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Taguchi Method Applied to Environmental Engineering

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1. Introduction

Over the last decades, environmental concerns have become more critical and frequent. This is, mainly, due to population growth and the increase of industrial activities in which anthropogenic actions have reached catastrophic proportions resulting in changes of soil, air, and water quality [1].

Environmental pollution by industrial effluent is being characterized as one of the major causes of the aggravation of this problem. Residues, in general, produce diversified compounds, containing, frequently, pollutants that are toxic and resistant to conventional treatments such as coagulation/flocculation or biodegradation [2], and they are eventually discharged, in most of the cases, in an inadequate way causing severe damages. Regarding the environmental problem, researchers were driven to study the feasibility of new techniques and methodologies, as well as, the emission and pollutant discharge control. In order to apply the pollution control and to attend environmental legislation, patterns and quality indicators were established. In terms of water quality: oxygen concentration, phenols, Hg, pH, temperature, among other requirements [3].

Companies search for new environmental alternatives to treat generated residuals. The environmental reality is demanding for further actions to mitigate industrial impacts on water. Therefore, water treatment has become a mandatory investment to industries, institutions, and others with the aim to attending environmental laws, as well as ISO 14000 series.

In this context, textile sector can be referred because of its great industrial area that generates a high volume of effluent deeply colored and containing high concentration of organic compounds, which if not treated, may cause serious damage to environmental contamination [4].

Hazardous waste treatment and the presence of organic pollutants in water have increased the use of alternatives to environmental matrixes such as the use of Advanced Oxidation Processes (AOPs) to residual water treatment [5].

This work features the application of Design of Experiments; Taguchi L_9 Orthogonal Array; in the effluent treatment of polyester resin that is originated from textile industries and through the application of Advanced Oxidation Processes (Heterogeneous Photocatalysis - UV/TiO_2) in the study of chemical oxygen demand.

2. Advanced oxidation processes

Advanced Oxidation Processes (AOP's) and electrochemical methodologies are developed to treat the contaminants of drinking water and industrial effluents. The oxidation processes are based on reactive species generation that degrades a great variety of organic pollutants, in a quick and non-selective way. Reactive species are unstable and must be generated continuously "in situ", through chemical or photochemical reactions [6].

AOPs are defined as processes with great capacity of producing hydroxyl radicals (•OH), that are reactive species. The high standard potential of radical's reduction is demonstrated by Equation 1. This radical is capable of oxidizing a great variety of organic compounds to CO_2, H_2O and inorganic ions originated from heteroatoms [7-8].

$$\bullet OH + e^- + H+ \rightarrow H_2O \qquad\qquad E_o = 2,730 \text{ V} \qquad\qquad (1)$$

Its destructive process is one of its great advantages. Contaminants are chemically destroyed instead of undergoing a phase change that happens, for instance, in physical-chemical processes of adsorption, filtration, precipitation, coagulation, flocculation, sedimentation, flotation, membrane use, organic and inorganic adsorption, centrifugation, reverse osmosis, extraction, distillation and evaporation [9]. The final disposition of solid phases continues being a problem without any solution; therefore, a passive agent [10]. This reagent is very few selective, electrophilic character, easy to produce and detains kinetic reaction control [11].

The hydroxyl radicals can be obtained from strong oxidants, as H_2O_2 and O_3 combined or not with UV radiation, with salts of Iron II or III, combined or not, with radiation, photocatalysis with TiO_2 or water photolosys with UV radiation [12].
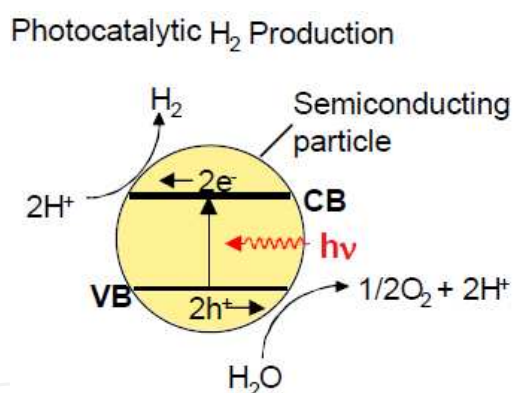
The organic matter (OM) present in the system is attacked by hydroxyl radical at the moment that it is generated, and as a consequence of this process, the effluent is degraded to other intermediate products described in Equation 2[13].

$$\bullet OH + OM \rightarrow Intermediates \tag{2}$$

The several AOPs are split into two groups: Homogenous Processes and Heterogeneous Processes. The former occur in one single phase and use ozone, H_2O_2 or Fenton reagent (mixture of H_2O_2 with salts of Fe^{2+}) as hydroxyl radical generators. The latter uses semiconductors as catalysts (titanium dioxide, zinc oxide, etc.)[14].

3. Heterogeneous photocatalysis

Practical studies using TiO_2 have been developed; however, the reaction mechanism is not totally understandable, yet. Nevertheless, most of researchers agree on some mechanisms steps such as: the excitation of semiconductors species and the formation of h+BV and e-BC, the recombination process among them, O_2, H_2O adsorption and organic species on the semiconductor surface, "trapping" where chemical species donate or accept a pair of electrons e-/h+ preventing the recombination. It is believed that O2 is the main responsible specie to give continuity to the reactions started during the photo-oxidation process, reacting as a formed organic radical and promoting a complete mineralization [15-16]. Figure 1 shows the excitation scheme of the semiconductors.

Figure 1. Schematic illustration of electricity generation and hydrogen production by solar energy conversion using semiconducting materials. CB: Conduction band, VB: Valence band Source: [17].

Direct oxidation process occurs when a photogerated gap in the valence band of semiconductor reacts directly with the organic compound (Equation 3) [18].

$$R_1\big(ads\big) + h+BV \rightarrow R_1\big(ads\big) \tag{3}$$

Indirect oxidation process occurs when a photogerated gap in the valence band of semiconductor reacts with H_2O molecule adsorbed on the semiconductor surface producing hydroxyl radical that will oxide the organic material (Equation 4) [18-19].

$$\bullet OH + R_1 \rightarrow R_2 \tag{4}$$

Photocatalytic process has been efficiently used to degrade innumerable recalcitrant substances prior to the biological treatment.

4. Benefits of advanced oxidation processes

Advanced Oxidation Processes offer several advantages when compared to conventional oxidation processes [20-21].

- Assimilate a large variety of organic compounds;

- Complete mineralization of pollutants;

- Destroy resistant refractory compounds to other treatments, as for example, biological treatment;

- May be used in other processes as a pre or post-treatment;

- Used in effluents with high toxicity that can entail a certain difficulty in the biological process treatment;

- Enable *in situ* treatment;

- Do not create reaction by-products;

- Improve the organoleptic properties of the treated water;

- Contain oxidizing power with elevated kinetic reaction.

5. Chemical Oxygen Demand (COD)

Chemical Oxygen Demand (COD) measures the amount of oxygen consumed through the organic material in water and, also represents an essential parameter in the characterization study of sanitary wastewater and industrial effluents. COD is crucial when used along with BOD to analyze and evaluate wastewater biodegradability [22].

By determining COD, the oxidation-reduction reaction is performed in a closed system using potassium dichromate due to its high oxidative capacity and to its application in a large variety of samples and operational feasibility [23].

Sample results of COD using potassium dichromate as an oxidation agent are superior to BOD, because the high oxidative power of the potassium dichromate is greater if compared to the action of micro-organisms, except in rarely cases, as aromatic hydrocarbonetes and pyridine. BOD measures only the biodegradable fraction. The more this value approximates to COD more easily biodegradable the effluent is [22, 24-25].

6. Design of Experiments (DOE)

Design of Experiments has been widely used to optimize processes parameters and to improve the quality of products with the application of engineering concepts and statistics [26].

Design of Experiments is defined as a set of applied statistical planning techniques, conducting, analyzing and interpreting controlled tests with the aim to find and define factors that may influence values of a parameter or of a group of parameters [27].

DOE considers interaction among variables and may be used to optimize operational parameters in multivariable systems [28].

According to [29], design of experiments was studied as a relevant mathematical tool in the area of Advanced Oxidation Processes. Taguchi's Orthogonal Array L_9 was used in this work for the degradation of organic material of the polyester resin effluent and the percentage reduction of the total organic carbon obtained in the treatment was 39.489%. This removal of organic load corresponds to an average ratio of TOC removal. This condition is inclusive of the weight ratio of hydrogen peroxide at 183g, pH = 3, TiO_2 = 0.250 g/L and lamp intensity = 21 W.

Design of experiments was used by [30] in the degradation of organic material of the polyester resin effluent by advanced oxidation processes. Taguchi's Orthogonal Array L_{16} was used to select statistically the most significant factors in the process; being optimized, lately. It was concluded that more influent variables permitted a reduction of 34% COD of the polyester resin effluent.
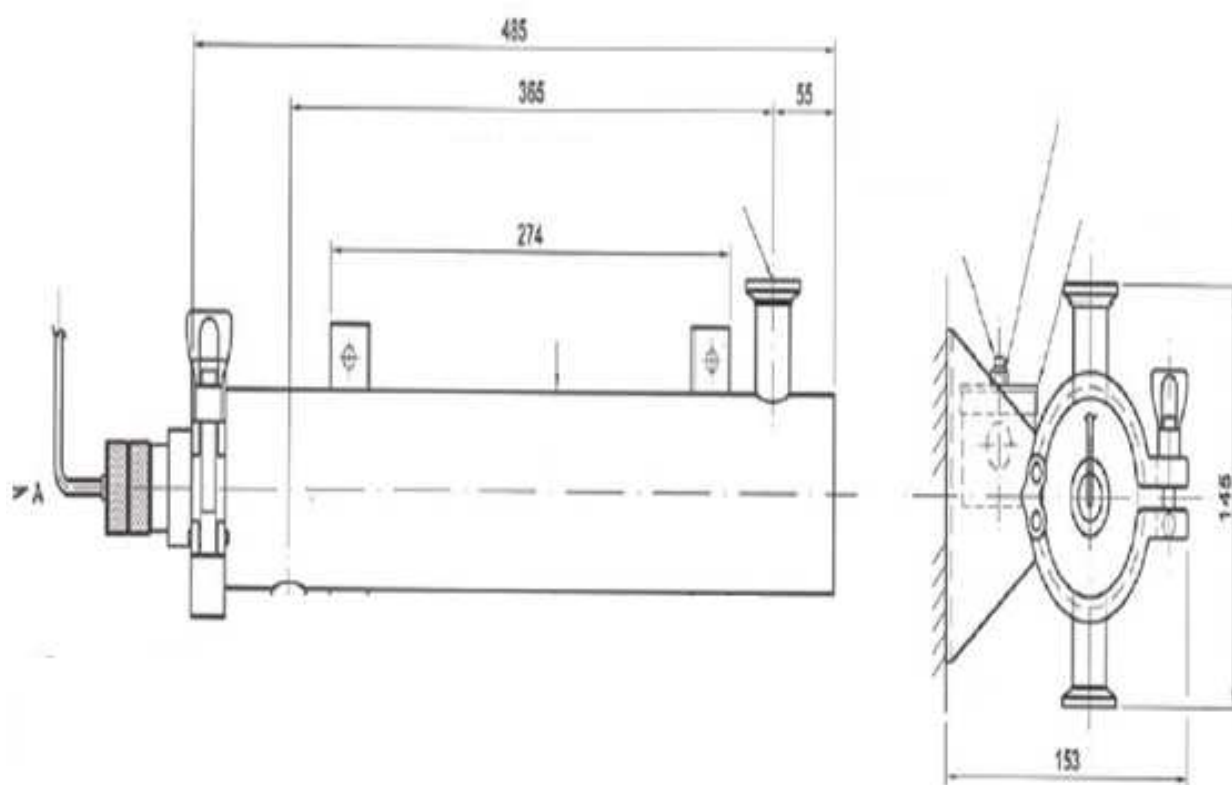
7. Taguchi method

According to [31], Taguchi's method is a powerful mathematical tool capable to find significant parameters of an ideal process through multiple qualitative aspects.

The application of Taguchi's method [32] consists of:

• Selecting the variable response to be optimized;

• Identifying factors (entry variables) and choosing the levels;

• Selecting the appropriate orthogonal array according to literature [33];

• Performing random experiments to avoid systematic errors;

• Analyzing results by using signal-to-noise ratio (S/N) and analysis of variance (ANOVA);

• Finding the best parameter settings.

There are independent variables or entries in the signal-to-noise ratio that compromises the performance of a process. For this reason, two categories are defined: controllable and non-controllable factors [34].

Figure 2. Tubular reactor used for photochemical treatment Source: [28]

Taguchi's method uses orthogonal arrays to study diverse factors with a reduced number of experiments [35]. Besides that, the method can offer other advantages as: process variability reduction, conformity of the expected result and, consequently, operational cost reduction [36].

The analysis of variance (ANOVA) is applied to Taguchi's statistical method to evaluate the significance of parameters used in the process [37].

8. Materials

The polyester resin effluent was conditioned in a chamber at 4 °C. The oxidation reaction of the effluent was performed in a tubular reactor of Germetec brand, Model GPJ-463/1, with nominal volume of approximately 1L, receiving radiation from a low-pressure mercury lamp type GPH-463T5L emitting UV radiation of 254 mm intensity of 15 W and 21 W, protected by a quartz pipe according to Figure 2.

The design of experiments followed these steps:

• 1L of effluent for 2L of distilled water was firstly added,

• Then, it was added TiO_2,

• The system for the effluent recirculation was turned on,

- H_2O_2 was added, and

- Simultaneously, the UV lamp was turned on.

9. Results and discussion

The design of experiments of polyester resin effluent was performed in a Taguchi's Orthogonal Array L_9. pH, titanium dioxide (TiO_2), ultraviolet lamp and hydrogen peroxide with concentration of 30% w/w were used in this process as controlled variables. Table 1 shows the variables and levels used in the degradation process.

Table 2 shows Taguchi's Orthogonal Array L_9, where experimental procedures were performed at random and, after each experimental procedure, chemical oxygen demand analysis were performed on each experimental condition.

Controlled Variables (Factors)	Level 1	Level 2	Level 3
A- Ph	3.0	5.0	7.0
B- TiO_2 [g/L]	0.083	0.167	0.25
C- H_2O_2 *[g]	120.0	151.0	182.0
D- UV [W]	Without	15	21

Table 1. Controlled Variables and Levels

Experiment	pH Factor A	TiO_2 Factor B	H_2O_2 Factor C	UV Factor D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Table 2. Taguchi's Orthogonal Array L_9, with 4 factors and 3 levels each

The COD of the effluent sample *in natura* was initially calculated with a mean value of 49990mg/L and, lately submitted to a pre-treatment. For each experiment, the COD of each sample *in natura* was calculated to an equal period of 60-minute-reaction. Then, the percentage of reduction of COD was calculated for each experiment and the results are shown in Table 3 and 4.

Experiment	pH Factor A	TiO_2 Factor B	H_2O_2 Factor C	UV Factor D	Replica 1 reduction of chemical oxygen demand (%)
1	1	1	1	1	73.44
2	1	2	2	2	78.39
3	1	3	3	3	72.77
4	2	1	2	3	82.35
5	2	2	3	1	69.81
6	2	3	1	2	80.70
7	3	1	3	2	71.13
8	3	2	1	3	78.71
9	3	3	2	1	71.13

Table 3. Result of replica 1 - percentage reduction obtained by experiments for an initial COD amount of 49990 mg/l.

Experiment	pH Factor A	TiO_2 Factor B	H_2O_2 Factor C	UV Factor D	Replica 2 reduction of chemical oxygen demand (%)
1	1	1	1	1	73.11
2	1	2	2	2	76.08
3	1	3	3	3	70.14
4	2	1	2	3	81.69
5	2	2	3	1	65.85
6	2	3	1	2	83.34
7	3	1	3	2	69.15
8	3	2	1	3	81.69
9	3	3	2	1	73.77

Table 4. Results of replica 2 – percentage reduction obtained by experiments, for an initial COD amount of 49990 mg/l.

For experiments performed in the first replica, it is noticeable that oxidative processes reduced COD until 82.345% of the initial amount, being experiment 4 the one with best experimental condition for the degradation experiment, consisting of pH of 5, titanium dioxide of 0.083 g/L, hydrogen peroxide of 151 g and ultraviolet lamp intensity of 21 watts.

The design experiments of the second replica featured that advanced oxidation processes reduced COD until 83.34% of the initial amount, being experiment 6 the one with best experimental variables conditions at pH of 5, titanium dioxide of 0.25g/L, titanium peroxide of 120 g and ultraviolet lamp intensity of 15 Watts.

Factors	Sum of Squares	DF	Mean Sum	Fisher Test	P-Value
Intercept	101741.1	1	101741.1	33172.66	0.000000
pH	40.2	2	20.1	6.56	0.017495
TiO$_2$	0.2	2	0.1	0.03	0.975085
H$_2$O$_2$	264.7	2	132.3	43.15	0.000024
UV	149.6	2	74.8	24.39	0.000232
Error	27.6	9	3.1		

Table 5. Analysis of Variance for polyester resin effluent degradation

Table 5 shows ANOVA factor involved in the polyester resin effluent treatment with the Heterogeneous Photocatalytic Process. The analysis of variance with 95% trust, critical F equal to 4.26 and p-value lower than 5% demonstrated that hydrogen peroxide (F= 6.56 and P-value= 1,7%), temperature (F= 43,15 and P-value= 0,0024%) and lamp intensity(F= 24,39 and P-value= 0,0232%), were significant in the COD removal process.
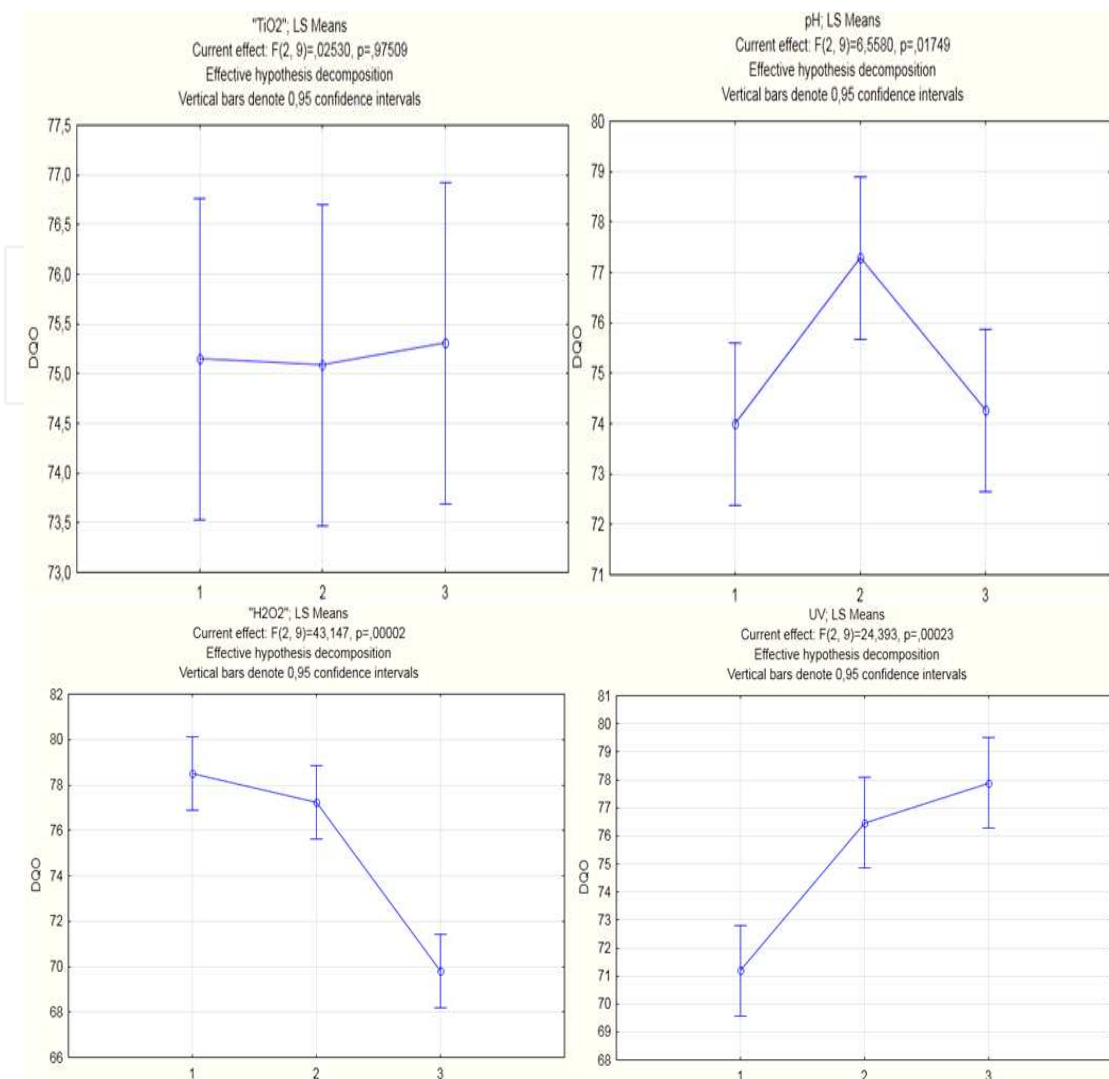
Taguchi's L$_9$ statistical design of experiment (Figure 3) showed more significant parameters for the organic material degradation of the effluent, corresponding to pH=5 adjusted to medium level, TiO$_2$ adjusted to any level, H$_2$O$_2$ concentration = 120g and the ultraviolet lamp intensity adjusted to maximum level of 21 W.

According to [38], the influence of peroxide and temperature is related to the efficiency ratio in the use of this compound and its accelerated decomposition in the reactional medium.

Figure 4 shows the most significant factors to a percentage reduction of Chemical Oxygen Demand. The graph of surface response shows an increase in the degradation of polyester resin compounds by the increase in the UV lamp intensity for lower hydrogen peroxide ratio. The highest percentage reduction is of 83%.

10. Conclusions

Advanced Oxidation Process (Heterogeneous Photocatalysis) for the Taguchi design of this work was evaluated, in which values are found to be significant to the chemical oxygen
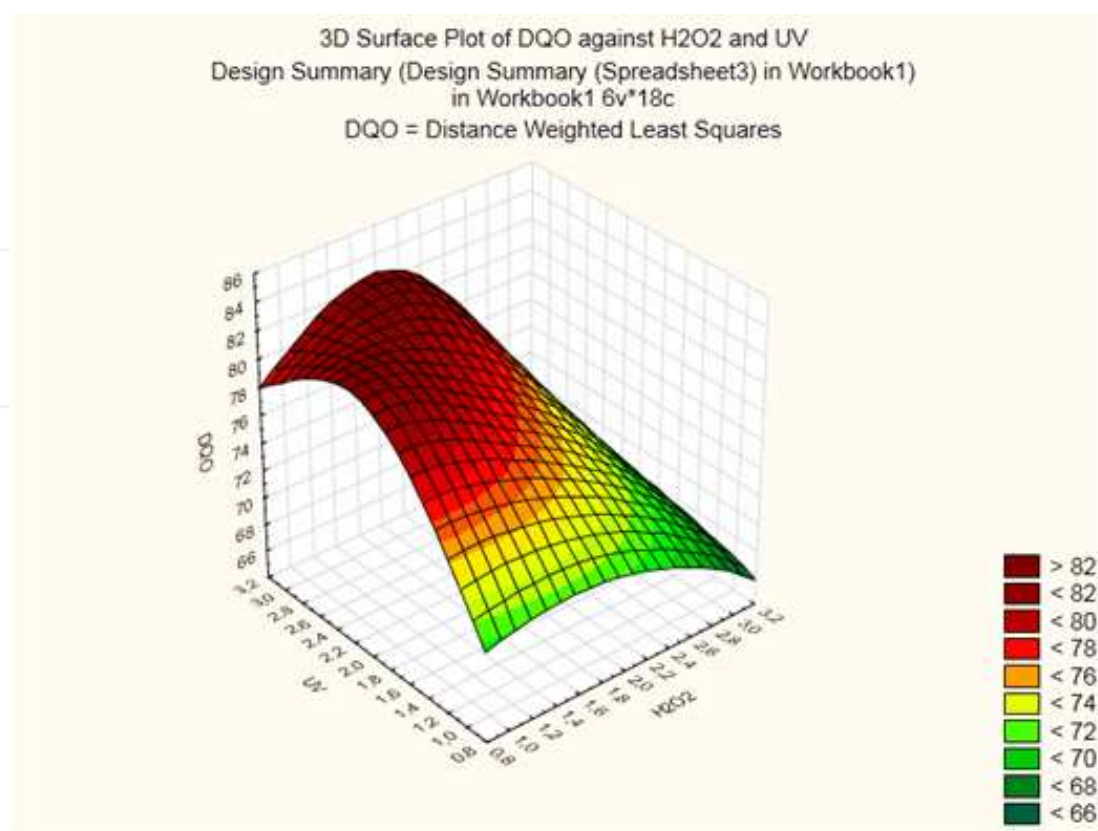
Figure 3. Graphic of Average Impact Factors

demand removal. This design of experiment verified that the highest COD reduction is related to the increase in peroxide hydrogen concentration of 120g, pH=5 and use of UV lamp, since the mechanism of photocatalysis requires energy to the degradation of organic matter of the effluent. Taguchi L_9 Orthogonal Array found was 83%, which demonstrates efficiency on the use of design of experiments and alternative methodologies to the degradation of organic load of polyester resin effluents.

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Figure 4. Graphic of influential factors on COD removal

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