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Removal of ammonia nitrogen in wastewater by microwave radiation

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ABSTRACT

Ammonia nitrogen contaminated wastewater has posed great threat on the safety of water resources. In this study, a novel process, microwave (MW) radiation, was developed for the removal of ammonia nitrogen from wastewater. The effects of pH, radiation time, aeration and initial ammonia concentration on the removal were investigated. pH and radiation time showed significant influence on the removal of ammonia nitrogen. The largest removal was obtained at pH 11 in 3 min. Initial ammonia concentration and aeration had minute influence. The mechanism of ammonia removal was proposed as the formation of molecular ammonia (NH_3) and the subsequent evaporation of NH_3 by MW radiation. Compared with conventional heating (CH) mode, MW radiation led to higher ammonia removal. Both thermal and non-thermal effects were responsible for the removal, but the contribution of thermal effect was more significant. In the end, coke-plant wastewater containing high concentration of ammonia nitrogen was treated by MW radiation. With the initial ammonia concentration of 5000 mg/L at pH 11, ammonia was reduced to 350 mg/L at 750 W by 10 min MW radiation. It could be proposed that MW radiation was an effective method for the removal of ammonia nitrogen from wastewater.

1. Introduction

The rapid development of human activities has caused a worldwide increase of nitrogen (N) and phosphorus (P) in rivers, lakes and coastal waters. It leads to considerable eutrophication and an increase in the frequency of toxic algae blooms [1,2]. The concentrations of nitrogen and phosphate in the water of China are all-pervading high [3]. The discharge of wastewater containing high concentration of ammonia nitrogen is one of the most important factors. Great efforts have been devoted to the removal of ammonia nitrogen from wastewater. Traditional methods include biological denitrification [4], ammonia-stripping [5], chemical precipitation with magnesium ammonium phosphate [6], electrochemical conversion [7] and so on. Biological denitrification is the most common process in the treatment of ammonia nitrogen wastewater. But the process is only suitable for the removal of relatively low ammonia concentration due to the requirement of appropriate C/N ratio [8]. Ammonia stripping makes use of stripping tower and consumes much energy [7]. Chemical precipitation needs additional reagents, which may introduce new pollutants to water body. Electrochemical method often uses expensive metal or metal oxide as electrodes, and also consumes large quantity of energy [7]. As a consequence, it is necessary to develop cost-effective method for the removal of ammonia nitrogen from wastewater.

In recent years, microwave (MW) radiation has attracted a great deal of attention due to the molecular-level heating, which leads to homogeneous and quick thermal reactions [9]. MW radiation has been applied in the field such as organic and inorganic synthesis [10,11], polymerization processes [12], biological aspects [13] and extraction in analytical chemistry [14]. Researchers have attempted the use of MW radiation in environmental remediation. It has been applied to the removal of dyes in wastewater [15], and the remediation of soils contaminated with organics [16,17] and heavy metals [18]. Concerning the mechanism of MW radiation, it has been generally assumed that energy absorption involves only dissipation by heat and only thermal effects should be considered [11,19]. Nevertheless, some other researches claimed the existence of non-thermal effects [20–22]. A comprehensive review on thermal and non-thermal effects in MW radiation was reported by Eskicioglu et al. [23]. Despite the high number of publications, neither the chemical mechanism of MW interaction with materials, nor microbial destruction mechanism of MW in biological systems is fully understood [23]. There are many reasons for the conflicting results on thermal and non-thermal effects in MW field, such as the performance of MW and conventional heating (CH) with different techniques used, lack of details on heating apparatus, materials and methods especially related to temperature monitoring and control

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[24]. Therefore, many non-thermal experiments cannot be accepted by scholars.

According to our knowledge, there are no reports on the removal of ammonia nitrogen from wastewater by MW radiation. In this study, we use MW radiation to remove ammonia nitrogen from both simulated and real ammonia nitrogen wastewater. The objectives are: (1) to optimize the operating conditions for the removal of ammonia nitrogen by MW radiation; (2) to explore the removal mechanism of the ammonia nitrogen by MW radiation and (3) to treat real coke-plant wastewater containing high concentration of ammonia nitrogen.

2. Experimental

2.1. Chemicals

Ammonium chloride (99.5%, analytical reagent, Tianjin Kermel Chemical Reagent Development Center, China) was used as the source of ammonia nitrogen. Deionized water was used for the preparation of solutions. All other reagents were above analytical grade. Real ammonia nitrogen wastewater was obtained from the Coke Company of Wuhan Iron and Steel (Group) Corporation, China.

2.2. Procedures and equipments

The schematic diagram of the experimental apparatus is shown in Fig. 1. A modified domestic microwave oven (750 W, 2450 MHz, Haier Co., China) with different power setting was used as MW source. A 300-mL glass column reactor was placed in the oven. The reactor was filled with 100 mL of wastewater and radiated by MW under different conditions. The initial pH of the solution was adjusted by NaOH (2.5 mol/L). The aeration rate was maintained at 1 L/min by an air compressor. A thermometer was used for the measurement of temperature at the end of radiation. The top of the column was connected to a condensing system. The generated contaminant vapor passed through two bottles containing 100 mL of H_2SO_4 solution (0.5 mol/L). Each experiment was performed in duplicate.

As a comparison, an electric oven (1000 W, 50 Hz, Shanghai Yuejin Iatrical Apparatus Factory, China) was used to heat wastewater. A thermometer was also used for the measurement of temperature. The removal efficiencies of ammonia nitrogen were compared with those obtained by MW radiation.

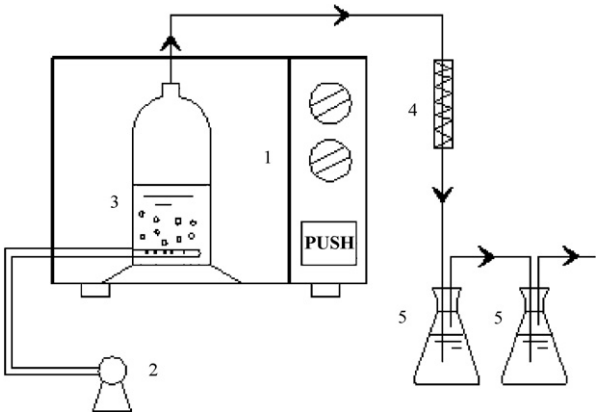

Fig. 1. Schematic diagram of the microwave reactor system. (1) MW oven, (2) air compressor, (3) glass reactor, (4) condenser, (5) absorption vessel.

2.3. Analysis

In the MW treatment process, the volume of wastewater slightly decreased due to evaporation of water. When the wastewater was cooled to room temperature at the end of experiment, deionized water was added into the reactor to keep the same initial volume of the wastewater. Ammonia nitrogen was measured by WT-1 portable apparatus of ammonia nitrogen (Wuhan Water Environmental Protection Company, China), which was based on the standard method [25]. pH was measured with a pH meter (pHS-25, Shanghai Leici Instrument Factory, China).

3. Results and discussion

3.1. Optimization of operation parameters

In order to achieve the maximal removal of ammonia nitrogen from wastewater by MW radiation, the operation conditions were first optimized. Four factors, including initial pH, MW radiation time, aeration and initial ammonia concentration were investigated.

3.1.1. Effect of pH

Fig. 2(a) illustrates the removal of ammonia nitrogen at different pH. The optimal pH was found to be 11, which resulted in 98% ammonia removal. There was always a pH dependent equilibrium between soluble ammonium ion NH_4^+ and dissolved molecular

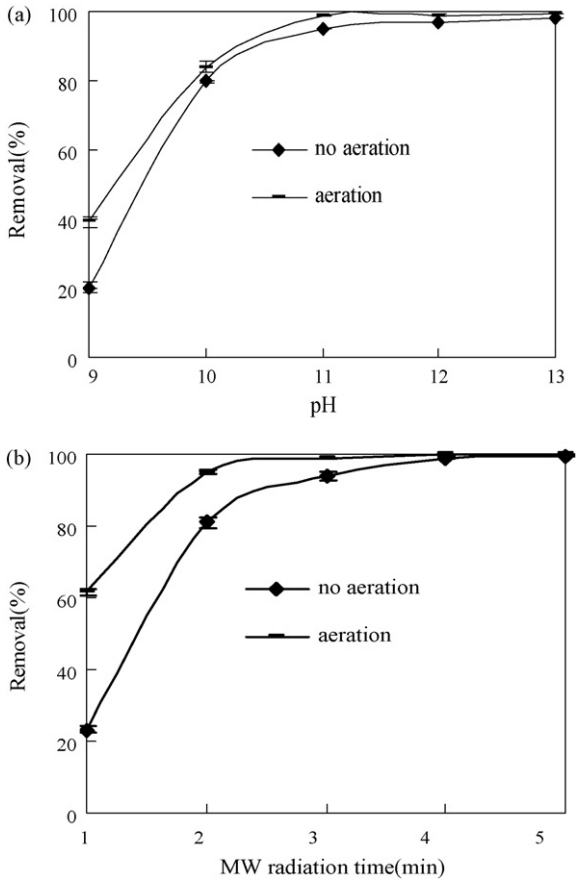

Fig. 2. Influence of pH (a) (500 mg/L ammonia nitrogen, 750 W MW power, 3 min radiation time) and MW radiation time (b) (500 mg/L ammonia nitrogen, 750 W MW power, pH 11) on the removal of ammonia nitrogen by MW with and without aeration.

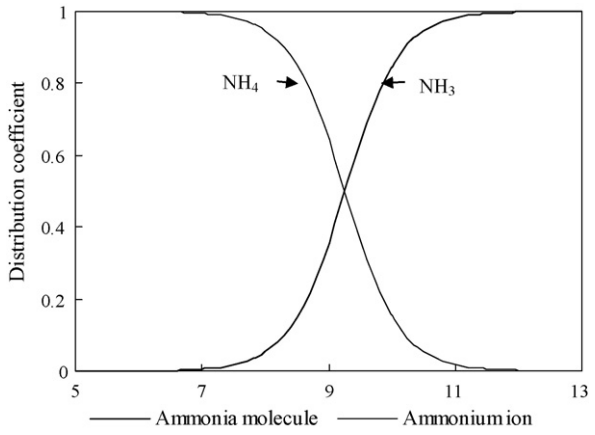

Fig. 3. Distribution coefficients of NH_3 and NH_4^+ at different pH (20 °C).

ammonia NH_3 in wastewater [26].

$$NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^- \quad (1)$$

$$\alpha_{NH_3} = \frac{[NH_3]}{[NH_3] + [NH_4^+]} \quad (2)$$

In reaction (2), α_{NH_3} was the distribution coefficient of NH_3. It was the fraction of NH_3 in total ammonia. $[NH_3]$ was the concentration of NH_3, and $[NH_4^+]$ was the concentration of NH_4^+ in water. Fig. 3 shows the distribution coefficients of NH_3 and NH_4^+ at different pH. It has been established that temperature has minute influence on the distribution coefficients [27]. In acidic and neutral media, ammonia nitrogen is presented as NH_4^+. In basic solution, non-volatile NH_4^+ is converted to volatile NH_3. High pH favors ammonia volatilization by driving the equilibrium between NH_3 and NH_4^+ to molecular ammonia.

Fig. 2(a) shows that the removal of ammonia reached 98.2% at pH 11 with aeration. From Fig. 3, it could be seen that 98.2% ammonia nitrogen was in the form of NH_3 at pH 11. The removal of ammonia was in good agreement with the proportion of molecular ammonia in the solution. It implied that all the volatile molecular ammonia was removed from the solution by MW radiation. At the end of radiation, pH of the solution decreased from 11 to about 10 due to the escape of NH_3. No significant increase of ammonia removal was observed when solution pH was further increased. When the wastewater was radiated by MW, the polar molecules in solution rotated fleetly (2450 million times/s), which resulted in rapid heating of the solution [28]. Consequently, the molecular movement in wastewater was greatly enhanced, which was highly advantageous for the evaporation of volatile molecular ammonia from liquid to gas. When the temperature of solution rose to boiling point, molecular ammonia could be stripped from the solution by the gas bubbles produced. The additional aeration was beneficial for the escape of volatile molecular ammonia. Besides, MW radiation might reduce the activation energy of reaction system and weaken the intensity of molecular chemical bond [29]. This effect was also advantageous for the removal of ammonia. This would be discussed in the later section.

Welander et al. [30] investigated the physical treatment of ammonia nitrogen by air stripping, and pH 10.5 was chosen to be the optimal pH to achieve high ammonia removal. Bonmati and Flotats [5] addressed that pH 11.5 was required for complete ammonia removal by air stripping regardless of the working temperature of 80 °C. In our experiments, considering the removal efficiency and the minimal dosage of alkali added, we chose pH 11 as the optimal value.

3.1.2. Effect of MW radiation time

Fig. 2(b) demonstrates that the removal of ammonia increased with radiation time and attained a plateau of 98.2% after 3 min. More heat could be generated with longer MW radiation time. Thus, the solution temperature became higher, which induced more impetuous and rapid molecular motion. This benefited the escape of ammonia nitrogen from solution. In addition, longer aeration time might contribute to the larger removal of volatile molecular ammonia. Since almost all the ammonia nitrogen in solution could be removed in 3 min MW radiation and aeration, and longer radiation time would result in an excessive cost, 3 min was considered to be the optimal radiation time. Liao et al. [31] investigated the removal of ammonia from wastewater by aeration and demonstrated that 130 h was needed to achieve 90% removal at pH 11.2.

3.1.3. Effect of aeration

The effect of aeration on the removal of ammonia by MW radiation is presented in Fig. 2. It could be seen that the removal was enhanced by aeration to some extent. Aeration brought a lot of air bubbles into the solution. This might result in turbulence and agitation [32]. Therefore, the mass transfer of ammonia nitrogen in the solution was enhanced, which benefited the volatile of molecular ammonia. Single aeration method was used by us to remove the ammonia nitrogen in wastewater, the aeration rate was 1 L/min, and aeration time was 3 min. It has been found that 10–11% ammonia nitrogen was removed from the solution. Kim et al. [7] reported that the ammonia molecule at pH 12 could be air-stripped from solution by fine gas bubbles produced during electrolysis. Because the electrolysis only produced small quantities of gas bubbles, only 9–11% ammonia was removed [7]. In the present study, large quantities of gas bubbles were produced by aeration, which attributed to the removal of molecular ammonia. But the aeration time was just 3 min. Therefore, the removal of ammonia was increased by only 10–11% when aeration was applied.

It had been found that higher pH and longer MW radiation time were favorable for the removal of ammonia. It could be seen from Fig. 2 that aeration showed no significant influence on the removal under the optimal conditions. Fig. 2(a) suggests that aeration significantly enhanced the removal of ammonia at pH below 10 and showed negligible effect at pH above 10. Similar result was also found by Liao et al. [31], who demonstrated that the increase of aeration rate led to minute increase of ammonia removal by aeration method at pH 11.5. The removal of ammonia was increased by aeration only when MW radiation time was less than 3 min (Fig. 2(b)). When MW radiation time exceeded 3 min, MW radiation without aeration was sufficient to remove almost all the ammonia in wastewater. It was indicated that the heating and molecular movement produced by MW radiation had great potential for ammonia removal.

3.1.4. Effect of initial ammonia concentration

Table 1 summarizes the removal of ammonia at different initial concentrations. It could be seen that ammonia could be largely removed by MW radiation, even at high initial concentrations. When the initial concentration increased from 500 mg/L to 12,000 mg/L, a slight decrease of removal efficiency was observed, but the removal efficiencies were still above 95%. Cheung et al. [32] concluded that the mass transfer of ammonia from liquid to air was proportional to its initial concentration. Therefore, the removal efficiencies were proportional to the initial concentration. When the initial concentration increased, the removal concentration of ammonia nitrogen from wastewater increased, but the removal efficiency was almost unchanged.

Summarily, trial results suggested a significant effect of pH and MW radiation time on ammonia nitrogen removal. The effect of the

Table 1
Removal of ammonia nitrogen at different initial concentrations

	Initial concentration (mg/L)				
	500	1270	2360	6000	12,000
Residue concentration (mg/L)	8	32.8	62.0	216.1	473.1
Removal (%)	98.4	97.4	97.3	96.4	96.1
S.D. (%)	0.54	0.14	0.08	0.29	0.46

Note: 750 W MW power, pH 11, 3 min radiation time and with aeration.

initial concentration and aeration on the removal was minute. The removal of ammonia nitrogen increased with the increase of pH and MW radiation time. The optimal condition was obtained as pH 11 and 3 min MW radiation.

3.2. Removal mechanism of ammonia nitrogen by MW radiation

3.2.1. Contribution of thermal and non-thermal effects

In general, thermal and non-thermal effects are responsible for the enhancement by MW radiation. Thermal effect is related to the heat generated by the absorption of microwave energy by water and other polar molecules, both characterized by a permanent or induced polarization [22]. Whereas, non-thermal effect is claimed to change the chemical, biochemical, or physical behaviors of systems while temperature and other parameters remain unaltered. Extensive researches had been conducted to investigate the non-thermal effect associated with MW radiation [11,19–22]. Non-thermal effect was revealed to enhance the crystallization rate of SnO under MW radiation by Wu et al. [21] and inactivation of two thermophilic and thermostable enzymes by Porcelli et al. [22]. However, Zhang et al. [11] and Shazman et al. [19] reported that no non-thermal effects could be found in their works. Critics of the non-thermal effect often claimed that differences of the effect could be attributed to poor temperature measurement and control of experimental conditions that resulted in systematic error. The existence or not of non-thermal effects continued to be an area of considerable debate and research.

The relationship between temperature and ammonia removal is shown in Fig. 4. The ammonia removal was minute at low temperature and increased sharply at temperature above 80 °C, particularly when the wastewater was boiling. It could be speculated that the ammonia was removed mostly at high temperature. There was a fundamental difference between MW radiation and CH process. In CH process, energy was transferred from the surface to material inside via convection, conduction and radiation [28]. However, heat was generated simultaneously in the whole container under MW

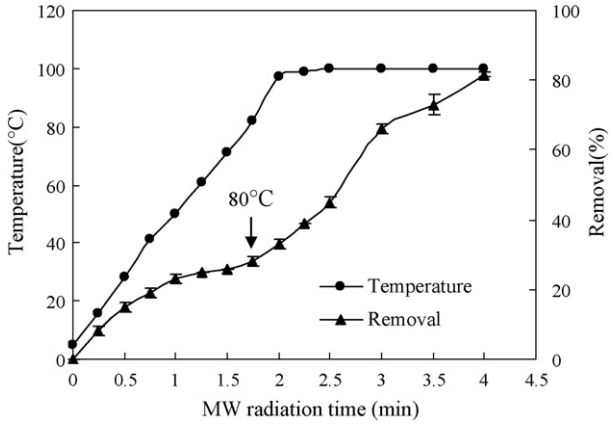

Fig. 4. Relationship between temperature and ammonia removal (500 mg/L ammonia nitrogen, 350 W MW power, pH 11 and no aeration).

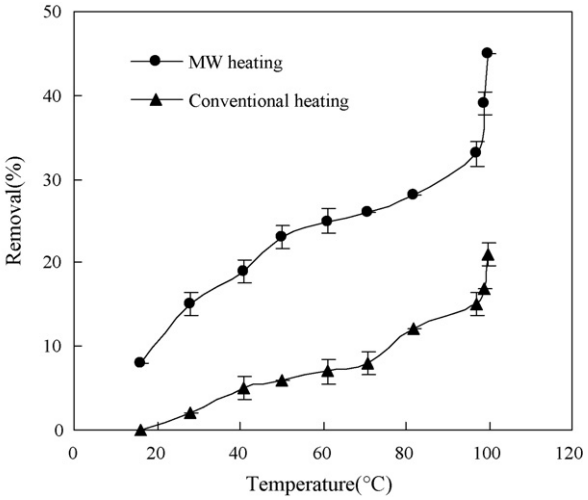

Fig. 5. Comparison of the ammonia removal by MW heating (350 W) and CH (1000 W) (500 mg/L ammonia nitrogen, pH 11, and no aeration).

radiation [28]. The mechanism of heat generation was a partial dissipation of the electromagnetic field energy and its conversion into heat. The alternating electromagnetic field induced the rotation of the dipoles of polar substances, such as H_2O. The intermolecular friction resulted in the generation of substantial amount of heat [33], and then the solution temperature in the container rapidly rose to a high level. This was the thermal effect of MW radiation. Longer MW radiation time induced higher temperature, which subsequently induced more impetuous molecule motion and faster mass transfer and benefited the elimination of ammonia nitrogen. Since the ammonia was removed mostly at high temperature in our study, it could be deduced that thermal effect played a key role on ammonia removal.

In order to confirm whether non-thermal effect was attributable to ammonia removal, an electric cooker was used to heat the wastewater. The wastewater was heated from the same initial temperature to the same final temperature by MW radiation and CH. As shown in Fig. 5, MW radiation demonstrated larger removal of ammonia nitrogen compared with CH. MW energy induced molecular motion by rotation of dipoles and migration of ions [34]. Heating by MW radiation depended on the dipole relaxation time and ionic conductivity [35]. This nonionizing electromagnetic radiation was absorbed at molecular level and manifested as changes

Table 2
Mass of ammonia nitrogen after the MW treatment

	pH				
	9	10	11	12	13
Ammonia nitrogen in wastewater (mg)	30.40	10.03	0.61	0.48	0.38
Ammonia nitrogen in H_2SO_4 (mg)	22.88	41.58	49.10	48.44	48.64
Sum (mg)	53.28	51.61	49.71	48.92	49.02

Note: 500 mg/L initial ammonia nitrogen in 100 mL solution, 750 W MW power, 3 min radiation time and with aeration.

Table 3
Characteristics of the influent and effluent wastewaters

Characteristics	COD (mg/L)	NH_3 (mg/L)	Volatile phenol (mg/L)	Cyanide (mg/L)	Sulfide (mg/L)
Influent wastewater	7860	5000	1710	56.2	459
Effluent wastewater	7050	1350	651	10.6	34.8

in vibrational energy of the molecules. Ammonia nitrogen wastewater consisted of a large amount of molecular H_2O and NH_3. And the N–H···O and O–H···N intermolecular hydrogen bond existed in the wastewater [36,37]. Molecular H_2O and NH_3 were both polar molecules and could be polarized by MW radiation. MW radiation caused dipoles to rotate and line up rapidly (2450 million times/s). Therefore, the frequent pendulum vibration of molecular H_2O and NH_3 led to the fracture and weakening of the intermolecular hydrogen bond between NH_3 and H_2O, which was beneficial for the escape of molecular ammonia from liquid phase to gas phase. It was clear that thermal effect could not lead to the molecular rotation or vibration. As a result, it could be concluded that thermal effect played a key role on the removal of ammonia by MW radiation, and non-thermal effect enhanced the removal to some extent.

3.2.2. Mass balance of nitrogen

The mass balance of nitrogen in the MW radiation process is shown in Table 2. The initial mass of ammonia nitrogen in the solution was 50 mg. The ammonia removed was completely collected in the bottles filled with H_2SO_4 solution. And the concentrations of nitrate and nitrite ions were also determined. There were no increase of both nitrate and nitrite ions in the MW process. This meant that ammonia nitrogen did not convert into other nitrogen form. It can be concluded that the ammonia nitrogen in wastewater was removed by volatilization from the liquid to gas. The mechanism was believed to be similar to that of ammonia-stripping method [5], which converted NH_4^+ into volatile NH_3 in basic solution and then stripped the molecule from liquid to gas. The difference was that the removal of molecular ammonia by MW radiation was via thermal and non-thermal effects generated by the radiation.

3.3. Treatment of coke-plant wastewater

High concentrations of organics and ammonia nitrogen are commonly present in industrial wastewater such as coke-plant, tannery, textile, landfill leachate and fertilizer wastewater [38]. The real coke-plant wastewaters used in this study were obtained from a conventional steam-stripping tower of Coke Company in Wuhan Iron and Steel (Group) Corporation, China. In the steam-stripping tower, the raw wastewater was stripped of ammonia partially at 80–90 °C and at a pH of 11–12 to reduce ammonia but chemical oxygen demand (COD) remained unchanged. Two waste streams – influent and effluent of the steam-stripping tower – were sampled as the real coke-plant wastewaters. The wastewaters contained toxic organic pollutants, such as phenol, cyanide and sulfide, and also contained a high concentration of ammonia nitrogen. The characteristics of the influent and effluent wastewaters were shown in Table 3.

Treatment of these coke-plant wastewaters were very difficult, mainly due to the presence of high ammonia concentration, and even the conventional steam-stripping tower could not remove the ammonia nitrogen effectively. MW radiation provided an alternative technique; it showed encouraging performance on the removal of ammonia from the wastewater. At pH 11, 5 min MW radiation reduced the effluent ammonia concentration from 1350 mg/L to 50 mg/L, and 10 min radiation reduced the influent ammonia from 5000 mg/L to 350 mg/L, when the MW power was 750 W, the volume of the wastewater was 100 mL. Therefore, MW radiation was preliminarily feasible for the removal of ammonia from real wastewater, even with the simultaneous existence of high concentration of organics. With the rapid development of MW technology, MW power can achieve as high as hundreds of kilowatt, and the waveguide can be designed flexibly. It is possible to provide the target sufficient heat in a very short time by MW radiation. Therefore, the engineering application of MW radiation to remove ammonia nitrogen from wastewater is practical. However, much work is still needed prior to the practical application.

4. Conclusions

A fundamental research had been carried out to explore the removal of ammonia nitrogen in wastewater by MW radiation. The influence of operating parameters and the mechanism of ammonia removal were investigated. Conclusions were drawn as follows.

(1) MW radiation had been proved to be an effective technique for the removal of ammonia nitrogen in wastewater. Large removal of ammonia nitrogen was achieved by MW radiation in a short time. pH and MW radiation time showed heavy influence on ammonia removal, while aeration and initial ammonia concentration presented minute effect. Higher pH and longer MW radiation time resulted in larger removal efficiencies.
(2) The mechanism of ammonia removal by MW radiation was the volatilization of the molecular ammonia (NH_3), which was produced from ammonia ion (NH_4^+) at high pH. Both thermal and non-thermal effects contributed to the removal of ammonia. Thermal effects played a key role on the removal, and non-thermal effect enhanced the removal to some extent.
(3) MW radiation can be considered as an alternative approach for the removal of high concentration of ammonia nitrogen from practical coke-plant wastewater. 10 min MW radiation could reduce ammonia concentration from 5000 mg/L to 350 mg/L at pH 11, when the MW power was 750 W, the volume of the wastewater was 100 mL.

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