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ORIGINAL PAPER

Efficient oxidation of bisphenol A with oxysulfur radicals generated by iron-catalyzed autoxidation of sulfite at circumneutral pH under UV irradiation

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Abstract There is actually a need for efficient methods to clean waters and wastewaters from pollutants such as the bisphenol A endocrine disrupter. Advanced oxidation processes currently use persulfate or peroxymonosulfate to generate sulfate radicals. There are, however, few reports on the use of sulfite to generate sulfate radicals, instead of persulfate or peroxymonosulfate, except for dyes. Here we studied the degradation of the bisphenol A using iron(III) as catalyst and sulfite as precursor of oxysulfur radicals, at initial pH of 6, under UV irradiation at 395 nm. The occurrence of radicals was checked by quenching with *tert*-butyl alcohol and ethanol. Bisphenol A degradation products were analyzed by liquid chromatography coupled with mass spectrometry (LC–MS). Results reveal that iron(III) or iron(II) have a similar oxidation efficiency. Quenching experiments show that the oxidation rate of bisphenol A is 47.7 % for $SO_4^{\cdot -}$, 37.3 % for $SO_5^{\cdot -}$ and 15 % for $HO^{\cdot}$. Bisphenol A degradation products include catechol and quinone derivatives. Overall, our findings show that the photo-iron(III)–sulfite system is efficient for the oxidation of bisphenol A at circumneutral pH.

Keywords Bisphenol A · Iron · Sulfite · Oxysulfur radical · Hydroxyl radical · Advanced oxidation processes

Introduction

Advanced oxidation processes involving hydroxyl radicals ($HO^{\cdot}$) and sulfate radicals ($SO_4^{\cdot -}$) have strong oxidation capacity for degrading pollutants in wastewater treatment (Shukla and Oturan 2015; Lee et al. 2015). At present, advanced oxidation processes based on sulfate radicals have received much attention since $SO_4^{\cdot -}$ has several advantages than $HO^{\cdot}$. For instance, the standard reduction potential of $SO_4^{\cdot -}$ ($E^0 = 2.5$–3.1 V, normal hydrogen electrode) (Neta et al. 1988; Minero et al. 2006) is higher than $HO^{\cdot}$ ($E^0 = 1.8$–2.7 V, normal hydrogen electrode) (Buxton et al. 1988) at circumneutral pH. Moreover, $SO_4^{\cdot -}$ is more selective for oxidation than $HO^{\cdot}$ (Anipsitakis and Dionysiou 2004). In advanced oxidation processes based on sulfate radicals, $SO_4^{\cdot -}$ is usually generated by using various means (e.g., transition metal catalysis, irradiation or pyrolysis) to stimulate persulfate or peroxymonosulfate (Zhou et al. 2015). While compared to persulfate or peroxymonosulfate, sulfite is usually cheaper and more environmentally friendly as well as iron which is abundant on earth (Chen et al. 2012; Yates et al. 2014). Currently, reports on the advanced oxidation processes based on sulfate radicals using sulfite to replace persulfate or peroxymonosulfate for the degradation of organic pollutants are much rare. Several works on iron(II)/(III)–sulfite system with or without irradiation have been explored by our group (Chen et al. 2012; Zhang et al. 2013; Zhou et al. 2014a, b, 2015).

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However, previous studies mainly focused on the decolorization of azo dyes rather than oxidation of other organic pollutants, which limited the recognition of the new iron–sulfite system. Bisphenol A, an extensively used industrial chemical, usually behaves as an endocrine disruptor in living organisms (Zhu and Zuo 2013). In recent years, an increasing number of new technologies and methods have been studied for removal of bisphenol A since its high resistance to traditional wastewater treatment as a kind of phenolic organic pollutant (Huang et al. 2013; Rodriguez et al. 2010; Hammedi et al. 2015). In this work, bisphenol A was employed as the model pollutant to inspect the oxidation capability of photo-iron(III)–sulfite system. A comparison between iron(II) and iron(III) in this system was firstly studied. Then, effects of sulfite concentration, light intensity and initial concentrations of bisphenol A were investigated. Afterward, free radical quenching experiments were performed to determine the contributions of reactive free radicals in this system. In addition, the initial degradation products of bisphenol A were identified via liquid chromatography coupled with mass spectrometer (LC–MS). The results of this work can provide practical ideas and theoretical basis for new advanced oxidation processes based on sulfate radicals systems containing sulfite salts on the transformation and degradation of bisphenol A and its analog.

Experimental

Photochemical reaction

Light-emitting diode plates that emit ultraviolet A ($\lambda_{\max} = 395$ nm) was used as the light source. All experiments were performed in a 500-mL beaker and in an open environment. A 400 mL solution containing bisphenol A [chemically pure (higher than 95 %)] and iron(III)/iron(II) at the desired concentration were firstly added into the 500-mL beaker and constantly stirred with a polytetrafluoroethylene-coated magnetic stirrer. Then, the pH of the solution was adjusted to the desired value by using dilute NaOH or H_2SO_4. Subsequently, sulfite solution was added, and then, the pH was then quickly readjusted to the desired value (±0.05). Purified water with 18 MΩ cm resistivity was used throughout this work. At specific time intervals, a sample aliquot of 5 mL was removed, and then, 1.5 mL of ethanol was added to terminate the reaction.

Analysis

The analysis of bisphenol A concentration was performed using an LC-10A high-performance liquid chromatography (Shimadzu, Japan) equipped with a Shimadzu VP-ODS-C18 chromatographic column (25 cm × 4.6 mm, 5 μm) fixed at 28 °C for column temperature. Samples (20 μL) were injected using a mobile phase containing water–methanol (30/70, v/v) at a flow rate of 0.8 mL min^{-1}. The detection wavelength of bisphenol A was set at 278 nm.

To understand the reaction mechanisms better, we determined the initial oxidation products of bisphenol A after 30 min of reaction. The pH value of the reaction solution was adjusted to pH 2 to stop the reaction since it is hard to form iron(III)–sulfite complex and the oxidation rate of S(IV) induced by iron(III) at pH 2 was about 100-fold lower as that at pH 3 (Seinfeld and Pandis 1998). Then, the sample was frozen for 12 h and lyophilized for the purpose of pre-concentration. The dried sample was dissolved in aqueous solution before analysis with an Agilent 1100 LC–MS. The separation was performed on a Supelco C18 reverse-phase column (4.6 mm × 250 mm, 5 μm) at an eluent flow of 1 mL min^{-1}. A solution containing 40 % water with 0.01 % acetic acid and 60 % methanol was used as the mobile phase. Product ion scans were obtained in negative ion mode over a scan range of 50–500 *m/z*.

Results and discussion

Comparison between iron(II) and iron(III) in photo-iron–sulfite system

The degradation curves in Fig. 1 suggest that the chain reactions are about the same under ultraviolet A irradiation when adding either iron(II) or iron(III), whereas without irradiation using iron(III) is more effective than using iron(II). This probably happens because decomposition of $FeSO_3^+$ complex [reaction (2)] is the rate-determining step in the whole reaction chain (1–8) (Zhou et al. 2014b; Chen et al. 2012). $FeSO_3^+$ complex as the radical precursor can be either formed from iron(II) through reactions (3) and (4) or from iron(III) via reaction (1).

Compared with reaction (2) which is a slow reaction, iron(II) can be oxidized into iron(III) apace under UV irradiation. So, the slight difference between the formation time of $FeSO_3^+$ from iron(II) and from iron(III) can be ignored under irradiation. While without UV irradiation, the difference between from iron(II) and from iron(III) is more obvious.

$$Fe^{3+} + HSO_3^- \rightarrow FeSO_3^+ + H^+ \quad (\log K_1 = 2.45) \tag{1}$$

$$FeSO_3^+ \rightarrow Fe^{2+} + SO_3^{\cdot-} \quad \left(k_2 = 0.19\ \text{s}^{-1}\right) \tag{2}$$

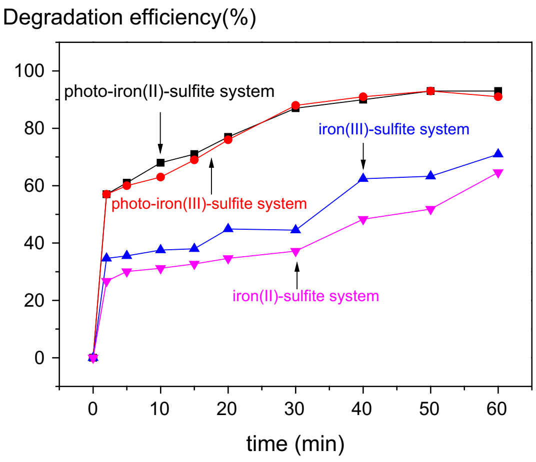


Fig. 1 Degradation of bisphenol A in different iron(III)/(II)–sulfite systems with or without light. Conditions: [bisphenol A]$_0$ = 1 mg L^{-1}, $[Fe_2(SO_4)_3]_0$ = 0.05 mmol L^{-1} or $[FeSO_4]_0$ = 0.1 mmol L^{-1}, $[Na_2SO_3]_0$ = 1 mmol L^{-1}, initial pH = 6. Number of the light-emitting diode plates: 4. Kinetics *curves* show no significantly difference between photo-iron(II)–sulfite system and photo-iron(III)–sulfite system under ultraviolet A irradiation, whereas without irradiation using iron(III)–sulfite system shows a higher degradation efficiency than using iron(II)–sulfite system. Moreover, the curves also reveal a fast stage and a slow stage on the degradation process of bisphenol A

$$Fe^{2+} + HSO_3^- \rightarrow FeHSO_3^+ \quad (\log k_3 = 4) \tag{3}$$

$$FeHSO_3^+ + 1/4O_2 \rightarrow FeSO_3^+ + H_2O \quad \left(k_4 = 1.69 \times 10^3\ \mathrm{L\ mol^{-1}\ s^{-1}}\right) \tag{4}$$

$$SO_3^{\cdot -} + O_2 \rightarrow SO_5^{\cdot -} \quad \left(k_5 = (1{-}2.3) \times 10^9\ \mathrm{L\ mol^{-1}\ s^{-1}}\right) \tag{5}$$

$$SO_5^{\cdot -} + HSO_3^- \rightarrow SO_3^{\cdot -} + HSO_5^- \quad \left(k_6 \le 3 \times 10^5\ \mathrm{L\ mol^{-1}\ s^{-1}}\right) \tag{6}$$

$$Fe^{2+} + HSO_5^- \rightarrow SO_4^{\cdot -} + Fe^{3+} + OH^- \quad \left(k_7 = 10^4{-}10^7\ \mathrm{L\ mol^{-1}\ s^{-1}}\right) \tag{7}$$

$$SO_5^{\cdot -} + HSO_3^- \rightarrow SO_4^{2-} + SO_4^{\cdot -} + H^+ \quad \left(k_8 = \sim 1.2 \times 10^4\ \mathrm{L\ mol^{-1}\ s^{-1}}\right) \tag{8}$$

The results in Fig. 1 also indicated a two-staged kinetics curves, including a fast stage which could degrade about 60 % of bisphenol A in 2 min and a slow one afterward. This phenomenon was also observed during the decolorization of azo dye Orange II in photo-iron(II)–sulfite systems (Zhang et al. 2013), which is believed to be caused by the quick consumption of dissolved oxygen and sulfite (Brandt et al. 1999). In the fast stage, the concentration of dissolved oxygen and sulfite was relatively abundant which leads to produce more reactive sulfur species. Afterward, the dissolved oxygen and sulfite was dropped sharply, and the concentrations of reactive sulfur species decreased which retarded the degradation of bisphenol A.

Effect of initial concentration of sulfite

In fact, Fig. 2 reveals that the degradation efficiency of bisphenol A in photo-iron(III)–sulfite system with 0.2 or 3 mmol L^{-1} of sulfite ions was lower than that with 1.0 mmol L^{-1} sulfite ions. Small dosage of sulfite ions (e.g., 0.2 mmol L^{-1}) cannot produce enough free radicals for bisphenol A degradation. Yet, when excessive sulfite ion was added, the reaction of HSO_3^- with $SO_4^{\cdot -}$ or HSO_5^- [reactions (9) and (10)] will reduce the concentrations of $SO_4^{\cdot -}$. Hence, the optimal dosage ratio of iron(III) to sulfite ion is between 1:15 and 1:10. In subsequent experiments, the ratio of iron(III)/sulfite 1:10 was used when considering the degradation efficiency of bisphenol A.

$$SO_4^{\cdot -} + HSO_3^- \rightarrow HSO_4^- + SO_3^{\cdot -} \tag{9}$$

(Wine et al. 1989)

$$HSO_5^- + HSO_3^- \rightarrow 2SO_4^{2-} + H_2O \tag{10}$$

(Zhang et al. 2000)

Effect of light intensity

Results in Fig. 3 show that higher light intensities led to higher efficiencies of bisphenol A degradation, but bisphenol A degradation rates in slow stage under different numbers of light-emitting diode plate were similar. This enhancement on bisphenol A degradation can be attributed to the reinforced decomposition of $FeSO_3^+$ complex under light-emitting diode irradiation as shown in reaction (2),

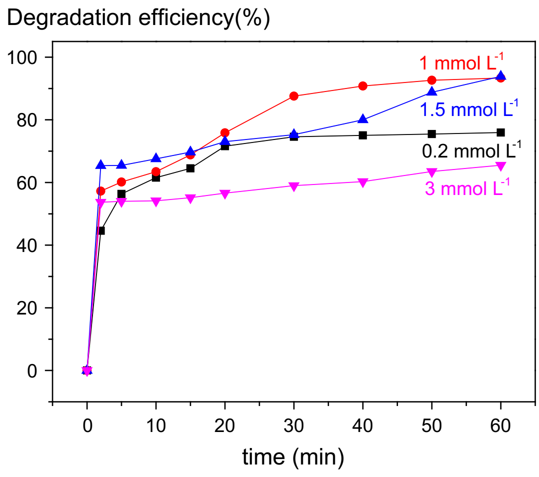


Fig. 2 Effect of initial concentration of sulfite ion on the efficiency of bisphenol A degradation in the photo-iron(III)–sulfite system. Conditions: [bisphenol A]$_0$ = 1 mg L^{-1}, $[Fe_2(SO_4)_3]_0$ = 0.05 mmol L^{-1}, $[Na_2SO_3]_0$ = 0.2–3 mmol L^{-1}, initial pH = 6. Number of light-emitting diode plates: 4. The *curves* show that the excess or lack of sulfite ion will reduce the degradation efficiency of bisphenol A. The optimal concentration of sulfite is 1 mmol L^{-1}

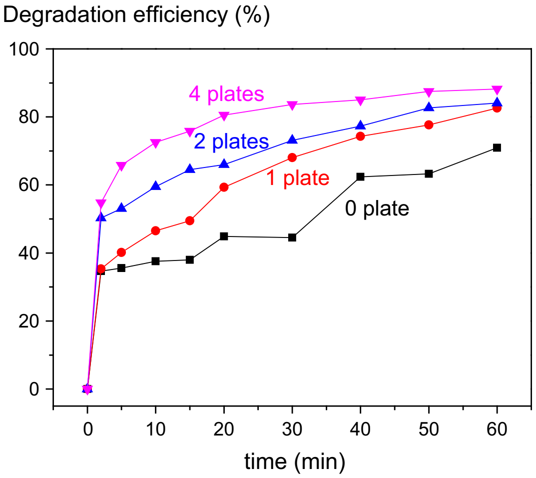


Fig. 3 Effect of different light intensities on the efficiency of bisphenol A degradation in the photo-iron(III)–sulfite system. Conditions: [bisphenol A]$_0$ = 1 mg L^{-1}, [$Fe_2(SO_4)_3$]$_0$ = 0.05 mmol L^{-1}, [Na_2SO_3]$_0$ = 1 mmol L^{-1}, initial pH = 6. The number of light-emitting diode plates varied as 0, 1, 2 and 4. The efficiency of bisphenol A degradation increased as the light intensity ascended from 0 (dark) to 32 W (4 light-emitting diode plates)

which leads to the higher concentration of oxysulfur radicals generated. Therefore, the effect of irradiation mainly occurs in the fast stage of the reaction. However, $SO_4^{\cdot-}$ generated involves dissolved oxygen, and the dissolved oxygen depleted sharply in the fast stage. Although the dissolved oxygen can recover slowly from the air with the reaction proceeding, the sulfite depletion still restrained the reaction. Therefore, due to the limitation of low concentrations of dissolved oxygen and sulfite, the effect of irradiation in the slow stage is not as significant as that in the fast stage of the reaction. But irradiation is still a simple method to enhance the efficiency of bisphenol A degradation by using more light-emitting diode irradiation, although the light-emitting diode light at the wavelength of 395 nm is very week compared with those lamps of higher energy like xenon lamp or high-pressure mercury lamp which have been used extensively. Furthermore, sunlight is believed to be effective in this photo-iron(III)–sulfite system as well.

Identification of radicals

The photo-iron–sulfite system involves free oxysulfur radicals including $SO_4^{\cdot-}$, $SO_3^{\cdot-}$ and $SO_5^{\cdot-}$ described via reactions (1–8). $HO^{\cdot}$ could be generated from $SO_4^{\cdot-}$ via reactions (11) and (12), which might have contributed to the degradation of bisphenol A. To better understand the reaction mechanisms, we identified the main reactive free radicals. Ethanol and *tert*-butyl alcohol were employed as the radical scavenger. The rate constant for the reaction between ethanol and $HO^{\cdot}$ is (1.2–2.8) × 10^9 L mol^{-1} s^{-1}, which is about 50 times higher than that for the reaction between ethanol and $SO_4^{\cdot-}$, which is (1.6–7.7) × 10^7 L mol^{-1} s^{-1} (McLachlan et al. 1996). Thus, ethanol in sufficient amount can capture both $HO^{\cdot}$ and $SO_4^{\cdot-}$. While the rate constant for the reaction between *tert*-butyl alcohol and $HO^{\cdot}$ is in fact (3.8–7.6) × 10^8 L mol^{-1} s^{-1}, which is 1000 times higher than that for reaction between *tert*-butyl alcohol and $SO_4^{\cdot-}$, which is (4–9.1) × 10^5 L mol^{-1} s^{-1} (Anipsitakis and Dionysiou 2004). The scavenger experiments were undergoing with 500 mmol L^{-1} of ethanol or *tert*-butyl alcohol. In the absence of scavenger, bisphenol A degradation efficiency reached 89.9 %, while in the presence of 500 mmol L^{-1} ethanol, bisphenol A degradation efficiency decreased to 33.5 %, which means that this 33.5 % degradation was contributed to the $SO_5^{\cdot-}$ or $SO_3^{\cdot-}$ since the rate constant between $SO_5^{\cdot-}$/$SO_3^{\cdot-}$ and ethanol are very low ($k \leq 10^3$ L mol^{-1} s^{-1}) (Hayon et al. 1972). In addition, $SO_3^{\cdot-}$ tends to be oxidized by dissolved oxygen to $SO_5^{\cdot-}$ very fast. Therefore, $SO_5^{\cdot-}$ caused the 33.5 % of bisphenol A degradation when the concentration of dissolved oxygen was higher than bisphenol A. Moreover, 76.4 % of bisphenol A were degraded with 500 mmol L^{-1} of *tert*-butyl alcohol added in the mixture showing that about 42.9 % of the degradation were attributed to $SO_4^{\cdot-}$ and that $HO^{\cdot}$ was responsible for about 13.5 % of the degradation.

So, in such system we can estimate that three radicals, $SO_4^{\cdot-}$, $SO_5^{\cdot-}$ and $HO^{\cdot}$, are involved in the organic compounds oxidation, contributed to about 47.7, 37.3 and 15 %, respectively, to the total degradation efficiency of bisphenol A (89.9 %). pH higher than 7:

$$SO_4^{\cdot-} + HO^- \rightarrow SO_4^{2-} + HO^{\cdot} \quad \left(k_{11} = 4.6 - 9.1 \times 10^7 \text{ L mol}^{-1} \text{ s}^{-1}\right) \tag{11}$$

(Liang and Su 2009)

All pH values:

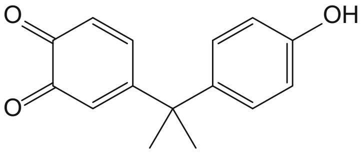
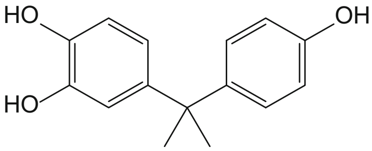
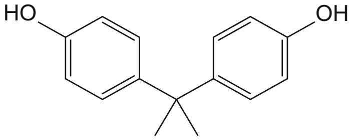
$$SO_4^{\cdot-} + H_2O \rightarrow SO_4^{2-} + HO^{\cdot} + H^+ \quad \left(k_{12} = 10^3 - 10^4 \text{s}^{-1}\right) \tag{12}$$

(Neta et al. 1988)

Initial products

Through LC–MS, three chemicals were identified including bisphenol A, bisphenol A-*o*-catechol and bisphenol A-*o*-quinone as shown in Table 1, in which the latter two were considered as the initial degradation products of bisphenol A. Both products of catechol and quinone derivatives could be formed by either $HO^{\cdot}$ or $SO_4^{\cdot-}$. For $HO^{\cdot}$, bisphenol A molecule was attacked through hydroxyl addition which is subsequently oxidized by O_2 to generate

Table 1 Major chemicals identified by liquid chromatograph coupled with mass spectrometer

Retention time (min)	Molecular weight	Name	Structure
6.4	242	Bisphenol A-*o*-quinone	
7.6	244	Bisphenol A-*o*-catechol	
10.9	228	Bisphenol A	

bisphenol A-*o*-catechol. Thereafter, H atoms on the hydroxyl group of catechol are extracted by $HO^{\cdot}$ and produce bisphenol A-*o*-quinone (Sajiki and Yonekubo 2003; Zhou et al. 2004). While the pathway of $SO_4^{\cdot -}$ oxidation is different from $HO^{\cdot}$, $SO_4^{\cdot -}$ acted as an electrophile abstracts an electron from one of the aromatic rings and forms positively charged aromatic intermediates. Then, the latter intermediate can transform to catechol derivatives (bisphenol A-*o*-catechol), via nucleophilic addition of $HO^{\cdot}$, or via nucleophilic addition of H_2O and subsequent substitution and deprotonation. Afterward, the bisphenol A-*o*-catechol can be oxidized to the corresponding quinone (bisphenol A-*o*-quinone) (Jiang et al. 2013). Although very few reaction of $SO_5^{\cdot -}$ with organic compounds have been reported, $SO_5^{\cdot -}$ has a comparatively higher standard reduction potential $E^0 = 0.95$ V (normal hydrogen electrode, pH 7) or $E^0 = 1.12$ V (normal hydrogen electrode, pH 4) for $SO_5^{\cdot -}/HSO_5^{-}$ (Das et al. 1999), which may act as oxidant for bisphenol A like $SO_4^{\cdot -}$ through hydrogen abstraction and electron transfer (Neta and Huie 1985).

Conclusions

Photo-iron–sulfite system can degrade bisphenol A effectively. Under the same conditions, the iron(III)–sulfite system and iron(II)–sulfite system almost have the same capability of bisphenol A degradation under irradiation. The optimal dosage ratio of iron(III) to sodium sulfite ranged from 1:10 to 1:15, with the degradation efficiency under both ratios being very similar. At specific concentrations of iron(III) and sodium sulfite, the efficiency of bisphenol A degradation was higher at higher light intensity. Radical scavenging tests confirmed that transformation of bisphenol A was caused by $SO_4^{\cdot -}$ (47.7 %), $SO_5^{\cdot -}$ (37.3 %) and $HO^{\cdot}$ (15 %). The initial degradation products were identified as bisphenol A-*o*-catechol and bisphenol A-*o*-quinone. Current work extends the application of photo-iron–sulfite system as a new advanced oxidation processes based on sulfate radicals and can provide practical basis to treat wastewater containing bisphenol A or its analogs.

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