

Taguchi Method Applied in a Treatment of Dairy Wastewater Using Advanced Oxidation Process

Ricardo Batista Penteado
Production Engineering
Department, Univ.
Estadual Paulista/UNESP,
Guaratinguetá, Brazil,

Délio Guerra Filho
Chemical Engineering
Department, São Paulo
State University/
USP/EEL, Lorena, Brazil

**Rubia Fernanda Toledo
de Oliveira**
Production Engineering Dep
artment, Univ. Estadual
Paulista/UNESP,
Guaratinguetá, Brazil,

Messias Borges Silva
Production Engineering
Department, Univ.
Estadual Paulista/UNESP,
Guaratinguetá, Brazil,

ABSTRACT

One of the most usable treatments for the dairy wastewater is the chemical type, because of the amount of organic matter present and easily biodegradable. This work has an objective to verify the degradability of wastewater in a dairy industry and to test an alternative for its treatment through the advanced oxidative process (AOP). The sample of the wastewater, presented some organic load from 2200 to 5500 mg L⁻¹ and the applied process was the treatment with UV/H₂O₂, where was studied the variables of the photochemical process, such as: the pH, the temperature, the concentration of the hydrogen peroxide and the UV irradiation. A factorial design proposed by Taguchi L16 was decisive to get a result. In this process, the variable responses were the removal of the chemical oxygen demand (COD) and turbidity. Up to here point, the experiments were carried out and were observed in the oxidative process the low pH had an important role. Considering the variable pH, the average of the reduction percentage when pH is equal 5 is 45,03%, while that for the pH is equal 10 the average was 8,32%. The maximum reduction of turbidity was 99,1%, while that in, the highest value for the percentage of COD reduction was 71,96%, both for the condition where pH is 5.

KEYWORDS

Dairy wastewater, Advanced oxidation process, Taguchi Method, Turbidity, Chemical Oxygen Demand.

1. INTRODUCTION

The preoccupation with the quality of environment grew up in the last decades due to the evident degradation of our environmental resources. This environmental impact affects directly the humans through its effects on health, agricultural production, as well as effects on forests and rivers.

Among the many environmental problems affecting society today, the process of contamination by industrial activities is one of the most worrisome.

The dairy industries are not an exception, they have been associated as an impacting agent, mainly due to the high level of organic materials present in their wastewater.

According with Sarkar et al (2006) water is used in all steps of the dairy industry including cleaning, sanitization, heating, cooling and floor washing, for this reason the requirement of water is huge.

The effluent from the dairy industry are considered pollutants, mainly because of organic material contained in it and should be

treated before of their discharges. Their constituents, feed on algae that consume dissolved oxygen, bringing danger to fish habitat and threat some recreational activities (CARTA, et al 2004).

Dairy wastewaters have invariably high level in nutrients (Nitrogen, phosphorus and potassium) and dissolved organic material (e.g. oils and fats, lactic acid, etc.), therefore it has a high demand of biological oxygen. In addition, this effluent contains high concentrations of dissolved salts. The use of acid and alkaline solutions for cleaning causes influence on their characteristics and results in a variation of its pH (SENGIL, ÖZACAR, 2006).

Studies report that dairy industries generate about 2.5 liters of effluent per liter of processed milk, can reach the order of thousands of cubic meters per day (PERLE, et al 1995; CARTA, et al 1999; VOURCH, et al 2008).

In milk processing, the operations that generate significantly pollutants are: washing and disinfection of equipment, rupture or bursting of cartons containing milk, losses: on bottling and lubricants transport systems. Such pollutants are composed of milk solids, detergents, disinfectants and lubricants (DANALEWICH et al, 1998).

According to Leal, et al (2006), these industries often encounter difficulties to follow the requirements imposed by the stringent rules for wastewater discharge. Thus, the search for a low-cost technique that is effective is increasing and may contribute to reduce the environmental impacts generated by the dairy industry.

Currently, there are several alternatives to treat effluent of the dairy industries using physical and chemical processes, which can contribute to the efficiency of processes organic. One of them is the use of advanced oxidation processes (AOPs) that has been recently an alternative promising, because it has a degradation power of organic compounds present in the wastewater.

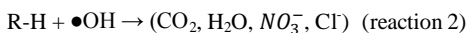
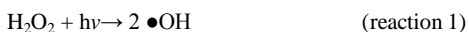
Advanced oxidation processes (AOPs) are those processes which are based on production and utilization of hydroxyl radicals (•OH). Hydroxyl radicals are extremely unstable and reactive because of their high oxidation potential (MAHAMUNI and ADEWUYI, 2010; GOGAT and PANDIT, 2004; YIN et al, 2008). These radicals have an oxidation potential of 2.8 V, staying below only of Fluor, which is 3.03 V (Domenech et al, 2001)

The most common AOP methods are the combinations of ozone, hydrogen peroxide, ultrasound and ultraviolet (UV) radiation (YIN et al, 2008). Therefore, the oxidation power of

H₂O₂ can be enhanced via a number of treatment scenarios including O₃/H₂O₂, UV/H₂O₂ and H₂O₂/ultrasound process).

It is known and accepted that the first step in the UV/H₂O₂ process is the attack of the photon against the hydrogen peroxide molecule and the subsequent formation of hydroxyl radical •OH (Eskicioglu et al, 2008, Guimarães et al, 2008).

The Hydrogen peroxide absorbs ultraviolet light and uses the energy obtained in this way breaks down the bond O-O, which results in the formation of two radicals •OH (reaction 1) that act by degrading organic matter to form simpler compounds and reaction (2)



The combination of hydrogen peroxide with UV irradiation is one of the most easy to generate hydroxyl radicals, as the absorption of UV photons by the hydrogen peroxide does dissociate into hydroxyl radicals (reaction 1) which react quickly, nonselectively with most organic compounds (Rodríguez et al, 2000). The hydroxyl radicals attack organic pollutants and initiate a series of oxidation reactions that can lead to their total mineralization, eliminating toxic substances and increasing the biodegradability of organic pollutants (Zayas et al, 2007).

According to Matilainen and Sillanpää (2010), the rate of oxidation depends on some factors like: radical, oxygen, pollutant concentrations, pH, temperature, the presence of ions and the type of pollutant.

Recently, researchers have been discussing about the main problem to use the AOP's and one problem to solve is the high cost of reagents, such as ozone and hydrogen peroxide, as well as the electrical energy to generate UV radiation (ESPLUGA et al, 2002).

With this purpose, many researchers are discovering other ways to optimize their process using different kinds of techniques, and one of the most common methods is the Design of Experiments (DOE), that can be utilized to optimize process in many sectors of the economy.

The concept of Design of Experiment was first introduced in early 1920 in a small station of Agricultural Research in England, by a scientist called Sir. Ronald Fisher, where was shown as an experiment could be conducted in the presence of many natural conditions variables, such as: temperature, soil conditions and rainfall. (Franceschini and Macchietto, 2008; Kim and Kalb, 1996).

Since then, the method has been often utilized as a form to evaluate many kinds of processes, for example: (Davies, 1975) studied the advantages of statistical design in the investigation of titrimetric methods, using a factorial experiment design study of the ferric iron-ascorbic acid titration system. Following this thought (Vicente et al, 1998) proposed the use of a factorial experiment design and response surface methodology optimize biodiesel production, while (Vanclay, 2006) uses the design of experiments to evaluate inter- and intra-specific interactions in mixed plantings of forest trees.

For Besseris (2008) and De Souza et al., (2011), one of the most attractive and powerful methods of the design of experiment and that have been tested for single product characteristic improvement, has been the Taguchi method.

Taguchi experimental design have being extensively used as a tool for designing process or products to be robust to variations, being commonly used in many areas providing a systematic and efficient approach for finding the near optimum combination of design parameters for the product to be functional, exhibit a high level of performance, and robust to noise factors (Antony et al, 2006).

The steps of the Taguchi experimental design to (Barrado et al., 1996) and (Besseris, 2008) are:

Selection of the output variable, identify the factors that affect the output variable(s), select the appropriate orthogonal array, assign factors and interactions to the columns of the array, perform experiments, statistical analysis, determine the significant factors and confirmatory experiments, if it is necessary.

According to (Rosa et al, 2009) and (Chen et al, 2010) Taguchi's parameter design method normally selects an appropriate formulation of the S/N ratio and calculates the S/N ratio for each treatment. The S/N ratio is a logarithmic function used to optimize the process or product design, minimizing the variability. To minimize variability, the level of factor which produces the greatest value of S/N ratio must be chosen.

There are three types of S/N ratios: nominal the best, the larger the best, and the smaller the best.

In an attempt to improve the efficiency of the advanced oxidation processes (UV/H₂O₂) this work propose the use of the DOE technique, more specifically, the Taguchi's Method to reduce the total organic carbon (TOC), turbidity and chemical oxygen demand (COD) in dairy wastewater industry.

2. EXPERIMENTAL PROCEDURE

2.1 Effluent

The wastewater analyzed was collected in a company processing and bottling of UHT milk, located in Barra Mansa state of Rio de Janeiro, with an average generation of 300 m³ of effluent a day. The effluent was collected near the Biological Station, after going through the screening system.

Its pH ranged between 5.0 to 10.0 and after collected, it was adjusted to a level below then 2.0; it was stored in a cold chamber, 4 ° C, in order to maintain their physical and chemical characteristics throughout the research.

2.2 Design of Experiment

For the optimization of response variables (dependent variables) was performed statistical design for the experiments, according to the Taguchi method using an orthogonal array L16. The response variable was given depending on the analysis of COD, it is used to verify the efficiency of degradation of the effluent.

The Table 1 shows the levels chosen for each variable in treatment POA's. The levels for each factor were selected based on previously conducted test and respecting the physical boundaries that allowed their preparation and the number 1 represents a low level and the number 2 the high level.

Table 1 – Factors and their respective experiment levels

Column	Factors	Levels	
		1	2
A	pH	5,0	10,0
B	Temperature °C	25	35
C	Concentration H ₂ O ₂ (mM/L)	15	30
D	Radiation UV	with	without

A factorial design is represented by an orthogonal array Taguchi L16 was used for the treatment POA as shown on Table 3 and the symbols for each factor with their interaction is shown on the Table 2.

Table 2: Symbols used for factors and interactions

Symbol	Denomination
A	pH
B	Temperature °C
C	Concentration H ₂ O ₂ (mM/L)
D	Radiation UV
AB	Interaction pH x Temperature
AC	Interaction pH x Concentration H ₂ O ₂
AD	Interaction pH x Radiation UV

The allocation of each factor in the respective columns of orthogonal array was made using the linear graphs as can be seen in Figure 1. Each point in the figure represents a factor and each row represents an interaction. The numbers represent the columns where the factors and interactions are located.

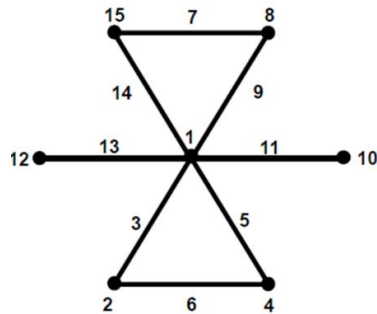


Figure 1: Linear graphs of the orthogonal array L16

Where ● Factor Column and ✕ Interaction Column

Table 3 – Factors and their respective interaction codified in the L16 Orthogonal Array

Run	1	8	9	10	11	12	13
	A	B	AB	C	AC	D	AD
1	1	1	1	1	1	1	1
2	1	2	2	2	2	2	2
3	1	1	1	1	1	2	2
4	1	2	2	2	2	1	1
5	1	1	1	2	2	1	1
6	1	2	2	1	1	2	2
7	1	1	1	2	2	2	2
8	1	2	2	1	1	1	1
9	2	1	2	1	2	1	2
10	2	2	1	2	1	2	1
11	2	1	2	1	2	2	1
12	2	2	1	2	1	1	2
13	2	1	2	2	1	1	2
14	2	2	1	1	2	2	1
15	2	1	2	2	1	2	1
16	2	2	1	1	2	1	2

2.3 Analytical Method

All of reagents utilized were analytically pure being used H₂O₂ 30% w/w in all photo-oxidizing procedures with NaOH and H₂SO₄, both of them in 0.5 eq/L concentration were used to obtain the desired pH through medium reaction initial.

The temperature and pH measure were made via pH meter pG2000 Gehaka, previously calibrated with tampon solution pH 4.0 and 7.0 and the photo-oxidizing process was performed in plug-flow reactor Germetec GPJ 463-1, emitting in 254 nm, with low pressure radiation source of 21W.

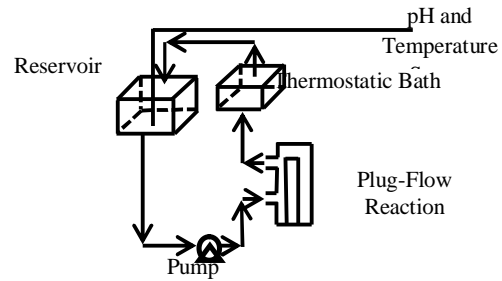


Figure 2- Laboratorial scheme. Adapted from (Guimarães et al, 2008)

At the end of each experiment, the system that has washing purposes, was filled with slightly acid solution and then re-circulated with distilled water. After discharged and re-circulated, the system was disassembled and the reactor filled with nitric acid 10% solution for cleaning purposes.

The temperature in each experiment was kept constant through Opherm DC1 thermostatic bath, $T_i \pm 2$ °C, where T_i stands for each experiment temperature, from 22 to 45 °C.

2.3.1 Turbidity

The determination of turbidity was performed in a turbidimeter model TB1000, with tungsten lamp, silicon photovoltaic detector, operating range from 0 to 1000 NTU and 2% accuracy.

2.3.2 Chemical Oxygen Demand

The determination of COD was carried out according to standard methodology, based on the closed reflux system, followed by determination Spectro-photometric. This analysis is based on chemical oxidation of organic matter by potassium dichromate, the high temperature and controlled, and in acid containing catalyst.

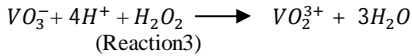
The analysis consists of adding 2.0 mL of a sample in a tube type Hach, containing 2.5 mL of 0.67% (w/v) of silver sulfate in sulfuric acid, inhibited by mercury sulphate 0.500 mL digester solution, potassium dichromate further 0.300 mL of distilled water, heated in a digester at 150 °C for two hours. After this time, the tube was cooled to room temperature, where was made the absorbance measure in a spectrophotometer SpectroFent, model 600, using a wavelength of 610 nm.

The absorbance obtained was inserted into a standard equation, developed with a solution of Potassium Phthalate, with a theoretical COD of 1000 mg O₂ L⁻¹, obtained under the same conditions.

To calculate the total expanded uncertainty (k = 2) the outcome of COD, it was considered as sources of uncertainty in the preparation of primary standards of Potassium Biphthalate and potassium dichromate, the standards of the analytical curve and its corresponding correlation, preparation of the digester tube with all reagents and samples and their repeatability, giving a

value of 4.57% (Peixoto et al, 2008). Thus, analytical results of COD values with uncertainty $\pm 4.57\%$ compared to the nominal value of COD of the effluent were considered zero.

The levels of the residual hydrogen peroxide were evaluated by spectrophotometric method, based on reaction with ammonium metavanadate. This method is based on the reaction between vanadate ions and hydrogen peroxide in acid medium (Reaction3), leading to a red color due to the formation of the cation peroxyvanadim:



The determination was performed by adding 1.0 mL sample to 2.0 mL ammonium vanadate solution of 0.1mol.L^{-1} , and the absorbance of this solution measured at a wavelength of 446 nm.

Every COD determinations were performed according to Standard Methodology (APHA, 1998).

3. RESULTS AND DISCUSSION

3.1. Chemical Oxygen Demand

All the samples were carried out following the same time of procedure, where the degradation rate were measured after passed 360 minutes of the beginning of the experiments for both variable response COD and Turbidity.

The results of the experiments were organized in a table, as can be shown at the Table 4.

Table 4: Reduction Percentage of COD after treatment

Chemical Oxygen Demand (COD)	
Run	%Reduction
1	66,69
2	0,00 ^(*)
3	54,40
4	71,96
5	52,03
6	41,77
7	14,60
8	58,80
9	8,26
10	0,00 ^(*)
11	0,00 ^(*)
12	9,02
13	22,44
14	0,00 ^(*)
15	0,54
16	26,27

^(*)COD Values considering the uncertainty in the analytic measures

The results of the effects over the mean responses percentage reduction of chemical oxygen demand are shown in the Figure 3.

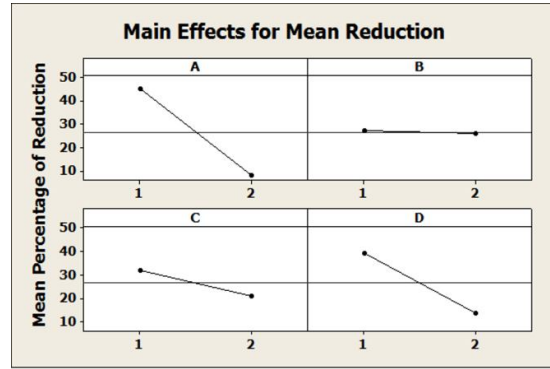


Figure 3: Effect of the factors over the COD Reduction

The Figure 3 shows more significant influence of pH followed by radiation UV and little or no significant influence were observed for H₂O₂ Concentration and Temperature. Considering the variable pH, the average of the reduction percentage when pH is equal 5 is 45,03, while that for the pH is equal 10 the average was 8,32%. It shows that the mean of the COD reduction for the level 1 was more than five times greater than for the level 2, proving that for to obtain the maximum COD reduction, this variable must be adjusted at the pH=5.

At the same way, the second controllable factor most important was the UV radiation, where can be found a average for the COD reduction in presence of UV radiation of 39,43%, being almost three times higher than the value found out without the presence of UV radiation, that was 13,91%, considered low compared with the other.

For the factor Temperature, the average of the difference between the effects at higher level and the lower level are not so significant comparing with the UV radiation and pH, showing that temperature, both at the higher level and lower level, the average of the variable response is almost same.

For the concentration of H₂O₂, the behavior showed in the Figure 3, which showed be more efficient when adjust the H₂O₂ Concentration at the level 1. It can be explained due to not necessarily use of higher concentrations of H₂O₂ favors the kinetics of the reaction. After the reaction starts, the steps of propagation can be prevented by the excess of hydrogen peroxide acting. When H₂O₂ is in excess there may be a parallel reaction, which decreases the rate of degradation organic matter (reaction 4). This is because the excess H₂O₂ acts capturing radical's hydroxyl (Guimarães et al, 2008).



Figure 4 shows the effect of the interaction over the mean response, where there is no significance of the factor A with the factors B, C and D.

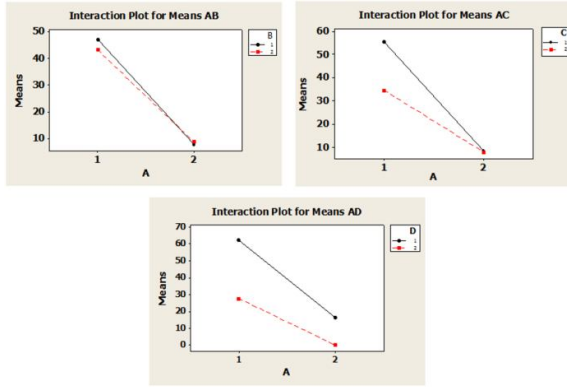


Figure 4: Effect of the interaction AB, AC and AD over the response COD Reduction

Table 5 shows ANOVA of the factors involved in the exploratory investigation to take a conclusion about influence of the factors in the process.

Table 5: ANOVA of factors and interactions for COD

Factors	DF	Seq SS	AdjMs	F	P
A	1	5392,0	5392,0	29,94	0,001
B	1	7,8	7,8	0,04	0,841
C	1	458,0	458,0	2,54	0,149
D	1	2605,1	2605,1	14,47	0,005
AB	1	23,1	23,1	0,13	0,729
AC	1	405,4	405,4	2,25	0,172
AD	1	335,4	335,4	1,86	0,209
Residual	8	1140,6	180,07		
Error					

The Table 5 shows that the factors pH and UV Radiation (D) were only significant in the process, proving that there is no influence of the factors Temperature and H₂O₂ Concentration. Thus, the best condition of adjustment for the controllable factors, deduced from the analysis of the mean response in order to have a higher COD percentage of reduction is: The pH must be adjusted to 5.0 that is the level 1, for the Temperature factor (B) and Concentration of H₂O₂, the mean of the factor when it is at the level 1 is almost same of the level 2, for this reason we can choose one level that can be more economic in the process or do the same experiment for both cases and compare both of them to verify what is more profitable, and finally the process must be carried out in presence of UV Radiation to maximize the percentage of COD reduction.

With the two factors most significant to this process was possible build a surface to know the behavior or evolution of the variable response, where it can be seen at the Figure 5.

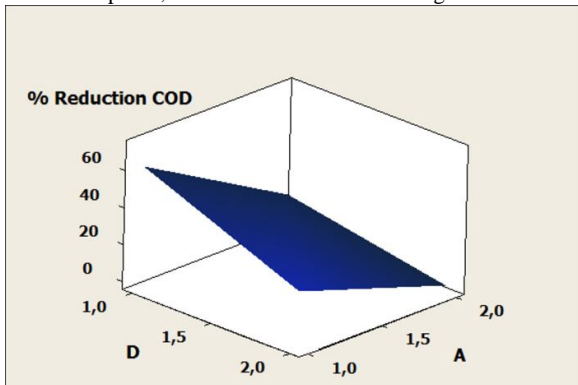


Figure 5: Surface Plot for the COD Reduction vs factor A and D

The Figure 5 shows the surface plot for the reduction percentage of COD considering the factor A and B. Analyzing the behavior of the variable response depending on both factors, the conclusion is that the reduction percentage of COD grows up as the pH decrease from 10 to 5 in presence of UV Radiation.

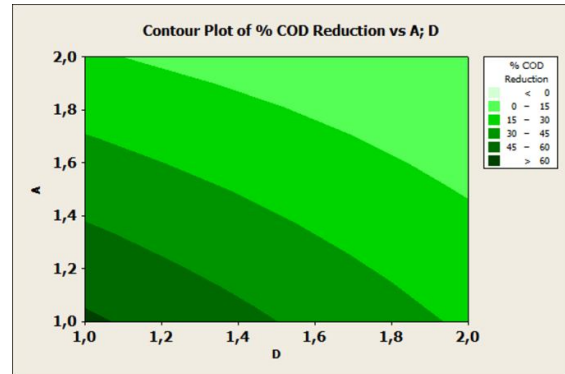


Figure 6: Contour Plot for the COD Reduction vs factor A and D

Proving what was concluded about the Figure 5, the Figure 6 represents the contour plot for the variable response depending on the variable A and B. The area in dark shade of blue is the area with the major reduction percentage, while the dark shade of green is the area with the lower reduction percentage of COD, then when both factors are in the high level, the reduction percentage of COD is minimum and when both factors are at the level 1, the reduction will be maximum.

3.2. Turbidity

For the turbidity analysis, was used the same thought of the COD reduction. After accomplished the orthogonal array the Table 6 was made with the results of the experiments.

Table 6: Reduction Percentage of Turbidity after treatment

Run	Reduction%
1	92
2	94,8
3	77,8
4	99,01
5	93,1
6	94,32
7	36,27
8	97,38
9	42,03
10	38,99
11	60,21
12	73,96
13	67
14	45,27
15	66,71
16	40,23

Looking at the Table 6 and comparing the results with the Table 4 is easy to find that this AOP is more efficient talking about Turbidity, which is shown by the percentage of reduction. In the Table 6 for example, the maximum reduction of turbidity was 99,1%, while that in the Table 4, the highest value for the percentage of COD reduction was 71,96%, both for the condition where pH is 5, Temperature is 35°C, H₂O₂ Concentration in 30 mM/L in presence of UV Radiation.

At the same way, the Figure 7 and Figure 8 show the effect of the factors and interactions over the variable response.

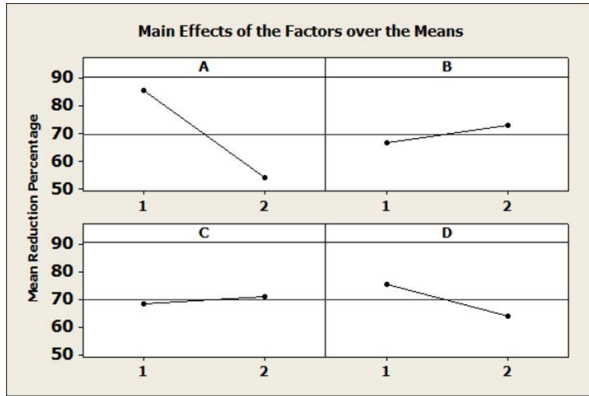


Figure 7: Effect of the factors in the Turbidity Reduction

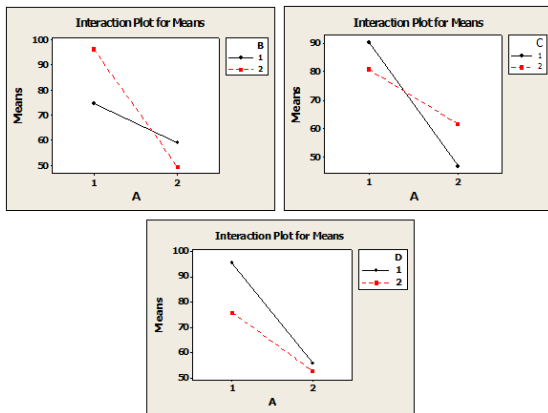


Figure 8: Effect of the interaction in the Turbidity Reduction

Table 7: ANOVA of factors and interactions for Turbidity

Factors	DF	Seq SS	AdjMs	F	P
A	1	3103,047	3103,047	13,72995	0,00599
B	1	28,409	28,409	0,12570	0,73209
C	1	10,989	10,989	0,04862	0,83099
D	1	868,186	868,186	3,84144	0,08566
AB	1	1428,084	1428,084	6,31880	0,03616
AC	1	252,651	252,651	1,11790	0,32125
AD	1	94,770	94,770	0,41933	0,53541
Error	8	1808,046	226,006		

In the Figure 7 is shown that the factor A is the most important causing the higher effects over the response when it is changed from the low level to the high level, while that for the Figure 8 is shown the influence of the interaction AB and AC, but making the analysis with the Table 7, only the interaction AB is influent in the process, and the same way, the factor A is the factor that causes more influence in the results.

The curiosity in this case is that, the factor B is not one factor influent in the process when it is alone, but when it in presence of the variable A causes great influence showing to be a very significant interaction reaching a reduction average of 93,78% when the Temperature is at the level 2 and the pH is at the level 1.

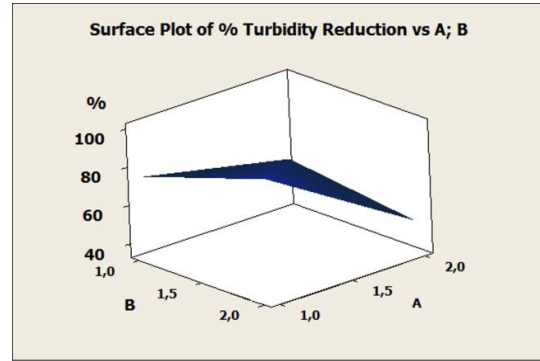


Figure 9: Surface Plot for the Turbidity Reduction vs factor A and B

The Figure 9 proves with the non-planar surface the influence of the interaction AB over the variable response Turbidity, where the behavior of the both variables can be seen in the Figure 10.

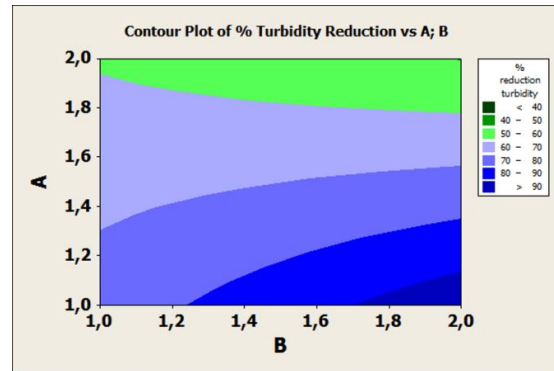


Figure 10: Contour Plot for the COD Reduction vs factor A and B

The Figure 10 represents the contour plot for the turbidity reduction depending on the variable pH and temperature, where the maximum reduction percentage is reached when the pH is settled at the level 1 and the temperature at the level 2.

Finally, the best condition of adjustment for the controllable factors and increase the reduction percentage of turbidity is adjust the factor A (pH) at the level 1, the factor B (Temperature) and Concentration of H₂O₂ factor C, must be done the same that was proposed for COD, and finally the process must be carried out in presence of UV Radiation to maximize the percentage of COD reduction.

Comparing both cases, the conclusions were the same, showing that in this process there are two dominant factors, which are pH and UV radiation, being these the factors really relevant when it comes to advanced oxidation process in dairy wastewater.

4. CONCLUSION

The oxidation process showed an estimated potential to reduce the organic load which represents a viable alternative to be applied as a pre-treatment of effluent.

The use of an orthogonal array by Taguchi has helped in the choice of factors and parameters of the test application, set the sequence of experiments and the properties measured, after which the experiments were able to identify the influence of each factor and interactions over the variable responses turbidity and chemical oxygen demand.

The best condition of adjustment for the controllable factors and increase the reduction percentage of Chemical Oxygen Demand is adjust the pH to 5.0 that is the level 1, the Temperature factor (B) and Concentration of H₂O₂, the mean of the factor when it is at the level 1 is almost same of the level 2, for this reason we can choose one level that can be more economic in the process or do the same experiment for both cases and compare both of them to verify what is more profitable, and finally the process must be carried out in presence of UV Radiation to maximize the percentage of COD reduction.

The conclusions for turbidity were the same comparing with the chemical oxygen demand, showing that in this process there are two dominant factors, which are pH and UV radiation.

Finally, we may conclude that the methods were successful, because the objective proposed in the study was well conducted step by step by each method, it is possible to determine the best arrangement of factors in this process.

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