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Wastewater management using cold plasma - Degradation of organic dyes in liquid phase

Manejo de aguas residuales utilizando plasma frío - Degradación de colorantes orgánicos en fase líquida

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Abstract

The use of a plasma reaction system for the treatment of wastewater was evaluated by analyzing the degradation of organic dyes in liquid phase. The reaction system allows the treatment of liquids with plasma generated reactive species in tiny bubbles that increase the contact area between the plasma and the liquid, thus reducing treatment time. The system capability to degrade organic matter was studied using organic dyes in aqueous solution. The discoloration reaction of organic dyes such as methylene blue (MB), methyl orange (MO), and methyl red (MR), as well as the reduction of total organic carbon (TOC), and chemical oxygen demand (COD) were analyzed to determine the efficacy of the system and determine its potential use to degrade organic pollutants in aqueous phase. It was found that after 5 minutes of treatment the solutions practically lost the color, and a significant part of the organic matter.

Keywords: wastewater management, degradation, discoloration, dyes, atmospheric plasma.

Resumen

Este El uso de un sistema de reacción por plasma para el tratamiento de aguas residuales fue evaluado mediante el análisis de la degradación de colorantes orgánicos en fase líquida. El sistema de reacción permite el tratamiento de líquidos mediante especies reactivas generadas por plasma dentro de pequeñas burbujas, lo que incrementa el área de contacto entre el plasma y el líquido, y por tanto reduciendo el tiempo de reacción. Se estudió la capacidad del sistema para degradar materia orgánica usando colorantes orgánicos en solución acuosa. Se analizó la reacción de decoloración, así como la reducción de carbón orgánico total, y demanda química de oxígeno para colorantes orgánicos tales como, azul de metileno, naranja de metilo, y rojo de metilo, a fin de determinar la eficacia del sistema y determinar el potencial del mismo para su uso en la degradación de contaminantes orgánicos en soluciones acuosas. Se encontró que después de cinco minutos de tratamiento, las soluciones prácticamente perdieron todo el color, así como una parte significativa de la materia orgánica.

Palabras clave: manejo de aguas residuales, degradación, decoloración, colorantes, plasma atmosférico.

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1 Introduction

The importance of environmental protection has led the way to new research on the development of materials and techniques which allow to clean or reduce toxic substances that are released into the nature (Villabona-Ortíz *et al*., 2021; Lavado-Meza *et al*., 2021; Puentes-Morales *et al*., 2021). Natural ecosystems are under increasing threat from human activities associated with increasing industrialization (Mollah *et al*., 2000; Jiang, *et al*., 2014). Industrial effluents contain different chemicals and organic matter which are causing toxicological and environmental problems. Generally, the treatment of these residues involves the removal of suspended solids, and procedures to remove contaminant agents, organic and inorganic, which are dissolved in the effluent (Gupta and Suhas, 2009). A characteristic treatment to remove contaminants is chemical oxidation (Brisset *et al*., 2011). An alternative to these methods is the use of cold plasma technology which involves the generation of highly reactive chemical species that promote the treatment of effluents (Wang *et al*., 2012). By introducing a gas within a liquid and applying a discharge in the system, it is possible the formation of plasma inside the gas bubbles. Thus, the highly reactive plasma species (ions, free radicals, and electrons) will be in contact with the liquid at the interphase promoting chemical reaction (Yang, *et al*., 2012; Takai, 2008). The study of such systems is constantly growing due to the importance of finding new technologies and more efficient methods to achieve objectives that are more difficult to accomplish with other techniques. The main advantage of the use of cold plasmas for treating liquid is the fact that the generated reactive species are in direct contact with the solution (Magureanu *et al*., 2010; Magureanu *et al*., 2011; Li *et al*., 2014; Bian *et al*., 2013), these species accelerate the decomposition reaction due to energy transfer caused by collisions with the contaminants in the liquid phase.

These systems can be used for the decomposition of organic molecules such as dyes in wastewater (Mollah *et al*., 2000; Jiang, *et al*., 2014; Reddy *et al*., 2013; Shin *et al*., 2000; Abdelmalek *et al*., 2006; Huang *et al*., 2012; Doubla *et al*., 2007; Huang *et al*., 2010; Lachheb *et al*., 2002). In the present work a reaction system is proposed with a configuration adapted to optimize the capacity to generate reactive species within solutions of organic dyes achieving their degradation. The proposed system consists of an electrode submerged in the solution to be treated (which is grounded); the reactive species are generated by producing dielectric barrier discharge between the electrode (a glass covered stainless steel tubing) and the solution. This discharge generates reactive species from a precursor gas and pushed into the solution through a gas diffuser. The advantage of the proposed configuration over previously reported configurations is the increased contact area between the bubbles and the solution.

Degradation of organic matter has been studied by several researchers by monitoring total organic carbon (TOC) and chemical oxygen demand (COD), finding that for dyes the change in TOC is slower than the change in discoloration (Lachheb *et al*., 2002; Tichonovas *et al*., 2013; Reddy and Subrahmanyam, 2012; Zhang *et al*., 2010; Zhou *et al*., 2011). In this study the proposed reaction system was characterized studying the following variables during the reaction process: discoloration factor; TOC and COD change rate; as well as CO_2 production by mass spectroscopy. Is worth noting the importance of analyzing the products of the reaction as well as operating conditions to optimize the process and find potential applications.

2 Materials and methods

2.1 Reagents

The materials used are oxygen (99%, Praxair); methylene blue (Hycel); methyl orange (Hycel); methyl red (Hycel); sodium phosphate dibasic (Sigma); sodium phosphate monobasic (Sigma), and distilled water was used in all solutions prepared.

2.2 Apparatus

Plasma reaction system: Plasma processing systems vary in different aspects such as geometry, components, and design materials. The design of a reaction system depends mainly on its application. Figure 1 shows the schematic of the plasma reaction system in liquid phase that was designed for the treatment of dyes in solution.

The system uses two stainless-steel electrodes, one is a solid rod, and the other is hollow, which allows the precursor gas to be fed to the system. The hollow electrode (connected to a high voltage power supply) is covered by borosilicate glass, which has

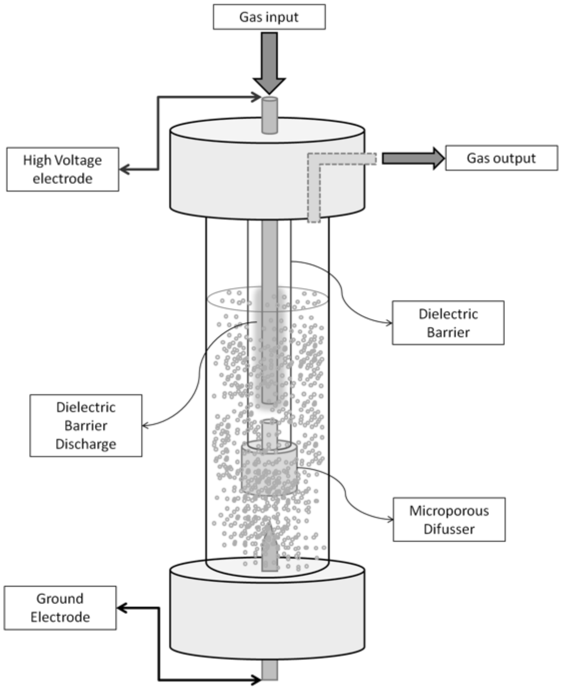

Fig. 1. Schematic of the plasma reaction system in liquid phase.

a diffuser at the end with purpose generating tiny bubbles and increase the contact surface area between reactive species and the liquid phase. The grounded solid rod is in direct contact with the solution (thus making it the ground electrode). The partial discharges are generated at the end of the hollow electrode in a section of the glass tube between the stainless-steel tubing and the liquid phase. The reactive species generated in this zone (ions, molecular fragments, free radicals, electrons) are pushed through the diffuser by the precursor gas flow, and the degradation reaction is carried out in the interface of the bubbles and the solution. This configuration allows for the active zone of the discharge to interact directly with the aqueous phase, which in turn produces other reactive species that contribute to the degradation of organic matter. The active zone of the discharge for oxygen includes O, O_2, O_3, oxygen ions, free radicals, UV light, and high energy free electrons. Since the active zone is in direct contact with water in the interface, it generates other reactive species ($OH^\bullet$, OH^-, H_2O_2, $H^\bullet$, etc.) due to collisions, UV exposure, and chemical reaction (Lukes, Locke, and Brisset, 2012; Meichsner *et al.*, 2013).

2.3 Procedure

The procedure involves placing a 20 mL liquid sample within the reaction system and start a precursor gas flow (300 cm^3/min), connect the hollow electrode to the high voltage power supply (50 kV) and initiate the discharge. Oxygen was used as precursor gas at atmospheric pressure. Reaction times (t) used varied from 15 to 300 seconds. Initial concentrations (C_o) for methylene blue, methyl orange, and methyl red were 100 mg/L. All the solutions were prepared using phosphates as buffer to avoid a change in pH and avoid interference in the results due to pH sensitivity of the organic dyes.

2.4 Reaction characterization analyses

2.4.1 UV-VIS Spectrophotometry

Concentration quantification for the organic dyes was carried out using a Genesys 10 UV UV-Vis spectrophotometer. The wavelength number for methylene blue analysis was 666 nm, for the methyl orange was 465 nm, and for methyl red was 430 nm. Finally, a discoloration factor (F_D) was calculated using the equation:

$$F_D = \frac{\text{Absorbance (initial)-Absorbance (treated)}}{\text{Absorbance (initial)}} \quad (1)$$

2.4.2 Chemical Oxygen Demand (COD)

Chemical oxygen demand was determined using a Hach DR 2800 spectrophotometer with the potassium dichromate technique, and a Hach DRB 200 sample digester. The tests were performed sequentially during the reaction process to obtain measurements of the difference between the initial and final state of the samples.

2.4.3 Total Organic Carbon (TOC)

The analysis to obtain the total organic carbon of the samples and to observe the changes after the reactions were performed using a Hach DR 2800 spectrophotometer, and a Hach DRB 200 sample digester.

2.4.4 Mass spectrometry

In the case of methylene blue, the formation of CO_2 during the reaction of discoloration was measured qualitatively *in situ* using an RGA Prisma Plus mass spectrometer. For this purpose, an initial 200 mg/L methylene blue solution was used with oxygen flows ranging from 12.5 to 100 cm^3/min. The experimental test was carried out for 25 minutes, despite reaction time being only 300 seconds, with the objective to detect the CO_2 evolution afterwards.

3 Results and discussion

3.1 Effect of treatment time on the discoloration of dyes

Discoloration experiments for the dyes were carried out using a neutral pH of 7. Figure 2 shows discoloration factor changes with treatment time. Equation 1 shown in section 2.4.1 was used to calculate F_D, considering the maximum wavelength absorption value obtained previously for each dye. From figure 2 it is observed that F_D values increase with respect to treatment time for all the dyes tested. After 90 seconds of treatment the rate of change for F_D slows down. The maximum values of discoloration factor were achieved after 5 minutes of treatment. For methylene blue the discoloration factor was 0.99, while for methyl red and methyl orange was 0.98.

Dye solutions were analyzed using a UV-Vis spectrophotometer after different treatment times. Spectra for methylene blue (a), methyl orange (b), and methyl red (c), are shown in figure 3. The spectra show a decrease in absorbance for the characteristic peaks of each dye.

This decrease represents a degradation of the chemical structure after the dyes had been treated with plasma, reducing the presence of functional groups responsible for the color in the organic dyes and decomposing them to form CO_2 and low levels of non-toxic organic intermediate compounds (Chandana *et al*., 2015; Sanito *et al*., 2022). The maximum change

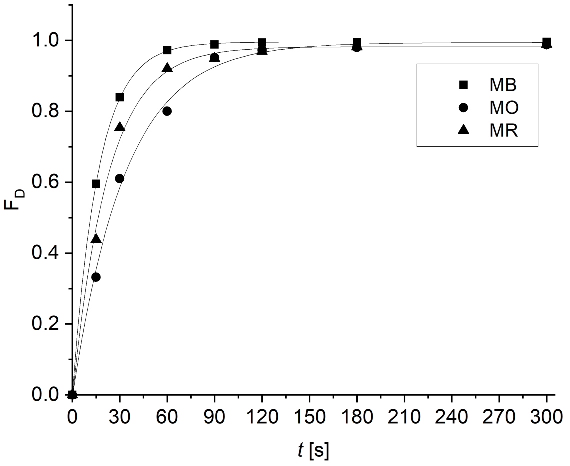

Fig. 2. Discoloration rates for dyes with initial concentrations of 100 mg/L.

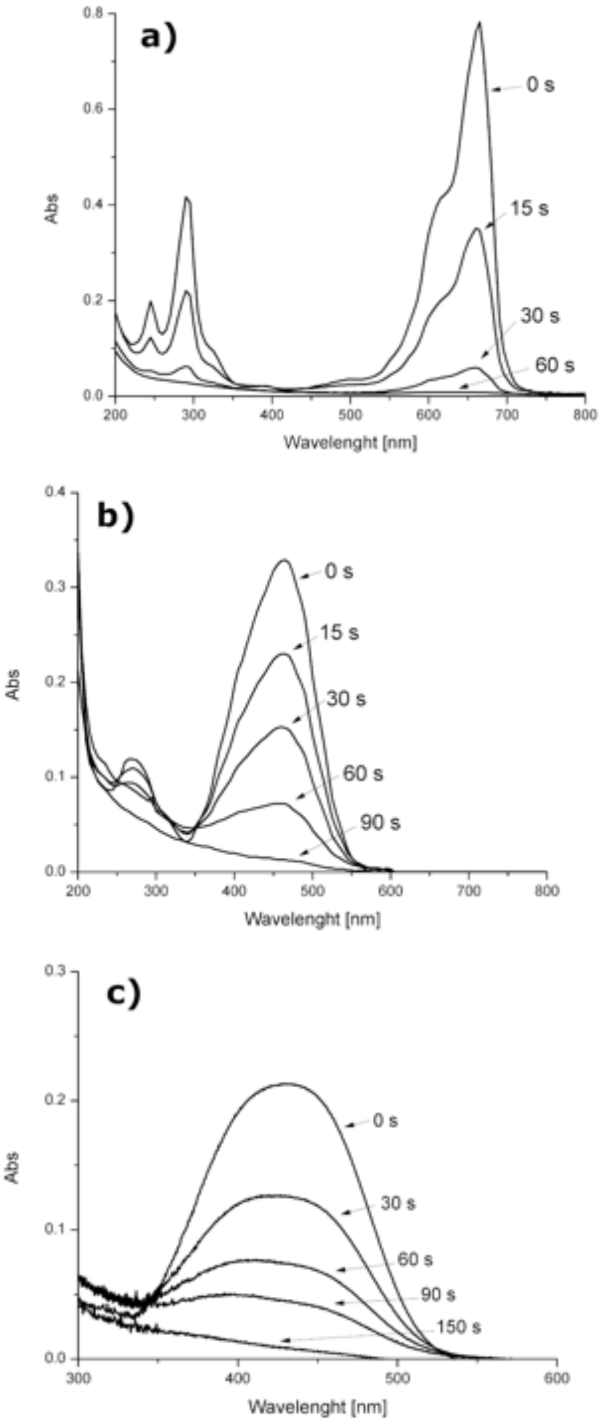

Fig. 3. UV-Vis Spectra for methylene blue (a), methyl orange (b), and methyl red (c).

is observed at times of 150 seconds, at which point the peaks are practically nonexistent.

Figure 3 shows the UV-Vis spectra for the organic dyes studied, where the absorbance of the characteristic peaks decreases as treatment time increases. It also shows that for methylene blue, and methyl orange the color practically disappears after 90 seconds of treatment. The decrease in the intensity of the peaks is evidence the plasma treatment is degrading the chromophore groups that give the dyes its characteristic color. After 5 minutes of treatment with plasma, no color is detected for any of the dye solutions (initial concentrations of 100 mg/L), which is a vast improvement over previously reported treatments, where it took at least 30 minutes to achieve 90% discoloration. Another advantage of the reduced treatment time is the possibility to build a continuous treatment system instead of the batch plasma systems previously reported in the literature.

3.2 Chemical Oxygen Demand (COD)

The degradation of the organic dyes was followed by analyzing the chemical oxygen demand of the solution. Figure 4 shows the evolution over time of the percentage of COD removed from the different dyes (compared to the initial COD). Methylene blue is the dye that degrades most rapidly. On the other hand, methyl orange shows a slower decrease of COD compared to the other two dyes. After 5 minutes of treatment, methyl red reached a lower COD, of approximately 52% of the initial value. Though, there is considerable reduction of COD for all the dyes studied, after 5 minutes of plasma treatment the values are not zero, which suggests there are still potentially oxidizable intermediate compounds within the treated solution after the discoloration reaction of the dyes (Chandana *et al.*, 2015; Sanito *et al.*, 2022).

3.3 Total Organic Carbon (TOC)

When comparing figures 2 and 4 it can be deduced that the discoloration chemical reaction is carried out faster than the degradation or mineralization of the dye. For the organic dyes studied (methylene blue, methyl orange, and methyl red), in 300 seconds the discoloration is practically complete; however, there are still organic matter to be degraded. Figure 5 shows the reduction of total organic carbon for the plasma treated samples at different times. The presence of organic carbon at 300 seconds of treatment is evidence that there are still intermediary compounds of the degradation process of the different dyes, and that the plasma treatment not only discolors the solutions but also provides a mineralization process that unfortunately is slower and does not end at 300 seconds of treatment as in the case of discoloration. The decrease of the total organic carbon with respect to the time of treatment, indicates the degree of mineralization of the dyes, which means the organic carbon on the dyes can be converted to CO_2, C and H_2O (Huang *et al.*, 2012; Huang *et al.*, 2010; Lachheb *et al.*, 2002).

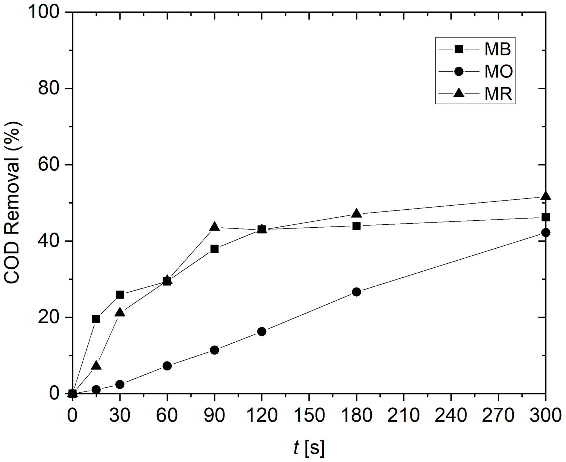

Fig. 4. COD reduction percentage for methylene blue, methyl orange and methyl red plasma treated solutions.

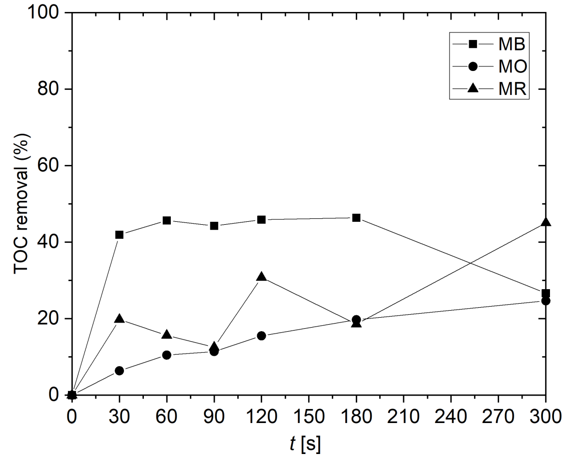

Fig. 5. Total organic carbon reduction for plasma treated solutions of organic dyes.

Table 1. Mass spectrometry results for CO_2 generation of methylene blue plasma treated solutions (initial concentration 200 mg/L).

Flow [cm^3/min]	12.5	25	100
Final F_D	0.2	0.52	0.97
Area under the curve [A*s]	5.80E-09	5.88E-09	1.35E-08

3.4 *Mass Spectrometry: Methylene Blue*

For methylene blue, mass spectrometry was used to qualitatively verify the formation of CO_2, according to the following stoichiometric reaction of the total oxidation of the dye (Lachheb *et al.*, 2002):

$$C_{16}H_{18}N_3S^+ + \frac{51}{2}O_2 \rightarrow 16CO_2 + 3NO_3^- + SO_4^{2-} + 6H^+ + 6H_2O \qquad (2)$$

A buffer solution containing 200 mg/L of methylene blue was used to maximize the signal obtained with the mass spectrometer, and experiments were performed at three different oxygen flows (12.5, 25 and 100 cm^3/min). The treated volume of solution was 20 cm^3, and the analysis of the generated gas was performed *in situ* with a residual gas analyzer, as described in the methods section.

Table 1 shows that the area under the curve in the mass spectra for CO_2 was bigger as the flow increased, which indicates that the amount of CO_2 generated is bigger for higher flows. These results agree with the fact that with the flow of 100 cm^3/min a discoloration factor of 0.97 was obtained, while a lower flow of just 12.5 cm^3/min only achieved a discoloration factor of 0.20.

The RGA Prisma Plus analyzer to verify the formation of reaction products *in situ*, such as CO_2, whose signal is shown in figure 6. This figure shows the formation of CO_2 immediately after initiating the plasma. The experiment begins with the power supply turned off (no plasma) for a period of 300 seconds, and immediately after the power supply is turned on, thus initiating the plasma, and generating reactive species, the degradation reaction takes place.

The mass spectrometry tests performed consisted of monitoring the signal once the reaction system is switched on and the plasma is ignited; when the reactive species are produced and carried into the solution. The signal obtained, considering the variables of the system, as well as the dead volumes and the ignition stage is evidence of CO_2 formation.

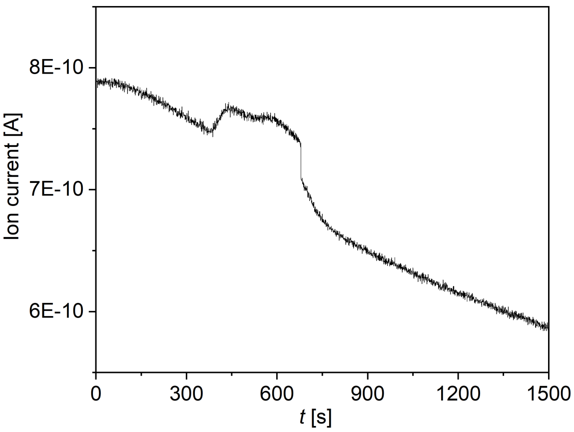

Fig. 6. RGA mass spectra for CO_2 formation during the plasma degradation reaction of methylene blue.

When the plasma reaction system is under operating conditions the CO_2 signal increases considerably, and a drop is observed the moment the power supply is switched off. Figure 6 shows a signal for CO_2 at 369 seconds, which drops once the reaction system is turned off. The 69 seconds of difference between the initiating of the treatment (at 300 seconds) and the showing of the signal is the lag due to displacement of the dead volume between the solution and the analyzer.

Conclusions

Three different dyes were treated in the plasma reaction system proposed in this study. Relatively fast discoloration reactions were achieved, obtaining discoloration factors of approximately 0.99 for the three dyes studied after 5 minutes of treatment.

These solutions were not only discolored but also degraded and mineralized, which was verified by the elimination of TOC and COD, as well as CO_2 formation from the treated methylene blue solution as evidenced by residual gas analysis.

Even though the discoloration rate is almost total after 5 minutes of treatment, the mineralization process does not achieve the same rate at this treatment time. The fact that there are TOC and COD in the treated solution of the dye treated after 5 minutes is evidence that there is still organic matter that need to be degraded.

The results presented in this study show a significant improvement over previously reported plasma treatments for the discoloration of dyes. Additionally, the evidence of degradation of the organic dyes presents the viability of the treatment, not only for discoloration of dyes, but removal of organic matter in effluents. Also, the proposed treatment system allows for the possibility of building a continuous treatment system for the removal of pollutant from effluents.

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Nomenclature

Abbreviations	
Abs	absorbance
COD	chemical oxygen demand
F_D	discoloration factor
MB	methylene blue
MO	methyl orange
MR	methyl red
TOC	total organic carbon
Symbols used	
C_o	[mg/L] initial concentration
t [s]	time

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