

# Microwave assisted catalytic wet air oxidation of H-acid in aqueous solution under the atmospheric pressure using activated carbon as catalyst

Yaobin Zhang<sup>a,b</sup>, Xie Quan<sup>a,\*</sup>, Shuo Chen<sup>a</sup>, Yazhi Zhao<sup>a</sup>, Fenglin Yang<sup>a</sup>

<sup>a</sup> School of Environmental and Biological Science and Technology, Dalian University of Technology, Dalian 116024, China

<sup>b</sup> Department of Environmental Science and Technology, Dalian Railway University, Dalian 116028, China

Received 19 August 2005; received in revised form 7 December 2005; accepted 20 February 2006

Available online 5 April 2006

## Abstract

Catalytic wet air oxidation (CWAO) is a promising method for the treatment of heavily contaminated wastewater. However, its application is restricted due to severe operation conditions (high pressure and high temperature). A microwave (MW) assisted oxidation method was investigated aiming to treat heavily contaminated wastewater under milder conditions. H-acid (1-amino-8-naphthol-3, 6-disulfonic acid) was selected as target compound to evaluate the performance of this novel process. The removal of H-acid and TOC (total organic carbon) for H-acid solution of 3000 mg/L reached as high as 92.6% in 20 min and 84.2% in 60 min, respectively under optimal conditions. The existence of activated carbon and oxygen proved to be critical for effective treatment. The activated carbon acted not only as a catalyst for H-acid decomposition, but also as a special material for the absorption of MW energy. Air was supplied to the reactor as an oxygen source at constant flows. The amino group in H-acid was converted ultimately into nitrate, and sulfonic group into sulfate. This observation gave an evidence of H-acid mineralization although other organic intermediates were unable to be determined. The value of BOD<sub>5</sub>/COD (ratio of 5d biochemical oxygen demand to chemical oxygen demand) increased from 0.008 to 0.467 indicating a significant improvement of biodegradability for the solution, which is beneficial for the further biological treatment of the wastewater.

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**Keywords:** Microwave; H-acid; Activated carbon; Wastewater treatment

## 1. Introduction

H-acid (1-amino-8-naphthol-3, 6-disulfonic acid) is an important intermediate for dye manufacturing, and was widely used in the syntheses of direct, acidic, and reactive dyes [1]. In addition, H-acid has been found its application in pharmaceutical industry. Common concentrations of H-acid in the industrial effluents range from 1000 to 10000 mg/L because of its good solubility in water. Another characteristic of H-acid is its resistance to biodegradation. Normal processes of wastewater treatment based on the physical, chemical, or microbiological methods are not effective for the treatment of wastewater containing H-acid [2].

Catalytic wet air oxidation (CWAO) is a promising technology to degrade non-biodegradable organic substances in industrial effluents even to the degree of mineralization. The advantages of CWAO are its ecological safety and the capability to improve biodegradability of wastewaters. Therefore, it is an alternative method to treat industrial effluents containing H-acid [3]. However, disadvantages of CWAO are apparent either. Severe operation conditions of high temperature (180–315 °C), high pressure (2–25 MPa), and demand of noble metallic catalysts [4,5], have restricted its wide application in the developing countries or for small enterprises. It is of significance to innovate the technology upon CWAO aiming at effective treatment of wastewater under milder conditions (lower temperature, lower pressure and using cheaper catalysts).

In recent decades, microwave (MW) irradiation has attracted the attention of chemists due to its characteristic of molecular level heating which leads to quick thermal reactions [6]. For many catalytic reaction systems, reaction rates were accelerated

\* Corresponding author. Tel.: +86 411 84706140; fax: +86 411 84706263.  
E-mail address: quanxie@dlut.edu.cn (X. Quan).

and product selectivity could be modified when MW irradiation was used rather than conventional heating [7–9]. Temperature profile measurements on the surface of catalyst under MW heating exhibited hot zones where catalytic reactions could be enhanced [10].

Recently, the application of MW technique has been reported in the field of environmental treatment, such as, remediation of soil [11], purification of air [12] and regeneration of activate carbon (AC) [13]. A few of studies performed under MW irradiation were concerned with wastewater treatment. It was reported that MW irradiation was applied in the CWAO process where the low value of phenol conversion was obtained, which was interpreted to the formation of a high-energy plasma which is believed to be a Maxwell–Wagner effect [14]. In addition, some researches showed that an integrated MW and UV-illumination method resulted in a higher efficiency of degradation of pollutants [15–17].

This paper described a MW assisted method for the removal of non-biodegradable organic substances in aqueous solution. The characteristics of the method including critical factors and pathway for the formation of course products were discussed. H-acid was employed as target substance, and AC was used as catalyst.

## 2. Experimental

### 2.1. Materials

Commercial AC (analytical grade, 872 m<sup>2</sup>/g of BET area, 4% of ash content) with a particle size of 1.0–2.0 mm was purchased from Tianjin Chemical Reagent Co., China, and was pretreated with following procedure: immersed in a 10% HCl solution for 4 h, washed with distilled water, and dried at 105 °C for 2 h.

H-acid (C.P.) was purchased from Shanghai Chemical Reagent Co., China.

### 2.2. Description of reactor system

A MW oven (Whirlpool T120, 1000 W, Huizhou Whirlpool Co., China) modified was used as MW source. A  $\phi$  50 mm hole was drilled on its cover, and a copper pipe was inserted to prevent MW emission [18]. The quartz column reactor ( $\phi$  32 mm  $\times$  350 mm) was installed into MW furnace through the hole of the copper pipe, and the top of the column was connected to a vertical water-cooling system. Two bottles, charged with 10 mL distilled water and 10 mL, 0.1 mol/L NaOH solution, respectively, were used to absorb and purify the off-gas after condensation. Air compressor was used to supply air. All experiments were carried out under the atmospheric pressure. The experimental setup is shown in Fig. 1.

### 2.3. Experimental methods

In order to reduce the effect of H-acid adsorption on AC during the experiment course insulating in the decrease of its concentration, the AC used in the reaction should be pre-saturated with H-acid prior to MW treatment. Thus, it is believed that the

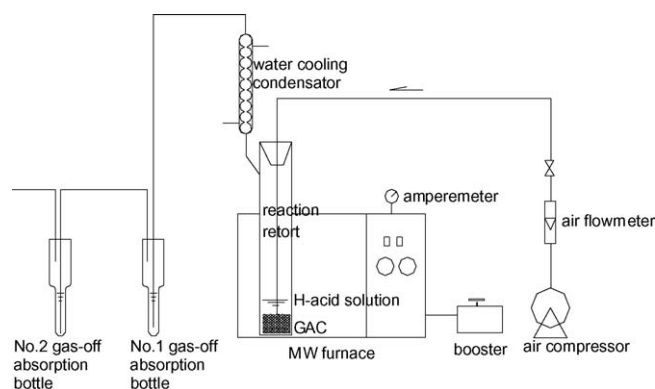


Fig. 1. Experimental setup.

decrease of H-acid concentration during the irradiation experiment resulted from degradation rather than from adsorption.

Before preparation of the saturated AC, it is necessary to know equilibrium adsorption amount of H-acid on AC. Based on our previous work [19], adsorption isotherm of H-acid on AC at the room temperature fitted to the following curve:  $q = 3.14C^{1/2.34}$  (where  $q$  is equilibrium adsorption amounts, and  $C$  is equilibrium concentration). Thus, when  $C$  was 3000 mg/L,  $q$  was 96.1 mg H-acid/g AC.

The AC was immersed into a given amount of H-acid solution (according to the equilibrium amount) to get adsorption equilibrium with 3000 mg/L H-acid in solution, which was the concentration of test solution. The AC saturated by H-acid was then taken out, and dried in the air for 24 h. Afterwards, 10 g of the AC saturated and another 20 mL of 3000 mg/L H-acid solution were added into the reaction retort. MW power was switched on, and air was supplied to the retort at a given flow.

### 2.4. Analysis

The solution and AC were taken out from the retort after irradiation, and were centrifuged (to cool down) for 15 min at 5000 rpm. The solution was analyzed directly using high performance liquid chromatograph (HPLC) and TOC analyzer. AC was dried in the air for 24 h, and then extracted with 25 mL of 0.1 mol/L NaOH solution (shaking at 250 rpm for 2 h and ultrasonic extraction for 30 min). The extraction procedure was repeated three times. Afterwards, all extracts were combined and diluted to 100 mL for TOC analysis.

The liquids in the adsorption bottles were analyzed for evaluation of mass loss through evaporation.

The concentration of H-acid was determined by HPLC (Jasco, PU1575/UV1580, Japan) with ODS column (5  $\mu$ m, 4.6 mm  $\times$  250 mm). Detection wavelength was set at 234 nm, and mobile phase was 1.0 mL/min of 0.05 mol/L Na<sub>2</sub>SO<sub>4</sub>:methanol=98:2. Total organic carbon (TOC) was determined by TOC analyzer (Shimadzu, TOC-V<sub>CPH</sub>, Japan). NH<sub>4</sub><sup>+</sup>, NO<sub>2</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, COD and BOD<sub>5</sub> were measured in accordance with the National Standard Methods (China, 1982). Ultraviolet (UV) spectrum was recorded by UV-vis spectrophotometer (JASCO, V550, Japan).

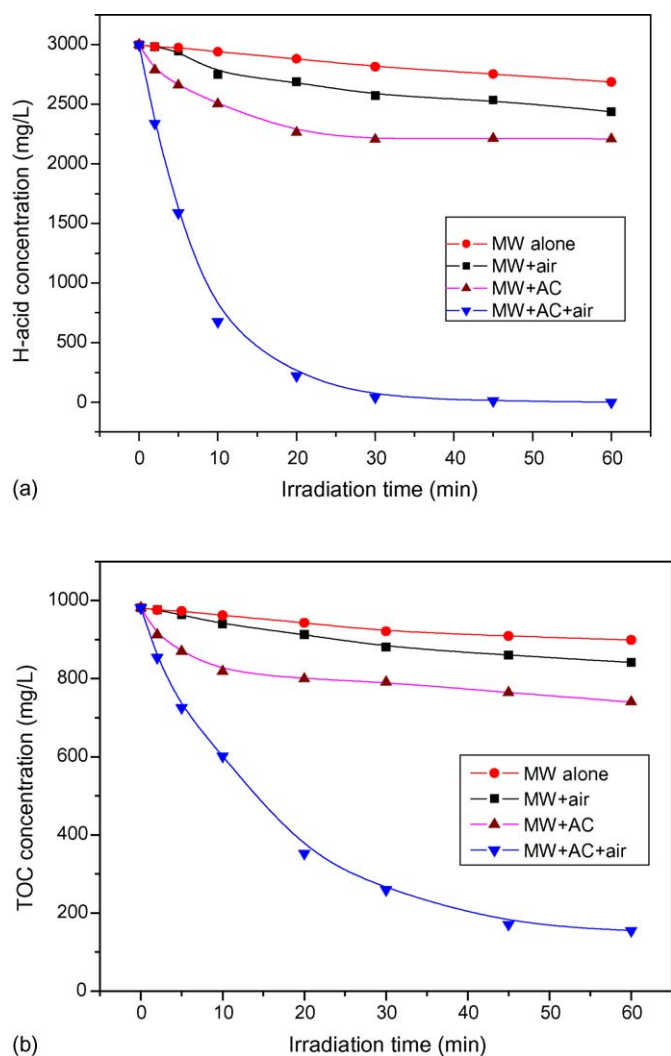


Fig. 2. Decay of H-acid concentration (a) and TOC (b) in the solution during MW irradiation under different conditions.

The temperature variation in the reactor during MW irradiation was recorded with a sheltered type-K thermocouple probe.

### 3. Results and discussion

#### 3.1. Efficiency of MW assisted process on H-acid degradation

In order to identify the critical factors that influence the degradation of H-acid, experiments were carried out respectively

Table 1  
TOC mass distribution in the aqueous phases

Condition	Initial amount of TOC (mg)	TOC after treatment (mg)				TOC amount removal (%)
		In solution	In first absorption bottle	In second absorption bottle	Sum	
MW	19.6	18.0	1.0	0.3	19.3	1.5
MW + air	19.6	16.8	1.8	0.5	19.1	2.6
MW + AC	19.6	14.8	1.9	0.6	17.3	11.7
MW + AC + air	19.6	3.1	0.5	0.1	3.7	81.1

under the conditions of (1) 800 W MW, (2) 800 W MW + air flow (0.5 L/min), (3) 800 W MW + 10 g AC, and (4) 800 W MW + 10 g AC + air flow (0.5 L/min). Fig. 2 displays the variations of the H-acid concentration and TOC in the solution treated with reaction time under the four conditions. The results show that the concentration of H-acid decreased significantly in the initial 20 min under the MW irradiation in the presence of AC and air flow, and the removal of H-acid reached 92.6%, however, this value only dropped a little during 20–60 min. Accordingly, TOC decreased steadily at the same time (Fig. 2(b)), reaching to 84.2% in 60 min. With the MW and AC, only 26.4% removal of H-acid and 24.6% removal of TOC were observed in 60 min. This might be partially due to the insufficiency of oxygen in the retort. The oxygen dissolved in the solution (8.2 mg/L) and the air resided in the head space might participate in the reaction. For MW alone or MW with air flow, the concentrations of H-acid and TOC were decreased slightly. A TOC mass distribution in the aqueous phases was determined to get more information about mass distribution, and the results are listed in Table 1. As seen in the table, under the condition (1), (2), (3), and (4), 6.6% (1.3 mg), 11.7% (2.3 mg), 12.7% (2.5 mg), and 3.1% (0.6 mg) among 19.6 mg of total TOC were found in the absorption bottles. It indicated that loss in the concentration and TOC for the condition (1) (MW alone) and (2) (MW + air flow) was mainly from evaporation of H-acid.

In the presence of AC and air, the changes of adsorption amount of TOC on AC along with irradiation time are displayed in Table 2. It steadily decreased from 32.1 to 30.3 mg TOC/g AC in 60 min, which more indicated that decrease of TOC in the solution resulted from degradation but not from adsorption.

It could be concluded from the above results that the MW assisted method had a good capability for degradation of H-acid (even to the degree of mineralization) in an aqueous solution, and the simultaneous existence of AC and air supply was critical for this process.

#### 3.2. Functions and characterization of AC

Major functions of AC in the process are defined as two aspects. Firstly, it is a material for absorbing MW energy and

Table 2  
Changes of adsorption amount of TOC on AC during the experiment course (mg TOC/g AC)

Irradiation time (min)	0	20	40	60
Adsorption amount (mg TOC/g AC)	32.1	31.5	30.9	30.3

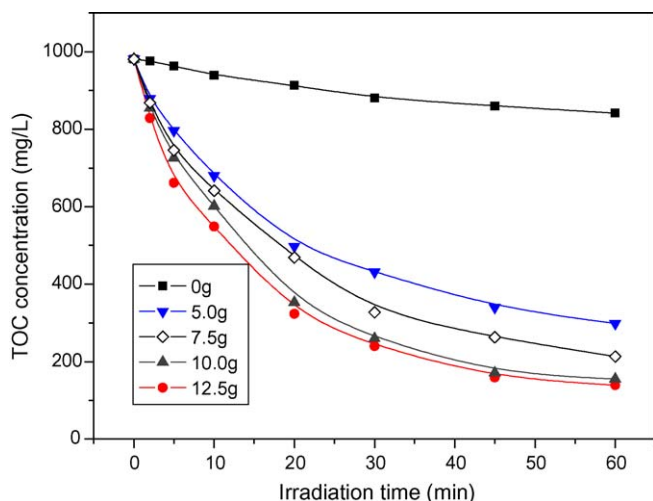


Fig. 3. Effect of AC dose on TOC concentration in H-acid solution.

converting it into heat. Our previous work showed that the rate of temperature increase for AC was 10 times as high as that for water under the same MW irradiation. There should be a temperature gradient from AC to water phase when water and AC exist together. Locally superheating within AC micro-surfaces might benefit the initiation of chemical reactions [20]. Secondly, AC can serve as catalyst. Fortuny et al. [21] and Aguilar et al. [22] reported the catalytic effect of AC in CWAO. Its catalytic activity was found to be eight times higher than a commercial copper oxide catalyst in CWAO for phenol degradation. As shown in Fig. 2 and Table 1, H-acid could not be decomposed without AC. Therefore, the presence of AC was one of critical factors for this MW assisted process.

Fig. 3 illustrates the effect of AC dose on the removal of TOC in the solution treated. It indicated that TOC removal increased with increasing AC dose. There was obvious improvement in the TOC removals when the AC dose increased from 5.0 to 7.5 g or from 7.5 to 10.0 g. However, the difference of TOC removals between 10 and 12.5 g AC dose was negligible. It implied that there was an economically optimal demand for AC dose. Temperature of solution bulk was recorded simultaneously and is shown in Fig. 4. The temperature rose steadily up to 102–103 °C with irradiation time elapsed for all cases of different AC doses, and maintained the temperature afterwards. It can be seen that

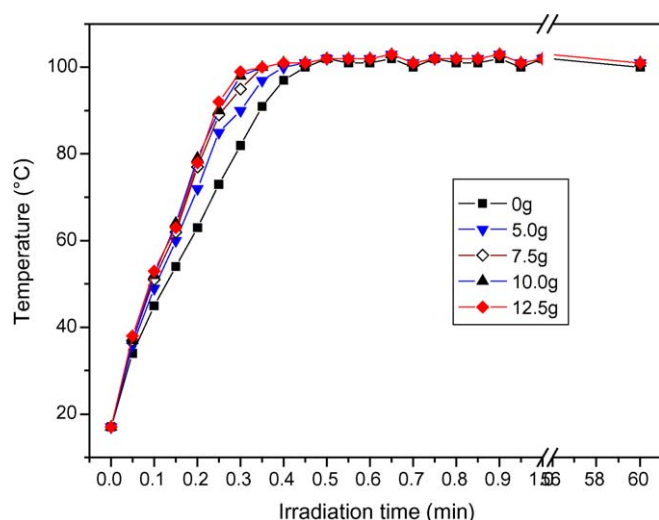


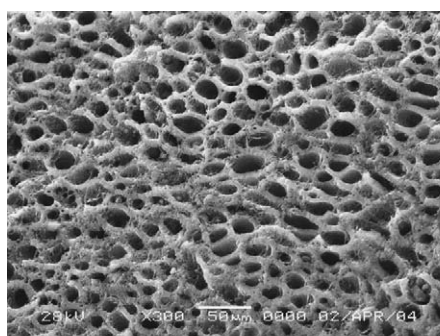
Fig. 4. Temperature records for mixed phase of AC and water (20 mL) at different AC doses.

the larger AC dose was, the more rapidly temperature rose, but when AC dose ranging from 10.0 to 12.5 g, the temperature rising courses were almost similar to the case of TOC removal. It meant that increase of AC dose had less contribution to the rise of temperature when excessive amount of AC was applied.

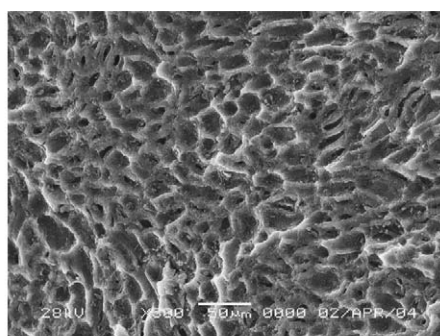
Surface morphology of AC was investigated using scanning electron micrographs (SEM, Fig. 5) to compare the AC before and after MW treatment of H-acid under the reaction conditions. The surface morphology of the AC after MW treatment seemed a little bit changed with more pores with smaller size. BET measurements indicated that after MW treatment the surface area and the total pore volume increased slightly from 872 to 931 m<sup>2</sup>/g and from 0.53 to 0.58 cm<sup>3</sup>/g, respectively (Fig. 6).

### 3.3. Other key factors influencing the process

Air supply – similar to the case in conventional CWAO, the MW assisted process was highly dependent on oxygen amount because oxygen supplies electron acceptor to the oxidative reaction. Fig. 7 shows that TOC decayed along with MW irradiation time. Air was supplied under the atmospheric pressure. It is reasonable to note that TOC removal increased as air flow rose in the initial course (about 0–40 min). However, in the end of the



(a) before MW treatment



(b) after MW treatment

Fig. 5. SEM for AC before (a) and after (b) MW treatment of H-acid under the reaction conditions.

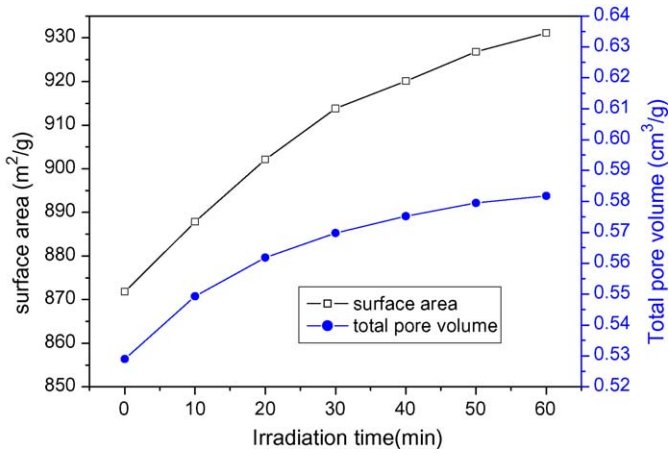


Fig. 6. Variation of the surface properties of AC after MW irradiation of H-acid under the reaction conditions.

course, TOC residues reached to similar values. The TOC decay for the case without air supply was supposed to partially result from decomposition of H-acid because of the limited existence of oxygen in the reaction retort, and partially due to evaporation, as discussed in Section 3.1.

MW power – MW is an energy composed of electric field and magnetic field. It is a form of electromagnetic waves with the wavelengths of 1 mm–1 m (frequency 300 MHz–300 GHz), corresponding to energy from 10<sup>-6</sup> to 10<sup>-3</sup> eV [23]. This energy is insufficient to cleave chemical bonds directly. However, the interaction of MW electric field with polar materials leads to internal heating of the polar materials, which is quite different from conventional heating, where materials are heated through heat conduction and dispersion from external energy sources. Hence, selective heating, which could act only to the desired points, is feasible by means of MW irradiation [24]. Increase of MW power might enhance the selective heating. The variation of temperature of the reaction phase under different MW powers was recorded (Fig. 8). It is clear that the temperature rose at different rates under different MW powers, but ultimately reached to the same value of 102–103 °C (the boiling point).

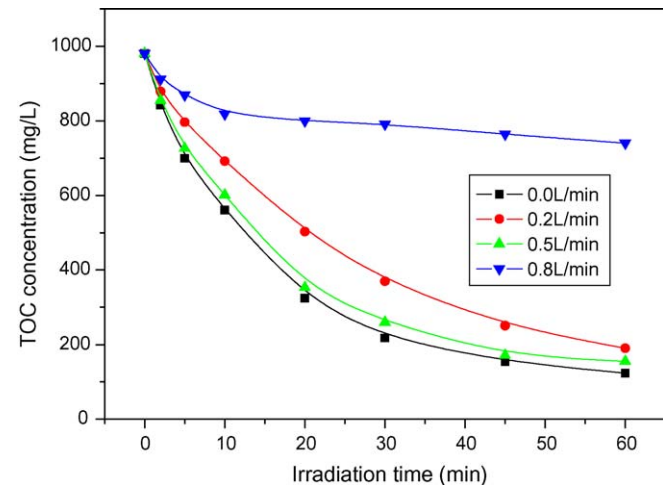


Fig. 7. Effect of air flow on TOC concentration in H-acid solution.

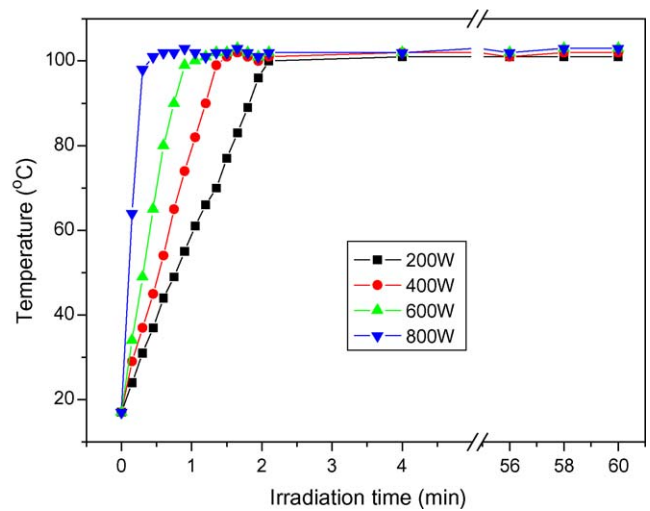


Fig. 8. Temperature records for mixed phase of AC (10 g) and water (20 mL) at different MW powers.

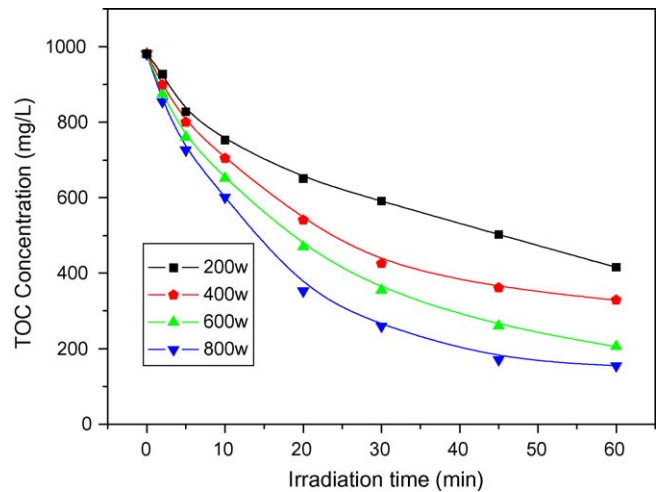
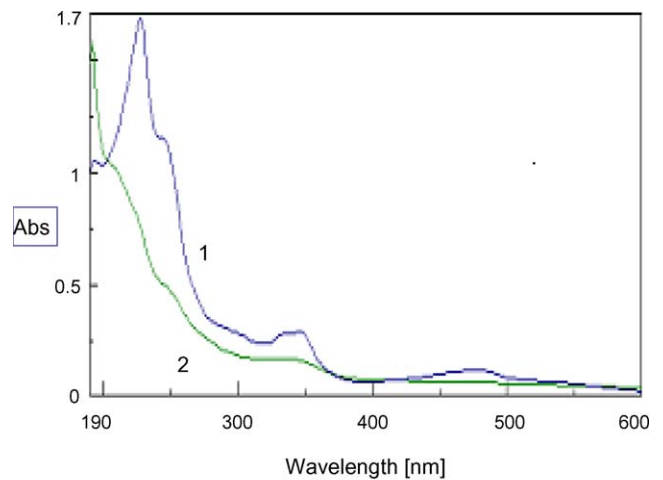


Fig. 9. Effect of MW power on TOC concentration in H-acid solution.



1. before treatment 2. after treatment

Fig. 10. UV Absorption spectra of H-acid solution before and after 30 min treatment.

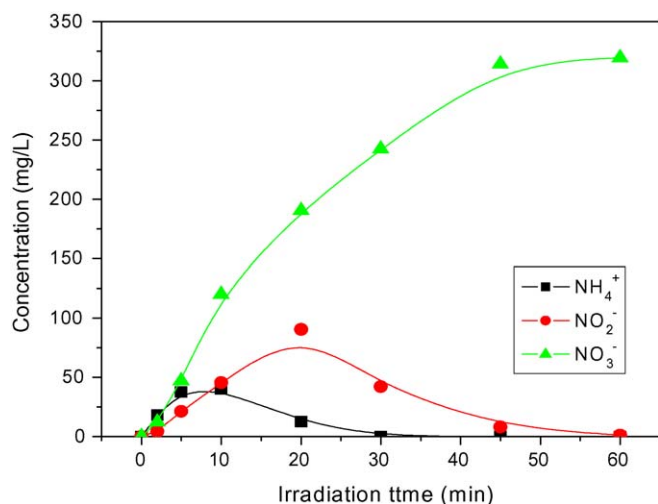
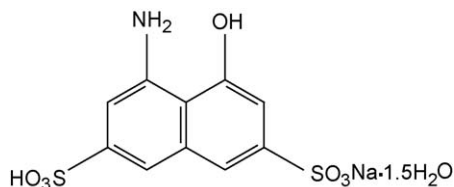


Fig. 11. Variation of  $\text{NH}_4^+$ ,  $\text{NO}_2^-$  and  $\text{NO}_3^-$  concentrations in the H-acid solution in the course of MW treatment.

According to the heat equation ( $Q = Cm\Delta t$ , where  $Q$ ,  $C$ ,  $m$  and  $\Delta t$  represents energy absorbed, specific heat, weight and raising temperature, respectively), when power increased from 200 to 800 W, the efficiencies of absorbed energy increased from 21.5% to 35.9%. Correspondingly, the degradation of H-acid was enhanced as the MW power increased. TOC removals in the solution increased from 57.6% to 84.2% in 60 min irradiation time when MW power ranged from 200 to 800 W (Fig. 9).

#### 3.4. Characterization of course products

According to the molecular structure of H-acid below, the course products after the MW reaction could be characterized by several ways.



The MW experiment conditions were 10 g of AC dose, 0.5 L/min of airflow, 20 mL 3000 mg/L of H-acid and 800 W of MW power. The H-acid solution after treatment was determined and the results are discussed as follows.

The UV absorption spectrum for H-acid solution before and after 30 min treatment is compared as shown in Fig. 10. Naphthalene structure in H-acid is capable of strong absorption of UV in the range of 210–260 nm as shown in the figure (spectrum 1). After 30 min of MW treatment, the absorption was apparently decayed. It indicated that the naphthalene ring was possibly destroyed.

Amino group in H-acid could be converted into inorganic species such as  $\text{NH}_4^+$ ,  $\text{NO}_2^-$  and  $\text{NO}_3^-$  if H-acid was mineralized. In order to confirm this suggestion, the concentrations of  $\text{NH}_4^+$ ,  $\text{NO}_2^-$  and  $\text{NO}_3^-$  were determined during MW irradiation and the results are illustrated in Fig. 11. The concentrations

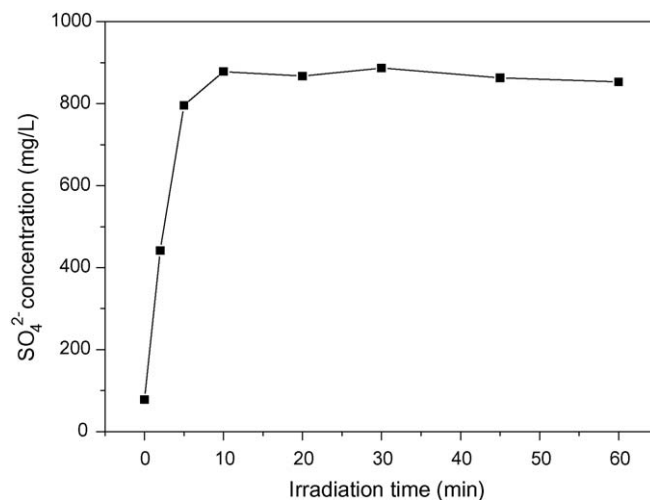


Fig. 12. Variation of sulfate concentration in the H-acid solution in the course of MW treatment.

of  $\text{NH}_4^+$  and  $\text{NO}_2^-$  increased with treatment time in the first stage, and reached their highest concentrations at 8 and 20 min, respectively, and then decayed gradually until the levels was undetectable. The concentration of  $\text{NO}_3^-$  increased steadily up to 314.1 mg/L (70.9 mg/L in nitrogen) in 45 min, and kept constant thereafter. Because the total nitrogen amount in original solution was 114 mg/L (corresponding to 3000 mg/L H-acid), about 62.2% nitrification of amino group was estimated.

Fig. 12 illustrates the changes of  $\text{SO}_4^{2-}$  in the solution. Sulfonic groups in H-acid were separated from naphthalene rings to convert into  $\text{SO}_4^{2-}$ . The concentration of  $\text{SO}_4^{2-}$  increased rapidly in the initial 10 min, approaching the highest level of 878 mg/L, which stood for 66.7% of total sulfur in the H-acid. After 10 min, the concentration of  $\text{SO}_4^{2-}$  remained almost constant until the end of experiment.

Other organic course products were unable to be identified with GC-MS and LC-MS. However, the decay of TOC and the variation of nitrogen and sulfur elements in the solution indicated the mineralization of H-acid during MW treatment.

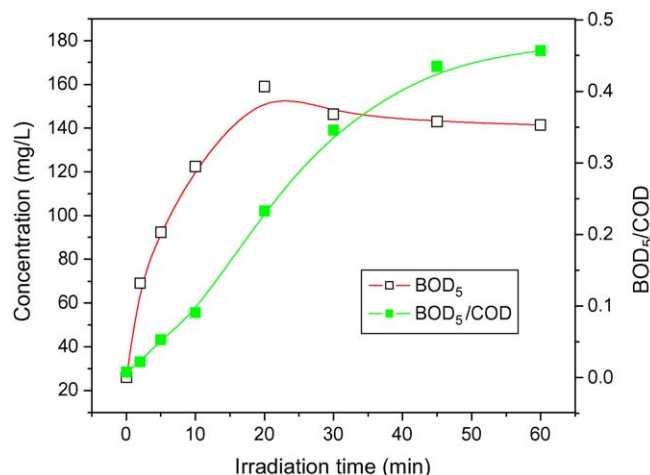


Fig. 13. Variation of  $\text{BOD}_5$  and  $\text{BOD}_5/\text{COD}$  in the H-acid solution in the course of MW treatment.

Biodegradability of the solutions was evaluated by the values of BOD<sub>5</sub>/COD (Fig. 13). Before treatment, BOD<sub>5</sub>/COD of initial H-acid solution was 0.008. During the course of MW assisted treatment, BOD<sub>5</sub>/COD increased steadily and reached 0.467 in 60 min. This improvement in BOD<sub>5</sub>/COD value is significant for in situ wastewater treatment, because microbiological process is suggested after CWAO treatment for further economical purification of wastewaters.

#### 4. Conclusions

The MW assisted method, or we call it MW assisted catalytic wet air oxidation (MW/CWAO) because air was applied as oxygen source, was effective for the degradation of non-biodegradable organic substances (such as H-acid) in aqueous solution. Existence of AC, which acted as a catalyst and the material for MW energy absorption, and oxygen supply were proven to be critical factors for performance of the process. Organic substances could experience sequent oxidative reactions, and were ultimately mineralized during the treatment. Because the process improved biodegradability of wastewater apparently, it is beneficial for the further purification of wastewater by biochemical method. This process can be operated under the atmospheric pressure. Therefore, it will reduce the operation risk resulting from high pressure and high temperature within conventional CWAO.

#### Acknowledgement

Authors acknowledge the financial support from National Natural Science Foundation of China (Project No. 20337020).

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