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# Simultaneous desulfurization and denitrification by microwave reactor with ammonium bicarbonate and zeolite

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#### ABSTRACT

Microwave reactor with ammonium bicarbonate ( $NH_4HCO_3$ ) and zeolite was set up to study the simultaneous removal of sulfur dioxide ( $SO_2$ ) and nitrogen oxides ( $NO_x$ ) from flue gas. The results showed that the microwave reactor filled with  $NH_4HCO_3$  and zeolite could reduce  $SO_2$  to sulfur with the best desulfurization efficiency of 99.1% and reduce  $NO_x$  to nitrogen with the best  $NO_x$  purifying efficiency of 86.5%. Microwave desulfurization and denitrification effect of the experiment using ammonium bicarbonate and zeolite together is much higher than that using ammonium bicarbonate or zeolite only.  $NO_x$  concentration has little effect on denitrification but has no influence on desulfurization,  $SO_2$  concentration has no effect on denitrification are 211–280 W and 0.315 s, respectively. The mechanism for microwave reduced desulfurization and denitrification can be described as the microwave-induced catalytic reduction reaction between  $SO_2$ ,  $NO_x$  and ammonium bicarbonate with zeolite being the catalyst and microwave absorbent.

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#### 1. Introduction

Sulfur dioxide  $(SO_2)$  and nitrogen oxides  $(NO_x)$ , mainly emitted from fossil fuel combustion, are considered to be the major source of air pollution. Sulfur dioxide is generally accepted to be the most important precursor to acid rain [1]. Nitrogen oxides contribute a lot to photochemical smog, acid rain, ozone depletion and greenhouse effect [2,3]. Almost all NO<sub>x</sub> (95%) sources are produced from the transportations (49%) and power plants (46%) [3]. In China, coal combustion produces about 24 million tons SO<sub>2</sub> and 7.7 million tons NO<sub>x</sub> per year that leads to more than \$13.3 billion dollars loss [4]. The new policy against pollution further appeals to the desulfurization and denitrification of flue gas. Therefore, desulfurization and denitrification of flue gas attracted much more attentions.

In recent years, many technologies have been used to reduce the emission of sulfur dioxide ( $SO_2$ ) and nitrogen oxides ( $NO_x$ ). Among which the flue gas desulfurization (FGD) and selective catalytic reduction (SCR) are most effective for  $SO_2$  and  $NO_x$  removal, respectively. The catalytic technologies are more attractive because of their low cost and high efficiency [5–7]. Some research has been conducted in catalytic desulfurization, the catalyst such as  $MOS_2$  or CuO-CeO<sub>2</sub> [1,8]. Extensive research has been undertaken using hydrocarbons (CH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, C<sub>3</sub>H<sub>6</sub>) [6,7], alcohols (CH<sub>3</sub>OH, CH<sub>3</sub>CH<sub>2</sub>OH) [7] and ammonia [9,12] as reductants for denitrification over various catalytic materials, such as Al<sub>2</sub>O<sub>3</sub> [6,7], ZrO<sub>2</sub> [10], CuO[11], TiO<sub>2</sub> [12] and zeolite [5,9,13,14]. The zeolite catalyst, which reduced the nitrogen oxide selectively with the hydrocarbon as a reducing agent under the rich oxygen condition, was developed [9,13,14]. Compared to the individual control techniques, simultaneous SO<sub>2</sub> and NO<sub>x</sub> removal is advantageous due to less equipment demanded. Simultaneous SO<sub>2</sub> and NO<sub>x</sub> removal from stationary sources could be achieved with high efficiency using copper on alumina catalyst sorbents (CuO/Al<sub>2</sub>O<sub>3</sub>) [15], or using a dual bed of potassium-containing coal-pellets and calcium-containing pellets [16].

In environment protection, microwave has been widely used in the areas of waste treatment, contaminated soil remediation, recycling of rubber tyres, activated carbon applications and the treatment of volatile organic compounds (VOCs) [17]. SO<sub>2</sub> and NO<sub>x</sub> emissions from coal combustion were simultaneously reduced by calcium magnesium acetate [18]. It was reported that the reaction efficiency of microwave reduction of NO<sub>x</sub> could be up to 98% when microwave energy was applied continuously [19]. It was also applied to a pyrolytic carbon such as activated carbon and char, enhancing the reaction of sulfur dioxide (SO<sub>2</sub>) and nitrogen oxides (NO<sub>x</sub>) with carbon [20]. The simultaneous treatment with the accelerated electronic beams and the microwaves could increase the removal efficiency of NO<sub>x</sub> and SO<sub>2</sub>, about 80% of NO<sub>x</sub> and more

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**Fig. 1.** Experimental flow loop of reduction of  $SO_2$  and  $NO_x$  with microwave ammonium bicarbonate over catalyst zeolite. (1)  $SO_2$  gas cylinder, (2)  $NO_x$  gas cylinder, (3) air compressor, (4) the bottle of gas mixture, (5) flow meter, (7) quartz tube, (7) microwave reactor, (8) outlet port, (G) sampling port.

than 95% of SO<sub>2</sub> were removed by precipitation with ammonia [21]. Under microwave, NO<sub>x</sub> gases adsorbed onto a char bed could be reduced to nitrogen and carbon oxides concurrently the char could be regenerated for reuse [22].

In this paper, experimental investigations were conducted to simultaneously desulfurization and denitrification from stimulated flue gas by the microwave with bicarbonate ammonium (NH<sub>4</sub>HCO<sub>3</sub>) and zeolite. The influence of inlet concentration, empty bed residence time (EBRT), microwave power on simultaneous desulfurization and denitrification were discussed, and the mechanism for microwave-induced catalytic SO<sub>2</sub> and NO<sub>x</sub> reduction was elicited.

#### 2. Materials and methods

#### 2.1. Microwave reaction system

The experimental flow loop used in the study was shown schematically in Fig. 1. A constant input microwave power of 119–462 W was used and the microwave frequency was 2450 MHz. The microwave reactor consisted of guartz tube (o.d. 25 mm and 85 mm long) with ammonium bicarbonate (NH<sub>4</sub>HCO<sub>3</sub>) and zeolite was set up to study removal of SO<sub>2</sub> and NO<sub>x</sub> from stimulated waste gas. The SO<sub>2</sub> and NO<sub>x</sub> supplied from the gas cylinders, were first diluted with the compressed air, passed through air mixture bottle and flowed upwards through the microwave reactor. The flow meter and the valve were used to monitor the gas flow through the reactor. SO<sub>2</sub> and NO<sub>x</sub> concentrations were monitored by the analysis device of S2000 flue gas, and gas flow rate was monitored by the rotameter and the mass flow controllers. In the process of the experiments, the simulated SO<sub>2</sub> and NO<sub>x</sub>-containing waste gas were supplied to the microwave reactor, at a flow rate of 200-5401/h (empty bed residence time (EBRT), 0.204-0.536 s).

#### 2.2. Measurement methods

The periodic measurements of the gas concentration from sampling ports in the quartz tube and the gas flow of the quartz tube in the microwave reaction system were carried out by using the following devices. S2000 flue gas device was used for the analysis of sulfur dioxide and nitrogen oxides. The rate of the gas flow was measured by 'Model LZB-1' flow meters with the units of 0.01 m<sup>3</sup>/h.

#### 3. Results and discussion

3.1. The influence of microwave power on simultaneous desulfurization and denitrification using ammonium bicarbonate only

Fig. 2 shows the effect of simultaneous desulfurization and dentrification by microwave reactor with different microwave powers using ammonium bicarbonate as reducing agent. It has no effect of



**Fig. 2.** Influence of microwave power on simultaneous  $SO_2$  and  $NO_x$  removal with ammonium bicarbonate only (reaction conditions: gas flow = 0.4 m<sup>3</sup>/h; inlet concentration of  $SO_2$  = 770 mg/m<sup>3</sup>; inlet concentration of  $NO_x$  = 180 mg/m<sup>3</sup>).

the microwave desulfurization with ammonium bicarbonate only. Fig. 2 indicates that no dentrification happened with less than 119 W, and the purifying efficiency of NO<sub>x</sub> is gradually increased from 0% to 11.1% when the microwave power is increased from 119 W to 280 W. However, the removal of NO<sub>x</sub> keeps no change with more than 280 W. In general, the effect of microwave dentrification using ammonium bicarbonate only is very low under our experimental conditions. The possible reason for this could be that the catalytic surface reaction temperature of microwave desulfurization and denitrification was 200–250 °C, obviously lower than the temperature of selective non-catalytic reduction (SNCR), 900–1100 °C, under microwave heating.

## 3.2. The influence of microwave power on simultaneous $SO_2$ and $NO_x$ removal using zeolite only

The effect of simultaneous desulfurization and dentrification by microwave reactor with different microwave powers using zeolite only is shown in Fig. 3. It has no effect of the microwave denitrification with zeolite, the possible explanation for this is that zeolite is not adsorptive  $NO_x$  due to absorb microwave energy. Fig. 3 indicates that zeolite is adsorptive  $SO_2$  under no microwave, and the purifying efficiency of  $SO_2$  is gradually decreased from 37% to 0% by means of microwave desorption when the microwave power is increased from 119 W to 280 W. The efficiency of desulfurization is zero with more than 280 W.



**Fig. 3.** Influence of microwave power on desulfurization and denitrification with zeolite only (reaction conditions: gas flow =  $0.4 \text{ m}^3/\text{h}$ ; inlet concentration of SO<sub>2</sub> = 770 mg/m<sup>3</sup>; inlet concentration of NO<sub>x</sub> = 180 mg/m<sup>3</sup>).



**Fig. 4.** Influence of desulfurization and denitrification with different microwave powers using ammonium bicarbonate and zeolite (reaction conditions: gas flow =  $0.4 \text{ m}^3/\text{h}$ ; inlet concentration of SO<sub>2</sub> =  $770 \text{ mg/m}^3$ ; inlet concentration of NO<sub>x</sub> =  $180 \text{ mg/m}^3$ ).

## 3.3. The influence of microwave power on simultaneous desulfurization and denitrification with ammonium bicarbonate and zeolite

Fig. 4 shows the influence of microwave power on simultaneous desulfurization and denitrification using ammonium bicarbonate as reducing agent and zeolite as catalyst. The conversion of desulfurization efficiency increases from 85% with 119 W microwave power to 98.1% with 280 W, and the conversion of denitrification efficiency increases from 58.5% with 119 W to 84.2% with 280 W, showing excellent desulfurization and denitrification effect by microwave reactor with the ammonium bicarbonate and zeolite.

Comparing Figs. 2 and 3 with Fig. 4, the microwave desulfurization and denitrification effect using ammonium bicarbonate and zeolite together are obviously much higher than that using ammonium bicarbonate or the zeolite only. The experimental results show that the optimum microwave power on desulfurization and denitrification simultaneously is supposed to be 211–280 W.

Based on the experiments as above, we assume that  $SO_2$ , NO<sub>x</sub> from flue gas can react with NH<sub>3</sub> decomposed from ammonium bicarbonate to produce sulfur, nitrogen when ammonium bicarbonate and zeolite are used together under microwave, which is critical to desulfurization and denitrification simultaneously. The ammonium bicarbonate does not absorb microwave energy and hence microwave would not induce SO<sub>2</sub> and NO<sub>x</sub> reduction without microwave absorbent such as zeolite. Zeolite does absorb microwave energy but require the reducing agent such as ammonium bicarbonate. The use of both zeolite and ammonium bicarbonate combined with microwave energy would induce SO<sub>2</sub> and NO<sub>x</sub> catalytic reduction reaction significantly. Thus, a major mechanism for microwave reduced desulfurization and denitrification can be described as the microwave-induced catalytic reaction (MICR) among SO<sub>2</sub>, NO<sub>x</sub> and ammonium bicarbonate with zeolite being the catalyst and microwave absorbent.

The principal reaction for the reduction of SO<sub>2</sub>, NO<sub>x</sub> by NH<sub>4</sub>HCO<sub>3</sub> can be represented as:

$$3SO_2(g) + 4NH_4HCO_3(s) \rightarrow 3[S](s) + 10H_2O(l) + 4CO_2(g) + 2N_2(g)$$
(1)

$$4NO(g) + 4NH_4HCO_3(s) + O_2(g) \rightarrow 4N_2(g) + 10H_2O(g) + 4CO_2(g)$$
(2)

where [S] denotes the various sulfur species  $(S_1, S_2, \ldots, S_8)$  in the gas phase. From thermo dynamic data it can be calculated that most of the sulfur will exist as diatomic molecules [1].

The decomposition of ammonium bicarbonate produces  $NH_3$ ,  $H_2O$ , and  $CO_2$  under microwave. The microwave-induced catalytic reduction (MICR) reaction among  $SO_2$ ,  $NO_x$  from the flue gas and ammonia ( $NH_3$ ) decomposed by ammonium bicarbonate can also proceed in a different way, giving rise to the product S,  $N_2$  and  $H_2O$  with zeolite acting as catalyst. Therefore, we assumed that the mechanism of the reactions might be followed as below:

$$NH_4HCO_3 \rightarrow NH_3 + H_2O + CO_2 \tag{3}$$

$$SO_2 + 8NH_3 + 2O_2 \rightarrow 4N_2 + 6H_2O + S$$
 (4)

$$4NO + 4NH_3 + O_2 \rightarrow 4N_2 + 6H_2O$$
(5)

$$6NO + 4NH_3 \rightarrow 5N_2 + 6H_2O \tag{6}$$

$$2NO_2 + 4NH_3 + O_2 \rightarrow 3N_2 + 6H_2O \tag{7}$$

## 3.4. The influence of empty bed residence time on simultaneous desulfurization and denitrification

The influence of empty bed residence time (EBRT) on simultaneous desulfurization and denitrification is presented in Fig. 5, under the conditions of microwave power of 259 W, inlet concentration of 660 mg/m<sup>3</sup> SO<sub>2</sub>, and inlet concentration of NO<sub>x</sub> 180 mg/m<sup>3</sup> with ammonium bicarbonate and zeolite. As keeping the inlet concentration of SO<sub>2</sub> and NO<sub>x</sub> at 660, 180 mg/m<sup>3</sup> respectively, the flow meter in the process should be changed step by step to make the mass loading of SO<sub>2</sub> and NO<sub>x</sub> changed proportionally, and the system should be stabilized for 3 min after every adjustment. In our experiment, 7-step adjustment was conducted to increase the empty bed residence time (EBRT) from 0.204 s at 0.54 m<sup>3</sup>/h air flow rate to 0.536 s at 0.2 m<sup>3</sup>/h air flow rate.

With empty bed residence time increasing, the efficiency of desulfurization increases from 88.9% to 99.1%, whereas the purifying efficiency of NO<sub>x</sub> increases from 77.8% to 86.5%. This indicates the longer empty bed residence time is a benefit on the removal of SO<sub>2</sub> and NO<sub>x</sub>, in case of the empty bed residence time (EBRT) it is too short to reduce SO<sub>2</sub>, NO<sub>x</sub> to sulfur, nitrogen before release. The



**Fig. 5.** Influence of empty bed residence time (EBRT) on desulfurization and denitrification (reaction conditions: the microwave power = 259 W; inlet concentration of SO<sub>2</sub> =  $660 \text{ mg/m}^3$ ; inlet concentration of NO<sub>x</sub> =  $180 \text{ mg/m}^3$ ).



**Fig. 6.** Influence of concentration of SO<sub>2</sub> in inlet on desulfurization and denitrification (reaction conditions: the microwave power = 259 W; the empty bed residence time (EBRT) = 0.315 s).

type of zeolite and the length of the quartz tube with catalyst and reducing agent are the key elements.

From Fig. 5, in our experimental conditions, we can assume the optimum empty bed residence time is 0.315 s in the microwave desulfurization and denitrification system, and about 98% sulfur dioxide or 83% nitric oxide in the gas stream is converted.

## 3.5. The influence of $SO_2$ and $NO_x$ concentration on simultaneous desulfurization and denitrification

Keeping the microwave power (259 W) and the empty bed residence time (EBRT) (0.315 s) fixed, the influence of concentration of SO<sub>2</sub> and NO<sub>x</sub> in inlet on simultaneous desulfurization and denitrification with ammonium bicarbonate and zeolite are presented in Figs. 6 and 7 separately. The SO<sub>2</sub> and NO<sub>x</sub> feed rate were increased stepwise retaining the air flow constant so that the  $SO_2$  and  $NO_x$ mass loading changed proportionally. After adjusting the SO<sub>2</sub> and  $NO_x$  concentrations the system was allowed to stabilize for 3 min before changing to another mass loading. The conversion of desulfurization efficiency reduces from 97.6% with 670 mg/m<sup>3</sup> to 79.5% with  $2100 \text{ mg/m}^3 \text{ SO}_2$  concentration, yet the denitrification effect changes small within the increasing inlet concentration of NO<sub>x</sub> from  $120 \text{ mg/m}^3$  to  $200 \text{ mg/m}^3$ . NO<sub>x</sub> concentration has little effect on denitrification but has no influence on desulfurization, SO<sub>2</sub> concentration has no affect on denitrification. This illustrates that the microwave reactor is efficient in purifying the waste gas whose



**Fig. 7.** Influence of concentration of NO<sub>x</sub> in inlet on desulfurization and denitrification (reaction conditions: the microwave power = 259 W; the empty bed residence time (EBRT) = 0.315 s).

 $SO_2$  concentration is between 670 and 2100 mg/m<sup>3</sup> and whose  $NO_x$  concentration is between 120 and 200 mg/m<sup>3</sup>.

#### 4. Conclusions

In order to facilitate the discussion of the results, we can summarize the main conclusions as follows:

- 1. The microwave reactor with the mixture of ammonium bicarbonate ( $NH_4HCO_3$ ) and zeolite can be used for simultaneous removal of sulfur dioxide ( $SO_2$ ) and nitrogen oxides ( $NO_x$ ) from waste gas. The best  $SO_2$  and  $NO_x$  purifying efficiency achieved 99.1 and 86.5%, respectively.
- 2. The effect of microwave dentrification is very low with ammonium bicarbonate (NH<sub>4</sub>HCO<sub>3</sub>) only. No effect on microwave desulfurization with ammonium bicarbonate. No effect on microwave denitrification with zeolite. From this study, it can be concluded that SO<sub>2</sub> is desorbed onto zeolite under microwave.
- 3. The microwave desulfurization and denitrification effect using ammonium bicarbonate and zeolite together are much higher than that using ammonium bicarbonate or the zeolite only.  $NO_x$  concentration has little effect on denitrification but has no influence on desulfurization,  $SO_2$  concentration has no effect on denitrification.
- 4. The optimal microwave power is 211–280 W, and the optimum empty bed residence time is 0.315 s.
- 5. Zeolites does absorb microwave energy for microwave-induced  $SO_2$  and  $NO_x$  reduction reaction with ammonium bicarbonate. The mechanism of microwave reduced desulfurization and denitrification can be described as the microwave-induced catalytic reaction between  $SO_2$ ,  $NO_x$  and ammonium bicarbonate with zeolite being the catalyst and microwave absorbent.

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