Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2013.809971

52 (2014) 4982–4989
July

Taylor & Francis
Taylor & Francis Group

Oxidation of bromocresol green by different advanced oxidation processes: Fenton, Fenton-like, photo-Fenton, photo-Fenton-like and solar light. Comparative study

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Received 11 June 2012; Accepted 12 May 2013

ABSTRACT

The aim of this study is to test some advanced oxidation techniques (AOTs) in the absence of light (Fenton and Fenton-like) and in its presence (photo-Fenton and photo-Fenton-like) to obtain total decolorization of an anionic dye in aqueous solution: the bromocresol Green (CI Reactive Green 15 and abbreviated as BCG). Experiments were conducted on a laboratory-scale set up with all these processes. Best operatory conditions were found to be of pH 3, hydrogen peroxide to iron (II or III) molar ratio of 30:1, artificial (254 nm) and solar light. Besides, several parameters were investigated like pH, oxygen, dose of H_2O_2, light effect, and reaction time. The obtained results showed that color removal followed the increasing order: $Fe(III)/H_2O_2 < Fe(II)/H_2O_2 < Fe(II)/H_2O_2/Solar < Fe(II)/H_2O_2/UV_{254nm} < Fe(III)/H_2O_2/UV_{254nm}$. This improvement could be related to a better production of radicals $OH^{\cdot}$. In another hand, decolorization kinetic followed pseudo-first-order type in all cases and no synergic effect was observed when Cu^{2+} ion (10^{-4}M) was added to the solution of the dye.

Keywords: Advanced oxidation processes; BCG; Photo-Fenton; UV; Solar light

1. Introduction

The effluents produced by the textile industry, present a major problem of pollution leading to severe damages to aquatic life (fauna and flora) [1,2] and to the human health through drinking water and consumption of food [3]. To overcome this problem, several decontamination methods were developed in the past like classical oxidation and reduction, biological treatment [4], coagulation [5], adsorption [6]. Unfortunately, these methods were unsuccessful, since they could involve problems such as sludge for precipitation, transfer of pollution from one phase to another for adsorption and coagulation, resistance to biodegradation for some dyes having complicated structure (presence of many aromatic cycles) [7]. An alternative way to these methods was to use new ones called advanced oxidation techniques (AOTs), since they could provide a definite solution to this problem by giving a possible mineralization. They exist both in the absence of light (Fenton and Fenton-like) and in the presence of light (photo-Fenton, photo-Fenton-like, UV/H_2O_2, O_3/UV, and semi-conductor/UV) [8–12].

Among these processes, Fenton or Fenton-like appear to be very interesting because of their simple

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implementation, which consists of simple equipment and availability of the used chemical reagents like H_2O_2, ferrous, and ferric ions. However, the only disadvantage for both systems is their use in acidic medium (pH=3) to prevent formation and precipitation of hydroxides. Thus, the mechanism of oxidation of pollutants is as follows (Eqs. (1)–(5)) [13–16]:

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + OH^\bullet + H_2O \quad (1)$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HOO^\bullet + H^+ \quad (2)$$

$$Fe^{3+} + HOO^\bullet \rightarrow Fe^{3+} + O_2 + H^+ \quad (3)$$

$$R - H + OH^\bullet \rightarrow R^\bullet + H_2O \quad (4)$$

$$R^\bullet + Fe^{3+} \rightarrow R^+ + Fe^{2+} \quad (5)$$

Moreover, the combination of light (artificial or natural) to the Fenton or Fenton-like, could improve the degradation of pollutants since the production of $OH^\cdot$ is greatly increased.

The goal of this study is to decolorize anionic dye BCG using a molecule model (belonging to the triphenylmethan) by some AOPs in the absence of light (Fenton, Fenton-like), and in the presence of artificial and solar light (photo-Fenton, photo-Fenton-like). Since this dye is hardly biodegradable, AOTs would be necessary to destroy it and its by-products. The effect of different parameters in bleaching process such as pH, H_2O_2 dose, O_2, and Cu^{2+} will be investigated. Moreover, an optimal ratio (H_2O_2-to-iron (II) or (III)) will be investigated too in order to produce maximum of $OH^\bullet$ radicals.

2. Experimental

2.1. Material

Bromocresol Green (BCG) was purchased from Fluka chemical company and used without further purification. The hydrogen peroxide solution (33% Fluka), salts of iron (II) ($(NH_4)_3$ $Fe(SO_4)_26H_2O$, Labosi 99%), iron (III) **(**$Fe(ClO_4)_39H_2O$, Carlo Erba 99%), and ions of Cu^{2+} ($CuSO_4{\cdot}5H_2O$, Fluka 99%) were prepared from ultra pure water from a MILIPORE unit at pH=3.0, to prevent formation and precipitation of hydroxyls. Besides, pH was adjusted with NaOH or HCl to reach the desired value. Moreover, the dissolved oxygen (which was 8 ppm at room temperature) was removed from the aqueous solution of BCG by bubbling nitrogen gas for about 2 h. Then, the reactor was closed (by means of paraffin) during the irradiation process.

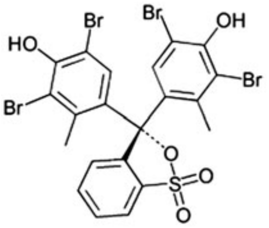

Fig. 1. Structure of bromocresol green.

Fig. 1 shows the structure of the dye. Its high solubility in water is attributed to the presence of the sulfonate group [17].

2.2. Photo-reactor and source light

Aqueous solutions were irradiated at 254 nm in cylindrical reactor quartz (100 cm of length and 2 cm in diameter), located on one of the principal axis of the assembly and equipped with three symmetrical externals low-pressure mercury lamps (germicide lamp, Philips TUV 15 W) emitting mainly at 254 nm [17].

In solar light, aqueous solution of the dye were degraded in presence of H_2O_2 and salt of Fe (II) using a pilot plant containing a reservoir delivery, a pump, and an assembly of tubes on Pyrex. They are located closely to each other and in a vertical position towards natural light. Samples were withdrawn at regular time intervals and analyzed immediately [18] (Fig. 2).

2.3. Procedure and analysis

The irradiation experiments were carried out with 100 ml of solution with 6×10^{-5} M of BCG. The UV–vis absorption spectrum was recorded from 200 to 800 nm using Unicam Helios "α" a UV–vis spectrophotometer at $\lambda_{max} = 444$ nm. The residual concentrations of the substrate at different times were obtained at $\lambda_{max} = 444$ nm using a calibration curve.

3. Results and discussion

3.1. UV-vis spectrum of BCG

The UV-vis spectra of BCG are reported in Fig. 3. At natural pH mainly three absorption bands are observed, each characterized by a molar absorption coefficient ε and located at 280 nm ($\varepsilon = 9{,}16\,51\,mol^{-1}\,cm^{-1}$), at .444 nm ($\varepsilon = 16{,}5451\,mol^{-1}\,cm^{-1}$ being the most intense), and at 616 nm ($\varepsilon = 7{,}49\,01\,mol^{-1}\,cm^{-1}$), respectively [19]. The natural pH of

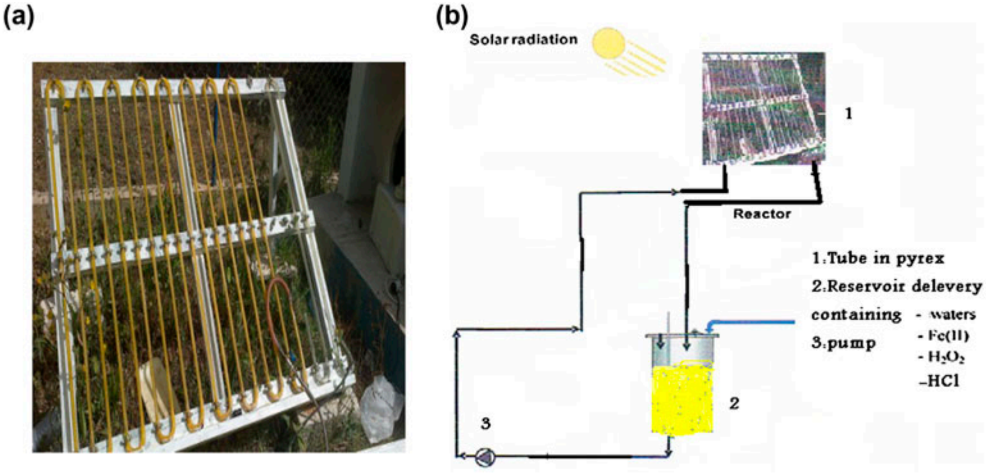

Fig. 2. Setup showing the pilot plant solar light: (a) picture of the solar reactor and (b) scheme of the device.

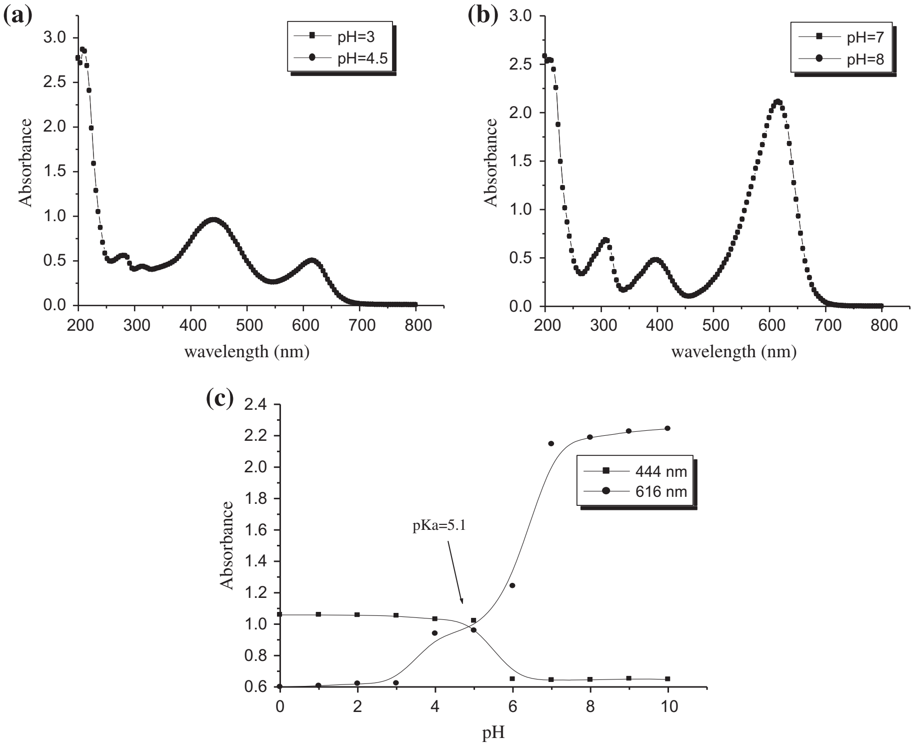

Fig. 3. UV–vis spectrum of BCG, $[dye]_o = 6 \times 10^{-5}$M. Influence of pH at (a) pH = 3 and 4.5; (b) pH = 7 and 8; and (c) evolution of pKa.

the aqueous solution containing 6×10^{-5}M dye is about 4.5. In addition, the pH affected the behavior of the substrate, mainly, in basic medium ($pH \geqslant 8$) where we observed a deprotonation of the dye molecule. Indeed this high value of pH is responsible of the blue shift and the change in the molar absorption coefficient. By contrast, we observed no shift of all bands in acidic medium ($pH < 4$).This could indicate, therefore, no change in the structure. The experimental value of the pKa was about 5.1 and agreed well with that found in the literature: 4.9.

3.2. Absence of light

3.2.1. Effect of pH and concentration of H_2O_2 on the elimination of BCG by Fenton and Fenton-like processes

It is well known that Fenton and Fenton-like reactions are effective at pH = 3.0 [16]. Taking this into

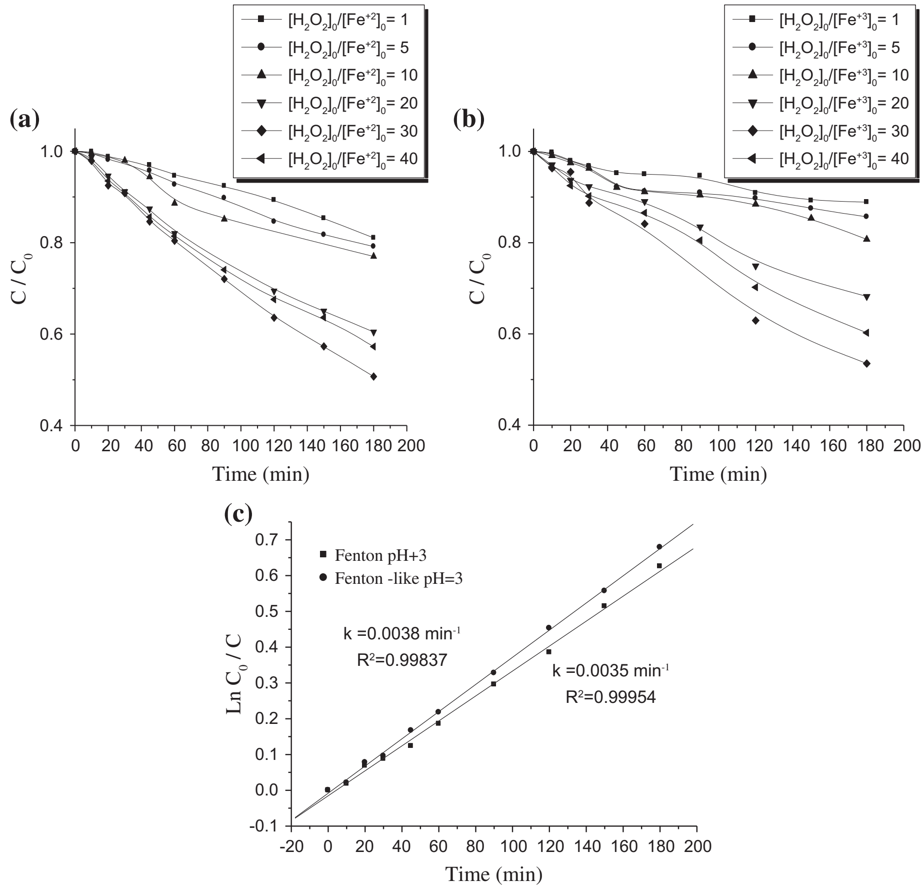

Fig. 4. Effect of ratio $[H_2O_2]/[Fe^{2+}]$ on the decolorization of BCG during: (a) Fenton process, (b) Fenton-Like process, and (c) kinetics decolorization of this dye (linear transform Ln (C_0 C_t) vs. time) at ratio 30:1. Initial conditions: (C_0 (dye) $= 6 \times 10^{-5}$ M. C_0 ($Fe^{2+} = Fe^{3+}$) $= 10^{-4}$ M and pH = 3.0).

account, a set of experiments were performed to obtain best color removal from the related ratio: H_2O_2/Fe^{+2} or H_2O_2/Fe^{+3}. As recommended by the literature, the ratio ranged generally from 10:1 to 40:1 [20–22]. Therefore, this best efficiency was achieved for 30:1 (Fig. 4). As can be seen in both figures, we observed a decolorization process reaching color removal of 50% for Fenton and of 46% for Fenton-like, for a reaction time of 180 min for both processes. This could be attributed to an effective participation of $OH^\bullet$ in the oxidation of the dye. We observed also an enhancement of this efficiency as the concentration of H_2O_2 increased from 10^{-4} M to 3×10^{-3} M. Thus, this enhancement might be due to the catalytic production of $OH^\bullet$ and Fe^{2+}. However, for high concentration of H_2O_2 ($[H_2O_2] \geqslant 3 \times 10^{-3}$ M), we observed a scavenging effect of the $OH^\bullet$ by H_2O_2 itself:

$$OH^\bullet + H_2O_2 \rightarrow HO_2^\bullet + H_2O \tag{6}$$

The kinetic study showed that the degradation processes for the ratio 30:1 followed a pseudo-first-order, where the constant k is equal to 3.8×10^{-3} min^{-1} for Fenton and to 3.5×10^{-3} min^{-1} for Fenton-like processes.

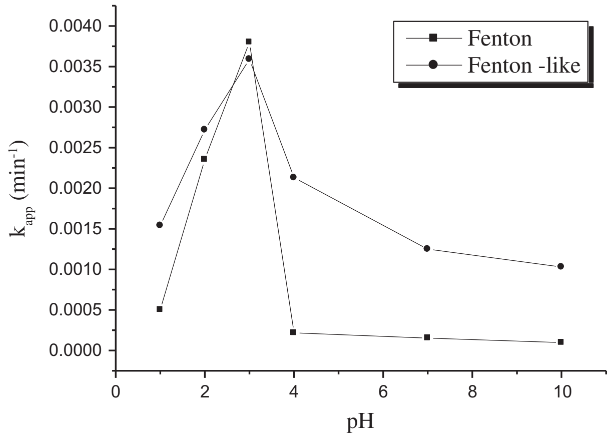

Fig. 5. Effect of pH on decolorization process of BCG in aqueous medium by Fenton and Fenton-like. Initial conditions: C_0 (dye) $= 6 \times 10^{-5}$ M. C_0 ($Fe^{2+} = Fe^{3+}$) $= 10^{-4}$ M. C_0 (H_2O_2) $= 3 \times 10^{-3}$ M and pH = 3.

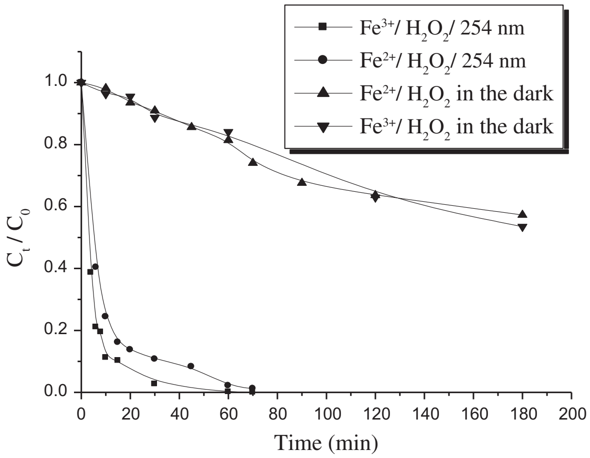

Fig. 6. Comparative study between Fenton, Fenton-like, photo-Fenton, and photo-Fenton-like processes (C_0 (dye) $= 6 \times 10^{-5}$ M. C_0 ($Fe^{2+} = Fe^{3+}$) $= 10^{-4}$ M. C_0 (H_2O_2) $= 3 \times 10^{-3}$ M, pH = 3, $\lambda = 254$ nm).

Moreover, the effect of pH (ranging from 1–10) in the bleaching process of the dye (6×10^{-5}M) was investigated too and reported in Fig. 5 for Fenton and Fenton-like systems. Thus, by considering this figure we observed that:

- In very acidic medium (pH < 2) the efficiency was low due to a weak participation of radical $OH^{\bullet}$ which are scavenged by this medium (reaction 7)

$$OH^{\cdot} + H^+ + e^- \rightarrow H_2O \qquad (7)$$

- In basic medium, we observed also a sensitive diminution of the efficiency linked to the formation and precipitation of iron $Fe(OH)_3$, stopping the formation of radicals $OH^{\bullet}$

The value of pH, which was equal to 3.0, corresponds to that from literature.

3.3. Effect of light

3.3.1. Photo-Fenton and photo-Fenton-like processes

The solutions of the dye, prepared under the same experimental conditions, were achieved in the presence of artificial (254 nm) and natural light (solar). They ended up with a better color removal than previously.

3.3.1.1. Effect of artificial light (254 nm). The oxidation of the dye was greatly enhanced by light for both systems. In this case, total decolorization was reached in approximately 60 min for photo Fenton-like and 70 min for photo-Fenton processes. Indeed, in the first 10 min, the efficiency of the degradation was 1.2 times faster for $Fe^{3+}/UV/H_2O_2$ comparatively to $Fe^{2+}/UV/H_2O_2$ (Fig. 6). Thus, it appeared that this route is more rapid than that obtained by Fenton and Fenton-like. This enhancement could be attributed to the important production of radical $OH^{\bullet}$ from different sources like [23,24].

- The reaction between Fe^{2+} and Fe^{3+} with H_2O_2.
- The photolysis of H_2O_2 at 254 nm.
- The photoreduction of species resulting from the hydrolysis of Fe^{3+} ($Fe(OH)_2^+$) and Fe^{2+} ($Fe(OH)^+$).

3.3.2. Effect of oxygen in photo-Fenton process

To evaluate the effect of oxygen, experiments were conducted under same conditions which were estab-

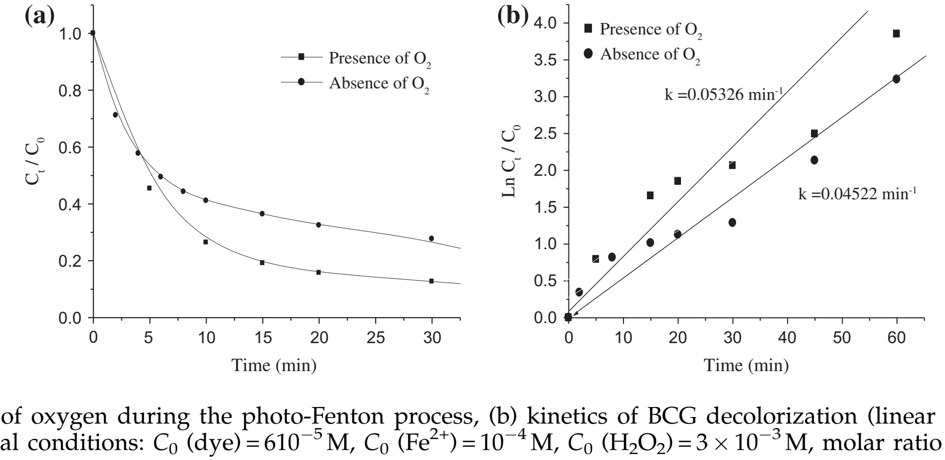

Fig. 7. (a) Effect of oxygen during the photo-Fenton process, (b) kinetics of BCG decolorization (linear transform ln (C_0/C_t) vs. time. Initial conditions: C_0 (dye) $= 610^{-5}$ M, C_0 (Fe^{2+}) $= 10^{-4}$ M, C_0 (H_2O_2) $= 3 \times 10^{-3}$ M, molar ratio $Fe^{2+}/H_2O_2 = 1{:}30$, and pH = 3, $\lambda = 254$ nm.

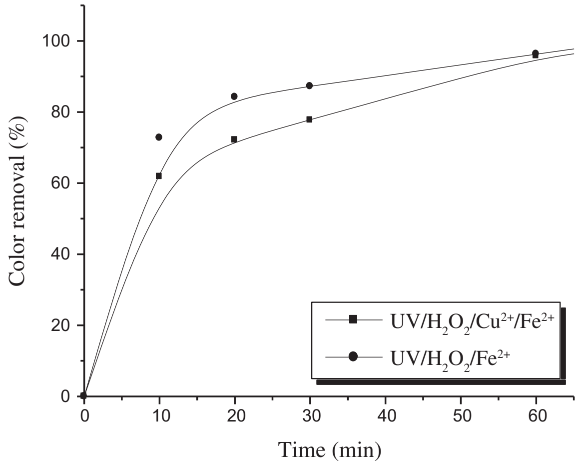

Fig. 8. Effect of Cu^{2+} during photo-Fenton process. Initial condition: C_0 (dye) = 6×10^{-5} M, C_0 (Fe^{2+}) = 10^{-4} M, C_0 (Fe^{3+}) = 10^{-4} M, C_0 (H_2O_2) = 3×10^{-3} M, C_0 (Cu^{2+}) = 10^{-4} M, pH = 3, λ = 254 nm.

lished previously ([BCG] = 610^{-5}M, H_2O_2/Fe^{2+} = 30:1, and pH=3.0). The results illustrated in the Fig. 7(a), showed that the efficiency of color removal was equal for both processes during the first 5 min and decreased beyond this time to reach 73.7% in the absence of O_2 and 87.3% in its presence, for a reaction time of 30 min. Moreover, Fig. 7(b) reveals that photo-Fenton system followed a pseudo-first-order kinetic with rate constants equal to 5.3×10^{-2} min^{-1} in the absence of O_2 and 4.5×10^{-2} min^{-1} in its presence. This slight improvement could be explained by an oxidation of iron (II) again, where we observed the production of $O_2^{\bullet-}$ and H_2O_2. Once H_2O_2 is formed, it could react with Fe^{2+} to produce and to reinforce the radical species $OH^{\bullet}$ [25].

$$Fe^{2+} + O_2 \rightarrow Fe^{3+} + O_2^{\bullet-} \tag{8}$$

$$Fe^{2+} + O_2^{\bullet-} + 2H^+ \rightarrow Fe^{3+} + H_2O_2 \tag{9}$$

$$2O_2^{\bullet-} + 2H^+ \rightarrow H_2O_2 + O_2 \tag{10}$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet} \tag{11}$$

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^- \tag{12}$$

3.3.3. *Effect of copper (Cu)*

The principal interest of copper was to study the synergetic effect in the photo-Fenton system. Indeed, according to the bibliographies data, copper (Cu (II)) could improve the photo-Fenton in the bleaching process. This improvement is related to the reduction of Cu (II)–Cu (I) [26]:

$$Cu^{2+} + R^{\bullet} \rightarrow Cu^+ + \text{Products} \quad [26] \tag{13}$$

To evaluate the benefit of the synergic effect, experiments were achieved under the following conditions: 5 ml of Cu^{2+} (10^{-4}M), [BCG] = 6×10^{-5} M, $[H_2O_2]/[Fe^{2+}]$ = 30:1, and at 254 nm. In Fig. 8, the results of decolorization are described and we observed no appreciable difference between both systems: $Fe^{2+}/H_2O_2/UV_{254nm}$ (99.2%) and $Fe^{2+}/H_2O_2/Cu^{2+}/UV_{254nm}$ (97.3%) [16].

3.3.3.1. Effect of solar light. The decolorization process, occurring in solar light, was achieved in typical

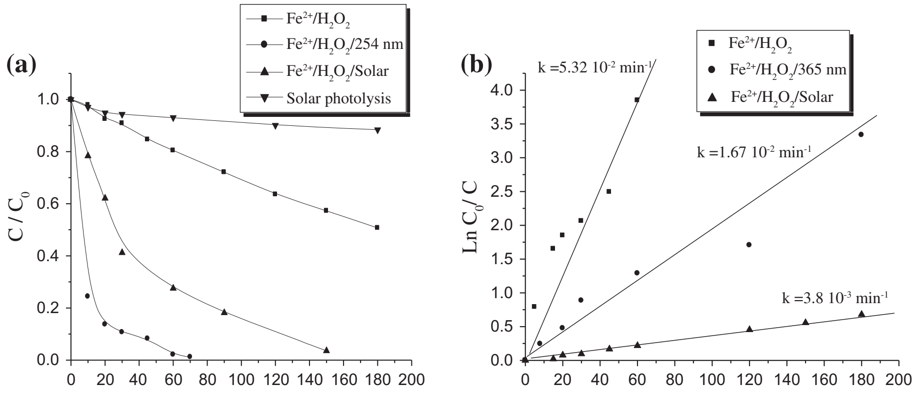

Fig. 9. Effect of light on the bleaching process of BCG: (a) in presence of Fe^{2+}/H_2O_2, solar photolysis, and solar/Fe^{2+}/H_2O_2 and (b) kinetics of BCG decolourization ((linear transform ln (C_0/C_t) vs. time). Initial conditions C_0 (dye) = 6×10^{-5} M, C_0 (Fe^{2+}) = 10^{-4} M, C_0 (H_2O_2) = 3×10^{-3} M, molar ratio Fe^{2+}/H_2O_2 = 1:30, and pH = 3.

Table 1
Values of $t_{1/2}$ and apparent rate constants parameters characterizing the different systems

Systems	$t_{1/2}$ (min^{-1})	R^2	Apparent rate constants (min^{-1})
Photolysis in solar light	Not reached	–	–
Fe^{2+}/H_2O_2	Not reached	0.9995	3.8×10^{-3}
254 nm/Fe^{2+}/H_2O_2	7.35	0.9838	5.21×10^{-2}
Solar/Fe^{2+}/H_2O_2	25.4	0.9883	1.67×10^{-2}

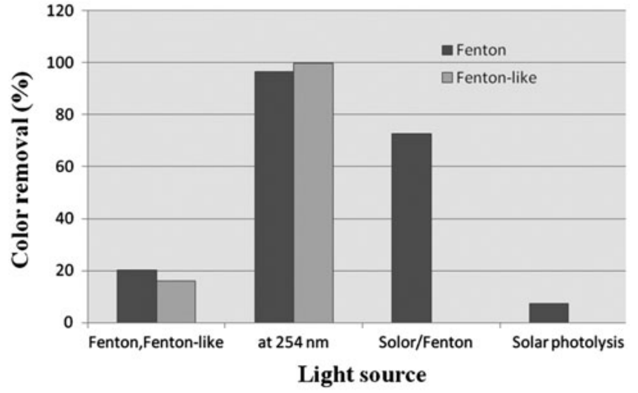

Fig 10. Effect of light on the degradation of BCG. Initial C_0 (dye) = 6×10^{-5}M, C_0 (Fe^{2+} and Fe^{3+}) = 10^{-4} M, C_0 (H_2O_2) = 3×10^{-3} M, and pH = 3.0. Reaction time: 60 min.

summer days and under the same experimental conditions. In Fig. 9(a), we observed that efficiency resulting from oxidizing process (photo-Fenton in artificial light) was about 99.2% in 70 min, whereas in natural light it was about 75.5%. The photooxidation of the substrate in natural light involving H_2O_2 and Fe^{2+} gave appreciable results due to the photoreduction of species like Fe $(III)_{aq}$ or $Fe(OH)^{+2}$, leading thereby, to an important production of radicals $OH^\bullet$ [15]. However, in the absence of iron salt and hydrogen peroxide, the direct photolysis of the substrate in solar light was not significant: about 12% for a reaction time of 180 min:

$$Fe^{3+} + H_2O_2 + h\nu \text{ (solar)} \rightarrow Fe^{2+} + OH^\bullet + H^+ \quad (14)$$

However, in the presence of artificial light, the efficiency was still higher as compared with that obtained in the dark and the solar light. This fact may be explained by a high energy, since we operated at 254 nm. A kinetic study of the following processes as Fe (II)/H_2O_2, Fe (II)/H_2O_2/UV_{254nm}, and solar/Fe (II)/H_2O_2, allowed us to evaluate the apparent rate constant (k_{app}) for each system from a linear regression ln C_0/C vs. time (min).We observed also that the obtained straight lines all have a high correlation coefficient (>0.9).Thus, the examination of Fig. 9(b) shows that no differences exist among all three systems but only in the first 20 min. In order to get a better knowledge of the color removal, we have calculated $t_{50\%}$ graphically for each system (which is a time to reduce to an half the initial dose of the substrate).This computation is useful in a sense, where it allows us to compare correctly the performance of each process (Table 1).

3.4. Comparison of the various processes studied in decolorization of BCG

In Fig. 10, we present the results obtained for color removal (expressed in%) as a function of the irradiation wavelength. We observed an increase of the efficiency as we coupled light (artificial or solar) to Fenton system: 20.2% for Fe^{2+}/H_2O_2, 96.3% for UV/Fe^{2+}/H_2O_2, 99% for UV/Fe^{3+}/H_2O_2, and finally 72.5% for solar/Fe^{2+}/H_2O_2. These efficiencies have all been measured in 60 min. However, the efficiency found in photolysis (solar light) was negligible (7.4% for the same time). This is in agreement with the fact that photo-Fenton and photo-Fenton-like dominate all processes including the solar one, due to a high production of reactive species (radicals $OH^\bullet$) comparatively to that of Fenton. We confirmed this increasing order followed by the efficiency:

Solar photolysis < Fe(III)/H_2O_2 < Fe(II)/H_2O_2 < Fe (II)/H_2O_2/Solar light < Fe(II)/H_2O_2/UV_{254nm} < Fe(III)/H_2O_2/UV_{254nm}.

4. Conclusion

In the present study, treatment of an anionic dye solution using Fenton and Fenton-like in one part and photo-Fenton and photo-Fenton–like under artificial and natural light in another part, was taken into account. Based on the obtained results, we observed that the optimal value of pH was equal to 3.0 and the most favorable ratio of H_2O_2 to iron was 30:1 for both oxidation states of iron II and III. Under these experimental conditions, 49.8 and 45.8% degradation were achieved for Fe^{2+}/H_2O_2 and Fe^{3+}/H_2O_2, respectively. However, a better efficiency was obtained when compared with that in dark solution when artificial and natural light were coupled to both systems. The total color removal was within 60 and 70 min for Fe^{2+}/

H_2O_2/UV_{254nm} and $Fe^{3+}/H_2O_2/UV_{254nm}$, respectively. Additionally, degradation of the dye was enhanced with the increase of H_2O_2 concentration but to a certain limit of H_2O_2 when we observed a scavenging effect from the ratio H_2O_2 to Fe^{3+}:40:1. In general, photo-Fenton and photo-Fenton-like appeared to be suitable for the degradation of the dye since they reinforce the production of radicals OH. These investigations showed that these processes might be used for the treatment of wastewater on industrial scale.

The influence of oxygen particularly in the photo-Fenton system showed a moderate "slow down" of the decolorization rate, mainly, beyond a reaction time of 5 min. Besides, no synergy effect was observed when adding Cu^{2+} in the same process.

In the other hand, the decolorization process in the presence of solar light and on typical summer days was found to be more efficient than that obtained in dark conditions. The application of the Fenton process using solar energy might be interesting in the treatment method of wastewater in industrial applications.

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