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Degradation of bisphenol A by UV/H_2O_2 oxidation in aqueous solution containing nitrate and alkalinity

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ABSTRACT

The goal of the present study was to investigate the effect of nitrate and bicarbonate on the removal of bisphenol A (BPA) by conducting bench-scale UV/H_2O_2 operations under a variety of reaction time and initial concentrations of H_2O_2, NO_3^--N and HCO_3^-. Although 100% removal efficiency of BPA was observed in 2 min in the absence of HCO_3^-, only 76.4 and 67.0% removal was achieved in the presence of 65 and 159 mg L^{-1} HCO_3^-, respectively. In the presence of 5 mg L^{-1} of NO_3^--N and 65 mg L^{-1} of HCO_3^-, the BPA removal efficiency was 87 and 76.5%, respectively. In the presence of both NO_3^- and HCO_3^-, 51.2% of BPA was removed. It was reduced to approximately 50% less than both are absent. The efficiency of BPA removal depends not only on nitrate but also on bicarbonate in aqueous solution. The scavenging effect of bicarbonate was more noticeable than nitrate. The effect was about 33% under same conducted test conditions. The scavenging effect on BPA removal was largest when there were both nitrate and bicarbonate in aqueous solution. The efficiency of BPA was almost halved by them.

Keywords: Bicarbonate; BPA; Hydroxyl radical; Nitrate; Scavenger

1. Introduction

Endocrine disrupting compounds (EDCs) are exogenous environmental chemicals that interfere with normal hormone activities by acting as receptor mimics, agonist/antagonists, altering the synthesis and metabolism of natural hormones or modifying hormone receptor levels [1]. Numerous pharmaceuticals and EDCs are detected in surface water, ground water supplies or sewage effluent worldwide [2]. An example is bisphenol A [2,2-bis(4-hydroxyphenyl)propane; BPA], which is widely used as a raw material for polycarbonate and epoxy resins and is known to accumulate in nature without decomposition causing reproductive damages to a variety of animal species [3]. Because of its wide usage and its endocrine disrupting effects, BPA has been regarded as a representative material among endocrine disruptors and causes reproductive damage to aquatic organisms

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[4–8]. BPA was detected at considerably high levels ranging from 0.44 to 53 nmol L^{-1} in surface waters [9] and 5.7 to 75 mmol L^{-1} in landfill leachates [10]. Persistent EDCs appear to remain for periods ranging from days to months in the surface waters into which they are discharged [11]. Therefore, in the last decade, there is an increasing interest in effective remediation technologies for the destruction of BPA in contaminated water environments [12].

Advanced oxidation processes (AOPs) were developed and used as potentially powerful methods capable of transforming the pollutants into harmless substances [13]. AOPs are based on the generation of very reactive non-selective transient oxidizing species such as the hydroxyl radicals ($^{\cdot}OH$) which were identified as the dominant oxidizing species [14]. For effective oxidations of refractory organic compounds, the hydroxyl radicals must be generated continuously *in situ* through chemical or photochemical reactions due to their instability. Generation of hydroxyl radicals is commonly accelerated by combining some oxidizing agents such as ozone (O_3), hydrogen peroxide (H_2O_2), UV radiation, ferrous and ferric salts (Fe^{2+} and Fe^{3+}) and catalysts such as TiO_2. Among various AOPs, UV/H_2O_2 process is the most extensively investigated. The process is well known for generating hydroxyl radicals by photolyzing the peroxide HO–OH bond with UV light below 300 nm [13].

Hydroxyl radical has a redox potential of 2.85 V, and thus, decomposes all organic matter unselectively with an extremely high second-order reaction rate constant (10^6–10^{10} M^{-1} s^{-1}) is believed to be capable of mineralizing most organic pollutants into carbon dioxide [13]. Hydroxyl radical can oxidize a broad range of organic pollutants quickly and non-selectively [15]. A drawback resulting from such a high degree of reactivity is that the hydroxyl also reacts with “non-target” materials in solution such as nitrate, carbonate, bicarbonate, chloride, and humic substances, all of which are referred to as radical “scavengers”. Hence, the presence of such compounds increases the $^{\cdot}OH$ demand to accomplish a desired degree of organic compound removal, which in turn increases the oxidant consumption rate and the cost of the process.

Wastewaters and treated wastewaters contain a variety of organic and inorganic species that interfere with oxidation reactions of $^{\cdot}OH$ with the target contaminants. Typical concentrations of NO_3^--N are known to range from 2 to 5 mg L^{-1} in biologically well-treated wastewater treatment plant effluents. The addition of HCO_3^- to the coagulation units of wastewater treatment plants is a common practice to stabilize the pH for enhanced treatment performance. Concentrations of HCO_3^- were detected 65–159 mg L^{-1} (as $CaCO_3$) in second treated wastewater treatment plant effluents.

Nitrates and nitrites adsorb UV light in the range of 230–240 nm and 300–310 nm and, consequently, high nitrate (>1 mg L^{-1}) or high nitrite (>1 mg L^{-1}) concentrations have been shown to limit the effectiveness of UV technologies [16,17]. Nitrate photolysis has three distinct mechanisms, which result in the formation of nitrite (NO_2^-), peroxynitrite (ONO_2^- ONO_2^-) and $^{\cdot}NO_2$. One of these mechanisms is the direct route to NO_2^- formation from NO_3^- photolysis, with a low quantum yield [12,13]. The considerable production of NO_2^- can be expected via two other reactions, which produce the intermediates ONO_2^- and $^{\cdot}NO_2$. These intermediates are eventually undergone thermal reactions to NO_2^- form as a stable product [12,14]. The formation of hydroxyl radicals ($^{\cdot}OH$) can also be expected, together with one of the intermediates, $^{\cdot}NO_2$, from nitrate photolysis. As NO_2^- accumulates, it can acts as an $^{\cdot}OH$ scavenger.

Both carbonate and bicarbonate will scavenge hydroxyl radicals to create carbonate radicals which, in turn, react with other organic or inorganic compounds present, albeit at a much slower rate [18,19]. The rate constants, k, for the reactions of the hydroxyl radical with carbonate and bicarbonate are 3.8×10^8 and 8.5×10^6 M^{-1} s^{-1}, respectively [20].

There were very limited numbers of studies, though, in which the effects of nitrate and bicarbonate in an aqueous solution were systematically determined for the UV/H_2O_2 process [15,18,21,22]. In our previous studies [19,20,23,24], effects of nitrate on the UV photolysis were investigated for the degradation of Volatile Organic Compounds (VOCs), 2,4-dichlorophenol, DEP, DEHP, and BPA in aqueous solutions.

The goal of the present study was to investigate the effect of nitrate and bicarbonate on the removal of BPA by conducting bench-scale UV/H_2O_2 operations under a variety of reaction time and initial concentrations of H_2O_2, NO_3^--N and HCO_3^-. Therefore, the results of this study can provide the referable information about the proper application of UV irradiation with hydrogen peroxide for the removal of BPA in aqueous solution containing nitrate and bicarbonate. The identification of the intermediates from the BPA oxidation was not within the scope of this study.

2. Materials and method

2.1. Bench-scale AOP system for UV/H_2O_2 oxidation

All the solvents and reagents used in this study, including bisphenol A (99%, Sigma-Aldrich, USA), hydrogen peroxide (28% w/w, Duksan, Korea), KNO_3

(99%, Samchun, Korea) and $KHCO_3$ (99%, Daejung, Korea), were of analytical grade. All experiments (UV alone, UV/H_2O_2) were carried out in a bench-scale UV/H_2O_2 system (Fig. 1), which consisted of a 28 L raw water tank, a 1 m^3 h^{-1} capacity raw water feed pump, a 5 L H_2O_2 tank and a 18 L UV reactor which were installed and connected to the bench-scale AOP system to study the BPA's oxidation. The reactor and tank are made of a stainless steel (STS316L). Also, they were interconnected with stainless steel pipes. The UV lamp in the reactor (HanSung Ultraviolet Co. LTD), with a nominal power of 65 W, emitted radiation at 254 nm. All headspaces inside the reactors and pipes were completely eliminated during the experiments. All the experiments were performed in the batch mode at room temperature, and the pH levels in the reactors were not controlled.

The aqueous solution (28 L) that was poured into the raw water tank was prepared by dissolving 0.256 g of BPA standard power and 1.01 g of KNO_3 in 28 L double distilled water. To completely mix the solution, the mixture was carefully performed with a stirrer and was circulated in the raw water tank with the use of the installed agitator (IKA-RW 20, IKA-Werke GmbH & Co. KG) for 2 min. Aliquots (799.93 μL) of H_2O_2 were injected before the aqueous solution was circulated to the UV reactor, and the UV reactor was filled with the aqueous solution to prevent any headspace inside it. This aqueous solution was again completely mixed for a period of time circulated in the UV reactor, without UV irradiation. The UV lamp in the reactor was turned on to initiate the oxidation reaction. Samples (2 mL) were regularly collected according to the predetermined schedule.

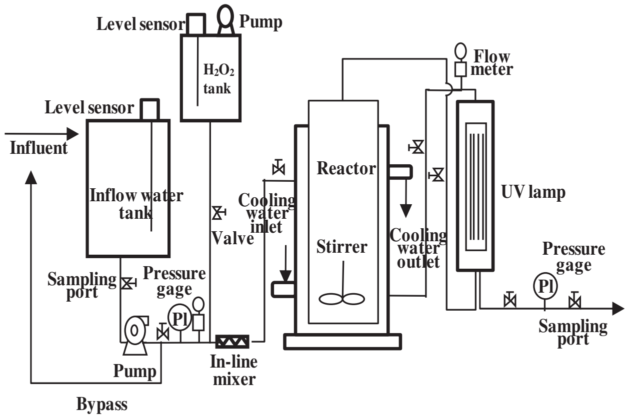

Fig. 1. Schematic diagram of the bench-scale AOP system for UV/H_2O_2 oxidation.

2.2. Analytical methods

BPA was analysed by high-performance liquid chromatography (HPLC) system (Infinity 1290 liquid chromatography, Agilent Technology) with a UV/VIS detector. The HPLC systems equipped with a pump, autosampler, thermostated column compartment and variable wavelength detector. The wavelength used for the HPLC analysis of BPA was 280 nm. Eclipse Plus C18 column (2.1 × 100 mm, 1.8 μm particle size, Agilent Technology) was used for the separation of BPA. Only 100% of acetonitrile was used as a binary mobile phase. The flow rate and the injection volume were 0.3 mL min^{-1} and 1 μL, respectively. Under these conditions, the separation time was less than 2 min. The samples were injected directly into the chromatograph, without any previous sample preparation process.

3. Results and discussion

3.1. Degradation of BPA by UV alone and UV/H_2O_2 processes and impact of nitrate

Fig. 2(a) shows the removal efficiency of BPA as functions of reaction time in the presence of NO_3^- in UV photolysis operations. As the reaction time increased, the removal efficiency of BPA gradually increased in UV photolysis operation. In the UV photolysis without any H_2O_2 addition, the removal efficiency at a reaction of 30 min was about 42.8% with nitrate addition and about 29.6% without nitrate addition, respectively. It is quite interesting to compare the removal efficiencies in the two operations. In the presence of NO_3^-, removal efficiencies in the reactor were higher than those in the reactor where NO_3^- was absent, and these results were observed for the tested operation time. It is possibly due to the oxidation of BPA by hydroxyl radicals. NO_3^- can be attributed to the hydroxyl radical production from the NO_3^- absorption of UV [25], even there is no H_2O_2 dissolved in the solution. The results indicate that NO_3^- has clearly enhanced the removal of BPA through the following nitrate photolysis [26–28]:

$$NO_3^- + hv + H+ \rightarrow {}^{\cdot}NO_2 + {}^{\cdot}OH \qquad (1)$$

Fig. 2(b) shows removal efficiency of BPA as a function of time. In the UV/H_2O_2 reaction, for the first 0.5 min BPA was rapidly removed and complete removal was achieved within 2 min with an initial H_2O_2 concentration of 150 mg L^{-1} and no nitrate addition. Also, BPA was completely removed at a reaction

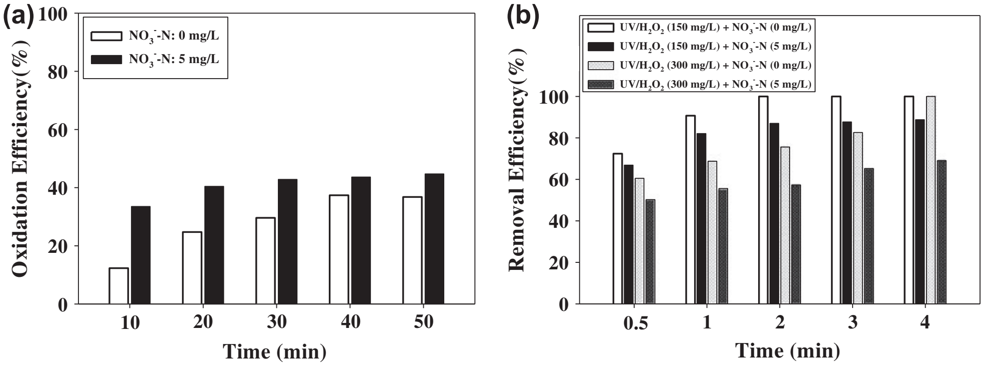

Fig. 2. Profiles of the removal of BPA as a function of time with respect to different initial concentrations of NO_3^--N; initial concentration of BPA: 4×10^{-5} mol L^{-1}; UV intensity: 20 mW cm^{-2}. (a) UV irradiation alone, (b) UV irradiation with initial H_2O_2 concentrations of 150 and 300 mg L^{-1}.

time of 4 min in condition of initial H_2O_2 concentration of 300 mg L^{-1} and in the absence of nitrate. The results indicated that the UV photolysis of H_2O_2 was a more effective method for removing BPA than the direct UV photolysis. These results clearly show that H_2O_2 photolysis provides another source of hydroxyl radicals, as shown in the given chemical reaction [29].

$$H_2O_2 + hv \rightarrow 2{}^{\cdot}OH \qquad (2)$$

However, in presence of 5 mg L^{-1} NO_3^--N, the removal efficiency of BPA was about 87.0% with an initial H_2O_2 concentration of 150 mg L^{-1} and at a reaction time of 2 min and about 69.0% at a reaction time of 4 min with an initial H_2O_2 concentration of 300 mg L^{-1} and NO_3^--N in the UV/H_2O_2 operation. The adverse effect of NO_3^- on the removal of BPA could clearly be shown under the operating conditions. The noticeable scavenging effect of nitrate on the H_2O_2 photolysis of BPA was because the photolysis of NO_3^- produced NO_2^- ions, which were accumulated during the reaction, as mentioned earlier [25,26]. Following reactions explains that due to the OH scavenging effect of NO_2^-, which is generated photochemically, the removal efficiencies of BPA were decreased (see Eqs. (3) and (4)) [29]. NO_3^- was also reported to cause a significant “inner filter” effect that could reduce the fraction of the incident UV absorbed by H_2O_2, which resulted in a significant reduction in the efficiency of the UV/H_2O_2 system [26]. It is very interesting that the longer the reaction time is, the larger the scavenging effect is.

$$NO_3^- + hv \rightarrow NO_2^- + O \qquad (3)$$

$$NO_2^- + {}^{\cdot}OH \rightarrow {}^{\cdot}NO_2 + OH^- \qquad (4)$$

3.2. Effect of initial HCO_3^- concentration on UV/H_2O_2 process to degrade BPA

The effect of bicarbonate on removal of BPA is presented in Fig. 3. The removal efficiencies (%) of BPA as a function of the reaction time were tested in the presence of 0, 65, and 159 mg L^{-1} of HCO_3^-. Although 100% removal efficiency of BPA was observed in 2 min in the absence of HCO_3^-, only 76.4 and 67.0% removal was achieved in the presence of 65 and 159 mg L^{-1} HCO_3^-, respectively, in the same time.

Bicarbonate react readily with the hydroxyl radical to produce the carbonate radical anion, CO_3^- [20,30]

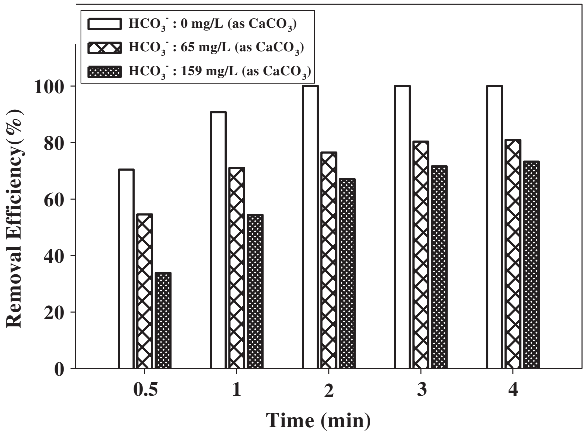

Fig. 3. Profiles of the removal efficiencies (%) of BPA as a function of the reaction time with respect to different initial concentration of HCO_3^- in the bench-scale UV/H_2O_2 operation; initial concentrations of BPA and H_2O_2: 4×10^{-5} mol L^{-1} and 150 mg L^{-1}; UV intensity: 20 mW cm^{-2}. HCO_3^- concentration for each treatment is given in parentheses.

which is also an active species known to participate in aquatic oxidation reactions.

$$HCO_3^- \rightleftarrows H^+ + CO_3^{2-} \tag{5}$$

$$^{\bullet}OH + HCO_3^- \rightarrow {}^{\bullet}CO_3^- + H_2O \tag{6}$$

$$^{\bullet}OH + CO_3^{2-} \rightarrow OH^- + {}^{\bullet}CO_3^- \tag{7}$$

The presence of HCO_3^- in solution also interferes with H_2O_2 for UV light, decreasing the fraction it normally absorbs. This in turns lows down the rate of hydroxyl radical generation [31].

Fig. 4(a) shows the removal efficiency (%) of BPA as functions of initial HCO_3^- concentrations (0, 65, and 159 mg L^{-1}). In the presence of 65 mg L^{-1} HCO_3^-, the removal efficiency of BPA was 47.0, 76.5 and 51.4%, respectively, at initial H_2O_2 concentrations of 100, 150, and 300 mg L^{-1}. However, HCO_3^- concentration increased to 159 mg L^{-1}, only 39.8, 67.0, and 46.5% of BPA were removed, respectively, at the same condition. The removal efficiencies rather linearly decreased, as the HCO_3^- concentration increased due to the scavenging effect of HCO_3^-.

Fig. 4(b) shows the effect of initial H_2O_2 concentration on BPA removal efficiency in 0, 65, and 159 mg L^{-1} of HCO_3^-. The removal efficiencies rather linearly decreased, as the HCO_3^- concentration increased due to the scavenging effect of HCO_3^-. The complete removal of BPA was achieved with initial H_2O_2 concentration of 150 mg L^{-1} in the absence of HCO_3^-. The enhancement of removal by addition of H_2O_2 is due to the increase in the hydroxyl radical concentration. At low H_2O_2 concentration, H_2O_2 cannot generate enough hydroxyl radical and the removal rate is limited [15]. However, the removal efficiency of BPA decreased with increasing initial H_2O_2 concentration of more than 150 mg L^{-1}; the addition of H_2O_2 increased from 150 to 300 mg L^{-1} the removal efficiency was decreased from 100 to 75.6% in the same operating conditions. It was reported that high concentration of H_2O_2 could restrain the reaction [32]. Above the optimum H_2O_2 concentration, H_2O_2 can act as a free radical scavenger [33].

$$H_2O_2 + {}^{\bullet}OH \rightarrow {}^{\bullet}HO_2 + H_2O \tag{8}$$

3.3. *Effect of nitrate and bicarbonate on UV/H_2O_2 process to degrade BPA*

Fig. 5(a) shows the removal efficiencies (%) of BPA as functions of time at the four different conditions in initial H_2O_2 concentration of 150 mg L^{-1}. To observe the inhibition in the presence of scavengers, 5 mg L^{-1} of NO_3^--N, 65 and 159 mg L^{-1} of HCO_3^- were added to the solution, respectively. And it was found that removal efficiency of BPA was 88.8, 81.0, and 73.3%, respectively, in 4 min. It can be explained that scavenging effect of HCO_3^- is higher than NO_3^--N under conducted test conditions.

Fig. 5(b) shows the removal efficiencies (%) of BPA as a function of initial concentration at the four different conditions. When initial H_2O_2 concentration was 150 mg L^{-1}, complete removal efficiency was shown in the absence of both nitrate and bicarbonate. In the presence of 5 mg L^{-1} of NO_3^--N and 65 mg L^{-1} of HCO_3^-, the BPA removal efficiency was 87 and 76.5%, respectively. The figure shows HCO_3^- has more scavenging effect on BPA oxidation than NO_3^--N. In

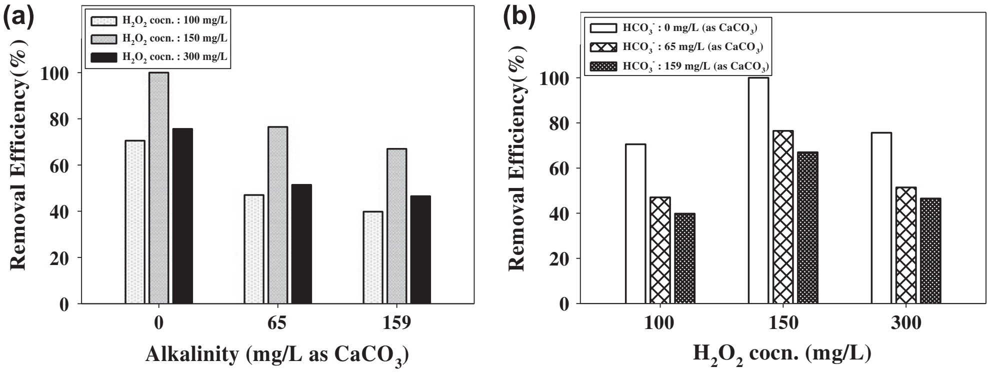

Fig. 4. Removal efficiencies (%) of BPA as functions of: (a) initial HCO_3^- concentrations (0, 65, and 159 mg L^{-1}) and (b) initial H_2O_2 concentrations (100, 150, and 300 mg L^{-1}) in the bench-scale UV/H_2O_2 operation; reaction time: 2 min; initial concentration of BPA: 4 × 10^{-5} mol L^{-1}; UV intensity: 20 mW cm^{-2}.

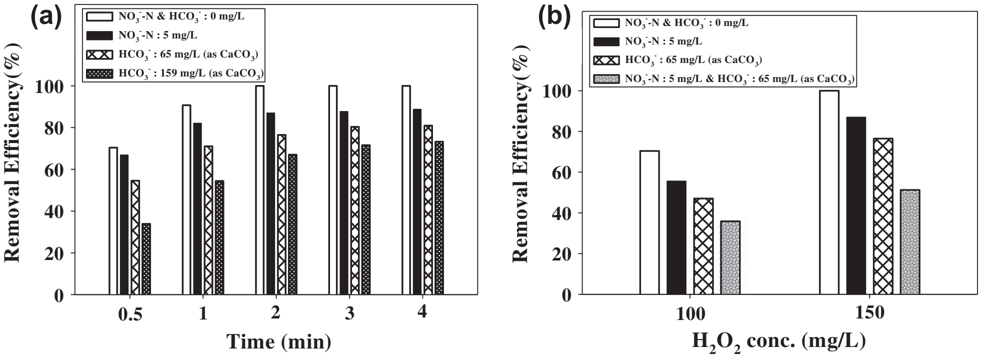

Fig. 5. Removal efficiencies (%) of BPA as functions of (a) time and (b) initial H_2O_2 concentration at the four different conditions in the bench-scale UV/H_2O_2 operation; initial concentration of BPA: 4 × 10^{-5} mol L^{-1}; UV intensity: 20 mW cm^{-2}; (a) initial H_2O_2 concentration of 150 mg L^{-1}; (b) reaction time: 2 min.

the presence of both NO_3^- and HCO_3^-, 51.2% of BPA was removed. The removal efficiency (%) of BPA was decreased about 50% compared with the absence of both. At initial H_2O_2 concentration of 100 mg L^{-1}, it also shows same tendency. It was 70.4% of removal efficiency in absence of both NO_3^- and HCO_3^-, and it was 35.9% when both are present.

The removal efficiencies (%) of BPA of real wastewater effluent as functions of H_2O_2 concentrations of 100, 150, 200, and 300 mg L^{-1} in Fig. 6. It shows that the UV photolysis of H_2O_2 was effective for BPA removal with a H_2O_2 concentration of 150 mg L^{-1} in this experiment conditions. During this experiment, the BPA concentrations of real wastewater effluent ranged from 8 to 10 μ L^{-1} with NO_3^--N concentrations of 3–4 mg L^{-1} and HCO_3^- concentrations of 65–77 mg L^{-1}.

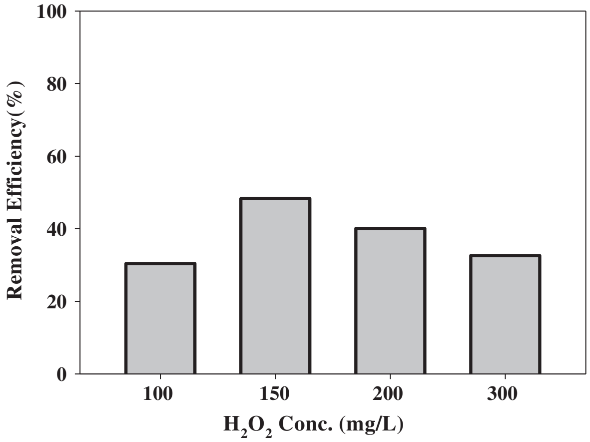

Fig. 6. Removal efficiencies (%) of BPA of real wastewater effluent as functions of initial H_2O_2 concentration in the bench-scale UV/H_2O_2 operation; initial concentration of BPA: 8–10 μ L^{-1}; UV intensity: 20 mW cm^{-2}; NO_3^--N: 3–4 mg L^{-1}; HCO_3^-: 65–77 mg L^{-1}; reaction time: 2 min.

It appears because NO_3^--N and HCO_3^- reacted least as hydroxyl radical scavenger at the H_2O_2 concentrations of 150 mg L^{-1} under tested conditions. The addition of 150–300 mg L^{-1} H_2O_2 decreases the removal efficiency from 48.3 to 32.6% in the same operating conditions. It was reported that high concentration of H_2O_2 could act as a free radical scavenger.

4. Conclusions

In this research, study on degradation of BPA with UV/H_2O_2 oxidation process in synthetic solution containing both nitrate and bicarbonate alkalinity was discussed through the bench-scale experiments. The conclusions obtained are summarized as follows:

(1) UV photolysis combined with H_2O_2 was effective in removing BPA in aqueous solution, while the UV photolysis alone without addition of H_2O_2 was ineffective. BPA could completely be removed with an initial H_2O_2 concentration of 150 mg L^{-1} and 2 min of reaction time in the UV/H_2O_2 process and absence of nitrate.

(2) In the UV/H_2O_2 process with nitrate addition, the scavenging effect of nitrate on BPA removal was about 13% with an initial H_2O_2 concentration of 150 mg L^{-1} and 2 min of reaction time.

(3) The efficiency of BPA removal depends not only on nitrate but also on bicarbonate in aqueous solution. The scavenging effect of bicarbonate was more noticeable than nitrate. The effect was about 33% under same conducted test conditions. The scavenging effect on BPA removal was largest when there were both nitrate and bicarbonate in aqueous solution. The efficiency of BPA was almost halved by them.

(4) The reason might be because both nitrate and bicarbonate hindered the BPA removal by acting as ·OH scavengers under the operating conditions described above.

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