



Discoloration of a textile dyes mixture in an aqueous solution using a corona discharge

Josefina Vergara Sánchez^a, César Torres Segundo^{a,*}, Esteban Montiel Palacios^a, Aarón Gómez Díaz^b, Pedro Guillermo Reyes Romero^b, Horacio Martínez Valencia^c

^aLaboratorio de Análisis y Sustentabilidad Ambiental, Escuela de Estudios Superiores de Xalostoc, Universidad Autónoma del Estado de Morelos, Xalostoc, Ayala, Morelos, C.P. 62715, México, emails: cesar.torres@uaem.mx (C.T. Segundo), vergara@uaem.mx (J.V. Sánchez), esteban.montiel@uaem.mx (E.M. Palacios)

^bLaboratorio de Física Avanzada, Facultad de Ciencias, Universidad Autónoma del Estado de México, Instituto Literario No. 100 Col. Centro, Toluca, Estado de México, C.P. 50000, México, emails: agomezd@uaemex.mx (A.G. Díaz), pgrr@uaemex.mx (P.G.R. Romero)

^cLaboratorio de Espectroscopia, Instituto de Ciencias Físicas, Universidad Nacional Autónoma de México, A. P. 48-3, Cuernavaca, Morelos, C.P. 62251, México, email: hm@icf.unam.mx (H.M. Valencia)

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ABSTRACT

A batch reactor and a corona discharge were used for the degradation of a mixture of azo dyes, Acid Black 52 and Acid Black 210, in an aqueous solution using iron filings (Fe^{2+}) as a catalyst. The plasma at atmospheric pressure was generated on the surface of the liquid using a constant electrical power of 80 W. The physicochemical properties of the liquid, electrical conductivity, pH, volume, and temperature were measured. Ultraviolet-visible spectrophotometry was used to determine the kinetics of the oxidation process. In addition, optical emission spectroscopy, CO_2 production, total organic carbon (TOC), and chemical oxygen demand experiments were also carried out. The dye mixture illustrated discoloration of 95%–99% at the treatment time of 180 min and a 92% reduction in TOC. In the optical emission spectrum of the plasma, species such as OH, H_α , H_β , N_2 , and Na were identified. The electron temperature and electron density of the plasma were also calculated.

Keywords: Plasma; Textile dyes; Total organic carbon; Chemical oxygen demand; Optical emission spectroscopy

1. Introduction

A large amount of water used in the industry is poured into natural tributaries, which causes serious damage to aquatic ecosystems, mainly because the color of the water blocks the entry of sunlight necessary for photosynthesis, reducing dissolved oxygen levels [1]. Of the total amount of water used in the industry worldwide, 25% is used in the textile industry [2]. The wastewater from the textile industry and tanneries with ineffective treatment mechanisms contains large amounts of non-biodegradable

organic chemicals. These chemicals, most notably dyes, are extremely harmful to the environment. In the absence of proper treatment, dyes contaminate large volumes of water. Dyes hamper the photosynthetic process of the aquatic systems because they consume oxygen and cause the eutrophication of water bodies; they also create anaerobic conditions releasing greenhouse gases [3]. Dyes are stable and toxic compounds. As a result, their degradation tends to be incomplete in wastewater treatment plants (WWTPs), which operates using conventional physical, chemical, and biological methods.

* Corresponding author.

There are other methods for the removal of textile dyes from water. The first method requires the use of activated carbon as an absorbent medium. This method has yielded promising results due to its high porosity, high surface area, large volume of mesopores, and relatively high adsorption rates [4]. The second involves technologies based on adsorption or filtration membranes. These technologies have attracted considerable attention due to their efficient, cost-effective, and ecological attributes, for example, the reported modification of wool fibers for the efficient adsorption of dyes [5]. The third concerns the use of nanoparticles of an organic or inorganic origin. Nanoparticles have high porosity, small sizes, and large surface areas, which maximize their interaction with dye molecules, hence high adsorption efficiency [6].

At present, several advanced oxidation processes (AOPs) are used as alternatives for the degradation of organic dyes, namely, ozone (O_3) and hydrogen peroxide (H_2O_2), Fenton ($H_2O_2 + Fe^{2+}$), photo-Fenton process ($H_2O_2 + Fe^{2+} + UV$), O_3 , and ultraviolet (UV) radiation, H_2O_2 , and UV [7], and cold plasma [8]. Such processes rely on the formation of hydroxyl radicals, which are capable of oxidizing contaminants to smaller and less polluting molecules or even mineralizing them, turning them into CO_2 , H_2O , and inorganic ions from atoms. Plasma can be defined as a partially or fully ionized gas that contains electrons, ions, and neutral particles; it can be produced through a wide variety of electrical discharges in the laboratory [9].

There also exist a great variety of methods that can be used to eliminate contaminants in water. These methods are classified into three groups: primary treatments, for example, aeration, neutralization, and sedimentation; secondary treatments, for example, coagulation and flocculation; and a third group that is subdivided into (a) absorption, (b) separation, and (c) oxidation, which contain biological, chemical, and cold plasma [10]. In this study, a corona discharge, a kind of atmospheric cold plasma, was applied for the

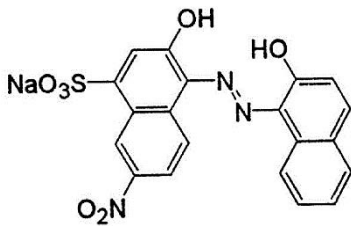
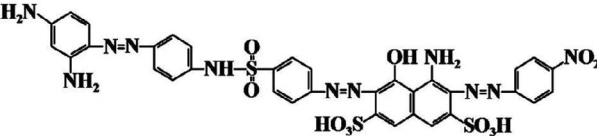
degradation of dyes in water. This plasma was produced by a DC voltage power supply.

This type of cold plasma at atmospheric pressure is an AOP in which different oxidizing species such as radicals (H^* , O^* , and OH^*) and molecules (H_2O_2 , O_3) are produced. Also, in this AOP, different physical and chemical effects such as pyrolysis, UV photolysis, and electrohydraulic cavitation combine. The use of plasma at atmospheric pressure in the treatment of wastewater is known for its excellent performance in the degradation of organic pollutants and, also, because it does not cause secondary contamination [11]. The effluents produced by industries, particularly the textile industry, contain a large variety of mixed contaminants used in the dyeing process, which renders the study of the elimination of mixed contaminants of the above effluents necessary [12–16].

Total organic carbon (TOC) represents the amount of carbon found in an organic compound (in this case, the dye). An alternative way to measure the mineralization of the dye is to determine the carbon dioxide released during the plasma treatment, because, through the oxidation process, the carbon present in the organic compound is gradually fractionated into molecules of simpler compounds (e.g., CO_2 in the form of gas), hence the importance of its measurement for wastewater treatment. The dyestuffs used in the textile industry and tanneries, particularly AB52 and AB210 with respective molecular weights of 461.38 and 938.02 g/mol, are toxic and highly resistant to washing; therefore, they cannot be eliminated through the conventional processes used in WWTPs. Thus, it is necessary to use AOPs. The dyes were obtained from a textile company where they are used to dye leather and wool. Both AB52 and AB210 dyes have at least one azo group ($-N=N-$) in their molecule. This chromophore group is responsible for the absorption of light, yielding the molecule color. The molecular structure of these dyes is described in Table 1 [17,18].

This study evaluates the effect of corona discharge on the degradation of the AB52/AB210 mixture in an aqueous

Table 1
Molecular structure of the Acid Black 52 (AB52) and Acid Black 210 (AB210) dyes

Dye	Molecular structure	Molecular weight (g/mol)	Maximum wavelength λ_{max} (nm)
AB52 C20H12N3O7NaS Color Index No. 15711 CAS No. 5610-64-0		461.38	570
AB210 C34H25K2N11O11S3 Color Index No. 300825 CAS No. 99576-15-5		938.02	465 and 604

solution at an initial concentration of 0.5 mM each by atmospheric plasma produced at the liquid–air interface. In this investigation, iron filing (Fe^{2+}) was used to accelerate the reaction. The reaction kinetics was determined by UV-vis (visible) spectrophotometry, TOC, and chemical oxygen demand (COD). The results are expected to provide useful information on the subsequent application of the present process to wastewater treatment on an industrial scale.

2. Methodology

2.1. Experimental system

A batch reactor was modified for the generation of stable atmospheric cold plasma in liquid samples (Fig. 1). The corona discharge was produced with a DC power supply (Mod HP 6525A, 4 kV–50 mA) at a constant electrical power of 80 W during the treatment. The plasma treatment time was between 15 and 180 min. The sample to be treated comprised 0.5 mM AB52, 0.5 mM AB210, and 1.0 mM iron filing. The initial volume of the sample was 250 mL. A Hach HQ40d (United States) potentiometer was used to perform the pH and electrical conductivity measurements, and a Hach DR3900 spectrophotometer (United States) was used to measure the absorbance in the range of 320–800 nm. COD and TOC were determined using Hach's low-ranking reagents (United States).

The experiment was carried out in the Environmental Analysis and Sustainability Laboratory of the Autonomous University of the State of Morelos.

2.2. Preparation of the AB52/AB210 mixture

Both AB52 and AB210 dyes were weighed on an analytical balance and were dissolved in water in a beaker. This mixture was placed in a volumetric flask. Iron filing (Fe^{2+}) was added, and the volume was adjusted to 250 mL. The concentrations in the mixture contained 0.5 mM AB52, 0.5 mM AB210, and 1.0 mM iron filing. The pH was adjusted to 6.0. This was then stirred to mix the sample. A Hach HQ40d multimeter (United States) was used to measure the pH, electrical conductivity, and temperature. The initial temperature was 25°C, and the initial pH remained

invariably 6.0. Electrical conductivity was measured in $\mu\text{S}/\text{cm}$. A Hach DR3900 spectrophotometer (United States) was used to measure the initial absorbance of the dye mixture solution. To do this, 1.0 mL of the dye mixture solution was taken, diluted with 2.0 mL of distilled water, and placed in a quartz cell. The sample was measured using a spectrophotometer. In this case, the characteristic absorbance peaks were at 467 and 585 nm for the mixture of the two dyes.

2.3. Dye mixture characterization

Knowing the initial absorbance (ABS_i) of the solution of the dye mixture and measuring the absorbance at a certain time (ABS_t) allowed us to obtain the discoloration percentage of the contaminant in the following way [19]:

$$\% \text{ Discoloration} = \frac{\text{ABS}_i - \text{ABS}_t}{\text{ABS}_i} \times 100\% \quad (1)$$

The Hach method (United States) was used to determine TOC and COD. To obtain TOC, medium-range vials (15–150 mg/L) were used, while for COD, low-level vials (3–150 mg/L) were used. The vials were measured in a Hach DR3900 spectrophotometer (United States). The analysis was carried out every 30 min for 3 h. All solutions were made from analytical grade chemicals. The experiment was performed five times under the same initial conditions.

2.4. Plasma characterization

Optical emission spectroscopy (OES) was performed when the corona discharge was generated on the surface of the solution. This analysis was performed using an optical fiber, which passed through the sample container and was located just in front of the place where luminescence was produced. This allowed the direct measurement of the entire luminescence with a StellarNet EPP2000 spectrometer. The spectrometer operated in a wavelength range of 200–1,100 nm, with an integration time of 1,000 ms; five samples were averaged for the collection of spectra. The emission

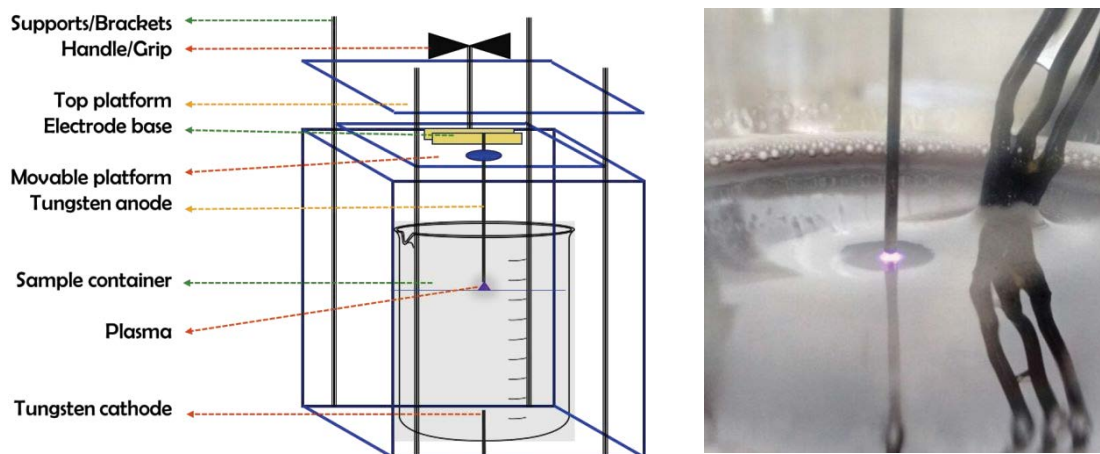


Fig. 1. Batch reactor.

spectra were qualitatively analyzed to assign chemical species to the peaks.

The corona discharge used in the treatment enabled the calculation of the electron temperature and electron density using the intensities of several spectral lines, assuming that the population of the emitting levels followed the Boltzmann distribution [20] and the system had a local thermodynamic equilibrium in some small fraction of the plasma. To calculate the electron temperature, the following equation was used:

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

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 their corresponding transition probabilities. These values are from the NIST atomic spectra database lines [21]. I_1 and I_2 are the relative line intensities of the lines in question; λ_1 and λ_2 are the wavelengths of the lines, which were experimentally measured.

The value of the electron density of the plasma used in the treatment was also obtained using T_e (i.e., the electron temperature). The electrons are responsible for most of the processes taking place in the corona discharge, ionization, dissociation, and recombination processes with water molecules. In this study, to calculate the electron density, the Saha-Boltzmann equation was used:

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

where T_e is the electron temperature, E_i is the ionization energy of the species, and k is the Boltzmann constant [22–24].

3. Results

Fig. 2 shows the comparative absorbance spectra of the AB52 (570 nm) and AB210 (465 and 604 nm) synthetic dyes and their mixture. When the dyes were combined, the maximum peaks were observed to suffer a shift in their wavelengths. The concentration of the AB210 and AB52 dyes was 0.5 mM in 250 mL. As shown in Fig. 2, the absorbance of the mixture turned out to be the sum of the absorbance of each dye because the two dyes were independent with no physical union or chemical bond.

The absorbance value was found to be higher, which increased its resistance to elimination. In general, it is difficult to treat water with a textile dye, mainly because this will increase the complexity of the treatment when it contains more than one dye. However, the actual effluents contain more than one component, and the study of the degradation processes of mixtures with chromophores is very useful for the treatment of real effluents [15].

Fig. 3 shows the absorbance spectra of the dye mixture in an aqueous solution in a UV-vis spectrophotometric range of 320–800 nm. The maximum wavelength in the absorption spectra of the mixture is 467 nm. The absorption spectra were obtained every 15 min for the analysis of the

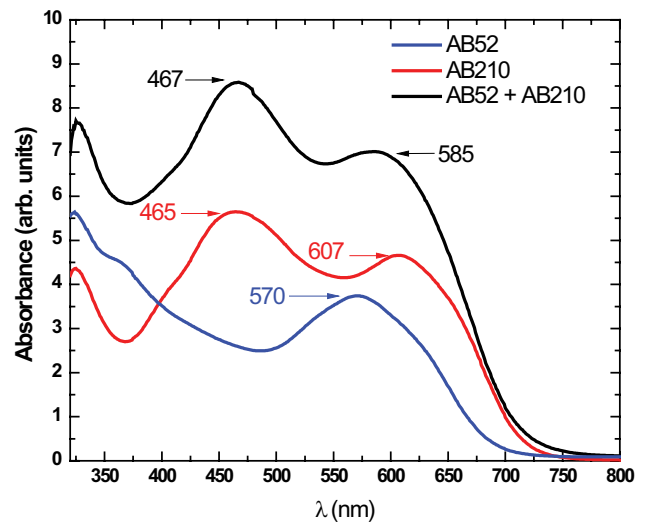


Fig. 2. Absorbance spectra of dyes and their mixture.

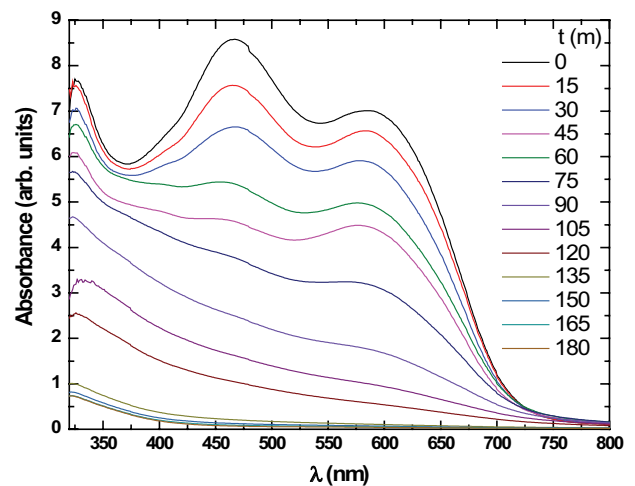


Fig. 3. Mixture absorbance as a function of treatment time with iron filings (Fe^{2+}).

decolorization process of the solution containing the dye mixture. Once the absorption spectra of the solution were obtained, the absorbance value of the peak at 467 nm was taken and inserted into Eq. (1), yielding the percentage of solution discoloration.

Fig. 4 shows the discoloration percentage of the dye mixture solution as a function of treatment time. The Fig. illustrates the good efficiency (discoloration 87.82%) of the plasma treatment using iron filing at 120 min of reaction. This will allow the remaining dye to be removed from the solution by some conventional method after 120 min. The figure also suggests that the plasma treatment yielded discoloration greater than 95% in 135 min, with it later falling gently to 99.1% in 180 min. These results recommend carrying out the treatment for 120 min and using another type of process for the remaining contaminant in the solution.

The optical emission spectrum of the plasma was obtained in a range of 200–1,100 nm. Fig. 5 shows the OES

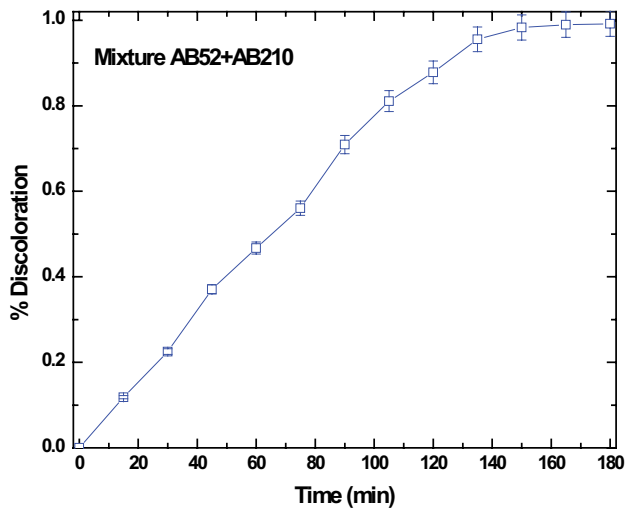


Fig. 4. Color removal percentage as a function of the treatment time of the AB52/AB210 mixture.

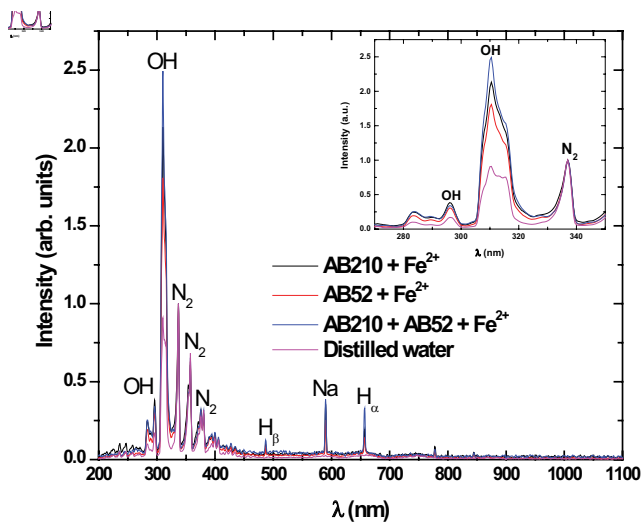


Fig. 5. Optical emission spectrum.

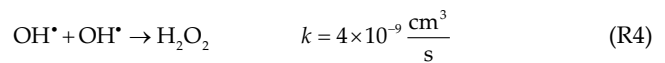
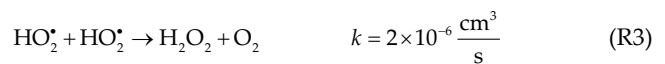
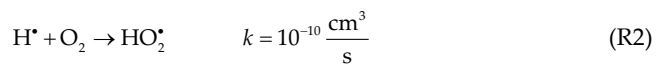
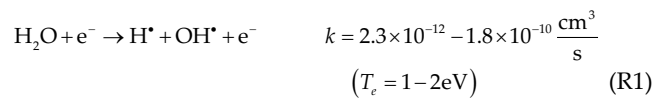
spectrum of distilled water (distilled water was considered as a reference to observe the changes in the emission spectrum of the plasma applied to the solution with dye and catalyst), the AB52 and AB210 dyes, and their mixture in a range of 200–1,100 nm. Dye solutions have Fe^{2+} as a catalyst. This wavelength interval was chosen to properly observe the region where lines and emission bands were identified.

The optical emission spectrum was normalized for nitrogen, because, in all four cases, the atmosphere used included air, and, in the air, the most abundant gas was nitrogen. In addition, the bands of nitrogen in intensity remained constant. The intensity of the HO emission bands was found to be higher for the mixture and the AB210 dye, maybe because of the greater amount of oxygen in the solution due to the presence of the dyes, which aid the generation of OH. In this type of system, the generation of active species such as H_{α} , H_{β} , N_2 , OH, and Na through OES (Fig. 5)

was observed (Table 2) [21]. Furthermore, the characteristic doublet of sodium in the spectra with pollutants (D-lines), commonly present in water, was also noticed.

The generation of corona discharge on the surface of water supplies the electrons in the solution. These electrons can interact with the water molecules and dissociate them. This will produce a chain of reactions that aid the production of H_2O_2 , which will, in turn, aid the production of OH^{\bullet} radical in the mixture.

The literature reports that hydrogen atoms, hydroxyl radicals, and hydrogen peroxide, which are the main oxidative species produced in the system, are generated at the plasma-liquid interface [12]. For the corona discharge on the surface of the water, the chemical reactions proposed are R1–R4:



These reactions describe the identification in the emission spectrum of the plasma of the OH band, which is the main radical that aids the decomposition of organic matter in the solution. Another possible process arising from the presence of nitrogen in generating the plasma in the air is described in Reaction 5, where OH^{\bullet} radical can also be generated through the dissociative excitation process of H_2O (gas) by excited nitrogen atoms [25]:



To characterize the corona discharge used in the treatment, the temperature and electron density were obtained. To determine the temperature of the applied plasma, in the process of degradation of the dye in water, the values of H_{α} and H_{β} were considered and inserted into Eq. (2), obtaining a value of 3.4 eV for AB52, 8.4 eV for AB210, and 4.42 eV for the AB52/AB210 mixture. Considering the value obtained from the electron temperature and hydrogen ionization energy, using Eq. (3), the value obtained from the electron density of the plasma was 8.69×10^{12} particles/cm³ for AB52, 3.68×10^{14} particles/cm³ for AB210, and 3.22×10^{13} particles/cm³ for the AB52/AB210 mixture.

To compare the effect of the plasma treatment on the degradation process of the dye mixture by corona discharge, the initial pH value was the same (pH = 6). The change in pH and electrical conductivity, as a function of treatment time with the electric discharge, is shown in Fig. 6. During the treatment of the solution using plasma and with iron filing

Table 2
Principal species identified in the treatment's optical emission spectrum of the corona discharge

Species	λ (nm)	Transition	Energy (eV)
H_{α}	656.5	2–3	1.8887
H_{β}	487.0	2–4	2.5497
OH	284.0	$A^2\Sigma^+ - X^2\Pi$	3,064 Å system
	309.5		
N_2	337.0	$C^3\Pi_u - B^3\Pi_g$	Second positive system
	357.5		
	375.5		
	380.5		
Na	590.0	$2p^63s - 2p^63p$	2.1023

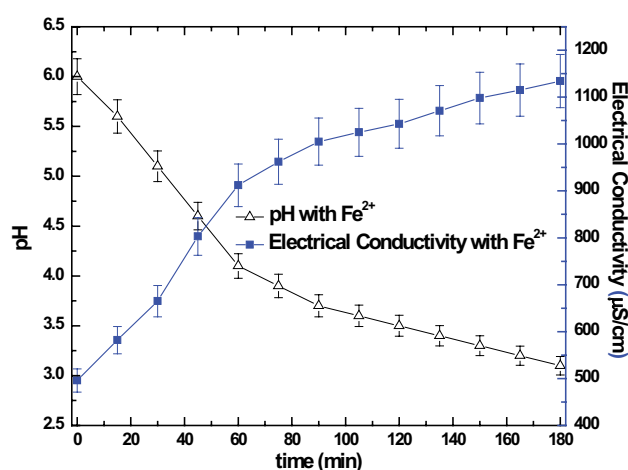


Fig. 6. pH and electrical conductivity of the AB52/AB210 mixture with the catalyst as a function of time.

used as a catalyst, the pH decreased from 6.0 to 3.1, which indicates a total decrease of 48.3%. In addition, the electrical conductivity of the water increased from 496 to 1,134 $\mu\text{S}/\text{cm}$, which illustrates an increase of 2.28 times compared with the initial value. The temperature has a direct impact on the rise in electrical conductivity. When the temperature increases, the water viscosity decreases, causing larger electrical conductivity. The pH gradually decreased in tandem with an increase in treatment time, showing a reduction of 6.7%, 15.0%, 23.3%, and 31.6% in 15, 30, 45, and 60 min, respectively; the degree of decrease for the rest of the time was only 16.7%. The greatest decay occurred in the first interval. From the beginning of the treatment until the 60th minute, the behavior was not linear. The organic acids and carbon dioxide formed from the degradation of the dye decreased the pH and increased the solution's conductivity. This variation occurred as a result of the hydrogen ions generating H^+ when plasma was applied to the solution, increasing the acidity of the medium. As a result of the electric current being transported by an ion medium in the solution, the electric conductivity increased in tandem with an increase in the ion concentration.

The discoloration of the solution serves as a primary measure for the evaluation of the degradation efficiency of the chemical compounds used as dyes. Fig. 7 shows the discoloration of the water by the effect of the plasma treatment as a function of the exposure time for the solution of the binary mixture. This discoloration process is due to the interaction of the free radicals generated by the plasma, which are highly oxidizing and able to break the chromophore bonds of the dye mixture. This condition can be observed separately for each dye.

The measurement of CO_2 is important in the treatment of this type of dye solution, because it affirms the mineralization of the organic pollutant by the plasma applied. Using the MQ135 air quality sensor, based on the Arduino hardware platform, along with open-source software resources, the concentration of carbon dioxide was measured during the plasma degradation process, yielding the highest concentration in the first 90 min (with 70.9% of the pollutant eliminated). This value gradually dropped to 63 ppm in 180 min, mainly due to a reduction in the concentration of the dye in the solution. The values obtained for CO_2 are consistent with those reported in the literature on water treatment [9].

Fig. 8 shows the reaction of the emission of CO_2 as a function of treatment time by the plasma of the solution with the dye mixture and iron filings and shows the measurement of CO_2 only when the dye mixture was heated, precluding the solution from emitting CO_2 since plasma is vital to the transformation of contaminants into CO_2 . The figure also shows the reaction of the water temperature with the dissolved dye and catalyst. The temperature increased when plasma was applied to the solution surface. The average rates of change for the intervals of 0–15, 15–30, 30–45, 45–60, and 60–75 min were 1.50°C, 1.22°C, 0.62°C, 0.39°C, and 0.18°C, respectively. At the 75th minute, the value reached 85.4°C, which practically remained constant during the rest of the treatment. The maximum temperature of 88.5°C was also achieved; however, this did not affect the degradation process.

The magnitude of the CO_2 emission was found to be directly linked to TOC, which means that when the magnitude decreased, the carbon was eliminated by the plasma in the form of carbon dioxide. The presence of the dye mixture in the aqueous solution only illustrates the initial concentration of each dye (0.5 mM). Moreover, a decrease in the concentration of the dyes corresponding to a decrease in the value of TOC for the solution was attained at different treatment intervals. TOC was obtained to determine whether the contaminant had been effectively removed from the solution. This finding provides a general overview of the degradation process of the dye and its by-products. The TOC value can be interpreted as an approximation of the amount of contaminants present in the sample. Fig. 9 shows the values of TOC and COD as the functions of the time of interaction with the plasma with and without the catalyst. The figure illustrates that the presence of the catalyst in the solution aids the decomposition process of the organic compound.

The magnitudes of TOC and COD gradually decreased until reaching the elimination percentage of the solution with a Fe^{2+} of 92.0% and 89.6%, respectively (Table 3). Table 3

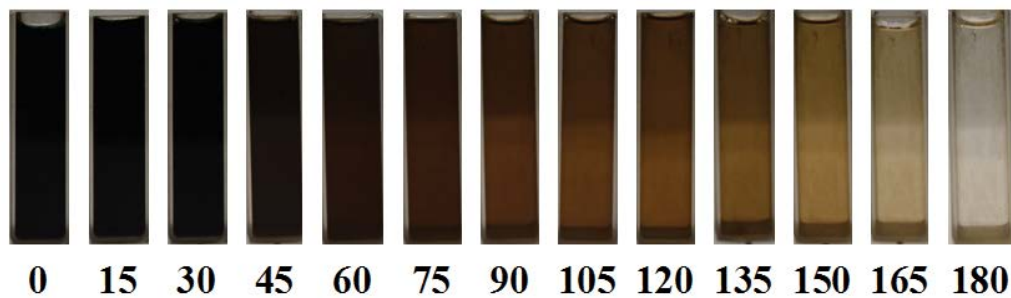


Fig. 7. Degradation images of the dye mixture by corona discharge treatment in time function (measured in minutes).

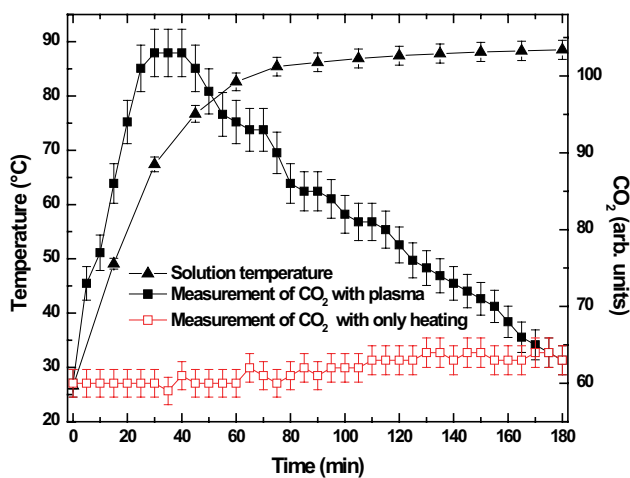


Fig. 8. CO₂ emission graphs during the degradation process vs. treatment time.

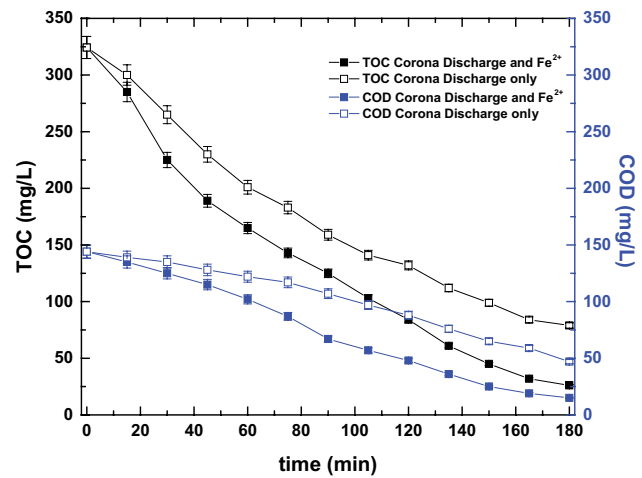


Fig. 9. TOC and COD graphs of the functions of the treatment time of the dye mixture, using Fe²⁺ as the catalyst.

Table 3
TOC and COD results

Mixture treatment by plasma	Initial		Final	
	TOC (mg/L) ±3%	COD (mg/L) ±4%	TOC (mg/L) ±3%	COD (mg/L) ±4%
AB52 + AB210 3 h	324.3	144.1	79 (75.6%)	47 (67.4%)
AB52 + AB210 + Fe ²⁺ 3 h			26 (92.0 %)	15 (89.6%)

shows the TOC and COD results in mg/L at a 3 h interval of treatment and elimination percentage. In addition, the treatment values of the mixture without a catalyst are presented, suggesting limited efficiency (TOC = 75.6% in 3 h) and the need for the use of a catalyst.

Previous studies have highlighted the efficiency of Fe²⁺ (1.0 mM) as a catalyst in treatment with plasma, yielding a degradation value greater than 90% in 2 h for both AB52 (0.5 mM) and AB210 (0.5 mM). However, in this research, when the concentration in the mixture reached 0.5 mM for each dye, the solution exhibited considerable resistance to the mineralization of the contaminants, necessitating the treatment of the solution for 3 h.

4. Conclusions

The Fe²⁺ catalyst enabled the treatment of the dissolved dye mixture in a batch reactor to obtain the discoloration and abatement of TOC and COD (Table 3). After 135 min of treatment, the degradation percentage remained above 95%, with the plasma generated at 80 W. The study showed that treatment by corona discharge can be successfully applied to water contaminated with dyes used in the textile industry at concentrations ≤0.5 mM. The optical emission spectrum of the corona discharge was obtained and observed to be active in species, such as OH, H_α, H_β, and N₂, mainly due to atmospheric plasma and the presence of Na in distilled water. The magnitude of carbon dioxide as a function

of treatment time was measured. It was initially found to be high in the first 45 min, but it later decreased alongside the TOC value verifying the mineralization of the dyes. Degradation by-products were readily biodegradable and less toxic than the dyes in the effluent, and these can be further oxidized in WWTPs through biological treatment. The values of pH (≈ 3.0), electrical conductivity ($\approx 1,100 \mu\text{S}/\text{cm}$), and temperature ($\approx 89^\circ\text{C}$) suggest that the water obtained in the effluent requires pretreatment before it can be used for any other purpose.

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