



Ultrasonic irradiation-induced degradation of low-concentration bisphenol A in aqueous solution

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ABSTRACT

The treatment of bisphenol A (BPA)-contaminated water has attracted great interest recently. In this contribution, ultrasound (US) was applied to remove low-concentration BPA in aqueous solution at the frequency of 20 kHz, and the effects of ultrasonic intensity and ozone on BPA removal were evaluated for the first time. Considering the coexistence of halomethanes and BPA in chlorinated drinking water, BPA was degraded under US in the presence of CCl₄. In addition, the main intermediates resulting from BPA ultrasonic degradation were identified by gas chromatography–mass spectrometry. On the basis of these studies, [•]OH radical induced oxidation was identified as the major destruction pathway during BPA sonolysis.

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1. Introduction

Bisphenol A (BPA) is a representative endocrine disrupting compound due to its large consumption as a monomer for the production of polycarbonate and epoxy resins, unsaturated polyester–styrene resins and flame retardants [1,2]. BPA is not only found in industrial wastewater, but also can be encountered in raw water. It is reported that BPA exhibited estrogenic activity [3], which increases the rate of proliferation of breast cancer cells and induces acute toxicity to freshwater and marine species [4]. BPA can be degraded by microorganisms, however, it is hard to be completely eliminated by conventional biological treatment method, which inevitably leads to the existence of low-concentration BPA in aqueous solution [5]. Under these circumstances, the effective removal of BPA is usually limited to physicochemical methods, such as photo-catalytic oxidation, Fenton oxidation and ozonation [6–8]. However, these processes require the addition of catalysts and oxidants to the solution, which may result in the high expenses and secondary pollution [9,10].

Nowadays, the application of ultrasound (US) in wastewater treatment has attracted great interest [11–15]. In principle, ultrasonic irradiation generates cavitation bubbles in aqueous solutions, which repeats a cycle of formation, growth and collapse in accordance with ultrasonic waves. An implosive collapse of

cavitation bubbles by adiabatic compression results in a specific environment with extremely high temperature (up to 5000 °C) and high pressure (about 5×10^7 Pa). As a result, organic compounds at the bubble/water interface can be thermally decomposed and a large amount of reactive radicals, such as HO[•], are generated via the thermal dissociation of water. These initiate a series of radical reactions resulting in the decomposition of pollutants generating compounds with lower molecular weight and lower toxicity [16].

In fact, US has been applied to degrade BPA in water since 2006. Torres et al. [17–19] made great contributions in this aspect. On the one hand, they achieved complete mineralization and determined BPA intermediates using high-pressure liquid chromatography–mass spectrometry (HPLC–MS) [19,20], but these intermediates were different from that of O₂-mediated sonolysis of BPA identified by Kitajima et al. [20]. On the other hand, Torres et al. [19] made a comparative study of ultrasonic cavitation and Fenton's reagent for BPA degradation in deionised and natural water. Ioan et al. [21] demonstrated that ultrasonic treatment was a suitable way to improve the Fenton oxidation of BPA.

It is noteworthy that the initial concentrations of BPA in aqueous solution for above-mentioned sonolysis are at the level of mg/L, much higher compared to its concentration in raw water. Consequently, in this study, low-concentration BPA (100 μg/L) was used to be degraded in dependence on ultrasonic intensity and in presence of O₃, CCl₄. Considering the complexity and inconsistency in the intermediates of BPA sono-degradation, the degradation path-

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ways were discussed based on the measured intermediates by gas chromatography–mass spectrometry (GC–MS).

2. Materials and methods

2.1. Materials

BPA was obtained from Shanghai Chemicals Factory and used without further purification. All chemicals used were of analytical grade and the solutions in the experiment were prepared in milli-Q water. Solution pH was adjusted using H_3PO_4 (0.01 mol/L) or NaOH (0.01 mol/L) solution.

2.2. Ultrasonic device

The experimental setup consisted of a cylindrical water-jacketed glass vessel (75 mL) and an ultrasonicator (JY92-2D, Inc. Scientz, China), which was connected with an immersible horn via a titanium alloy rod (6 mm in diameter). The reactor was double-walled-type and water was circulated between the walls in order to keep the temperature constant at $25 \pm 0.5^\circ C$, the temperature was monitored with a thermocouple.

2.3. Sonolysis and analysis

For a typical experiment of ultrasonic degradation, 50 mL of a 100 $\mu g/L$ BPA aqueous solution was placed in the glass vessel and the titanium alloy rod was immersed beneath the liquid surface of the solution (2 cm). The ultrasonic irradiation was then conducted with a frequency at 20 kHz and samples were taken periodically. The concentrations of BPA were determined using a high-pressure liquid chromatograph (HPLC, Aligent 1100) equipped with a multiple wavelength UV diode array detector. The detection wavelength was set at 228 nm and the detector temperature was kept at $30^\circ C$. A mixture of acetonitrile–water solution (60/40, v/v) at a flow rate of 1 mL/min was used as the eluent throughout analysis. The BPA intermediates after 1 h ultrasonic degradation were identified by a HP 5890–5971 GC–MS system after pretreatment including extraction and silylation. Helium was used as carrier gas with a flow rate at 1 mL/min. The GC oven temperature was held constant at $40^\circ C$ for the first 5 min, then increased up to $280^\circ C$ with a rate of $6^\circ C/min$ and finally stayed constant for 10 min.

3. Results and discussion

3.1. Ultrasonic irradiation-induced BPA degradation under different ultrasonic intensities

As pictured in Fig. 1, about 33.2%, 44.9%, 51.1% and 55.0% of BPA was decomposed at the ultrasonic intensities determined by calorimetry of 20 W/cm^2 , 40 W/cm^2 , 60 W/cm^2 and 80 W/cm^2 , respectively. This indicates that increasing ultrasonic intensity was favorable for BPA degradation, which was in agreement with findings from Guo et al. [22] during degradation of 2,4-dinitrophenol. A linear relation between $\ln(C_t/C_0)$ and irradiation time for sono-oxidation at different ultrasonic intensities was obtained, indicating that BPA degradation followed pseudo-first-order kinetics. The corresponding rate constants of BPA sonolysis at different ultrasonic intensities are compared in Fig. 2. It is noted that the rate constants increased exponentially with the increase of ultrasonic intensity. The beneficial effect of ultrasonic intensities could be ascribed to the increased cavitation effect. However, too high ultrasonic intensity can make cavitation bubbles grow largely in negative pressure-phase of ultrasonic wave, which not

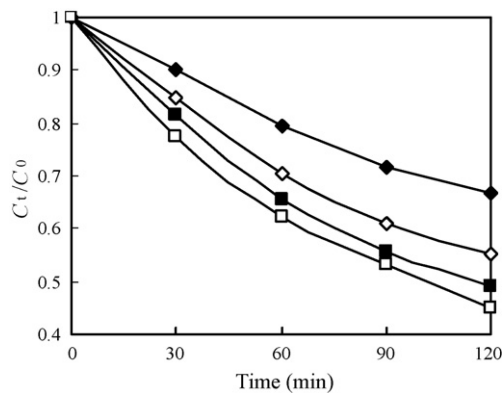


Fig. 1. BPA degradation under different ultrasonic intensities. BPA concentration: 100 $\mu g/L$; ultrasonic frequency: 20 kHz; pH: 6.5; temperature: $25 \pm 0.5^\circ C$; (◆) 20 W/cm^2 ; (◇) 40 W/cm^2 ; (■) 60 W/cm^2 ; (□) 80 W/cm^2 .

only results in inadequate collapse of cavitation bubbles, but also forms the ultrasonic screen. As a result, this led to a slower increase of BPA degradation rate constants at higher levels of ultrasonic intensity.

In general, two different pathways can be discussed for the degradation of organic compounds under the circumstance of high temperature and pressure induced by the ultrasonic irradiation: (1) homolytic breaking of the bonds of volatile compounds in cavitation bubbles and (2) reaction with $\bullet H$ and $\bullet OH$ radicals. Generally, non-volatile organic compounds are believed to be decomposed via the reaction with radicals [23]. Rehorek et al. [23] studied the ultrasonic decomposition of dyes and observed that the concentration of radicals increased exponentially with increasing ultrasonic intensity. Hence, it can be expected that there existed an exponential relationship between the rate constant and the ultrasonic intensity if pollutant decomposition is controlled by a radical-oxidation reaction. Consequently, it can be primarily concluded from Fig. 2 that the ultrasonic degradation of BPA in aqueous solution is mainly attributed to the $\bullet OH$ radical oxidation.

3.2. Effect of O_3 on BPA degradation

O_3 is usually used to remove persistent organic pollutants in water in combination with ultrasonic irradiation [24,25]. In our experiments, 50 mL of 100 $\mu g/L$ BPA was treated using US and O_3 with different flow rates, and BPA degradation efficiencies are compared in Fig. 3. Meanwhile, BPA was irradiated at the ultrasonic intensity of 60 W/cm^2 , O_3 flow rate of 10 mL/min and the solution

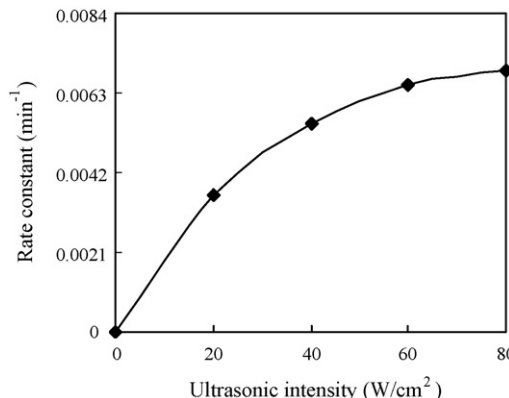


Fig. 2. Dependence of BPA degradation rate constants on ultrasonic intensities.

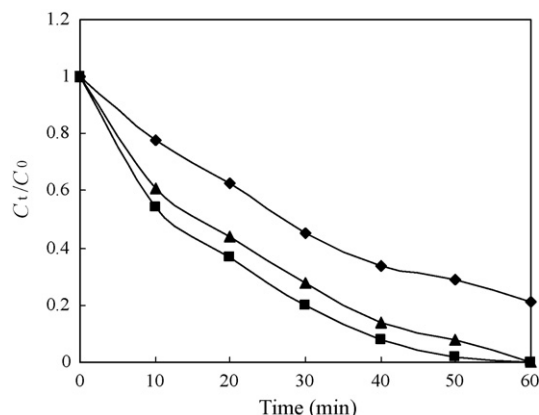


Fig. 3. BPA ultrasonic degradation under different O₃ flow rates. BPA concentration: 100 μg/L; ultrasonic frequency: 20 kHz; ultrasonic intensity: 60 W/cm²; pH: 6.5; temperature: 25 ± 0.5 °C; O₃ flow rate: (♦) 5 mL/min; (▲) 10 mL/min; (■) 20 mL/min.

pH value of 6.5. The removals of BPA in the US, O₃ and US/O₃ systems are described in Fig. 4.

It is noteworthy from Fig. 3 that BPA ultrasonic removals increased with the increase of O₃ flow rates. However, it is found that further increasing O₃ flow rate only slightly enhanced BPA degradation efficiency when O₃ flow rate was higher than 10 mL/min, which is ascribed to the destroys of cavitation bubbles at the high O₃ flow rate. At the same time, it can be gained from Fig. 4 that BPA was completely removed after 60 min treatment in the combined US/O₃ system. In contrast, only 34.6% and 63% of BPA were degraded in the single US and O₃ systems. It is observed that BPA degradation fitted pseudo-first-order kinetics in all degradation systems, and the reaction rate constants of BPA in US/O₃, US and O₃ systems were 0.0482 min⁻¹, 0.0063 min⁻¹ and 0.015 min⁻¹, respectively. The results indicate that BPA degradation in the combined US/O₃ process showed a synergistic effect. Generally, Eq. (1) was used to assess the synergistic effect in a couple process:

$$f = \frac{k_{US/O_3}}{k_{US} + k_{O_3}} \quad (1)$$

where f is the synergistic index and k_{US} , k_{O_3} , and k_{US/O_3} are the rate constants for BPA degradation in the US, O₃, and US/O₃ systems, respectively. The synergistic index in US/O₃ system was calculated to be 2.26, indicating a considerably synergistic effect in this coupled system.

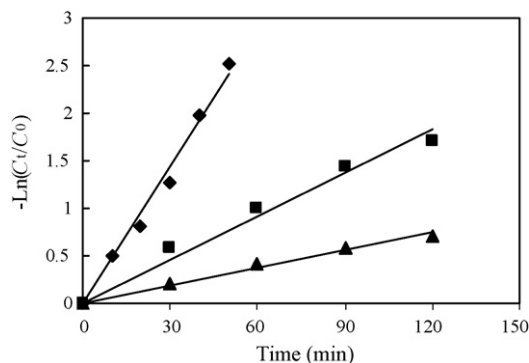


Fig. 4. BPA degradation in US, O₃, and US/O₃ systems. BPA concentration: 100 μg/L; ultrasonic frequency: 20 kHz; ultrasonic intensity: 60 W/cm²; O₃ flow rate: 10 mL/min; pH: 6.5; temperature: 25 ± 0.5 °C; (▲) US; (■) O₃; (♦) US/O₃.

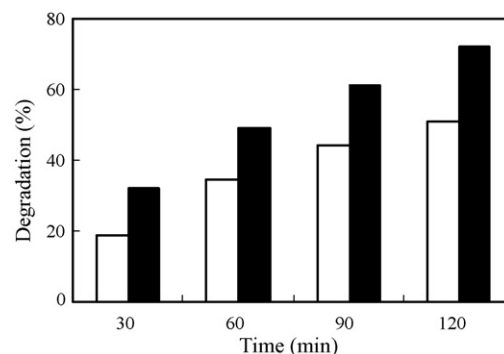


Fig. 5. BPA degradation in the presence and absence of CCl₄. BPA concentration: 100 μg/L; CCl₄ concentration: 25 μg/L; ultrasonic frequency: 20 kHz; ultrasonic intensity: 60 W/cm²; pH: 6.5; temperature: 25 ± 0.5 °C; empty: BPA; filled: BPA/CCl₄.

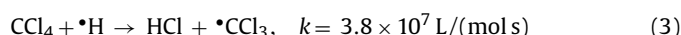
In the US/O₃ system, the mass transfer and decomposition of O₃ can be enhanced by US. As a result, O₃ concentration in liquid phase and the amounts of active free radicals, such as •OH, •O₂⁻ and •O₂H, are increased [26,27], which results in the higher degradation efficiency of BPA in the US/O₃ system. Weavers et al. [28] also drew a similar conclusion after degradation of aromatic compounds using a combination of sonolysis and ozonolysis.

3.3. Degradation of the mixture of BPA and CCl₄

In chlorinated drinking water, there may exist some halomethanes. In order to make clear the effect of these halomethanes on ultrasonic degradation of BPA, we prepared 50 mL mixture containing 100 μg/L BPA and 25 μg/L CCl₄, followed by ultrasonic irradiation at an ultrasonic intensity of 60 W/cm². The removal efficiencies of BPA in the mixture are shown in Fig. 5.

The degradation efficiency of BPA in the US/CCl₄ system was higher compared to that in the US system. This indicates that US has a promising future in removing mixtures containing BPA and some halomethane in aqueous solution.

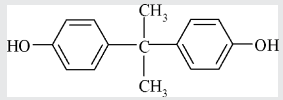
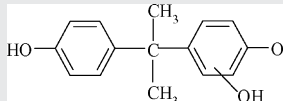
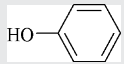
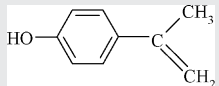
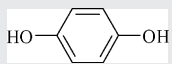
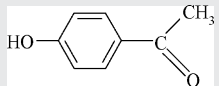
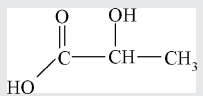
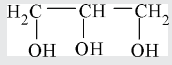
When adding CCl₄ to BPA solution in the US process, the following reactions may occur:



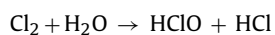
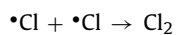
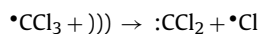
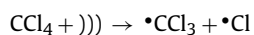
According to the high rate constant (Eq. (3)), CCl₄ is a hydrogen atom scavenger. Consequently, CCl₄ inevitably restrained the recombination reaction between hydrogen atom and •OH radicals in sonochemical degradation process. Considering that there is no reaction between CCl₄ and •OH radicals [29], an increase in •OH radical concentration can be achieved, which is contributed to BPA degradation.

In addition, CCl₄, as a hydrophobic organic compound, is prone to enter the cavitation bubbles and undergoes degradation by pyrolytic cleavage. The C–Cl bond in CCl₄ is preferentially broken at high temperature to produce large amount of •Cl radicals compared to the H–O bond in H₂O due to the fact that the C–Cl bond energy in CCl₄ is 73 kcal/mol and the H–OH bond energy in H₂O is 119 kcal/mol. The generation of •Cl radicals initiates a series of reactions to produce oxidants, such as HClO and chlorine-containing radicals (•Cl, •CCl₃ and :CCl₂), which facilitates the oxidation of BPA in aqueous solution (Eqs. (4)–(7)). In our study, BPA solution pH decreased from the initial value of 6.5 to a final of 4.6 after 2 h ultrasonic irradiation in the presence of CCl₄, which is partially attributed to the formation of HClO and HCl during the degradation

Table 1
The main intermediates during BPA ultrasonic irradiation alone

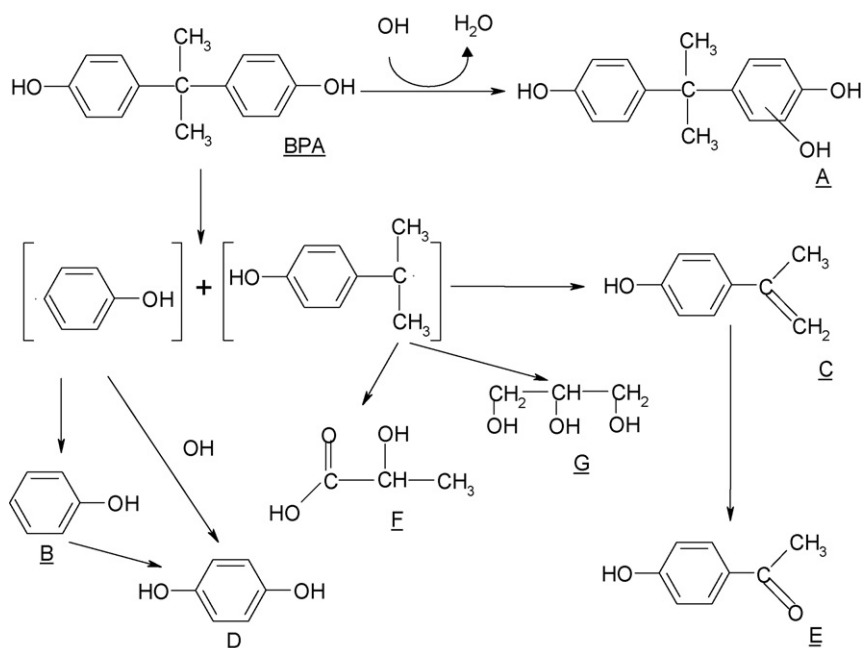
| Compound list | Retention time (min) | Main fragment ions (<i>m/z</i>) | Molecular weight before and molecular weight after silylation | Molecular structure |
|---------------|----------------------|-----------------------------------|---|--|
| BPA | 35.10 | 372, 357, 207, 73 | 228, 372 |  |
| A | 36.85 | 460, 445, 357, 207, 73 | 244, 460 |  |
| B | 11.20 | 166, 151, 77, 73 | 94, 166 |  |
| C | 19.20 | 206, 191, 91, 73 | 134, 206 |  |
| D | 20.02 | 254, 239, 108, 73 | 110, 254 |  |
| E | 23.50 | 208, 193, 151, 89, 73 | 136, 208 |  |
| F | 11.62 | 147, 117, 73 | 90, 234 |  |
| G | 17.18 | 205, 147, 73 | 92, 308 |  |

process [30]:



3.4. Identification of intermediates during BPA ultrasonic irradiation

The intermediates of BPA sonolysis are relatively complicated. Torres et al. [17] and Kitajima et al. [20] determined the intermediates during BPA ultrasonic irradiation, but they came to a different conclusion. Kitajima et al. [20] identified only one intermedi-



Scheme 1. Main degradation pathways of low-concentration BPA during ultrasonic treatment.

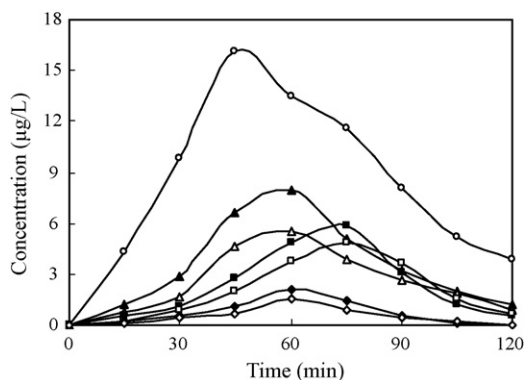


Fig. 6. Concentrations of the intermediates during BPA ultrasonic irradiation. BPA concentration: 100 µg/L; ultrasonic frequency: 20 kHz; ultrasonic intensity: 60 W/cm²; pH: 6.5; temperature: 25 ± 0.5 °C. (○) Monohydroxylated BPA; (▲) phenol; (△) 4-isopropenylphenol; (■) hydroquinone; (□) 4-hydroxyacetophenone; (◆) 2-hydroxypropionic acid; (◇) glycerol.

ate (2,3-dihydro-2-methylbenzofuran) during BPA sonolysis in O₂-saturated water. However, Torres et al. [17] found several intermediates, such as monohydroxylated BPA, 4-isopropenylphenol, quinone of monohydroxylated BPA, dihydroxylated BPA, quinone of dihydroxylated BPA, monohydroxylated-4-isopropenylphenol and 4-hydroxyacetophenone. To determine the intermediates of low-concentration BPA under US and gain its sonodegradation pathways, we identified the intermediates of 100 µg/L BPA using GC-MS after 1 h ultrasonic irradiation. The BPA intermediates are listed in Table 1. Four intermediates, monohydroxylated BPA, hydroquinone, 4-isopropenylphenol and 4-hydroxyacetophenone, were also determined by Torres et al. [17] in degrading BPA. Besides, phenol, 2-hydroxypropionic acid and glycerol were identified in our study. As presented in Scheme 1, the ultrasonic degradation of BPA was mainly attributed to the attack of •OH radicals resulting from water dissociation. The first •OH reactions yielded monohydroxylated BPA, whereas the breaking of C–C bond between isopropyl and benzene ring produced •C(CH₃)₂C₆H₄OH and •C₆H₅OH radicals. The former radicals were then converted into 4-isopropenylphenol, and the latter ones into phenol and hydroquinone. Subsequent hydroxylation of 4-isopropenylphenol and phenol led to the generation of 4-hydroxyacetophenone and hydroquinone. Besides, ultrasonic cavitation made isopropyl split from benzene ring in the •C(CH₃)₂C₆H₄OH radicals, and further formed 2-hydroxypropionic acid and glycerol [7].

At the same time, the concentrations of BPA intermediates were determined in our study. As Fig. 6 indicates, the concentrations of all intermediates increased simultaneously with BPA degradation, and subsequently decreased with ultrasonic irradiation. It can be seen that monohydroxylated BPA was the most significant intermediate, the highest concentration was determined to be 16.1 µg/L after 45 min ultrasonic irradiation. In contrast, the concentrations of 2-hydroxypropionic acid and glycerol were relatively lower, which were restricted to 2 µg/L.

4. Conclusions

Ultrasonic intensity and ozone markedly affect BPA sonochemical degradation in aqueous solution. US has been demonstrated as a more effective method to remove BPA in the presence of CCl₄. Besides, •OH radical-mediated oxidation is found to be the major destruction pathway during BPA sonolysis, and the main intermediates include monohydroxylated BPA, phenol, 4-isopropenylphenol,

hydroquinone, 4-hydroxyacetophenone, 2-hydroxypropionic acid and glycerol.

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