



Degradation of bisphenol A and 4-tert-octylphenol: a comparison between ultrasonic and photocatalytic technique

Pushparaj Pokhalekar, Mousumi Chakraborty*

Department of Chemical Engineering, SV National Institute of Technology, Surat 395007, Gujarat, India,

email: pushparaj.pokhalekar@yahoo.com (P. Pokhalekar), Tel. +91 2612201642; Fax: +91 2612227334; emails: mch@ched.svnit.ac.in, mousumi_chakra@yahoo.com (M. Chakraborty)

Received 24 August 2014; Accepted 21 March 2015

ABSTRACT

Bisphenol A (BPA) and 4-tert-octylphenol (OP) are well-known endocrine disrupting compounds. Ultrasonic and photocatalytic degradation of BPA and OP were carried out by optimizing different parameters such as effects of the pH, ultrasonic frequency, power and the dosing amount of the sodium persulfate (oxidizer)/TiO₂ photocatalyst. The results showed that the maximum ultrasonic degradation of BPA and OP was obtained at a pH value of 5, 45 kHz frequency, 500 W power and with sodium persulfate concentration ranging from 100 to 200 mg/L under bubbling of air, whereas for photocatalytic degradation, maximum degradation was observed at a pH value of 5, and TiO₂ dosing ranging from 50 to 150 mg/L. Photocatalytic degradation was found to be more efficient and cost-effective than ultrasonic degradation.

Keywords: Ultrasonic and photocatalytic degradation; Bisphenol A; 4-tert-Octylphenol; Kinetic study

1. Introduction

Nowadays, advanced oxidation processes (AOPs) are highly efficient methods to remove low to trace amount of organic compounds from both aqueous and gaseous wastes [1]. AOPs are well known for remediation of such contaminated water/effluent without generating any sludge of hazardous character [2]. Generally, AOPs involve generation and utilization of hydroxyl radicals, which are highly reactive in nature having an oxidation potential of 2.33 V and able to oxidize toxic and nonbiodegradable organic compounds in the wastewater [3].

Ultrasonic degradation involves use of ultrasound that generates cavitation which helps in formation,

growth and subsequent collapse of microbubbles [4]. This results in local hot spot having temperature 5,000 K and pressure 1,000 atm, respectively [5–8]. Two main reaction mechanisms have been proposed for ultrasonic degradation. The first is pyrolysis of hydrophobic compound with high volatility at high temperature and pressure inside the bubble, and second is generation and utilization of hydroxyl radical to oxidize the organic matter which is hydrophilic and less or nonvolatile in nature [9].

Photocatalytic degradation is also considered a cleaner and greener technology for the removal of toxic organic and inorganic pollutants from water and wastewater [10]. Generally, photocatalysis involves a photocatalyst (anatase-type TiO₂) in the presence of UV irradiation (usually UV-A with a wavelength 315–400 nm) [11]. The efficacy of photochemical process

*Corresponding author.

depends strongly on the rate of generation of the free radicals along with the extent of contact of the generated radicals with the contaminant molecules.

An endocrine disrupting chemicals (EDCs) are defined as xenobiotic and exogenous substance which can affect adversely on natural action of endocrine system of animals and human such as synthesis, secretion, transport and binding [12]. Among these EDCs Bisphenol A (BPA) and 4-tert-octylphenol (OP) are highly toxic in nature and mainly present in sediments and almost in every water supply, such as surface waters, wastewater and even drinking water up to 670 µg/kg and 0.1 µg/L [13]. BPA and OP are pollutants, which cause not only endocrine disruption but also cancer [14]. Generally, BPA is used for manufacturing of raw materials of epoxy and polycarbonate resins [15,16] and also in the manufacturing of medical device and dental materials [17]. OP belongs to alkylphenol ethoxylates group and is widely used as nonionic surfactants in large variety of industrial and commercial applications [18].

Many researchers have studied on degradation of phenolic compounds as well as BPA [19–24] and OP [25–27] using ultrasonic and photocatalytic degradation technique. However, to the best of our knowledge, comparative study of these two techniques has not been explored much so far. In this context, degradation rates of BPA and OP have been studied in the presence of sodium persulfate/TiO₂ under ultrasonication and UV illumination, respectively. Effects of the pH, ultrasonic frequency, power and the dosing amount of the sodium persulfate (oxidizer)/TiO₂ photocatalyst on degradation rate were also evaluated. Kinetic study was also performed varying operating conditions of the system. Finally, a comparison of energy consumption of these two different techniques was also estimated.

2. Experimental

2.1. Materials

BPA (CAS number 80-05-7), OP (CAS number 140-66-9), sodium persulfate (Na₂S₂O₈) and anatase-type titanium dioxide (TiO₂) were obtained from Sterling Labcare, India, and used without further purification. All other chemicals used were of analytical grade. Stock solution of BPA (100 mg/L) and OP (10 mg/L) was prepared by dissolving it in demineralized water and stored carefully throughout the experiment.

2.2. Experimental procedure for ultrasonic degradation

The sonication process was carried out at low frequency 25–45 kHz and 500 W power supply.

Experiment was conducted at room temperature in 120-ml flat-bottom beaker using a sample volume of 40 mL. US Environmental Protection Agency considers exposures up to 50 µg/kg/d of BPA to be safe, and for alkylphenol, concentration should not exceed 6.6 µg/L in fresh water and 1.7 µg/L in saltwater. Accordingly, feed solution containing low concentration of BPA and OP was taken (2 and 5 mg/L, respectively) for degradation study. A total of 50 mg/L concentration of Na₂S₂O₈ was used in all experiments except the experiment, where effects of concentration of Na₂S₂O₈ on degradation rate were studied. During sonication, BPA and OP concentration was monitored at regular time (1–10 h) interval using UV absorption spectra (HACH, Germany). BPA and OP showed maximum absorption at wavelength 224, 276, 193, 220 and 276 nm, respectively.

2.3. Experimental procedure for photocatalytic degradation

Photodegradation (125W UV) experiment was conducted in glass reactor of capacity 150 mL using a sample volume of 120 mL. The reaction mixture was bubbled continuously at a moderate airflow rate at room temperature. The temperature of reactor was maintained by cooling water to jacketed side of the reactor by submersible pump. Initial concentration of BPA and OP was 2 and 5 mg/L, respectively. A total of 50 mg/L concentration of TiO₂ was used in all experiments except the experiment, where effects of concentration of TiO₂ on degradation rate were studied.

To ensure reproducibility of the results, all experiments were repeated twice under identical experimental conditions and data were found to be reproducible within ±2% variation.

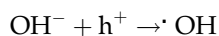
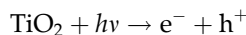
2.4. Chemistry of ultrasonic degradation

When aqueous solutions are irradiated with ultrasound, the H–O bond in water is homolytically cleaved to form hydroxyl radicals and hydrogen atoms. This process is the result of cavitation, whereby very high temperatures and pressures are generated within an imploding bubble. The most common reaction is dimerization of the hydroxyl radical producing hydrogen peroxide and the amount of H₂O₂ generated is often used as a measure of the effectiveness of the ultrasonic process. Compounds may undergo complete mineralization at high temperature and pressure within an imploding bubble.

2.5. Chemistry of photocatalytic degradation

TiO₂ acts as electron traps, enhancing the electron-hole separation and the subsequent transfer of the

trapped electron to the adsorbed O_2 acting as an electron acceptor. After the photogeneration of electrons and holes by photons of appropriate energy, reaction enables the positive photoholes h^+ to react with OH^- adsorbed species to create $\cdot OH$ which is generally assumed to be oxidative agents.



3. Results

3.1. Ultrasonic degradation of BPA and OP

3.1.1. Effect of pH

The effect of pH for BPA and OP degradation was studied between pH ranges of 4–9. Maximum rates of degradation of BPA and OP (~40% within 60 min) were observed at a pH value of 5 for both the substances. It is found that at lower pH, the compounds exist mainly in molecular form, whereas at basic pH, it is completely ionic in nature. Hydrophilic neutral form of the molecules diffuses more readily to the hydrophobic bubble–liquid interface, where the concentration of hydroxyl radical is maximum which degrades the organic compound [19].

3.1.2. Effect of frequency

The experiment was carried out at low frequency ranging from 25 to 45 kHz. It is found from literature that frequency can affect cavitations by modifying bubble number, bubble size, cavitations' threshold and temperature reached during cavitations [20]. The average size and number of cavitations' bubbles increase with increasing frequency of sonication [21]. The maximum degradation (~40% within 60 min) rate was observed at 45 kHz frequency, so this frequency was maintained in all other experiments.

3.1.3. Effect of ultrasonic power

The effect of power has been investigated at different power supply, i.e. from 10 to 100% W of the total power consumption. Maximum degradation rates were observed at 100% power supply, i.e. 500 W power for both BPA and OP. Increased power supply will generate more number of active cavitations bubbles, and implosive energy increases with applied

power supply, leading to an increase in the amount of hydroxyl radical generated [20,28]. The calorimetric efficiency was calculated as 0.32% (the ultrasonic energy dissipated in the reactor was $\approx 2\%$ of the total electrical power input) [29]. The degradation trend of BPA (up to 75 min) and OP (up to 60 min) is shown in Figs. 1 and 2, respectively.

3.1.4. Effect of aeration

The effect of aeration was studied at moderate air-flow rate. It was observed that with aeration, major spectra of BPA increased continuously at 224 and 276 nm wavelengths (Fig. 3). This was because of formation of different intermediates and same trend was also observed by Ioan et al. in sono-Fenton process [21] and Katsumuta et al. in photo-Fenton process [22]. This increase in absorbance remained about 3–4 h and then slowly decreased with time. Continuous decrease in absorbance was observed for OP (Fig. 4) which attributed that there was no intermediate formation and degradation rate increased in the presence of air [23].

3.1.5. Effect of sodium persulfate

Sodium persulfate is a strong oxidizer which oxidizes BPA and forms intermediates in the presence of air easily. It was observed that higher the concentration of sodium persulfate higher the rate of degradation of BPA. The time required to degrade BPA with concentration of $Na_2S_2O_8$ is shown in Tables 1 and 2. BPA intermediates (Table 3) were identified by GC–MS technique. In case of OP, there was no intermediate formation and 100% degradation was observed within

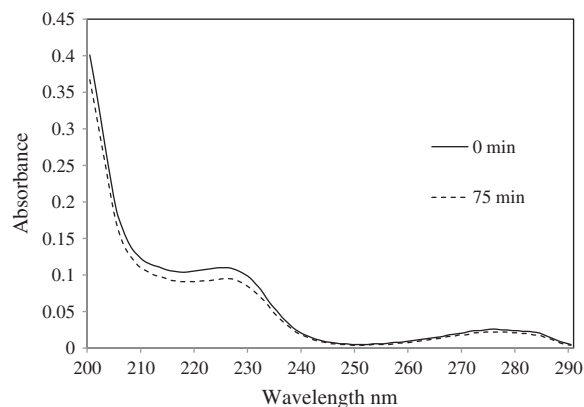


Fig. 1. Effect of ultrasonic power on ultrasonic destruction of BPA at Co: 2 mg/L, ultrasonic frequency: 45 kHz, pH 5, ultrasonic power: 500 W, time: 75 min.

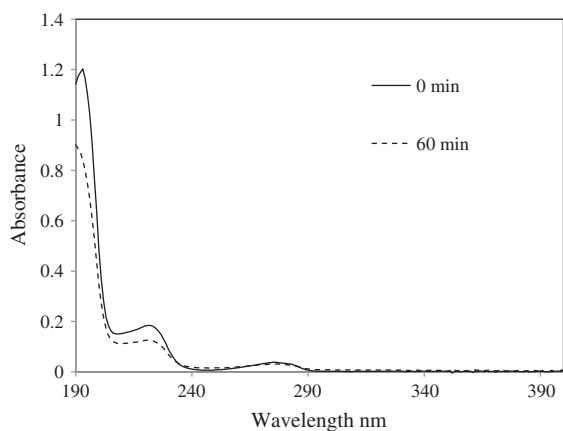


Fig. 2. Effect of ultrasonic power on ultrasonic destruction of OP at Co: 5 mg/L, ultrasonic frequency: 45 kHz, pH 5, ultrasonic power: 500 W, time: 60 min.

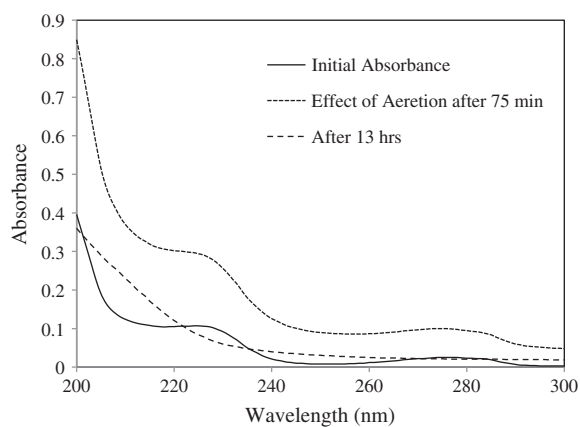


Fig. 3. Effect of aeration on ultrasonic destruction of BPA at Co: 2 mg/L, time: 75 min, pH 5, ultrasonic power: 500 W, ultrasonic frequency: 45 kHz.

2 h at 200 mg/L $\text{Na}_2\text{S}_2\text{O}_8$ concentration (Table 4). The reaction (at very high temperatures and pressures within an imploding bubble) begins with hydroxyl radical attack at the ortho- and para-positions of the aromatic ring followed by further oxidation, ring opening and oxidation to diacids. This diacidic compounds undergo complete mineralization. Also, tertbutyl group of OP promotes oxidation at high temperature and pressure via formation of tertbutyl alcohol.

3.2. Photocatalytic degradation of BPA and OP

3.2.1. Effect of pH

The effect of pH for BPA and OP photocatalytic degradation was studied between pH ranges of 3–6

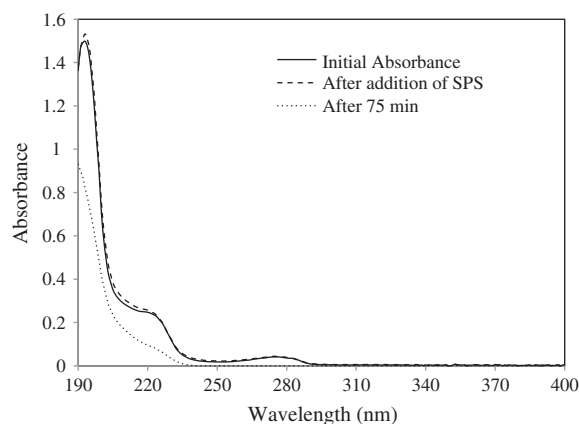


Fig. 4. Effect of aeration on ultrasonic destruction of OP at Co: 5 mg/L, time: 60 min, pH 5, ultrasonic power: 500 W, ultrasonic frequency: 45 kHz.

and 5–9, respectively. BPA is a weak organic acid ($\text{p}K_{\text{a}1} = 9.6$ and $\text{p}K_{\text{a}2} = 10.2$) which can dissociate in solution as either an anionic or dianionic species. Generally, when $\text{pH} = \text{p}K_{\text{a}}$, the undissociated and ionic species exist at equal concentration in solution. When $\text{pH} < \text{p}K_{\text{a}}$, the undissociated species is predominant, and when $\text{pH} > \text{p}K_{\text{a}}$, the ionic species is predominant. So under acidic condition ($\text{pH} < 7$), the reaction would be mainly between $\cdot\text{OH}$ and undissociated BPA. At lower pH, higher the adsorption of substrate on TiO_2 surface, higher is the degradation. When the pH increases, the rate of degradation decreases; because the change of surface charge of TiO_2 leads to a supplementary repulsive phenomenon [30]. The maximum degradation ($\sim 60\%$ within 60 min) was observed at pH 5 for both BPA and OP. This enhancement in degradation at relatively lower pH has been attributed to the fact that BPA and OP remain in molecular form at lower pH and degraded by hydroxyl radical generated at bubble-liquid interface [24,25].

3.2.2. Effect of initial concentration of BPA and OP

The BPA and OP degradation was studied at 2–8 and 3–10 mg/L of initial concentration, respectively (Figs. 5 and 6). In both cases, it was observed that higher the concentration, lower the degradation. From the result, it was concluded that there was not enough dosage of TiO_2 (100 mg/L) to provide active radicals for the degradation of OP at higher concentration. Also, higher concentration caused formation of by-products to a greater extent, which might absorb some photons or consumed additional active radicals [31].

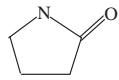
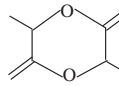
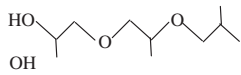
Table 1
Effect of sodium persulfate concentration on ultrasonic destruction of BPA

Sr. No.	pH	Initial BPA concentration (mg/L)	Sodium persulfate concentration (mg/L)	Time required to disappear peaks at 224 and 276 nm (h)	% degradation at different time interval calculated from COD measurement			
					15 min	30 min	45 min	60 min
1	5	2	50	4.5	20	33	37	40
2	5	2	100	2	30	45	63	70
3	5	2	150	1.5	35	52	65	80
4	5	2	200	1	50	63	84	100
5	5	2	250	1	50	73	92	100

Table 2
Effect of initial concentration of BPA on ultrasonic destruction of BPA

Sr. No.	pH	Initial BPA concentration (mg/L)	Sodium persulfate concentration (mg/L)	Time required to disappear at 224 and 276 nm (h)	% degradation at different time interval calculated from COD measurement			
					15 min	30 min	45 min	60 min
1	5	2	200	1	50	63	84	100
2	5	5	200	2	35	48	55	60
3	5	10	200	3	13	22	38	52
4	5	20	200	10	8	16	21	28

Table 3
GC-MS analysis of BPA intermediates (ultrasonic degradation)

Peak No.	Retention time (min)	Molecular weight	Molecular structure
1	5.12	99	
2	7.40	144	
3	13.84	192	

3.2.3. Effect of TiO₂ loading

Plot of $\ln(C/C_0)$ vs. time showed that degradation followed first-order kinetics.

$$\ln(C/C_0) = -kt \quad (1)$$

The photocatalytic degradation of both BPA and OP was carried out at different TiO₂ concentration ranging from 50 to 200 mg/L. From Figs. 7 and 8, it was

observed that 150 mg/L and 100 mg/L TiO₂ concentration was optimum for both BPA and OP degradation as first-order rate constant values were found to be maximum, i.e. 0.0144 and 0.0231 min⁻¹, respectively. The increase in degradation rate with increase in TiO₂ concentration may be explained by the fragmentation of catalyst which produces higher surface area. Thereafter with further increase in catalyst loading to 200 mg/L, the degradation rate starts declining. This is due to the screening effect, that is, above a

Table 4
Effect of Sodium persulfate concentration on ultrasonic destruction of OP

Sr. No.	pH	Initial OP concentration (mg/L)	Sodium persulfate concentration (mg/L)	Time required to disappear peaks at 220 and 276 nm (h)	% degradation at different time interval calculated from COD measurement			
					30 min	60 min	90 min	120 min
1	5	5	50	3.5	33	39	47	55
2	5	5	100	3	38	49	55	60
3	5	5	150	2.5	45	62	75	90
4	5	5	200	2	60	78	86	100
5	5	5	250	2	65	85	93	100

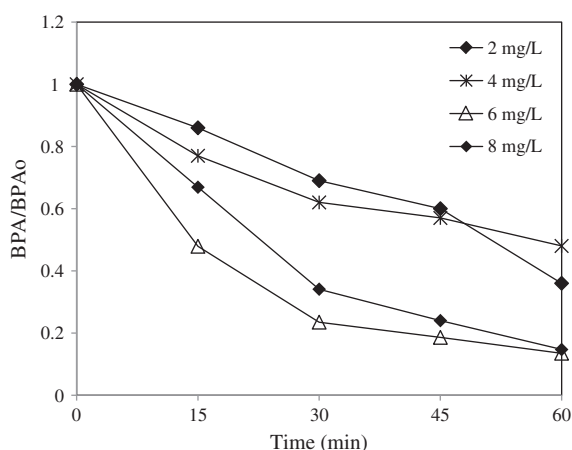


Fig. 5. Effect of initial concentration of BPA on ultrasonic destruction of BPA at ultrasonic frequency: 45 kHz, pH 5; ultrasonic power: 500 W, time: 60 min.

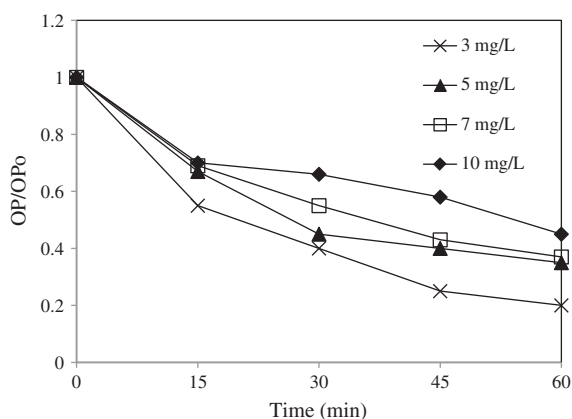


Fig. 6. Effect of initial concentration of OP on ultrasonic destruction of OP at ultrasonic frequency: 45 kHz, pH 5; ultrasonic power: 500 W, time: 60 min.

certain amount of catalyst loading (150/100 mg/L), the turbidity of the solution increases and ultraviolet rays start getting scattered, hence reducing the optical path [26]. Intermediates like hexadecane, eicosane, heptacosane and demeclocycline, tetradecane, heptacosane and hexadecanewere identified by GC–MS for BPA and OP degradation.

4. Energy consumption

For ultrasound degradation, the electrical energy in kilowatt hours (kWh) required to degrade contaminants per unit volume e.g. (1,000 L) [32].

$$E = \frac{P_{elec} \times t \times 1,000}{V \times 60 \times \log(C_{A0}/C_A)} \quad (2)$$

where P_{elec} = total power input to the system (kWh), t = time required to degrade (min), V = volume of

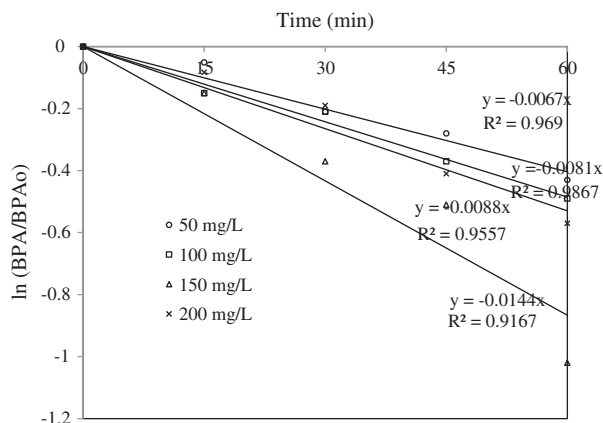


Fig. 7. Kinetic study of photocatalytic degradation of BPA using different concentration of TiO_2 at pH 5, time: 60 min.

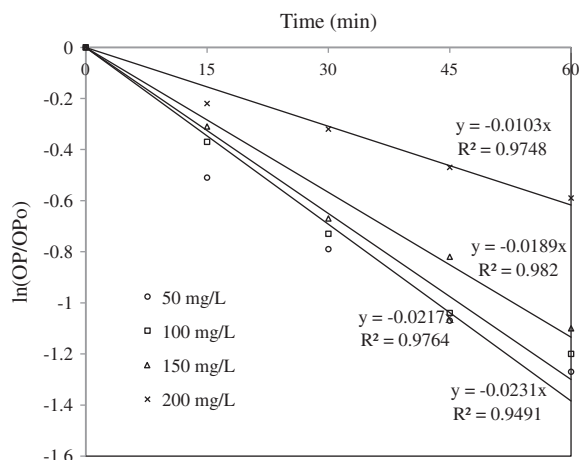


Fig. 8. Kinetic study of photocatalytic degradation of OP using different concentration of TiO_2 at pH 5, time: 60 min.

reaction mixture (L), C_{A0} = initial concentration (mg/L) and C_A = final concentration (mg/L).

For photocatalytic degradation, energy consumption was calculated by this equation

$$\text{Daily consumption (kWh)} = \text{Wattage} \times \text{hours used per day} / 1,000 \quad (3)$$

The evaluation of energy consumption was calculated for 100% degradation. Time taken for 100% degradation was substituted in the above equation to calculate daily consumption of the power.

Cost of ultrasonic degradation for BPA and OP was found to be Rs. 3,744 and Rs. 864, respectively. While for photocatalytic degradation, electrical energy required was directly calculated from power consumption, which was found to be Rs. 288 and 216, respectively, quite cheaper than ultrasonic degradation.

5. Conclusion

This study showed the potentialities of ultrasonic and photocatalytic degradation in water purification. Both the processes were observed to be strongly pH dependent. Efficiency of ultrasonic degradation was found to vary with ultrasonic frequency, power and the dosing amount of the sodium persulfate (oxidizer). On the other hand, efficiency of photocatalytic degradation mainly depend on dosing amount of the TiO_2 photocatalyst and followed first-order kinetics. Hundred per cent degradation of BPA and

OP was observed within 1–2 h at 200 mg/L concentration of $\text{Na}_2\text{S}_2\text{O}_8/\text{TiO}_2$ for sonication and photocatalytic degradation. Comparing energy consumption for both the processes, it was observed that photocatalytic degradation is more economical than ultrasonic degradation.

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