

Direct red 83 textile dye degradation using photoperoxidation and photo-fenton: kinetic studies, toxicity and neural networks modeling

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ABSTRACT

Textile dyes have been observed in aquatic matrices that receive effluents from different textile industries. These compounds have the peculiarity of being resistant to the physical, chemical and biological treatments commonly used in wastewater treatment plants. Thus, alternative treatments such as advanced oxidative processes (AOP) have been considered in order to promote the degradation of this type of pollutant, being the photoperoxidation and photo-Fenton processes the most used. Therefore, the present work evaluated the efficiency of these AOP in the degradation of direct red 83 dye. It was found a greater efficiency of the photoperoxidation process, especially in the degradation of functional groups observed at 289 nm. This AOP presented a pseudo first order reaction kinetics, with rapid decay in the first minutes. The MLP (5-21-2) neural network model was able to satisfactorily predict the degradation of the dye under study. Finally, it was found that the proposed process showed no adverse effects when studying the toxicity in bacteria.

Keywords: ANN; Advanced oxidative processes; Treatment

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1 INTRODUCTION

The textile industry has been standing out in the Brazilian scenario, as it has directly participated in the growth of the country's economy. However, due to the diversity of this industry, especially in the dyeing sector, environmental problems have been observed even after the treatment of the wastewater, which is performed through conventional processes (MARTINS et al., 2011). The dyes presented in these effluents have chromophoric groups (responsible for color fixation) and aromatic groups (usually toxic) (SILVA et al., 2017).

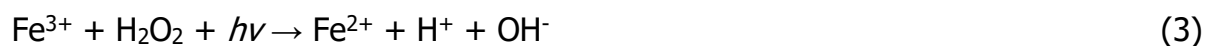
The concern about the presence of coloring substances in the aquatic environment is related to the toxicity arising from the aromatic groups present in their molecular structures. In dyes that have one or more azo groups (-N = N-), it is known that they can cause carcinogenic and mutagenic effects in animals, besides having allergenic effects (ZANONI; YAMANAKA, 2016; RAWAT; MISHRA; SHARMA, 2016). Additionally, the persistence of such compounds may favor the process of eutrophication in the environment and promote the reduction of solar radiation penetration, compromising the water body (SARANRAJ, 2013).

Thus, advanced oxidative processes (AOPs) have been employed as a viable alternative for the degradation of unwanted compounds as they efficiently convert recalcitrant groups into non-toxic products (ASAITHAMBI et al., 2015). The use of hydrogen peroxide as well as ultraviolet (UV) or solar radiation assist in the generation of hydroxyl radical. These, in turn, react with a vast amount of substances by removing electrons and/or hydrogen atoms from them (NAVARRO et al., 2017).

Hydroxyl radicals can react with the substances to be treated through three different mechanisms. Among these mechanisms are hydrogen abstraction reactions, which generates organic radicals (Equation 1); and union to molecular oxygen, producing peroxide radical (Equation 2). Such mechanisms give rise to chain reactions, responsible for the mineralization of the organic substrate. Thus, the result of the treatment is a clean process that should form carbon dioxide, water and inorganic salts (BRITO; SILVA, 2012).



In cases where a light source is combined with hydrogen peroxide, the treatment is called photoperoxidation. However, the treatment response is often better when using the photo-Fenton process, which combines radiation, H₂O₂ and iron ions. In this process there is a photo decomposition of H₂O₂, through the regeneration of Fe²⁺ ions, from Fe³⁺ ions previously formed in the Fenton reaction (COSTA et al., 2014). Equation 3 represents the reaction of the photo-Fenton process.



Leite et al. (2016) state that the main indication of the efficiency of photo-Fenton dye treatment is related to the disappearance of the absorption band of the chromophoric groups. Salvador, Marcolino Júnior (2012) managed to promote the degradation of 99% of chromophoric clusters presented in QR-19 blue and reactive orange 16 dyes through this type of AOP.

However, it should be noted that regardless of the process employed, it is important to analyze the speed at which pollutant degradation reactions occur, since a very long exposure time is not feasible on an industrial scale for large effluent volumes. Another relevant factor concerns the evaluation of toxicity presented by the contaminant before and after treatment. Different species have been analyzed in order to verify the toxic potential of polluting substances, such as the bacteria. Nascimento et al. (2018) performed toxicity tests with *Escherichia coli* and *Salmonella enteritidis* strains to evaluate the behavior of reactive grey BF-2R dye before and after photo-assisted degradation. The authors found that the photo-Fenton process employing UV-C radiation did not interfere with the development of *E. coli* strains. However, this same treatment inhibited the growth of *S. enteritidis* strains, thus showing the need to perform this evaluation when working with AOP.

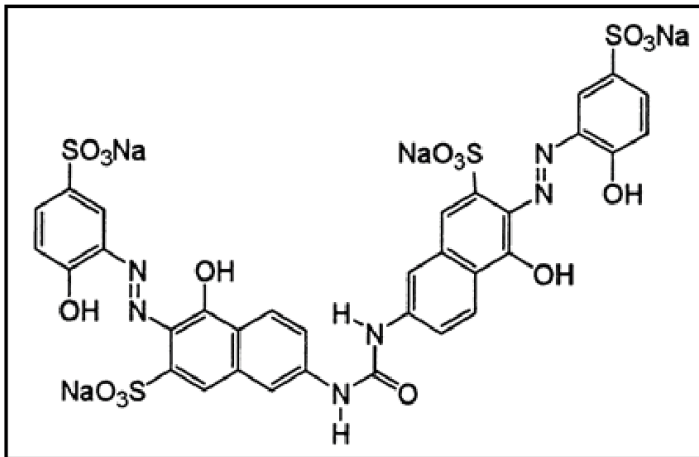
The present work was developed, then, with the purpose of evaluating the application of photoperoxidation and photo-Fenton AOP in the treatment of aqueous solution containing direct red 83 textile dye. In addition, the kinetic behavior of the treatment was evaluated and neural network modeling was used to predict process efficiency by varying different parameters.

2 MATERIALS AND METHODS

2.1 Study of the degradation of aqueous solution of direct red 83 dye

Direct red 83 azo dye (DR83) (Figure 1) has a molecular mass of 992.77 g.mol⁻¹, C.A.S 15418-16-3 and formula C₃₃H₂₀N₆Na₄O₁₇S₄. Samples of the dye solution before and after treatment were analyzed by ultraviolet-visible spectrophotometry (ThermoScientific, Genesys 10S model) at 289 and 544 nm, characteristic of the substance.

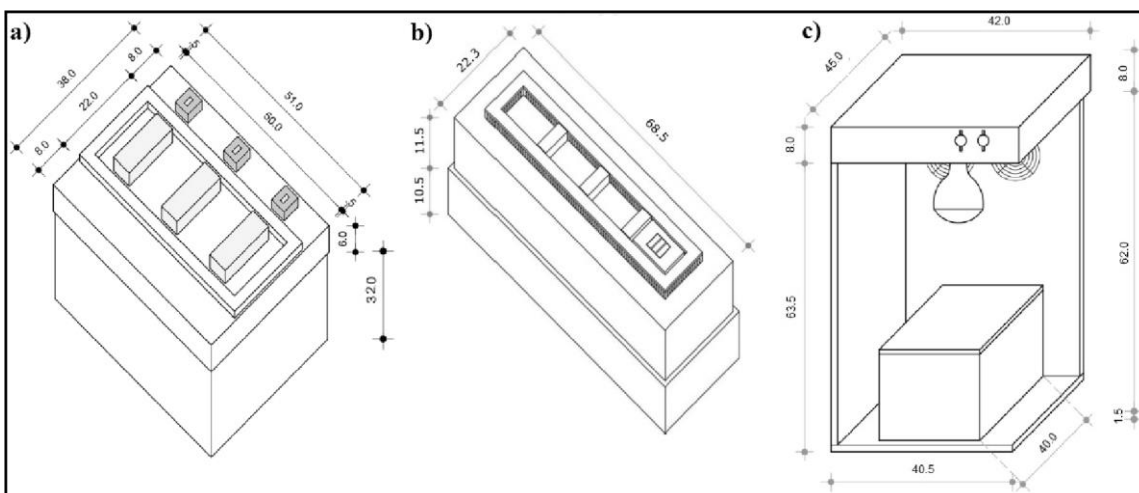
Figure 1 – Structural formula of direct red 83 dye



Source: Pellicer et al. (2018)

For the degradation tests, the advanced oxidative processes (AOPs) photo-Fenton and photoperoxidation were employed. For this, three bench reactors were used. The first with UV-C radiation (consisting of 3 30W (Phillips) lamps, arranged in parallel), the second with UV-A radiation (containing 3 15W (Philips) lamps, arranged in parallel) and the third with artificial *sunlight* (using a 300 W power sunlight (Osram)), as shown in Figure 1.

Figure 2 – Schematic drawing of the reactors: a) UV-C, b) UV-A and c) sunlight



Source: Zaidan et al. (2017), Charamba et al. (2018), Santana et al. (2017)

At first, for 60 min, 50 mL of dye solution with different concentrations (5, 15 and 30 mg.L⁻¹) were submitted to both types of AOP and previously selected radiation. For the photo-Fenton process the initial pH was adjusted to 3. The concentrations of iron ([Fe] (FeSO₄.7H₂O, Vetec) and hydrogen peroxide ([H₂O₂]) (Modern Chemistry) employed were 2.5 and 100 mg.L⁻¹, respectively.

Considering the AOP and the radiation that presented the highest dye degradation, the degradation conditions optimization stage was started. For this study, the H₂O₂ concentration was varied from 20, 30, 40, 50, 60, 80 to 100 mg.L⁻¹, while [Fe] was fixed at 2.5 mg.L⁻¹. The solutions were exposed to radiation for 60 min.

2.2 Evaluation of degradation kinetics of direct red 83 dye

After the determination of the best AOP and the optimization of the process conditions for dye degradation, a reaction kinetic study was performed. To this end, 1 L of each dye solution at the three concentrations under study were subjected to radiation for 180 min. Aliquots of 5 mL were taken within 10 to 180 min. The results were submitted to adjustment to the pseudo-first order kinetic model proposed by Chan and Chu (2003) presented in Equation 4.

$$C = C_0 \cdot \left(1 - \frac{t}{\rho + \sigma t} \right) \quad (4)$$

In which, C is the dye concentration (mg.L⁻¹) after the treatment in a reactional time t (min), and C_0 is the initial concentration of the dye (mg.L⁻¹). The parameters ρ and σ represent the reaction kinetics (min) and the oxidative capacity of the system (dimensionless), respectively.

2.3 Mathematical modeling by artificial neural networks

Mathematical modeling of experimental data using artificial neural networks (ANN) was performed aiming at predicting the dye degradation, by considering the variables involved in the degradation process. For this purpose, Statistica software (version 10.0) was used. The neural networks employed were Multilayer Perceptron (MLP) and Radial Base Function (RBF). Identity, logistic, hyperbolic, exponential, sine and hyperbolic tangential activation functions were tested. The selected inputs were: initial dye concentration ([Dye]₀) (mg.L⁻¹), time (min), [H₂O₂] (mg.L⁻¹), [Fe] (mg.L⁻¹) and pH. Thus, an analysis of the relevance of each variable in the dye degradation percentage was

performed. The selected output consisted of dye degradation in the λ studied. The sampling method used was randomized with data divided into 70% for training, 15% for testing and 15% for validation. The ANNs were trained using 200 iterations and 10 repetitions, totaling 2000 iterations. The best results were based on the highest values of the linear regression coefficients (R^2) of training, testing and validation; as well as the smallest errors obtained.

2.4 Bacteriological toxicity assessment

The microbiological toxicity test was performed with the dye solutions before and after treatment in sterile ultrapure water. Each sample (450 μL) was incubated (24h at 36 ± 1 ° C) together with the UFPEDA 224 *Escherichia coli* and *Salmonella enteritidis* UFPEDA 414 cell suspension (50 μL ; 10^5 CFU.mL⁻¹ in 0.15M NaCl) and half Mueller Hinton broth (100 μL). The negative control corresponded to bacterial cells treated with sterile ultrapure water. Before and after the incubation period, the contents of each treatment were transferred to 96-well microtiter plates for reading at 600 nm (OD600) to determine the growth percentage. This was compared to the negative control. Additionally, aliquots of the treatments were seeded in Petri dishes containing Mueller Hinton Agar (MHA) to assess cell viability after 48h incubation at 37 ± 1 ° C. Each assay was performed in triplicate and three independent experiments were performed.

3 RESULTS AND DISCUSSION

3.1 Study of the degradation of aqueous solution of direct red 83

Degradation tests were performed at the pre-determined concentrations (5, 15 and 30 mg.L⁻¹) in order to evaluate the photo-Fenton AOP and photoperoxidation, as well as the different radiation types. The results obtained regarding the degradation percentages are presented in Table 1.

Table 1 shows that photoperoxidation and photo-Fenton processes employing UV-A radiation were not efficient for the degradation of DR83 at the three concentrations evaluated. UV-C radiation, when used combined with photoperoxidation, proved to be effective with degradation greater than 79% at both wavelengths. It was also verified that the photo-Fenton process was not efficient with regard to the degradation of aromatic groups ($\lambda = 289$ nm) when the initial dye concentration was 5 mg.L⁻¹ for all studied

radiations. This result indicated that for dye concentrations below 5 mg.L⁻¹, a more detailed study of the variables involved in the process should be performed.

Table 1 – Degradation percentages obtained for the different concentrations in each studied λ

AOP	Radiation	% of degradation					
		5 mg.L ⁻¹		15 mg.L ⁻¹		30 mg.L ⁻¹	
		289 nm	544 nm	289 nm	544 nm	289 nm	544 nm
Photoperoxidation	UV-A	NDO	NDO	1.7	3.11	NDO	3.9
	UV-C	79.5	99.1	93.9	97.0	88.1	94.9
	<i>Sunlight</i>	84.4	91.1	32.9	58.0	10.6	26.1
Photo-Fenton	UV-A	NDO	87.5	59.6	96.1	79.5	99.6
	UV-C	NDO	85.4	70.9	99.4	85.5	99.6
	<i>Sunlight</i>	3.9	98.6	83.9	100.0	91.1	99.7

NDO = No degradation observed

Another important result was obtained by analyzing the sunlight/photoperoxidation system alone, considering the high degradation rates only for the concentration of 5 mg.L⁻¹. The tests were repeated and the low efficiency of the system was found at concentrations equal to and/or above 15 mg.L⁻¹.

Table 2 – Degradation of DR83 dye employing UV-C radiation and photoperoxidation by varying H₂O₂ concentration

H ₂ O ₂ concentration (mg.L ⁻¹)	% of degradation					
	5 mg.L ⁻¹		15 mg.L ⁻¹		30 mg.L ⁻¹	
	289 nm	544 nm	289 nm	544 nm	289 nm	544 nm
100	97.5	99.7	94.1	99.3	75.9	90.1
80	96.7	99.6	94.2	99.1	74.9	91.4
60	98.6	99.4	89.4	98.1	74.6	90.3
50	97.3	99.6	93.1	98.6	67.9	87.6
40	96.4	99.6	93.5	98.6	62.4	82.8
30	96.9	98.8	90.4	98.0	57.9	78.3
20	96.5	99.3	75.6	91.9	56.3	76.7

Given the above, the UV-C/photoperoxidation radiation system was chosen to promote the degradation of the dye, and therefore, the influence of the process variables were evaluated. In the first phase of the optimization process, solutions containing the

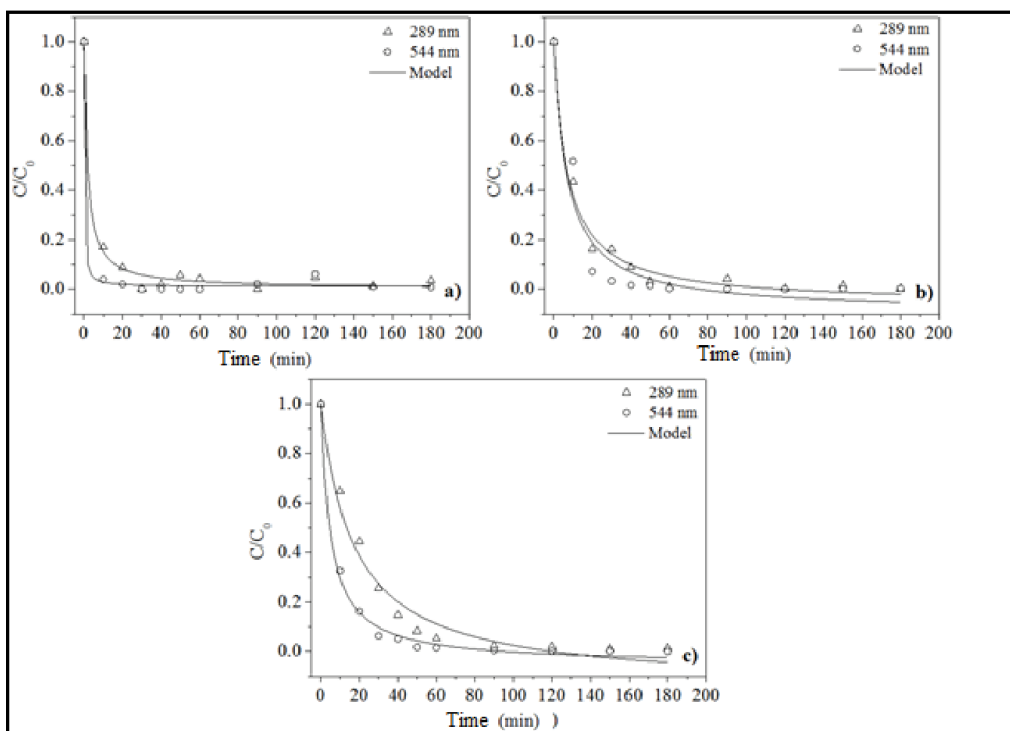
three DR83 concentrations were exposed to the selected system, employing different concentrations of H_2O_2 (20, 30, 40, 50, 60, 80 and 100 mg.L^{-1}) for 60 min. The degradation percentages obtained are shown in Table 2.

From the data shown in Table 2, it was concluded that increasing the concentration of H_2O_2 to greater than 80 mg.L^{-1} is not worthy once the degradation percentage obtained is approximately the same or even smaller compared when using 80 mg.L^{-1} . Thus, this value of H_2O_2 concentration was used in subsequent studies.

3.2 Evaluation of degradation kinetics of direct red 83 dye

The kinetic study of the photoperoxidation process was performed using 80 mg.L^{-1} [H_2O_2] and UV-C radiation. Assays were performed under atmospheric pressure and temperature of 50 ± 1 ° C. For this, 1 L of the dye solution (in the three studied concentrations) was submitted to the treatment, being removed aliquots of 5 mL from it. From the results obtained, it was verified the adequacy of the data to the pseudo-first order model proposed by Chan and Chu (2003), as can be observed in Figure 3.

Figure 3. Kinetics of direct red 83 dye degradation with adjustment to the model proposed by Chan and Chu at initial concentrations of: a) 5 mg.L^{-1} , b) 15 mg.L^{-1} ; c) 30 mg.L^{-1}



It was possible to verify, through the analysis of Figure 3, the stabilization of the system after 30, 50 and 60 min of exposure. The degradations obtained were greater than

99, 98 and 97% for the concentrations of 5, 15 and 30 mg.L⁻¹, respectively, for both dyes. The values of the linear regression coefficients and the other kinetic parameters of the oxidation reaction can be observed in Table 3.

Table 3 – Kinetic Parameters

Parameters	5 mg.L ⁻¹		15 mg.L ⁻¹		30 mg.L ⁻¹	
	289 nm	544 nm	289 nm	544 nm	289 nm	544 nm
1/ρ ^a	0.57	5.24	0.15	0.16	0.07	0.21
1/σ ^b	1.00	1.00	1.06	1.07	1.04	1.05
R ²	0.99	0.99	0.98	0.94	0.97	0.99

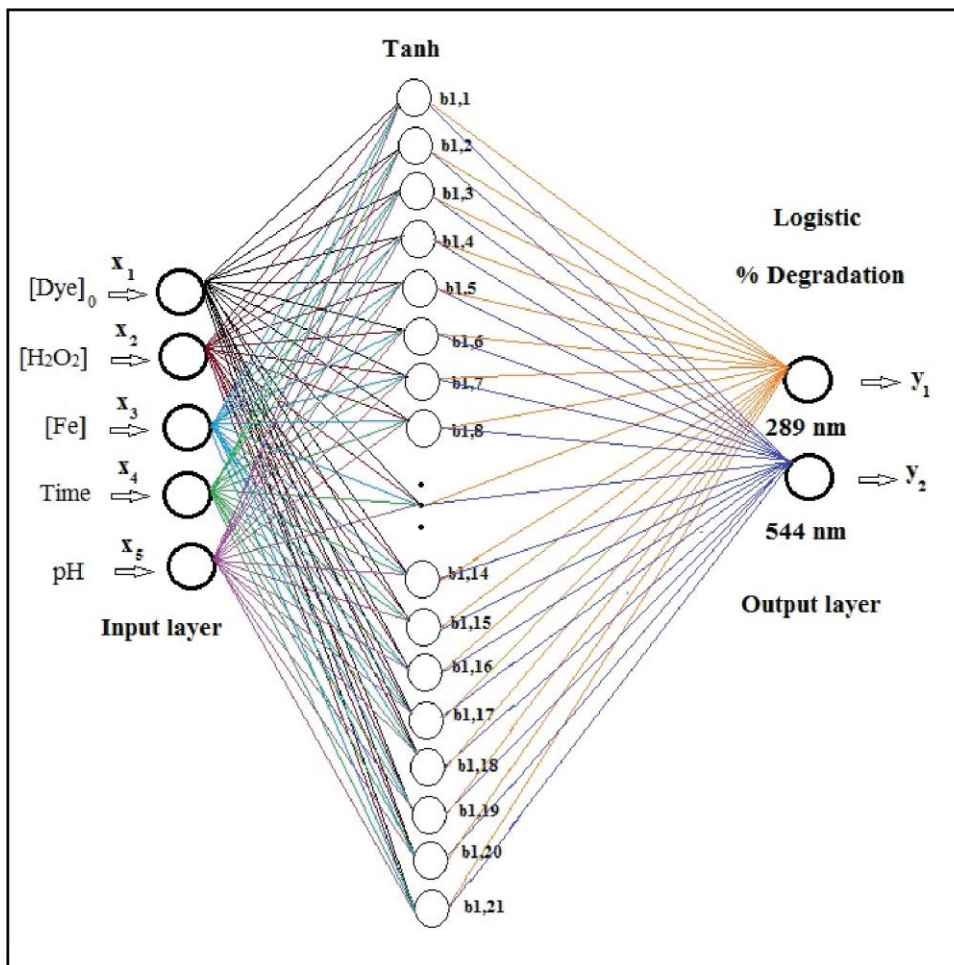
1/ρ^a: initial removal rate; 1/σ^b: maximum oxidation capacity.

The R² values presented in Table 3 showed a good fit of the experimental data to the Chan and Chu model. The initial removal rates (1/ρ) were higher at 544 nm λ for the three concentrations studied, obtaining faster compound reduction ratios. The maximum oxidation capacities of the system (1/σ) were similar for the three concentrations in both. Thus, it can be stated that the kinetic behavior of dye degradation via photo-peroxidation is similar for the studied concentrations (5, 15 and 30 mg.L⁻¹).

3.3 Mathematical modeling by artificial neural networks

The effect of the variables [Dye]₀, time, [H₂O₂], [Fe] and pH were evaluated in the photoperoxidation process using UV-C radiation. Experimental data obtained under different operating conditions were modeled using ANN. Among the ANNs employed, it was found that the RBF function was not as accurate to describe the data, and therefore the best result was obtained when applying the MLP network. This, in turn, resulted in 21 hidden layer neurons (5-21-2), whose architecture is shown in Figure 4. It was found that the training algorithm employed was based on the Broyden, Fletcher, Goldfarb and Shanno methods (BFGS). The activation functions of the input and output layers were Tanh and Logistic, respectively.

Figure 4 – ANN MLP` s architecture 5-21-2



From the results obtained with ANN MLP 5-21-2 it was possible to determine the relative importance of the input variables in the dye degradation efficiency. For both λ , the variables influenced the dye degradation in a similar way, with pH being the most influential factor (on average 37.76%). Then, important contributions were given by the variables $[\text{Dye}]_0$ ($\approx 24.12\%$), Time ($\approx 19.81\%$) and $[\text{Fe}]$ ($\approx 13.25\%$). The variable $[\text{H}_2\text{O}_2]$ presented the lowest contribution among the studied factors ($\approx 5.06\%$).

The ANN model developed to predict dye degradation was tested for accuracy. In this case, the values predicted by the network were compared with those obtained in the corresponding experiments, as can be observed in Figure 5.

Figure 5 – Parity graphs of the models used in training, testing and validation for the studied λ

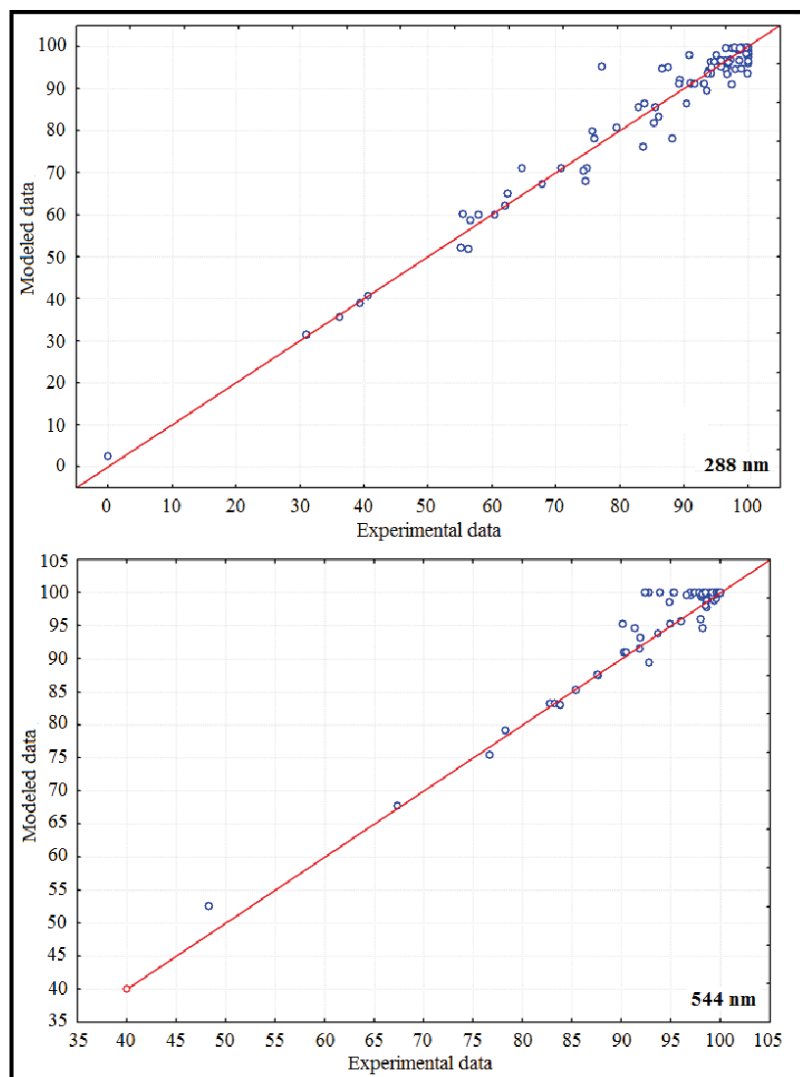


Figure 5 represents the comparative analysis between the calculated and experimental dye degradation values for the data set distributed among the training, testing and validation subgroups. From the analysis of this figure, it was found that the data showed no dispersion trends, confirming that the MLP 5-21-2 was adequate to predict the performance of the DR83 dye degradation process. In addition, the reliability of the ANN was verified from the linear regression coefficients (R^2) obtained for the output variable, which were higher than 0.98 and 0.97 for the λ of 289 and 544 nm, respectively. Similar results were obtained by Nascimento et al. (2018) that by employing ANN, it was verified that MLP type with BFGS training algorithm was able to predict the degradation of reactive gray textile azo dye using AOP with UV-C radiation.

3.4 Bacteriological toxicity assessment

In this study, samples of the solution containing the CR83 dye were evaluated at the three initial concentrations (5, 15 and 30 mg.L⁻¹) before and after treatment. The results obtained for the two bacteria studied are shown in Table 4.

Table 4 – Growth percentage and cell viability of *Escherichia coli* and *Salmonella enteritidis* bacteria

<i>Escherichia coli</i>			
Samples	$t_{24h} - t_{0h}$	Growth (%)	Viability
	Average $\pm \sigma$ (DO ₆₀₀)		
Water (control)	0.310 \pm 0.017	100.0	+++
Before the treatment (5 mg.L ⁻¹)	0.248 \pm 0.019	77.98	++
Before the treatment (15 mg.L ⁻¹)	0.297 \pm 0.023	89.65	++
Before the treatment (30 mg.L ⁻¹)	0.279 \pm 0.018	89.80	++
After the treatment (5 mg.L ⁻¹)	0.250 \pm 0.026	80.63	++
After the treatment (15 mg.L ⁻¹)	0.302 \pm 0.016	97.37	+++
After the treatment (30 mg.L ⁻¹)	0.298 \pm 0.001	95.92	+++
<i>Salmonella enteritidis</i>			
Water (control)	0.256 \pm 0.022	100.0	+++
Before the treatment (5 mg.L ⁻¹)	0.187 \pm 0.029	74.45	++
Before the treatment (15 mg.L ⁻¹)	0.241 \pm 0.027	90.12	++
Before the treatment (30 mg.L ⁻¹)	0.203 \pm 0.013	79.28	++
After the treatment (5 mg.L ⁻¹)	0.197 \pm 0.023	76.94	++
After the treatment (15 mg.L ⁻¹)	0.253 \pm 0.028	98.76	+++
After the treatment (30 mg.L ⁻¹)	0.265 \pm 0.007	103.44	+++

Through the analysis of Table 4 and according to the percentage of bacterial growth, it was possible to observe that the dye DR83 presented low toxicity. It was also found that AOP treatment did not give toxicological effects to the solution, which can also be observed by the increase of cell viability. By analyzing cell growth in isolation, it was found that there was no significant difference for the obtained optical density (OD) values of the samples before and after treatment, as well as in relation to the negative control.

4 CONCLUSION

The use of photoperoxidation and photo-Fenton as treatments to promote the degradation of the direct red 83 dye proved to be efficient in the deterioration of the chromophore group, responsible for conferring the characteristic coloration of the studied compound. However, it was noticed that by analyzing the decrease in absorbance in the length of 289 nm, only the photoperoxidation process was efficient for the three concentrations studied. This optimized treatment presented a nonlinear pseudo-first order kinetics, with adjustment greater than 0.94 (R^2) for the two λ and the three concentrations evaluated. It was also found that the ANN MLP model (5-21-2) can satisfactorily predict the percentage of direct red 83 dye degradation based on the input variables: $[Dye]_0$, $[H_2O_2]$, $[Fe]$, time and pH. Finally, it was found that the possible intermediates formed after degradation did not have toxicological effects against strains of bacteria *Escherichia coli* and *Salmonella enteritidis*, thus confirming the efficiency of the treatment with regard to the toxicity of the formed solution.

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