$ [4–6], some gas phase oxidations including ozone, ClO_2, non-thermal plasma and transition metal oxides catalysts [7, 8] have been investigated to convert insoluble NO to soluble NO_2. However, higher NO oxidation efficiency is limited by higher operating cost. If a certain amount of NO could be co-captured in absorption process, the overall treatment cost will be greatly slashed. Numerous aqueous absorbents, such as H_2O, NaOH, $Ca(OH)_2$, aqueous sulfite, ammoniacal cobalt(II) solutions, ferrate(VI), Fe(II)-EDTA and other complex component solutions, have been

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investigated in the removal of NO and NO_2 [9–18]. Compared with other approaches, the aqueous sulfite absorption method is one of the promising processes due to its high removal efficiency, moreover sulfite is an abundant reaction intermediate in the typical wet flue gas desulfurization [19].

$CaSO_3$ slurry can be a reasonable absorbent for the co-capture of multi-pollutants, as it is the leading byproduct in the limestone scrubbing solution and reacts with NO_2 in preference to O_2 [17]. Nevertheless, the low solubility of $CaSO_3$ slurry limits the reaction of NO_2 and SO_3^{2-}, thus scientists are currently investigating alternative cost-effective additives for enhancing NO_2 absorption through enriching the SO_3^{2-} concentration. Tang et al. [13] indicated the $MgSO_4$ as an effective additive could significantly increase the NO_2 absorption efficiency from 70.8 to 86.0 %. Some metal and non-metal additives for enhancing NO_2 absorption in insoluble calcium sulfite slurry were also investigated. Wang et al. [17] reported that the $FeSO_4$ was the most effective additive with absorption efficiency of 95 %. In addition, with 0.5 mol L^{-1} ammonium sulfate added, the NO_2 removal efficiency increased significantly from 67 to 90 %.

To the best of our knowledge, the chemical behavior of NO_2 absorption process in $CaSO_3$ slurry has not yet been systematically investigated. This study investigated the effects of $CaSO_3$ concentration, NO_2/NO mole ratio and SO_2 and O_2 concentrations on NO_x absorption in $CaSO_3$ slurry. The liquid and gas products formed in $CaSO_3$ slurry were investigated, and the chemical behavior of NO_2 absorption process in $CaSO_3$ slurry was discussed. In addition, different additives employed to enhance the absorption process were also studied.

Experimental section

Experimental setup

The schematic of the experimental system setup is illustrated in Fig. 1. It consists of a reaction gas supply unit, a cylindrical flue gas treatment reactor and a set of analytical instruments.

A cylinder with an inner diameter of 300 mm and a length of 600 mm (a volume of 5 L) was used as the bubbling reactor, redundant solution was stored in the overflow tank, and outer layer of the reactor was filled with water from water bath to control reaction temperature. The liquid stirring speed was controlled at 110 rpm. A cooling pipe was used to cool and dehydrate the outlet flue gas to protect the flue gas analyzer.

Experimental methods

The simulated flue gas was prepared with air, N_2, CO_2, SO_2, NO, and NO_2. A set of mass flow controllers was used to adjust the flow rate. All of the experiments were performed at atmospheric pressure and 325 K. The simulated gas consisted of 12 % CO_2, 300 ppm SO_2, 0–200 ppm NO, 0–200 ppm NO_2, 0–10 % O_2, using N_2 as balance gas, with a total flow rate of 4 L min^{-1}. While in the study of reaction products of NO_2 absorption in $CaSO_3$ slurry (see "Reaction products of NO2 absorption in CaSO3 slurry"), the simulated flue gas consisted of 5 % O_2, 260 ppm NO_2, and inert gas Ar, which is marked red in Fig. 1. The concentrations of NO_x, O_2 and SO_2 were monitored by a flue gas analyzer (Testo 350, Germany). Sulfite ions (SO_3^{2-}) in the liquid phase were measured by an iodometric titration method. The concentrations of NO_3^- and NO_2^- in the liquid phase were tested by ion chromatogram (Metrohm 792, Switzerland; column: Asupper5/250). The qualitative analysis of gaseous products after reactor was performed by a Fourier transform infrared spectrometer (In situ FTIR, Nicolet 6700, USA).

And the initial pH (5.5–6) was moderated by the additional HCl. The $CaSO_3$ used in this study is $CaSO_3{\cdot}2H_2O$ (98 % purity) and deionized water, and the total $CaSO_3$ slurry volume in reactor was 2 L. Additives tested in this study include $MgSO_4$, Na_2SO_4, $FeSO_4$, $MgSO_4/Na_2SO_4$, $FeSO_4/Na_2SO_4$, which were introduced to the slurry with a concentration ranging from 0 to 0.5 mol L^{-1}. Meanwhile, the mole ratio of complex additives was 1/1.

The absorption efficiencies of SO_2 and NO_x were calculated as

$$\text{Removal efficiency}\ (\%) = \frac{C_{\text{in}} - C_{\text{out}}}{C_{\text{in}}} \times 100\ \%, \qquad (1)$$

here C_{in} (ppm) is the concentrations of SO_2 or NO_x measured at inlet of the bubbling reactor; C_{out} (ppm) is the outlet concentrations of SO_2 and NO_x.

Results and discussion

Effect of $CaSO_3$ concentration on SO_2 and NO_2 absorption

Some experiments were carried out to investigate the effect of $CaSO_3$ concentration on NO_2 absorption rate, and the results were shown in Fig. 2. From Fig. 2a, b, it is obvious that the higher $CaSO_3$ concentration is favorable to the absorption of NO_2. It can be found from Fig. 2a that the absorption efficiency of NO_2 increased with $CaSO_3$ concentration from zero to 0.05 mol L^{-1}, thereafter, remained

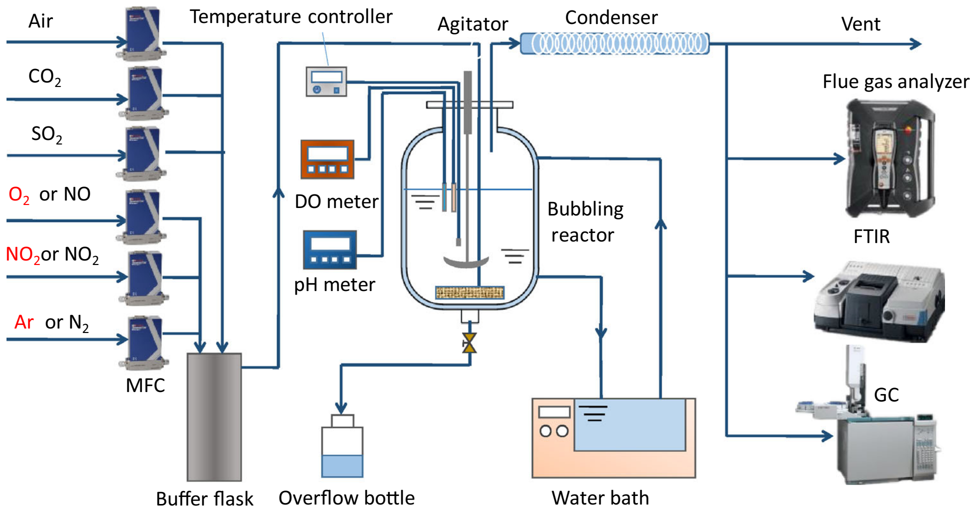

Fig. 1 Schematic diagram of the experimental setup

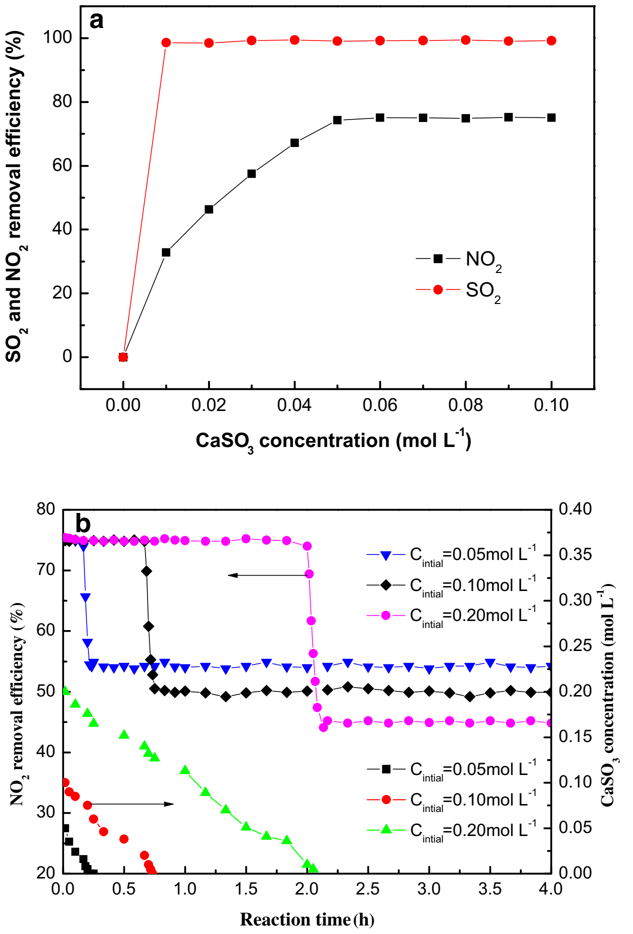

Fig. 2 **a** Effect of $CaSO_3$ concentration on SO_2 and NO_2 absorption. **b** Variation of NO_2 removal efficiency and $CaSO_3$ concentration in absorption solution with reaction time. (The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of O_2, CO_2, SO_2 and NO_2 are 5, 12 %, 300 and 200 ppm, respectively)

stable with increasing $CaSO_3$ concentration at above 0.05 mol L^{-1}, and the highest absorption efficiency of NO_2 about 75.2 % was obtained. This phenomenon is not only the cause of the existence of more SO_3^{2-} ions, but also more likely attributed to the absorbent particles that were suspended in the $CaSO_3$ slurry which could provide more reactive surface; the absorption rate would increase with solid content within a certain range, which is in accordance with the conclusion drawn by Dagaonkar et al. [20]. Also, when $CaSO_3$ concentration was changed from zero to 0.1 mol L^{-1}, the absorption efficiency of SO_2 kept stable, different from NO_2, the removal efficiency of SO_2 is slightly affected by $CaSO_3$ concentration and almost retains 99.0 %, and it indicates that SO_2 is easier removed. Figure 2b shows the relation of the NO_2 removal efficiency and $CaSO_3$ concentration with reaction time at different $CaSO_3$ concentrations. As shown in Fig. 2b, when $CaSO_3$ concentration was above 0.05 mol L^{-1}, the presence of more $CaSO_3$ absorbent (0.05–0.2 mol L^{-1}) resulted in a significant improvement in NO_2 absorption performance. Although the variety trend of NO_2 absorption was similar, a longer efficient time was kept. However, a sharply declining happened in a short time, and NO_2 removal efficiency finally retained at around 50 %. This trend is due to the fact that the SO_3^{2-} concentration decreases can lower chemical reaction rates. Moreover, the decreases of SO_3^{2-} concentration influence the gas–liquid mass transfer.

The results indicate that compared with solubility SO_3^{2-} absorbent, although the low solubility of $CaSO_3$ will limit the NO_2 absorption efficiency, the maximum removal efficiencies for NO_2 reached 75.2 %, so $CaSO_3$ slurry is a suitable absorbent, because $CaSO_3$ can also be oxidized by NO_2 prior to by O_2, which is consistent with the conclusion drawn by Tang et al. [13].

The overall reaction of the NO_2 absorption in $CaSO_3$ slurry can be written as follows:

$$4NO_2(aq) + SO_3^{2-}(aq) + 2H_2O(aq) \rightarrow 3NO_2^-(aq) + NO_3^-(aq) + SO_4^{2-}(aq) + 4H^+. \quad (2)$$

According to Eq. (2), when NO_2 is absorbed by $CaSO_3$ slurry, $SO_3{}^{2-}$ is oxidized to $SO_4{}^{2-}$, and NO_2 is reduced to $NO_3{}^-$ and $NO_2{}^-$. Meanwhile, SO_2 absorption in $CaSO_3$ slurry will be complementary to $SO_3{}^{2-}$, and then, desulfurization and denitrification processes will be combined reasonably.

Effect of O_2 concentration on SO_2 and NO_2 absorption

The effect of O_2 concentration on the SO_2 and NO_2 absorption were investigated, and the results are displayed in Fig. 3.

In Fig. 3, it is obvious that the presence of O_2 resulted in a significant decrease of NO_2 absorption performance in $CaSO_3$ slurry, and it is partly due to the fact that $SO_3{}^{2-}$ can also be oxidized to $SO_4{}^{2-}$ by O_2. The effective operating time in absence of O_2 was obviously longer than that of 5 % O_2 concentration, the result revealed that O_2 concentration is an important parameter during the $CaSO_3$ absorption process. In this study, there is almost no influence of the variation of O_2 concentration on the SO_2 absorption.

Figure 4 shows the effect of O_2 concentrations on $NO_2{}^-$, $NO_3{}^-$ and the DO concentrations in $CaSO_3$ slurry. $NO_3{}^-$ concentration steadily increased with the increase of O_2 concentration, while $NO_2{}^-$ concentration decreased. That is because the $NO_2{}^-$could also be oxidized to $NO_3{}^-$ in the presence of O_2. Also, the formation of more $NO_3{}^-$ is due to the reaction of NO_2 and H_2O. The reactions with the participation of oxygen in this study can be summarized by the following chemical equations.

$$2CaSO_3(aq) + O_2(g) \rightarrow 2CaSO_4(aq), \quad (3)$$

$$4NO_2(aq) + 2H_2O(aq) + 2O_2(g) \rightarrow 4HNO_3(aq), \quad (4)$$

$$2HNO_2(aq) + O_2(g) \rightarrow 2HNO_3(aq). \quad (5)$$

Figure 4 also showed that the DO concentration in $CaSO_3$ slurry was almost invariable at about 0.46 mg L^{-1} as the O_2 concentration increased from 0 to 10 %. The result indicates that the mass transfer resistance of O_2 dissolving may lie on liquid phase.

Effect of NO_2/NO ratio on SO_2 and NO_x absorption

Since the effect of NO_2/NO ratio on the removal of SO_2 and NO_x is an important factor, NO_2/NO ratios were investigated and the results are shown in Fig. 5.

As shown in Fig. 5, the SO_2 and NO_x absorption in $CaSO_3$ slurry is closely related to NO_2/NO ratio, because the SO_2 removal efficiency in the absence of NO_2, which is much lower than that in the presence of NO_2 (99.5 %), was 94.2 %. This result reveals that the existence of NO_2 can facilitate SO_2 absorption. It could be ascribed to that the dissolved NO_2 reacted with $CaSO_3$ slurry, which leads to decrease of the $SO_3{}^{2-}$ concentration. On the other hand, the increasing of $SO_3{}^{2-}$concentration, due to the products of SO_2 dissolution into $CaSO_3$ slurry, may promote the reaction Eq. (2) moving to the right. However,

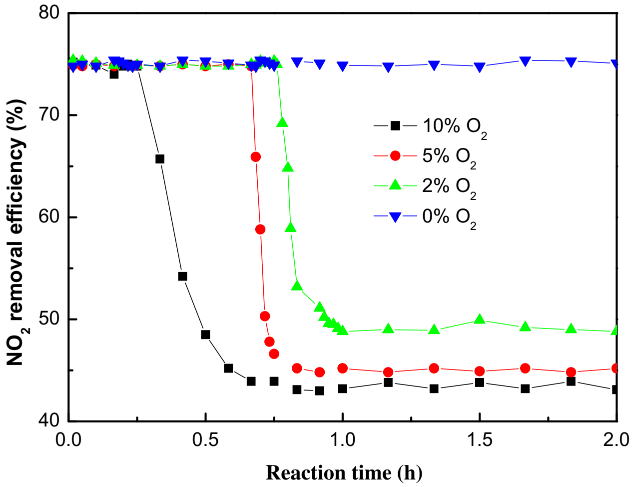

Fig. 3 Effect of O_2 concentration on NO_2 absorption. The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, the initial $CaSO_3$ concentration is 0.1 mol L^{-1}, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of CO_2, SO_2 and NO_2 are 12 %, 300 and 200 ppm, respectively

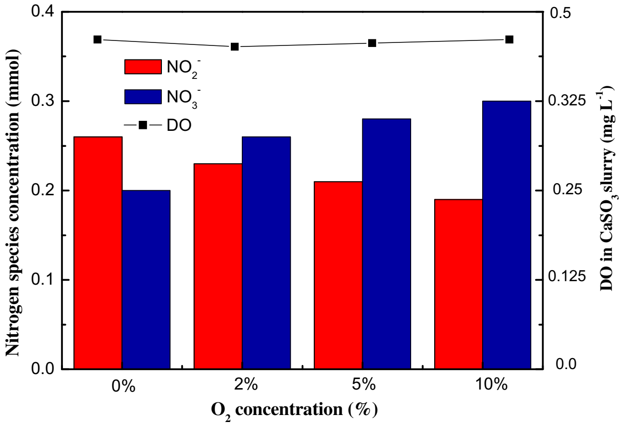

Fig. 4 NO_2 absorption product compositions and DO in $CaSO_3$ slurry. The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, the initial $CaSO_3$ concentration is 0.1 mol L^{-1}, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of O_2, CO_2, SO_2 and NO_2 are 5, 12 %, 300 and 200 ppm, respectively; the sampling time is 30 min

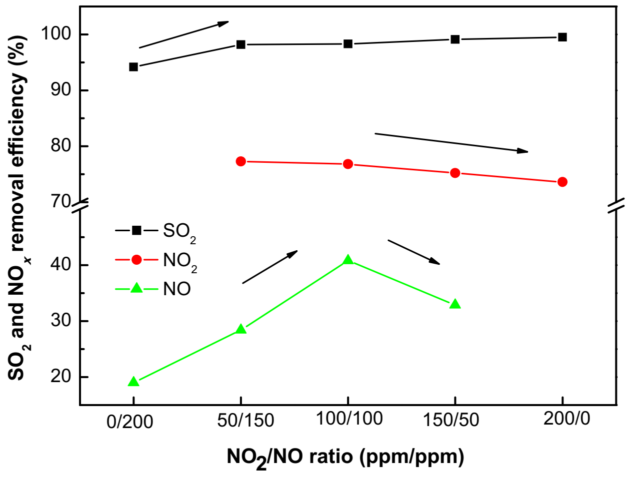

Fig. 5 Effect of NO_2/NO ratio on SO_2 and NO_x absorption in $CaSO_3$ slurry. The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, the initial $CaSO_3$ concentration is 0.1 mol L^{-1}, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of O_2, CO_2, SO_2, NO and NO_2 are 5, 12 %, 300, 0–200 and 0–200 ppm, respectively. The reaction time is 30 min

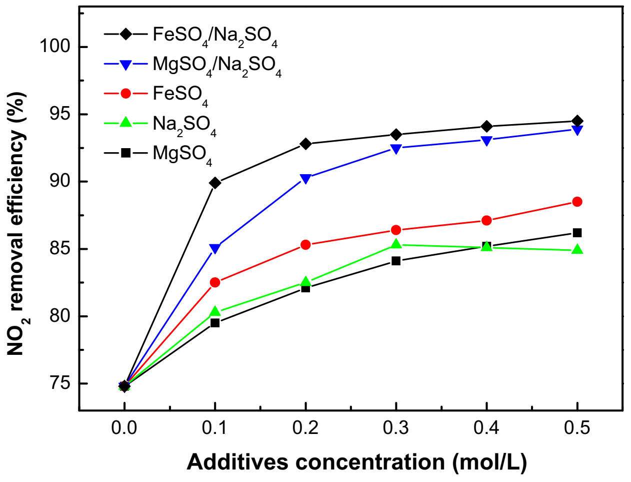

Fig. 6 Effects of individual and complex additives on NO_2 absorption in $CaSO_3$ slurry. The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, the initial $CaSO_3$ concentration is 0.1 mol L^{-1}, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of O_2, CO_2, SO_2 and NO_2 are 5, 12 %, 300 and 200 ppm, respectively; the reaction time is 30 min

the SO_2 removal efficiency had a slight increase with the NO_2 concentration from 50 to 200 ppm. Also, with the increase of the NO_2/NO ratio, the concentration of NO_2 had a slight decrease, which indicates that NO_2 absorption reaches equilibrium and further NO_2 absorption is inhibited. Figure 5 also shows that the NO removal efficiency kept low range without NO_2 in $CaSO_3$ absorbent system, which is decided by the insoluble property of NO. Moreover, NO removal rate increased firstly and then decreased, according to reaction (6), the NO_2 is advantageous to the absorption of effect on the absorption of NO at lower concentration of NO_2; then when the NO_2 concentration is large enough, the NO absorption rate shows decreasing trend. This change is consistent with the study of Gao et al. [11]. They investigated the effect of the presence of NO_2 on NO absorption and indicated that the existence of NO_2 may promote NO absorption in $(NH_4)_2SO_3$ absorbent; this result was also similar with the case of NO_x absorption into $CaSO_3$ slurry. When NO and NO_2 coexist in inlet flue gas, the reaction between NO, H_2O and NO_2 occurred in the liquid phase.

$$NO(g) + H_2O(aq) + NO_2(g) \rightarrow 2HNO_2(aq). \quad (6)$$

Effect of additive agents on SO_2 and NO_2 absorption

To improve the dissolution of $CaSO_3$ slurry, the additives, including individual $FeSO_4$, $MgSO_4$, Na_2SO_4, and complex $MgSO_4/Na_2SO_4$, $FeSO_4/Na_2SO_4$ had been selected to enhance the deNO_x performance (Fig. 6).

According to the preceding results, about 75 % of NO_2 was removed in the $CaSO_3$ system without additives. As shown in Fig. 6, with the concentration of additives varied from 0 to 0.5 mol L^{-1}, all individual and complex additives had contributed to NO_2 absorption at different levels, and the bubble was finer and smoother when additives were added into absorption system. Meanwhile, complex additives were more effective additives with absorption efficiency reaching about 95 %. While for individual additives, the maximum absorption efficiency reached around 87 %. The performance of additives varies typically as the following order $MgSO_4 < Na_2SO_4 < FeSO_4 < MgSO_4/Na_2SO_4 < FeSO_4/Na_2SO_4$. It is possible that ${SO_4}^{2-}$ could preferentially react with Ca^{2+} in $CaSO_3$ slurry to form $CaSO_4$, and the existence of ${SO_4}^{2-}$ increases the concentration of dissociated ${SO_3}^{2-}$, The related reactions appear as following [17]. On the other hand, another reason of enhancing the deNO_x performance by additives is that the metal ions Mg^{2+} might react with ${SO_3}^{2-}$ to form other soluble sulfite species, and Na^+ might improve the gas–liquid mass transfer of absorption system. Fe^{2+} has catalytic effects on the oxidation of aqueous sulfur dioxide solutions, which may also benefit the contribution of soluble ${SO_3}^{2-}$, all individual and complex additives had contributed to shift the reaction (2) to the right, and the ${SO_3}^{2-}$ ions in the system are enriched.

$$Ca^{2+} + SO_4^{2-} \leftrightarrow CaSO_4, \quad (7)$$

$$CaSO_3 \leftrightarrow Ca^{2+} + SO_3^{2-}. \quad (8)$$

Reaction products of NO_2 absorption in $CaSO_3$ slurry

The potential reaction products of NO_2 absorption in $CaSO_3$ slurry were NO_3^- and NO_2^-; using IC analysis, SO_4^{2-}, NO_3^- and NO_2^- were detected. NO_3^- and NO_2^- were formed in solution because of the reactions between NO_2 and H_2O and between NO_2 and SO_3^{2-}, respectively.

To further investigate the products of NO_2 absorption and nitrogen species fate in reaction system, a typical measurement of NO_2 absorption in $CaSO_3$ solution was carried out (Fig. 7).

As shown in Fig. 7a, the absorption efficiency of NO_2 remained stable at around 70 % before 20 min, however, a sharp decline occurred from 20 to 30 min; thereafter, the NO_2 removal efficiency was originally stabilized. Also, when the reaction time was 15, 30 and 60 min, respectively, the obtained samples of the absorption liquid were tested by the ion chromatograph. The ion chromatogram of absorption solution (reaction time is 30 min) is presented in Fig. 7b. Figure 7c points out that the amount of nitrate and nitrite concentrations showed an increasing trend as time went on; nevertheless, the experimental sum of total nitrate and nitrite concentrations were not equivalent to the calculated values, about 10 % other nitrogen species existed in this system. This is mostly due to the fact that N–S compound and N–S compound decomposed [15, 21]. Figure 7d shows the in situ FTIR spectra of all chemical individuals of outlet flue gas, especially for nitrogen species. It can be found that the absorbance peaks at 2930, 2287, 1720, 1600, 1326, and 887 cm^{-1} were presented; the bands were assigned to NO_2, N_2O, N_2O_5, NO_2, HNO_3, HNO_3, respectively [22]. According to the IC and in situ FTIR results, the main NO_2 absorption products in $CaSO_3$ absorption system are nitrate and nitrite in liquid phase; meanwhile, the consideration of gaseous products, including N_2O, N_2O_5, HNO_3 and so on, is indispensable.

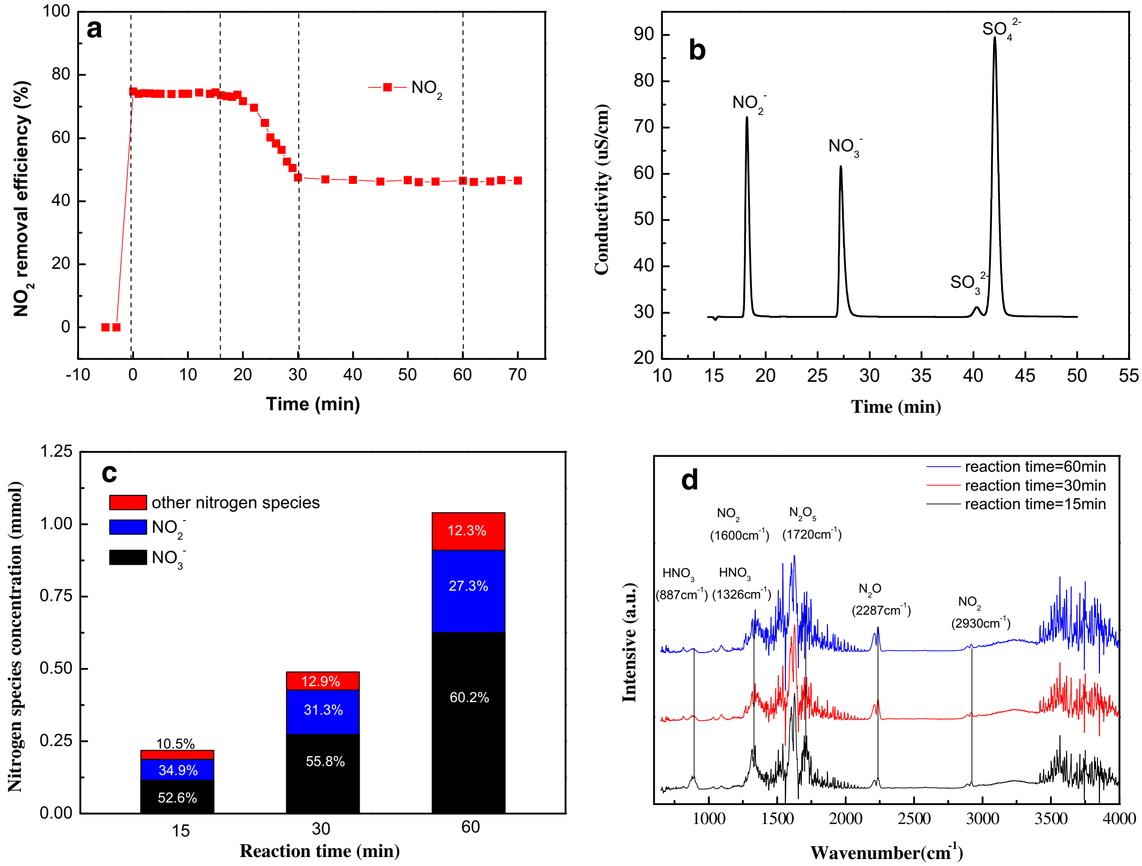

Fig. 7 **a** Typical experiment of NO_2 absorption in $CaSO_3$ solution; **b** ion chromatogram of NO_2 absorption in $CaSO_3$ solution; **c** nitrogen equilibrium of NO_2 absorption in $CaSO_3$ slurry; **d** in situ FTIR spectra of all nitrogen individuals of outlet flue gas. The reaction temperature is 325 K, the $CaSO_3$ slurry pH is 6.0, the initial $CaSO_3$ concentration is 0.06 mol L^{-1}, flue gas flow rate was 4.0 L min^{-1}, and the concentrations of O_2 and NO_2 are 5 % and 260 ppm, respectively, Ar as the balance gas, the reaction time is 70 min

Conclusions

A series of experiments were carried out to study the absorption of SO_2 and NO_x in $CaSO_3$ slurry with or without additives and the formation of liquid and gas products. The experimental results can be summarized as follows:

1. NO_2 removal efficiency increases with $CaSO_3$ concentration at the range from 0 to 0.2 mol L^{-1}, and the highest NO_2 absorption efficiency is about 75 %, while SO_2 removal efficiency will not be affected.
2. The existence of O_2 in flue gas is disadvantage to the sulfite concentration in solution in the system. NO_3^- is the major absorption product in the presence of O_2, while $NO_3^-$and NO_2^- concentrations are almost equilibrium in the absence of O_2.
3. SO_2 and NO_x absorption in $CaSO_3$ slurry is strongly related to NO_2/NO ratio. When NO and NO_2 coexist in inlet flue gas, the existence of NO_2 may promote NO absorption in $CaSO_3$ absorbent, the best NO_2/NO ratio for NO and NO_2 co-absorption is 1/1.
4. The individual and complex additives have contributed to NO_2 absorption at different levels, complex additives are more effective additives, and the performance of additives varies typically in the following order $MgSO_4 < Na_2SO_4 < FeSO_4 < MgSO_4/Na_2SO_4 < FeSO_4/Na_2SO_4$.
5. In addition to the liquid phase products, gaseous N_2O_5, N_2O and HNO_3 products in outlet flue gas can be obviously detected. The new reaction of NO_x absorption and conversion in $CaSO_3$ slurry could be explained.

Acknowledgments This work was supported by the National High Technology Research and Development Program (863) of China (No. 2012AA062503) and the Environmental Nonprofit Industry Research Project of China (No. 201209005).

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