Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2021.26632

212 (2021) 254–260
February

Enhanced degradation of textile wastewater by plasma-irradiated titanium dioxide

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Received 1 June 2020; Accepted 3 October 2020

ABSTRACT

Two techniques, contact glow discharge plasma and photocatalysis, have been combined by using plasma energy to activate TiO_2 and enhance chemical oxygen demand (COD), colour and turbidity removal of a real textile wastewater. Optimization of the photocatalyst amount and the suspension pH has been carried out by means of central composite design statistical method. The phytotoxicity of the treated wastewater has been assessed through *Lactuca sativa* seeds bioassays. The obtained results confirmed that the discharge plasma can induce the photoactivation of TiO_2. The most significant factor for COD and turbidity reductions was pH, and TiO_2 concentration for colour reduction. It was possible to remove about 94% of colour and 59% of COD, and at least, 33% of turbidity with 0.4 g L^{-1} of TiO_2 at acid medium. After applying toxicity tests to treated samples, radical growth of *Lactuca sativa* seeds was higher than that found for the control (i.e., 114%).

Keywords: Contact glow discharge plasma; Textile wastewater; Titanium dioxide; Plasma

1. Introduction

The textile industry wastewater is extensively addressed in literature. It is already attested that this kind of wastewater is highly coloured, frequently toxic, recalcitrant and resistant to conventional treatments. The most common types of dyes are azo and anthraquinonic dyes, which are suspected carcinogens [1]. Due to the presence of colour and toxic compounds, advanced treatment processes are proven to be the more adequate alternative for treating these kinds of effluents.

Advanced oxidation processes (AOPs) have emerged as innovative tools for textile wastewater treatment and organic pollutant mineralization, based on the input of energy in the reaction medium in order to generate highly non-selective oxidising compounds. Among the AOPs, the application of electrical plasma technology arises as a highly efficient technique [1–5].

Plasmas can be produced by the formation of high electric field due to a potential difference between two electrodes. In water solutions, the electrical discharge process leads to the formation of $OH^{\bullet}$ and $H^{\bullet}$ via dissociation (Eq. (1)), ionization (Eqs. (2) and (3)) and vibrational/rotational excitation of water molecules (Eqs. (4)–(6)) [2,6]. The hydroxyl radical is a powerful oxidant, known as one of the most reactive oxygen species. It has redox potential of 2.72 V in acidic and 1.90 V in neutral solutions. Hydrogen radical ($H^{\bullet}$) is a conjugated acid, which acts as reducing agent in acidic medium, and has a reduction potential of –2.31 V [7,8]. Hydrogen peroxide and ozone may also

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be formed (Eqs. (7) and (8)). Additionally, $O^\bullet$ ($E°$ = 2.42 V [9]) radicals formed (as in Eqs. (5) and (6)) or by dissociation of O_2 can react with H_2O and boost the formation of $OH^\bullet$ (Eq. (9)) [5].

$$H_2O + e^- \rightarrow OH^\bullet + H^\bullet + e^- \quad (1)$$

$$H_2O + e^- \rightarrow 2e^- + H_2O^+ \quad (2)$$

$$H_2O^+ + H_2O \rightarrow OH^\bullet + H_3O^+ \quad (3)$$

$$H_2O + e^- \rightarrow H_2O^* + e^- \quad (4)$$

$$H_2O^* + H_2O \rightarrow H_2 + O^\bullet + H_2O \quad (5)$$

$$H_2O^* + H_2O \rightarrow 2H_2 + O^\bullet + H_2O \quad (6)$$

$$O^\bullet + O_2 \rightarrow O_3 \quad (7)$$

$$OH^\bullet + OH^\bullet \rightarrow H_2O_2 \quad (8)$$

$$O^\bullet + H_2O \rightarrow 2OH^\bullet \quad (9)$$

Water containing plasmas have UV light emission as a result of excited species relaxation to lower energetic states, due to collisions between electrons and neutral molecules [2]. Thus, if TiO_2 is present, the UV light enables the excitation of its surface and generation of electron–hole (e^-/h^+) pairs (Eq. (10)) [1,10]. Furthermore, UV light can directly degrade organic compounds by photolysis, and also dissociate the hydrogen peroxide and ozone thereby causing hydroxyl radicals generation in plasma system (Eqs. (11) and (12)) [2,11,12].

$$TiO_2 + h\nu \rightarrow TiO_2(e^-_{cb} + h^+_{vb}) \quad (10)$$

$$H_2O_2 + h\nu \rightarrow 2OH^\bullet \quad (11)$$

$$O_3 + H_2O + h\nu \rightarrow O_2 + 2OH^\bullet \quad (12)$$

Some contact glow discharge reactors have been studied for water and wastewater treatment. In contact glow discharge plasma reactors (CGDP), a light emitting plasma is sustained by direct current (DC) glow discharges between an electrode, the anode or the cathode and the liquid electrolyte surrounding it [13]. Most of the studies concerning to CGDP for wastewater treatment mainly focus on the use of $OH^\bullet$ radicals to oxidize different compounds. However, UV light, O-based and reduction species are also be available and take place on the contaminant degradation process. Additionally, the combination of plasma and photocatalysis is mostly applied to degradation of model molecules, such as medicines and dyes.

With this background, the subject of this paper was the investigation of electric discharge in a textile wastewater seeking the utilization of CGDP not only as $OH^\bullet$ source but also as UV source for activation of TiO_2 and promotion of simultaneous plasma and photocatalytic oxidation of a raw textile wastewater.

2. Methods

2.1. Wastewater samples

Textile wastewater samples were collected after equalization tank in a wastewater treatment plant of an industrial laundry. The activities of the industry are the processing of Denim articles and the dyeing of textile articles. The main dyes used are indigo carmine and reactive dyes. The wastewater was properly stored and characterized according to the following physical–chemical parameters: pH, turbidity, chemical oxygen demand (COD), and UV-visible absorption spectra.

2.2. GDP apparatus

A schematic diagram of the experimental setup used in this study is shown in Fig. 1. A jacket glass batch with 250 mL was used as GDP reactor. The DC high-voltage power unit (variable voltage of 0–700 V and current of 0–600 mA) was supplied by a Variac variable transformer and a self-designed voltage amplifier. The purpose of the amplifier is to amplify the output voltage of the Variac by means of four capacitors (470 mF/25 V), so that it is possible to reach the working voltage. The total area of 2.5 cm^2 of a stainless-steel plate cathode was submersed into the suspension, while a 0.5 mm diameter tungsten anode was maintained at the surface of the suspension, with the submersion of approximately 1 mm in length. During operation, 200 mL of the solution were magnetically stirred at a constant rate. Voltage was maintained at approximately 600 V and current at 500 mA. Water was used as cooling fluid in the cooling jacket in order to avoid boiling of the wastewater, since the electrical discharge can heat the medium.

The reaction vessel was open to atmosphere, and the plasma was generated under atmospheric air. In electrical liquid discharge, the current transfer is strongly affected by liquid conductivity. Taking into account that the textile wastewater usually has high salt concentrations, no electrolytes were added to conduct the current.

2.3. Effect of pH and catalyst

Central composite design was utilized to investigate the combined effect of two independent variables (pH and TiO_2 concentration) by 11 sets of experiments including three replications at the central point. Reaction time was 120 min. Table 1 shows the ranges and levels of independent variables, defined after exploratory experiments. Independent variable values were rounded to a number of decimal places suitable to be measured in the available equipment. Anatase phase TiO_2 (14 $m^2 g^{-1}$) was purchased from Kronos®.

Response surface analysis was used to match the data to an empirical quadratic polynomial as follows (Eq. (13)) [14]:

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_i \sum_{<j=2}^{k} \beta_{ij} X_i X_j + e_i \quad (13)$$

where Y is the response, X_i and X_j are the variables, β is the regression coefficient, k is the number of factors studied and optimized in the experiment, and e_i is the random error.

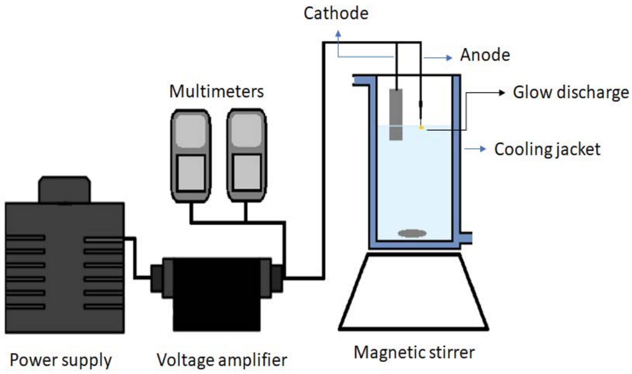

Fig. 1. Experimental set-up of the open-air contact glow discharge plasma reactor.

ANOVA (analysis of variance) has been applied to define interactions between the variables and the responses for statistical parameters assessment. Statistical significance was checked by F-test and the accuracy of the fitted polynomial was assessed by the value of R^2. The significant model terms were evaluated by the probability value (p-value) at 95% confidence interval.

2.4. Wastewater toxicity evaluation

Lactuca sativa seed is one of the most common plant species recommended by the US Environmental Protection Agency and the US Food and Drug Administration for phytotoxicity bioassays [15,16]. According to Priac et al. [17], these bioassays are simple, inexpensive and only require a relatively small amount of sample. In this sense, triplicate toxicity assays were performed on effluent samples. Thus, Petri dishes with qualitative filter paper of compatible diameter were prepared and 20 lettuce seeds (*Lactuca sativa*) with 99% germination index were arranged. The plates were watered with 14 mL of solution (positive and negative controls, raw and treated effluent without pH adjustment) and placed in an incubator with an average temperature of 22°C ± 2°C for 120 h. There were two distinct controls, one negative only with distilled water and three positives with 0.5, 1.0 and 2.0 mol L^{-1} NaCl solutions.

After 120 h of incubation, the percentage of germination and radicle growth was determined by measuring the radicle length of germinated seeds. The relative germination of the samples was calculated according to Eq. (14), and the percentage of relative radicle growth inhibition according to Eq. (15).

$$RG = \frac{GSS}{GSC} \times 100 \tag{14}$$

RG – relative germination %; GSS – number of germinated seeds in the sample; GSC – number of germinated seeds in the negative control.

$$GI = RGI \times RG \tag{15}$$

GI – germination index %; RGI – relative growth index (RGI = radicle length of the sample/radicle length of the negative control).

Relative growth index has been classified into three categories: inhibition of the radicle elongation if 0 > RGI > 0.9; no significant effects if 0.9 < RGI < 1.1 and stimulation of the radicle elongation if RGI > 1.1.

3. Results and discussion

3.1. Textile wastewater characteristics

The characteristics of the collected wastewater are presented in Table 2. By comparing the results with Brazilian environmental regulations, is can be noted that odour, turbidity and COD are against the limits for disposal in aquatic environment [18–20]. It is worth noting that these results refer to the characteristics of the effluent at the time of collection. Changes of dyes used in the dyeing process can induce remarkable fluctuation in wastewater characteristics, and the variation in the physico-chemical properties of real textile effluents can be observed in several industries throughout the world [21].

3.2. Response surface analysis

Experimental values of COD, colour and turbidity removals under different experimental conditions are presented in Table 3. Data were analysed by the ANOVA. Optimal values of the parameters were estimated by the response surface analysis of the independent and the dependent variables.

In respect to the experimental design presented in Table 1, a second-order polynomial equation in terms of significant factors (p < 0.05) was found in order to demonstrate the empirical relationships between the independent variables and the response. Fitted models for the codified coefficients with 95% of confidence are presented in Eqs. (16)–(18) for COD, colour and turbidity reductions, respectively.

Table 1
Independent variables and experimental range for degradation of textile wastewater by contact glow discharge plasma

Factor	Symbol	Range				
		–1.41[a]	–1	0	+1	+1.41[a]
pH	X_1	3.17	4.00	6.00	8.00	8.83
TiO_2 loading (mg L^{-1})	X_2	0.00	0.10	0.40	0.70	0.82

[a]Axial points were $X_i = \pm\alpha$, $\alpha = \sqrt[4]{2^k}$, where k = 2 is the number of independent variables.

Table 2
Characteristics of the raw textile wastewater

Parameter	Unity	Value
pH	–	6.98
Turbidity	NTU	169
Conductivity	mS cm^{-1} (25°C)	2.15
COD	mg L^{-1}	292
Total area under the absorbance curve	a.u.	223.856
Odour	–	Present

$$Y_{COD} = 26.82 - 7.53X_1 - 3.5X_2 + 5.13X_1^2 - 3.1X_1X_2 \quad (16)$$

$$Y_{Colour} = 92.84 - 2.77X_2^2 - 1.48X_1X_2 \quad (17)$$

$$Y_{Turb} = 33.37 + 8.69X_1 + 7.63X_2 - 20.09X_1^2 \quad (18)$$

In the polynomial expression, negative coefficients of the terms mean negative influences on the response. The adjusted models presented R^2 values of 63.22% for COD, 62.3% for colour and 89.4% for turbidity, respectively. The most significant factors were pH (X_1) for COD and turbidity reductions, and TiO_2 concentration (X_2) for colour reduction.

The three-dimension surface plots were drawn to illustrate the main and interactive effects of the independent variables on the dependent ones. Figs. 2a–c show the quadratic effect of both pH and TiO_2 on COD, colour and turbidity reductions, respectively.

It can be noted from Eqs. (16)–(18) that pH has a negative effect over parameters reduction. It means that efficiency might increase in acid medium. The same behaviour has been reported by Guo et al. [22]. Solution acidity induces OH$^\bullet$ formation obtained from positive holes (h$^+$) on TiO_2 surface [1] and the increase on the formation of ozone [22,23].

Furthermore, the TiO_2 zero charge point (pH$_{ZPC}$) is 6.8. If the suspension pH is higher than pH$_{ZPC}$, TiO_2 surface is

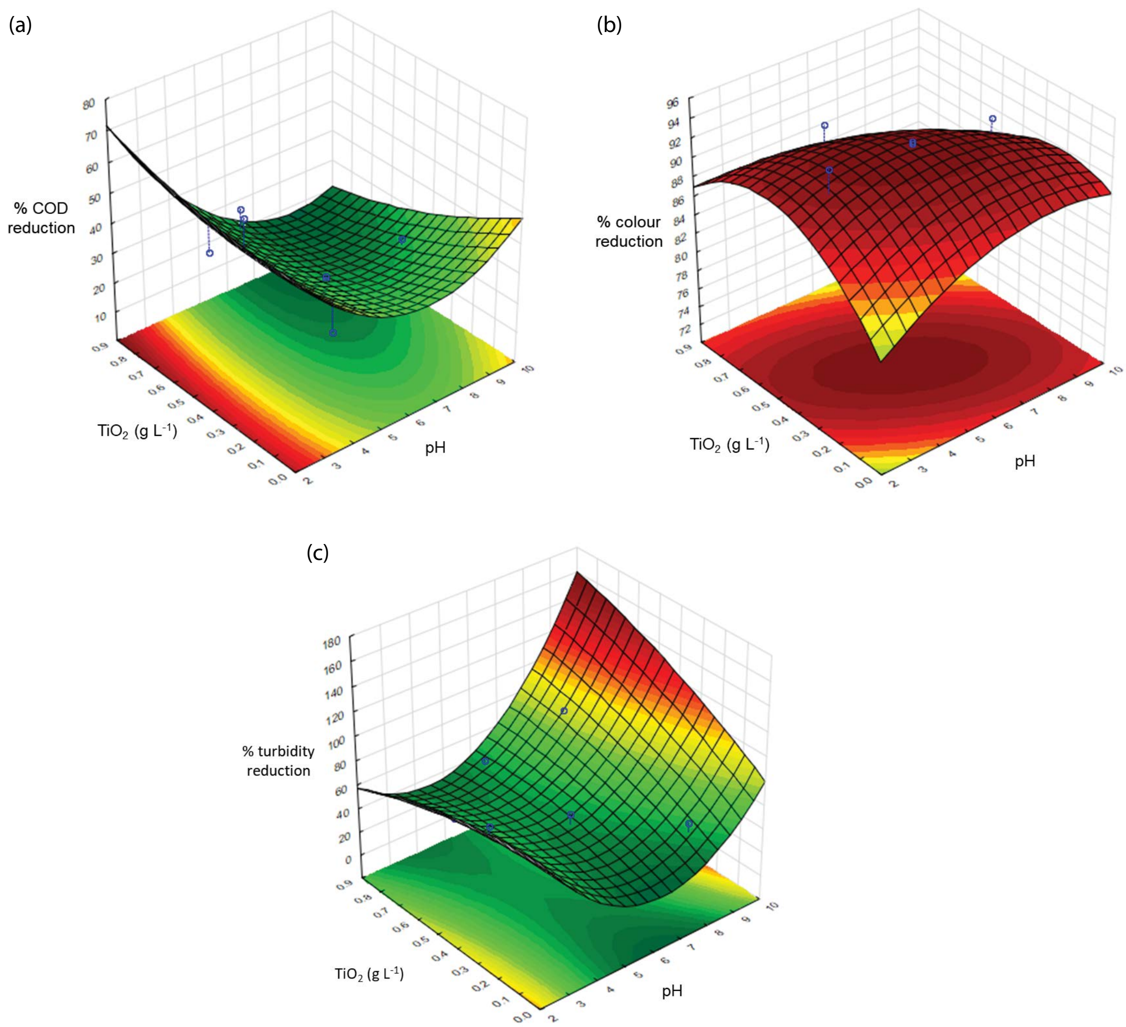

Fig. 2. Response surface plots showing the effect of pH and TiO_2 concentration on reduction of (a) COD, (b) colour and (c) turbidity of the textile wastewater by contact glow discharge plasma reactor (≈600 V).

negatively charged, favouring the adsorption of cationic species, and if the suspension pH is lower than pH_{ZPC}, the TiO_2 surface is positively charged, favouring the adsorption of anionic species. Taking into account that the main types of dyes present on the wastewater are indigo carmine and reactive dyes, both anionic, at acid medium the adsorption of both the dyes is favoured [24].

Guo et al. [22] investigated the influence of pH and graphene oxide/TiO_2 nanocomposites on the degradation of flumequine in water by pulsed discharge plasma, and reported that with 0.2 g L^{-1} of catalyst, synergistic factor has raised four-fold. Regarding to pH, the highest kinetic constant was observed for the pH value of 3.00. The authors highlighted that oxidation potentials of $OH^\bullet$ and O_3 can became lower in alkaline solution.

The decolouration of Acid Orange 7 (50 mg L^{-1}) has been studied by means of TiO_2 catalysed pulsed discharge plasma, and the best result was about 98% of decolouration after 45 min at a TiO_2 concentration of 0.6 g L^{-1} without pH adjustment [25]. Synergistic effects of pulsed discharge plasma and multi-walled carbon nanotubes of TiO_2/γ-Al_2O_3 composite were applied to the degradation of the same dye (20 mg L^{-1}), reaching 94% of decolouration after 60 min of discharge with 5 g L^{-1} of catalyst with no pH adjustment. Authors assigned the photocatalytic activity of the catalyst to the photo-induced electron absorption and the electron trap effects [26]. Comparing these results to this work, 94% of discolouration has been reached within 120 min of discharge, but with only 0.4 g L^{-1} of TiO_2. Ghezzar et al. [1] investigated the enhancement of degradation and biodegradability of a textile industry wastewater by arc discharge plasma in the presence of TiO_2. After 180 min, degradation increased from 87.7% to 96.0% with the addition of 3 g L^{-1} of catalyst. These results highlight the effectiveness of the present work, since at optimized conditions (0.4 g L^{-1} of TiO_2, pH 3.2), COD reduction was 48% and colour reduction was 94% in a shorter period of time and with a much smaller amount of catalyst. This efficiency is remarkable since it deals with a real wastewater with no previous treatment.

3.3. Seed cultivar

Treating wastewater by AOPs can possibly generate toxic by-products. In this sense, toxicity evaluation of the wastewater before and after treatment is mandatory. Bioassays were conducted using a sample of raw wastewater and treat samples from assays 9, 10 and 11 (Table 3). Table 4 presents the relative germination and germination index for both, treated and untreated samples, calculated according to Eqs. (14) and (15).

The roots of seeds that were watered with the raw wastewater had germination index of 69.8% when compared with the negative control samples (inhibition effect). On the other hand, roots of the seeds that have been watered with the treated wastewater have grown larger than the roots of seeds in negative control (GI = 114% – stimulation effect). Exposure to the plasma probably has resulted in the release of sulphate, phosphate and nitrogen ions from the organic part of dyes present in wastewater, thus

Table 3
Central composite design with experimental and predictive values

Run	Experimental conditions		Reduction of COD (%)		Reduction of colour (%)		Reduction of turbidity (%)	
	pH	TiO_2 (mg L^{-1})	Experimental	Predictive	Experimental	Predictive	Experimental	Predictive
1	4.00	0.10	31.9	41.4	86.7	88.3	44.4	47.0
2	4.00	0.70	30.8	40.6	88.0	90.7	33.1	42.2
3	8.00	0.10	29.4	32.7	89.5	90.5	54.4	43.9
4	8.00	0.70	15.7	19.3	85.0	87.0	84.0	80.0
5	3.17	0.40	58.7	47.8	94.0	91.8	69.8	61.2
6	8.83	0.40	28.6	26.5	92.2	90.7	75.7	85.7
7	6.00	0.00	41.2	34.3	89.1	88.0	17.8	23.5
8	6.00	0.82	31.9	24.9	89.4	86.7	47.9	44.2
9	6.00	0.40	25.5	27.1	93.0	92.9	35.5	40.4
10	6.00	0.40	26.8	27.1	92.1	92.9	36.7	40.4
11	6.00	0.40	27.9	27.1	93.3	92.9	49.7	40.4

COD: Chemical oxygen demand.

Table 4
Relative germination and growth inhibition of *Lactuca sativa* seeds

Sample	RG (%)	Radicle length (cm)	RGI (%)	GI (%)	Remarks
Negative control	100	4.63 ± 0.13	1	100	–
Untreated sample	95	3.40 ± 0.24	0.73	69.8	Inhibition
Treated sample	100	5.26 ± 0.36	1.14	114	Stimulation

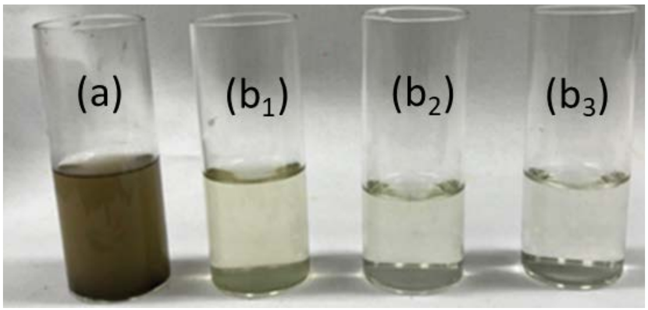

Fig. 3. Textile wastewater samples before (a) and after plasma/TiO_2 treatment (b_1, b_2, b_3). Conditions: pH = 6.00; TiO_2 = 0.4 g L^{-1}, reaction time: 180 min.

possibly acting as nutrients in the medium [1]. Torres et al. [27] evaluated the toxicity of a real textile wastewater treated by coagulation/flocculation followed by electrochemical oxidation. The authors reported that the treated wastewater did not show significant inhibition of *Lactuca sativa* germination rate, but not showing stimulation effect as observed in the present work.

In this sense, we can assume that the TiO_2-boosted plasma treatment was successful in treating and reducing phytotoxicity of the textile wastewater. Beside the risk of toxicity underestimation when only one bioassay is used, the final characteristics of the treated wastewater confirm that the process has been effective. Additionally, toxicity tests served as an important indicator on the efficiency of the process, since the formation of the most toxic compounds may not be registered neither by COD, decolourization nor turbidity analyses.

Fig. 3 shows the photograph of untreated and treated samples from the central point essays (runs 9, 10 and 11).

Summarizing, the experiments have shown that the CGDP method can be successfully used in the treatment of real textile wastewater and the process efficiency can be enhanced by the addition of TiO_2 photocatalyst. Additionally, the degradation products are less toxic than the raw material present in the wastewater.

4. Conclusions

Combination of contact glow discharge plasma and TiO_2 photocatalyst significantly enhances the degradation of real textile wastewater. The photocatalyst concentration and pH were statistically significant on COD, colour and turbidity reduction efficiency. In the investigation, the best amount of TiO_2 was 0.4 g L^{-1} and the best pH was 3.17. Under optimal conditions, 94% of colour and 59% of COD have been reduced after 120 min. *Lactuca sativa* seeds bioassays showed that after treatment, the wastewater promoted the stimulation of radicle growth. The relatively simple TiO_2-mediated degradation process has shown to be more efficient than only plasma treatment.

Acknowledgements

The authors would like to thank Federal University of Technology – Parana for the financial support.

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