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Removal of ammonia nitrogen in wastewater by microwave radiation: A pilot-scale study

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ABSTRACT

A large removal of ammonia nitrogen in wastewater has been achieved by microwave (MW) radiation in our previous bench-scale study. This study developed a continuous pilot-scale MW system to remove ammonia nitrogen in real wastewater. A typical high concentration of ammonia nitrogen contaminated wastewater, the coke-plant wastewater from a Coke company, was treated. The output power of the microwave reactor was 4.8 kW and the handling capacity of the reactor was about 5 m^3 per day. The ammonia removal efficiencies under four operating conditions, including ambient temperature, wastewater flow rate, aeration conditions and initial concentration were evaluated in the pilot-scale experiments. The ammonia removal could reach about 80% for the real coke-plant wastewater with ammonia nitrogen concentrations of 2400–11000 mg/L. The running cost of the MW technique was a little lower than the conventional steam-stripping method. The continuous microwave system showed the potential as an effective method for ammonia nitrogen removal in coke-plant water treatment. It is proposed that this process is suitable for the treatment of toxic wastewater containing high concentrations of ammonia nitrogen.

1. Introduction

Water quality deterioration and eutrophication of lake, river and costal waters in China have attracted more and more attention in the last few decades [1]. Most lakes are commonly undergoing the eutrophication process, water quality has decreased and lake ecosystems are being declined [2]. High concentrations of ammonia nitrogen are commonly present in industrial wastewaters such as coke-plant, tannery, textile, landfill leachate and fertilizer wastewater [3]. The discharge of these industrial wastewaters is one of the most important sources of ammonia nitrogen. In the past years, great efforts have been devoted to the removal of ammonia nitrogen from wastewater. Traditional methods for the removal of high concentration of ammonia nitrogen include biological systems [4], chemical precipitation [5], supercritical water oxidation [6,7], steam-stripping [8,9] and so on. The ammonia concentration after biological treatment is still high because the high concentration of ammonia leads to the low ratio of C/N [10]. As a result, biological processes are usually difficult to meet the discharge standards [11]. Chemical precipitation needs additional reagents, which may introduce new pollutants to the water body [5]. Supercritical water oxidation is operated at high temperatures (>400 °C) and high pressures (>20 MPa) [6]. In most coke companies, the steam-stripping method is widely used for the removal of ammonia nitrogen [8]. Steam-stripping method uses a large stripping tower, which consumes much energy, and the ammonia concentration in effluent is often very high [9]. As a consequence, it is necessary to develop a cost-effective technique for the removal of high concentrations of ammonia nitrogen in industrial wastewater.

In the last several years, microwave (MW) technique was used in environmental remediation, especially in wastewater treatment. It had been applied to eliminate dyes [12], invasive organisms [13], pentachlorophenol [14], phenol [15] and so on in wastewaters. Recently, we found that MW radiation could be used to remove ammonia nitrogen in wastewater, and large removal efficiencies were achieved in the bench-scale experiments [16]. It was noted that pH and radiation time had significant influence on the removal of ammonia nitrogen, initial ammonia concentration and aeration had minute influence. The mechanism for the ammonia removal was proposed as the evaporation of NH_3 by MW radiation. Although MW technique is proved to be effective for the removal of high concentration of ammonia nitrogen in wastewater, it is still doubtful for the full scale application.

As the continuity of the previous bench-scale study, a continuous pilot-scale MW system was designed in this study to remove ammonia nitrogen in wastewater. A typical high concentration of ammonia wastewater, the coke-plant wastewater from Coke Company of Wuhan Iron and Steel (Group) Corporation in Wuhan city,

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was treated. The removal efficiencies of ammonia nitrogen and the energy consumption were investigated, and a comparison between MW technique and conventional steam-stripping method was also performed. This effort was undertaken in order to obtain useful information for the scale-up of MW process to full scale application.

2. Experimental

2.1. MW energy calculation

The capacity of the pilot-scale reactor was designed to be about 5 m^3 wastewater per day. Eq. (1) was used to calculate the power of the MW reactor based on energy balance and temperature difference between inlet and outlet of the MW heating cavity [17].

$$Q = Cm\Delta T \qquad (1)$$

where Q is the absorbed power in W, C is specific heat capacity of wastewater in J/(kg °C) (assumed 4.2×10^3 J/(kg °C), the same as H_2O), m is the mass flow rate in kg/s (0.0579 kg/s, the density of the wastewater was assumed 1×10^3 kg/m^3, the same as H_2O) and ΔT is the change of temperature in °C.

In our previous work, it was found that thermal effect played a key role on ammonia removal [16]. The removal was minute at low temperatures and increased sharply at temperature above 80 °C, particularly when the wastewater was boiling [16]. In order to get a high ammonia removal, we intended to heat the wastewater to approximately 100 °C by MW radiation. In the coke company, the initial temperature of the wastewater was about 75 °C, the wastewater was firstly heated to 90 °C, and then pumped into the stripping tower to strip off the ammonia. The wastewater used in this study was the influent of a conventional steam-stripping tower in the coke company. Temperature of the used wastewater was about 90 °C, but the wastewater needed to be mixed with lye to adjust the pH and went through the pipeline to the MW reactor, which decreased the temperature to about 80 °C. Then the temperatures of the influent and effluent wastewater were 80 °C and 100 °C, respectively, with the difference of 20 °C. The total heat needed to treat wastewater (5 m^3/d) was calculated to be 4.8 kW ($Q = Cm\Delta T = 4.2 \times 10^3$ J/(kg °C) $\times$ 0.0579 kg/s $\times$ $(100 - 80)$°C = 4863.6 W $\approx$ 4.8 kW). Presently, the magnetron, which is widely used as a MW generator [18], generates a useful power of 600 W. So eight magnetrons were needed to generate a maximum output power of 4.8 kW. Palafox [19] developed a MW apparatus with a maximum output power of 5 kW to degrade plastic wastes, four magnetrons were used and each of them were controlled by a separated switch so that the output power could be controlled at 25, 50 or 75% of the maximum. In this present work, the eight magnetrons were also controlled by eight separated switches on the MW control panel.

2.2. MW reactor design

The schematic diagram of the MW reactor is shown in Fig. 1. The output power of the reactor was 4.8 kW, and the frequency was 2450 MHz. The shell of the MW reactor was made by stainless steel in order to resist corrosion and facilitate fabrication. A cylindrical-shaped glass tube was inserted vertically in the cavity chamber of the MW unit and fixed in place with an angle iron bracket. The glass tube was specially made of boron glass which could stand very high temperatures and with a great intensity, with an inner diameter of 22 cm and a length of 205 cm. The glass tube consisted of three parts, the upper one, the middle one and the bottom one. The three parts were connected by flanges. Effective water volume of the reactor was about 28 L. A bubble tube made of boron glass was placed in the glass tube to provide fine air bubbles. Fine air bubbles increased the air/water contact surface area, which enhanced the contaminant phase-transfer process [20]. The MW unit was comprised of magnetrons, waveguides and other accessories. In order to achieve a uniform heating, eight MW magnetrons were arranged evenly around the glass tube. Four magnetrons can be seen in Fig. 1, with the other four magnetrons situated behind them.

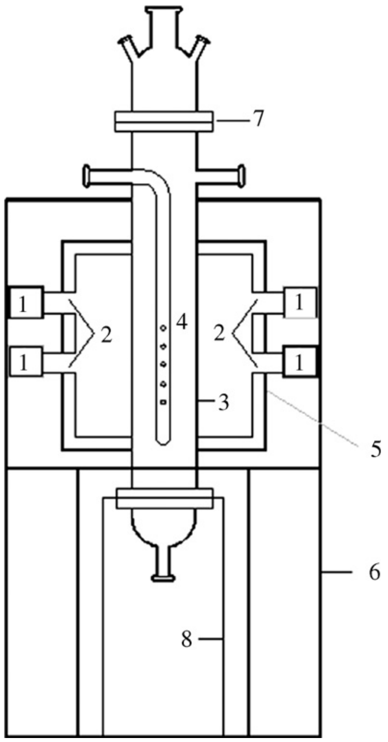

Fig. 1. Schematic diagram of the MW reactor. (1) Magnetron, (2) wave guide, (3) glass tube, (4) bubble tube, (5) cavity chamber, (6) shell, (7) flange, (8) bracket.

2.3. Continuous pilot-scale system

The pilot-scale system is shown schematically in Fig. 2. It consisted of a MW reactor, lye mixing chamber, two flowmeters (LZB-15F, Hangzhou Heshan instrument Factory, China), two pumps (MP-20RX, Zhejiang Xishan Pump Ltd., China), air compressor (HG370, Shanghai Fuli Electromotor Factory, China) and other accessories. Since the pH of the wastewater was above 11, the material of the pipes for connection of each part should resist corrosion. Therefore, galvanized steel pipes were used here due to their great capability to resist corrosion in strongly alkaline pH. The continuous pilot-scale system was installed at Coke Company of Wuhan Iron and Steel (Group) Corporation in Wuhan city.

The wastewater used was the influent of a conventional steam-stripping tower in the coke company. The initial ammonia concentration in the real coke-plant wastewater varied from 2400 mg/L to 11,000 mg/L and the initial pH was about 9. The lye used was a mixture of sodium hydroxide (industrial pure) and tap water at concentration of about 3 g/L. At the start of the run, wastewater was mixed with lye in the mixing chamber to reach pH around 12, at which the ammonia nitrogen in the wastewater was converted into ammonia molecule [16]. Then mixed wastewater was pumped into the reactor from the bottom of the glass tube at different flow rates. When the glass tube was filled with wastewater, the switches on the control panel of MW reactor were turned on. The magnetrons launched electromagnetic wave through wave guide to the reactor, and continuous-flow wastewater was treated by MW radiation. Clean air was supplied to the reactor by an air compressor through the bubble tube to facilitate ammonia nitrogen

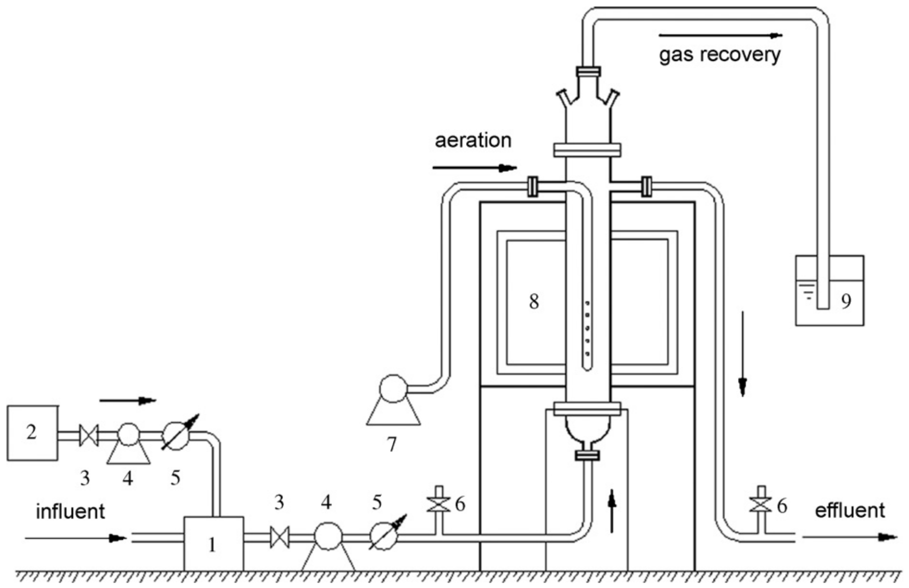

Fig. 2. Flow sheet of the continuous-flow pilot-scale system, (1) mixing chamber, (2) lye, (3) valve, (4) pump, (5) flowmeter, (6) sampling port, (7) air compressor, (8) MW reactor, (9) dilute sulphuric acid.

transfer from the aqueous phase to vapor phase. The wastewater flowed continuously from the bottom to the top of the glass tube. Ammonia gas produced in the MW and aeration process was recovered through a collapsible tube by dilute sulphuric acid to produce ammonium sulphate, which could be used as fertilizer. In the aeration experiments, each experiment was conducted using an airflow rate of about 30 L/min. Two regulating valves were attached to control the flow rates of wastewater and lye. Water samples of the influent and effluent were taken at different running times. Ammonia concentrations in the samples were measured by WT-1 portable apparatus of ammonia nitrogen analysis (Wuhan Water Environmental Protection Company, China). Removal efficiencies were calculated as $E = 1 - (C_{out}/C_{in})$. Effluent pH was measured by a portable pH meter (Hi8424new, Hanna, Italy).

3. Removal of ammonia in the pilot-scale system

In this pilot-scale study, the effects of four operating conditions, including ambient temperature, wastewater flow rate, aeration condition and initial average concentration were investigated. The experimental conditions for each experiment are listed in Table 1. All experiments were conducted between July and September 2007 at the ambient temperatures of 24–37 °C. Each experiment lasted 90 min. The removals for each experiment are displayed in Fig. 3. It could be seen that ammonia removal increased with MW radiation time at the beginning of the process and attained a plateau after about 40–70 min, indicating that the system was steady-state after 40–70 min. It was observed that the wastewater in the MW reactor was boiling at steady state. Fig. 4 shows the pH change under different operation conditions. The initial pH of the wastewater was adjusted to around 12 and the pH decreased due to the escape of NH_3 during the MW process. A linear relationship between pH and ammonia removal was found in the five experiments, the equations and the relationship coefficiencies between pH and removal is shown in Table 2.

3.1. Ambient temperature

The main difference between experiment 1 and experiment 2 was ambient temperature, which affected the temperature of the wastewater in microwave reactor. As shown in Fig. 3, a higher ambient temperature demonstrated higher ammonia removal. The ammonia removal achieved 84% when ambient temperature was 35 ± 2 °C, but only 78% when the temperature was 26 ± 2 °C. In bench-scale study, it was concluded that higher temperature of wastewater induced more impetuous molecule motion and faster mass transfer and benefited the elimination of ammonia nitrogen [16]. Temperature of influent wastewater from the coke company was about 90 °C. When wastewater went through pipelines to MW reactor, the temperature decreased because of the dissipation of heat and the mixing with lye. When ambient temperature was high, less heat was lost, and the temperature of wastewater flowed into the reactor would be higher, which was beneficial for the removal of ammonia nitrogen. It can be concluded that cold weather conditions reduced the effectiveness of MW reactor. The pH drops in the process also suggested the removal of ammonia, because the escape of ammonia from wastewater led to the decrease of pH. The pH of experiment 1 decreased from 11.8 to about 10.2, and the pH of experiment 2 decreased from 11.6 to about 10.2.

3.2. Wastewater flow rate

Experiment 3 was run under similar conditions as experiment 1, except the flow rate of wastewater. Fig. 3 reveals that the ammo-

Table 1
Experimental conditions.

Experiment no.	Initial average concentration (mg/L)	Bubble flow rate (L/min)	Wastewater flow rate (L/min)	Ambient temperature (°C)
1	2400	30	2	35 ± 2
2	2600	30	2	26 ± 2
3	2900	30	3	35 ± 2
4	2850	0	3	35 ± 2
5	11000	0	3	35 ± 2

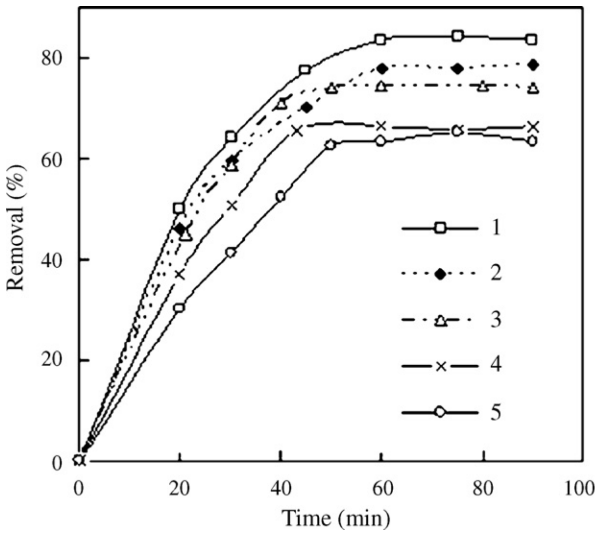

Fig. 3. Ammonia nitrogen removal under different operating conditions (1, 2, 3, 4, 5 denote experiments 1, 2, 3, 4, 5, respectively).

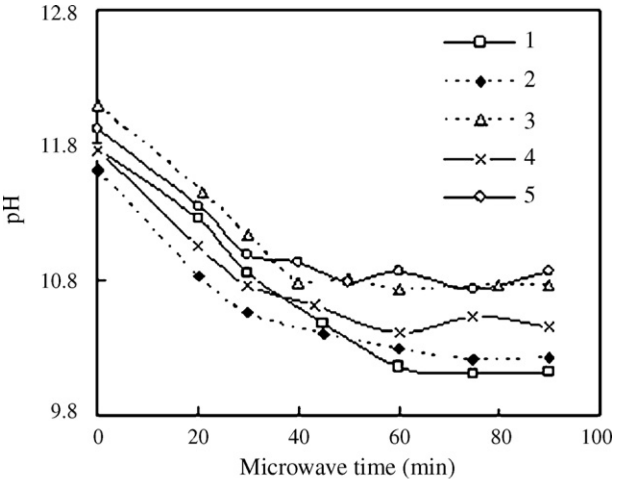

Fig. 4. pH under different operating conditions (1, 2, 3, 4, 5 denote experiments 1, 2, 3, 4, 5, respectively).

nia removals were 84% and 74% at the flow rate of 2 L/min and 3 L/min, respectively. The system needed about 60 min at 2 L/min and about 40 min at 3 L/min to reach steady-state. It is evident that there was a higher heating effect produced at lower flow rate due to the increased residence time inside the microwave reactor. The potential application for microwave heating is dependent on the dielectric properties of the target material [20]. In this study, wastewater containing high concentration of ammonia was the only target material. Water is a typical non-symmetric molecule, which makes it a good MW absorber [21]. Hence, a uniform bulk heating could be achieved in the MW process. Fig. 4 shows that the pH of experiment 1 reduced from 11.8 to about 10.2, and then reached a plateau after 70 min, and pH of experiment 3 reduced from 12.1 to about 10.7, and then reached a plateau after 40 min.

Table 2
The equations and relationship coefficiencies between removal of ammonia nitrogen and pH during the MW process (y and x denote removal of ammonia nitrogen and pH, respectively).

Experiment	Equations	Coefficient (R^2)
1	$y = -29.243x + 380.71$	0.9817
2	$y = -54.644x + 637.75$	0.9892
3	$y = -42.134x + 527.45$	0.9889
4	$y = -50.103x + 591.61$	0.9295
5	$y = -62.337x + 735.61$	0.8616

3.3. Aeration condition

Experiment 3 and experiment 4 had the same operation conditions except aeration. The two systems both reached steady state after 40 min, with the removal and pH remaining both almost unchanged. The airflow rate of 30 L/min was used in experiment 3. Fig. 3 demonstrates that the ammonia removal with aeration was about 9% higher than that without aeration. This result was consistent with bench-scale experiments [16], where aeration showed no significant influence on the removal when the operating conditions were favorable for ammonia removal. Herein, the operating conditions were not sufficient to remove all the ammonia in wastewater. Thus, aeration resulted in the increase of ammonia removal.

3.4. Initial concentration

The only difference between experiments 4 and 5 was the initial concentration, but almost the same removal efficiencies and pH drops were obtained. The same results were also found in bench-scale experiments.

Summarily, a low ambient temperature and higher flow rate reduced the effectiveness of the MW reactor. Both the bench-scale and pilot-scale experiments demonstrated that initial concentration had minute influence on ammonia removal, and the removal could be enhanced by about 9–10% with aeration. The ammonia removal efficiency of the pilot-scale system could reach 74–84% for real coke-plant wastewater with aeration and a high ambient temperature. Furthermore it could be observed that the results from the pilot-scale study generally conformed to those from the laboratory-scale study.

4. Economical analysis

Developing a cost-effective technique depends on various factors such as effectiveness, cost, safety, and ease of operation. The test results indicated that the continuous pilot-scale microwave system in the present study could be an effective method for ammonia removal. An energy consumption and economical analysis were necessary, in order to determine the practicability of this technology.

We compared the economical expenditure of the MW technique with that of the conventional steam-stripping method. Since both methods needed to adjust the wastewater pH to 11 before treatment, the comparison was focused on the energy consumption. In a MW system, electric energy is used as the energy source and translated into MW energy [22]. The energy conversion efficiency from electric energy to microwave energy is 50–70%, with the conversion efficiency of our MW reactor being 65%. Since the output MW power value of our MW reactor was 4.8 kW, the input electric power value should be approximately 7.4 kW, and the treat capacity of the reactor was about 5 m^3 wastewater per day. Hence, the total energy consumed per hour by MW system to heat the wastewater was 35.5 kWh/m^3. The market price of electricity for industrial use was $ 0.081/kWh in China. Thus, the running cost for treating 1 m^3 wastewater with an energy input of 7.4 kW would be about $ 2.88. Regarding the steam-stripping method in the Coke Company of Wuhan Iron and Steel (Group) Corporation, the cost was mainly spent on the steam. According to the running record of the system in the Coke company, 0.3 m^3 steam (5 Mpa) was needed to treat 1 m^3 wastewater, and the average market price of steam was $ 11.70 per m^3. Hence, the cost needed to treat 1 m^3 wastewater was about $ 3.51. It could be concluded that the running cost of the MW technique was a little lower than steam-stripping method. The temperature of the glass tube in the MW cavity would always be very high under MW radiation, which led to a short life of the glass tube. Thus, it needed to be replaced regularly. The regular replacement of the glass tube in the MW cavity would also increase the running costs of the MW system. Hence, the economical advantage of the MW technology was not obvious compared with conventional steam-stripping method. The key point affected the economical cost of the MW technique is the energy utilization. In the present system, only 65% electric power could be transformed into microwave energy. If the energy utilization enhanced, the energy consumption and the economical cost would decrease.

Boldor et al. [13] have demonstrated that MW radiation was effective for ballast water treatment, but they thought the environmental advantages of this technology cannot be overlooked because of current high treatment costs. The cost of their system without a heat exchanger was calculated about \$ 2.55/m^3. Nevertheless, the authors did not consider the energy conversion efficiency from electric energy into MW energy. If they considered it, the cost would be enhanced 40% at least. They thought this technique could be added as a supplemental technology to the palette of existing treatment methods, and another implementation option would be to use the technology in conjunction with other treatment methods [13]. Mavrogianopoulos et al. [23] used the MW for soil disinfection and disinfestation. Their experiments carried out in real scale showed that MW present numerous advantages, such as compactness of the equipment, rapid switching off and on, and pollution-free environment as there are no products of combustion, but energy demand of the technology was large. The author thought that a combination of solarization and microwaves was proposed as an energy efficient technique of using microwave for soil disinfestation. Thus, more work needs to be done to enhance the energy utilization to reduce the energy consumption and the economical cost.

5. Advantages and disadvantages of MW technique

5.1. Advantages of MW technique

Compared with the conventional steam-stripping method, the MW technique has the following advantages:

(1) The removal efficiency of ammonia nitrogen by MW radiation is higher than that of the steam-stripping method. MW heating is fast and the molecular-level heating leads to homogeneous and quick thermal reactions [24,25]. Besides, the particular non-thermal effect enhances the ammonia removal effectively [16]. Both effects result in the high removal of ammonia nitrogen, which could reach above 95% in lab-scale experiments and about 80% in pilot-scale experiments. Whereas, only 60% removal is reached by the steam-stripping method.
(2) In the steam-stripping method, since a large quantity of steam is added to the stripping tower, the effluent water volume increases by about 40%, which increases the consequent investment and treatment cost. In MW radiation, because of the high temperature in the reactor, partial wastewater volatilizes and ammonia wastewater is concentrated after MW treatment. Although the ammonia removal efficiencies are analyzed as 74–84% with aeration and a high ambient temperature, the real removal efficiencies would be higher. Because of the volatilization of the wastewater, the effluent water volume decreases slightly. This leads to a decrease of investment needed for the building of the facilities.
(3) Water quality after MW radiation is better than that after steam-stripping treatment. MW technique is a green chemistry, MW has a strong sterilization capability and it can effectively inactivate the bacteria and enzyme in wastewater [26,27].

5.2. Disadvantages of MW technique

Although MW technique has some advantages, there are still some disadvantages:

(1) MW radiation consumes electric energy and converts electric energy to heat. When it is used to treat wastewater, a large amount of energy is needed because the specific heat capacity of water is very high. This results in high power consumption. Thus, the application area of the technique is restricted. The optimal application field of this technique is the toxic industrial wastewater which contains high concentrations of ammonia nitrogen and is hard to be treated by conventional methods, such as wastewater from coke-plant, tannery, textile and landfill leachates.
(2) The temperature of the glass tube will always be very high under MW radiation, which leads to a short life of the glass tube. Thus, the glass tube in the MW cavity needs to be replaced regularly, which increases the running cost of the system.

6. Conclusions and perspectives

Continuous pilot-scale MW technique was found to be effective for the removal of high concentration ammonia wastewater from the Coke company. The removal of ammonia nitrogen would reach about 80%. Low ambient temperature and higher flow rate reduced the effectiveness of the MW reactor, and initial concentration and aeration had minute influence on the removal. The comparison between the MW technique and the steam-stripping method showed that the MW technique had many advantages. However, the economical advantage of the MW technology was not obvious compared with the conventional steam-stripping method.

Presumably, the optimal application field of this MW technique is the toxic industrial wastewater which contains high concentrations of ammonia nitrogen and is hard to be treated by conventional methods, such as wastewater from coke-plants, tanneries, textiles and landfill leachates. Current and future studies should focus on optimizing the system for maximum power utilization and energy efficiency by recovering part of the process heat through heat exchangers. Using a heat exchanger system would reduce the energy costs, and enhance the removal efficiencies [13]. Overall, the continuous microwave system has shown the potential as an effective method for ammonia nitrogen in coke-plant water treatment, but more work needs to be done to reduce the cost of this technology. Our research is therefore appropriate allowing some recommendations to be made, which will perhaps guide future research.

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