

## Research Article

# Evaluation of the Degradation Process of Diethyl (3H-1-Ethoxy-3-phenoxazinylidene) Ammonium Chloride in Water, after Exposure to Nonthermal Plasma at Atmospheric Pressure

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The present study characterizes the degradation of diethyl dye (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride in water (concentration of 20.00 mg/L), after its exposure to nonthermal plasma of air at atmospheric pressure. The exposure time of the solution of interest to the plasma was 250 minutes per treatment. Four different nonthermal plasma generation conditions were considered (15, 20, 25, and 30 mA) at a voltage of 2000 V. The change in dilution was determined by UV/VIS spectrophotometry (absorbance and concentration), chemical oxygen demand (COD), pH, and bioassays with lettuce seeds (*Lactuca sativa*). Given the increase in temperature of the sample under study due to exposure to plasma, the change in each of the measured parameters was also explored as an exclusive function of the increase in temperature (27°C to 44°C). The measurements did not show the influence of temperature on the changes observed in the parameters of interest in this experiment. In general, the maximum degradation obtained from the samples analyzed at the end of the treatments corresponds to the 30 mA treatment. For this, reductions of 70.4% in absorbance, 71.5% of dye concentration, 81.6% of COD, and 51.85% of pH were achieved. The germination percentage of the lettuce seeds obtained was 74%. The degradation of the dye in the solution of interest depends on the plasma generating parameters high power and the high degradation rate.

## 1. Introduction

Water pollution is a global problem. Industry is one of the main sources of water pollution as it produces pollutants that are harmful to humans and the environment. In developing countries, around 90% of wastewater is discharged without treatment to different bodies of water and approximately two million tons of industrial, domestic, and agricultural waste are discharged into rivers or canals [1].

In particular, the textile industry requires a large amount of water for its production processes. From 100 to 200 L of water are necessary to produce one kilogram of textile products. The wastewater generated by these industries is

highly polluted due to the presence of colorants, surfactants, inorganic salts, and different chemical compounds used in their processes [2]. As a consequence, these effluents are present in high concentrations of biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SSs), total solids (STs), heavy metals, and salts, in addition to having low levels of biodegradability. Therefore, these effluents are considered difficult to treat [3, 4].

Currently, there are different types of treatment for wastewater from the textile sector, such as physicochemical [5–8] and biological [9–11] methods. These technologies have shown advantages in the treatment of these effluents; however, some of these methods present restrictions in their

application, as they are not considered economically feasible due to their high cost, the formation of by-products of undesirable reactions, and the production of sludge, which decreases its efficiency [12].

Plasma technology is an alternative for treating effluents from the textile industry. Additionally known as the fourth state of matter, this technique consists of a partially ionized gas consisting of electrons, ions, and free radicals [13]. Plasmas can be classified as thermal and nonthermal. In thermal plasmas, the temperature inside them reaches millions of degrees (107–109°C), the same for electrons as for heavy species. There are other types of thermal plasmas, with certain industrial applications, which are generated at high pressures, above 133 millibars (133 mbar), just over a tenth of an atmosphere. On the other hand, nonthermal plasmas are characterized by the fact that the temperature of heavy species (neutral particles and ions) is close to room temperature (25–100°C). Cold plasmas are usually produced at low pressure ( $p < 133$  mbar) in reactors with different geometries. In such reactors, plasmas are generated by direct current, radio frequency, microwave, or pulsed discharge systems [14].

Nonthermal plasmas are an emerging technology, with different applications such as the elimination of different types of compounds, among which are the following compounds: volatile organic compounds (VOCs), odors, organic materials such as chlorofluorocarbons (CFCs), and combustion gases (such as nitrogen oxide ( $\text{NO}_x$ )) and sulfur oxide ( $\text{SO}_x$ )), and particle reduction. Thus, they constitute a promising alternative for the treatment of textile effluents. In addition, they have advantages over other treatment systems, since their cost is moderate and they do not generate toxic by-products and operate at room temperature [15].

From this perspective, in a hybrid gas-liquid electrical discharge reactor, nonthermal plasmas can form directly on the surface of the water, and chemically active species generated at the gas-liquid interface can easily enter the water for inactivation of microorganisms and/or degradation of compounds.

In this study, the changes generated in a solution of diethyl dye (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride at an established concentration of 20.00 mg/L after exposure to nonthermal plasma are analyzed. This colorant is of a basic type depending on its application form. It is widely used in different industries that produce paint, ceramics, plastics, leather, and inks, in addition to the textile industry in fiber dyeing. It is identified by CAS number 2787-91-9.

## 2. Materials and Methods

**2.1. Preparation of the Study Solution.** 0.25 g of diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride colorant is poured into 2500 mL of distilled water and mixed thoroughly (the dye was weighed using an ADAM brand weighing machine, model PW 254). A stock solution obtained a concentration of  $1 \times 10^{-4}$  g/mL.

The experimental solution was achieved using 100 mL of the stock solution which is diluted in 400 mL of the distilled

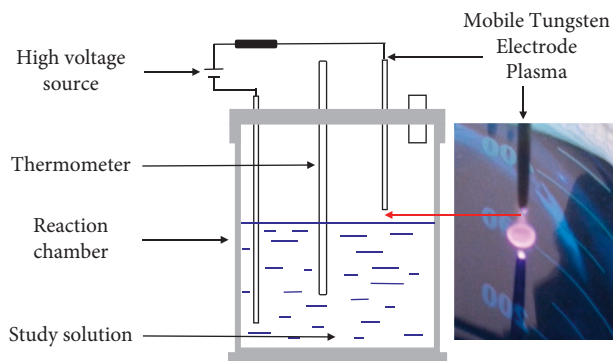


FIGURE 1: Scheme of the experimental device used for the treatment of the solutions by nonthermal plasma.

water. Through this procedure, a new concentration of 20.00 mg/L was obtained as the final working concentration.

**2.2. Experimental System.** A diagram of the experimental device is shown in Figure 1. It consists of a glass container that can be hermetically closed (reaction chamber) with a volume of 1.0 liters, two tungsten electrodes, a mercury thermometer, and a high voltage source. One of the electrodes is fixed, and it always remains within the study solution. The other electrode is mobile (it is never in contact with the study solution) and can be moved closer to or farther away from the surface of the solution for plasma generation. The thermometer is always immersed in the interest solution and can measure its temperature at any time.

To expose the dye solution to plasma treatment, 500 mL of the solution is placed in the reaction chamber of the experimental device used and the nonthermal plasma is then generated. The generation of the plasma is achieved by adjusting the corresponding values of potential difference (V) and electric current (I) in the high voltage source (SPELLMAN SL600). In particular, four different plasma treatments were considered. The parameters for each of the treatments are shown in Table 1. Five repetitions were carried out for each treatment. The results presented are the average value of the measurements taken.

**2.3. Measurement of Interest Parameters.** Once the solution under study is placed inside the reaction chamber, it is hermetically closed; the plasma generation parameters are set, one of the electrodes is introduced into the solution, the high voltage source is put into operation, and the other electrode is adjusted at approximately 2 mm from the surface of the solution to generate the plasma.

After 25 minutes of exposure of the solution under study to plasma, the temperature of the solution is monitored, the treatment is stopped, a sample is extracted to measure the absorbance (Hach DR 3900 spectrophotometer) and the pH (HI 9813-6N potentiometer) and determine the germination percentage of (*Lactuca sativa*) lettuce seeds. Once this has been performed, the exposure of the solution to the

TABLE 1: Parameters considered for the realization of this experiment. Treated volume of contaminated water per experiment: 500 mL.

Treatment	Treatment time (minutes)	Concentration (mg/L)	Voltage (volts)	Electric current (mA)
T1	250	20.00	2000	15
T2	250	20.00	2000	20
T3	250	20.00	2000	25
T4	250	20.00	2000	30

nonthermal plasma is resumed, repeating this procedure until 250 minutes are reached, and the experiment is concluded.

COD is measured at the beginning and end of the treatment (Hach DR 3900 Spectrophotometer). The percentage of degradation of the analyzed pollutant is determined in terms of the absorbance and the concentration calibration curve.

**2.4. Toxicity Bioassays.** Plasma treatment experiments have shown that different types of dye are effectively degraded. However, the change in toxicity of the study solution before and after the treatments is not reported. In this study, seeking to respond to this variable, bioassays are considered for all experiments and in each test performed.

The biological organisms used were uncured seeds (without fungicides or pesticides) of lettuce (*Lactuca sativa*), with a germination percentage higher than 90%. The germination percentage analysis methodology was followed [16].

For this test, five repetitions were performed for each treatment. A negative control (distilled water) and a positive control (solution of interest without exposing the plasma to a concentration of 20.00 mg/L) were used.

**2.5. COD.** Hach's method was used to determine COD. To obtain COD, ultra-low vials (1–40 mg/L) were used. All solutions were prepared with analytical grade chemicals.

**2.6. Percentage of Degradation.** The following equation is used to calculate the percentage of degradation:

$$\% \text{degradation} = \left(1 - \frac{C}{C_0}\right) \times 100\%. \quad (1)$$

In (1), the value of  $C$  is the concentration of the study dye at a certain treatment time and  $C_0$  is the initial concentration of the solution dye without treatment. The absorbance values were related to the respective dye concentrations by a linear function

$$y = 0.1793x + 0.0357. \quad (2)$$

**2.7. Calibration Curve.** Figure 2 shows the calibration curve obtained at a wavelength  $\lambda = 635$  nm. It is used to relate the absorbance values to the concentration (mg/L) of the dye diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride. This curve was obtained by performing 11 different dilutions of a solution with a known concentration of dye.

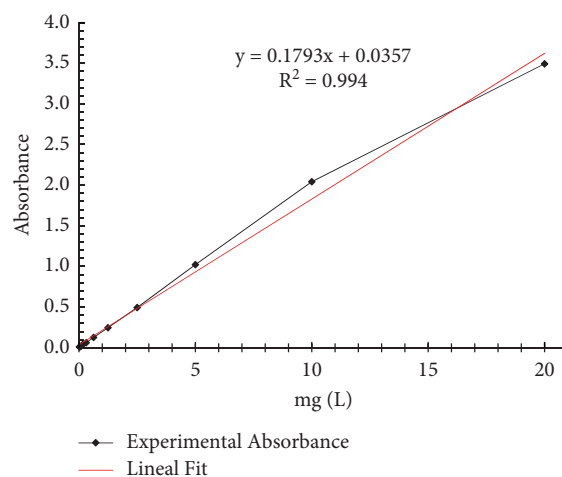


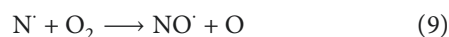
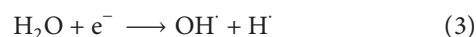
FIGURE 2: Diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride calibration curve.

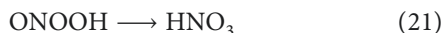
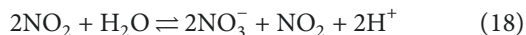
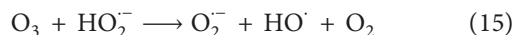
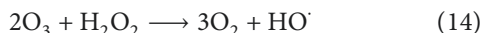
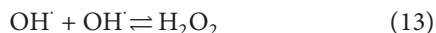
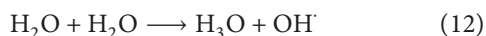
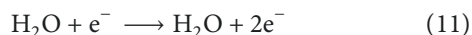
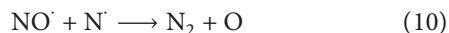
Figure 2 shows the calibration curve of the diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride dye, whose adjustment equation is  $y = 0.1793x + 0.0357$ .

### 3. Results and Discussion

It is observed in Figure 1 that the interaction of the study solution with plasma occurs on its surface (purple coloration due to  $N_2$  in the air [17]). However, given the characteristics of the experimental system (one electrode immersed in the solution with dye and the other in the surrounding air), and due to the generation of different chemically active species, changes are made in the entire solution studied.

The chemically active species that may be present and that explain the mechanism of dye degradation when subjected to plasma are the following [18]. Reactions are as follows:





Reactions (3)–(11) (primary species) are favored and stabilized by the water in the experimental system [19, 20]. They have the possibility of forming secondary species. Reactions (12)–(22) are capable of inducing chemical degradation reactions of organic compounds in the medium.

The interaction of high-energy electrons resulting from the generation of nonthermal plasma in the study solution induces different processes such as hydrolysis and ionization by electron impact; which in turn favor recombination processes and the appearance of different species [21].

One of the changes experienced by the study solution after the plasma treatments is the increase in the temperature of the solution due to the plasma. To determine the possible effects that this temperature increase can generate in the dye solution, the solution of interest was heated until the maximum temperature value reached in the plasma experiments. Similar experiments have been carried out using a different dye [17]. This action was carried out to contrast the obtained results of absorbance (percentage of degradation and concentration) and pH. In all cases, no effect was observed due to the increase in temperature from 27°C to 44°C.

**3.1. Absorbance.** The plasma that had contact with the water heated the sample to a maximum temperature of 44°C. The initial temperature was 27°C, which was nearly the room temperature of the laboratory. Figure 3 shows the changes in the absorbance values of the solution of interest as a function of the plasma treatment time and as a function of the increase in temperature of the solution. It is observed that the absorbance values of the solution of interest do not change

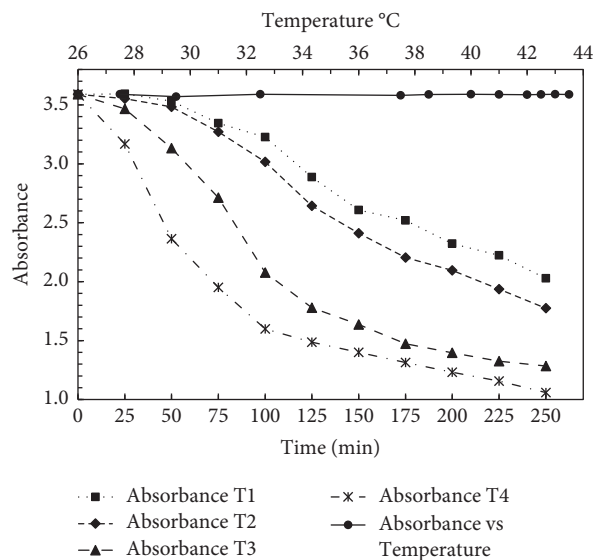


FIGURE 3: Change in absorbance of the diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution as a function of time of exposure to plasma treatment and temperature.

with increasing temperature. On the other hand, the plasma-treated solution shows a decreasing behavior in absorbance.

It should be noted that the last two treatments (T3 and T4) had a faster absorbance decrease behavior than the rest (T1 and T2), especially the T4 treatment, which during the first 125 minutes had a very noticeable reduction with respect to others.

Using (2) of the calibration curve and the absorbance values measured by spectrophotometry at different treatment times, it is possible to calculate the corresponding values of the solution concentration change.

According to (1), an increase in the percentage of dye degradation can be inferred in terms of the decrease in the value of the concentration in the solution, and the above equation can be expressed as a function of exposure time to plasma and the increase in the electrical power of plasma.

For treatment T4, the initial value of concentration is 20.00 mg/L, but after 250 min of treatment, the final dye concentration is 5.70 mg/L, which is equal to a dye loss of 71.5%. In this treatment, the degradation is maximum.

The concentration values of the dye determined by (2) have an associated maximum interval of uncertainty of  $\pm 1\%$ .

**3.2. Chemical Oxygen Demand (COD).** COD was determined for final samples of the treatments (250 min) (Figure 4). Every day, they show a decrescent behavior, which allows to infer a decrease in the concentration of pollutants as a function of the treatment characteristics. COD provides information about the necessary oxygen for oxidizing organic matter in the solution, and in these cases, the COD decreases for all treatments.

In general terms, the changes found in the chemical oxygen demand of the solution at the end of the treatments decreased between 8.3% and 81.6%. The COD values in the graph show a dependence on the physical variables that

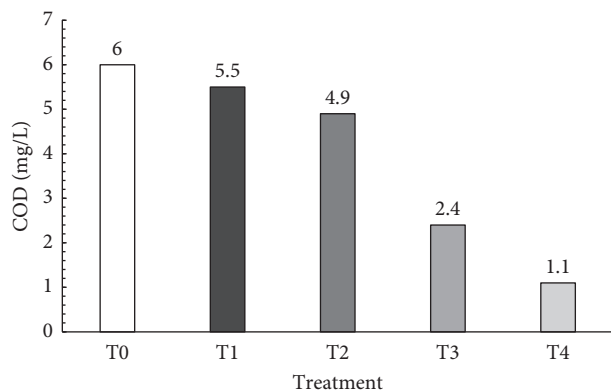


FIGURE 4: COD of diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution samples as a function of treatment. T0 is the diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution sample without treatment.

generate plasma. The higher the power of plasma, the higher the change in COD.

It is observed that the greater efficiency of the treatment with respect to the decrease in COD occurs for the T4 treatment. Corresponding to this treatment, the COD decreases to a final value of 1.1 mg/L, which is equivalent to a decrease of 81.6%.

**3.3. pH.** Figure 5 shows the behavior of the change in the hydrogen ion concentration  $[H^+]$  of the aqueous solution of diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride when exposed to nonthermal plasma with variation in intensity of current for 250 minutes. The behavior of this variable is also presented as a function of the increase in temperature of the diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution.

In general, the solution undergoes a change in pH (it becomes more acidic). It is observed that the pH values show a decrease as a function of the plasma treatment time. A decrease is also observed as a function of the increase in the intensity of the electric current used in each of the treatments. The behavior of the pH change is similar for all treatments; however, it happens at different rates of change depending on the generating power of the plasma.

On the other hand, it is observed that in the initial 100 minutes the pH values experience the greatest decrease and it happens faster. For the remaining treatment time, the pH continues to decrease but at a slower rate. Similar results have been found regarding the behavior of the pH of solutions that are subjected to air plasma treatments [22, 23].

The diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution that was subjected to temperature change (27°C to 44°C) did not show significant pH changes. The pH measurements were performed when the solution of interest returned to room temperature (27°C).

The minimum pH value (2.60) is observed in the final interval of the fourth treatment, that is, when a current intensity of 30 mA is applied during 250 minutes of exposure.

Due to the configuration of the experimental device in which the plasma is generated (an electrode immersed in the

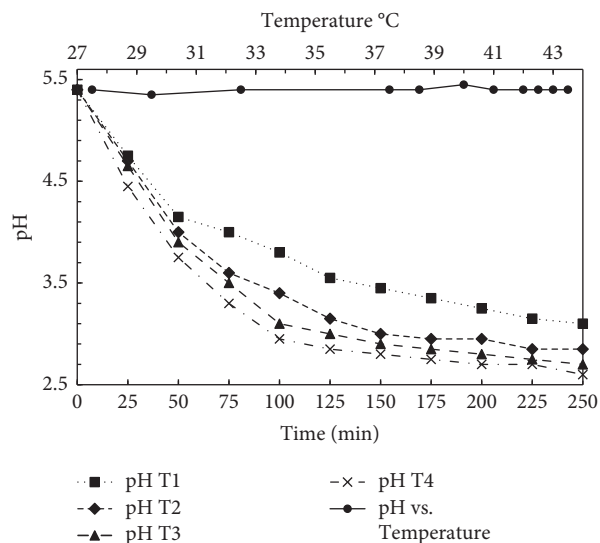
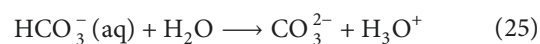
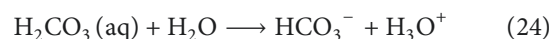


FIGURE 5: pH of the diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution as a function of plasma exposure time, treatment, and temperature.

study solution and another in air), the generation of hydroxyl and hydronium radicals is present, as well as multiple chemical reactions to which they are attributed should be responsible for lowering the pH of the solution of interest.

Basically, the solution under study is in direct interaction with the surrounding air located inside the reaction chamber. The air contains  $CO_2$ , a compound that, when reacting with the plasma system, contributes to a greater generation of hydronium ions. Reactions are as follows:



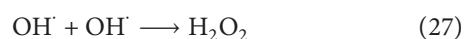
Electrical discharges in interaction with the water surface and the presence of air induce a change in solution pH [24–26] due to the generation of  $HNO_3$ ,  $HNO_2$ , and other by-products that can be formed in plasmas of moist air or water.

In hybrid discharge systems,  $NO$  and  $NO_2$  molecules are formed through the reactions of energetic electrons with  $N_2$  and  $O_2$  in the gas phase [27], which react with  $H_2O$  molecules forming  $HNO_3$  and  $HNO_2$  that decrease the pH [28].

On the other hand, since the system feeds on high-energy electrons, causing ionization, dissociation, and the recombination processes with water molecules,  $OH$  and  $H$  radicals are generated, as described in reactions (26) and (27) [18, 29].



The  $OH$  radicals in the solution recombine generating hydrogen peroxide [18, 30]:



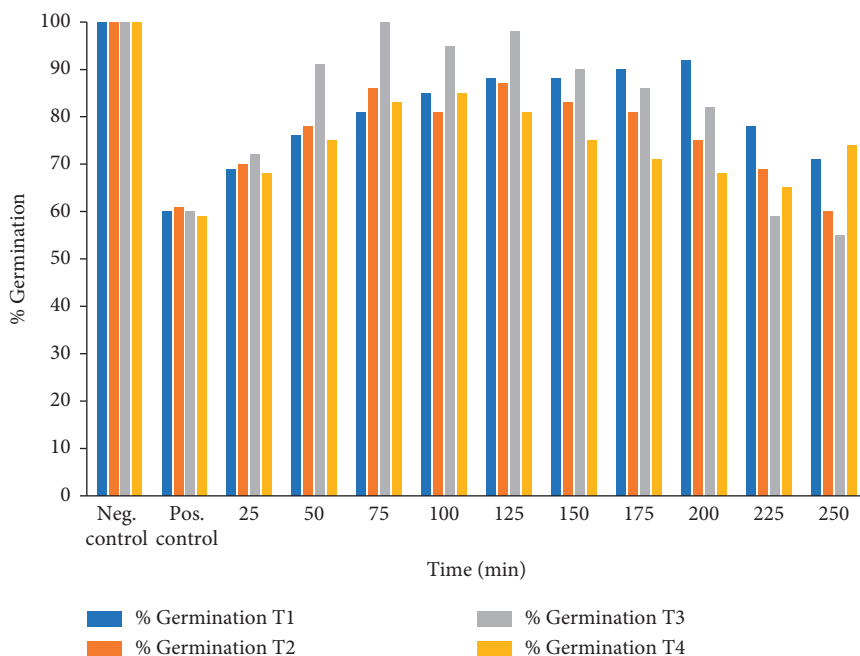


FIGURE 6: Percentage of germination of diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride as a function of the plasma exposure time and treatment.

In general, previous studies using nonthermal plasmas have shown that mixed experimental systems, such as the one used in the present experiment, induce a decrease in the pH of the study solution [24–31].

**3.4. Percentage of Germination.** The results obtained from the germination percentages for each of the treatments as a function of time are shown in Figure 6. The positive control indicates that diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution is relatively toxic. Only 60% of seeds germinated on average for all samples. However, the toxicity decreases as the solution is exposed to plasma and as time passes. The germination percentage increases for each treatment. It is observed that as the time of exposure to plasma increases, the number of germinated seeds increases until it reaches maximum time and then decreases.

The maximum germination percentage corresponds to T3 treatment (lower toxicity). 100% of seeds germinate in the interval of 75 to 125 minutes.

The decrease in the percentage of germination after an exposure time is an indicator of the presence of residual toxic chemical compounds derived from the decomposition of the original dye molecule. These residual compounds are manifested for all treatments, and as expected it happens for different times depending on the electrical power of the plasma.

The lower the electrical power of plasma generation, the longer the exposure time to reach the maximum germination percentage. Treatment T1 reaches the relative maximum at minute 200, treatment T2 at minute 125, treatment T3 at minute 75, and treatment T4 at minute 100. Such maximum values would be better determined on the exposure time scale, if the time intervals for taking the measurement were shorter.

In general, a behavior of the decrease and after the increase in toxicity is observed. It is necessary to prolong the treatments over time to determine, in terms of the germination percentage, at what time the exposure should end.

All diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride solution samples remained toxic at the end of each treatment. Even when other variables (absorbance, percentage of degradation, and COD) have been positively modified, the germination percentage is still below 80%. However, exposure times of the solution of interest to plasma are observed in which the germination percentage is greater than 80%.

There are different technologies applied for the treatment of textile effluents, such as conventional physical, chemical, and biological methods, which operate efficiently and are commercially available; however, some technologies are highly specific and expensive. Furthermore, they are not applied to a wide variety of dyes, and they do not solve the problem of discoloration [10].

On the other hand, advanced oxidation processes (AOPs) constitute an alternative in the treatment of hardly biodegradable compounds. Within these AOPs are photochemical processes [5, 32, 33] and nonphotochemical processes [34, 35].

It has been observed that photochemical processes have the possibility of direct degradation (normally with UV), without adding a chemical reagent, but one of the main disadvantages of these processes is their high-energy consumption, especially electricity, which makes them economically very demanding and of little application. Nonphotochemical processes originate from highly reactive species (mainly the hydroxyl radical) through the transformation of chemical species or through the use of different forms of energy, with the exception of light irradiation. One

of these processes is nonthermal plasma, which is an emerging alternative for the treatment of textile effluents and can be applied to different types of compounds, and unlike other treatment methods, it has a moderate cost and does not generate toxic by-products, in addition to not requiring support fuel, so it does not generate secondary emissions [15].

#### 4. Conclusions

Nonthermal plasmas at atmospheric pressure were shown to be functional in the treatment of water with diethyl (3H-1-ethoxy-3-phenoxazinylidene) ammonium chloride dye. Dye degrades gradually in a great percentage. The plasma treatment time of the solution of interest is a factor that determines the decrease in absorbance (the longer the treatment time, the lower the absorbance value). Likewise, the plasma generating values (potential difference and electrical current) show the decrease in absorbance; the greater the electrical power, the greater the degradation value. Since the absorbance decreases in all cases, it is concluded that the dye molecule is being reduced. On the other hand, the final COD value of each experiment shows that the pollutant used is being decreased since the COD values are reduced. That is, the products of the element to be degraded require less oxygen. The pH values as well as the final percentage of germination suggest that the water obtained in this treatment requires pretreatment before it can be used for any purpose.

#### Data Availability

All data used to support the findings of this study are included within the article.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Acknowledgments

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