Evaluation of an aqueous solution degradation of textile dye Permalon Rhodamine B by non-thermal plasma treatments

Evaluación de la degradación de una solución acuosa de colorante textil Permalon Rhodamine B mediante tratamientos de plasma no térmico

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Key Handbooks

The main contributions to the generation of science and technology in this research are mainly reflected in obtaining knowledge for wastewater treatment through non-conventional technologies, such as plasma treatments. In particular, it seeks to establish a methodology that allows more efficient treatments. The key aspects that must be understood to apply to the generation of universal knowledge are the chemical and physical changes generated under this methodology, as they are key to adequate wastewater treatment and optimization of the results obtained. From this perspective, the conclusion derived from this research can be summarized in the adequate degradation of the pollutant under study, which other methodologies have not achieved. The authors of this chapter do not have any scholarship (CONAHCYT, PRODEP or external) and come from State Public Institutions (Autonomous University of the State of Morelos). The keywords most commonly used are plasma, degradation, rhodamine, wastewater, electrical conductivity, total dissolved solids, chemical oxygen demand, and organic carbon.

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Abstract

The degradation generated by plasma treatments of the dye Permalon Rhodamine B in aqueous solution (123 mg/L) was evaluated. The degradation was quantified as a function of the exposure time (240 min) and the plasma generation current (20.0, 30.0, 40.0 and 50 mA) at a voltage of 2000 V. The changes (degradation) were monitored by Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC). Total dissolved solids (TDS) and electrical conductivity (EC) were also measured. The degradation of the dye in terms of TOC in the solution was maximum (97 %) for the 50 mA plasma treatment. As the current increases, the COD in the solution also increases. For the 50 mA treatment, the COD increased by 27%. The TDS and EC increased as the exposure time increased. The higher the current and exposure time, the greater the degradation of the dye

Objectives	Methodology	Contribution
Air plasma degrading — Evaluate physical and chemical changes	The solution is subjected to plasma — Plasma reactor — Treated solution — Measured: TDS, EC, COD and TOC	TDS — EC — Analysis — COD — TOC
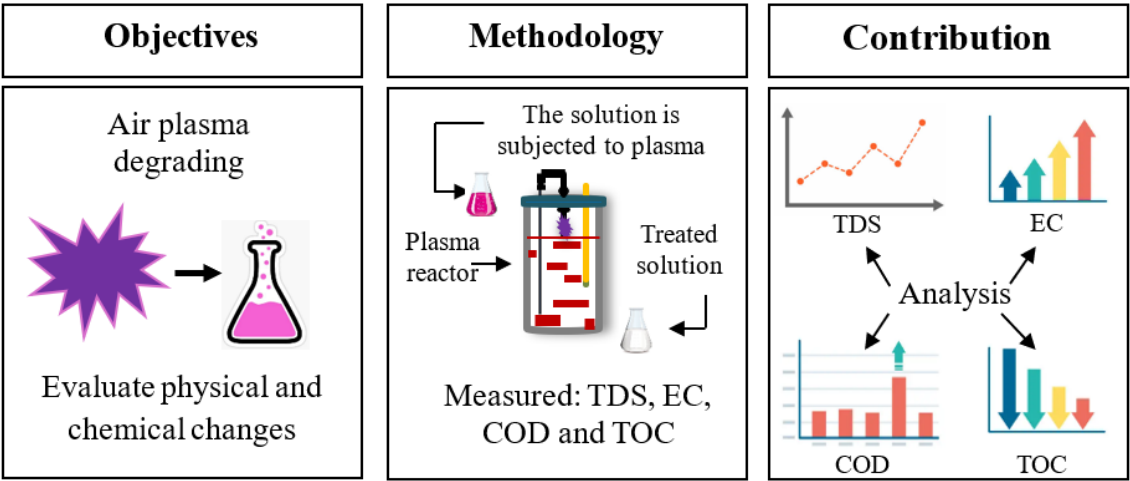
Plasma, degradation, rhodamine B

Resumen

Se evaluó la degradación generada mediante tratamientos de plasma del colorante Permalon Rhodamine B en solución acuosa (123 mg/L). Se cuantificó la degradación como función del tiempo de exposición (240 min) y de la corriente de generación del plasma (20.0, 30.0, 40.0 y 50 mA) a un voltaje de 2000 V. Los cambios (degradación) se monitorearon mediante Demanda Química de Oxígeno (DQO) y Carbono Orgánico Total (COT). También se midieron solidos disueltos totales (SDT) y conductividad eléctrica (CE). La degradación del colorante en términos del COT en la solución fue máxima (97 %) para el tratamiento de plasma de 50 mA. Conforme la corriente incrementa, la DQO en la solución también aumenta; para el tratamiento de 50 mA la DQO incrementó un 27 %. Los SDT y la CE aumentaron conforme el tiempo de exposición sucede. A mayor corriente y tiempo de exposición, mayor degradación del colorante.

Objetivos	Metodología	Contribución
Degradar por plasma de aire — Evaluar cambios físicos y químicos	La solución se somete a plasma — Reactor de Plasma — Solución modificada — Se mide: SDT, CE, DQO y COT	SDT — CE — Análisis — DQO — COT
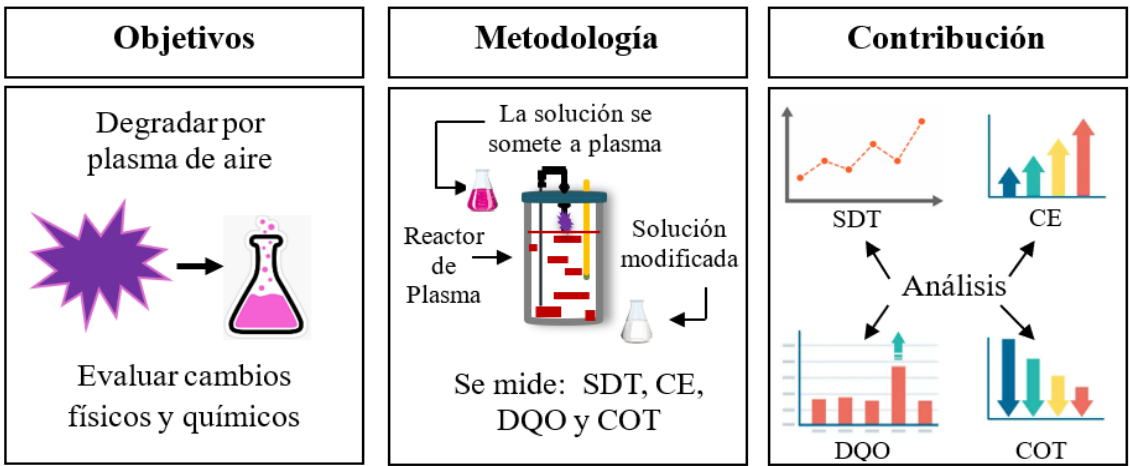
Plasma, degradación, rhodamine B

Introduction

The dye Rhodamine B is one of the most widely used substances in the industry. It is mainly used in the textile industry, paints, manufacturing pens, dyes, explosives, carbon sheets, stamp inks, and even biscuits (Imam & Babamale, 2020; Hamdaoui, 2011).

This dye is one of the most toxic due to its high stability and non-biodegradability, which is why it is present in textile industrial wastewater. On the other hand, since it prevents the passage of light and inhibits the bioprocesses of aquatic plants and microorganisms that cohabit in water bodies, generating anoxic conditions (Sharma et al., 2022), as well as inducing inflammation of the organs of living beings (Seerangaraj et al., 2021; Nasr et al., 2023), it becomes a highly harmful compound (Sharma et al., 2022). In general, apart from the Rhodamine B dye, a large amount of toxic and dangerous organic or inorganic compounds are dumped into bodies and water currents, destroying the ecological system and human health because they have not been previously treated or degraded by some wastewater treatment methodology (Nasr et al., 2023).

Alternative procedures are currently being developed and researched to address the problem of water body pollution. In particular, advanced oxidation processes (AOP) are studied, representing a promising option of techniques and an active field of research for removing dyes. These processes include atmospheric pressure air plasmas, where degradation occurs when reactive molecules, ions and/or radical species created during the plasma discharge in the gaseous or liquid phase attack organic molecules and oxidize them (Ma et al., 2021; Aggelopoulos et al., 2024; García et al., 2017; Aggelopoulos., 2022; Pandiyaraj et al., 2021; Ghezzar et al., 2009).

In general, these non-thermal plasma degradation processes have the advantages of high efficiency, no secondary pollution and low energy consumption (Petrović et al., 2024; Hentit et al., 2014). Considering the degradation characteristics of atmospheric pressure air plasmas and the multiple advantages of this methodology, the present experimental research aims to evaluate the degradation percentage generated by this methodology in a solution of the dye Permalon Rhodamine B at an initial concentration of 123 mg/L. The degradation is evaluated by measuring Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC). The behaviour of total dissolved solids (TDS) and electrical conductivity (EC) is also determined; all of this is a function of the time of exposure of the solution to the treatments and the plasma generation parameters.

The analysis and observation of the degradation behaviour of the dye of interest in aqueous solutions through this research provide a solid basis for developing effective methodologies in treating industrial wastewater with similar characteristics. This experiment uses atmospheric pressure air plasma, which can offer an innovative and efficient alternative to removing contaminants. In the methodology section, the experimental device's particularities, the dye's chemical properties, and the techniques selected to evaluate the variables of interest are detailed and presented. The results section presents graphs illustrating the evolution of chemical oxygen demand (COD), total organic carbon (TOC), total dissolved solids (TDS) and electrical conductivity (EC) of the solution over the exposure time and as a function of the power of the plasma used. These data allow a precise analysis of the effectiveness of the treatment. Finally, in the conclusions section, the most significant observations are highlighted, which could guide future research and applications in wastewater treatment.

This approach not only underlines the importance of understanding the dynamics of dye degradation but also highlights the potential of using advanced technologies, such as plasma, to improve the quality of treated water.

Methodology

The experiment was carried out considering an initial concentration of 123 mg/L. To obtain this value (123 mg/L), 246 mg of Permalon Rhodamine B dye powder was added to 2.0 L of distilled water. The dye was weighed using an ADAM brand analytical balance, model PW 254. This concentration was chosen for all the experiments performed. The Permalon Rhodamine B dye is identified with C.I: 45170. It was used without any purification or physical or chemical changes. Its formula and molecular weight are $C_{28}H_{31}ClN_2O_3$ and 479.01 g/mol, respectively.

The plasma treatment process is carried out as follows. It begins by exposing 500 ml of the solution of interest at a concentration of 123 mg/L to the air plasma. The plasma is generated with specific determined values (voltage and current) in a high-voltage source (SPELLMAN SL600), as shown in Figure 1 (Ávila *et al.*, 2023).

Box 1
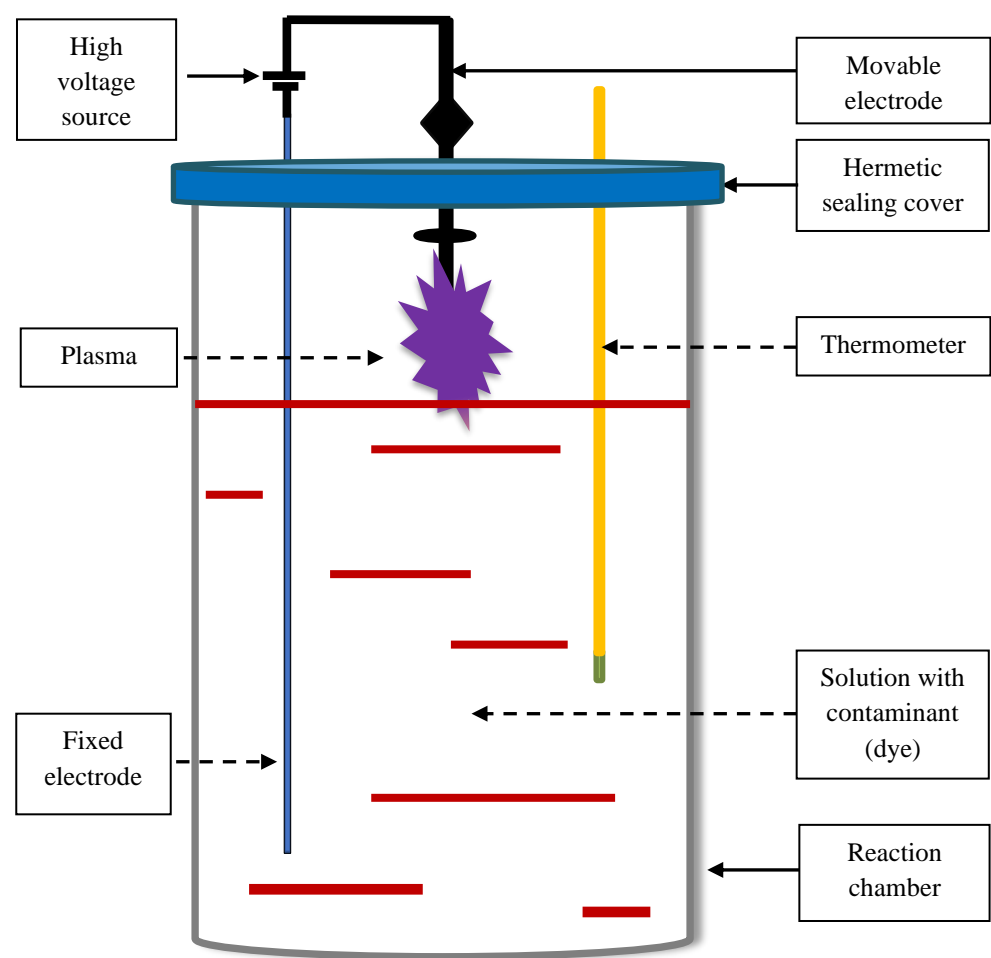
Figure 1
Diagram of the solution exposure to plasma and components of the experimental device

Source: Own elaboration

The device consists of a hermetically sealed glass chamber with a capacity of 1.0 L, two tungsten electrodes, a mercury thermometer, and a high-voltage source. In this device, one electrode is immersed in the solution of interest. At the same time, the other, which is movable, is placed at a distance of approximately 2 mm to facilitate plasma generation. This plasma is formed at the interface between the air in the reaction chamber and the solution, as illustrated in the diagram. The thermometer remains immersed in the solution, allowing the temperature to be measured at all times. Four treatments were carried out with different electric currents and the same voltage (2000 V). Likewise, the values of exposure time (240 minutes), concentration (123 mg/L), and volume of solution treated (500 mL) were kept constant. All treatments started with a temperature value of 28 °C. The electric current values used to generate the plasma are specified in Table 1.

Box 2

Table 1
Electric current parameters used to generate the air plasma in the present experiment

Treatment	Electric current (mA)
T1	20
T2	30
T3	40
T4	50

Source: Own elaboration

The parameters of interest were measured every 20 minutes for electrical conductivity (EC) and total dissolved solids (TDS) (HI 9813-6N potentiometer). In these cases, the plasma treatment is interrupted to perform the measurement. Once the necessary measurements have been taken, the sample solution is added to the reaction chamber and the treatment is continued. This procedure is repeated until the 240 minutes have elapsed. On the other hand, the measurement of chemical oxygen demand (COD) and total organic carbon (TOC) (Hach DRB 200 digester, Hach DR 3900 spectrophotometer) was carried out before starting the respective treatment and at the end of the experiment.

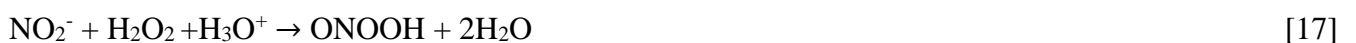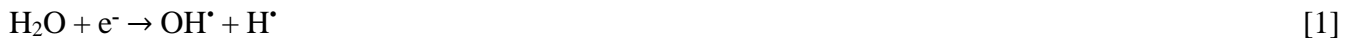The values of chemical oxygen demand and total organic carbon were determined according to the procedure established by the Hach method. Vials ranging between 3 - 150 mg O_2/L and 15 - 150 mg C/L were used. All testing was performed with analytical-grade chemicals.

Results and discussion

The changes generated in the degradation of the dye in an aqueous solution are presented after exposure to air plasma generated by different electric currents and atmospheric pressure. The changes concerning electrical conductivity, total dissolved solids, chemical oxygen demand, and total organic carbon of the solution are analyzed over time.

In this treatment, the chemically active species that may be present and explain the mechanisms of dye degradation when subjected to plasma are the following (Safenraider *et al*., 2020; Alarcón *et al*., 2022). Reactions [1] – [20].

$$H_2O + e^- \rightarrow OH^\bullet + H^\bullet \tag{1}$$

$$O_2 + e^- \rightarrow 2O \tag{2}$$

$$O_2 + O \rightarrow O_3 \tag{3}$$

$$N_2 + e^- \rightarrow N^\bullet + N^\bullet \tag{4}$$

$$O + N_2 \rightarrow NO^\bullet + N^\bullet \tag{5}$$

$$NO^\bullet + O^\bullet \rightarrow NO_2 \tag{6}$$

$$N^\bullet + O_2 \rightarrow NO^\bullet + O \tag{7}$$

$$NO^\bullet + N^\bullet \rightarrow N_2 + O \tag{8}$$

$$H_2O + e^- \rightarrow H_2O^+ + 2e^- \tag{9}$$

$$H_2O^+ + H_2O \rightarrow H_3O^+ + OH^\bullet \tag{10}$$

$$OH^\bullet + OH^\bullet \rightleftharpoons H_2O_2 \tag{11}$$

$$2O_3 + H_2O_2 \rightarrow 3O_2 + HO^\bullet \tag{12}$$

$$O_3 + HO_2^{\bullet-} \rightarrow O_2^{\bullet-} + HO^\bullet + O_2 \tag{13}$$

$$2NO^\bullet + O_2 \rightarrow 2NO_2 \tag{14}$$

$$2NO_2^- + 2H^+ \rightleftharpoons NO^\bullet + NO_2 + H_2O \tag{15}$$

$$2NO_2 + H_2O \rightleftharpoons 2NO_3^- + NO_2 + 2H^+ \tag{16}$$

$$NO_2^- + H_2O_2 + H_3O^+ \rightarrow ONOOH + 2H_2O \tag{17}$$

$$ONOOH \rightleftharpoons NO_2 + HO^\bullet \tag{18}$$

$$ONOOH \rightarrow HNO_3 \hspace{8cm} [19]$$

$$ONOOH + H_2O \rightleftharpoons ONOO^- + H_3O^+ \hspace{6cm} [20]$$

Reactions [1] – [8] (primary species) are favoured and stabilized in water, given the experimental system (Lukes *et al.*, 2014; Cadorin *et al.*, 2015). These have the possibility of forming secondary species (Reactions [9] – [20]) that are capable of inducing chemical degradation reactions of organic compounds in the medium. On the other hand, the interaction of high-energy electrons resulting from the generation of non-thermal plasma in the study solution induces different processes, such as hydrolysis and electron impact ionization, that favour recombination processes and the appearance of different species (Bruggeman *et al.*, 2016).

Electrical conductivity and total dissolved solids

Given the conditions under which this experiment was carried out (atmospheric pressure, ambient temperature, electric current for generating the plasma, treated volume, solution concentration and exposure time), it can be observed in Figures 2 and 3, respectively, that both the electrical conductivity and the total dissolved solids of the solution increase. The electrical conductivity of a solution increases as the amount of dissolved ions increases. Total dissolved solids include substances that dissociate into ions (such as salts, acids and bases) and those that do not, but in general, the direct relationship between CE and TDS is clearer when the dissolved solids generated are ions.

Mainly, an increase in electrical conductivity and total dissolved solids for all treatments as a function of exposure time is observed in Figures 2 and 3. The longer the exposure time, the greater the generation of ions (electrical conductivity and total dissolved solids). The above expression suggests that plasma exposure influences the solution, possibly generating ionic species or radicals that increase electrical conductivity (More *et al.*, 2020; Ávila *et al.*, 2023).

Box 3
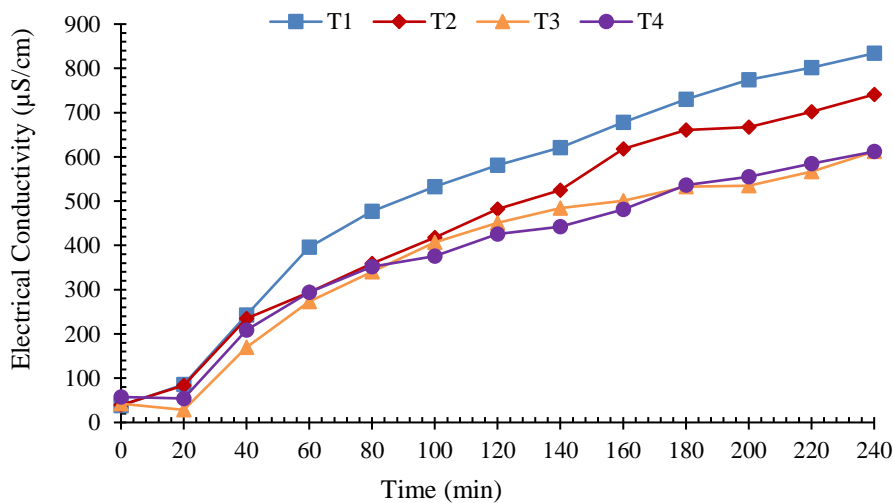
Figure 2

Behaviour of electrical conductivity as a function of time and electric current

Source: Own elaboration

The most significant increase (~94.6%) was observed for treatment T1, with the initial value changing from 38.1 to 834 µS/cm. Treatments T3 and T4 saw a minor change, reaching only 612 µS/cm and below treatment T2 (741 µS/cm). This behaviour indicates that the electric current used to generate the plasma during the treatment has a particular effect on generating ionic species in the Rhodamine B solution (Chen *et al.*, 2015).

Box 4

Figure 3

Change in total dissolved solids in the solution

As the current increases, a decrease in electrical conductivity and total dissolved solids is observed; these phenomena are more prominent at minute 240 of exp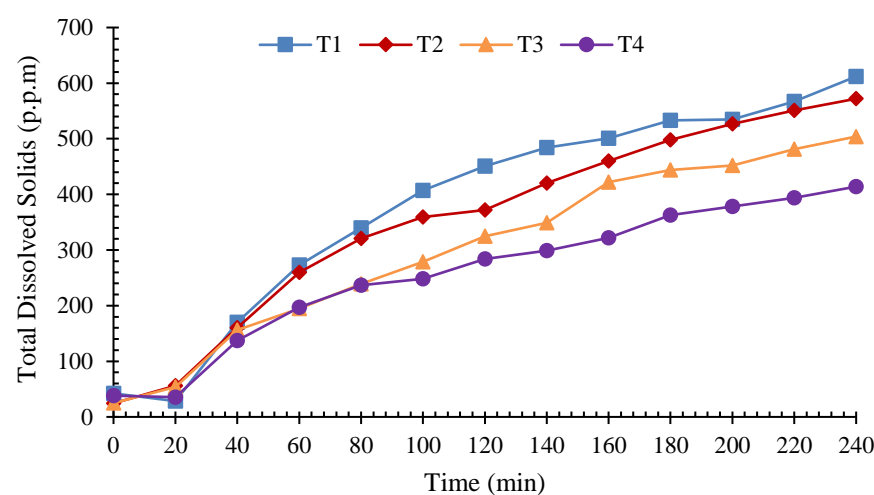osure for all treatments. Such behaviour is due to a more significant generation of ions or secondary products at a low plasma generation current. This type of transformation could be related to oxidation processes or dye degradation by the plasma (Komarov *et al.*, 2020). In particular, for total dissolved solids, a significant difference (32.3%) is observed between the final value reached by treatment T1 (20 mA) and the final value of treatment T4 (50 mA). In general, both electrical conductivity and total dissolved solids show an increasing behaviour as the exposure time increases and a decreasing one as the current of the treatments increases.

Chemical oxygen demand

The COD was determined for the final samples of the treatments (240 min) (Figure 3.3). For treatments T1 and T2, the COD decreases concerning the value of the untreated sample (6.5 and 5.1 %, respectively); however, it is observed that for all treatments, the COD value increases as a function of the electric current of plasma generation; which allows inferring an increase of products and byproducts generated by the interaction of the solution of interest with the plasma that requires more oxygen to oxidize.
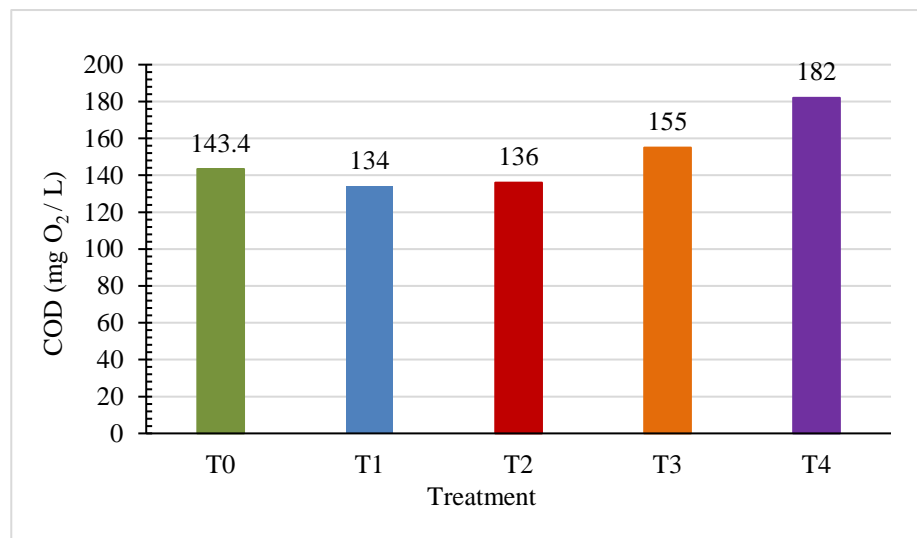
Box 5

Figure 3

Behavior of the change in the chemical oxygen demand of the solution at the end of the treatments, as a function of the plasma generation current

Overall, the percentage increase between the COD values of treatment T1 and T4 is 35.8%. The values depend on the treatment's physical variables. The higher the plasma generation current, the greater the change in COD. This increase in COD may be due to the decomposition of Rhodamine B under the influence of plasma, giving rise to a variety of organic by-products, such as phenolic compounds, organic acids or smaller molecular fragments, as well as new functional groups that can be more easily oxidized. The formation of carbonyl groups (C=O), carboxyls (COOH) or even smaller nitrogen-containing structures increases the oxygen demand (Chen *et al.*, 2015; Le Thi *et al.*, 2021). These by-products can be more difficult to oxidize than the original Rhodamine B molecule.

In general, the interaction of air plasma with the Rhodamine B solution not only decomposes the dye but also generates a variety of organic and inorganic by-products due to the oxidizing action of the reactive species generated in the plasma. This shows the complexity of the process and its potential to modify the original chemical structure of Rhodamine B significantly.

Total organic carbon

Figure 4 shows the change in the solution's TOC values for the four treatments after 240 minutes of exposure. In this case, a decrease in the values is observed depending on the electric current used to generate the plasma.

Box 5
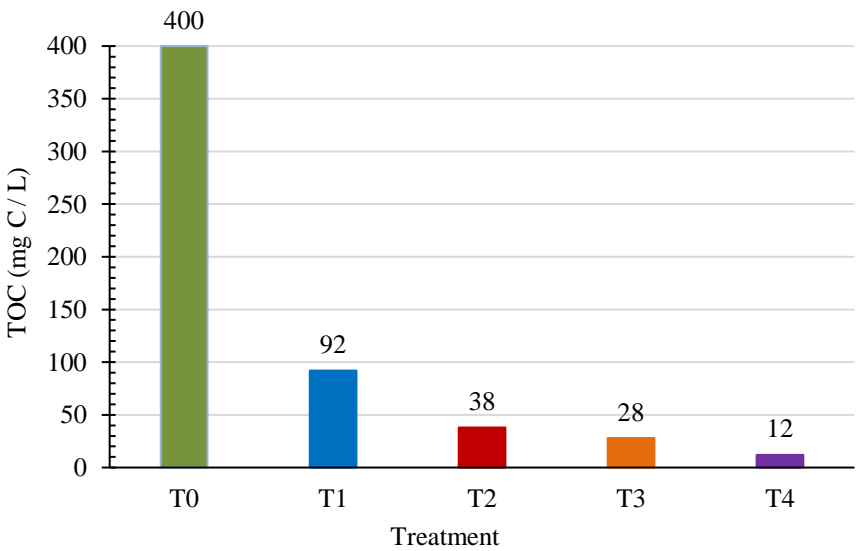
Figure 4

Change in total organic carbon values in solution as a function of electric current

Source: Own elaboration

At 240 minutes of exposure to plasma generated with an electric current of 50 mA, the TOC value decreased from 400.0 mg C/L to 12.0 mg C/L, which corresponds to a dye removal of 97.0 %, while for the minimum electric current value (20 mA) it decreased to 92 mg C/L, which corresponds to a removal value of 77.0 %.

Table 3.1 shows the measured and percentage values of COD and TOC for each treatment at 240 minutes of exposure. These data show that increasing the plasma electric current generates a more significant change in the TOC removal values.

Box 6

Table 2

Percentage change of dye in solution as a function of plasma generation current

Electric current (mA)	COD (mg O₂ / L)	Change (%)	TOC (mg C / L)	Change (%)
00	143.4	Initial value	400.0	Initial value
20	134.0	06.5 ↓	92.0	77.0 ↓
30	136.0	05.1 ↓	38.0	90.5 ↓
40	155.0	08.0 ↑	28.0	93.0 ↓
50	182.0	26.9 ↑	12.0	97.0 ↓

Source: Own elaboration

The results obtained from TOC suggest that plasma treatment mineralizes organic matter. In particular, it has been reported that mainly intermediate products are generated, such as benzoic acid, paradioxybenzene, benzoquinone, and benzene, among others, as well as some carboxylic acids, such as formic, oxalic, and malonic acids. All carboxylic acids can be decomposed into inorganic carbon (Gai *et al.*, 2014).

Conclusions

Given the specific characteristics of this type of treatment and the chemically active species that may be present, the degradation of the dye, when subjected to plasma, had a particular behaviour. In general, both the electrical conductivity and the total dissolved solids showed an increase in behaviour as the exposure time increased and a decrease in behaviour as the treatment current increased. The chemical oxygen demand changed concerning the initial value of the solution, which indicates modifications to the original molecule and its decomposition into byproducts that require more oxygen for higher values of plasma generating current. On the other hand, the total organic carbon of the solution decreased as the treatment current increased, thus achieving the mineralization of the dye. This type of plasma-based treatment offers a potentially efficient approach for the removal of organic contaminants, such as Rhodamine B, in an aqueous solution since it not only degrades the original compound but can also contribute to the reduction of the organic load by generating simpler and possibly less contaminating products. However, the variability in the products formed and the possible formation of toxic or difficult-to-remove byproducts must be carefully evaluated in large-scale applications to ensure the safety and effectiveness of the process.

Conflict of interest

The authors declare that they have no conflict of interest. They have no financial interests or personal relationships that could have influenced this book.

Authors' contribution

Gómez-Anzures, Uriel Yosimar: Supported the execution of the experiments and application of the research methods and techniques. He contributed to the laboratory analysis.

Alarcón-Hernández, Fidel Benjamín: Design and implementation of the project idea, the research methods and techniques applied. Supported the development of the experimentation, performed the data analysis and wrote the work.

Fuentes-Albarrán, María del Carmen: Contributed to the design of the research, type of research, laboratory analysis, analysis of collected data and writing the article.

Gadea-Pacheco, José Luis: Supported the laboratory analysis. Worked on writing the article.

Availability of data and materials

All data used to support the findings of this study are included in the work.

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Abbreviations

COD	Chemical Oxygen Demand
TOC	Total Organic Carbon
TDS	Total Dissolved Solids
EC	Electrical Conductivity
AOP	Advanced Oxidation Processes

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