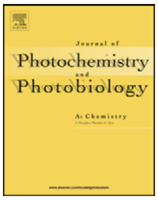
Journal of Photochemistry and Photobiology A: Chemistry 203 (2009) 131–136

Contents lists available at ScienceDirect

Journal of Photochemistry and Photobiology A: Chemistry

journal homepage: www.elsevier.com/locate/jphotochem



Photodegradation of butylparaben in aqueous solutions by 254 nm irradiation

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ARTICLE INFO

Article history:
Received 19 September 2008
Received in revised form 31 December 2008
Accepted 12 January 2009
Available online 21 January 2009

Keywords:
Estrogenic compounds
Butylparaben
Photolysis
Quantum yield

ABSTRACT

Photolytic degradation of the endocrine disruptor butylparaben in aqueous media upon 254 nm irradiation has been investigated. The influence of photon fluence rate, initial butylparaben concentration and pH of reaction solution on reaction rate was studied. The effect of additives: *t*-butanol–hydroxyl radical scavenger, humic acids and nitrate ions—constituents of natural surface water, on reaction course was also determined. Bioassays for toxicity and COD and TOC measurements of photolysed solutions were performed as well. The quantum yield of butylparaben decay in neutral aqueous solution was established to be 0.0033 ± 0.0004.

1. Introduction

Nowadays, the occurrence of endocrine disrupting compounds (EDCs) in the aquatic environment has been recognized as an emerging worldwide issue. Pesticides, some pharmaceuticals, brominated flame retardants, PVC phthalate plasticizers, organotin compounds used as PVC stabilizers and antifouling agents, polycyclic aromatic hydrocarbons, alkylphenols, bisphenol A, and parabens are representatives of the class compounds mimicking or antagonizing hormone functions in aquatic organisms and humans. The potential and real detrimental effects of those anthropogenic compounds on wildlife and humans have been highlighted in several recent papers [1–4]. The continuous discharge of EDCs into the environment results in chronic exposure of aquatic organisms to these compounds and their metabolites, because their natural transformations and removal are compensated by the continuous input into the environment.

Parabens were first introduced in 1930s and currently are the most commonly used preservatives in cosmetics, food commodities, pharmaceutical and industrial products [5]. They are found in creams, pastes, beauty products, glues, fats and oils. Parabens are a family of alkyl esters of 4-hydroxybenzoic acid. Among them methylparaben, ethylparaben, propylparaben, butylparaben and benzylparaben are most often used, which are differentiated from each other by solubility and antimicrobial activity. The average total paraben exposure per individual in the United States is estimated to be approximately 76 mg/day (cosmetics and personal care products 50 mg/day, drugs 25 mg/day and food 1 mg/day) [6]. The broad application of parabens results in their presence in surface water, for example butylparaben was found in UK (South Wales) at concentrations slightly exceeding 50 ng dm^{-3} [7]. The common use of parabens is the result of their inexpensive production, their broad spectrum of activity against yeast, molds, bacteria, and that they have been for a long time considered to be relatively non-toxic to human. However, recent evidence suggests estrogenic activity of parabens [8,9]. Particularly, propylparaben and butylparaben adversely affected the secretion of testosterone and the function of the male reproductive system to rats and mice [10,11]. Additionally Canosa et al. [12] found the formation of halogenated byproducts of parabens in chlorinated water. The major sources of the release of EDCs into aquatic environment surprisingly at a first glance appear to be wastewater treatment plants [7,13]. Generally, EDCs are present in water at low concentration levels which makes their removal in conventional sewage treatment plant difficult and costly. Only modern techniques, like nanofiltration or reverse osmosis, show good enough efficiency in the removal of some of these substances from water stream [14,15].

Ultraviolet light irradiation is an established method of drinking water disinfection and a developing method for wastewater purification. These technologies are mainly accomplished using low-pressure mercury lamps that emit, primarily, monochromatic light at 254 nm. In addition to its disinfection efficiency, UV can also degrade organic compounds by direct photolysis as a consequence of light absorption or by an indirect process assisted by the addition of hydrogen peroxide. In surface water indirect photolysis may also occur by the production of oxidants during irradiation of some components of surface water, e.g. humic substances or nitrate ions. The results of the study on photodegradation of pollutants using

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doi:10.1016/j.jphotochem.2009.01.006

radiation below 300 nm to some extent can be extrapolated to environmental conditions [16]. Particularly important is information on the degradation degree and toxicity of formed photoproducts.

There are few degradation studies of EDCs using UV radiation. Hansen et al. [17] reported degradation of xenoestrogens in tap water and in treated sewage spiked with 17 various EDCs by UV irradiation and more efficient advanced oxidation process (AOP) UV/H_2O_2. The electrical energy necessary for 90% removal of butylparaben in UV photolysis amounts to 28 kWh for 1 m^3 of effluents, while for AOP it is reduced to about 8 kWh m^{-3}. The degradations carried out in tap water assure higher removal degree of almost all investigated EDCs, indicating on inhibiting effect of natural water components on photodegradation.

The influence of environmentally relevant parameters: pH, salinity and addition of humic acids was also studied during 254 nm photolysis of methyltins compounds [18]. The results indicate a strong photolysis inhibition in the presence of sodium chloride and humic acids. The degradation of 17α-ethinylestradiol (EE2) in phosphate buffered pure water and two types of natural waters: after sand filtration and after granular activated carbon filtration using low-pressure (LP) and medium-pressure (MP) mercury lamps was investigated by Canonica et al. [19]. The efficiency of EE2 depletion was higher for MP ($\phi = 0.055$) than for LP ($\phi = 0.026$) source of irradiation. The photodegradation of EE2 was strongly influenced by the type of water. The clear enhancement of the decay in both investigated natural waters was observed which can be ascribed to the presence of various photooxidants in natural waters. The photolysis at 254 nm of bisphenol A in pure water, surface water and wastewater was investigated by Neamţu and Frimmel [20]. The degradation was more efficient in wastewater than in pure water which the authors explain by the potent formation of reactive oxygen species and possible proceeding of Fenton reaction in wastewater. The decay of bisphenol A in solution was also confirmed by disappearance of its estrogenic activity.

The objects of the UV degradation studies were also other endocrine disrupting compounds like antimicrobial agent triclosan [21]; flame retardants brominated diphenyl ethers [22]; antioxidant butylated hydroxyanisole [23].

Our aim was to investigate the photodegradation of butylparaben (BP) in aqueous solution at 254 nm irradiation depending on initial BP concentration, pH and oxygen content in the reaction mixture and on fluence of the irradiation. The effect of radical scavenger on the reaction course was also tested. The obtained results were used in quantum yield estimation. The influence of synthetic natural water on BP photolysis was also studied.

2. Experimental

The experiments were carried out in quartz test tubes of the capacity about 10 cm^3 placed in a merry-go-round at the temperature 25 °C. The source of radiation was two exposure panels equipped with five removable UVC lamps each (Luzchem), located on both sides of the merry-go-round at a distance 15 cm from its center. The optical length in the tubes was equal to 0.85 cm. The lamps emitted above 86% radiation at the wavelength 254 nm. The reaction solution of BP concentration in the range 1×10^{-5} to 2×10^{-4} M was prepared in distilled water treated in Millipore Milli-Q Plus System from stock solution of BP (Fluka) in methanol (p.a., POCH, Poland). An appropriate volume of BP stock solution was introduced into the flask, methanol was evaporated under slight nitrogen stream and buffered water was added. This solution was treated in an ultrasonic bath for 1 h, transferred into the test tubes and sealed with parafilm. The pH of reaction medium was adjusted by a phosphate buffer for pH 5 and 7 and by sodium hydroxide for pH 11 (Na_2HPO_4, KH_2PO_4, NaOH; p.a., POCH, Poland). In some cases the reaction solution was saturated with oxygen or nitrogen to perform experiments in oxygenated and deoxygenated conditions, respectively. Synthetic natural waters were prepared by adding humic acid (Fluka) or KNO_3 (p.a., POCH, Poland) to pure water according to [24]. *Tert*-butanol (p.a., Fluka) was used as a radical scavenger.

The reaction progress was traced by determination of the decrease of BP concentration using a Waters HPLC apparatus with an auto-sampler and UV diode array detector. The chromatograph was equipped with a Nova-Pak 150/C18 column. The mobile phase was a mixture of acetonitrile and acidified water (0.01% H_3PO_4) at the flow rate 1 cm^3 min^{-1}. For the first 12 min of the analysis the eluent consisted of water, over the next 8 min was changed to 70/30 acetonitrile/water (%, v/v) and this composition was maintained for the last 10 min of the analysis. Detection wavelength was 257 nm.

Oxygen concentrations were measured using a Diamond General 733 Clark style microelectrode. The absorption spectra were measured on a Unicam UV 300 spectrophotometer.

The toxicity of BP solutions before and after irradiation was assayed toward *Pseudomonas putida*. The viability of bacteria population after 24 and 48 h of exposure to the tested solutions was determined using the spread-plate technique. The growth of microorganisms was evaluated by Koch's dilution method to establish the number of living cells in the population. The determinations were done in triplicate. The percent of bacterial growth inhibition was calculated from the formula: $I = (\ln B_0 - \ln B_n)/\ln B_0 \times 100\%$, where I is the inhibition, B_0 is the number of cells in 1 cm^3 of the control sample after time t, and B_n is the number of cells in 1 cm^3 of the test sample after time t.

The total organic carbon TOC was measured on a HACH IL 550 TOC-TN apparatus.

The chemical oxygen demand, COD was determined by dichromate standard method using a HACH-LANGE apparatus.

Actinometry experiments were conducted using uranyl oxalate solution [25]. The calculated photonic flux entering reaction space at 254 nm changed from 2.47×10^{18} to 9.27×10^{18} quanta dm^{-3} s^{-1} (4.10×10^{-6} to 1.54×10^{-5} einstein dm^{-3} s^{-1}) depending on the number of working lamps, which corresponds to irradiance rate ranging from 11.8 to 44.4 W m^{-2}.

Data analysis was performed using Origin (Microcal Software Inc., USA). The initial reaction rates were calculated by differentiating exponential curve that fitted experimental points (C, t) at the correlation factor higher than 0.97.

3. Results and discussion

3.1. Preliminary study

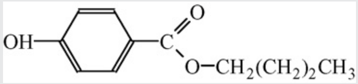
Preliminary investigation included the determination of some physicochemical properties of butylparaben, particularly spectrophotometric ones. Table 1 shows established parameters along with some literature data.

The absorption spectra of BP are pH dependent. A maximum absorption band of undissociated molecule is centered at 256 nm, very close to emission peak of the LP lamp. The solutions of BP at pH about 8 show second band located at 297 nm, which became dominant at pH above 9 (Fig. 1). The determined acid–base equilibrium constant was equal to 8.24 which was in good agreement with the literature value (Table 1).

3.2. Influence of parameters

Changes in acidity and alkalinity of the reaction medium often influence the photoreaction rate due to various electron distributions in the molecule of interest. The photolysis of BP was studied at three pH values: 5, 7 and 11. During the photolysis without a phosphate buffer (natural pH 6.17) the changes of pH were not

Table 1
Physicochemical properties of butylparaben.

Property	Information	References
Molecular weight	194.23 	CAS [94–26–8]
Physical state	White crystalline powder	[26]
Melting point	68–69 °C	[26]
Water solubility	15 mg/100 g at 25 °C; 0.15/100 g at 80 °C 0.77 µM at 25 °C; 7.5 µM at 80 °C	[26]
Soluble in	Chloroform, acetone, alcohol, ether	[26]
Molar decadic absorption coefficient at pH 7 (for anion pH 10)	$\lambda = 254$ nm; $\varepsilon = 15{,}400\ M^{-1}\ cm^{-1}$ $\lambda_{max} = 267$ nm; $\varepsilon = 16{,}080\ M^{-1}\ cm^{-1}$ Anion, $\lambda_{max} = 297$ nm; $\varepsilon = 23{,}300\ M^{-1}\ cm^{-1}$	This work
Acid–base equilibrium constant (OH group)	$pK_a = 8.2$ $pK_a = 8.32$ $pK_a = 8.24 \pm 0.22$	[27] [28] This work
Octanol–water partition coefficient	$\log K_{OW} = 3.57$	[26]

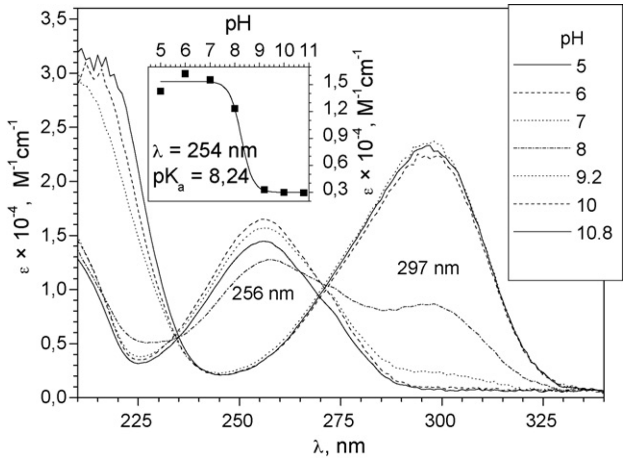


Fig. 1. Absorption spectra of butylparaben: $C_0 = 8 \times 10^{-5}$ M. Inset: determination of acid–base equilibrium constant of BP.

observed, probably due to a too short reaction time. Fig. 2 presents changes in normalized BP concentration during irradiation for various pH. The most rapid BP decomposition occurred at pH 5 and practically at pH 7. The slowest BP decay was observed at pH 11. The main reason is the existence of anion form of BP in the alkaline solution where the absorption peak moved from 256 to 297 nm and the absorption coefficient at wavelength emitted by the LP lamp drastically decreased.

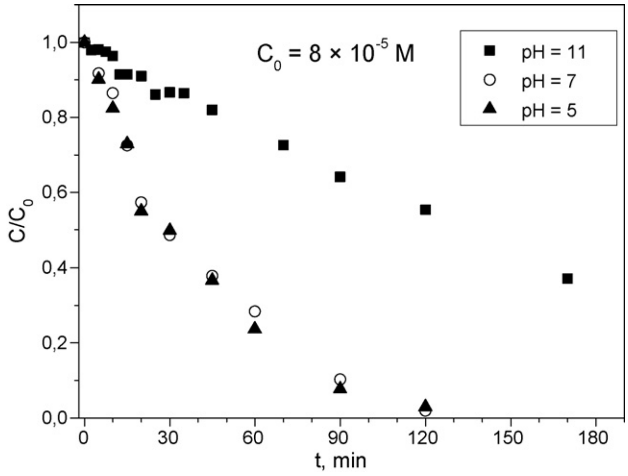


Fig. 2. The effect of pH on BP degradation upon irradiation at 254 nm. $C_0 = 8 \times 10^{-5}$ M and $E_0 = 1 \times 10^{-5}$ einstein $dm^{-3}\ s^{-1}$.

The next series of experiments were aimed at checking the influence of photon fluence rate on the reaction course (Fig. 3a). The photolysis rate of BP carried out with an increasing number of turned on lamps practically linearly increased upon the fluence rate. The 90% reduction of BP concentration for fluence rate above 1×10^{-5} einstein $dm^{-3}\ s^{-1}$ was achieved after above one and a half hour of irradiation. The disinfection dose applied for water was estimated to be about 400 J m^{-2} [19], for BP this value of fluence caused practically any BP degradation (Fig. 3b, upper axis). At the lowest fluence rate the 90% BP concentration reduction was achieved after 263 min which overcomes the disinfection dose by almost 500 times.

The content of oxygen in solution during photolytic degradation of BP has no significant influence on the course of reaction, particularly in the initial period (Fig. 4a). The BP decay rates in aerated and oxygenated solutions were similar. The photolysis of BP solution saturated with nitrogen in the initial period run very close to photolysis of the solutions containing oxygen. Only in the final period of reaction the decay of BP is retarded. After 2 h of photolysis of BP solution in oxygenated conditions the reduction of the compound is 93.5%, in aerobic 88%, while in deoxygenated conditions it is 64.4%. The study of BP photolysis in the presence of hydroxyl radical scavenger *t*-butanol indicated the absence of such radicals in BP degradation. The decay curves of BP concentration were run almost identical without and with 0.1 M of *t*-BuOH (Fig. 4b).

Fig. 5a shows the changes in BP concentration during photolysis for various initial BP concentrations. Interesting is the dependence of initial photolysis rate on initial BP concentration (Fig. 5b).

For lower concentrations, below about $(8–9) \times 10^{-5}$ M, the rate was increased with rising BP concentration, while for a higher initial concentration the reaction rate was decreased. At the higher concentration the molecules of BP are closer to each other which resulted probably in much frequent dissipation of absorbed energy without any chemical changes, which is called concentration quenching [29]. At higher concentration diminishes also the homogeneity of absorption in the reaction space. At BP concentration 8×10^{-5} M almost total absorption (99.9%) occurs in about 1 cm layer, which corresponds to the diameter of reaction test tubes. Hence for BP concentration circa 8×10^{-5} M the highest rate of photolysis is observed. Similar phenomena were observed at higher fluence rate 1.5×10^{-5} einstein $dm^{-3}\ s^{-1}$.

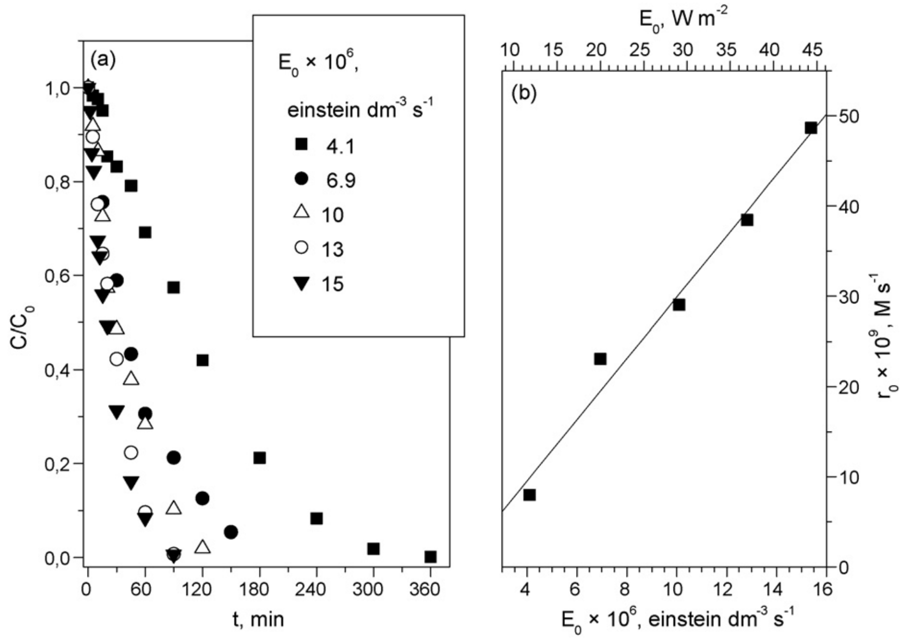


Fig. 3. Disappearance of BP upon irradiation with various photon fluence rates (a). Dependence of BP decay rate on fluence rate (b). pH 7; [BP] = 8 × 10^{-5} M.

Bioassays for toxicity are carried out on living organisms and they can be a basis for the evaluation of real hazards in the environment of chemically treated pollutants. The toxicity of BP aqueous solutions before and after 3 h of irradiation was determined toward bacteria *P. putida* for three pH values 5.3, 7 and 11. Unirradiated BP solutions did not demonstrate toxicity after 24 and 48 h of exposition in accordance with predictions as BP was used as a component of drugs and food commodities. After photolysis, when BP was absent, the solutions were also atoxic toward *P. putida* both at pH 7 and 11. Only for photolysis performed at pH 5.3 the 10% inhibition growth of *P. putida* was observed after 24 and 48 h of incubation.

The measurement of chemical oxygen demand (COD) or total organic carbon (TOC) permits to estimate the level of mineralization of degraded compounds. During photolysis a monotonic slow decrease of the TOC was observed. After 3 h of irradiation the highest TOC drop—19% was determined for pH 5, while for pH 11 the TOC decrease was nearly 11%, which is shown in Fig. 6. Similarly, we observed the decrease of COD parameter.

The influence of the presence in reaction mixture of surface water natural components, i.e. humic acids and nitrate ions on BP degradation is shown in Fig. 7. The addition of humic acids caused the decrease of BP decay rate probably due to the competition of humic acids for UV radiation or quenching of excited triplet state of BP [30]. The presence of nitrate ions has no effect on BP decay; so photolysis of NO_3^- leading to hydroxyl radicals formation does not accelerate the decomposition of the target compound [31].

3.3. Quantum yield determination

The attack of photon on BP molecule most probably occurs on carbonyl carbon causing the break of carbon–aromatic ring

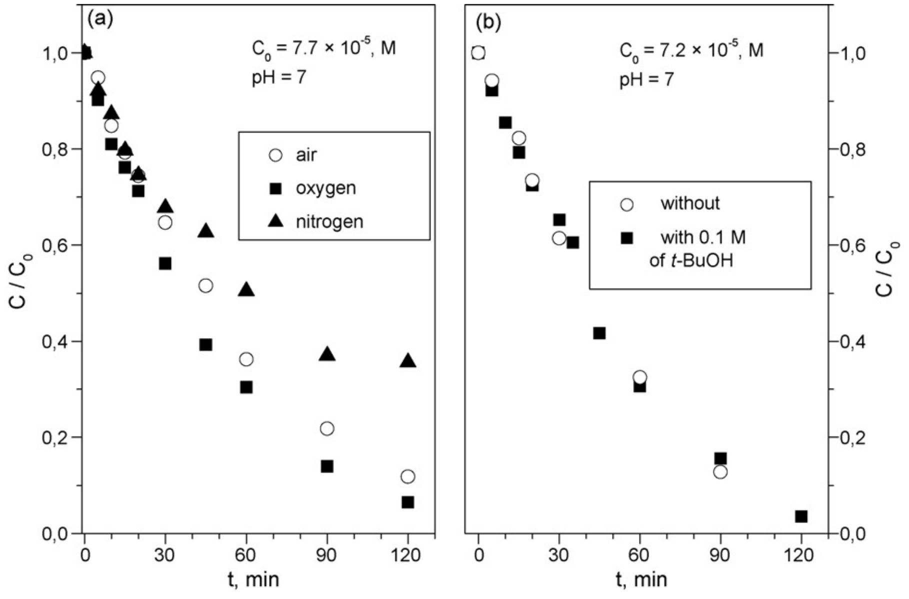


Fig. 4. Oxygen influence on BP degradation (a) and the effect of radical scavenger *t*-butanol on BP decay (b) during irradiation at 254 nm. $E_0 = 1 \times 10^{-5}$ einstein dm^{-3} s^{-1}.

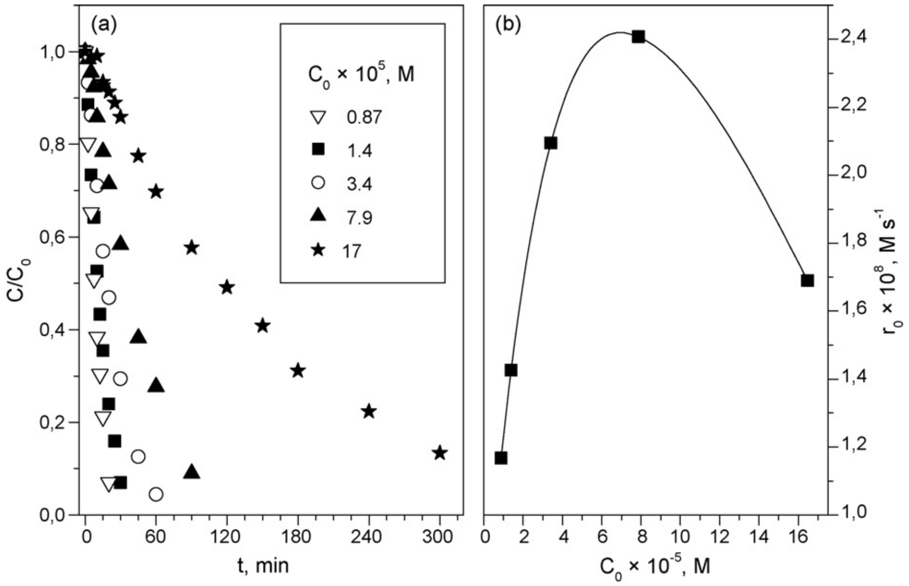


Fig. 5. Degradation of BP during photolysis for various initial BP concentrations (a). Dependence of the reaction rate on BP initial concentration (b). pH 7; $E_0 = 1 \times 10^{-5}$ einstein dm^{-3} s^{-1}.

bond or carbon–oxygen bond which was found in dimethyl- and diethyl-phthalate photolysis [32]. In our study we observed on chromatograms two photoproducts unfortunately unidentified and surely none of the peaks could be ascribed to 4-hydroxybenzoic acid which can exclude the break of oxygen–carbon (*n*-butyl group) bond. The hydrogen abstraction in hydroxyl group with phenoxyl radical formation postulated by Mazellier and Leverd [33] for *t*-octylphenol photolysis seems to be doubtful, at least in the first stage of the reaction. The photolysis of methyl benzoate performed in the gas phase leads to main primary products such as benzaldehyde and formaldehyde [34] which is in agreement with the photolysis of phthalate esters. In any case, the presence of carboxyl and hydroxyl groups attached to aromatic structure makes the mechanism of BP photolysis quite complicated.

The excitation of BP leads to triplet state that can return to ground state or undergo transformation to products, which can be represented by the following reactions:

$$\mathrm{BP} + h\nu \rightarrow \mathrm{BP}^*, \quad k_1 E_\mathrm{a} \tag{1}$$

$$\mathrm{BP}^* \rightarrow \mathrm{BP}, \quad k_2[\mathrm{BP}^*] \tag{2}$$

$$\mathrm{BP}^* \rightarrow \text{products}, \quad k_3[\mathrm{BP}^*] \tag{3}$$

Taking into account the above reactions for the BP decay, the reaction rate is as follows:

$$-\frac{\mathrm{d}[\mathrm{BP}]}{\mathrm{d}t} = k_1 E_\mathrm{a} - k_2[\mathrm{BP}^*] \tag{4}$$

Applying quasi-stationary assumption for the excited BP molecule concentration we obtain:

$$-\frac{\mathrm{d}[\mathrm{BP}]}{\mathrm{d}t} = \frac{k_1 k_2}{k_2 + k_3} E_\mathrm{a} = \phi E_\mathrm{a} \tag{5}$$

where $\phi = k_1 k_2/(k_2 + k_3)$ is the apparent quantum yield of BP decay.

For the initial reaction time, when BP molecule is solely absorbing species, absorbed UV light E_a can be expressed using

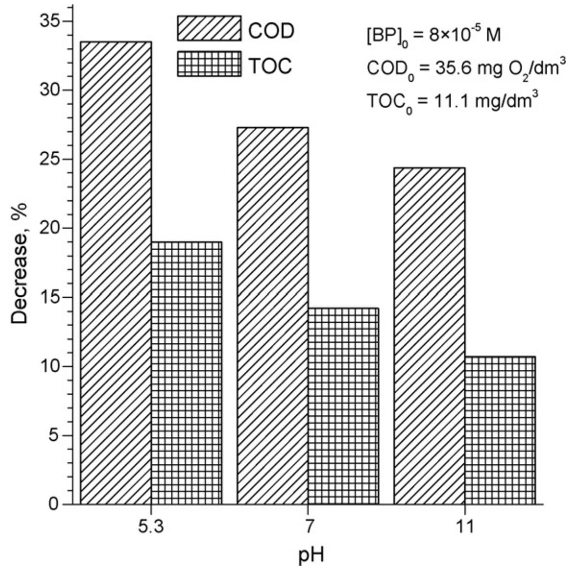


Fig. 6. Decrease of COD and TOC of BP solution after 180 min of irradiation for various pH values. $E_0 = 1 \times 10^{-5}$ einstein dm^{-3} s^{-1}.

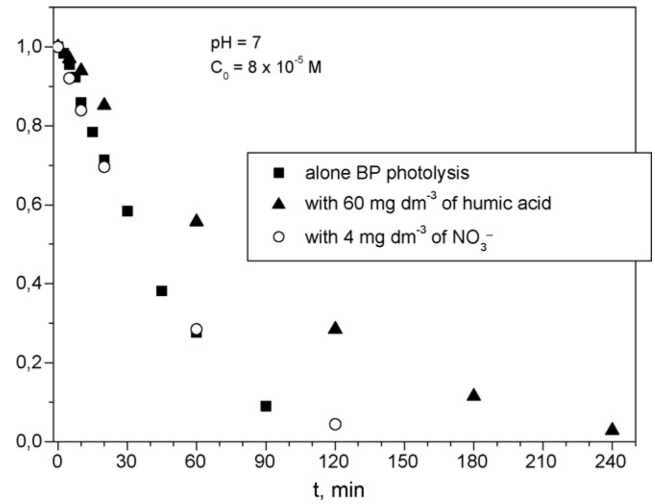


Fig. 7. The course of BP photolysis alone and in the presence of humic acid and potassium nitrate. $E_0 = 1 \times 10^{-5}$ einstein dm^{-3} s^{-1}.

Lambert–Beer law and Eq. (5) becomes:

$$r_0 = -\frac{d[BP]}{dt} = \phi E_0[1 - \exp(-2.3\,\varepsilon b[BP])] \quad (6)$$

Expression (6) was used to estimate an apparent quantum yield of the BP decay. For the studied range of photon fluence rate (see Fig. 3b) and for initial BP concentrations up to 8×10^{-5} M the value of ϕ equal to 0.0033 ± 0.0004 was obtained. In the alkaline solution, where BP anion exists, the quantum yield of the order 6×10^{-4} was determined.

4. Conclusions

The photolysis of BP initiated by the low-pressure mercury lamp (254 nm) occurs very slowly. 99% reduction of BP concentration was attained after 1.5 h of irradiation at irradiance equal to about 44 W m^{-2} (1.5×10^{-5} einstein dm^{-3} s^{-1}). The relationship of photon fluence rate on the reaction rate is rectilinear. The determined quantum yield of BP decay at pH 7 amounts to 3.3×10^{-3}. In alkaline solution, the photolysis of BP anion is about five times slower. The performed COD and TOC measurements indicate also weak mineralization of BP yielded during 254 nm photolysis. The disinfection doses of UV radiation usually applied for drinking water treatment are insufficient for BP decomposition. In our opinion the degradation of BP by photochemical route for practical purposes requires application of more efficient methods like advanced oxidation processes, for example photocatalysis or photolysis in the presence of hydrogen peroxide. The BP decomposition during photolysis proceeds without participation of hydroxyl radical.

The presence of humic acids inhibited reaction rate and the presence of nitrate ions do not affect photolysis. However the influence of some natural components of surface water on photolysis demands further studies. The toxicity tests did not demonstrate the effect of unirradiated and irradiated BP solutions on the viability of bacteria *P. putida*. Only a slight inhibition growth (10%) of bacteria was observed in the solutions of pH 5 after 24 and 48 h of incubation.

Acknowledgement

The study was financed by the Polish Ministry of Science and Higher Education from resources for science in 2006–2009 as the research project no. N207 032 31/1438.

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