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Degradation of an azo dye by a fast and innovative pulsed light/H_2O_2 advanced oxidation process

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ABSTRACT

Pulsed light (PL) is a food processing technology initially intended for microbial inactivation that can potentially be applied in other UV-light based processes. PL lamps emit high intensity broad spectrum light from UV to infrared. This research tested the use of PL as part of an advanced oxidation process (AOP) for degrading polluting dyes. Experiments were performed in a batch reactor, and the efficiency of the AOP under different parametric values: dye concentration, pH and H_2O_2 doses on Direct yellow 106 decolourization was followed by spectrophotometry. Effects on chemical oxygen demand and electrical efficiency were also determined. Decolourization process follows a pseudo-first order kinetic. It was improved by increasing H_2O_2 concentrations and low pH; while there was little influence of dye concentration, perhaps due to the reactor configuration. The highest constant rate observed was 0.0410 cm^2/J. The decolourization by PL/H_2O_2 fits to a typical mechanism of a conventional UV/H_2O_2 process; since no direct photolysis or thermal effects were observed. Mineralization was incomplete likely due to the highly resonant structure of the dye. An electrical energy per order of 1009 kWh/m^3/order was calculated. Ninety % of colour removal was reached at 40 J/cm^2, which is achievable in less than 30 s by most of PL systems existing in the market. The PL/H_2O_2 process seems to be useful for decolourization of wastewater and could offer the advantage of a very fast degradation.

1. Introduction

From the past few decades the extensive use of pesticides, industrial chemicals and different types of dyes in cosmetic, paper and leather is well known. A large amount of pollutants are disposed continuously into water bodies from the above sources, leading to undesirable accumulation of toxic compounds that entail serious environmental problems [1]. The textile industry is considered the most polluting industrial sector due to the quantity and constituents of the effluents produced [2]. Therefore, it is logical to look for an economical and feasible technique for remediation of such hazardous environmental effluents [3]. In fact, about 1–20% of world dye products enter into textile wastewater during the dying process. Due to their good properties, azo dyes are the most widely used. It is estimated that their total production exceed 7.000,000 ton per year, representing over 60% of the total dye production, followed by the anthraquinone group [4–7]. The main adverse effects of azo dyes in the environment are their inhibitory effect on aquatic photosynthesis, ability to deplete dissolved oxygen and toxicity to flora, fauna and humans. Also, if the dyes are broken down anaerobically, aromatic amines are generated, which are very toxic, carcinogenic, genotoxic and mutagenic compounds [8].

One of the strategies to reduce the disposal of dyes into water bodies is directed to recover them from wastewater and reuse them as raw material in new dyeing steps, as well as obtaining less contaminated wastewater that can be reused. Following this strategy, our research group has recently developed a method based on the use of cyclodextrins for the encapsulation of different dyes, such as Direct yellow 106 [9]. Since dye retention is incomplete, an additional strategy to eliminate the remaining dye from wastewater is required.

In recent years, advanced oxidation processes (AOPs) have been proved as an excellent method for the degradation of environmentally hazardous material [1]. Among these methods,

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homogeneous chemical oxidation using ultraviolet radiation (UV) in the presence of H_2O_2 has been widely studied. The UV/H_2O_2 process involves the photolysis of hydrogen peroxide to generate hydroxyl radicals (•OH), which are very effective in the oxidation and mineralization of most organic pollutants [10].

Pulsed light technology is a kind of photonic technology where electrical energy is stored in capacitors over a relatively long time, and then released in a shorter time to a xenon lamp, which emits pulses of a high intensity broad spectrum light, that includes UV light [11]. PL can deliver high fluences in a significantly shorter time than UV continuous (CW UV) processes, producing more rapid effects both in time and fluence. This technology has been mainly developed and applied for inactivation of microorganisms on food, food contact surfaces and transparent liquids, and it is currently applied at industrial level for decontamination of caps and cups used in food packaging. In recent years, the scope of potential applications of PL technology has been extended to other fields such as protein modification [12], inactivation of enzymes [13] or enrichment of food vitamin content [14], the latter currently applied at industrial scale. Since PL has an important UV component, a brand new application of this technology could be its use as part of an AOP for degradation of polluting dyes. Therefore, the aim of this research has been to assess the performance of PL as part of an AOP, for the degradation of polluting dyes, studying specifically the effect of H_2O_2 and dye and concentrations and pH on the rate of decolourization. Moreover, dye mineralization and electrical efficiency of the process was evaluated.

2. Materials and methods

2.1. Chemicals

C. I. Direct yellow 106 (DY106) (CAS: 12222-60-5) was kindly provided by Comercial Química Massó (Barcelona Spain). As can be observed in Fig. 1 [15], the chemical structure of DY 106 is a challenge for its degradation by an AOP since is a stable molecule, including in its structure an azo link, an ethylene group and benzene-sulphonic moieties. Also, the presence of naphthalene groups sharing carbons with 1,2,3-triazole groups give this dye a high conjugation degree. Hydrogen peroxide solution (30%) and other compounds such as HCl and NaOH, were purchased from Merck (Germany). Work solutions were prepared by dissolving the required quantity in demineralized water.

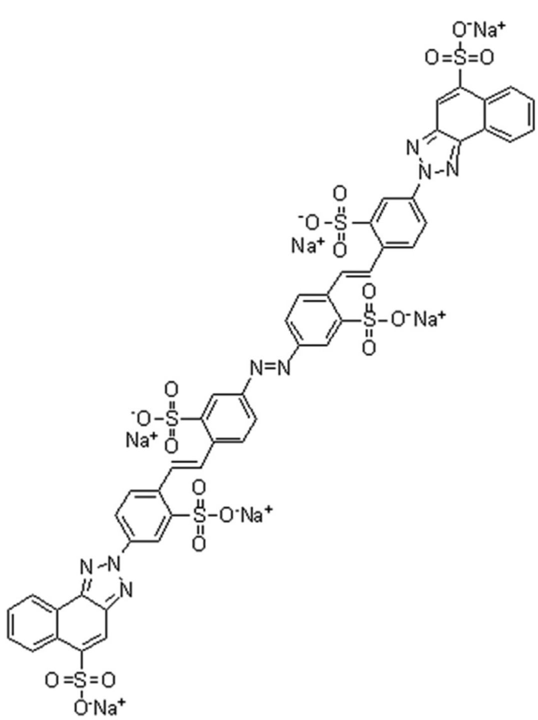

Fig. 1. Molecular structure of Direct yellow 106.

2.2. Apparatus

Pulsed light treatment was performed in a XeMaticA-Basic-1L system (Steribeam, Kehl, Germany). The system produces a broad-spectrum pulsed light spanning from infrared to ultraviolet light with a pulse width of 200 μs The treatment chamber has dimensions of 20 cm wide, 14 cm high, 10 cm deep, and it is equipped with one 19-cm long xenon lamp placed at the top of the chamber. A stirrer (Topolino, IKA, Staufen, Germany) was incorporated to the chamber for inter-pulse sample homogenization. The system applied a discharge voltage of 2.5 kV, which generates 500 J/pulse. Samples received an incident fluence of 2.14 J/cm^2 pulse, which was determined by analysis of in-built photodiode readings using a PC-Lab 2000 LT PC oscilloscope (Velleman Instruments, Gavere, Belgium), and manufacturer performance charts. At this discharge voltage, the UV fraction of the spectrum is 21%. Different fluencies were obtained by increasing the number of pulses.

2.3. Experimental procedure

A Petri dish without cover with 15 mL of sample was placed on the stirrer below the centre of the lamp. This volume (2.7 mm height) was selected as the minimum required to fully covering the plate bottom. The distance between the lamp and the sample surface was 6.7 cm. Tests were performed based in a reaction mixture consisting in 20 mg/L of dye and 600 mg/L H_2O_2 dissolved in demiwater, at the natural pH of this mixture (9.15); this solution was named “standard reaction mixture” in order to easily describe the different experimental conditions. For decolourization measurements, an aliquot of 1.5 mL of sample was collected at specific fluence intervals, and the absorbance at 400 nm was measured. Afterwards, the aliquot was poured back into the Petri dish in order to keep constant the volume of sample. The same procedure was used to monitor changes in the UV–vis absorption spectrum.

Based in the standard reaction mixture, different tests were carried out changing dye concentration (0–25 mg/L), pH (2–13.5), or H_2O_2 doses (0–2000 mg/L). For the evaluation of pH effect, the solution of dye was adjusted to the different pHs by addition of HCl or NaOH.

Independent tests using the standard reaction mixture were performed for the determination of changes in chemical oxygen demand (COD), since this determination is destructive and does not allow sample reutilisation. Consequently, the COD destruction curve was built, for each fluence tested, from independent samples. To this, the standard reaction mixture was treated until reach the desired fluence, and then an aliquot of 2 mL of sample was collected and analysed for COD.

All tests have been carried out at room temperature (23 °C) at least per duplicate.

2.4. Analysis

Spectrophotometric measurements were carried out by using a UV–Vis spectrophotometer (UV-1700, Shimadzu, Japan). A 400 nm wavelength was selected to monitor decolourization because the dye solution exhibits its highest absorbance at this wavelength in the visible range (Fig. 2). The absorbance was linearly correlated

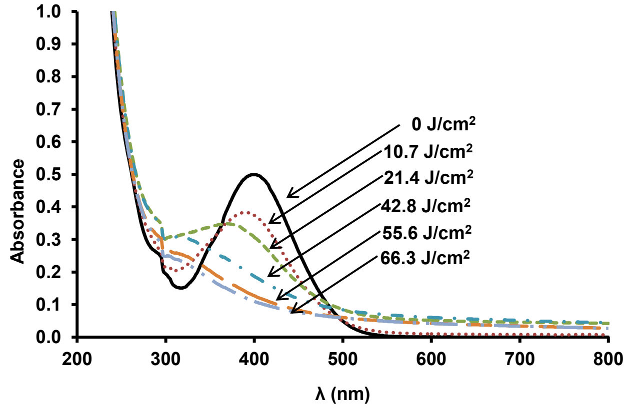

Fig. 2. UV–vis spectra of Direct yellow 106 undergoing a PL/H_2O_2 process. [dye] = 20 mg/L, $[H_2O_2]$ = 600 mg/L, pH = 9.15.

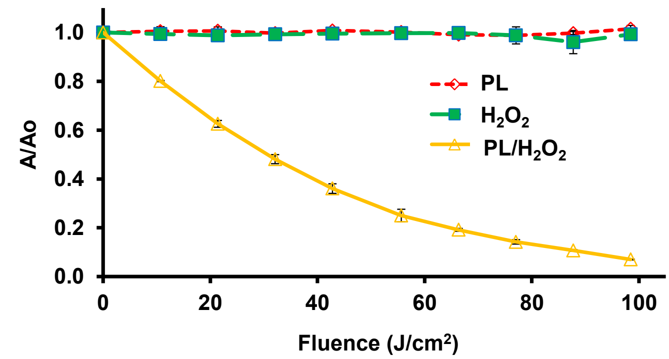

Fig. 3. Decolouration of Direct yellow 106 dye by PL/H_2O_2 in comparison with the effect of PL and H_2O_2 alone. Error bars means standard deviation.

(R^2 = 0.999) to dye concentration over the studied range. COD was determined by the closed reflux colorimetric method using Lovibond (Dortmund, Germany) ampoules and a Lovibond MultiDirect tintometer. Also, pH was measured by a Crison pH meter (Basic 20, CRISON, Barcelona, Spain). Temperature through treatments was monitored by an infrared thermometer (ScanTemp 410, TFA Dostmann, Wertheim, Germany). Amperage during the capacitor charging process was measured by a HT73 Digiclamp ammeter (HT Instruments, Barcelona, Spain).

2.5. Calculations

Absorbance at 400 nm vs. fluence data were regressed for the pseudo-first order kinetic using Eq. (1):

$$\ln A/A_o = -k\,F \quad (1)$$

where A is the absorbance at fluence F(J/cm^2), A_o the initial absorbance and k the constant rate (cm^2/J).

The figure-of-merit electric energy per order E_{EO} (kWh/m^3/order) was used to calculate the electric efficiency of the process using Eq. (2) [16]:

$$E_{EO} = \frac{Pt\,1000}{V\log\frac{c_i}{c_f}} \quad (2)$$

where *P* is the power (kW) of the system, *t* (h) is the duration of the current input to the PL system, V (m^3) is the treated volume, and c_i and c_f the initial and final dye concentrations (mg/L) respectively, obtained from the calibration curve; a factor of 1000 was used to convert litres in m^3. The power was estimated taking into account the Spanish electrical network voltage (220 V), and amperemetric measurements.

Data was analysed by using Excel 2010 (Microsoft, USA).

3. Results and discussion

3.1. PL direct photolysis and PL/H_2O_2 process

Firstly, the effect of PL alone on the decolourization of DY 106 was tested. As can be seen in (Fig. 3), the results obtained showed that PL not cause a direct photolytic effect on colour solution, maybe as explained before, due to the highly stable chemical structure of this dye. The use of H_2O_2 alone, which was tested to control its possible individual effect, has no influence on the decolourization process. However, when used as a part of a H_2O_2 AOP, PL is effective for decolourization. The PL/H_2O_2 process reaches nearly 90% of colour removal, showing faster initial colour degradation up to 40 J/cm^2 and a progressive declining of decolouration rate, which is typical of a first-order reaction commonly observed in the decomposition of dyes by an UV/H_2O_2 process. In this sense, the decolourization of the dye by the PL/H_2O_2 AOP can be described according to the widely known mechanism of generation of hydroxyl radicals upon photolysis of H_2O_2 according to Eq. (3) [17]; where the UV part of the PL emission provides the required photons for the generation of hydroxyl radicals. The hydroxyl radical show a high oxidation potential and can attack both the dye and the products of photolysis [18], by abstracting hydrogen atoms or by adding to double bonds [17].

$$H_2O_2 + h\nu \rightarrow 2{\bullet}OH \quad (3)$$

An unwanted side effect of PL is sample heating [11], therefore temperature is usually monitored during PL treatments to assess potential thermal effects. The temperature rise at the end of all tests was lower than 3 °C, thus ruling out any heat contribution to the process.

The UV–visible absorption spectrum of DY 106 has peaks at wavelengths of 400 nm and 204 nm (Fig. 2). The peak at 400 nm corresponds to the azo linkage [19] and the UV peak includes both the aromatic groups together with the background absorbance of H_2O_2. The change in absorption spectrum with the progress of the PL/H_2O_2 treatment (Fig. 2), concomitant with its degradation curve, shows the progressive disappearance of the peak at 400 nm, which can be ascribed to the attack of OH radical to the azo group leading to the opening of the –N=N- double bond [20]. No hypsochromic or bathochromic shifts have been observed, which may indicate limited changes in the generation of degradation products; although it can also be noticed that, the original dye exhibits a minimum absorbance in the UV range at 316–319 nm, that increases without forming a peak, with the progress of the treatment, becomes stable between 21 and 43 J/cm^2 and then decreases. The formation of new peaks or absorption bands in photolytic degradation of dyes is due to formation of the intermediate products [21].

3.2. Effect of dye concentration

The results of decolourization obtained at different initial dye concentrations are shown in Fig. 4. As can be seen in Table 1, pseudo-first order rate constants show that the decolourization rate under different dye concentrations are quite similar in the range of 5–15 mg/mL, decreasing at concentrations of 20 mg/mL.

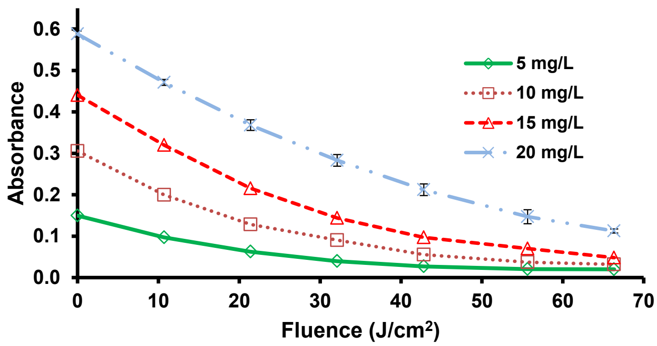

Fig. 4. Effect of dye concentration on the decolouration of Direct yellow 106 dye by PL/H_2O_2.[H_2O_2] = 600 mg/L, pH = 9.15. Error bars are smaller than symbols.

Table 1
Pseudo-first rate constants and figure-of-merit electric energy per order for decolourization of different concentrations of Direct yellow 106. [H_2O_2] = 600 mg/L, fluence up to 66 J/cm^2, pH = 9.15.

[Dye] (mg/L)	k (cm^2/J)	R^2	E_{EO} (kWh/m^3/order)
5	0.0320	0.96	827
10	0.0353	0.99	737
15	0.0338	1.00	750
20	0.0253	1.00	1006

It is known that higher dye concentrations produce slow decolourization rates, due to the lower light penetration [18]. However, the relative insensitivity of this process at low dye concentrations can be attributed to the thin sample film used in the experiments, just 2.4 mm thick, which allows a high light transmission through the sample. For example, according to the Beer-Lambert law, at 400 nm between 72 and 91% of light that penetrates the sample depend on dye concentration.

A remarkable finding that can be observed in Fig. 4 and others is that a high level of decolourization can be attained at 66 J/cm^2; this fluence is achieved by 31 light pulses. The PL system used in the current work has an important difference with others, is manually handled at 30–60 s time intervals since has not a cooling system to prevent lamp overheating. However, typical commercial PL systems can operate at a pulse repetition rate of 2–3 Hz [22,23]. That means that the energy required for the effects reported herein can be achieved in much less than 30 s, faster than conventional UV/H_2O_2 treatments that require many minutes or hours. This feature allow lower reactor residence times and a higher capacity for processing large volume of wastewaters.

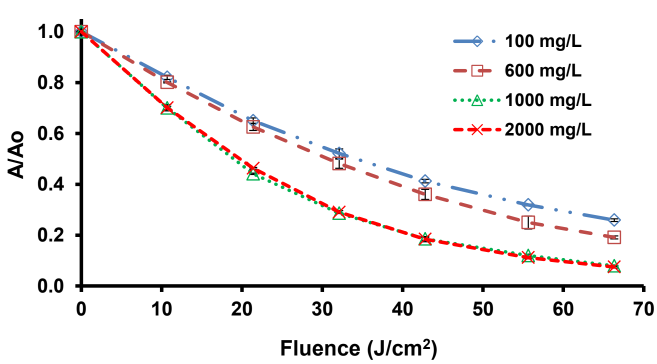

Fig. 5. Effect of H_2O_2concentration on the decolouration of Direct yellow 106 dye by PL/H_2O_2.[dye] = 20 mg/L, pH = 9.15. Error bars are smaller than symbols.

3.3. *Effect of H_2O_2 concentration*

The effect of different concentrations of H_2O_2 (0–2000 mg/L) in DY 106 decomposition was evaluated. As can be observed in Fig. 5, decolourization rate increases with increasing H_2O_2 concentrations up to 1000 mg/L, likely due to a higher generation of OH radicals. The decolourization seems to reach a maximum at 1000 mg H_2O_2/L because a concentration of 2000 mg H_2O_2/L does not yield a further increase. The pseudo-first reaction rate (Table 2) increases up to 88% when we increase the concentration of H_2O_2 from 100 to 1000 mg/L. However, only an increase of 3% was observed when H_2O_2 increases from 1000 to 2000 mg/L. It has been reported that at high H_2O_2 concentrations, the solutions undergo self-quenching of hydroxyl radicals [24] as represented in Eqs. (4) and (5) [17], or dimerize to H_2O_2 according to Eq. (6) [24].

$$\bullet OH + H_2O_2 \rightarrow H_2O + HO_2\bullet \quad (4)$$

$$\cdot OH + HO_2^- \rightarrow HO_2\cdot + OH^- \quad (5)$$

$$2\bullet OH \rightarrow H_2O_2 \quad (6)$$

3.4. *Effect of pH*

The effect of pH on decolourization of DY 106 was also assessed. Decolourization was favoured by low sample pH (Fig. 6), with small differences in the range 4.0–9.2, and an appreciable lower rate (Table 3) at pH 13.5. The natural pH of the solution is 9.15; therefore, its acidification might be used to increase the efficacy of the treatment. Other studies have reported similar results [24,25], that have been explained by the chemical properties of H_2O_2 and its photolysis products. Under alkaline conditions, H_2O_2 is converted to its less reactive conjugate base according to Eq. (7) [26], which decreases the concentration of the hydroxyl radical; moreover, hydroxyl radical deactivates at high pH by reacting with HO_2^- [24]. In contrast, an acidic medium allows a faster reaction of radicals [24].

$$2\bullet OH \rightarrow \bullet O^- + H_2O \quad (7)$$

3.5. *Mineralization*

The mineralization of the dye was tested using the COD as an indicator. COD determination measures the amount of oxidant that reacts with the sample under controlled conditions [27]; the mineralization of dyes decreases the concentration of organic compounds present in samples and consequently, the COD. It was observed an initial fast degradation reaching almost 50% up to 30 J/cm^2 (Fig. 7), which remains constant until the end of the treatment (up to 77 J/cm^2). This result could be a consequence of a fast degradation of the azo linkages of the molecule but a reduced degradation of highly recalcitrant uncoloured by-products, likely from the greatly stable aromatic part of the dye. The limited

Table 2
Pseudo-first rate constants and figure-of-merit electric energy per order for decolourization of Direct yellow 106 with different H_2O_2 concentrations. [dye] = 20 mg/L, fluence up to 66 J/cm^2, pH = 9.15.

[H_2O_2] (mg/L)	k (cm^2/J)	R^2	E_{EO} (kWh/m^3/order)
100	0.0206	1.00	1231
600	0.0253	0.99	1006
1000	0.0387	1.00	656
2000	0.0398	1.00	642

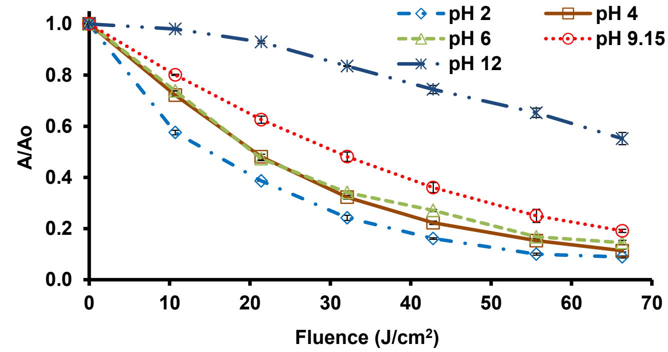

Fig. 6. Effect of pH on the decolouration of Direct yellow 106 dye by PL/H_2O_2.[dye] = 20 mg/L, [H_2O_2] = 600 mg/L. Error bars are smaller than symbols.

Table 3
Pseudo-first rate constants and figure-of-merit electric energy per order for decolourization of Direct yellow 106 at different pHs. [dye] = 20 mg/L, [H_2O_2] = 600 mg/L, fluence up to 66 J/cm^2.

pH	k (cm^2/J)	R^2	E_{EO} (kWh/m^3/order)
2.00	0.0410	1.00	688
4.00	0.0335	1.00	765
6.00	0.0300	0.99	862
9.15	0.0253	0.99	1006
13.50	0.0091	0.95	2798

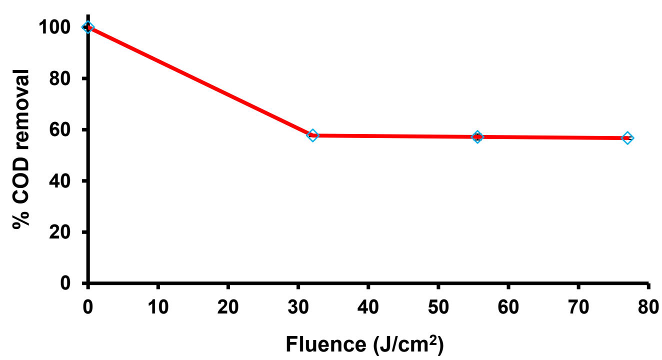

Fig. 7. Chemical oxygen demand (COD) removal through the progress of a pulsed light/H_2O_2 advanced oxidation treatment.[Direct yellow 106] = 20 mg/L, [H_2O_2] = 600 mg/L. Error bars are smaller than symbols.

mineralization level cannot be associated with a PL technology, since this is just a different way to generate hydroxyl radicals from H_2O_2. Similar results have been reported for other chemicals subjected to similar or different AOP, such as Acid orange 52 [28], Acid yellow 23 [21] and for a triazole [29]. Taking into account the variation of absorption spectra with the progress of AOP and incomplete mineralization (50%) achieved in this work, similar results have been reported by Neamtu et al. [30] for Reactive yellow 84 and Behnajady et al. [21], for Acid yellow 23 degradation with ZnO photocatalyst.

It is also possible that in addition to the aromaticity of the structure, its sulfonic moiety contributes to its stability, since it is known than the sulfonate group has a high electron withdrawing capacity that could help to stabilize aromatic species. No attempts to test mineralization under other conditions were done because complete mineralization would not be expected based on the findings that the mineralization degree observed for the standard reaction mixture was relatively low, the decolourization was not complete under any condition, and it is known that mineralization rates are always lower than decolourization rates [10,18,28,31].

3.6. Electric energy per order

The figure-of-merit E_{EO} was used to evaluate the electrical efficiency of the process. This figure was developed by an expert panel of the International Union of Pure and Applied Chemistry [16] for direct comparison of electric efficiency of AOPs, independently of their nature. The figure-of-merit E_{EO} is seldom reported for PL process, therefore it worth explaining how it was calculated in the current case. The original equation define its variables for a continuous process, where *Pt* is the energy billed by the power company and *t* the time invested to treat a volume *V* [16]. However, PL systems work in a discontinuous mode, where the electricity is taken from the mains during a relatively long time, to be stored in capacitors and then released to the lamp in a shorter time. Therefore, the time that the PL system takes electricity from the mains and the treating time are not neither simultaneous nor the same. In the current case, the duration of the light pulse is 200 μs but it takes 4 s to charge the capacitors to the desired voltage, this is the time (*t*) that must be used to calculate the E_{EO}. Since the measured amperage has been 1.43 A, the energy consumption is 0.011 kWh and the E_{EO} of this process for 20 mg/mL dye with 600 mg/mL H_2O_2 at pH 9.15 is 1009 kWh/m^3/order. This value is similar or smaller than those reported for other photolytic processes. For example, the E_{EO} for diazinon by a photocatalysis with ZnO nanocrystals is 1075–1389 kW h/m^3/order [31] and for the more complex dyes rhodamine B and methylene blue are 1500 and 3000 kWh/m^3/order, respectively [32].

The electric efficiency of this process can be improved by more than 30% to less than 700 kWh/m^3/order by either using a higher concentration of hydrogen peroxide (Table 2) or a lower pH (Table 3), both factors decrease the electrical energy necessary to decolourize DY 106 by the same magnitude. Using another type of system configuration such a flow-through reactor should render a higher energy efficiency because it will allow make profit of all photons generated by the lamp.

4. Conclusions

Pulsed light technology used alone has not effect on Direct yellow 106 degradation. However, when PL was used together H_2O_2 in an advanced oxidation process, it can degrade very fast the dye. The effects of this AOP can be explained by the formation of hydroxyl radicals as any other UV/H_2O_2 process, and follow a pseudo-first order kinetics. Decolourization increases with concentration of H_2O_2, reaching a maximum (asymptotic value) at high concentrations of oxidant agent. While a faster decolourization rate was observed at low pH, the concentration of dye showed a weak influence due to the thin film reactor configuration. A PL/H_2O_2 process may offer a much faster dye degradation rate than conventional CW UV/H_2O_2 processes, which should allow treating higher volume of wastewater. Additional tests including other dyes should be performed to further assess the potential of PL for dye degradation.

Acknowledgments

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