



## Microwave enhanced chemical reduction process for nitrite-containing wastewater treatment using sulfaminic acid

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Received 09 March 2009; revised 30 May 2009; accepted 05 June 2009

### Abstract

High-concentration nitrite-containing wastewater that presents extreme toxicity to human health and organisms is difficult to be treated using traditional biological process. In this study, a novel microwave-enhanced chemical reduction process (MECRP) using sulfaminic acid (SA) was proposed as a new manner to treat such type of wastewater. Based on lab-scale experiments, it was shown that 75%–80% nitrite ( $\text{NO}_2^-$ ) could be removed within time as short as 4 min under 50 W microwave irradiation in pH range 5–10 when molar ratio of SA to nitrite ( $\text{SA}/\text{NO}_2^-$ ) was 0.8. Pilot-scale investigations demonstrated that MECRP was able to achieve nitrite and chemical oxygen demand (COD) removal with efficiency up to 80% and 20%, respectively under operating conditions of SA concentration 80 kg/m<sup>3</sup>, SA/ $\text{NO}_2^-$  ratio 0.8, microwave power 3.4 kW, and stirring time 3 min. Five-day biological oxygen demand ( $\text{BOD}_5$ )/COD value of treated effluent after MECRP was increased from 0.05 to 0.36 (by 620%), which clearly suggested a considerable improvement of biodegradability for subsequent biological treatment. This study provided a demonstration of using microwave irradiation to enhance reaction between SA and nitrite in a short time, in which nitrite in wastewater was completely converted into nitrogen gas without leaving any sludge and secondary pollutants.

**Key words:** microwave-enhanced chemical reduction process, nitrite-containing wastewater, sulfaminic acid

**DOI:** 10.1016/S1001-0742(09)60074-7

### Introduction

Recently, the concentration of nitrite ion in water body has been significantly rising due to excessive industrial effluents, overused nitrogenous fertilizer and incomplete abiotic denitrification process in the soil (Seinfeld, 1989; Vorlop et al., 1992). Nitrite presents a serious toxicity to human health. For example, excessive nitrite accumulated in human body can form methaemoglobin and lead to “blue baby syndrome”. Besides, nitrite has a potential to convert into carcinogenic nitroamine and this compound may cause cancer and hypertension (Canter, 1997). For these reasons, the European Drinking Water Directive regulated the maximum permitted level of nitrite in drinking water to be 0.02 mg/L (calculated upon nitrogen, mg/L) (Dague, 1972; Holler et al., 2001). In China, the environmental standards for nitrite in surface water (calculated upon nitrogen, mg/L) of different water areas are 0.06 mg/L for grade I, 0.10 mg/L for grade III, 0.15 mg/L for grade III, 1.0 mg/L for grades IV and V (GB 3838-2002, 2002). Therefore, an emphasis must be placed on removing such high-concentration nitrite from wastewater before being discharged into natural water body.

Dimethyl sulfoxide (DMSO) is widely adopted as

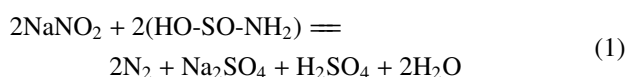
a chemical solvent for its low toxicity in electronic and chemical industries. However, the effluents of DM-SO production process contain high-concentration nitrite. Methods that can effectively remove nitrite include biological and physicochemical processes. Biological processes mainly involve courses of nitrification and denitrification, which require wastewater to be inherently biodegradable (Wang and Jing, 2005; Fux et al., 2004; Zhao et al., 1999). Additionally, biological process also puts a demand in concentration of nitrite in wastewater within a relatively low level; otherwise bacterium will be incapable of remaining viable and proliferating under toxic microenvironment. Physicochemical processes such as ion exchange, reverse osmosis, electro dialysis and liquid-phase catalytic reduction have been developed for the treatment of high-concentration nitrite wastewater (Albin et al., 1998; Florence et al., 2001; Hörold et al., 1993; Vorlop and Tacke, 1989; Yurii et al., 2000; Kormann et al., 1991). Nevertheless, these methods may often be problematic because of either much complex operation or great investment for post treatment. Despite obvious effectiveness of physicochemical processes for several certain types of nitrite wastewater treatment, each of them specifically accommodated certain type of nitrite wastewater.

The reaction between sulfaminic acid (SA) and nitrite in

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aqueous phase proceeds as follows (Xiao et al., 2005):



As given in Reaction (1), nitrite ( $\text{NO}_2^-$ ) is totally converted to  $\text{N}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{Na}_2\text{SO}_4$  and  $\text{H}_2\text{SO}_4$ , all of which produced are innocuous. However, the reaction that fails to fully proceed in short time might make SA wasted and leave much remaining nitrite in effluent. Hence, it should be inapplicable to directly utilize SA for nitrite removal in practical wastewater treatment.

Microwave is electromagnetic waves with wavelengths between 1 mm and 1 m (frequencies of 300 GHz to 300 MHz). Microwave radiation can make dipolar molecules rotate and ions migrate when applied to materials. The potential of microwave power encourages researchers to apply microwave for accelerating chemical reactions as an alternative to conventional conductive heating. Several benefits may be captured by the introduction of microwave irradiation such as rate enhancement, higher yields, and improved reaction selectivity. Many investigations on the application of microwave dielectric heating effects have been reported in the fields of organic synthesis (Zhang et al., 2003), chemical catalysis (Bond et al., 1993), solid state reactions (Abramovith et al., 1999), polymers (Estel et al., 2003; Menendez et al., 2002) and wastewater treatment (Bi et al., 2007; Zhang et al., 2005; Liu et al., 2004). The enhancement and advantage of microwave accelerating chemical reaction have been proved by several reports.

The main purpose of this study was to investigate the combinative effects of SA and microwave irradiation for removing high-concentration nitrite from wastewater in a microwave-enhanced chemical reduction process (MECRP). Significant operational parameters were optimized to maximize nitrite elimination for possible practical applications based on lab-scale and pilot-scale tests.

## 1 Materials and methods

### 1.1 Wastewater characterization

The real wastewater used in practical process was characterized as follows: nitrite ( $\text{NaNO}_2$ ) about 3000 mg N/L (calculated upon nitrogen), COD 8570–11,400 mg/L (including nitrite),  $\text{BOD}_5$  430–570 mg/L and pH 8–9. The synthetic wastewater used in lab-scale experiments was prepared with  $\text{NaNO}_2$  and distilled water, and the concentration of  $\text{NaNO}_2$  was 100 mg N/L (calculated upon nitrogen).

### 1.2 Chemicals

Sulfaminic acid (SA) ( $(\text{HO-SO}_2\text{-NH}_2)$ ) and  $\text{NaNO}_2$  used in lab-scale experiments were purchased from the Bodi Chemical Co., Tianjin, China (analytical grade, purity > 99.5%).  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  (Shuangchuan Chemical Co., Tianjin, China) were used to adjust solution pH. SA used in practical process (98% purity) was provided by Hebei Huanqiu Chemical Co., China.

### 1.3 Microwave generator

In lab-scale experiments, microwave generator (MCL-2) was reassembled from household microwave oven with a maximum power of 700 W and frequency of 2,450 MHz. In pilot-scale process of real nitrite-containing wastewater treatment, microwave generator was provided by Nanjing Jiequan Microwave Equipment Co., China. It had a structure of thin coil pipes for wastewater flowing through, which was more easily penetrated for the whole volume. The device was dominated by manipulative tank with a cooling system of circulating water. The microwave equipment holding the power of 0–16 kW could be conveniently controlled on its panel.

### 1.4 Operations of lab scale experiments

The lab-scale experiments were carried out in 200-mL beakers. A 100-mL synthetic nitrite-containing wastewater (100 mg N/L) was put into the beaker, and then followed by the addition of SA solution (10 g/L). In room-temperature stirring experiments, the solution was mixed at stirring rate of 150 r/min. In water bath heating experiments, the synthetic nitrite wastewater was heated up to 40°C in water bath prior to SA addition. While in microwave-irradiation experiments, the mixed solution was immediately transferred into microwave generator. Samples of the mixed solution were taken for physical and chemical analysis at certain time intervals.

### 1.5 Operations of pilot-scale MECRP

The schematic representation of MECRP in pilot-scale studies of practical process is given in Fig. 1 with the following steps: (1) SA solution and original nitrite wastewater were firstly pumped into mixing tower; (2) the mixture reacted in the tower, while gas and liquid was separated through separating boards inside; (3) the mixture was subsequently pumped into microwave generator for further reaction; (4) the mixture was then pumped into reaction tank and stirred before being discharged if reaction did not proceed sufficiently. After the treatment with MECRP, the removal of nitrite and COD was optimized.

### 1.6 Analytics and calculations

The COD and  $\text{BOD}_5$  in each water sample were measured following standard methods (APHA, 1995). Analysis of nitrite concentration was performed with spectrophotometer 752 (Shanghai 3rd Analysis Equipment Factory, China) by means of N-(1-naphthyl) ethylenediamine dihydrochloride spectrophotometric method. In order to check the reproducibility of the experimental results, random tests were performed for different experimental conditions.

Nitrite removing efficiency ( $R$ ) was calculated according to the following Eq. (2):

$$R = \frac{C_0 - C_e}{C_0} \times 100\% \quad (2)$$

where,  $C_0$  (mg/L) and  $C_e$  (mg/L) are nitrite concentrations before and after reaction, respectively. The COD removing efficiency was calculated in the same way.

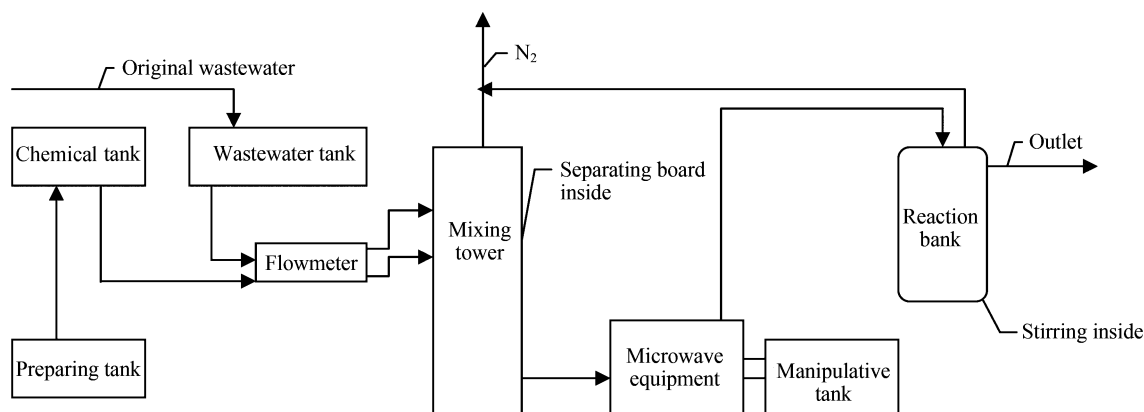


Fig. 1 Schematic legend of pilot-scale microwave-enhanced chemical reduction process (MECRP).

## 2 Results and discussion

### 2.1 Factors affecting nitrite removal

#### 2.1.1 Comparison of different reaction conditions

The reaction between SA and nitrite proceeded swiftly at the beginning. However, it hardly took place to a sufficient extent within short period. Hence, a longer hydraulic retention time (HRT) or SA waste was indicated. The acceleration using microwave or water bath was slight at initial stage when the reaction was violent. As shown in Fig. 2, for stirring experiments conducted in room temperature, 45.2% nitrite removal was achieved in the first 1 min at 150 r/min. Nevertheless, the nitrite removing efficiency reached 74.0% after 30-min stirring at room temperature and 76.3% after 4-min microwave irradiation at 50 W. Such difference in reaction time indicated that the reacting rate decreased rapidly after the first min just by stirring, and microwave irradiation could obviously enhance the reaction to a more sufficient extent in much shorter time.

A comparing study was performed among different influenced factors in terms of room temperature (20°C) stirring rate, water bath heating (40°C) and microwave irradiation. The temperature 40°C of water bath was selected in correspondence to the temperature after 4-min microwave irradiation (50 W), when the reaction under microwave irradiation condition fully proceeded. As can be seen from Fig. 2, upon the same reaction time,

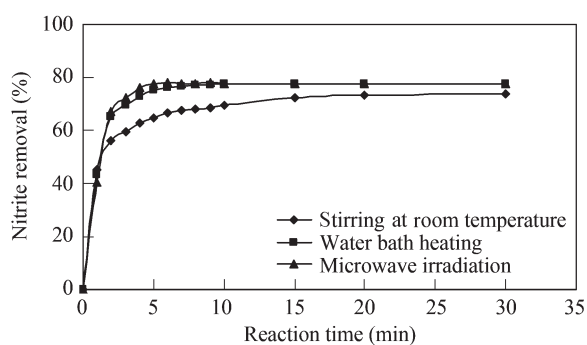


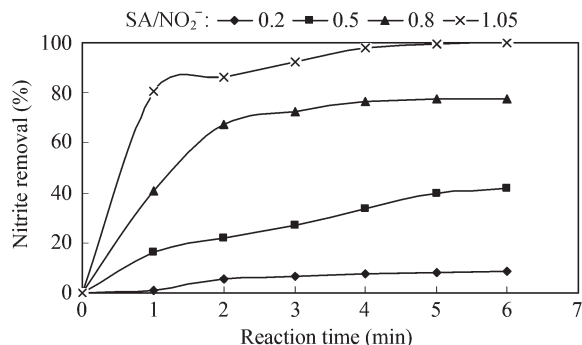
Fig. 2 Nitrite removal under room temperature (20°C) stirring (150 r/min), water bath heating (40°C) and microwave irradiation (50 W) at molar ratio 0.8:1 and sulfaminic acid concentration 10 g/L.

the nitrite removals under conditions of both water bath heating and microwave irradiation were higher than that under room temperature stirring. Furthermore, the same nitrite removal (74%) required reaction of 30 min under room temperature stirring while 3–4 min under water bath heating and microwave irradiation. This clearly indicated that high temperature was more favourable for nitrite removal than individual stirring and decreasing amount of SA and shortening HRT of the integrated treatment system could be accomplished.

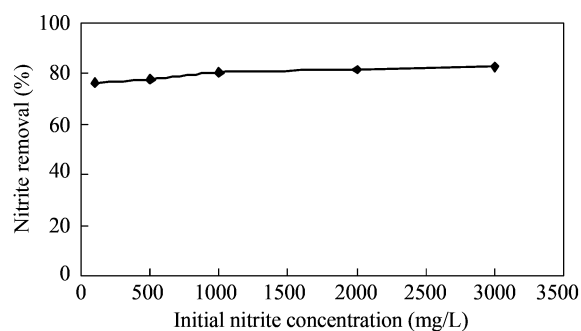
As addressed in previous studies, microwave had dielectric heating effects (Bi et al., 2007; Zhang et al., 2005; Liu et al., 2004), which would fasten the rate of chemical reaction. As illustrated in Fig. 2, the nitrite removal reached a sufficient extent after 4 min for microwave irradiation, and 8 min for bath heating. In comparison to traditional water bath heating, the microwave irradiation could either achieve a better nitrite removal upon the same reaction time or shorten total time for sufficient reaction. Hence, nitrite removal enhanced by microwave irradiation might attribute to both conductive and dielectric heating effects. Besides, the microwave technology had advantages in term of swiftness and more energy-efficiency, and should be preferable in practical utilizations for nitrite treatment.

#### 2.1.2 Effect of molar ratio of SA to nitrite

According to Reaction (1), the amount of input SA plays a key role in nitrite removal and the theoretical nitrite removal efficiency should be equal to the molar ratio of SA to nitrite ( $SA/NO_2^-$ ). As revealed in Fig. 3, nitrite removal efficiency was lower than theoretical values at  $SA/NO_2^-$  of 0.2 and 0.5 while it was almost equal to theoretical values at  $SA/NO_2^-$  of 0.8 and 1.05. This suggested that increasing  $SA/NO_2^-$  could achieve more sufficient  $NO_2^-$  removal. The  $SA/NO_2^-$  of 0.8 was determined as optimum input molar ratio of SA to nitrite for optimizing nitrite removal (above 80%), thus, reducing nitrite concentration to accepted level for post biotreatment due to the fact that lower nitrite concentration could make the effluent more biodegradable for its lower toxicity. Furthermore, some bacteria developed in biological process in terms of nitrite-oxidizing bacteria (NOB) were capable of converting nitrite to nitrate with nitrite serving as electron donor under aerobic concentration (Verstraete et al., 1977).



**Fig. 3** Nitrite removal as function of irradiation time at different molar ratios of SA to nitrite. SA concentration: 10 g/L, microwave power: 50 W.



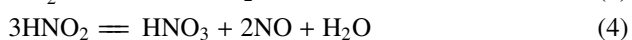
**Fig. 4** Effects of initial nitrite concentration on nitrite removal. Molar ratio of SA/NO<sub>2</sub><sup>-</sup>: 0.8:1; microwave power: 50 W; stirring time: 4 min.

### 2.1.3 Effect of initial nitrite concentration

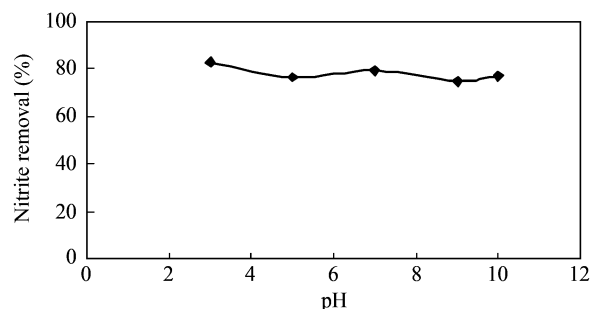
Experiments were also carried out to examine the effects of optimum SA/NO<sub>2</sub><sup>-</sup> on nitrite removal at high strength. The nitrite removal did not show significant difference for various initial nitrite concentrations (Fig. 4). In addition, at SA/NO<sub>2</sub><sup>-</sup> of 0.8, the nitrite removal was slightly increased for even high nitrite concentration. This is mainly attributed to the fact that the mixture of SA and high-concentration nitrite would have more probability to interact, which is responsible for the greater nitrite removal. The results implied that the optimum molar ratio of SA/NO<sub>2</sub><sup>-</sup> (0.8) could be available in nitrite removal over a wide initial nitrite concentration range.

### 2.1.4 Effect of pH

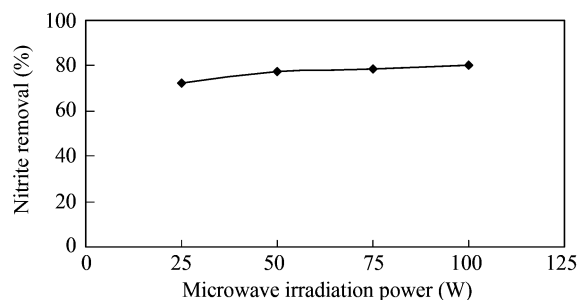
Five sets of experiments in parallel with initial pH of 3, 5, 7, 9 and 10 were taken to study the influence of solution pH on nitrite removal. According to the results given in Fig. 5, the nitrite removal with pH of 3, 9 and 10 reached 82.7%, 74.7% and 77.0%, respectively. The greater removal at pH 3 could be explained according to Reactions (3)–(5) (Xiao et al., 2005).



It was deduced that more HNO<sub>2</sub> was decomposed to NO and then further oxidized to NO<sub>2</sub> under acidic condition. However, it should be also noticed that both NO and NO<sub>2</sub> gas contribute much to atmosphere and had a potential corrosive influence on metallic equipments. Therefore, acidic



**Fig. 5** Nitrite removal as function of pH value. Molar ratio of SA/NO<sub>2</sub><sup>-</sup>: 0.8:1; SA concentration: 10 g/L; microwave power: 50 W, stirring time: 4 min.



**Fig. 6** Nitrite removal as function of microwave irradiation power. Molar ratio of SA/NO<sub>2</sub><sup>-</sup>: 0.8:1; SA concentration: 10 g/L, stirring time: 4 min.

reaction condition should be avoided. The experimental results demonstrated that at pH range 5–10, 75%–80% nitrite removal could be achieved without NO<sub>2</sub> production. Taking into account that wastewater in practical process often had pH value of 8–9, there is no additional need to adjust pH of original wastewater during the treatment. As a result, it could reduce much cost of chemicals for actively adjusting pH compared to some other processes for nitrite wastewater treatment (Albin et al., 1998; Florence et al., 2001; Hörold et al., 1993; Vorlop and Tacke, 1989; Yurii et al., 2000).

### 2.1.5 Effect of microwave irradiation power and time

Figure 6 clearly shows that the nitrite removal under microwave irradiation was promoted when irradiation power was increased from 25 to 50 W and then enhanced slightly with further increase in power. The most likely reason for such increase was that the reaction between SA and nitrite was an exothermic reaction. When increasing power or prolonging running time, the restraining high temperature showed more remarkable effects than the microwave-accelerating effect; and thus, no apparent contribution had been made to improve the nitrite removal. Based on the above possible reasons, reaction time of 4 min and power of 50 W were chosen for 100 mL synthetic wastewater treatment in lab-scale investigations.

## 2.2 MECRP for nitrite wastewater treatment

### 2.2.1 Proposal of MECRP

Based on the results of lab-scale experiments performed above, MECRP was proposed to deal with real high-concentration nitrite wastewater in practical process. In

**Table 1** Effect of operating parameters on nitrite removal in pilot-scale MECRP

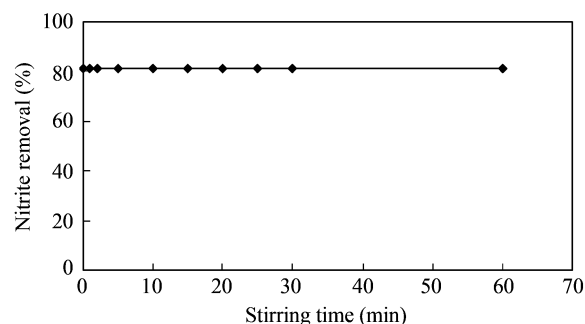
Experimental number	SA concentration (kg/m <sup>3</sup> )	Flow ratio ((L/hr):(L/hr))	Molar ratio (mol/mol)	Microwave power (kW)	Stirring time in reaction tank (min)	Nitrite removal (%)
1	20	600:120	0.20	2.8	3	19.5
2	20	800:160	0.20	3.4	5	16.5
3	20	1000:200	0.20	4.0	1	14.3
4	50	600:120	0.50	2.8	1	47.2
5	50	800:160	0.50	3.4	5	44.5
6	50	1000:200	0.50	4.0	3	42.4
7	80	600:120	0.80	2.8	1	82.8
8	80	800:160	0.80	3.4	3	81.2
9	80	1000:200	0.80	4.0	5	80.6
10	100	600:120	1.00	2.8	3	98.7
11	100	800:160	1.00	3.4	5	97.5
12	100	1000:200	1.00	4.0	1	95.4

this process, SA was added and mixed with nitrite wastewater for primary fast reaction, which was followed by microwave irradiation to accelerate the reaction to a more sufficient extent (Fig. 1).

### 2.2.2 Operating parameter optimization

The concentration of original nitrite in wastewater in practical process was about 3,000 mg N/L (calculated upon nitrogen) while the optimized nitrite removal approximately ranged from 80% to 90% in subsequent biological process. Table 1 shows operating parameters, including SA concentration, mass flow ratio (by adjusting molar ratio), microwave irradiation power as well as stirring time in reaction tank. As shown in Fig. 1, SA concentration was adjusted in preparing tank by varying certain concentration of SA solution. Flow ratio was adjusted by flowmeters, which presented the rate ratio of nitrite wastewater to SA solution and controlled the molar ratio of SA to nitrate (SA/NO<sub>2</sub><sup>-</sup>). Microwave power was adjusted by manipulative tank and evaluated by checking water temperature rising on panel. Stirring time in reaction tank was determined by judging the sufficient level of effluent. The nitrite removals in reaction tank were analyzed in some stirring intervals and compared to evaluate the enhanced performances of microwave irradiation.

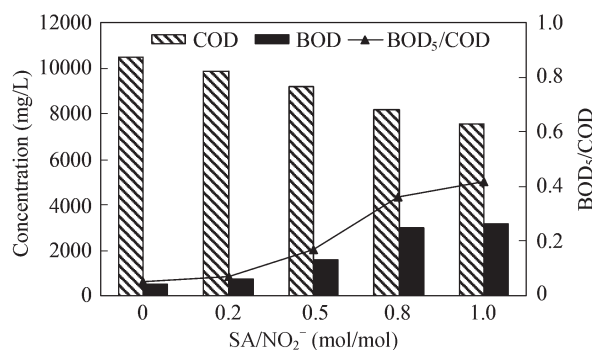
A series of pilot-scale experiments were conducted to identify the optimum operating parameters. With the comparison of nitrite removal in Table 1, the parameters in the 8th group appeared to be the optimum ones corresponding to maximum nitrite removal in the process. It operated under the conditions of SA concentration 80 kg/m<sup>3</sup>, flow ratio (nitrite wastewater to SA is (800 L/hr):(160 L/hr) (molar ratio 0.8), microwave power 3.4 kW and stirring time 3 min in reaction tank. The results obtained from the optimized group from pilot-scale studies were similar to that obtained from lab-scale experiments. However, temperature rising was found lower than that in lab-scale experiments. The temperature of mixture flowing through the microwave reactor rose by 5°C higher, which facilitated the reaction between SA and nitrite more completely. It was also confirmed by the nitrite removal experiment in reaction tank, i.e., the nitrite removal in reaction tank remained at the same level after 60 min stirring (Fig. 7). The results obtained above indicated a fact that the microwave irradiation remarkably accelerated the reaction to



**Fig. 7** Effects of stirring time in reaction tank on nitrite removal after microwave irradiation. Molar ratio of SA/NO<sub>2</sub><sup>-</sup>: 0.8:1; SA concentration: 80 kg/m<sup>3</sup>, microwave power: 3.4 kW.

a more sufficient extent by raising temperature magnitude of 5°C. It was speculated that the microwave generator in pilot-scale experiments had thin coil pipes inside that was more easily for the penetration of microwave irradiation. Therefore, the utilizing efficiency of microwave in pilot-scale experiments was much better than that in lab-scale experiments.

As discussed above, lowering nitrite concentration in wastewater could make the effluent after microwave irradiation much more biodegradable in subsequent biological process. The results of pilot-scale experiments in Fig. 8 showed that the addition of SA could remove partial COD and thus enhance the biodegradability of organic compounds as indicated by increasing BOD<sub>5</sub>/COD ratio.



**Fig. 8** Effect of SA to nitrite molar ratio (SA/NO<sub>2</sub><sup>-</sup>) on the change of COD, BOD and BOD<sub>5</sub>/COD in treated effluent of MECRP. Nitrite wastewater to SA flow ratio: 800 L/hr:160 L/hr; microwave power: 3.4 kW; stirring time: 3 min.

In fact, the COD removal was equal to the part that nitrite courted. Despite insignificance in COD removal observed, nitrite reduction largely decreased the toxicity of high-concentration nitrite in original wastewater to microorganisms in bioprocess. With the optimum parameters given in the 8th group, the MECRP achieved over 80% nitrite and about 20% COD removal while enhancing BOD<sub>5</sub>/COD ratio from 0.05 to 0.36 simultaneously. For the case of increase in either wastewater quantity or nitrite concentration, parameters tested in 9th–12th groups in Table 1 should be chosen, although this might account for more SA and energy consumption.

### 3 Conclusions

In this study, microwave-enhanced chemical reduction process (MECRP) was successfully developed and applied to treat high-concentration nitrite-containing wastewater in both lab-scale and practical process. With designed MECRP, over 80% nitrite and about 20% COD removal were achieved, and BOD<sub>5</sub>/COD of treated effluent was increased greatly from 0.05 to 0.36 (by 620%), both of which made it more available for subsequent biological treatment process.

The reaction between SA and nitrite could be substantially enhanced by microwave irradiation. For synthetic nitrite-containing wastewater in lab-scale experiments, best performances of nitrite removal were obtained by optimizing operating parameters as molar ratio of SA to nitrite (SA/NO<sub>2</sub><sup>-</sup>) of 0.8, irradiation time of 4 min and power of 50 W. A wide pH range 5–10 was applicable in pilot-scale MECRP for conversion of nitrite to nitrogen gas without NO<sub>2</sub> production. In contrast to stirring and water bath heating methods, microwave irradiation revealed several advantages in terms of swiftness, sufficiency and energy efficiency.

The pilot-scale experiments conducted with real wastewater showed that MECRP could achieved optimum nitrite and COD removal under operating conditions of SA concentration 80 kg/m<sup>3</sup>, SA/NO<sub>2</sub><sup>-</sup> 0.8, microwave power 3.4 kW and stirring time 3 min in reaction tank. This should be in well consistence with that obtained in lab-scale experimental results only except the lower temperature rising associated with the higher microwave irradiation efficiency.

### Acknowledgments

This work was partly supported by the National Natural Science Foundation of China (No. 50678045).

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