Study and construction of a sustainable solar photocatalytic reactor: indigo carmine blue food dye degradation

Carlos Teixeira Alves Filho[a],*, Maria Carolina de Almeida[b], Fernando Pereira de Sá[b], Tatianne Ferreira de Oliveira[c]

[a]*School of Civil and Environmental Engineering, Federal University of Goiás - UFG, Colemar Natal e Silva Campus, Avenida Universitária, nº 1488, Setor Universitário, 74690-900, Goiânia, Brazil, email: carlosteixeira@ufg.br*
[b]*Federal Institute of Education, Science and Technology of Goiás - IFG, Inhumas Campus, Avenida Universitária, Vale das Goiabeiras, 75402-556, Inhumas, Brazil, emails: maria.almeida@ifg.edu.br (M.C. de Almeida), fernandofpsa@gmail.com (F.P. de Sá)*
[c]*School of Agronomy, Federal University of Goiás-UFG, Samambaia Campus, Rodovia Goiânia-Nova Veneza Km-0, Caixa Postal 131, CEP 74690-900, Goiânia, Brazil, email: tatianne_ferreira_oliveira@ufg.br*

ABSTRACT

The disposal of effluents from food industries into surface waters represents a serious environmental problem, mainly due to the presence of dyes in their composition, which alters the photosynthetic capacity of aquatic plants. In addition, many of the by-products generated by their degradation can be toxic and non-biodegradable. In this research, a photocatalytic reactor constructed from quartz tubes was designed and built to be applied in an advanced oxidation processes (UV/TiO_2), for the degradation of indigo carmine blue food dye, in an aqueous solution. A factorial design 2^3 was performed, where three variables were studied: the dye concentration (20 and 30 mg/L); the pH of the solution (4 and 10), and the TiO_2 dosage (20 and 30 mg/L). The aqueous solution was subjected to constant circulation, inside the quartz tubes in order to carry out the experiments. The results showed that pH is the parameter with the greatest impact on degradation efficiency, which showed that at basic pH (10) a degradation rate of 99.91% was achieved, against 34.95% at acidic pH (4). It was concluded that the photo reactor using solar energy, presented itself as a strategic alternative for the removal of contaminants, including recalcitrant, such as synthetic dyes, which contribute to agro-industrial sustainability.

Keywords: Advanced oxidative process; Heterogeneous photocatalysis; Food coloring; Photocatalytic reactor

1. Introduction

The need to maintain stability in the supply of foodstuffs, in addition to the emergence of new products made available by the food industry, has had a negative impact on global water resources. The dyes are used only to make food more attractive and stimulate its consumption. Manufacturers use dyes to change or intensify food colors during their production process [1,2].

In the food industry, the application of synthetic food dye presents as something advantageous. It is stable to light, oxygen, and thermal variations, demonstrates a pH, enjoys a low acquisition or production cost, and has high dyeing capacity while maintaining a uniformity in the color obtained. These facts mean that synthetic dyes are widely used [3].

When waste from the aforementioned industries is released into the environment, without the degradation or prior removal of dyes, the problem arises and is characterized

* Corresponding author.

by pollution and the contamination of soil, surface, and groundwater [4].

When a dye is released into the receiving water body, even in low amounts of less than 1 ppm, it can absorb, reflect, and prevent the penetration of sunlight into aquatic environments, a fact that hinders the photosynthetic activity of plants and organisms in the aquatic ecosystem [5,6].

Indigo carmine blue food coloring can be obtained by synthesis in the laboratory, in powder form, is soluble in water, and is used only in industrial processes and in contact with humans, but can cause nausea, vomiting, hypertension, and allergies. According to Lopes et al. [7], in addition to Brazil, it is used in the United States, The European Union, and Japan.

It is an anionic, synthetic dye, classified as indigoid because it contains a carbonyl chromophore [8] and is recalcitrant to preliminary, primary, and secondary treatments, applied to domestic and industrial effluents in sewage and water treatment plants.

Thus, a possible technological resource to be applied is the advanced oxidation processes (AOP), a technology developed in recent decades, whose purpose is the induction of physical chemical reactions, capable of generating hydroxyl radicals (OH$^\bullet$), in sufficient quantities to oxidize the chemical substances present in effluents. AOPs can materialize inside photocatalytic reactors, built for the generation of and use of hydroxyl radicals (OH$^\bullet$). They possess a high oxidizing power obtained from semiconductors that absorb sunlight or artificial light and release electrons capable of producing reactive radicals that oxidize and mineralize organic compounds. By-products include organic salts, water, and carbon dioxide [9,10].

They are presented as an alternative to conventional oxidation methods, degrading persistent materials when conventional treatments do not attain the efficacy required. These processes can be heterogeneous or homogeneous, and may or may not use UV radiation. The application of these AOPs is promising when combined with the use of sunlight as an alternative treatment from an economic point of view because it provides energy thus contributing a level of sustainability to the process. It is worth mentioning that the use of sunlight implies cleaner production as it decreases the need to dispose of mercury lamps, while increasing the efficiency in the use of energy, and bringing economic and environmental benefits [11].

The objective of this study was to build a catalytic photo reactor and evaluate the degradation of indigo carmine blue food colorant through a UV/titanium dioxide (TiO$_2$) photocatalysis process. An experimental factorial design 2^3 was used to evaluate the influence of the pH variables (4 and 10), together with a concentration of titanium dioxide TiO$_2$ (20 and 30 mg/L), and dye concentration (20 and 30 mg/L) in the heterogeneous photocatalysis process.

2. Material and methods

2.1. Material and reagents

In the experiments, tubes produced from quartz with 99.995% purity in SiO$_2$, with an internal dimension of 16 mm and an external dimension of 20 mm, with a length of 500 mm, manufactured and marketed by "Action - Scientific Technology, Campinas - SP, Brazil" were used. Lettuce seeds (*Lactuca*

sativa L.), of the Simpson (Porto Alegre - RS, Brazil) select variety, purchased from local commerce, and were used for the execution of the effluent phytotoxicity test before and after treatment. The chemical reagents used were titanium dioxide TiO$_2$ P25 produced and distributed by Degussa Evonik Brazil, composed of 75% anatase and 25% rutile. The surface area measured from 20 to 50 nm, and used an indigo carmine blue food dye, with a minimum concentration of 85%, produced by Adicel Indústria e Comércio (Belo Horizonte - MG, Brazil) producing the molecular chemical formula C$_{16}$H$_8$N$_2$Na$_2$O$_8$S$_2$ (Fig. 1) and that of zinc sulfate (ZnSO$_4$).

2.2. Photocatalytic reactor design and construction

Initially, with the aid of AutoCAD software, a structural design of the photo reactor was developed to support the tubes, connectors, hoses, and component accessories for system operation (Fig. 2). The photocatalytic reactor is composed of a set of quartz tubes, arranged in parallel, under solar illumination with the effluent in constant circulation. The structure was constructed with USI SAC-300 type steel, with an anti-corrosion background, and the surface was intended to receive solar incidence, inclined in relation to the horizontal angle of 16° 40' 48' south, a value coincident with the latitude in the city of Goiânia.

The photo reactor was assembled in order to obtain a "tubular flow reactor (TFR)" type or an unconcentrated "tubular flow" [12], with forced flow, continuous and piston flows of 4.51 L/min, where four quartz tubes, containing 99.995% purity in SiO$_2$, an internal dimension of 16 mm, an external dimension of 20 mm and a length of 500 mm, arranged in parallel and joined by opaque and elastoplastic connectors, produced in high-density polypropylene, thus having a circuit with an inlet and outlet orifice. A raw effluent reservoir and a pump for pressurization and recirculation of the effluent under treatment were also installed. The new structure is mobile (Fig. 3) and equipped with pulleys, allowing for its displacement to sunny regions to avail of UV radiation and promote photodegradation reactions. At the end of each experiment the connectors were removed, and the quartz tubes were cleaned, sanitized, and dried. This is called the decay curve.

2.3. Experimental design: degradation kinetics

Photodegradation kinetics were performed using a factorial design 2^3, where three factors were studied: the concentration of dye introduced into the reactor (20 and 30 mg/L); the pH of the solution (4 and 10), and the TiO$_2$ dosage (20 and 30 mg/L), as shown in Tables 1 and 2. According to the National Institute for Space Research (INPE), using the Laboratory of Modeling and Studies of Renewable
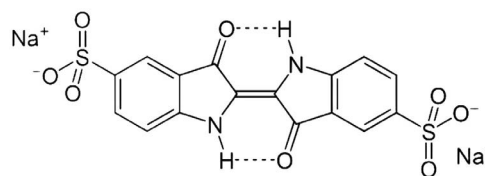

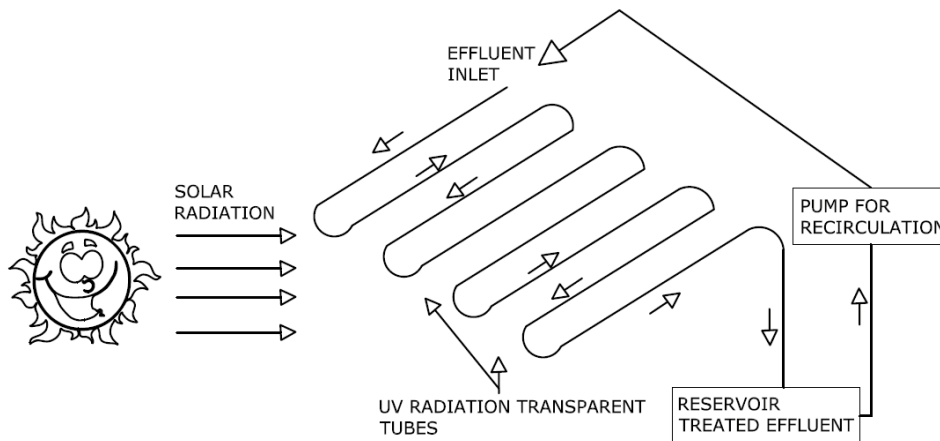Fig. 1. Molecular chemical formula of indigo carmine blue dye.

Fig. 2. Simplified scheme of the photocatalytic reactor.

Fig. 3. Photocatalytic reactor in operation.

Table 1
Factors and levels for three independent variables

Factor	Max. level (+)	Min. level (−)
Dye concentration (mg/L)	30	20
TiO$_2$ dosage (mg/L)	30	20
pH	10	4

Table 2
Values applied in the factorial design for three independent variables

Kinetics	TiO$_2$ concentration (mg/L)	Dye concentration (mg/L)	pH
1	30	30	10
2	30	30	4
3	30	20	10
4	30	20	4
5	20	30	10
6	20	30	4
7	20	20	10
8	20	20	4

Energy Resources (LABREN), the irradiation in the inclined plane at the local latitude and date of the experiments corresponded to 5,464 Wh/m^2 of energy per day, which was its extreme ultraviolet radiation index, a fact that allows maximum use of solar rays to perform the experiments.

2 L of the initial solution were inserted into the photo reactor and transported to an open environment, in the presence of solar lighting, to obtain light radiation in the visible and ultraviolet region and so, beginning the process of continuous circulation of the effluent with the use of a pressurization pump. During the advanced oxidation processes, samples were collected at different time intervals (0', 5', 10', 15', 20', 30', 40', 50', 60', 75', 90', 105' and 120' min) in order to evaluate the elimination of the contaminant in the solution. 3.5 mL of aliquot were collected in these time intervals and the samples were filtered in a syringe filter (0.45 μm) for removal of TiO$_2$ and packed in test tubes wrapped in aluminium foil, eliminating the possibility of light degradation after collection. After filtration, 3.5 mL of effluent was inserted into a quartz cuvette and the absorbance was measured and quantified by spectrophotometry in a Shimadzu UV-1800 (Kyoto, Japan) apparatus at the maximum absorption wavelength of the 610 nm solution samples, which was determined after scanning them from 300 to 700 nm.

The calculations of the final concentrations were obtained through the standard curve of the dye and the percentage of elimination was calculated by Eq. (1).

$$DRE = \left(\frac{C_i - C_f}{C_i}\right) \times 100 \tag{1}$$

where DRE represents the dye removal efficiency (%) over time; C_i and C_f represent, respectively, the initial and final dye concentration (mg/L).

2.4. Toxicity test

Toxicity assessment tests were performed using *Lactuca sativa* seeds according to the methodology proposed by Melo et al. [13] and United States Environmental Protection Agency [14]. The analysis consisted of observing lettuce seeds (*Lactuca sativa* L.), of the Simpson (Porto Alegre - RS, Brazil) select variety, the index and percentage of germination, and the development of the radicle, when in contact with effluent samples. Toxicity analysis was performed at intervals of 0′, 60′, and 120′ min for kinetics with higher and lower discoloration rates.

The positive control was performed in a solution of zinc sulfate ($ZnSO_4$), prepared at a concentration of 0.05 mol/L. The negative control was performed with distilled water.

As proposed by Viana et al. [15], the experiments were carried out in triplicate for each sample, both for the positive and negative control, where 24 Petri dishes with an internal diameter of 9 cm each, were washed, sanitized, and dried in an oven and then lined with qualitative filter paper. Then, with the aid of tweezers, 15 lettuce seeds (*Lactuca sativa* L.) were placed inside each plate, on the surface of the paper, with sufficient space between them for root growth and with 4 mL of the indigo carmine blue effluent added, to be analyzed later [16].

Therefore, to maintain internal moisture and prevent evaporation, the plates were wrapped in plastic polyvinyl chloride (PVC) film, packed in aluminium foil to avoid the incidence of light and then stored in a biochemical oxygen demand incubator, at a constant temperature of 22°C ± 2°C, for 120 h [15] and analyzed in relation to their root growth index [Eq. (2)] and germination index, [Eq. (3)], as recommended by Melo et al. [13].

All seeds with a radicle length greater than or equal to 1 mm were considered germinated [17].

$$RGI = \frac{SRL}{RLN} \tag{2}$$

where RGI represents the root growth index, SRL the sample root length (mm) and RLN is the root length in the negative control.

$$GI = RGI \times \left(\frac{GSS}{GSN}\right) \times 100 \tag{3}$$

where GI represents the germination index, RGI is the root growth index, GSS the number of seeds germinated in the sample, and GSN the number of seeds germinated in the negative control.

For validation of the test and to verify if the experiment had satisfactory results, the criteria of Viana et al. [15] were adopted, which provides for minimum germination of 90% of the seeds of the negative control plates and the coefficient of variation of root elongation [Eq. (4)] does not exceed 30%. If these criteria had not been met, the test would have been repeated, using another batch of seeds.

$$VC = \left(\frac{S}{X}\right) \times 100 \tag{4}$$

where VC is the variation coefficient, S is the standard deviation, and X is the mean.

The RGI was classified into three categories, according to the toxic effects presented: firstly, inhibition of root elongation when $0 < RGI < 0.8$; secondly, there was no significant effect (NSE) when $0.8 \leq RGI \leq 1.2$; finally, stimulation of root elongation (E) when $RGI > 1.2$ [15].

The toxicity classification was performed in accordance with the methodology of González et al. [18], where the NGI and NRI indices point to the level of effluent toxicity according to Table 3.
where NRI [Eq. (5)], represents the normalized root growth percentage index and NGI [Eq. (6)] the normalized residual germination percentage index.

$$NRI(\%) = \frac{ALRSC - ALGRNC}{ALGRNC} \tag{5}$$

where ALRSC represents the average length of the radicles in each sample and ALGRNC represents the average length of the germinated radicles in the negative control.

$$NGI(\%) = \frac{GERMsample - GERMcont.negative}{GERMcont.negative} \tag{6}$$

where sample GERM corresponds to the average percentage of germinated seeds in each sample and negative GERMcont. corresponds to the percentage of germinated seeds in the negative control.

2.5. Statistical analysis

The data obtained were analyzed using the STATISTICA 7.0 program (StatSoft Inc., Tulsa, Oklahoma, USA), with a significance level ($p < 0.05$), to determine response surfaces.

3. Results and discussions

3.1. Experimental design: degradation kinetics

Fig. 4 shows the degradation curves of indigo carmine blue dye as a function of time in the different experimental conditions applied.

Table 3
Toxicity classification

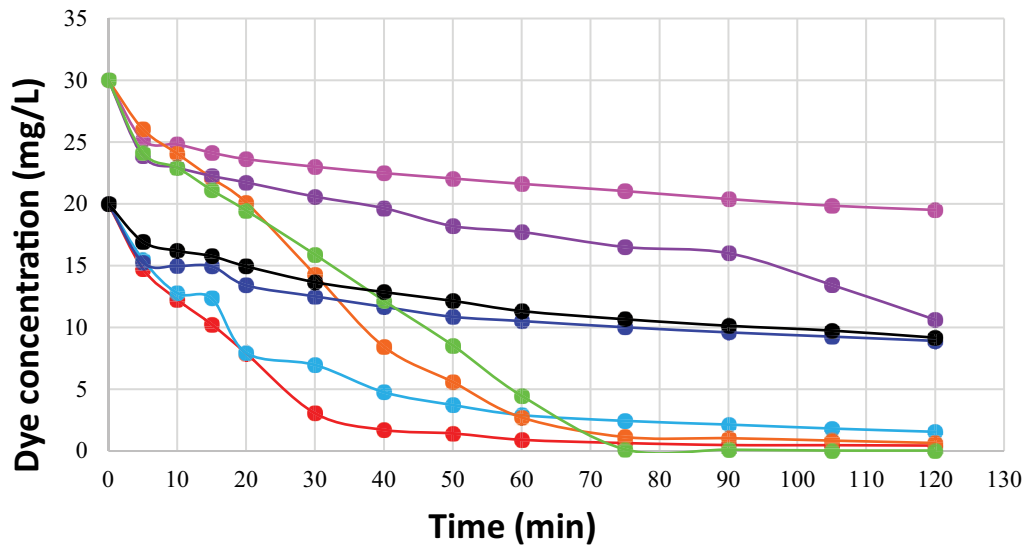| NGI (%) and NRI (%)	Toxicity
0 to –0.25	Lower toxicity
–0.25 to –0.5	Moderate toxicity
–0.5 to –0.75	High toxicity
–0.75 to –1.0	Very high toxicity

Fig. 4. Comparison between decay curves of indigo carmine blue dye as a function of time: C1 (●) 30 mg/L TiO_2, dye concentrate 30 mg/L, pH 10; C2 (●) 30 mg/L TiO_2, dye concentrate 30 mg/L, pH 4; C3 (●) 30 mg/L TiO_2, dye concentrate 20 mg/L, pH 10; C4 (●) 30 mg/L TiO_2, dye concentrate 20 mg/L, pH 4; C5 (●) 20 mg/L TiO_2, dye concentrate 30 mg/L, pH 10; C6 (●) 20 mg/L TiO_2, dye concentrate 30 mg/L, pH 4; C7 (●) 20 mg/L TiO_2, dye concentrate 20 mg/L, pH 10; C8 (●) 20 mg/L of TiO_2, dye concentrate 20 mg/L, pH 4.

Table 4
Results obtained in the experimental design of discoloration

| Experiment	Dye elimination rate (%)	Time spent planning (min)
1	97.85	120
2	64.63	120
3	97.85	120
4	55.42	120
5	99.91	120
6	34.95	120
7	92.25	120
8	54.16	120

The results obtained reveal that there was discoloration of the dye in the eight kinetics performed, but the variables studied, titanium dioxide TiO_2, pH, and initial concentration of the dye, impacted the treatment considerably.

As shown in Table 4, experiment number 5 reveals that it was possible to obtain a discoloration rate of 99.91%, which was the best result for the experimental condition studied. Experiment number 6 demonstrated the worst experimental condition, with only 34.95% discoloration obtained.

3.2. Effect of pH

The results obtained in the experimental design (Table 3) demonstrate that, for pH 10, the highest dye elimination rates were obtained, a fact that indicates that the basic pH is the operational parameter with the greatest influence on the photodegradation process of indigo carmine blue dye in aqueous solution when compared to other variables of the process. In order to understand this more fully, we need to explain heterogeneous photocatalysis, as follows.

According to Lima et al. [19], the pH of the effluent to be treated is the variable with the greatest importance in the photocatalytic degradation process. This is justified since the pH exerts influences on the ionic species of the solution and the physical and chemical properties of the photocatalyst, a fact that provides an increase or reduction in the efficiency of the discoloration process. At basic pH, there is a greater formation of OH• hydroxyl radicals, from the reaction between hydroxide ions and positive gaps [Eq. (7)]. Thus, it is possible to affirm that in an alkaline solution, there is a propensity to generate OH• radicals by oxidizing more hydroxide ions available on the surface of TiO_2, a fact that justifies a process efficiency increase [20].

$$TiO_2\left(h_{BV}^+\right) + OH^-\left(adsorbed\right) \rightarrow TiO_2\left(OH^\bullet\right) \tag{7}$$
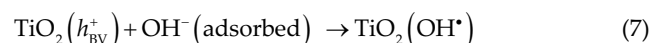
Similar studies for photocatalytic degradation of indigotine blue dye conducted by Naciri et al. [21], indicate that the rate of discoloration increases with an increase in pH, with a discoloration efficiency of 99% obtained in 125 min of treatment for pH 11, this being the pH value chosen as the ideal parameter.

3.3. Effect of TiO_2 concentration

It is also noted from this study that, individually, the concentration of TiO_2 is an important factor in the decolorization process of the indigotine blue dye. Statistically, it is the second variable with the greatest influence on the process. At doses of 30 mg/L, dye elimination of up to 97.85% was achieved.

Naciri et al. [21], performed a series of experiments using different amounts of TiO_2, from 0.125 to 3 g/L in solutions of 20 mg/L of dye. When the catalyst dosage was increased, the discoloration rate increased and reached the

maximum of 99% for 3 g/L of catalyst when the dye solution was irradiated for only 90 min. This is justified by the fact that the greater the amount of photocatalyst the greater the increase in the number of active sites, as these were directly proportional to the particle density of the photocatalyst in the radiation area, which results in an increase in the formation and amount of hydroxyl radicals. Therefore, it favored the photodegradation reactions [22].

3.4. Effect of initial dye concentration

Statistically, the effect of the initial dye concentration is the variable with the least influence on photodegradation.

It can be noted that for higher initial concentrations of dye, as is the case of kinetics C1 and C5, with 30 mg/L, the process only required more time to reach levels of degradation similar to kinetics with lower initial concentrations C3 and C7, with 20 mg/L. However, it is possible to note from Fig. 4 that from the second hour of treatment, the decay curves of kinetics C1, C3, C5, and C7 approach the highest rate of discoloration while C5 actually reached the highest rate. Similar studies carried out by Sood et al. [23], revealed that it is possible to obtain total photocatalytic degradation of the indigo carmine blue dye at different initial concentrations. However, the higher the initial concentration, the longer the treatment time should be.

Table 5
Regression coefficients for the discoloration capacity of the dye by advanced oxidation processes

| Factors	Regression coefficient	Standard error	$t(1)$	p-value	Estimates by range (95%)	
					Lower limit	Upper limit
Mean	74.6275	4.510000	16.54712	0.038426	17.323	131.9325
(1) Concentration of TiO_2 (mg/L)	–8.6200	9.020000	–0.95565	0.514433	–123.230	105.9900
(2) Concentration of dye (mg/L)	0.5850	9.020000	0.06486	0.958769	–114.025	115.1950
(3) pH	–44.6750	9.020000	–4.95288	0.126830	–159.285	69.9350
1 and 2	5.1900	9.020000	0.57539	0.667604	–109.420	119.8000
1 and 3	–6.8500	9.020000	–0.75942	0.586512	–121.460	107.7600
2 and 3	4.4150	9.020000	0.48947	0.710219	–110.195	119.0250
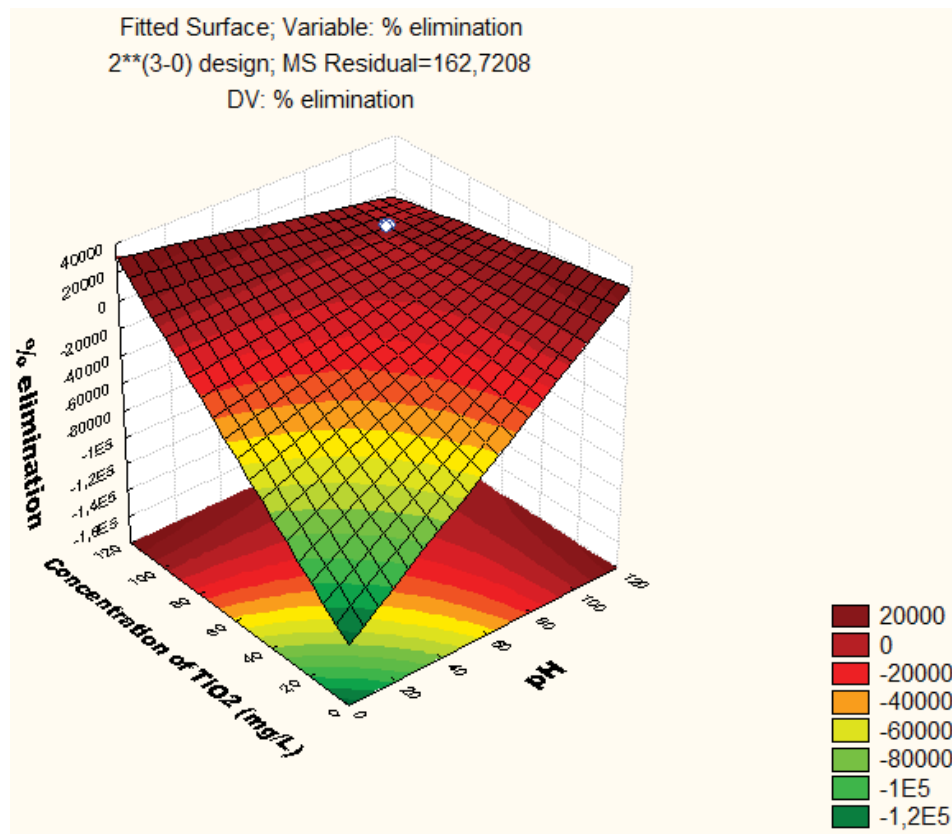
Fig. 5. Surface response.

4. Statistical analysis

From the results obtained from the degradation kinetics of indigo carmine blue dye as a function of time, the regression coefficients and *p*-values were calculated (Table 5), with a probability of significance of 0.05, whose objective was to analyze the influence of the variables studied: dye concentration introduced into the reactor, pH of the solution and TiO$_2$ dosage.

The results obtained from the three variables combined, dye concentration, TiO$_2$ concentration, and pH showed a statistically significant correlation in the elimination of the dye because the probability of significance *p*-value corresponds to 0.038426, which is lower than the probability of significance 0.05, adopted for statistical calculations.

The results also confirm that pH is the variable with the greatest influence on the photodegradation process. Thus, the more acidic the pH, the lower the elimination of the dye molecule. Similarly, lower TiO$_2$ concentrations had negative effects on AOP and higher initial dye concentrations had a positive influence on photodegradation.

The response surfaces (Fig. 5) confirm the results obtained experimentally, since they reveal higher rates of elimination of indigo carmine blue dye by AOP, for higher pH values and higher TiO$_2$ concentration values.

The effect is positive for the variable discoloration capacity, that is, directly proportional, when there is an increase in pH, there is also an increase in the dye elimination capacity. Similarly, for TiO$_2$ increments, there is a directly proportional increase in the dye's elimination capacity. Therefore, the Pareto graph (Fig. 6) confirms that pH is the variable with the greatest influence on the dye photodegradation process.

Thus, it can be stated that, although none of the independent variables has a probability of statistical significance, they still have a great impact on the dye elimination
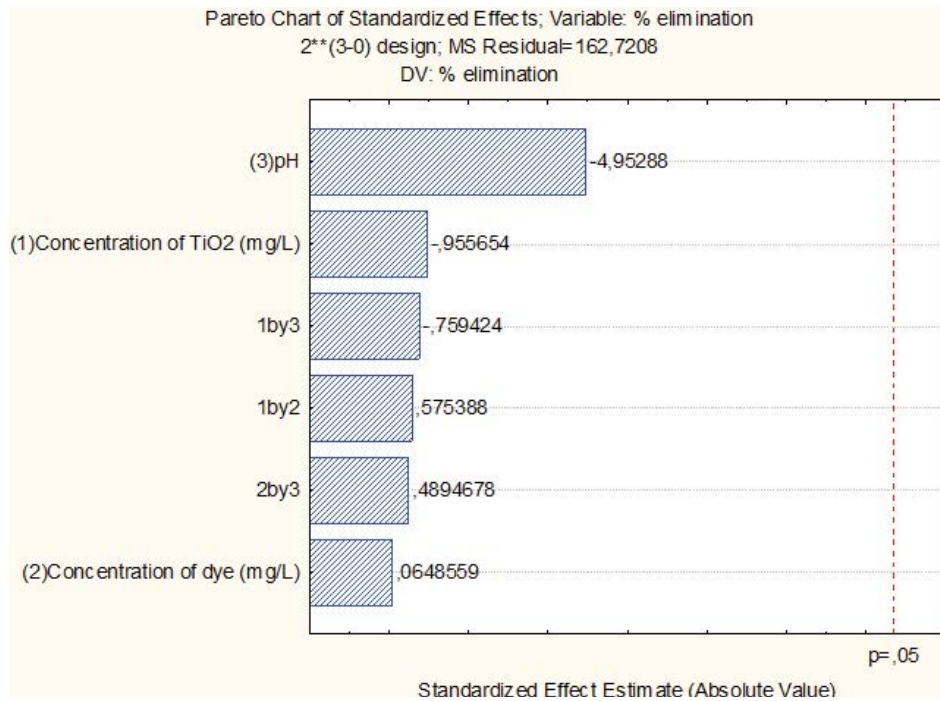

Fig. 6. Pareto diagram of standardized effects for response discoloration capacity by advanced oxidation processes.

Table 6
Statistical results of phytotoxicity analysis

Sample		% Germination	Coeff. variation (%)	RGI	NGI (%)	NRI (%)
Negative control	–	97.78	29.70	–	–	–
Experiment 03 – Treatment time (min)	0	84.444	65.605	0.714	–0.136	–0.288
	60	84.444	78.106	0.689	–0.136	–0.310
	120	88.889	59.432	0.811	–0.091	–0.190
Experiment 06 – Treatment time (min)	0	91.111	52.826	0.914	–0.068	–0.086
	60	93.333	66.073	0.731	–0.045	–0.269
	120	88.889	51.815	0.908	–0.091	–0.091
Positive control	–	0.000	–	–	–	–

capacity. Given the results obtained, it can also be said that the three factors or variables have approximate amplitudes of effect, as highlighted by [24,25]. The photodegradation process is a complex interaction with different factors at the same time as a process driven by a major factor.

4.1. Phytotoxicity test results

From the statistical analysis (Table 6), it was possible to argue in favour of the acceptance of the test, since for the negative control, 97.78% of the seeds germinated, and as the coefficient of variation of root elongation was 29.70, it meets the criteria of Viana et al. [15].

The results for the normalized residual germination percentage index (NGI) are between 0 to –0.25, which initially suggests being the effluent before and after the 120 min of low toxicity treatment. However, when establishing a relationship between the indices obtained in the statistical analysis and the phytotoxicity of the set, it can be concluded that, although the NGI indicates low toxicity, the normalized root growth percentage index (NRI) was between –0.25 to –0.5, a fact that affects the effluent of moderate toxicity, both before and after treatment.

The RGI obtained was 0.8 ≤ RGI ≤ 1.2, which pointed to no significant effect (NES) on root growth.

5. Conclusions

The experiments demonstrated that the reactor proposed and developed in this project is a sustainable treatment alternative, according to the evidence given by the high degradation rates of indigo carmine blue dye. The results revealed that pH is the variable with the greatest influence on the process. In alkaline solutions (pH 10), 99.91% degradation was obtained, while in acidic solutions (pH 4), the highest rate of discoloration was 64.63%.

From the results obtained by the decontamination of the effluent treated in this study, it is possible to conclude that the advanced oxidation processes (UV/TiO$_2$), using solar energy, presented itself as a strategic alternative for the removal of contaminants, including recalcitrant, such as synthetic dyes. In addition, it is an economically viable, sustainable treatment as it does not require energy expenditure. This research also provides strategies and indicators for the development of a new photocatalysis and TiO$_2$ coupling technology for wastewater treatment.

Acknowledgements

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