H_2O_2-BASED ADVANCED OXIDATION PROCESSES FOR WATER POLLUTANT DECONTAMINATION. H_2O_2 AS INDICATOR OF THE EFFECTIVENESS OF THE UV/H_2O_2 SYSTEM

Ainhoa Rubio-Clemente[1, 2], Edwin Chica[3], Gustavo A. Peñuela[1]

[1]Grupo de Diagnóstico y Control de la Contaminación (GDCON). Facultad de Ingeniería. Sede de Investigaciones Universitarias (SIU). Universidad de Antioquia UdeA, Calle 70, No. 52-21. Medellín. Colombia

[2]Facultad de Ciencias de la Salud. Universidad Católica de Murcia UCAM, Avenida de los Jerónimos, s/n. Guadalupe-Murcia. España

[3]Departamento de Ingeniería Mecánica, Facultad de Ingeniería, Universidad de Antioquia UdeA, Calle 70, No. 52-21. Medellín. Colombia

Abstract

AOPs based on hydrogen peroxide (H_2O_2) are very efficient in degrading pollutants persistent to conventional water treatments. In order to measure the effectiveness of these alternative technologies, H_2O_2 is regarded as a good indicator. The aim of this work was to determine the oxidizing potential of the UV/H_2O_2 system for removing anthracene (12 µg/L) and benzo[a]pyrene (3 µg/L) in natural water by monitoring the evolution of the H_2O_2 consumed. It was found that the optimal H_2O_2 dose achieving the highest removal of the target pollutants and mineralization of the organic matter was 10 mg/L.

Key words: *water treatment, advanced oxidation processes (AOPs), hydrogen peroxide, UV/H_2O_2 system, contaminants*

1. INTRODUCTION

Water is an essential natural resource for human and living beings. However, it is scarce and highly polluted. In order to solve the problem of water pollution, different treatment processes have been conventionally applied (Malato et al. 2002; Oller et al. 2011; Rubio-Clemente et al. 2014b). Among these techniques, chemical oxidation can be named and hydrogen peroxide (H_2O_2) has been used.

H_2O_2 has an oxidation potential of 1.76 V (Bratsch et al. 1989), able to oxidize a certain amount of pollutants. Nevertheless, it is a selective oxidizing agent and, consequently, some persistent contaminants can escape from H_2O_2 action (Elmorsi et al. 2010; Zuorro et al. 2014).

In this sense, H_2O_2 has been used in combination with ultrasonic waves, electromagnetic radiation, and/or catalysts, such as iron species and semiconductors, like TiO_2, WO_3, ZnO, etc., among other substances, to increase the efficiency of the traditional oxidation systems, leading to the application of the so-called advanced oxidation processes (AOPs). Although the performance of the process depends on the factors influencing each technique, including pH, temperature of the bulk, amount of catalysts and oxidizing agents, as well as frequency and input power in the case of sonochemistry-based processes, to name just a few (Sabate et al. 2001; Neyens & Baeyens 2003; Shemer & Linden 2007; Ghosh et al. 2010; Pontes et al. 2010; Homem & Santos 2011; Rubio-Clemente et al. 2014a, 2014b), these advanced oxidation technologies are characterized by the production of hydroxyl radicals (HO^o).

When H_2O_2 is irradiated with a lamp emitting UV-C light under 300 nm, such as the Hg low pressure germicidal lamps, whose emission wavelength is mainly located at 254 nm, HO^o are produced, as represented by Eq. 1. This process is known as UV/H_2O_2 system (Hofman-Caris & Beerendonk 2011) and H_2O_2 concentration is one of the most influential factors affecting the performance of the oxidation system. In this sense, H_2O_2 level evolution through the treatment time can be used as indicator of the efficiency of the process for treating water polluted with the contaminants of interest. This AOP can also be combined with ultrasound wave effect and the oxidizing action of O_3, obtaining the US/UV/H_2O_2 and UV/H_2O_2/O_3 systems, respectively, which result also in an efficient conversion

of pollutants from water (Fung et al. 2001; Behnajady et al. 2008; Srithep & Phattarapattamawong 2017).

$$H_2O_2 + hv \rightarrow 2HO^o \quad \text{Eq. 1}$$

Additionally, HOº can be cyclically produced by reacting H_2O_2 with iron ions that can be naturally present in water or that can be added to the water of study, as described by Eq. 2 and 3 (Rubio-Clemente et al. 2014a, 2014b, 2015). This reaction is so-called as the Fenton's reaction and the process, Fenton system. In addition, the Fenton's reaction can be accelerated by irradiating the reactants (Eq. 4), by using for example UV-A light, leading to the photo-Fenton process. UV-A radiation can be provided by different kinds of lamps, such as black-light lamps, emitting primarily at 365 nm. Furthermore, the production of HOº by means of the irradiation of Fenton process can take profit from the UV-A portion of the sun radiation spectrum. As deduced from Eq. 4, the number of HOº produced by introducing light in the system is higher than when working in the dark. On the other hand, it is worth noting that a reduced proportion of the HOº yielded can be formed from the photolysis of H_2O_2, although this phenomenon contributes to a lesser extent due to the low molar absorption coefficient of H_2O_2 at wavelengths higher than 300 nm. In Rubio-Clemente et al (2014b), a more detailed information about this and other AOPs can be found.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^o \quad \text{Eq. 2}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + HO_2^o \quad \text{Eq. 3}$$

$$Fe^{3+} + H_2O + hv \rightarrow Fe^{2+} + H^+ + HO^o \quad \text{Eq. 4}$$

HOº is a highly reactive chemical species, which exhibits an oxidation potential about 1.6-folds higher than H_2O_2 (Rubio-Clemente et al. 2014a). In addition, HOº effect is not selective; therefore, a large range of pollutants, both organic and inorganic substances, can be converted to H_2O, CO_2 and inorganic salts. Moreover, HOº has disinfection capacity (Penru et al. 2012; Souza et al. 2013; Zhang et al. 2013).

As previously mentioned, HOº generation can be carried out in various ways. Nonetheless, in spite of a wide number of studies have reported drastic reductions of pollutants from water by implementing the mentioned AOPs (Kavitha & Palanivelu 2004; Elmorsi et al. 2010; Homem & Santos 2011; Rubio Clemente et al. 2013; Zuorro et al. 2014; Rubio-Clemente et al. 2014a, 2014b, 2015), the efficiency of HOº is influenced by the nature and concentration of the contaminant to be degraded and the presence of other organic and inorganic water constituents (Kavitha & Palanivelu 2004; Pignatello et al. 2006; Rubio-Clemente et al. 2014a, 2014b).

Several contaminants can be found in water from low molecular weight compounds to high molecular weight substances, being aromatic compounds like polycyclic aromatic hydrocarbons (PAHs) of great interest. These compounds are composed of two or more fused benzene rings (Chen et al. 2015) that are arranged angular, cluster or linearly (Rubio-Clemente et al. 2014b). In Figure 1, the chemical structure of two representative PAHs can be found.



(a) (b)

Figure 1. Chemical structure of (a) anthracene and (b) benzo[a]pyrene.

PAHs have attracted the attention of different researches in the field of air, soil and water contamination because of their acute toxic, carcinogenic, mutagenic and teratogenic potential (Ukiwe et al. 2013; Rubio-Clemente et al. 2014b; Chen et al. 2015). Polynuclear aromatic substances exhibit different physico-chemical properties; however, in general, they have low water solubility, being detected in aqueous solutions at very low levels, in the range of μg/L or ng/L.

In this work, the efficiency of a H_2O_2-based AOP; particularly, the UV/H_2O_2 system due to the positive results reported for organic compound destruction (Elmorsi et al. 2010; Zuorro et al. 2014; Rubio-Clemente et al. 2014b), applied to the degradation of a mixture of PAHs, containing trace and ultra-trace levels of anthracene (AN) and benzo[a]pyrene (BaP), respectively, as representative PAHs because of the acute phototoxicity of AN and the carcinogenesis, mutagenesis and teratogenesis associated with BaP, utilizing an annular jacketed photo-reactor has been investigated by using H_2O_2 as indicator.

2. MATERIALS AND METHODS

2.1. Chemicals and reactants

All the chemical and reagents were used without previous purification. The target PAHs selected as model micropollutants for the UV/H_2O_2 performance were AN and BaP. These compounds have a purity of 99% (AN) and 96% (BaP), and they were purchased from Alfa Aesar (Ward Hill, MA, USA). Reagent grade H_2O_2 (30%, w/w) from J.T. Baker (ECATEPEC, Estado de Mexico, Mexico) was used as the oxidizing agent. Liquid chromatography analyses were carried out using HPLC-grade acetonitrile (Merck, Darmstadt, Germany) and AN (99%) and BaP (98%) certified analytical standards purchased from Dr. Ehrenstorfer (Augsburg, Germany) to calibrate and evaluate the proper performance of the chromatographic system during the determination of the target analytes. For H_2O_2 analyses, in addition to H_2O_2 (30% w/w), ammonium monovanadate (99.9%) and sulfuric acid (95-97%) also provided by Merck (Darmstadt, Germany) were used. Deionized water obtained by a Millipore system (Bedford, MA, USA) was utilized for preparing all solutions and for liquid chromatography analyses.

2.2. Experimental

The UV/H_2O_2 system was implemented in a multi-lamp jacketed photoreactor, allowing keeping constant the temperature of the bulk. The photoreactor has an effective volume of 2 L and is made of 3.3 borosilicate glass. Further descriptions about the reactor used are detailed in Rubio-Clemente et al (2017b). UV-C radiation was utilized and provided by Hg low pressure lamps (General Electric International Inc., Fairfield, CT, USA). Radiation intensity was measured by a UVX radiometer equipped with a UVX-25 sensor (UVP Inc., Upland, CA, USA) resulting to be 0.17 and 0.46 mW/cm^2 for 1 and 3 lamps, respectively. The action of H_2O_2 in combination with UV-C radiation was studied at three different concentrations (5, 10 and 15 mg/L). Additionally, aliquots were withdrawn at different time intervals during the reaction time. The samples were collected in borosilicate amber glass containers. They were stored in the dark at 4 °C and treated as soon as possible. The experiments were conducted in triplicate.

The natural water used was obtained from a Colombian reservoir (“El Peñol” dam), located in Guatapè (Antioquia), and was spiked with 12 μg/L AN and 3 μg/L BaP due to the target compounds were not initially detected. The water was characterized by its circumneutral pH, reduced organic matter content and low turbidity, allowing the diffusion of photons within the reactor solution, among other physicochemical characteristics.

2.3. Analytical determinations

AN and BaP were measured using a HPLC-FLD system series 1100/1200 (Agilent Technologies Inc., Palo Alto, CA, USA). The operating conditions were described elsewhere (Rubio-Clemente et al. 2017a). In turn, H_2O_2 identification and quantification was carried out with a UV-Vis spectrophotometer Evolution 300 from Thermo Scientific (Waltham, MA, USA). In Rubio-Clemente

et al (2017b), a detailed description of the H_2O_2 analytical procedure is reported. Additionally, the organic matter contained in the sample was measured with a Total Organic Carbon Analyzer TOC–Apollo 9000 from Teledyne Tekmar Instruments (Mason, OH, USA).

Standard stock solutions of H_2O_2, AN and BaP were prepared by diluting suitable amounts of correspondingly H_2O_2, AN and BaP reagents or certificate analytical standards, as appropriate. From the stock solutions, the calibration curves and analytical controls of the methods used for H_2O_2, AN and BaP determination in aqueous solution were constructed and prepared. The curves ranged from 0.1 to 15 mg/L for H_2O_2; from 75 to 3000 ng/L for AN; and from 30 to 3000 ng/L for BaP by using the certificate standards. AN and BaP reagent-grade were used for preparing the working solution of the natural water used.

3. RESULTS AND DISCUSSION

In order to measure the oxidation potential of one of the most efficient H_2O_2-based AOPs, the UV/H_2O_2 system was applied for treating a natural water containing 12 and 3 µg/L of AN and BaP, respectively. The efficiency of the advanced oxidation system was measured by monitoring the evolution of the organic matter of the sample, as well as the target compounds, focusing on H_2O_2 consumption throughout the treatment time. For this purpose, the photoreactor described above was used and 5, 10 and 15 mg/L of H_2O_2 were introduced in the system to act synergically with UV-C radiation. Once the working solution was prepared and homogenized, 3 UV-C lamps, which were previously warmed during 30 min, were turned on and aliquots were withdrawn for analyses.

The evolution of 5, 10 and 15 mg/L H_2O_2 content in the bulk during 90 min of irradiation time is represented in Figure 2.

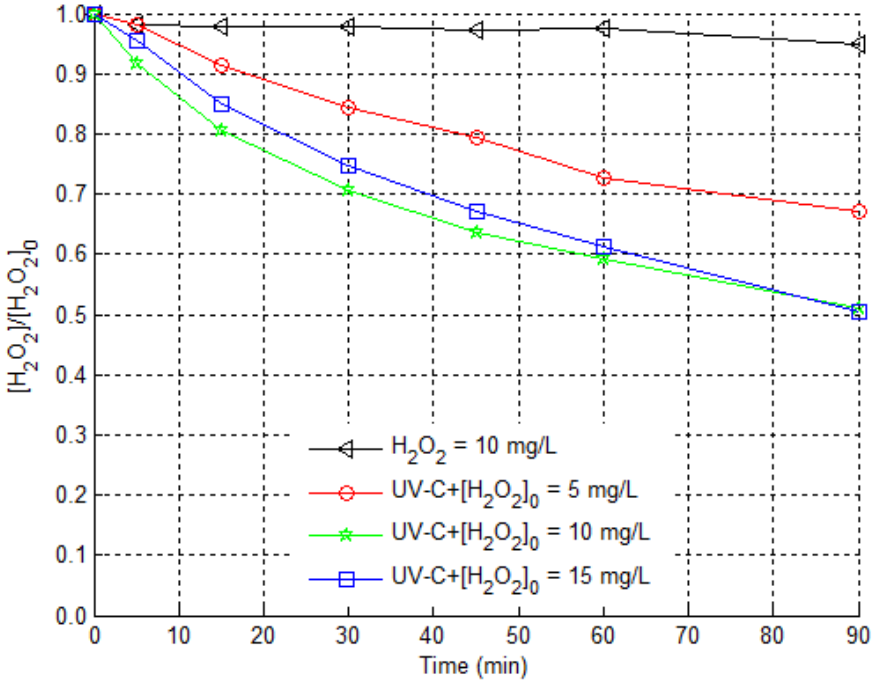


Figure 2. H_2O_2 evolution. $[AN]_0$= 12 µg/L; $[BaP]_0$= 3 µg/L; $[TOC]_0$= 2.04 mg/L; $[H_2O_2]_o$= 5, 10 and 15 mg/L; UV-C radiation= 0.46 mW/cm^2 (corresponding to a light power of 24 W); irradiation time= 90 min.

According to the figure, it can be observed that by working in the dark, the consumption of 10 mg/L H_2O_2 in the system remains practically constant during 90 min of treatment, since about 5% of the initial H_2O_2 was consumed. This can be explained from the low removal capacity of H_2O_2 under thermal conditions, as reported by Elmorsi et al (2010) when studied the degradation of $5x10^{-2}$ mM Mordant red 73 azo dye in water using 2.5 mM H_2O_2 at pH 3 and 25 ºC. Zuorro et al (2014) also found similar results for chloramphenicol in the presence of only H_2O_2.

On the other hand, when the system was irradiated under a UV-C radiation intensity of 0.46 mW/cm^2, corresponding to the use of 3 UV-C lamps (28 W of light power), a higher H_2O_2 decay was observed through the reaction time, resulting the initial dose of 10 and 15 mg/L H_2O_2 in similar evolution-profiles with time, and leading to the highest consumption of the initial H_2O_2 dose in the system. The reduction of H_2O_2 initial content as the oxidation system proceeds could be indicative of the photolysis extent of H_2O_2 by the action of UV-C photon flux reaching the bulk and, consequently, of the production of HO^o which could be reacting with the target pollutants and the natural organic matter of the water producing CO_2, H_2O and harmless inorganic salts. In order to know if the system is gaining oxidation capacity as the H_2O_2 content is being reduced, organic matter in terms of total organic carbon (TOC), was measured. Surprisingly, an increase in the TOC percentage removal was observed from about 14.76 to 45.03% by irradiating 5 and 10 mg/L H_2O_2, respectively. Nevertheless, by using 15 mg/L H_2O_2, TOC elimination was reduced. These contradictory results can be explained by the double role developed by H_2O_2 in the UV/H_2O_2 system. As expected, the TOC conversion extent was increased by raising the dose of H_2O_2 added to the system from 5 to 10 mg/L due to the increase in the number of HO^o within the bulk, which is related to the initial amount of the oxidizing agent, among other operating factors. However, contrary to expectations, a concentration of 15 mg/L H_2O_2 did not result in a higher TOC conversion, likely due to the HO^o scavenging role developed by H_2O_2 when found in excess, as described by Eq. 5. On the other hand, during the scavenging and recombination of HO^o, less reactive radical species, such as hydroperoxyl radicals (HO_2^o) are produced. Additionally, an extreme overdose of H_2O_2 can promote the recombination of the HO^o yielded ascribed to the excessive production of these radicals that do not react with the organic matter of the sample and the target pollutants (Eq. 6). The non-reacting HO^o could also interact with HO_2^o (Eq. 7), decreasing the overall oxidation efficiency of the system.

$$H_2O_2 + HO^o \rightarrow HO_2^o + H_2O \quad \text{Eq. 5}$$

$$HO^o + HO^o \rightarrow H_2O_2 \quad \text{Eq. 6}$$

$$HO^o + HO_2^o \rightarrow H_2O + O_2 \quad \text{Eq. 7}$$

This opposite effect developed by H_2O_2 has been widely reported for both the UV/H_2O_2 system and other AOPs where H_2O_2 is involved (Elmorsi et al. 2010; Homem & Santos 2011; Rubio-Clemente et al. 2014b; Zuorro et al., 2014).

In addition to the organic matter mineralization extent through the UV/H_2O_2 system, AN and BaP removal percentage was studied. In this case, due to the photochemical properties of these PAHs, only the action of 1 UV-C lamp without the presence of H_2O_2 lead to a removal of the target compounds higher than 99% in less than 90 min of treatment. However, intermediate products more toxic that the parent compounds were observed to be formed. By combining 10 mg/L H_2O_2 with 0.46 mW/cm^2 of UV-C radiation intensity no harmful degradation by-products were found.

From these findings, the optimal H_2O_2 dose to be combined with the action of 3 UV-C lamps for the maximal AN, BaP and TOC conversion under the working conditions was 10 mg/L. Additionally, the UV/H_2O_2 system has been proven to exhibit disinfection capacity (Penru et al. 2012; Souza et al. 2013; Zhang et al. 2013). In this sense, the advanced oxidation system tested might be a good candidate to produce drinking water providing the accomplishment of the parameters regulated in terms of water quality.

4. CONCLUSION

Water contamination with pollutants persistent to traditional water treatment processes is a matter of great concern. In this work, the combination of H_2O_2 with UV-C radiation was demonstrated to be very efficient for removing 12 µg/L AN and 3 µg/L BaP in a natural water matrix beyond 99% and organic matter, in terms of TOC, of approx. 45% after 90 min of treatment without the production of toxic reaction intermediates. The optimal H_2O_2 dose to be irradiated with an intensity of 0.46 mW/cm^2, under the studied operating conditions, allowing achieving these promising results was 10 mg/L.

The investigated advanced oxidation system could be a good option for producing water of quality providing the accomplishment of other water quality parameters established in the regulations.

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