Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2019.24749

170 (2019) 361–368
December

Comparative study of the degradation process of Azo textile dyes AB52 and AB210 in water using a type-Corona electric discharge

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Received 30 April 2019; Accepted 24 July 2019

ABSTRACT

In this study, the effect of maintaining the same initial chemical and physical conditions in the mineralization process, (decomposition of organic pollutants in aqueous solutions to CO_2, mineral salts and water) was evaluated for 2 azo-type dyes (N=N) using a cold plasma as agent precursor to the mechanism of elimination of pollutants in water. This paper reports the treatment of water with Acid Black 52 (AB52) and Acid Black 210 (AB210), using as a reaction accelerator iron filing (analytical grade) (Fe^{2+}). The kinetics of the reaction was determined by UV/VIS spectrophotometry, at the beginning and during the treatment of the samples the total organic carbon (TOC) and chemical oxygen demand (COD) value was determined. The discoloration values in 180 min are greater than 95% and the values of TOC and COD decrease, more than 90% depending upon the time of treatment. In addition, the optical emission spectrum of the plasma was obtained to determine the species present in the ionization processes of the corona discharge. The energy yield value was calculated in terms of G_{50}, these results provide information for the subsequent application of this process to degradation of organic compounds.

Keywords: Corona discharge; Azo textile dyes; TOC

1. Introduction

The discharges of wastewater from the industry into natural tributaries have serious consequences for aquatic ecosystems [1,2], because most of them change the coloration under the influence of the effluents containing dyes. Once inside the system, contaminants hinder the photosynthetic process because they consume oxygen and cause the eutrophication of water bodies, promoting the generation of anaerobic processes in which greenhouse gases are produced [3]. Dyes are the substances that are used in the textile, food, pharmaceutical and paper industries, these compounds have chromophore groups that are responsible for coloring the substance. According to the chromophore group, they are classified as azo dyes, arylmethanes, anthraquinones, among others [4]. Additionally, azo dyes are toxic, carcinogenic and mutagenic [5].

In a simplified way, conventional methods for wastewater treatment can be classified into biological and physicochemical methods, the last includes adsorption, coagulation, flocculation, sedimentation, filtration, among others. The main disadvantage of these is that they do not include

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any chemical reaction that could lead to a complete mineralization of the contaminant, but instead they produce secondary waste. On the other hand, the chemical methods, among which the advanced oxidation processes (AOP's) tend to be more efficient since they focus on the production of hydroxyl radicals ($OH^•$), highly reactive species, which promote the oxidation of organic compounds [6]. Within AOP's, plasma treatment at atmospheric pressure results in high efficiency of elimination of compounds, since it combines different physical and chemical effects applied to the system, such as pyrolysis, UV photolysis, electrohydraulic cavitation, as well as the formation of oxidizing species that favor the degradation of pollutants, such as radicals H, O, $OH^•$ and molecules H_2O_2, O_3, among others [7].

Several methods have been used to remove these contaminants from the water. Adsorption by activated carbon only changes them from one phase to another, which is also contaminated [8]. Biological processes are difficult to apply due to the toxicity of these compounds, in addition to, not achieving complete degradation of the dye, generating by-products even more toxic than the original contaminant [9]. The AOP's are based on the generation of $OH^•$. These radicals can degrade and even mineralize a wide variety of organic contaminants, including dyes [10–12].

The dyes AB210 and AB52 (Table 1) are azo dyes (N=N), these are classified by the color index (CI) and chemical abstracts service (CAS) number. They are used in different industries that produce paint, ceramics, plastic, leather, ink; in addition, in the textile industry, in the dyeing of fibers; which have a high resistance to washing, so that their disposal of wastewater cannot be done by conventional methods, being necessary the use of AOP's. Currently, several AOP's are used as alternatives for the degradation of organic pollutants, for example: ozone (O_3), O_3 and hydrogen peroxide (H_2O_2), O_3 and ultraviolet radiation (UV), Fenton (H_2O_2 + Fe^{2+}), photocatalytic Fenton process (H_2O_2 + Fe^{2+} + UV); H_2O_2 and UV [14] and cold plasma [15,16]. In the literature there are reported studies on the elimination of organic pollutants, using atmospheric plasma as a degradation method, having an acceptable efficiency compared to other processes [17–20].

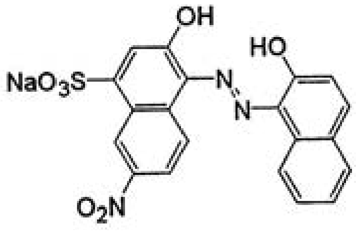
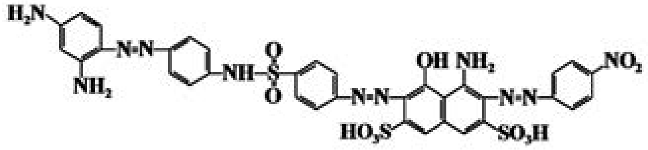
The AB210 and AB52 are used in the dyeing of wool, cotton and leather. Incomplete degradation of this compound can generate 4-nitroaniline, which is suspected to cause cancer in humans [21]. For this reason, the study of the process of degradation of dyes in water in controlled systems is of great interest. In this work, the degradations of Azo-type dyes AB52 and AB210 are studied when interacting with plasma generated at atmospheric pressure in the liquid-air interface.

2. Methodology

2.1. Experimental system

An experimental system was designed and built to produce a Corona-type electric discharge on liquid samples with organic pollutants, where samples of textile dyes are treated by means of a cold plasma at atmospheric pressure [2,15]. The luminescent discharge is generated with a direct current power supply (HP Mod. 6525 A, 4 kV–50 mA) keeping the applied voltage constant 2 kV, and current of 40 mA, at an 80 W constant electric power. Sampling was run at regular intervals of time increasing every 30 min, from 0 to 180 min. Fig. 1 shows the components of the experimental system used in the degradation of textile dye AB52 and AB210. The electrodes are tungsten rods, the upper electrode is movable and is adjusted in the center of the sample, 250 mL can be treated in the batch reactor. The pH and electrical conductivity (EC) measurements were performed with a HACH HQ40d multimeter (Loveland, Colorado, USA); using a HACH DR3900 spectrophotometer (Loveland, Colorado, USA) the absorbance was measured, using reagents low-ranking brand HACH (Loveland, Colorado, USA) chemical oxygen demand (COD) and total organic carbon (TOC) were determined.

Table 1
Molecular structure of the dyes AB52 and AB210 [2,13]

Dye	Molecular structure	Molecular weight (g/mol)	$\lambda_{máx}$ (nm)
AB52 $C_{20}H_{12}N_3O_7NaS$ CI: 15711 CAS No.: 5610-64-0		461.38	570
AB210 $C_{34}H_{25}K_2N_{11}O_{11}S_3$ CI: 300825 CAS No.: 99576-15-5		938.02	320 465 607

2.2. Preparation of solutions

A solution at 1.0 mM of the AB52 dye was prepared in a volume of 250 mL, to this solution was added to the necessary amount of iron filing (catalytic agent) to have a concentration of 1.0 mM. In an analogous and independent manner, the same procedure was carried out for the AB210 dye.

The initial and during treatment absorbance of the solutions were measured using a HACH DR3900 spectrophotometer (Loveland, Colorado, USA). Measurements of pH, EC, and temperature are made at the beginning and during the treatment with a HACH mod. HQ40d multimeter (Loveland, Colorado, USA). The Hach method (Loveland, Colorado, USA) was used to determine TOC and COD. To obtain TOC, medium-range vials (15–150 mg/L) were used, while low-level vials (3–150 mg/L) were used for COD. The vials were measured in a HACH Mod. 3900 spectrophotometer (Loveland, Colorado, USA). The analysis was carried out every 30 min for 3 h. All solutions were made from analytical-grade chemicals; the experiment was performed 5 times under the same initial conditions.

2.3. Characterization of plasma

2.3.1. Optical emission spectroscopy

Optical emission spectroscopy of the plasma was carried out using a StellarNet EPP2000 spectrometer (Tampa, Florida, USA) at a resolution of 1.5 nm. The light emitted by the plasma is taken to the spectrophotometer by means of an optical fiber. The spectrometer operates in a wavelength range of 200–1,100 nm, with integration time of 1,000 ms and 5 samples were averaged for the collection of spectra. The emission spectra were qualitatively analyzed to assign chemical species to the peaks.

For describe the plasma used for the treatment, it is possible to calculate the plasma electron temperature and the electron density using the intensities of several spectral lines assuming that the population of the emitting levels follows the Boltzmann distribution [22] and considering that the system has a local thermodynamic equilibrium (LTE) in some small fraction of this. For calculating electron temperature (T_e), the following expression was used [22].

$$T_e = \frac{E_m(2) - E_m(1)}{k}\left[\ln\left(\frac{I_1\lambda_1 g_m(2)A_m(2)}{I_2\lambda_2 g_m(1)A_m(1)}\right)\right]^{-1} \tag{1}$$

where E_m (i) is the energy of the upper levels of the lines, k is the Boltzmann constant, g_m (i) is the statistical weight of the upper levels and A_m (i) is the corresponding transition probabilities. I_1 and I_2 are the relative line intensities of lines in questions, λ_1 and λ_2 are wavelength of the lines, which were experimentally measured. Also, it is possible to obtain the value of the electron density of the plasma used in the treatment using the value of T_e. The electrons are responsible for most of the processes that are taking place in the Corona discharge, ionization, dissociation and recombination processes with water molecules. In this paper, for calculating the electron density, the Saha–Boltzmann equation was used [23–25].

$$n_e = 6\times 10^{21}\left(T_e\right)^{\frac{3}{2}}\left(\exp\left[-E_i / kT_e\right]\right) \tag{2}$$

where E_i is the ionization energy of the species, and k is the Boltzmann constant.

2.4. Characterization of treatment

The percentage of degradation is calculated using Eq. (3).

$$\%\ \text{Degradation} = \left(1 - \frac{C}{C_0}\right)\times 100\% \tag{3}$$

where C is the concentration measured of dye at a certain time of treatment and C_0 is the initial concentration of the dye; the concentration C was obtained from the calibration curve (C vs. Abs) of each dye, as described in previous studies [15].

3. Results

Fig. 2a shows the absorption spectra of each dye before being exposed to the treatment, identifying the most intense

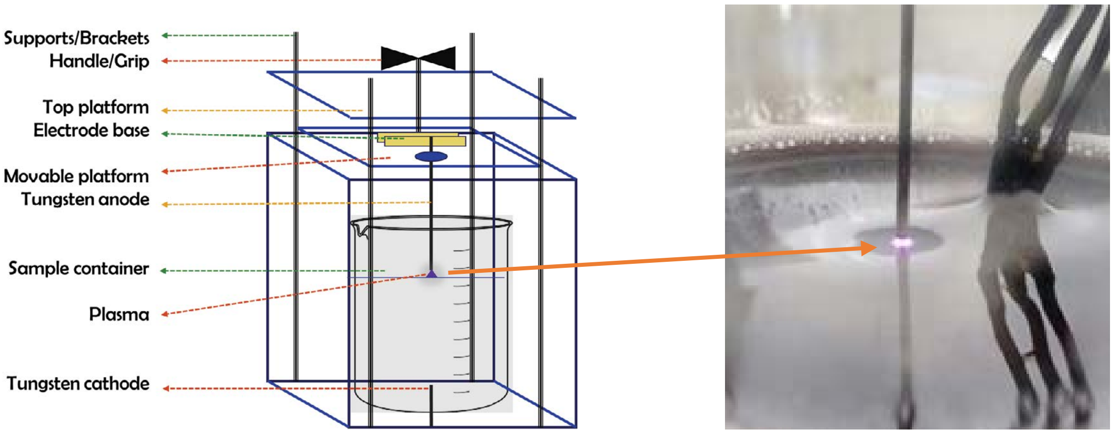


Fig. 1. Reactor for the treatment with atmospheric pressure plasma.

peaks at 570 for the AB52, while for the AB210 dye are in 465 and 607 nm. In addition, the absorption spectra are presented during the degradation process of the dyes AB210 (Fig. 2b) and AB52 (Fig. 2c). In Fig. 2b the gradual decrease of the absorbance of the dye AB52 is observed as a function of the treatment time, in Fig. 2c is identified that the absorption bands characteristic of the AB210 dye gradually decrease when the solution is treated by plasma.

The appearance of 1 peak for the AB52 and 2 peaks for the AB210 in the study region (320–800 nm) are due to Azo-type bonds (N=N), the AB52 has only one azo-type bond, while the AB210 has 3 (in the literature 320, 465 and 607 nm) [13]. When the plasma interacts with the solution, chemical variables of the solution, such as pH and EC, are affected, because of the system is supplied with high energy electrons, causing ionization, dissociation and recombination processes with water molecules, generating $OH^\bullet$ and $H^\bullet$ radicals, as described in reaction R1 [26]:

$$H_2O + e \rightarrow OH^\bullet + H^\bullet + e \tag{R1}$$

The recombination of $OH^\bullet$ radicals in the solution produces hydrogen peroxide, reaction R2 [27]

$$OH^\bullet + OH^\bullet \rightarrow H_2O_2 \tag{R2}$$

In this study iron filings were used as a catalyst, then for organic oxidation, the Fe^{2+} can directly involve in the catalytic formation of hydrogen peroxide into hydroxyl radicals in R3 [26]

$$H_2O_2 + Fe^{2+} \rightarrow OH^\bullet + OH^- + Fe^{3+} \tag{R3}$$

On the other hand, Fe^{3+} is transformed into Fe^{2+} consuming the hydrogen peroxide generating for the plasma

$$H_2O_2 + Fe^{3+} \rightarrow H^+ + Fe^{2+} + HO_2^\bullet \tag{R4}$$

Considering this type of reactions in the treatment by plasma (R1–R4), the generation of the hydroxyl radical can mineralize the initial organic compound and its by-products, as described by the reaction R5 [26].

$$\text{Intermediates} + OH^\bullet \rightarrow CO_2 + H_2O \tag{R5}$$

In Fig. 3a, it shows that the pH decreases gradually from 2.44 to 2.04 as a function of the treatment time for azo dye AB52, its behavior is decreasing and smooth. Regarding the EC, it is observed that in 180 min of treatment the magnitude of EC changes from 406 to 738 µS/cm for the AB52; while for the AB210 the pH starts at 8.27 and reaches 3.2; while the EC ranges from 90.7 to 221.9 µS/cm (Fig. 3b).

The pH does not adjust in the solutions, because in this work the purpose was to identify the effect of the use of the plasma and Fe^{2+} treatment only, and not to use another acidic solution to adjust the initial pH. This variation is because when the plasma is applied to the solution, hydrogen ions [H^+] are generated that lead to an increase in the acidity of the medium and, consequently, the EC is directly proportional

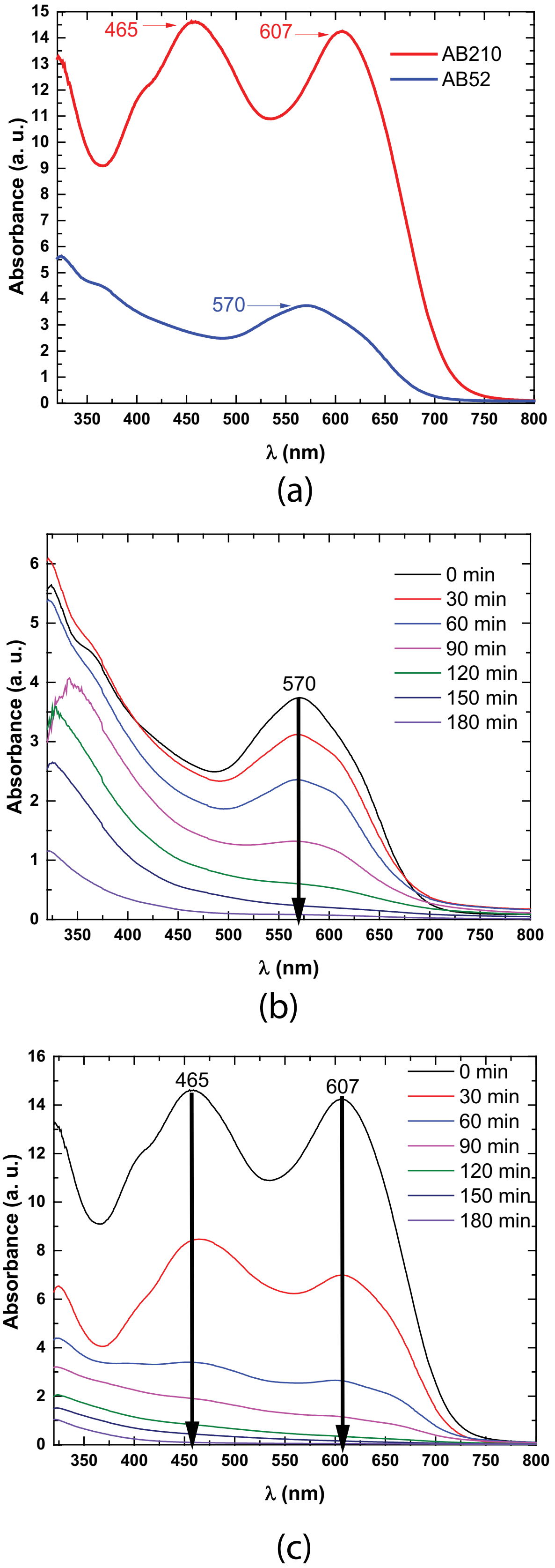
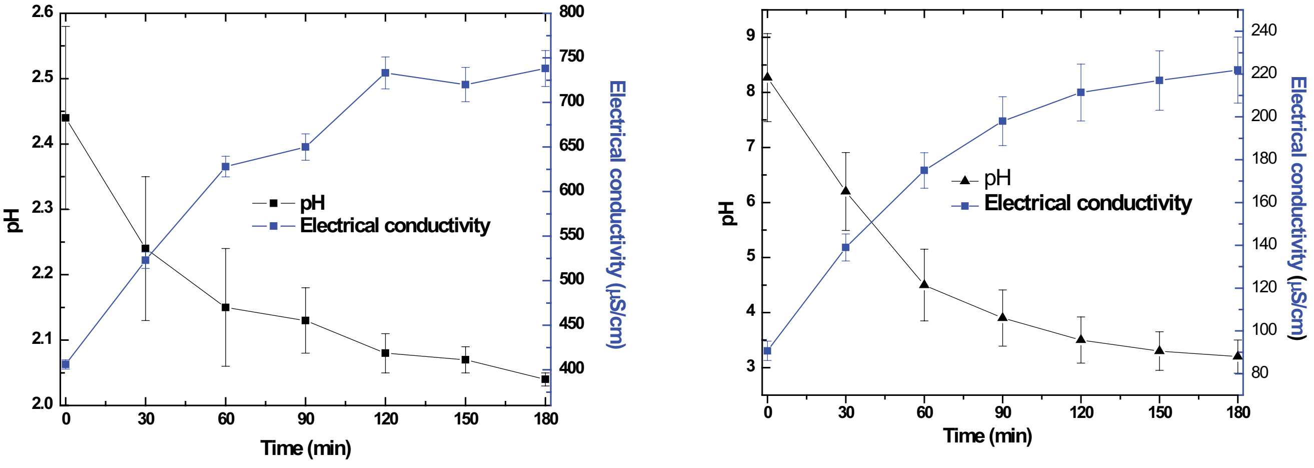


Fig. 2. (a) Initial absorption spectrum of the dyes, (b) degradation process of AB210 and (c) degradation process of AB52.



(a)

(b)

Fig. 3. Behavior of pH and electrical conductivity in the process of degradation of the 2 dyes (a) AB52 and (b) AB210, by plasma.

to the ion concentration of ions. When applying the plasma in the solution, it raises its temperature, as reported in previous studies [2,15] the temperature of the solution increases gradually as a function of the treatment time in a range of approximately 20°C–90°C, from 0 to 180 min of interaction with the plasma, without affecting the degradation of the contaminant. This increase in temperature has a direct effect on the increase of the EC and decrease in pH value, when the temperature increases the viscosity of the water decreases, causing a higher value of EC.

Fig. 4 shows the comparison of the removal percentage of the dyes under the same initial conditions, concentration of the dye and the catalyst, the percentage of degradation is calculated using Eq. (3); observing that AB210 has a greater resistance to being removed in the first minutes of treatment, this is due to the fact that having an azo triple bond, the energy necessary to dissociate the molecule is greater and for this reason the time for its degradation is greater; in 30 min the AB52 has been eliminated from the solution by 59.8%, while the AB210 51.3%, and in 60 min 87.9% and 82.6% for AB52 and AB210, respectively, after 180 min of treatment, the percentage degradation values are very similar and greater than 98%; which makes evident the efficiency of the degradation by plasma.

In order to corroborate that the organic compound has been mineralized and not only have the solutions been discolored, the TOC and COD of the samples under treatment are determined (Fig. 5). The initial values of TOC and COD were determined as described in [15], the initial value being higher for AB210 because it has more carbon and oxygen atoms, 34 and 11 respectively, while the AB52 has only 20 carbons and 7 oxygens.

In Fig. 5 is observed the decreasing behavior in exponential form of the TOC as a function of time for the 2 dyes, this as a consequence of exposure to luminescent discharge, and this suggests that there is a decomposition of the pollutant into the CO_2 form, that is, the pollutant is mineralized in carbon dioxide and water. Equally, the COD was determined, which shows a descendant behavior similar to the TOC, which allows to infer a decrease in the concentration of pollutants as a function of the time of interaction with the plasma. The COD provide information about the necessary oxygen for oxidize the organic matter in the solution, in these cases the COD decrease exponentially.

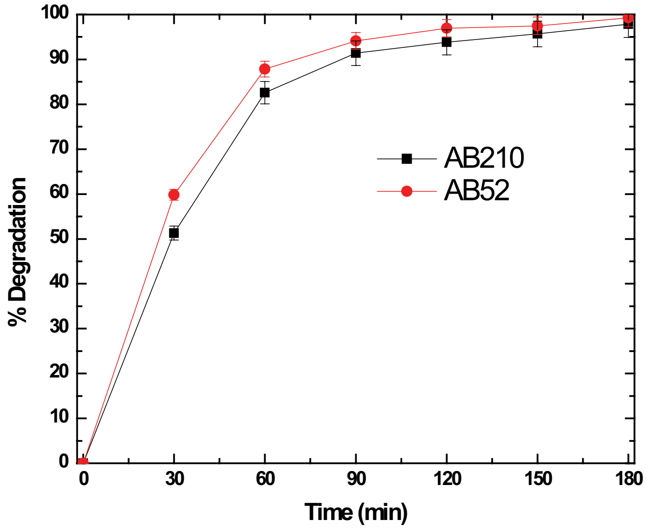
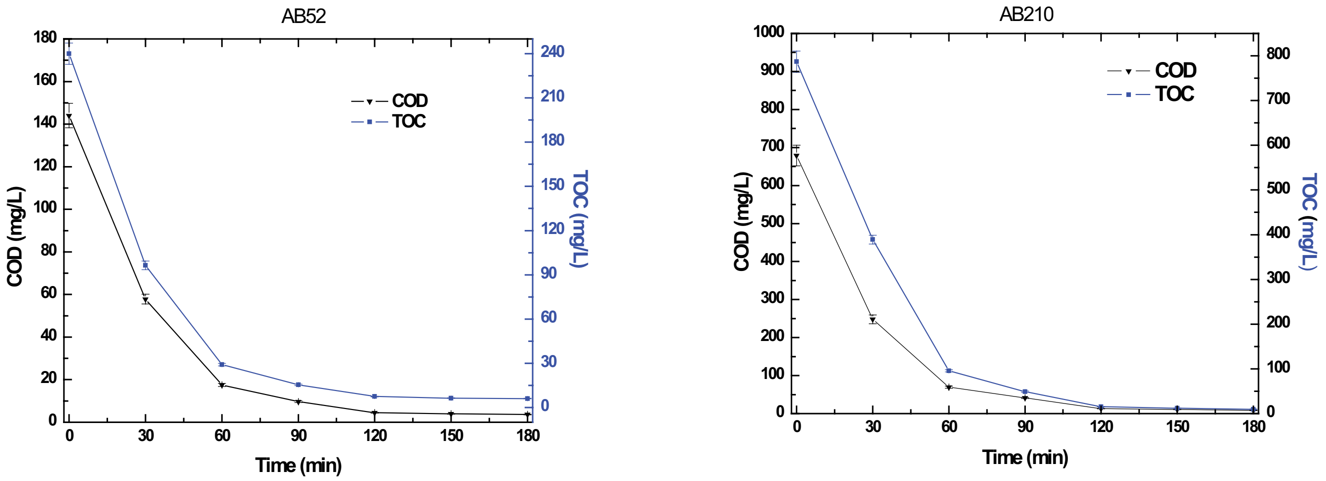


Fig. 4. Percentage of degradation of the dyes as a function of the time of treatment with plasma.

The TOC provides a general idea about the degradation process of the parent compounds (AB52 and AB210) and their byproducts as an ensemble; this amount can be interpreted as an approximation of the quantity of contaminant present in the sample. Fig. 6 shows that the rate of change in TOC/TOC_0 (normalized TOC) is different only in the first 30 min of treatment, being yield 9.3% greater for removal of the AB52, however during the rest of treatment, the values are very similar.

By fitting the values shown in Fig. 6 for TOC/TOC_0 with an exponentially decaying function ($y = e^{-kt}$) with $R^2 > 0.99$,



(a)

(b)

Fig. 5. Behavior of the chemical oxygen demand and total organic carbon during the treatment, (a) AB52 and (b) AB210.

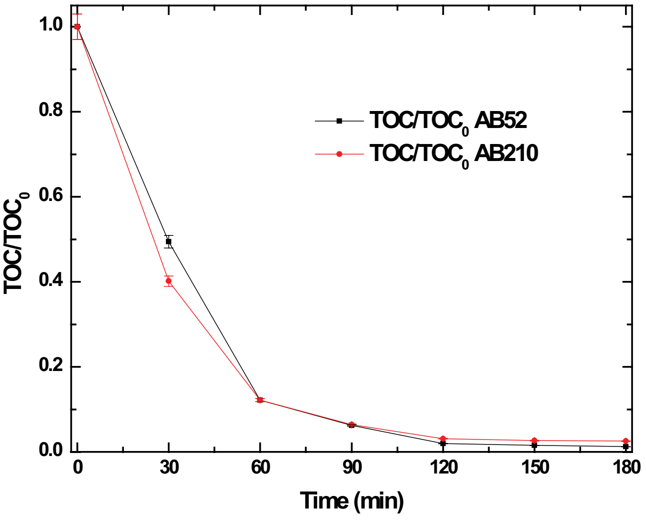


Fig. 6. Behavior of the TOC/TOC$_0$ during the plasma treatment.

the time constant for degradation (τ) of each compound can be obtained (Fig. 7). The results show that the time constant for the degradation of AB52 is lower than that of AB210. This confirms that the AB210 dye has a greater resistance to plasma degradation, compared to the AB52. This value can also be understood as the time necessary to eliminate half of the dye $t_{1/2}$.

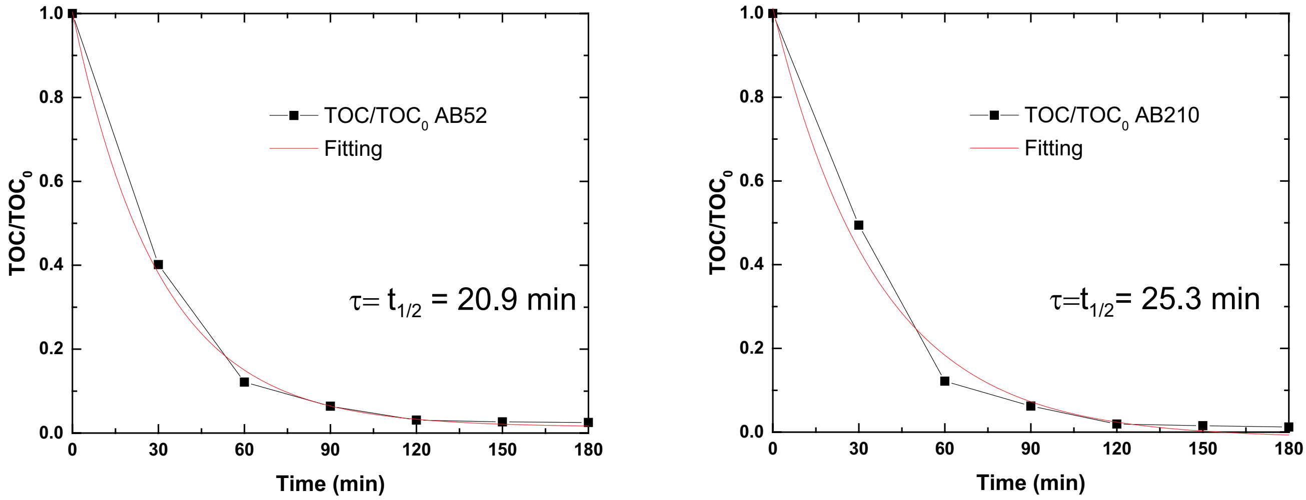
The energy yield value was calculated in terms of G_{50} [28], this yield value expresses the amount of pollutant converted in function of the energy supplied and required for 50% conversion of the pollutant:

$$G_{50} = 1.8 \times 10^6 \frac{C_0 V_0 M}{P t_{50}} \tag{4}$$

where C_0 is the molar concentration of the pollutant at $t = 0$, V_0 is volume of treated solution in L, M is molecular weight of the pollutant, P is electrical power of the reactor in W, and t_{50} is the time in s required for 50% conversion. G_{50} is expressed in g/kWh.

For the dye Acid Black 52; the initial concentration was $C_0 = 0.001$ M, initial volume was $V_0 = 0.25$ L, $M = 461.38$ g/mol, the electrical power was $P = 80$ W and using the time constant for degradation ($\tau = t_{1/2} = t_{50}$), $t_{50} = 1{,}254$ s. Substituting in Eq. (4), the calculated value of the G_{50} for this process is 2.07 g/kWh. For the dye Acid Black 210; the initial concentration was $C_0 = 0.001$ M, initial volume was $V_0 = 0.25$ L, $M = 938.02$ g/mol, the electrical power was $P = 80$ W and $t_{50} = 1518$ s. Substituting in Eq. (4), the calculated value of the G_{50} for this process is 3.47 g/kWh. These results indicate that the energy yield value is greater for AB210; this can be understood because the AB210 dye has a higher molecular weight and the plasma treatment takes almost the same time to degrade 50% of the contaminant. These values are similar to reported in the literature, for example for methyl orange G_{50}, with similar plasma reactor, was 0.024 g/kWh [17] and G_{50} value of 1.57 g/kWh was obtained by sonochemical for AB210 [29].

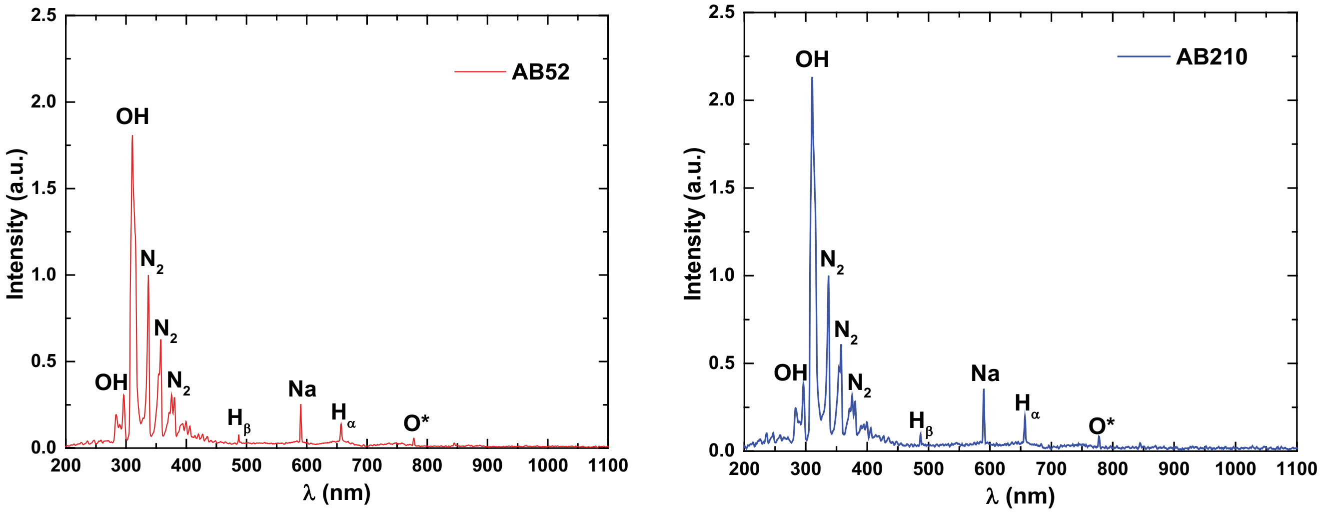
The energy supplied to generate the plasma produces several chemical species in excited states. These species in the gas phase were investigated using optical emission spectrum (OES) during corona discharge. Fig. 8 shows the emission spectrum in the wavelength range of 200–1,100 nm for the water treatment reactor by means of an atmospheric air plasma containing a dye sample (AB52 or AB210) at 80 W. Observing the emission spectrum of the plasma for both dyes, it is known that the highest emission occurs in the region close to ultraviolet radiation (250–400 nm). N_2 band of second positive system ($C^3\Pi_u$ — $B^3\Pi_g$) in 337.0, 357.5 and 375.5 nm were identified, It is also possible to identify the characteristic doublet of sodium in the spectra with pollutant (D-lines) in 590 nm. In both treatments, the band with the highest intensity belongs to the OH at 309.5 nm. In addition, H_α (656.5 nm), H_β (487.0 nm) were observed and low intensity; emissions from singlet O* are noted at 777 nm [30].



(a)

(b)

Fig. 7. Fitting curve of the TOC/TOC$_0$, (a): AB52 and (b) AB210.



a

b

Fig. 8. Optical emission spectrum of plasma by (a) AB52 and (b) AB210 treatment.

To determine the temperature of the applied plasma, in the process of degradation of the dye in the water, the values of H_α and H_β were considered and substituted in Eq. (1), obtaining a value of 3.4 eV for AB52 and 8.4 eV for AB210. It is possible to understand this difference in the magnitudes of T_e as a consequence of a greater number of hydrogen atoms in the solution with AB210 (25) compared with the solution with AB52 (12), remembering that the power applied to produce the plasma in both cases is 80 W.

Considering the value obtained from electron temperature and hydrogen ionization energy and substituted in Eq. (2), the value obtained from the electron density of the plasma is 8.69×10^{12} particles/cm^3 for AB52 and 3.68×10^{14} particles/cm^3 for AB210, the values obtained in this work are similar that the reported in the literature for a plasma at atmospheric pressure, electron density is around 10^{14} cm^{-3} and the electron temperature in the range of 1–10 eV [31].

5. Conclusions

With the results obtained using these 2 azo dyes, it is possible to ensure that the mineralization takes place in the form of CO$_2$ and water; The use of catalyst in the form of filings is very convenient to use, because it is possible to remove them from the final solution after of treatment. Through OES the species: OH, N$_2$, Na, H_α, H_β and O*, present in the plasma, are identified, in the spectrum the intensity of the OH band being greater for the treatment with AB210. The electron temperature and electron density calculated for the plasma used for the treatment is greater for the treatment of AB210. The energy yield value calculated, like other parameters, is higher for the degradation of AB210. In 60 min of treatment more than 80% of the dye has been mineralized, which would allow using a complementary treatment to degrade the rest of the dye and only use the plasma for 1 h. The pH as well as the final EC values suggest that the water obtained in this treatment requires a pretreatment before it can be used for any purpose. These results could be used to evaluate the degradation of other dyes of the same family (Azo type).

Acknowledgments

The authors would like to thank the projects DGAPA [IN-02916], CONACyT [268644], PROMEP [103.5/13/6626], PRODEP [DSA/103.5/15/6986], PII-43/PIDE/2013, 4307/2017/CI UAEMex and PRODEP [CA-5511-6/18-8304].

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