



Degradation of bisphenol-A using ultrasonic irradiation assisted by low-concentration hydrogen peroxide

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Abstract

This study investigated the degradation of bisphenol-A (BPA) by ultrasonic irradiation in the presence of different additives (H₂O₂, air bubbles and humic acid) under various operating conditions, i.e., ultrasonic frequency, power intensity and power density. The results demonstrated that the BPA degradation followed pseudo first-order kinetics under different experimental conditions. The optimum power intensities were 0.9, 1.8, and 3.0 W/cm² at the frequencies of 400, 670, and 800 kHz, respectively. At the fixed frequency (800 kHz), the degradation rate of BPA was shown proportional to the increase of power density applied. With this manner, the BPA sonolysis could be facilitated at H₂O₂ dosage being lower than 0.1 mmol/L; while BPA degradation was hindered at H₂O₂ concentration in excess of 1 mmol/L. Additionally, BPA removal was shown to be inhibited by the presence of aeration and humic acid during ultrasonic irradiation. The present study suggested that the degradation rate of BPA assisted by ultrasonic irradiation was influenced by a variety of factors, and high BPA removal rate could be achieved under appropriate conditions.

Key words: bisphenol-A (BPA); ultrasonic irradiation; power density; additives

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Introduction

Bisphenol-A (BPA) has been widely accepted as one of important monomers for the production of variety of chemical materials such as polycarbonate, epoxy resins, and flame retardants (Levy et al., 2004; Staples et al., 1998). For this reason, BPA has been frequently detected in both the industrial wastewater and drinking water sources recently (Guo and Feng, 2009). BPA is known as an endocrine-disrupting compound that has to be removed from drinking water due to its estrogenic activity and toxicity to human bodies. However, conventional processes for drinking water treatment may be of inefficiency for BPA elimination (Korshin et al., 2006; Kuruto-Niwa et al., 2002). Therefore, several alternative processes have been proposed. Of all the methods developed so far, advanced oxidation processes (AOPs) offers several particular advantages in terms of unselective degradation of BPA into the final mineralized form at a diffusion-limited reaction rate, which results from the production of highly oxidative hydroxyl radical ($\cdot\text{OH}$). Recently, the AOP induced by ultrasonic irradiation has been found a promising manner for aqueous BPA decomposition (Adewuyi, 2005; Guo

et al., 2006, 2007; Sivakumar et al., 2002). In general, ultrasonic degradation of organic compounds takes place via two pathways, i.e., homolytic bond breaking and reaction with radicals (Guo and Feng, 2009; Rehorek et al., 2004). The former one usually occurs with the cavitation of micro-bubbles being accompanied with extremely high temperature ($> 5000^\circ\text{C}$) and high pressure (c.a. $5 \times 10^7 \text{ Pa}$). While in the latter mechanism, ultrasonic irradiation provokes the generation of $\cdot\text{OH}$ through thermal dissociation of water, and the reactions for $\cdot\text{OH}$ generation and depletion under ultrasonic irradiation are given in Reactions (1)–(4) (Nie et al., 2008) as follows:



Ultrasonic irradiation has been addressed able to successfully degrade many types of pollutants like chlorinated hydrocarbons, aromatic compounds, textile dyes, phenolic compounds and esters (Gogate and Pandit, 2004). Recently, the degradation of BPA using ultrasonic irradiation has drawn a growing interest widely (Guo and Feng, 2009). Gültekin and Ince (2008) investigated the role of

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$\cdot\text{OH}$ in ultrasonic degradation of BPA, and found that the radicals played a dominant role in the reaction. In addition, Guo and Feng (2009) plus Loan et al. (2007) studied the effect of ultrasonic irradiation combined with ozone or Fenton process, and they demonstrated that ozone or Fenton agents was capable of enhancing the degradation efficiency of BPA. Nevertheless, these studies involved neither the natural water contaminated by BPA nor the possible effect of other organic matters existing in natural water body. Hence, several interesting questions in ultrasonic irradiation of BPA may arise. For example, it seems uncertain whether the optimal frequency was the same for different power intensities. Besides, we also wondered if the ultrasonic degradation of BPA could be impacted by aeration, the addition of H_2O_2 , as well as humic acid present in natural water sources.

The objective of this study is to explore the effects of frequency, power intensity, power density, H_2O_2 addition, aeration, and humic acid on the ultrasonic irradiation of BPA based on synthesized natural water samples. In particular, the key operating parameters, i.e. frequency, power intensity and power density were optimized to maximize the rate of BPA degradation by ultrasonic irradiation.

1 Materials and methods

1.1 Reagents

BPA (purity 99%) and humic acid (technical grade) were obtained from Sigma-Aldrich Corp. (USA). Unless stated otherwise, all the other chemicals (e.g., 30 % of H_2O_2 , KH_2PO_4 and Na_2HPO_4) were analytical grade and obtained from China National Medicines Corporation. All the solution used was made from Milli-Q deionized water (resistivity of $18.2 \text{ M}\Omega\cdot\text{cm}$). During the reaction, the pH was maintained about 7.0 with the addition of phosphate buffered solution.

1.2 Ultrasonic apparatus and experimental procedure

As shown in Fig. 1, a completely-mixing batch reactor was composed of a 250 mL glass cylinder vessel and an ultrasonicator (SF400, SF600 and SF800, the Shanghai Acoustics Laboratory, Chinese Academy of Sciences). The

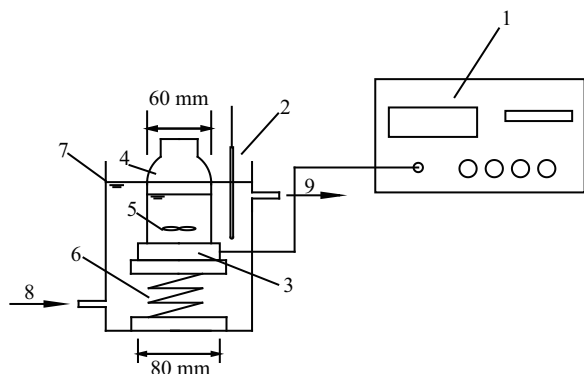


Fig. 1 A schematic diagram of experimental set-up in ultrasonic irradiation. (1) ultrasonicator; (2) temperature detector; (3) base-plate transducer; (4) glass vessel; (5) stirrer; (6) extension frame; (7) water-jacketed; (8) water inlet; (9) water outlet.

solution was mixed at 120 r/min by using a magnetic stirrer. The vessel was installed on the ultrasonic transducer, both of which were immersed by tap water. The tap water was continuously entered and discharged through the inlet port at the bottom and outlet port on the upper side, respectively, and this maintained the temperature inside the reactor constant ($30 \pm 1^\circ\text{C}$). The extension frame functioned to adjust the height of whole reactor. An air pump (ACO-9610, Haili Company, China) was employed to sparge air bubbles into the solution, and the aeration rate was controlled by a regulating valve. At each experimental run, a predetermined amount of BPA with different concentration solution was introduced to the vessel, and then the certain amount of H_2O_2 or humic acid was added to the solution to investigate their effect on the performances of ultrasonic irradiation. The reaction was initiated by starting ultrasonic irradiation. At each sampling time, 1 mL sample was collected for sample analysis.

1.3 Chemical analysis

The measurement of the concentration of BPA was performed using a high-performance liquid chromatograph (LC-2010AHT, Shimadzu, Japan) equipped with a BDS-C18 column (dimensions $150 \text{ mm} \times 4.6 \text{ mm}$, $5 \mu\text{m}$, Agilent Technologies, USA) and a UV detector at a wavelength of 280 nm. The mobile phase was the mixture of Milli-Q deionized water and HPLC-grade methanol (30:70, V/V) with a flow rate of 1.0 mL/min. The injection volume was $50 \mu\text{L}$.

2 Results and discussion

2.1 Effects of ultrasonic frequency, power intensity, and power density

In this section, the power inputs were set at a density of 33, 66 and 100 W, respectively, which were applied to the reactor containing solution of 200 mL. Ultrasonic degradation of organic matters in aqueous media generally follows first-order kinetics as described in Eq. (5) by Cloussi et al. (1999).

$$\ln(C/C_0) = kt \quad (5)$$

where, C_0 and C are BPA concentration at initial and time t , respectively, and k is the first-order kinetic rate constant.

A linear correlation between $\ln(C/C_0)$ and irradiation time was obtained at different ultrasonic frequencies (400, 670 and 800 kHz) and power intensities (0.9, 1.8, and 3.0 W/cm^2) (Fig. 2). For the ultrasonic frequencies and power intensities tests, BPA degradation could be described by pseudo first-order kinetics ($R^2 > 0.99$), and this result was in good agreement with the previously reported studies (Guo and Feng, 2009; Torres et al., 2008).

The observed rate constants of ultrasonic degradation of BPA obtained are compared in Fig. 3. It was noted that the optimal power intensity differed from the frequencies used. The optimal power intensities at 400, 670, and 800 kHz were 0.9, 1.8, and 3.0 W/cm^2 , which corresponded well

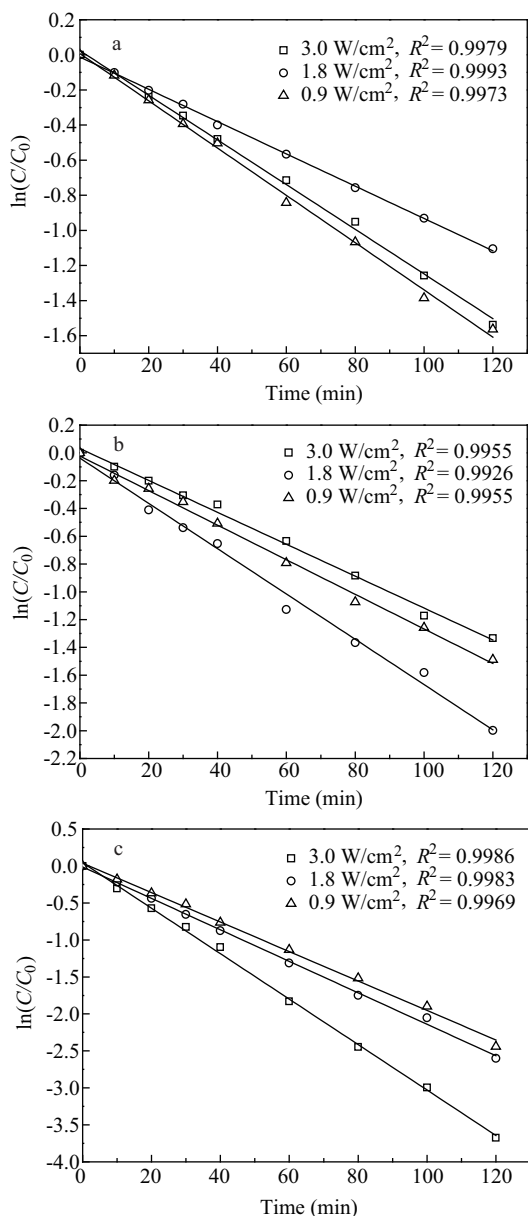


Fig. 2 BPA degradation as function of ultrasonic frequencies and power intensities 400 (a), 670 (b), and 800 (c) kHz. Experimental conditions: initial BPA concentration of 1 mg/L and power density of 0.5 W/mL.

with the observed rate constants of 0.0135, 0.0163, and 0.0307 min⁻¹, respectively. The most likely reason for this

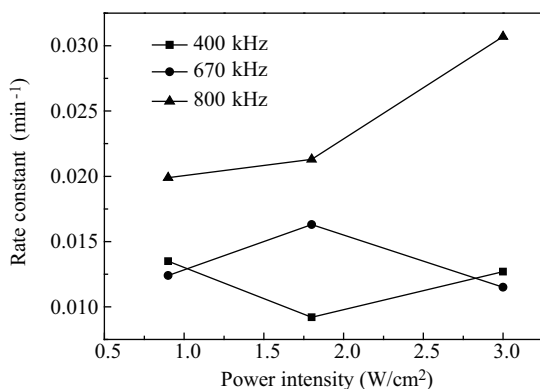


Fig. 3 Comparison of BPA degradation rate constants at different frequencies and power intensities. Experimental conditions: initial BPA concentration of 1 mg/L and power density of 0.5 W/mL.

difference may be some of power is transmitted into the reactor, leaving the remaining power transformed to heat (Ma and Zhao, 2005). For each frequency, the transmitting ratio did not appear invariable, and the best performance did not collaborate with the highest power intensity either. Obviously, the treatment can be accomplished much more efficiently with the higher frequency. This result appears to be in line with that reported by Petrier et al. (1996), who found the ultrasonic degradation proceeded at an increased rate 7.8 times for atrazine and 4.8 times for pentachlorophenol at frequency of 500 kHz than at 20 kHz. Higher ultrasonic frequency could promote the generation of free radicals in solution. However, owing to shorter bubble lifetime for higher frequencies, the free radicals may escape more readily from the bubbles, and migrate towards the liquid bulk prior to undergoing any recombination reactions (Psillakis et al., 2004). Jiang et al. (2006) investigated the degradation of 4-chlorophenol under a frequency in the range of 20–800 kHz, achieving the maximum removal at the optimal frequency of 200 kHz. Moreover, Torres et al. (2008) reported the greatest yield of free radicals at the lowest frequency. Such difference in the effect of frequency can be the consequence of the implementation of different power intensities and ultrasonic properties. Taken together, the frequency can affect the generation of free radicals in a complicated manner, which relies on a number of factors including the dynamics of bubble formation, solution chemistry (e.g., pH), and other ultrasonic characteristics (e.g., power intensity).

Another key operational parameter that should be addressed is power density (Desai et al., 2008). Gronroos et al. (2001) have shown that the power density was required above a cavitation threshold prior to producing cavitation in the bulk solution. The effect of the power density on the BPA sonolysis is illustrated in Fig. 4. For any particular power density, the BPA degradation proceeded in accordance with a pseudo first-order kinetic. Higher power density could result in faster BPA degradation because of more cavitation in a unit volume, and thus the production of more reactive radicals. Another likely reason may be attributed to the decrease in the power density resulting from an increase in the operating volume. The increased operating volume caused more dead zones where

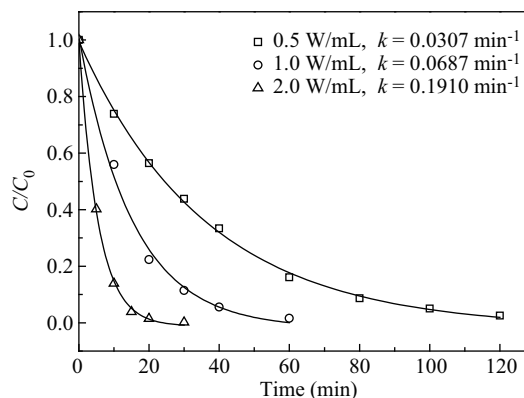


Fig. 4 BPA degradation as function of ultrasonic power densities. Experimental conditions: initial BPA concentration of 1 mg/L, ultrasonic frequency of 800 kHz, and power intensity of 3.0 W/cm².

the cavitation activity was lowered (Desai et al., 2008).

Overall, the rate of BPA degradation by ultrasonic is investigated as a function of frequency, power intensity and power density, which can be presented by Eq. (6).

$$k = x_1^a x_2^b x_3^c \quad (6)$$

where, x_1 , x_2 and x_3 is the ultrasonic frequency, power intensity and power density, respectively. a , b and c , which is the influencing coefficients of rate constant (k), can be calculated using the statistic toolbox provided by MATLAB software. Based on the values of a , b and c obtained, the power density is suggested to be the most significant parameter that controls the rate constant of degradation. The Eq. (6) is rearranged by Eq. (7) with the related coefficient R^2 of 0.7534.

$$k = \frac{x_3^{1.6935}}{x_1^{0.3974} x_2^{0.0841}} \quad (7)$$

2.2 Effect of H₂O₂ addition

Taking into account that higher power density is effective for faster BPA degradation, we applied a power input of 100 W and the solution volume of 100 mL, giving the power density of 1 W/mL in the subsequent experiments. During the ultrasonic treatment, relatively high local concentration of $\cdot\text{OH}$ are measured and significant quantities of H₂O₂ can be found as indicated by the reactions in Reactions (1)–(4). Since H₂O₂ is a crucial intermediate produced during ultrasonic irradiation, it is a common practice to add H₂O₂ into the system to improve the efficiency of treatment. Following the addition of H₂O₂, the variation of BPA concentration with respect to time also fitted to pseudo first-order kinetic model as shown in Fig. 5, and the observed rate constants and correlation coefficients are given in Table 1. The addition of 0.1 mmol/L H₂O₂ was demonstrated to greatly increase the observed rate constant from 0.0687 min⁻¹ to 0.0908 min⁻¹. Such improvement is in good consistence with the studies carried out by Kim et al. (2007), who reported that the increased rate of TOC removal by ultrasonic irradiation correlated positively to the increasing dosage of H₂O₂ below the concentration of 10 mmol/L. On the other hand, further increase in H₂O₂ dosage inhibited BPA degradation. Ultrasonic degradation

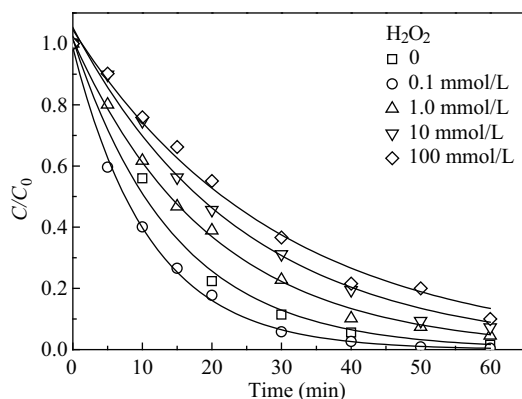


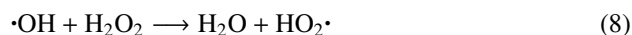
Fig. 5 Effect of H₂O₂ dosage on the ultrasonically-induced degradation of BPA. Experimental conditions: initial BPA concentration of 1 mg/L, ultrasonic frequency of 800 kHz, and power density of 1 W/mL.

Table 1 Summary of the parameters for kinetics models (pseudo first-order) on ultrasonic degradation of BPA at different H₂O₂ dosages.

Initial H ₂ O ₂ concentration (mmol/L)	BPA degradation rate constant (min ⁻¹)	R ²
0	0.0687 ± 0.0039	0.9945
0.1	0.0908 ± 0.0024	0.9982
1	0.0505 ± 0.0013	0.9979
10	0.0411 ± 0.0022	0.9905
100	0.0342 ± 0.0018	0.9899

with 1, 10, and 100 mmol/L H₂O₂ corresponded to the observed rate constants of 0.0505, 0.0411, and 0.0342 min⁻¹, respectively, all of which were below the level obtained by ultrasonic irradiation individually. Similar observations in the sonolysis of other organic species (e.g., phenol and microcystin-LR) have been reported in previous studies (Song et al., 2006; Zhao et al., 2004).

Promoted by ultrasonic irradiation, H₂O₂ is able to produce additional $\cdot\text{OH}$ (Gogate and Pandit, 2004), and thus improves the BPA degradation. However, over high H₂O₂ concentration may inhibit the radical-induced oxidation, because H₂O₂ serving as $\cdot\text{OH}$ -scavenger itself significantly compete with BPA for $\cdot\text{OH}$. The $\cdot\text{OH}$ scavenging reactions originating from H₂O₂ are presented Reactions (8) and (9), as reported by Olson and Barbier (1994). The amount of H₂O₂ generated during the ultrasonic irradiation here was sustained within an appropriate range, suggesting minimal need of external addition of H₂O₂.



2.3 Effect of aeration

The effect of aeration on BPA degradation is given in Fig. 6, which clearly shows that the rate of BPA degradation was inhibited by aeration. Ultrasonic degradation with 5 and 10 L/min air flow accounted for the observed rate constants of 0.0568 and 0.0301 min⁻¹, respectively, both of which were below the level for ultrasonic irradiation in the absence of aeration.

BPA is characterized as a relatively non-volatile compound, and the reaction between BPA and radicals proceeds at the interface of cavitation bubbles created by ultrasonic irradiation and bulk solution. Aeration exter-

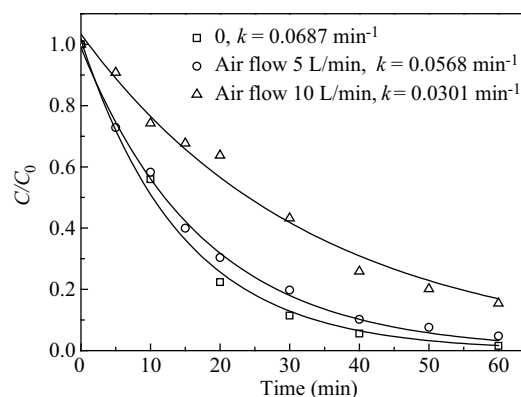


Fig. 6 Effect of aeration on the ultrasonically-induced degradation of BPA. Experimental conditions: initial BPA concentration of 1 mg/L, ultrasonic frequency of 800 kHz, and power density of 1 W/mL.

nally introduces more bubbles in the solution, as a result of facilitating the mass transfer of BPA to the interface of these bubbles and the bulk solution. Nonetheless, it was noticed that very limited amount of radicals are produced at the surface of the bubbles from aeration. This could provide a most likely explanation to the decreased overall BPA degradation rate inhibited by aeration. A higher aeration rate leads to more bubbles without radicals in the solution as a consequence of more severe inhibition. Besides, the available $\cdot\text{OH}$ outside the cavitation bubbles depend heavily on the temperature (Torres et al., 2008). For this reason, the aeration would also reduce the interface temperature of cavitation bubbles, which decreased the availability of usable $\cdot\text{OH}$ for BPA degradation. By investigating the effect of saturating gas on the production of $\cdot\text{OH}$, Yazici et al. (2007) found that the production rate of $\cdot\text{OH}$ decreased in the presence of the aeration at an air-flow rate of 0.27 L/min.

2.4 Effect of humic acid

In this study, humic acid was selected as the representative of NOM (natural organic matters) and the effect of NOM (0, 5 and 10 mg/L) on the ultrasonic degradation of BPA is shown in Fig. 7. The ultrasonic degradation of BPA was negatively impacted by humic acid. The BPA degradation rate in the presence of 10 mg/L NOM was 2.4 times lower than that in the absence of NOM. With the increase of NOM concentration, the inhibition degree increased. Naddeo et al. (2007) explained the ultrasonic irradiation of humic acid based on two mechanisms in terms of oxidation and physical collapse. Humic acid molecules in the vapor phase or the surrounding liquid of the collapsing bubbles are readily oxidized by $\cdot\text{OH}$, giving rise to disassociation or protonation of $-\text{COOH}$ and phenolic-OH groups (Joyce et al., 2003). In this study, humic acid may have a competition with BPA for $\cdot\text{OH}$, and this side reaction slows down the BPA degradation. This observation should be well consistent with that obtained by Zachary et al. (2001), who addressed that the addition of humic acid could result in a decreased rate constant for three types of polycyclic aromatic hydrocarbons.

The absorbance of UV_{254} during the ultrasonic irradiation was observed to remain constant (Fig. 8). The

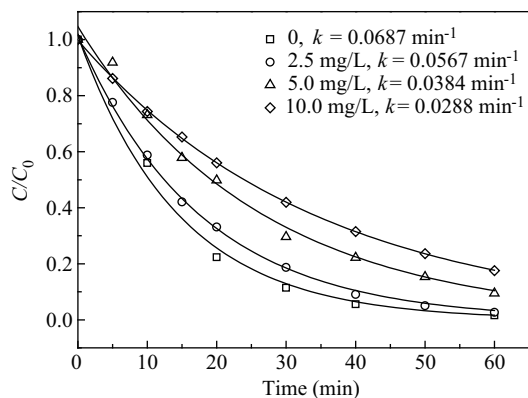


Fig. 7 Effect of humic acid on the ultrasonically-induced degradation of BPA. Experimental conditions: initial concentration of BPA of 1 mg/L, ultrasonic frequency of 800 kHz, and power density of 1 W/mL.

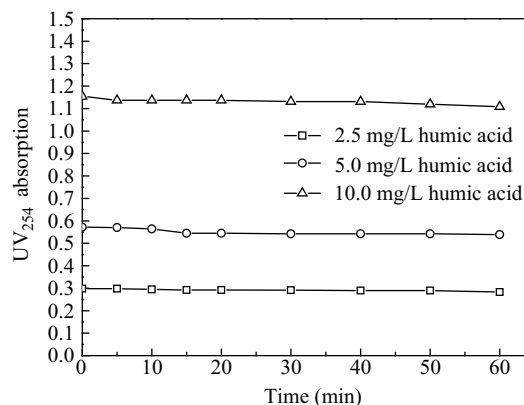


Fig. 8 Variation of UV_{254} absorption with time at different humic acid concentrations.

insignificant change of the UV_{254} absorption with the reaction time indicates the existence of chromophore and auxochromes. Under ultrasonic irradiation, the alteration of molecular structure of humic acid and BPA may occur, except those humic acid and BPA can hardly be mineralized to CO_2 and H_2O (Naddeo et al., 2007). In the previous studies (Torres et al., 2008), some aliphatic acids that come from opening aromatic rings, were identified in the solution by using LC-MS technology at the end of ultrasonic degradation of BPA. These compounds are known not only toxic but also readily biodegradable, and they can be eliminated by ultrasonic method combined with UV irradiation (Torres et al., 2007).

3 Conclusions

The ultrasonic irradiation is directly related to the $\cdot\text{OH}$ production as a result of BPA degradation. In this study, the degradation rate was demonstrated to depend on various factors, including ultrasonic frequency, power intensity, power density, addition of H_2O_2 , aeration, as well as humic acids. Under different experimental conditions, the degradation rate exhibited a pseudo first-order kinetic behavior ($R^2 > 0.99$). The optimal experimental conditions were determined to be the frequency of 800 kHz and the power intensity of 3.0 W/cm^2 . Increasing the power density could achieve higher rate of BPA degradation. Furthermore, the impact of various additives to the BPA degradation efficiency was observed different. The addition of H_2O_2 with a low concentration could facilitate BPA ultrasonic degradation efficiently. However, the further increased dosage of H_2O_2 (the mole ratio $\text{H}_2\text{O}_2:\text{BPA} > 200:1$) was shown to decrease the degradation efficiency of BPA. It was also found that the BPA ultrasonic degradation was inhibited by aeration and the presence of humic acids. This study provides a proof-in-concept demonstration of using ultrasonic irradiation as a promising manner for effective treatment of BPA in water under the optimized operational conditions.

Acknowledgments

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