STRATEGIES FOR HYDROGEN PEROXIDE DOSING BASED ON DISSOLVED OXYGEN CONCENTRATION FOR SOLAR PHOTO-FENTON TREATMENT OF COMPLEX WASTEWATER

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ABSTRACT

Hydrogen peroxide consumption is known to be one of the key parameters of the photo-Fenton process responsible for its high operating costs. The purpose of this study was to find out whether adding dissolved oxygen to the reaction medium in a continuous stream of pure oxygen or air with different H$_2$O$_2$ dosage strategies could improve the Dorfman mechanism, generating secondary oxidation species that could boost mineralization, and consequently, diminish H$_2$O$_2$ consumption. Experiments confirmed less efficient COD elimination. Additional DO slightly increased H$_2$O$_2$ consumption, as it initiated a series of reactions generating hydroxyl radical scavengers and H$_2$O$_2$ self-decomposition slowing down the reaction. At the same time, the study highlighted that automatic H$_2$O$_2$ dosing based on DO setpoints would be a highly attractive control option, however, more detailed studies on the initial amount of hydrogen peroxide and dosing rates must be undertaken.

Keywords: Automatic H$_2$O$_2$ dosing, Dissolved oxygen, Injection of pure oxygen or air, Wastewater decontamination

1. Introduction

The potential of the photo-Fenton process as an alternative technology for the treatment of wastewater containing organic pollutants not removable by conventional techniques (due to their high chemical stability, low biodegradability, or high toxicity) is widely recognized (Pignatello et al., 2006). However, its main drawback is its operating costs. In recent years, research has focused on making this advanced oxidation process less-expensive, for example, by using solar radiation instead of UV lamps as a photon source. In addition, a detailed economic evaluation of the photo-Fenton process pointed out that optimizing the amount of reagent required is essential to lower the final cost (Sánchez Pérez et al., 2013). The main reagents affecting operating cost are hydrogen peroxide and acid. Assuming that for highly polluted wastewater the process is carried out under optimal pH (around pH = 2.8) and addition of acid is unavoidable, the role of H$_2$O$_2$ dosing becomes essential to improve the treatment’s economic competitiveness. On one hand, hydrogen peroxide concentration is directly related to the amount of hydroxyl radicals generated, and on the other, hydrogen peroxide competes with pollutants for the

hydroxyl radical. Therefore, the amount of hydrogen peroxide added should be a balance between high enough mineralization rates (efficient hydroxyl radical reactions) and moderate hydroxyl radical scavenging.

The photo-Fenton process has been described by several authors as a reaction system including over 30 well-known reactions (Pignatello et al., 2006; Burns et al., 2010). Direct oxidation of Fe(II) by dioxygen is exceedingly slow at pH levels where Fe (III) is soluble and therefore does not contribute to Fenton of photo-Fenton reactions below pH 4. The most important of these reactions when hydrogen peroxide and oxygen are in solution can be summarized as follows:

\[
\begin{align*}
\text{Fe(II)} + \text{H}_2\text{O}_2 &\rightarrow \text{Fe(III)} + \text{HO}^* + \text{HO}^- & k=53-76 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 1} \\
\text{HO}^* + \text{R} &\rightarrow \text{R}^* & & \text{Reac. 2} \\
\text{R}^* + \text{O}_2 &\rightarrow \text{ROO}^* & k=10^8 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 3} \\
\text{ROO}^* + \text{RH} &\rightarrow \text{ROOH} + \text{R}^* & & \text{Reac. 4} \\
\text{ROO}^* + \text{H}_2\text{O} &\rightarrow \text{ROH} + \text{HO}_2^- & \text{pK}_a=4.8 & \text{Reac. 5} \\
\text{HO}_2^- + \text{H}_2\text{O} &\rightarrow \text{HO}^* + \text{H}_2\text{O}_2 & k=3 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 6} \\
\text{Fe(III)} + \text{H}_2\text{O}_2 &\rightarrow \text{Fe(II)} + \text{HO}_2^* + \text{H}^+ & k=9.1 \times 10^7 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 7} \\
\text{Fe(III)} + \text{HO}_2^* + \text{H}^+ &\rightarrow \text{Fe(II)} + \text{O}_2 + \text{H}_2\text{O} & k<2 \times 10^3 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 8} \\
\text{Fe(II)} + \text{HO}_2^* + \text{H}^+ &\rightarrow \text{Fe(III)} + \text{H}_2\text{O}_2 & k=1.2 \times 10^6 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 9} \\
\text{HO}_2^- + \text{H}_2\text{O} &\rightarrow \text{H}_2\text{O} + \text{HO}_2 & k=4.7 \times 10^7 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 10} \\
2\text{H}_2\text{O}_2 &\rightarrow 2\text{O}_2 + \text{H}_2\text{O} & & \text{Reac. 11} \\
2\text{HO}_2^- &\rightarrow \text{H}_2\text{O}_2 + \text{O}_2 & k=8.3 \times 10^5 \text{ M}^{-1}\text{s}^{-1} & \text{Reac. 12}
\end{align*}
\]

As reaction of hydrogen peroxide with hydroxyl radicals finally produces oxygen (Reac. 11 and 13), and this reagent is also decomposed into water and oxygen (Reac. 12), monitoring dissolved oxygen (DO) has been suggested as an easy, measurable tool for studying progress of the photo-Fenton process and H$_2$O$_2$ consumption efficiency (Gernjak et al., 2006; Prieto-Rodriguez et al., 2011; Santos-Juanes et al., 2011). Generation of too much oxygen is indicative of inefficient reaction conditions, whereas a decrease in dissolved oxygen concentration (to near zero) indicates participation of competitive superoxide radicals (Reac. 3-11) in the degradation of organic compounds and so low hydrogen peroxide concentrations available (Kunai et al., 1986). Therefore, based on DO measurements, different strategies for optimizing hydrogen peroxide consumption have been proposed. For example, (Ortega-Gómez et al., 2012) developed a system model (DO-H$_2$O$_2$) using the reaction curve method with an anti-windup proportional integral (PI) controller which achieved a 50% decrease in hydrogen peroxide consumption for decontamination of waste water containing paracetamol as a model pollutant. The behavior of the DO-H$_2$O$_2$ relationship has also been examined in tests of H$_2$O$_2$ automatic dosing strategies keeping DO between desirable set points (Prieto-Rodriguez et al., 2011). These studies also showed the importance of the Dorfman mechanism (Dorfman et al., 1962) during the first stages (Reac. 3-5) of photo-Fenton in which DO is actually consumed.

As a follow-up to such previous studies, different H$_2$O$_2$ dosing strategies, along with continuous supply of pure oxygen or air to the reaction medium, were analyzed to see whether external contribution of oxygen leads to lower H$_2$O$_2$ consumption for a specific level of degradation of complex wastewater.

2. Material and methods

2.1. Chemicals and reagents

Commercial formulations of five pesticides: Vydate® (10%, w/v, oxamyl, C$_7$H$_{13}$N$_2$O$_3$S), Metomur® (20%, w/v, methomyl, C$_3$H$_{10}$N$_2$O$_2$S), Couraze® (20%, w/v, imidacloprid, C$_{10}$H$_{12}$CIN$_3$O), Ditimur-40® (40%, w/v, dimethoate, C$_{3}$H$_{12}$NO$_3$PS$_2$), and Scala® (40%, w/v, pyrimethanil, C$_{12}$H$_{13}$N$_3$I) were used as received. Analytical standards (>98%) for chromatographic analyses were purchased from Sigma–Aldrich. Distilled water used in the pilot plant was supplied by the Plataforma Solar de Almería (PSA) distillation plant.
(conductivity < 10 μS cm⁻¹, Cl⁻ = 0.2–0.3 mg l⁻¹, NO₃⁻ < 0.2 mg l⁻¹, organic carbon < 0.5 mg l⁻¹). Solar photo-Fenton experiments were performed using iron sulfate (FeSO₄·7H₂O), reagent grade hydrogen peroxide (30%, w/v) and sulfuric acid for pH adjustment (around 2.6-2.8) to avoid iron salt precipitation, all provided by Panreac (analytical grade). Pure oxygen (industrial quality) was also used as purchased from Carburos Metálicos S.A.

2.2. Analytical determinations

Mineralization was followed by measuring the dissolved organic carbon (DOC) by direct injection of filtered samples with 0.2 μm syringe-driven filters into a Shimadzu TOC-VCSN analyzer with an NDIR detector. Total iron concentration was monitored by colorimetric determination with 1,10-phenanthroline, following ISO 6332, using a Unicam-2 spectrophotometer. Hydrogen peroxide concentration was measured in a spectrophotometer at 410 nm based on the formation of a yellow complex from the reaction of titanium (IV) oxysulfate with H₂O₂ following DIN 38409 H15. Chemical oxygen demand (COD) was measured with Merck® Spectroquant® NOVA 30 photometer (an external calibration curve was used).

2.3. Experimental set-up

2.3.1. Solar photo-reactor

Solar photo-Fenton experiments were performed in a Compound Parabolic Collector (CPC) solar pilot plant specially developed for solar photo-Fenton applications (Malato et al., 2009) and operated in batch recirculation mode. The reactor loop consists of a continuously stirred tank, a centrifugal recirculation pump (1.5 m³ h⁻¹), and connecting tubing and valves. The solar collector is composed of four 1.04-m² CPC units (total area of 4.16 m²). The total reactor volume is 75 L (V₁) and the total illuminated volume is 44.6 l (V). The temperature inside the reactor was kept at 35 °C using a temperature control system consisting of trace heating in the tubing, and cooling by a heat exchanger with a secondary cooling water cycle. For a detailed diagram of this solar reactor see (Maldonado et al., 2007).

Dissolved oxygen (DO) and pH in the pilot plant were measured online by WTW® Sensolyt IQ system electrodes. All the online data acquisition instruments transmitted their measurements by analogical output of their respective controllers to a series of distributed I/O modules (Advantech® ADAM 4000 series). Custom SCADA (Supervisory Control and Data Acquisition) software for this was programmed in National Instruments® Lab-VIEW 7.0 at the Plataforma Solar de Almería. In the experiments in which hydrogen peroxide was automatically dosed as a function of dissolved oxygen, 30% w/v H₂O₂ solution was added to the batch recirculation tank by an Alldos® diaphragm dosing pump (Alldos® Primus 208 Etron Profi E26-0.8, at 1 ml min⁻¹). This pump was activated by the abovementioned SCADA software when the DO low setpoint was reached, and turned off when it reached the high setpoint.

Solar ultraviolet radiation (UV) was measured by a global UV radiometer (KIPP&ZONEN, model CUV3). Equation 1 was used for treatment of data acquired during the experiments, where tₙ is the experimental time for each sample, UV (kJ l⁻¹) is the average solar ultraviolet radiation measured during Δtₙ, Aₙ is the total illuminated area of collectors (m²), V₁ is the total volume of the pilot plant and Qᵥᵥ is the accumulated energy (per unit of volume, kJ l⁻¹) incident on the reactor for each sample taken during the experiment.

\[ Q_{UV,n+1} = Q_{UV} + \Delta t_n \cdot \frac{A_n}{V_1}; \quad \Delta t_n = t_{n+1} - t_n \]

Eq. 1

2.3.2 Experimental Procedure

Based on previous related work (Prieto-Rodríguez et al., 2011) three different solar photo-Fenton treatments of waste water containing five commercial pesticides were carried out to determine whether H₂O₂ consumption could be reduced when working with a continuous supply of air or pure oxygen to remove at least 60% COD. This wastewater was used as a model because it was sufficiently
complex to simulate different waste waters, and because our group has had long experience with it, making evaluation of the effect of adding oxygen on a well-known photo-Fenton process more consistent. The main purpose was to try and reinforce the Dorffmann mechanism for removal of organics, lowering H$_2$O$_2$ consumption and reducing the amount of sunlight required, as the Dorffmann mechanism is a “dark process”.

Experiments performed were: (i) Solar photo-Fenton with pure oxygen injected directly into the recirculation tank and excess hydrogen peroxide of 200-500 mg l$^{-1}$; (ii) Solar photo-Fenton under the same conditions as before, but injecting air; and (iii) Solar photo-Fenton with air injection and automatic dosing of H$_2$O$_2$ based on DO setpoints, instead of an excess of hydrogen peroxide.

The covered solar photo reactor was filled with 7.5 l of water and an amount of each of the five commercial pesticides necessary for 40 mg l$^{-1}$ of DOC was added to the system, for a starting total DOC of 200 mg l$^{-1}$. Unfortunately, this could not be done with precision, since formulation ingredients usually caused foam and some of the DOC to not dissolve in water. As a consequence, the initial COD and DOC measured was usually not constant at the beginning of the treatment but the foam disappeared after a few minutes of treatment. After at least 15 min of homogenization, a sample was taken. Then, depending on the experiment, oxygen or air was injected into the reactor and maintained until the end of the experiment. Once DO was saturated in solution, the pH was adjusted to 2.8 with sulfuric acid. Afterwards, iron salt was added (20 mg l$^{-1}$ Fe$^{2+}$) and well homogenized for 15 min. Fe concentration was the optimum for CPC photoreactor according to previous optimization (Malato et al., 2004; Zapata et al., 2009). At that moment, collectors were uncovered and the first dose of H$_2$O$_2$ was added, starting photo-Fenton. Samples were taken every 15 min, quickly filtered and analyzed. For those experiments in which it was attempted to keep hydrogen peroxide in excess (between 200-500 mg l$^{-1}$) throughout the test, reagent consumed was continuously replaced.

3. Results and discussion

3.1. Solar photo-Fenton with injection of pure oxygen

At the beginning of the experiment, the reaction medium was over-saturated with pure oxygen to around 32 mg l$^{-1}$ of DO. Figure 1 presents the DOC and COD degradation profiles, changes in DO, and H$_2$O$_2$ consumption during the experiment. Once the photo-Fenton reaction began, DO quickly decreased during the first few minutes to 19 mg l$^{-1}$. After that, DO slowly but continuously decreased to below 1 mg l$^{-1}$, which is to now, the strongest COD reduction.

Reaction mechanisms in which dissolved oxygen is involved may take place during the first stage of the Fenton process, when it could be considered a soup-like radical. These mechanisms are maintained by the continuous injection of pure oxygen. That is, peroxy-organic radicals (ROO$^*$) produced by the Dorffman mechanism (Reac.3) can participate in another series of reactions (Reac. 4 and 5), generating hydroperoxyl radicals, which are less reactive than hydroxyl radicals, mainly generated in solution at acid pH (Reac. 6) (Pignatello et al., 2006), and can also react with the iron salts in solution (Reac. 9-10) generating more hydrogen peroxide and dissolved oxygen (Utset et al., 2000). The most favored reaction, under these conditions, is Reac. 10, which caused generation of more H$_2$O$_2$, and a negative effect according to Reac. 11 (very high kinetic constant), capturing hydroxyl radicals. Reac. 10 also converted Fe(II) into Fe(III), impeding the efficiency of the Fe(II)-based Fenton reaction (Reac. 1). On the other hand, an excess of hydrogen peroxide could also start to self-decompose by Reac.12. Due to these alternative mechanisms, overall decontamination slows down, as observed at the beginning of the treatment, where DOC remained constant for a longer time than in the experiment without O$_2$ as reported previously (Prieto-Rodriguez et al., 2011).

Taking into account that an input stream of pure O$_2$ was kept up until the end of the experiment, the DO profile observed in Figure 1 confirms its consumption throughout the process. Nevertheless, the heavy consumption of H$_2$O$_2$ in this experiment compared to the other one without injecting oxygen (Prieto-Rodriguez et al., 2011), in which 60% of COD (with 6.9 kJ l$^{-1}$) was removed after consuming only 12 mM
(408 mg l⁻¹) of H₂O₂ must be highlighted. In this experiment, 60% of COD was eliminated after 28.4 kJ l⁻¹ of accumulated UV energy and H₂O₂ consumption of 18 mM (612 mg l⁻¹). This shows that injection of pure oxygen into the reaction matrix has a negative effect on the solar photo-Fenton treatment of complex waste water.

Therefore, in an attempt to reduce this negative effect, in experiments described in Sections 3.2 and 3.3, air was continuously bubbled into the photo-reactor instead of pure oxygen.

Figure 1. DOC and COD, H₂O₂ consumption and DO concentration during solar photo-Fenton with pure oxygen injection and excess of H₂O₂. DOC during solar photo-Fenton with the same initial conditions previously reported (no O₂ injection) (Prieto-Rodriguez et al., 2011)

3.2. Solar photo-Fenton with air injection

Figure 2 shows the results of the experiment with air injection.

Figure 2. DOC and COD evolution, H₂O₂ consumption and DO concentration during solar photo-Fenton with air injection and excess H₂O₂
This test was carried out with the same initial operating conditions described in Section 3.1, except oxygen was continuously supplied by injecting air into the reaction tank, up to a maximum of 9.5 mg l\(^{-1}\) of DO.

As observed, COD remained almost constant during a shorter period of time and less accumulated energy was required for its degradation to begin, at approximately 13 kJ l\(^{-1}\) instead of 18 kJ l\(^{-1}\), as previously observed when pure oxygen was injected. In this case, 20.5 mM (695 mg l\(^{-1}\)) of \(\text{H}_2\text{O}_2\) was consumed to remove 60% of the initial COD \((Q_{\text{UV}} = 24.2 \text{ kJ } \text{l}^{-1})\). This high consumption also shows inefficient replacement of \(\text{H}_2\text{O}_2\) by \(\text{O}_2\) from air, at least at the beginning of solar photo-Fenton. However, DO was rather low during the experiment, increasing only at the end of the treatment, when 60% of COD was mineralized. This substantial increase in DO may have been due to inefficient use of \(\text{H}_2\text{O}_2\), because Reac.10 was generating \(\text{H}_2\text{O}_2\) at the same time as Reac.12 (\(\text{H}_2\text{O}_2\) decomposition) was producing oxygen, balancing the overall consumption of \(\text{H}_2\text{O}_2\). This demonstrates the active participation of DO due to other undesirable reactions.

Again, as discussed in the section above, both oxidants were in excess (\(\text{H}_2\text{O}_2\) and \(\text{O}_2\)) during most of the degradation process, but they were inefficient for removal of organics compared to \(\text{H}_2\text{O}_2\) alone. Although in this case air was used instead of pure oxygen, it is clear that DO was also in excess giving rise to the undesirable mechanisms described in Section 3.1.

### 3.3. Solar photo-Fenton process with air injection and automatic dosing of \(\text{H}_2\text{O}_2\)

This experiment was done to evaluate the efficiency of DOC degradation and \(\text{H}_2\text{O}_2\) consumption with DO from continuous injection of air, and using a very low amount of automatically controlled hydrogen peroxide. These operating conditions were maintained by an automatic \(\text{H}_2\text{O}_2\) dosing system with on-off control according to low and high DO setpoints based on previously published results (Prieto-Rodriguez et al., 2011). The DO control setpoints tested were 5 (on) and 7 (off) mg l\(^{-1}\) and the inlet flow rate was 1 mg l\(^{-1}\) min\(^{-1}\) of \(\text{H}_2\text{O}_2\). With these set points, DO decreased to a minimum of approximately 2 mg l\(^{-1}\) at the beginning, even though automatic \(\text{H}_2\text{O}_2\) dosing started when DO went below 5 mg l\(^{-1}\). Afterwards, the DO concentration increased due to accumulation and self-decomposition of \(\text{H}_2\text{O}_2\) to over 7 mg l\(^{-1}\), when automatic dosing turned off. In order to start the degradation chain reaction (first steps Reactions 1 and 2) an initial amount of 0.2 mM (8 mg l\(^{-1}\)) of \(\text{H}_2\text{O}_2\) was added right at the beginning.

![Figure 3. DOC and COD, \(\text{H}_2\text{O}_2\) consumption and DO concentration during solar photo-Fenton with continuous air injection and automatic dosing of \(\text{H}_2\text{O}_2\). DO setpoints (5 and 7 mg l\(^{-1}\)) are also shown (dotted lines). \(\text{H}_2\text{O}_2\) in solution is also shown](image)
H₂O₂ accumulated as observed in Figure 3. In this case, H₂O₂ reached a maximum of 148 mg l⁻¹ at 19.4 kJ l⁻¹ (see Figure 3), when rapid mineralization started. A sharp COD reduction from 17% (at 19.4 kJ l⁻¹) to 60% elimination was observed with H₂O₂ consumption of 11.5 mM (390 mg l⁻¹, see Figure 3). In this case, when 60% of the initial COD was removed (Qᵥᵥ = 36.3 kJ l⁻¹), around 900 mg l⁻¹ of H₂O₂ had been consumed. Therefore, the H₂O₂ consumption was higher than for the two previous cases (see Sections 3.1 and 3.2). Although automatic dosing based on DO setpoints had been shown to be efficient in previous work (Prieto-Rodriguez et al., 2011), in this case higher hydrogen peroxide consumption is related to the low initial hydrogen peroxide dose (8 mg l⁻¹), which did not permit proper evolution of Reacs. 2 and 3. As DO rapidly decreases from the beginning, a higher initial H₂O₂ would be recommendable. Excess oxygen supply seems also to have had a negative effect, as H₂O₂ was inefficiently consumed.

3.4. Comparison of key parameters

The main parameters used to observe the effectiveness of solar photo-Fenton in remediation of a complex waste water by direct injection of additional dissolved oxygen into the reaction medium are summarized in Table 1, which shows DOC elimination percentages, H₂O₂ consumption and the accumulated energy (Qᵥᵥ, kJ l⁻¹) needed to eliminate 60% of COD. We selected 60% COD elimination, because for complex and biorecalcitrant industrial waste water, this is usually the point at which wastewater biodegradability is sufficiently enhanced to end the chemical oxidation step, and proceed to final polishing with a conventional biological system (Ballesteros et al., 2009; Oller et al., 2011). Attending to the results, behavior of DOC was consistent as its degradation was always slower and reached lower degradation than COD. It is well known that to reduce COD is not needed complete mineralisation, as it is for DOC.

### Table 1. Percentage of DOC mineralized, accumulated energy and hydrogen peroxide consumption required to eliminate 60% of COD

<table>
<thead>
<tr>
<th>Exp.</th>
<th>Description</th>
<th>% DOC mineralized</th>
<th>Qᵥᵥ(kJ l⁻¹)</th>
<th>H₂O₂ consumed (mg l⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>i</td>
<td>Pure O₂ injection + H₂O₂ in excess (200-500 mg l⁻¹)</td>
<td>27.1</td>
<td>28.4</td>
<td>612</td>
</tr>
<tr>
<td>ii</td>
<td>Air injection + H₂O₂ in excess (200-500 mg l⁻¹)</td>
<td>49.6</td>
<td>24.2</td>
<td>695</td>
</tr>
<tr>
<td>iii</td>
<td>Air injection + H₂O₂ automatic dosage</td>
<td>44.3</td>
<td>36.3</td>
<td>900</td>
</tr>
</tbody>
</table>

4. Conclusions

It was demonstrated that an injection of air or pure oxygen in the photo-Fenton reaction medium along with excess H₂O₂ causes generation of radicals (HO₂⁻) that are less reactive than hydroxyl radicals, leading to longer irradiation and higher hydrogen peroxide consumption to be necessary for desired degradation of organics. So it may be concluded that aeration of the photoreactor is not recommendable.

Automatic control of H₂O₂ based on DO setpoints has been proven extremely useful and efficient for photo-Fenton to progress properly based on “online” measurements (DO), instead of “offline”, such as H₂O₂ experimental determination. Nevertheless, H₂O₂ consumption was higher when this reagent was controlled by DO setpoints than in the companion experiment with H₂O₂ continuously added in excess because of the extremely low starting dose of hydrogen peroxide. However, this would be easy to solve in any industrial implementation just by adding a larger amount of H₂O₂ at the beginning, and continuing beyond the automatic DO setpoint control.
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