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Ferrous-Assisted Corona Plasma Decolorization of Acid Black 194 and Auramine O in a Continuous-Flow Recirculation System

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Abstract

Atmospheric-pressure corona plasma is a promising advanced oxidation technology for treating textile effluents owing to its capacity to generate reactive oxygen and nitrogen species (RONS) at the plasma–liquid interface. This study comparatively evaluated the decolorization of Acid Black 194 (AB-194), an azo dye, and Auramine O (AO), a triarylmethane dye, using an atmospheric-pressure corona plasma system operated under recirculating-flow conditions and assisted by ferrous species prepared from ferrous sulfate heptahydrate in an acidic medium. A three-tungsten-tip-to-stainless-steel-plate geometry treated 5 L of solution at 250 mL min⁻¹.

Decolorization was monitored by UV–Vis spectrophotometry at 594 nm for AB-194 and 432 nm for AO, together with measurements of pH, electrical conductivity, and turbidity. Absorbance values recorded immediately after addition of the ferrous solution served as the baseline for plasma-induced decolorization. After 90 min, decolorization efficiencies of 98.66% for AB-194 and 97.74% for AO were achieved. The calculated times required to reach 50% decolorization were 14.27 min for AB-194 and 16.00 min for AO.

The average power values were 139.42 W for AB-194 and 133.71 W for AO. The specific energy consumptions were 41.83 and 40.11 kWh m⁻³, whereas the energy yields at 50% decolorization were 5.72 and 2.13 g kWh⁻¹, respectively.

Keywords: Atmospheric-pressure plasma; corona discharge; plasma–liquid interaction; dye decolorization; ferrous species; recirculating-flow reactor.

1. Introduction

Textile effluents pose a major environmental challenge owing to the persistence of synthetic dyes-compounds characterized by high chemical stability, low biodegradability, and intense coloration. During dyeing and finishing, a fraction of the applied dyes does not fully bind to the fibers and may be discharged into wastewater together with salts and auxiliary compounds [1–3]. Once discharged, these compounds reduce light penetration, impair photosynthesis, and may exert ecotoxicological effects on aquatic biota.

Azo dyes such as Acid Black 194 are characterized by an –N=N– chromophoric group linked to aromatic structures. This molecular architecture contributes to dye stability but also renders the dye resistant to conventional treatment. Under certain conditions, incomplete cleavage of the azo bond may produce aromatic intermediates of toxicological concern [3,4]. In contrast, triarylmethane dyes such as Auramine O contain conjugated aromatic systems that confer high color intensity and persistence in aqueous media. Comparing representatives of these two families allows assessment of whether the same system retains efficiency against structurally different contaminants.

Conventional treatments, including coagulation–flocculation, adsorption, filtration, and biological processes, can reduce effluent color; however, they often have limitations related to sludge generation, adsorbent saturation, regeneration

costs, or low efficiency toward recalcitrant aromatic compounds [5–8]. Against this background, advanced oxidation processes (AOPs) have attracted considerable attention for their capacity to generate highly reactive oxidants, including hydroxyl radicals, ozone, hydrogen peroxide, and excited species [9–11].

Non-thermal atmospheric-pressure plasmas provide an attractive physicochemical route for water treatment because they generate reactive oxygen and nitrogen species (RONS) directly at the plasma–liquid interface. In corona discharges, the electric field is intensified around the electrodes, promoting localized gas ionization and the formation of a stable plasma at atmospheric pressure [12–15]. These species can transfer into the liquid phase and react with dissolved organic molecules, cleaving chromophoric bonds and promoting partial or advanced oxidation.

The addition of ferrous species is motivated by the ability of Fe^{2+} to participate in redox cycles that convert plasma-generated oxidants, particularly H_2O_2 formed through $\cdot\text{OH}$ recombination, into additional hydroxyl radicals. In simplified form, Fe^{2+} reacts with H_2O_2 to produce Fe^{3+} , $\cdot\text{OH}$, and OH^- ; Fe^{3+} may subsequently be reduced back to Fe^{2+} by reducing species or radical intermediates, thereby sustaining secondary oxidation pathways. In plasma–liquid systems, this assistance promotes the conversion of relatively persistent species into short-lived, high-potential oxidants, intensifying attack on chromophoric bonds and aromatic structures [9,10,13,16,17].

Although numerous studies have reported dye degradation by non-thermal plasma, many systems operate in batch mode or at small volumes, which limits scale-up. Recirculating-flow operation can improve hydraulic reproducibility, control residence time, and more closely approximate industrial application conditions [15,18,19]. In addition, chemical assistance with ferrous species may promote additional radical-generation pathways and enhance oxidation efficiency without requiring additional optical stages [9,10,18].

This study aimed to comparatively evaluate the degradation of Acid Black 194 and Auramine O by atmospheric-pressure corona plasma in a continuous recirculating-flow system assisted by a ferrous sulfate heptahydrate-based catalyst. Decolorization efficiency, apparent kinetics, pH, conductivity, turbidity, discharge emission spectra, and the energy indicators SEC and G_{50} were analyzed to establish the influence of dye molecular structure on process performance.

2. Materials and methods

2.1 Reagents and solution preparation

Aqueous solutions of Acid Black 194 (AB-194) and Auramine O (AO) were prepared using tap water supplied to the Faculty of Sciences of the Autonomous University of the State of Mexico at Cerrillo Piedras Blancas, Toluca, State of Mexico. The initial concentration was 0.1 mM for both dyes, which is comparable to concentrations used in non-thermal-plasma degradation studies [17,19]. The absorption maxima were experimentally determined by UV–Vis spectrophotometry as 594 nm for AB-194 and 432 nm for AO [20,21].

A blank sample consisting exclusively of the same tap water used to prepare the dye solutions was analyzed as an experimental control. No dye, ferrous catalyst, sulfuric acid, or other reagent was added to this sample. Its pH, electrical conductivity, turbidity, and absorbance were measured to establish the baseline physicochemical and optical characteristics of the aqueous matrix. The corresponding values are presented in Table 1.

Parameter	Value (tap water)
pH	8
Electrical conductivity ($\mu\text{S cm}^{-1}$)	730
Initial turbidity (NTU)	0.37
Absorbance	0.00

Table 1. Physicochemical and optical characteristics of the tap-water blank used as the experimental control.

The absorbance values of the blank were used to verify that the water matrix did not produce substantial optical interference at the analytical wavelengths selected for AB-194 and AO. The blank measurements also provided a

reference for distinguishing changes associated with the dye, catalyst addition, and plasma treatment from the initial characteristics of the water.

For the dye-containing solutions, pH, electrical conductivity, and turbidity were recorded before catalyst addition, immediately after catalyst addition, and after 90 min of plasma treatment. The corresponding values are summarized in Table 2.

Parameter	Dye	Before catalyst addition	After catalyst addition	After 90 min
pH	AB-194	8.04 ± 0.16	6.38 ± 0.22	6.02 ± 0.32
pH	AO	8.40 ± 0.10	6.60 ± 0.00	5.75 ± 0.05
Electrical conductivity ($\mu\text{S cm}^{-1}$)	AB-194	732 ± 58	846 ± 16	796 ± 6
Electrical conductivity ($\mu\text{S cm}^{-1}$)	AO	675 ± 5	755 ± 10	780 ± 10
Turbidity (NTU)	AB-194	2.350 ± 0.900	36.640 ± 3.060	2.856 ± 0.590
Turbidity (NTU)	AO	2.565 ± 0.120	36.650 ± 3.050	2.425 ± 0.825

Table 2. pH, electrical conductivity, and turbidity of the AB-194 and AO solutions before catalyst addition, after catalyst addition, and after 90 min of plasma treatment.

The ferrous catalyst stock solution was prepared by dissolving 6.95 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ($278.01 \text{ g mol}^{-1}$) in 500 mL of distilled water, followed by the addition of 2.7 mL of H_2SO_4 . The resulting stock solution contained approximately $0.050 \text{ mol L}^{-1} \text{ Fe}^{2+}$. The catalyst was dosed at a ratio of 5 mL of stock solution per 250 mL of dye solution. Therefore, for the 5.0 L volume used in each treatment, a total of 100 mL of the ferrous stock solution was added. Considering the resulting total liquid volume of approximately 5.10 L, this dosage produced a final Fe^{2+} concentration of approximately 0.98 mmol L^{-1} . This dose was selected as a moderate intensification condition intended to promote ferrous-mediated radical pathways without the external addition of H_2O_2 . Accordingly, the system is described as ferrous-species-assisted plasma rather than as a conventional Fenton process [9,17].

2.2 Continuous-flow experimental system

The experimental system operated under continuous recirculation with a working volume of 5 L. The solution was pumped from the reservoir to the active reactor region and returned to the reservoir after contacting the discharge at the air–liquid interface (Fig. 1). Recirculation was provided by a Jingnuo JK-180 submersible pump (3 W, nominal maximum flow rate of 200 L h^{-1} , and maximum delivery head of 50–55 cm).

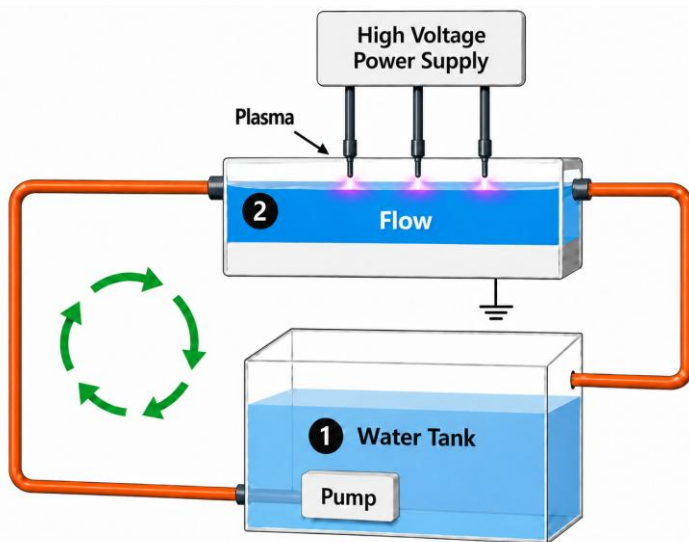


Fig. 1 Schematic of the catalyst-assisted corona plasma experimental system operated under recirculating-flow conditions.

The flow rate was determined volumetrically by collecting the discharge over timed intervals. Triplicate measurements confirmed a flow rate of 250 mL min⁻¹, which was applied consistently across all experiments. The discharge was powered by a Spellman SA4 direct-current power supply. The circuit included a 1100 Ω, 200 W maximum-dissipation limiting resistor for current protection and stabilization.

Voltage and current were recorded using two 6½-digit Siglent SDM3065X digital bench multimeters; one measured voltage and the other measured current directly. Absorbance was measured with a Thermo Scientific GENESYS 20 spectrophotometer (325–1100 nm; photometric resolution, 0.001 Abs; wavelength accuracy, ±2 nm). Optical emission was recorded with an Ocean Optics FLAME-T-UV-VIS spectrometer equipped with a 600 lines mm⁻¹ grating and operated using OceanView software.

pH and electrical conductivity were determined with a Hanna Instruments HI9811-51 multiparameter meter, with resolutions of 0.01 pH units and 10 μS cm⁻¹, respectively. Turbidity was measured using a Hach 2100Q turbidimeter with a resolution of 0.01 NTU. The specific procedures for plasma generation, electrical characterization, OES, and physicochemical analysis are described in Sections 2.3–2.11.

The general operating conditions are summarized in Table 3. Samples were collected every 15 min during the 90 min treatment.

Parameter	Experimental condition
Volume per run	5 L
Flow rate	250 mL min ⁻¹
Operating mode	Continuous recirculating flow
Discharge geometry	Three-tip-to-plate configuration
Electrode (anode)	Tungsten
Electrode (cathode)	Stainless-steel plate
Treatment time	0–90 min
Sampling interval	15 min

Table 3. Main experimental conditions of the catalyst-assisted corona plasma system.

2.3 Corona plasma discharge

The discharge was generated in air at atmospheric pressure using a direct-current-powered configuration consisting of three tungsten tips facing a stainless-steel plate. The high curvature of the tips locally intensified the electric field and promoted impact ionization in the air–liquid region, enabling operation in the corona regime [17,19,22–25].

2.4 Electrical characterization and discharge power

Electrical characterization was performed by simultaneously measuring the applied voltage and discharge current with the two Siglent SDM3065X multimeters. The 1100 Ω, 200 W resistor was included in the circuit as a limiting element; the current used in the calculations was read directly from the multimeter and was therefore not estimated from the voltage drop across the resistor.

Because voltage and current remained nearly constant during the stable operating stage, electrical power was calculated for each pair of readings as:

$$P = V I \quad (1)$$

where P is the electrical power (W), V is the applied voltage (V), and I is the discharge current (A). The average power used in the energy analysis was the arithmetic mean of the values calculated with Eq. (1) during the stable period of each treatment.

The temporal evolution of V, I, and P was used to verify discharge stability and rule out abrupt current increases associated with a transition to arc discharge. This behavior is consistent with a current-limited corona discharge [12,22,24,25].

2.5 Optical emission spectroscopy

Excited species generated by the discharge were characterized by optical emission spectroscopy (OES) using an Ocean Optics FLAME-T-UV-VIS spectrometer. Radiation from the air–liquid region was guided to the spectrometer through an optical fiber and acquired with OceanView software.

Signals were assigned by comparing the experimental wavelengths with reported transitions for species characteristic of air discharges in contact with water, including $\cdot\text{OH}$, N_2 , N_2^+ , H_α , H_β , Na I , and O I [16,22,26,27]. For each spectrum, relative intensity was normalized to the most intense emission. The band associated with $\cdot\text{OH}$ was related to water-dissociation processes and secondary reactions at the plasma–liquid interface [9,10,16].

OES was used as a non-invasive diagnostic of excited species. Intensities were interpreted qualitatively or semiquantitatively rather than as absolute concentrations of reactive species in the liquid phase.

2.6 Spectrophotometric analysis and decolorization efficiency

Dye concentration was monitored by UV–Vis spectrophotometry using the GENESYS 20 instrument. Samples were analyzed in 1 cm optical-path-length cells at 594 nm for AB-194 and 432 nm for AO. Before each measurement series, water from the same matrix was used as the instrumental blank.

Before treatment, absorbance–concentration calibration curves were constructed for each dye. Proportionality between the two variables was evaluated using the Beer–Lambert law [20]:

$$A = \varepsilon l C \quad (2)$$

where A is absorbance, ε is the molar absorption coefficient, l is the optical path length, and C is concentration. The linearity of the curves allowed absorbance to be related to concentration over the working range.

Decolorization efficiency was calculated from the relative decrease in absorbance [15,18]:

$$\text{Decoloration}(\%) = [(A_o - A_t)/A_o] \times 100 \quad (3)$$

where A_o is the initial absorbance of the solution after catalyst addition and A_t is the absorbance at time t. Equation (3) was applied to each sampling point.

2.7 Kinetic analysis

Decolorization kinetics were analyzed using an apparent pseudo-first-order model, assuming that the effective concentration of oxidizing species remained quasi-steady relative to the dye concentration during each treatment interval:

$$\ln(A_o - A_t) = kt \quad (4)$$

where k is the apparent rate constant (min^{-1}) and t is the treatment time (min). The value of k was obtained from the slope of the linear fit of $\ln(A_o/A_t)$ versus t, and goodness of fit was evaluated using R^2 . The time required to reach 50 % decolorization was determined from $t_{50} = \ln(2)/k$, derived from Eq. (4).

2.8 Energy indicators

The energy demand of the treatment was evaluated using the specific energy consumption (SEC) and the energy yield at 50 % decolorization (G_{50}). SEC was calculated as:

$$SEC = \frac{P_{\text{promt}}}{V_{\text{treat}}} \quad (5)$$

where SEC is expressed in kWh m^{-3} , $\bar{P}$ is the average system power, t is the treatment time, and V is the treated volume.

The G_{50} parameter represents the mass of contaminant degraded per unit of energy consumed to achieve 50 % decolorization, allowing comparison of energy efficiency among treatment systems [14,17,18]. G_{50} was calculated as:

$$G_{50} = 1.8 \times 10^6 \frac{C_0 V_0 M}{P t_{50}} \quad (6)$$

where C_0 is the initial contaminant concentration (mol L^{-1}), V is the treated volume (L), M is the dye molecular weight (g mol^{-1}), $\bar{P}$ is the average system power (W), and t_{50} is the time required to reach 50 % decolorization.

This methodology enables quantitative comparison of the energy performance of the treatments applied to AB-194 and AO and assessment of the catalyst influence on degradation rate and plasma–liquid-system energy consumption.

2.9 pH measurement

pH was measured before discharge ignition and every 15 min using the Hanna HI9811-51 instrument. The electrode was rinsed with distilled water between samples, and the same immersion and reading-stabilization procedure was maintained. Monitoring pH enabled analysis of plasma-induced changes that may be associated with reactive-species generation, formation of oxidized organic byproducts, and the presence of nitrogen-containing species in solution [10,11,16].

Changes in pH are a relevant indicator of the chemical impact of plasma on the aqueous matrix and of the stability of the treated effluent [13,15].

2.10 Electrical conductivity

Electrical conductivity was measured in the same samples and at the same sampling times using the Hanna HI9811-51. Results were expressed in $\mu\text{S cm}^{-1}$ to evaluate changes associated with the formation of ionic species during the process [13,15]. This parameter provides complementary information on the breakdown of complex organic structures and the release of charged species into solution [10,16].

2.11 Turbidity

Turbidity was determined at each sampling point using the Hach 2100Q and expressed in NTU. Before each measurement, the sample was gently mixed, the cuvette was checked for air bubbles, and placed in a fixed orientation. Turbidity analysis helps distinguish effective effluent decolorization from the possible formation of intermediates that increase medium opacity, thereby providing a more comprehensive process assessment [2,15].

2.12 Reproducibility and data treatment

Each treatment was performed in five independent replicates under the same volume, flow-rate, catalyst-dose, and exposure-time conditions. Results were reported as means; when available, error bars represented dispersion among replicates. Linear fits were evaluated using R^2 , and calculations were performed with consistent units. No inferential

statistics were applied, as the study aimed to compare kinetic, physicochemical, and energy trends between dyes rather than to test formal hypotheses.

3. Results and discussion

3.1 Calibration curves

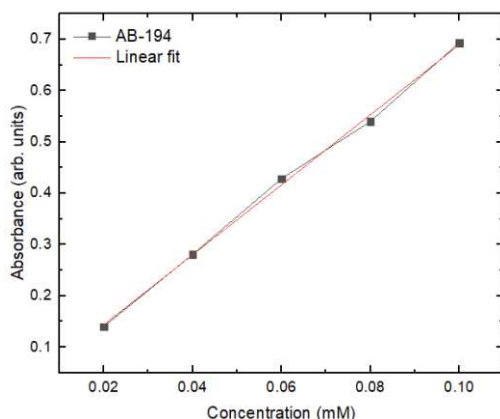


Fig. 2 Linear relationship between concentration and absorbance for AB-194

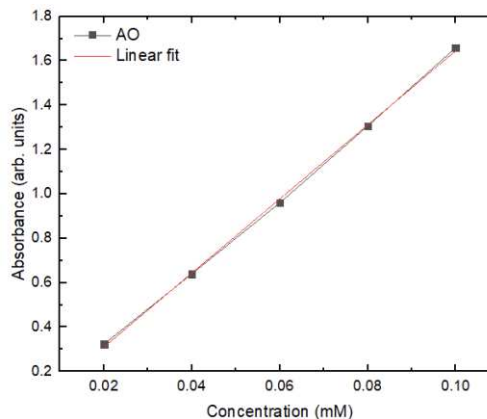


Fig. 3 Linear relationship between concentration and absorbance for AO.

Figs. 2 and 3 show a linear relationship between concentration and absorbance for both dyes, with coefficients of determination above 0.99. This behavior validates the Beer–Lambert law expressed in Eq. (2) and supports the use of absorbance as an indirect concentration variable during treatment. Prior calibration is essential to avoid interpreting instrumental changes as chemical degradation and to support the subsequent kinetic analysis.

3.2 Absorbance evolution and decolorization

Figs. 4 and 5 show the temporal evolution of AB-194 and AO absorbance during treatment. Two initial measurements were obtained for each dye: the first corresponded to the water–dye solution before addition of the ferrous stock solution, whereas the second was recorded after its addition and before plasma exposure. The post-addition measurement was used as the initial value for evaluating plasma-induced decolorization, thereby separating the immediate effect of the ferrous solution from the subsequent effect of plasma treatment.

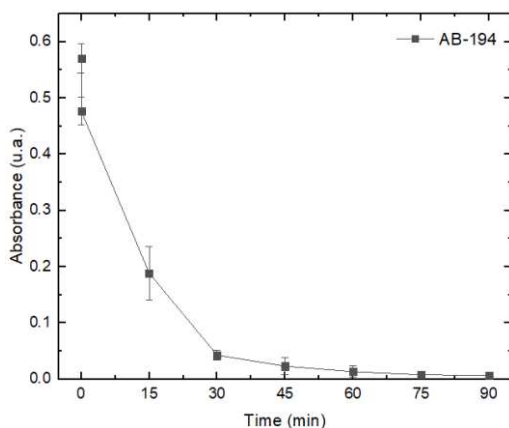


Fig. 4 Time-dependent absorbance of AB-194 during corona plasma treatment and catalyst-assisted plasma treatment.

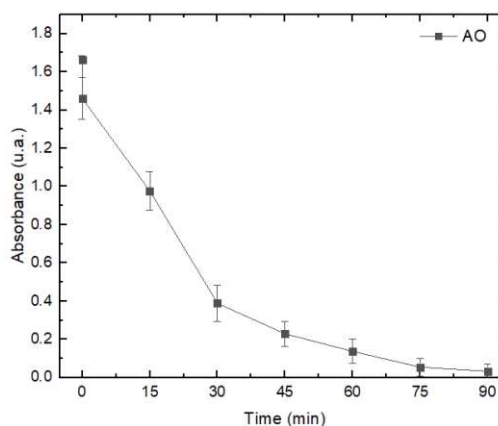


Fig. 5 Time-dependent absorbance of AO during corona plasma treatment and catalyst-assisted plasma treatment.

For AB-194, absorbance decreased from 0.5706 ± 0.026 to 0.4768 ± 0.024 after addition of the ferrous solution, corresponding to an initial reduction of approximately 16.4%. This decrease was greater than that expected from dilution alone, since adding 100 mL of stock solution to 5.0 L of dye solution increased the total volume by only approximately 2%. The observed change may therefore be associated with immediate interactions between the anionic dye and dissolved ferrous species, including changes in dye aggregation, speciation, ionic environment, or complex formation, which may modify the spectral response of AB-194 at its analytical wavelength. Because these effects were not independently characterized, this initial decrease should be interpreted as an effect of ferrous-solution addition rather than as confirmed dye degradation.

Absorbance then decreased rapidly during plasma treatment, reaching 0.1884 ± 0.048 at 15 min, 0.0426 ± 0.009 at 30 min, and 0.0064 ± 0.004 at 90 min. This decrease was accompanied by a progressive loss of solution color, as visually shown in Fig. 6 Using the absorbance recorded after addition of the ferrous solution as the reference, the final decolorization efficiency was approximately 98.66%.

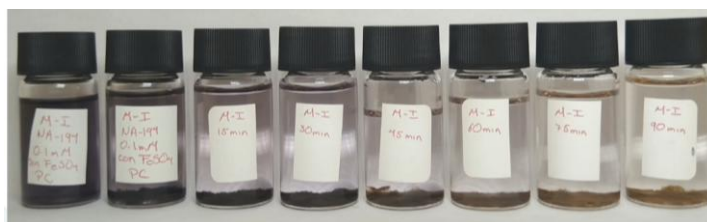


Fig. 6 Visual change in the color of the AB-194 solution during ferrous-species-assisted corona plasma treatment, from the initial condition to 90 min of treatment.

For AO, addition of the ferrous solution decreased the absorbance from 1.6626 ± 0.022 to 1.4606 ± 0.109 , corresponding to an initial reduction of approximately 12.15%. As in the AB-194 system, this decrease was substantially greater than that attributable to dilution alone. It may therefore be related to immediate interactions between AO and dissolved ferrous species, including changes in aggregation, ionic strength, molecular environment, or complex formation, which could modify the optical response of the dye at the analytical wavelength. Since these effects were not independently characterized, the initial absorbance decrease should likewise be interpreted as an effect of ferrous-solution addition rather than as confirmed chemical degradation.

During plasma treatment, AO absorbance progressively decreased to 0.9766 ± 0.102 at 15 min, 0.3888 ± 0.094 at 30 min, 0.2290 ± 0.066 at 45 min, 0.1370 ± 0.062 at 60 min, 0.0542 ± 0.046 at 75 min, and 0.0330 ± 0.037 at 90 min. This decrease was accompanied by a progressive loss of solution color, as visually shown in Fig. 7. Taking the post-addition absorbance as the reference, the final decolorization efficiency was approximately 97.74%.



Fig. 7 Visual change in the color of the AO solution during ferrous-species-assisted corona plasma treatment, from the initial condition to 90 min of treatment.

Both systems exhibited a pronounced decrease in absorbance during plasma exposure, consistent with the progressive disruption or transformation of the chromophoric structures responsible for color. AB-194 showed a faster absorbance decrease during the first 30 min and a slightly higher final decolorization efficiency than AO, with final values of 98.66% and 97.74%, respectively. This behavior indicates that the two dyes responded differently to the plasma–ferrous-species system, likely because of differences in molecular structure, charge, chromophore accessibility, and susceptibility to

attack by plasma-generated reactive species. Nevertheless, the decrease in absorbance demonstrates decolorization and should not, by itself, be interpreted as complete mineralization.

3.3 Kinetic interpretation

Decolorization kinetics were evaluated using an apparent pseudo-first-order model by plotting $\ln(A_0/A_t)$ versus time according to Eq. (4). For this analysis, A_0 corresponded to the absorbance measured after addition of the ferrous solution and before plasma treatment.

The linear fits yielded apparent rate constants of 0.0486 min^{-1} for AB-194 and 0.0433 min^{-1} for AO, with coefficients of determination of 0.9385 and 0.9929, respectively. From $t_{50} = \ln(2)/k$, the calculated times required to reach 50% decolorization were 14.27 min for AB-194 and 16.00 min for AO.

The ratio $k_{AB-194}/k_{AO} = 1.12$ indicates that, under the conditions studied, the apparent decolorization rate of AB-194 was approximately 12% higher than that of AO. Although both dyes exhibited similar overall kinetic behavior, AO showed a slightly lower apparent rate constant and a longer calculated half-decolorization time. These differences may be associated with variations in molecular structure, chromophore accessibility, charge, and susceptibility to attack by plasma-generated reactive species.

The high R^2 value obtained for AO indicates that its absorbance decay was well represented by the apparent pseudo-first-order model over the evaluated treatment period. In contrast, the lower R^2 value obtained for AB-194 may reflect its rapid initial decolorization during the first 15 min, which deviated from strict linear behavior. This deviation could be related to transient variations in the production and consumption of reactive species during the early stage of treatment. Therefore, the pseudo-first-order model should be regarded as a global empirical approximation of the experimental data rather than as a mechanistically rigorous description of the complete reaction pathway.

3.4 Electrical characterization of the discharge: voltage, current and power

Figs. 8–13 show the temporal evolution of voltage, current, and power during the treatment of Acid Black 194 (AB-194) and Auramine O (AO) by catalyst-assisted corona plasma. In both cases, a brief transient regime was observed at discharge initiation, followed by sustained electrical stabilization throughout the 90 min operating period, characteristic of a non-thermal corona regime under current-limited conditions.

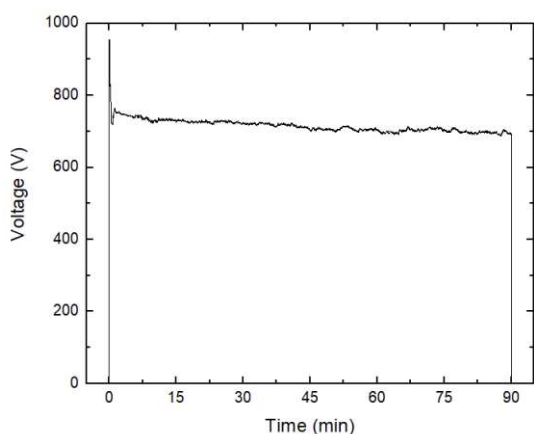


Fig. 8 Time-dependent voltage during the catalyst-assisted corona plasma treatment of AB-194.

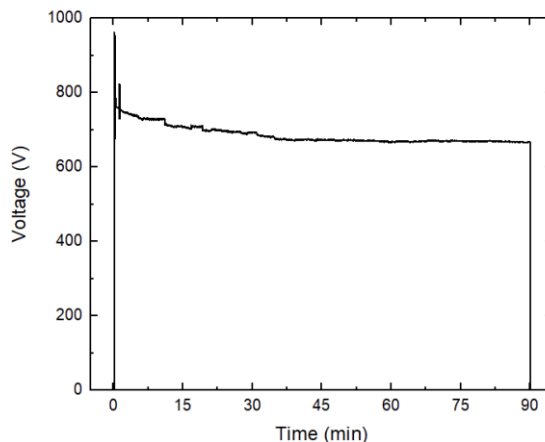


Fig. 9 Time-dependent voltage during the catalyst-assisted corona plasma treatment of AO

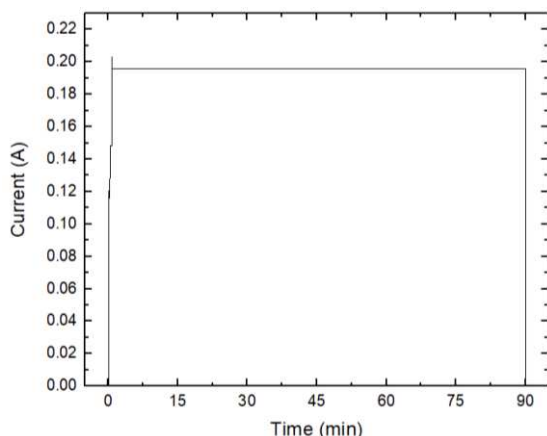


Fig. 10 Time-dependent current during the catalyst-assisted corona plasma treatment of AB-194.

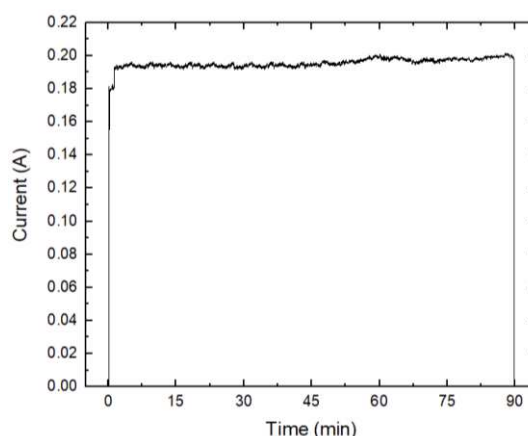


Fig. 11 Time-dependent current during the catalyst-assisted corona plasma treatment of AO

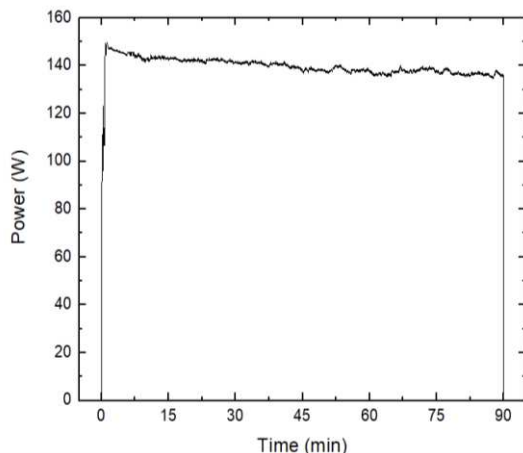


Fig. 12 Time-dependent electrical power during the catalyst-assisted corona plasma treatment of AB-194.

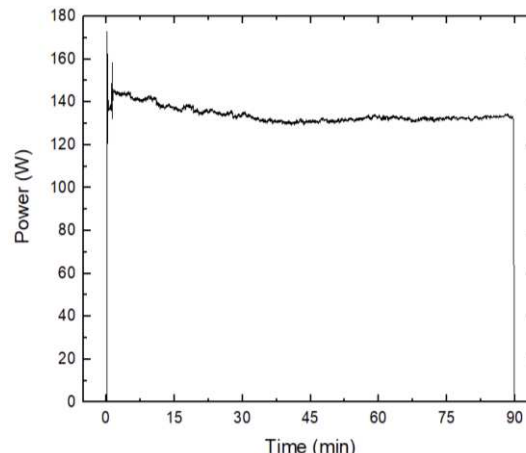


Fig. 13 Time-dependent electrical power during the catalyst-assisted corona plasma treatment of AO

For AB-194 treatment, the voltage initially approached 950 V during discharge establishment and subsequently stabilized within approximately 700–740 V. The current remained nearly constant at around 0.195 A during most of the treatment. Consequently, the experimental average power calculated from the instantaneous voltage and current signals using Eq. (1) was 139.42 W. The stability of the electrical signals indicates that the discharge remained in a stable corona regime without transitioning to an arc, thereby favoring continuous generation of reactive species at the plasma–liquid interface.

For AO, the electrical behavior was similar. The voltage stabilized at slightly lower values of approximately 660–700 V, whereas the current remained around 0.19–0.20 A during treatment. The average power calculated using Eq. (1) was 133.71 W. Although both systems operated under comparable electrical conditions, the small differences in voltage and power may be associated with the specific physicochemical properties of each dye solution, including conductivity, ionic character, and molecular structure.

The corresponding figures also show the absence of abrupt current oscillations or sudden power increases, confirming that the system maintained a stable atmospheric-pressure discharge regime throughout the experiment. This behavior is

consistent with reports on corona systems used for advanced oxidation processes and non-thermal-plasma water treatment [12–15,22].

3.5 pH evolution

Before catalyst addition, the AB-194 and AO solutions had pH values of 8.04 ± 0.16 and 8.40 ± 0.10 , respectively. Addition of the $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution prepared in an acidic medium immediately decreased the pH to 6.38 ± 0.22 for AB-194 and 6.60 ± 0.00 for AO. These latter values were taken as the initial plasma-treatment conditions at $t = 0$.

During the 90 min treatment, pH gradually decreased to 6.02 ± 0.32 for AB-194 and 5.75 ± 0.05 for AO, corresponding to changes of -0.36 and -0.85 pH units relative to the values measured after catalyst addition (Fig. 14 and 15). Acidification was more pronounced for AO and may be related to the formation of oxidized nitrogen species and lower-molecular-weight organic products during plasma–liquid interaction [10,11,16].

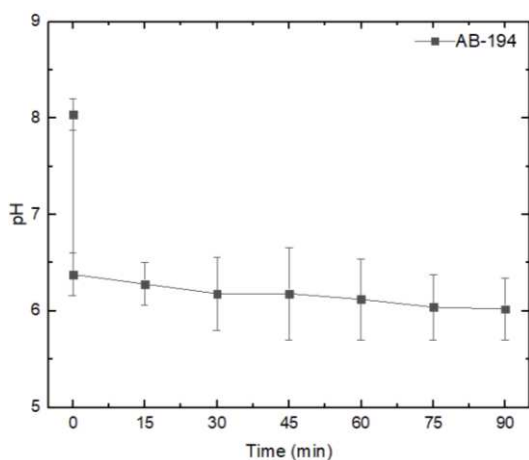


Fig. 14 Evolution of pH during the treatment of AB-194.

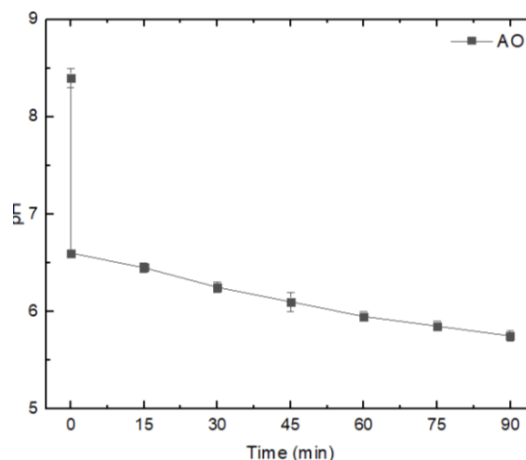


Fig. 15 Evolution of pH during the treatment of AO

However, because the catalytic solution was prepared in the presence of H_2SO_4 , the initial pH decrease should be attributed primarily to catalyst addition, whereas the subsequent variation reflects evolution of the medium during plasma treatment. The observed pH range may favor the persistence of ferrous species and secondary $\cdot\text{OH}$ -generation pathways; however, pH alone cannot determine the extent of mineralization.

3.6 Turbidity evolution

Before catalyst addition, the AB-194 (Fig. 16) and AO (Fig. 17) solutions had turbidities of 2.35 ± 0.90 and 2.565 ± 0.12 NTU, respectively. Addition of the $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution prepared in an acidic medium immediately increased turbidity to 36.64 ± 3.06 NTU for AB-194 and 36.65 ± 3.05 NTU for AO. These values were taken as the initial plasma-treatment conditions at $t = 0$.

A pronounced decrease in turbidity was observed during the first 30 min of treatment. For AB-194, turbidity decreased from 36.64 ± 3.06 to 3.714 ± 1.13 NTU, whereas for AO it decreased from 36.65 ± 3.05 to 3.10 ± 0.84 NTU. The values then remained relatively stable, reaching 2.856 ± 0.59 NTU for AB-194 and 2.425 ± 0.825 NTU for AO after 90 min of treatment.

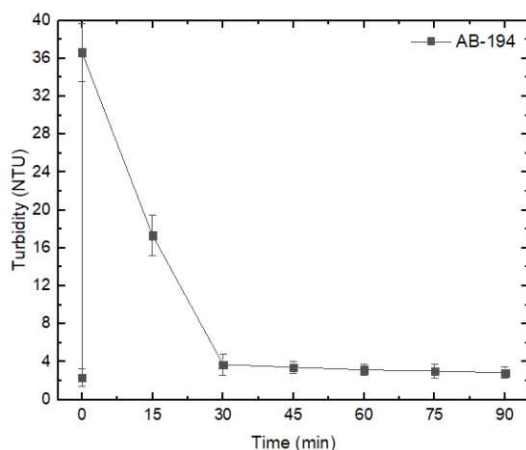


Fig. 16 Evolution of turbidity during the treatment of AB-194.

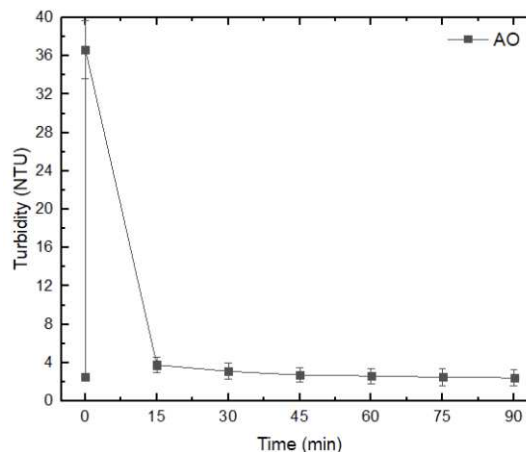


Fig. 17 Evolution of turbidity during the treatment of AO

Using the values measured after catalyst addition as the reference, turbidity decreased by approximately 92.2 % for AB-194 and 93.4 % for AO. The initial turbidity increase is attributed to iron hydroxide colloids or fine particles formed upon mixing the ferrous solution with the dye matrix. The subsequent decrease suggests removal, transformation, or sedimentation of the species responsible for light scattering; however, turbidity alone cannot establish mineralization or identify the nature of the particles present.

3.7 Electrical conductivity evolution

Before catalyst addition, the AB-194 (Fig. 18) and AO (Fig. 19) solutions had conductivities of 732 ± 58 and $675 \pm 5 \mu\text{S cm}^{-1}$, respectively. Addition of the catalytic solution immediately increased conductivity to $846 \pm 16 \mu\text{S cm}^{-1}$ for AB-194 and $755 \pm 10 \mu\text{S cm}^{-1}$ for AO. This initial increase is mainly attributed to ionic species introduced with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and H_2SO_4 in the catalytic solution.

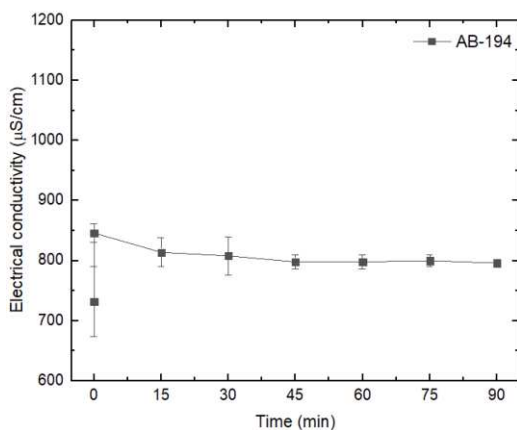


Fig. 18. Evolution of electrical conductivity during the treatment of AB-194.

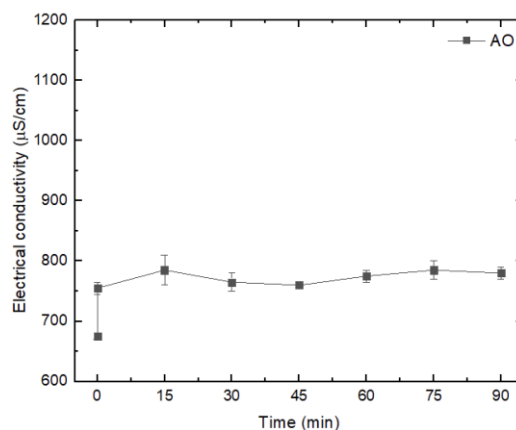


Fig. 19. Evolution of electrical conductivity during the treatment of AO

During the 90 min plasma treatment, the two systems behaved differently. For AB-194, conductivity gradually decreased from 846 ± 16 to $796 \pm 6 \mu\text{S cm}^{-1}$, corresponding to an approximate reduction of 5.9 % relative to the value measured after catalyst addition. For AO, conductivity showed moderate fluctuations and increased from 755 ± 10 to $780 \pm 10 \mu\text{S cm}^{-1}$, equivalent to an approximate increase of 3.3 %.

These variations may be related to changes in solution ionic composition during treatment, including the formation, transformation, or consumption of charged species. However, because the changes were moderate and experimental

dispersion was present, conductivity is interpreted only as a complementary indicator of modifications in the aqueous matrix rather than as a direct measure of mineralization.

3.8 OES characterization

The optical emission spectra obtained during AB-194 and AO treatment are shown in Figs. 20 and 21, and the main spectral assignments are summarized in Tables 4 and 5. Emissions associated with excited species characteristic of air discharges in contact with water were identified in both systems, including OH, N₂, N₂⁺, H_α, and H_β. The band observed at 305–314 nm was assigned to the OH(A²Σ⁺ → X²Π) system, whereas emissions near 336 nm and within 353–380 nm were associated with the second positive system of molecular nitrogen, N₂(C³Π_u → B³Π_g). The signal near 391 nm was attributed to the first negative system of ionized nitrogen, N₂⁺(B²Σ^u → X²Σ^g⁺), indicating excitation and ionization processes in the gas phase of the discharge. The Balmer H_β and H_α lines observed at approximately 487 and 656 nm, respectively, are also consistent with excitation of water-derived species in the plasma–liquid interaction region.

The AB-194 and AO spectrum also showed signals near 589 and 777 nm, tentatively assigned to Na I and O I, respectively. Na I emission may be related to dissolved salts or impurities in the aqueous matrix, whereas the O I signal is compatible with dissociation and excitation processes involving oxygen-containing species. Differences in intensity between the AB-194 and AO spectra may be associated with variations in the composition and physicochemical properties of each solution.

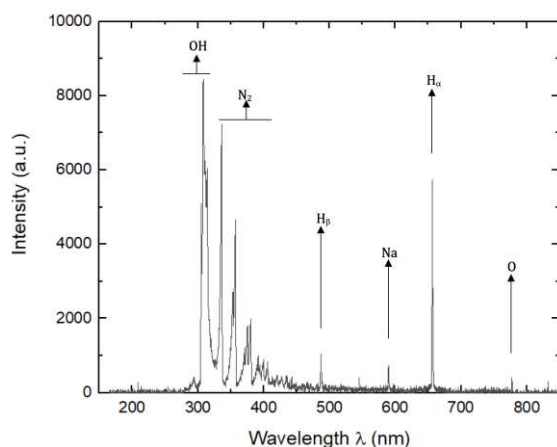


Fig. 20. Optical emission spectrum recorded at atmospheric pressure at the air–water interface during the treatment of AB-194.

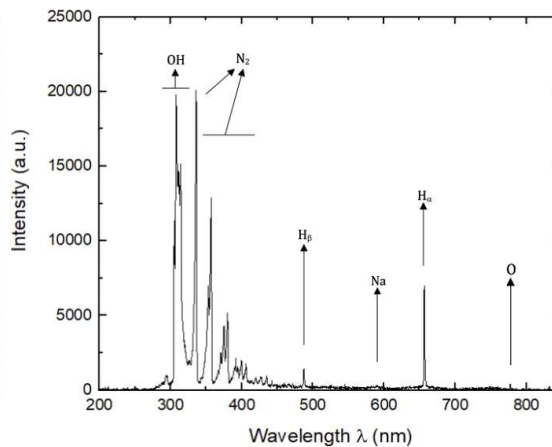


Fig. 21. Optical emission spectrum recorded at atmospheric pressure at the air–water interface during the treatment of AO.

Species	Experimental wavelength (nm)	Theoretical wavelength (nm)	Transition
OH•	305–311	308.90	A ² Σ ⁺ → X ² Π
N ₂	336	337.10	C ³ Π _u → B ³ Π _g
N ₂	353–380	357.70	C ³ Π _u → B ³ Π _g
N ₂ ⁺	391	391.40	B ² Σ ^u → X ² Σ ^g ⁺
H _β	487	486.10	n = 4 → n = 2
Na I	589	588.99	3p → 3s
H _α	656	656.30	n = 3 → n = 2
O I	777	777.19	3p ⁵ P → 3s ⁵ S ^o

Table 4. Optical emission species identified during plasma treatment of AB-194.

Species	Experimental wavelength (nm)	Theoretical wavelength (nm)	Transition
OH•	305–314	308.90	A ² Σ ⁺ → X ² Π
N ₂	336	337.10	C ³ Π _u → B ³ Π _g

N ₂	353–380	357.70	C ³ Π _u → B ³ Π _g
N ₂ ⁺	391	391.40	B ² Σ _u ⁺ → X ² Σ _g ⁺
Na I	589	588.99	3p → 3s
Hβ	487	486.10	n = 4 → n = 2
O I	777	777.19	3p ⁵ P → 3s ⁵ S ^o
Hα	656	656.30	n = 3 → n = 2

Table 5. Optical emission species identified during plasma treatment of AO.

Taken together, the identified emissions are consistent with simultaneous generation of RONS, which participate in the oxidative degradation pathways of the dyes during atmospheric-pressure corona plasma treatment.

3.9 Apparent electron temperature and electron density

The apparent electron temperature of the plasma was estimated from the H_α and H_β hydrogen emission lines identified by OES. This procedure assumes that the populations of the emitting levels can be approximated by a Boltzmann-type distribution; therefore, the resulting value should be interpreted as an apparent excitation temperature rather than the global thermodynamic temperature of the plasma [22,28,29].

The Boltzmann relation was used to estimate the apparent electron temperature:

$$T_e = \frac{E_{m(2)} - E_{m(1)}}{k_B} \left[\ln \left(\frac{I_1 \lambda_1 g_{m(2)} A_{m(2)}}{I_2 \lambda_2 g_{m(1)} A_{m(1)}} \right) \right]^{-1} \quad (7)$$

where E_m denotes the energy of the excited levels, k_B is the Boltzmann constant, I is the spectral intensity of the emission lines, λ is wavelength, g is the statistical weight, and A is the radiative transition probability [22,28,29].

For AB-194 treatment, experimental intensities of H_β = 1051.96 a.u. and H_α = 5735.98 a.u. were used, yielding an approximate electron temperature of 0.72 eV from Eq. (7). For AO, intensities of H_β = 1413.34 a.u. and H_α = 7008.58 a.u. yielded an electron temperature of 0.81 eV.

Electron density was estimated from the electron-temperature values using the Saha–Boltzmann equation [22,29,30].

$$n_e = 6 \times 10^{21} (T_e)^{3/2} \exp \left(-\frac{E_i}{k_B T_e} \right) \quad (8)$$

where n_e is the estimated electron density, k_B is the Boltzmann constant, and E_i is the ionization energy of atomic hydrogen. The value used in the calculation, $E_i = 13.5984$ eV, was obtained from the National Institute of Standards and Technology Atomic Spectra Database [31].

The calculated apparent electron-density values were $2.88 \times 10^{19} \text{ m}^{-3}$ for AB-194 and $2.80 \times 10^{20} \text{ m}^{-3}$ for AO. These values indicate electron populations capable of supporting excitation and ionization processes involved in the formation of reactive oxygen and nitrogen species [12,13,22]. Nevertheless, the electron densities derived from the Saha–Boltzmann equation should be regarded as apparent estimates because the method assumes local thermodynamic equilibrium and may be affected by departures from a Maxwellian electron-energy distribution, line-of-sight integration, and optical effects such as self-absorption. These limitations are particularly relevant for atmospheric-pressure non-thermal discharges, which generally operate under non-equilibrium conditions [22].

Dye	T_e (eV)	n_e (m ⁻³)
AB-194	0.72	2.88×10^{19}
AO	0.81	2.80×10^{20}

Table 6. Calculated apparent electron temperature (T_e) and estimated electron density (n_e) for AB-194 and AO treatments

3.10 Proposed catalyst-assisted degradation mechanism

Degradation is attributed to the combined action of species generated in the gas phase and secondary reactions in the liquid. Electron impact may promote H_2O and O_2 dissociation, producing $\cdot\text{OH}$ and $\text{O}\cdot$, while $\cdot\text{OH}$ recombination may generate H_2O_2 . In the presence of Fe^{2+} , H_2O_2 may react according to $\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \cdot\text{OH} + \text{OH}^-$, increasing hydroxyl-radical availability [9,10,13,16,17]. The OES signals of $\cdot\text{OH}$, N_2/N_2^+ , H_α , H_β , and O I are consistent with these pathways, although they do not quantify oxidant concentrations in the liquid phase.

The decolorization observed for AB-194 is consistent with susceptibility of the azo chromophore to oxidative attack. For AO, decolorization requires disruption of the conjugated triarylmethane system and may generate relatively persistent aromatic intermediates. Although AB-194 exhibited slightly faster apparent kinetics and a higher G_{50} , both dyes reached final decolorization efficiencies above 97%, indicating effective transformation of their chromophoric structures under the applied conditions.

3.11 Energy assessment

The energy performance of the system was evaluated using the energy yield at 50 % decolorization, G_{50} , defined in Eq. (6), and the specific energy consumption, SEC, defined in Eq. (5). These indicators were calculated from the initial dye concentration, treated volume, average electrical power, and treatment time.

For AB-194, the calculation used an initial concentration of 0.1 mM, a treated volume of 5 L, a molar mass of 758.74 g mol^{-1} , an average power of 139.42 W, and a 50 % decolorization time of 14.27 min, equivalent to 856.2 s. Equation (6) yielded a G_{50} value of 5.72 g kWh^{-1} .

For AO, the calculation used an initial concentration of 0.1 mM, a treated volume of 5 L, a molar mass of 303.83 g mol^{-1} , an average power of 133.71 W, and a 50% decolorization time of 16.00 min, equivalent to 960 s. Equation (6) yielded a G_{50} value of approximately 2.13 g kWh^{-1} .

SEC, calculated for the 90 min treatment using Eq. (5), was 41.83 kWh m^{-3} for AB-194 and 40.11 kWh m^{-3} for AO. The similarity between these values results from the use of the same treated volume and total treatment time in both experiments, together with relatively similar average powers.

The results in Table 7 show that AB-194 reached 50% decolorization slightly earlier than AO and exhibited a higher energy yield. The difference in t_{50} was modest, with values of 14.27 and 16.00 min, respectively, indicating broadly comparable apparent decolorization kinetics. In contrast, the higher G_{50} of AB-194 was influenced not only by its shorter t_{50} but also by its substantially greater molar mass.

Dye	P (W)	t_{50} (min)	G_{50} (g kWh^{-1})	SEC (kWh m^{-3})	Final decolorization (%)
AB-194	139.42	14.27	5.72	41.83	98.66
AO	133.71	16.00	2.13	40.11	97.74

Table 7. Comparison of kinetic and energy parameters for AB-194 and AO decolorization by Fe^{2+} -assisted corona plasma in a recirculating-flow system.

To contextualize system performance, Table 8 compares the G_{50} values obtained in this study with previous reports of dye degradation by atmospheric-pressure plasmas, including flow systems, catalyst-assisted treatments, and corona discharges applied to organic dyes. Because experimental conditions, initial concentration, volume, geometry, power, and operating mode differ among studies, this comparison should be interpreted as an order-of-magnitude reference rather than as a direct equivalence.

Reference	Contaminant	System	G_{50} (g kWh^{-1})	Flow/volume
This study	AB-194	Corona + catalyst, recirculation	5.72	Continuous recirculation, 5 L

This study	AO	Corona + catalyst, recirculation	2.13	Continuous recirculation, 5 L
Gómez et al. [32]	Basic Violet I	Corona plasma	0.63	Flow, 5 L
Zhang et al. [33]	Crystal Violet	Plasma + catalyst	6.91	Batch
Torres-Segundo et al. [34]	AB-194	Corona plasma + FeSO ₄	0.621	Batch, 250 mL
Torres-Segundo et al. [35]	Direct Orange 39	Plasma	0.825	Batch
Handoko et al. [17]	Reactive Blue 21	Corona + Fe ²⁺	0.27	Batch/plasma reactor
Segundo et al. [36]	Rhodamine B	Corona discharge	2.84	Batch, 250 mL

Table 8. Comparison of reported G₅₀ values for dye degradation by plasma technologies and catalyst-assisted systems.

4. Conclusions

Atmospheric-pressure corona plasma operated in a recirculating-flow system and assisted by ferrous species efficiently decolorized AB-194 and AO solutions, achieving efficiencies of 98.66% and 97.74%, respectively, after 90 min. The discharge exhibited stable electrical behavior during the analyzed operating period, with average powers of 139.42 W for AB-194 and 133.71 W for AO. Fitting to the apparent pseudo-first-order model yielded rate constants of 0.0486 min⁻¹ for AB-194 and 0.0433 min⁻¹ for AO, with coefficients of determination of 0.9385 and 0.9929, respectively, and times to reach 50% decolorization of 14.27 and 16.00 min. The specific energy consumption was 41.83 kWh m⁻³ for AB-194 and 40.11 kWh m⁻³ for AO, whereas the energy yields were 5.72 and 2.13 g kWh⁻¹, respectively. These results indicate that both dyes exhibited high final decolorization and broadly comparable apparent kinetics, although AB-194 showed a slightly faster response and a higher mass-based energy yield under the applied conditions.

Addition of the ferrous solution caused an immediate decrease in pH and initial increases in electrical conductivity and turbidity. During treatment, turbidity decreased by approximately 92.2% for AB-194 and 93.4% for AO, whereas conductivity decreased by approximately 5.9% for AB-194 and increased by approximately 3.3% for AO. Optical emission spectroscopy showed bands and lines associated with excited species such as OH, N₂, N₂⁺, H α , and H β , consistent with reactive chemistry at the plasma–liquid interface. The slightly faster apparent decolorization of AB-194 may be associated with susceptibility of its azo chromophore to oxidative attack, whereas AO required disruption of its conjugated triarylmethane structure. However, the decrease in absorbance primarily demonstrates alteration of chromophoric groups and does not confirm complete mineralization or the absence of transformation products. Key limitations include the absence of total organic carbon, residual iron, and toxicity measurements, which preclude confirmation of complete mineralization or product safety. Future work should therefore optimize Fe²⁺ concentration, identify the products formed, quantify mineralization, and assess system scale-up under controlled flow-rate, effective-contact-time, and energy-consumption conditions.

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Declarations

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César Torres, Horacio Martínez, Joséfina Vergara, Erik Torres; Resources: Pedro Guillermo Reyes; Supervision: Pedro Guillermo Reyes, Aarón Gómez, César Torres, Horacio Martínez, Joséfina Vergara, Erik Torres; All authors read and approved the final manuscript.

Competing Interests

The authors declare no competing interests.

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