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Optimization of the Coagulation-flocculation Process Using Ferric Chloride and Phosphate for the Reduction of Contaminants in the Slaughterhouses Wastewater

A.W. Pilco-Nuñez[1]*; K.M. Zevallos Palacio[2], A.Z. García Campos[3], J.A. Flores Salinas[4], J.A. Montaño Pisfil[5], R.E. Solis Farfan[6], S.A Trujillo Perez[7], A. Arroyo-Paz[8], J.A. Poma García[9]

[1,4]*Universidad Nacional de Ingeniería, Faculty of Chemical and Textile Engineering, Av. Túpac Amaru 210, Lima, Perú. E-mail: apilco@uni.edu.pe*

[2,3]*Universidad Nacional del Callao, Faculty of Environmental Engineering and Natural Resources, Av. Juan Pablo II 306, Bellavista, Callao, Perú.*

[5,6]*Universidad Nacional del Callao, Faculty of Electrical and Electronic Engineering, Av. Juan Pablo II 306, Bellavista, Callao, Perú.*

[7]*Universidad Nacional del Callao, Faculty of Chemical Engineering, Av. Juan Pablo II 306, Bellavista, Callao, Perú.*

[8]*Universidad Tecnológica del Perú, Faculty of Engineering, Av. Tacna y Arica 160, Arequipa, Perú.*

[9]*Universidad Cesar Vallejo, Faculty of Engineering and Architecture, Av. Larco 1770, Trujillo 13001, Perú.*

Abstracts: In this work, the coagulation-flocculation process was optimized using ferric chloride and phosphate for the reduction of pollutants in the wastewater from the Conchucos S.A., Lima. Parameters were measured in percentage reduction of chemical oxygen demand (COD), turbidity (NTU) and total phosphorus (PT mg/L), adding ferric chloride as coagulant and potassium dihydrogen phosphate as flocculant. The effects of four independent variables were investigated: ferric chloride dose (500-700 ppm), phosphate dose (700-900 mg/L), fast agitation speed (250-320 rpm) and slow agitation speed (90-100 rpm). The experimental data were optimized by the response surface method using a central composite design. The results show that the statistical models obtained F-values of 3.33, 4.27 and 4.16 for the percent reduction of COD, turbidity and total phosphorus, respectively. Furthermore, the statistical models developed to predict the responses were confirmed by significant probability values ($p<0.05$). On the fit of the models, an R2 of 0.61, 0.66 and 0.67 are shown for COD, turbidity and total phosphorus percentage, respectively. The optimum conditions were found experimentally at 700 ppm of ferric chloride dose, 900 ppm of phosphate, 320 rpm fast speed and 100 rpm for a reduction of 75.46% of COD, 83.47% of turbidity and 44.08% of total phosphorus presenting a desirability of 0.7.

Keywords: Ferric Chloride, Coagulation-flocculation, Wastewater, Slaughterhouse, Optimization.

1. INTRODUCTION

The growth of the world's population has led to an increase in improper wastewater discharges, increasing freshwater pollution, especially in developing countries [1]. For this reason, water and wastewater treatment is important for the continued development of society. Being, increasingly stringent standards for effluent discharge worldwide [2].

Slaughterhouses slaughter different animals such as cattle, pork and poultry, mostly for direct marketing of meat. The process depends on the type of animal slaughtered, with general processes such as slaughter, bleeding, evisceration, refrigeration and their respective by-products. The wastewater generated depends greatly on the type of slaughter of different animals, obtaining values with high concentrations of turbidity, phosphates, and electrical conductivity [2].

The wastewater generated by slaughterhouses includes water from the different processes such as slaughtering, plucking or removal of skins, evisceration and trimming, washing, disinfection and cooling, so it is usually composed of organic matter including proteins, blood residues, fats etc., [3]. Therefore, direct disposal of untreated slaughterhouse wastewater into the environment is associated with the occurrence of the eutrophication phenomenon, which affects the aquatic ecosystem and also corresponds to infection points affecting the health of vulnerable families who often have access to contaminated water [4].

Some research works point out different wastewater treatment alternatives ranging from physicochemical, biological and even electrochemical processes in the application of new advanced methods, as well as chemical coagulation coupled to Fenton processes [5]. Current commercial treatment technologies are based on biological and physicochemical processes that have high investment and operating costs [6]. Of all these, the physical-chemical treatment presents advantages that correspond to its simplicity, rather low chemical doses, short process time and even with its real possibility of being applied at pilot and commercial scale [7]. On the other hand, coagulation appears to be a low-cost treatment that involves the use of coagulant-flocculants with high efficiency in the removal of pollutants [8].

In this sense, the present research work optimized the coagulation-flocculation process using ferric chloride and potassium dihydrogen phosphate for the reduction of pollutants in the wastewater from the Conchucos S.A. Camal.

2. MATERIEL AND METHODS

The research design refers to a factorial design composed by the response surface method between groups: fast agitation speed, slow agitation speed, phosphate dose and ferric chloride dose; and the response parameters measured with 2 repetitions: the indicators of electrical conductivity, pH, COD and total phosphorus. This analytical model responds to a mixed factorial analysis of variance (ANOVA) forming an experimental arrangement. To carry out the research, the stages of the research design and its experimental development are shown in Fig. **1**.

Figure 1: characterization of the slaughterhouse wastewater.

2.1. Wastewater Characterization

The initial characterization of the slaughterhouse wastewater was performed during the operating hours of the slaughterhouse from July to November 2022. The parameters analyzed were Oils and Fats (mg/L), Turbidity (NTU), Biochemical Oxygen Demand (mg BOD5/L), Chemical Oxygen Demand (mgO2/L), Conductivity (mS/cm), pH (pH unit), Total Phosphorus (mg/L). The wastewater was collected in three 40L drums as shown in Figure **1** and transferred to the laboratory of the Environmental Engineering Faculty of the Universidad Nacional del Callao.

2.2. Test Jar

After collecting the wastewater, for the beginning of the investigation, the experiments were carried out in the jar test equipment (Figure **2**). This equipment is located in the laboratory of the Faculty of Environmental Engineering and Natural Resources of the Universidad Nacional del Callao. Subsequently, $FeCl_3$ and phosphate were prepared in different dosages.

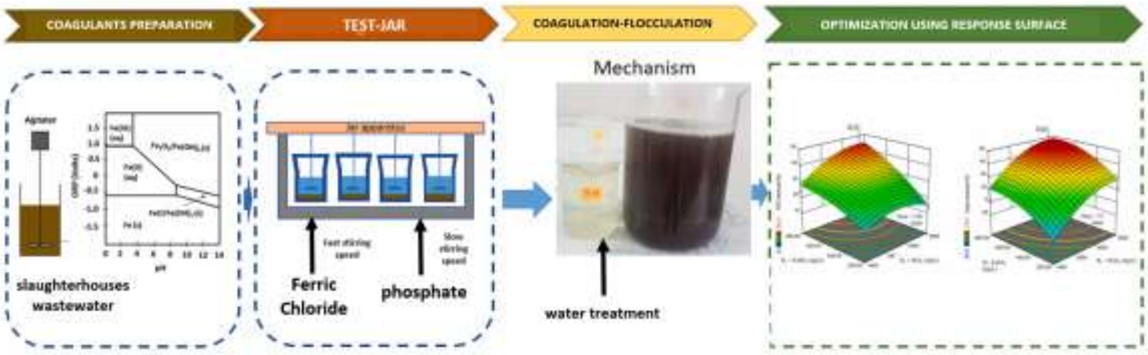

Figure 2: Flow chart for experimentation

2.3. Coagulant-flocculant selection

2.3.1. Ferric chloride salt ($FeCl_3$)

Ferric chloride is used in drinking water and wastewater treatment plants as an excellent coagulant, widely used for its high efficiency in the removal of organics and heavy metals; it is also used as an etching agent in lithography and photography, catalyst, mordant, oxidizing agent, disinfectant, pigment and feed additive.

2.3.2. Potassium dihydrogen phosphate salt (KH_2PO_4)

It is a highly soluble source of phosphorus and potassium. This agent made it possible to generate phosphate ions to enhance the coagulation-flocculation of ferric chloride with the polymerized wastewater.

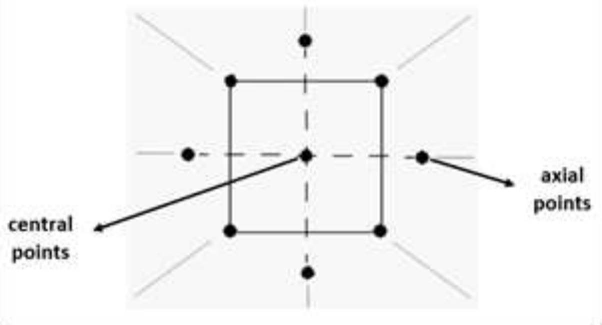

Figure 3: Graphical reference of the central composite design

2.4. Response Surface Model (RSM)

The Composite Central Design (CCD) response surface is the most commonly used design. Composite central designs are a factorial or fractional factorial design with central points, extended with a group of axial points (also called star points) that allow estimation of curvature (Figure **3**).

The factorial design allows comparing the effect of the manipulated factors on the different response parameters and obtaining a statistically significant difference. Analysis of variance (ANOVA) was used for data analysis and processing. Equation 1 shows the regression model used.

$$y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} {X_1}^2 + \beta_{12} {X_2}^2 + \beta_{23} {X_3}^2 \quad (1)$$

Table 1: Factors and their levels for the response surface method-composite central design

Factor	Unit	Levels	
		Low (-1)	High (+1)
Ferric chloride dosage	ppm	500	700
Dose of potassium dihydrogen phosphate	ppm	700	900
Fast stirring speed	rpm	250	320
Slow stirring speed	rpm	90	100

Where: y is a response, X_1 and X_2 are manipulated factors and β_0, β_1, β_{12} are unknown parameters.

For the validation of the model, the acceptability analysis was performed where the result is a function of the F-value and p-value and the model fit is a function of the R_2, adjusted R_2 and predicted R_2. For this research a central composite design was used consisting of a full factorial design (or a fraction of it) 24, where the level of the factors is coded to the usual -1, +1 standard values (this is called the factorial portion of the design) with 1 replicate, with 4 central points and 8 axial points at a distance α from the center of the design (this portion is called the axial portion of the design); developing 44 runs in the jar test (Table **1**).

This method will allow the effects of the 4 factors to be determined. The independent factors and their levels applied in the design are presented in Table **2**. For the ranges chosen for the factors, they were adjusted to the nearest possibility.

2.5. Desirability model to optimize

As part of the multiple response method, the desirability model will be used to obtain a concurrent objective function to represent all the transformed responses by combining the desired ranges for each response using Equation (2):

where, D, di and n are the desirability function, each individual response and the total number of responses, respectively. For simultaneous optimization, each response requires high and low values. Otherwise, if any response is found outside its desirability range, the overall desirability becomes equal to zero.

Table 2: Experimental arrangement for the model

N°	Factor 1	Factor2	Factor 3	Factor 4
	Iron Chloride	Phosphate	Fast speed	Slow speed
	ppm	ppm	Min	rpm
1	500	900	250	80
2	500	700	250	100
3	600	800	355	90
4	500	700	250	80
5	500	700	320	80
6	600	800	215	90
7	500	900	320	100
8	700	700	250	80
9	700	900	320	80
10	600	600	285	90
11	500	700	250	100
12	600	800	285	110
13	700	700	250	100
14	500	900	320	80

15	500	700	250	80
16	600	800	285	90
17	700	900	320	80
18	600	800	285	90
19	700	900	250	100
20	600	800	285	70
21	700	700	320	100
22	500	700	320	100
23	500	900	250	80
24	800	800	285	90
25	500	900	250	100
26	700	700	320	80
27	700	700	320	80
28	700	700	320	100
29	400	800	285	90
30	500	900	320	100
31	600	1000	285	90
32	700	900	250	100
33	700	900	320	100
34	700	700	250	80
35	700	900	320	100
36	600	800	285	90
37	700	700	250	100
38	700	900	250	80
39	600	800	285	90
40	700	900	250	80
41	500	900	250	100
42	500	700	320	100
43	500	900	320	80
44	500	700	320	80

TABLE 3: PHYSICO-CHEMICAL CHARACTERISTICS OF FEEDLOT EFFLUENTS

PARAMETERS	UNIT	RESULTS
OILS AND FATS	MG/L	25
CONDUCTIVITY	MS/CM	2.12
BIOCHEMICAL OXYGEN DEMAND	MGBOD /L5	759
CHEMICAL OXYGEN DEMAND	MGO /L2	4005.4
PH	PH UNIT	6.88
TURBIDITY	NTU	485
TOTAL PHOSPHORUS	MG/L	244.5

3. RESULTS

3.1. Characterization of the farm's wastewater

According to the analyses carried out on the wastewater from the Conchucos S.A. slaughterhouse, the following levels of contaminant removal were found for the physicochemical characteristics evaluated before being treated with the addition of ferric chloride and phosphate (Table **3**).

Table 4: Contaminant removal

Parameters	Average
%DQO	49.078 +/- 7.24
%Turbidity	75.89 +/- 5.65
Total Phosphorus	41.85+/- 9.20
pH	5.87+/- 0.37
Conductivity	6.74+/- 0.38

3.2. Coagulation-flocculation treatment

After treatment using ferric chloride and phosphate, the results show an average removal of 49.07% of contaminants represented by COD removal, 75.89% turbidity removal and 41.85% of total phosphorus. The treatment also slightly acidified the wastewater and increased the conductivity (Table **4**).

3.3. ANOVA analysis

The significant main and interaction effects of the factors influencing the parameters of responses in the litter water treatment were analyzed by the ANOVA method shown in Table **5**, Table **6** and Table **7**.

The significance of each model parameter was evaluated using the F -value test and p-values for each variable, including linear, interaction and quadratic interaction. As shown in Table **5**, Table **6** and Table **7** p-values less than 0.05 identify the model coefficients as significant.

As shown in Table **5**, Table **6** and Table **7**, the quadratic models developed for each response were found to be significant. F-values of 3.33, 4.27 and 4.16 were obtained for the percent removal of COD, Turbidity and total phosphorus, respectively. Furthermore, the accuracy of the statistical models developed to predict the responses was confirmed by small probability values ($p<0.05$). However, some terms of the models were also found to be non-significant ($p>0.10$).

Table 4: ANOVA for the quadratic response surface model %COD removed

Source	Sum of squares	GL	F-value	p-value	R2	R2 adjusted
$\%DQO=+38.20+1.64A+1.13B+0.7240C+0.1092D-0.9987AB+4.00AC-0.5110AD-2.83BC+1.10BD-0.8426CD+4.77A^2+3.12B^2+0.4026C^2+0.7443D^2$						
Model	2440.69	14	3.33	0.003	0.61	0.43
A-Doses of Ferric Chloride	81.58	1	1.56	0.2219		
B-Phosphate dosage	39.17	1	0.7481	0.3942		
C-Fast speed	15.98	1	0.3052	0.5849		
D-Slow speed	0.7272	1	0.0139	0.907		
AB	31.91	1	0.6096	0.4413		
AC	512.62	1	9.79	0.004		
AD	33.43	1	0.6385	0.4308		
BC	256.67	1	4.9	0.0348		
BD	153.81	1	2.94	0.0972		

CD	90.88	1	1.74	0.198		
A^2	560.47	1	10.71	0.0028		
B^2	239.26	1	4.57	0.0411		
C^2	3.99	1	0.0762	0.7845		
D^2	218.19	1	4.17	0.0504		
Residual	1518.25	29				
Lack of adjustment	316.51	10	0.5004	0.869		
Error	1201.74	19				
Total	3958.94	43				

3.3.1. COD Removal

Table **5** shows that for %DQO removed the influent iron chloride dosage concentration (A), phosphate dosage (B), fast stirring speed (C), slow stirring speed (D) showed a non-significant effect, while the quadratic of iron chloride dosage (A2) and phosphate dosage (B_2) do have significant effects. Also, the interaction between iron chloride dosage with fast speed (AC) and phosphate dosage with fast speed (BC) showed a significant effect.

3.3.2. Turbidity Removal

Table **6** shows that for the percentage of turbidity removed the influent iron chloride dosage concentration (A), phosphate dosage (B), slow stirring speed (D) showed a non-significant effect, while fast stirring speed (C), iron chloride dosage with fast speed (AC) and the interaction between fast and slow speeds showed a significant effect.

Table 6: ANOVA for the quadratic response surface model Turbidity removed

Source	Sum of squares	GL	F-value	p-value	R^2	R^2 adjusted
$\%Turbidity = +73.01 - 1.58A + 1.12B + 4.55C + 0.0399D + 0.8921AB + 4.43AC - 0.6199AD - 1.40BC - 0.1912BD + 1.09CD - 1.99A^2 + 1.74B^2 + 2.11C^2 + 0.2624D^2$						
Model	1908.79	14	4.27	0.0005	0.67	0.51
A-Doses of Ferric Chloride	75.82	1	2.37	0.1342		
B-Phosphate dosage	38.5	1	1.21	0.2812		
C-Fast speed	629.8	1	19.72	0.0001		
D-Slow speed	0.0969	1	0.003	0.9565		
AB	25.47	1	0.7974	0.3792		
AC	628.58	1	19.68	0.0001		
AD	49.19	1	1.54	0.2246		
BC	62.57	1	1.96	0.1722		
BD	4.68	1	0.1464	0.7047		
CD	152.12	1	4.76	0.0373		
A^2	97.43	1	3.05	0.0913		
B^2	74.73	1	2.34	0.1369		
C^2	110.06	1	3.45	0.0736		
D^2	27.12	1	0.8492	0.3644		
Residual	926.21	29				
Lack of adjustment	271.79	10	0.7891	0.6398		

Error	654.42	19				
Total	2835	43				

3.3.3. Total Phosphorus Removal

Table **7** shows that for the percentage of Total Phosphorus removed, the influent iron chloride dose concentration (A), its quadratic (A2) and the phosphate dose square (B_2), showed a significant effect.

The R^2 are significant parameters for the model of COD, Turbidity and Total phosphorus (>60%). While Adj-R^2 presents a representativeness in the range of 50%. Pred-R^2 shows the variation of the predicted data. In this research, the Pred-R^2 did not reach expected values (for COD, turbidity and Total Phosphorus removal). In addition, all coefficients were below the satisfactory level.

Table 7: ANOVA for the quadratic response surface model for total phosphorus removal

Source	Sum of squares	GL	F-value	p-value	R2	R2 adjusted
$\%PT=+58.42+6.70A-4.42B-0.9678\,C-0.9271D+1.59AB-2.66AC+0.5872AD+2.25BC-0.1879\,BD+0.5811CD-6.76A^2-7.51B^2-2.04C^2-0.5776D^2$						
Model	29486.22	14	4.16	0.0006	0.66	0.55
A-Doses of Ferric Chloride	8183.24	1	16.18	0.0004		
B-Phosphate dosage	3556.9	1	7.03	0.0128		
C-Fast speed	170.59	1	0.3373	0.5659		
D-Slow speed	313.03	1	0.6189	0.4378		
AB	484.19	1	0.9573	0.3359		
AC	1349.15	1	2.67	0.1132		
AD	263.77	1	0.5215	0.476		
BC	964.77	1	1.91	0.1778		
BD	27	1	0.0534	0.8189		
CD	258.25	1	0.5106	0.4806		
A^2	6723.79	1	13.29	0.001		
B^2	8289.25	1	16.39	0.0004		
C^2	611.15	1	1.21	0.2807		
D^2	785.05	1	1.55	0.2228		
Residual	14667.01	29				
Lack of adjustment	3675.95	10	0.6355	0.7664		
Error	10991.05	19				
Total	44153.23	43				

4. ANALYSIS OF THE EFFECTS

4.1. Effect of Ferric Chloride dosage

Figure **4** shows the significant effects of ferric chloride/ on the percentage of COD removed, Turbidity and Total Phosphorus. Ferric chloride is the most significant factor during treatment. According to Figure **4** the % COD removal remained constant as ferric chloride increased. The highest % COD removal efficiency achieved was over 40% and 70% for turbidity. Figure **5** shows that as ferric chloride increases the % total phosphorus removed increases.

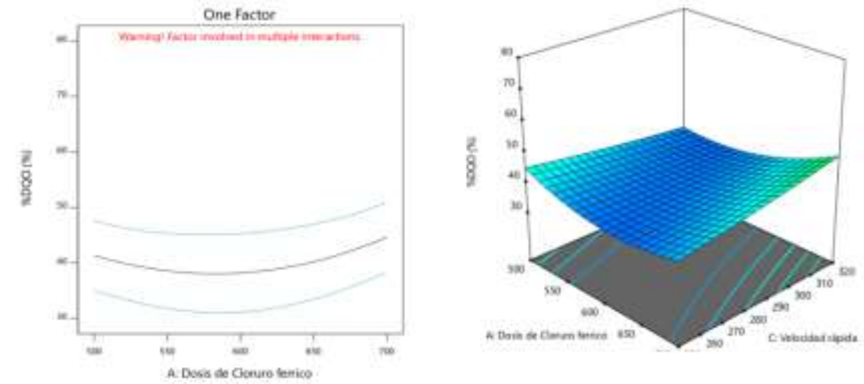

Figure 4: Plots of significant effects of ferric chloride dose on DQO

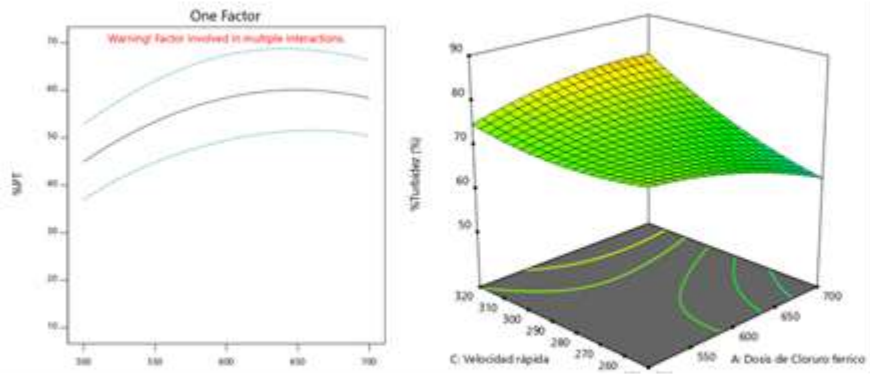

Figure 5: Plots of significant effects of ferric chloride dose on Turbidity and total phosphorus.

4.2. Effect of Phosphate dose

Figure **6** shows the significant effects of phosphate dosage on the %COD removed, the % Total Phosphorus removal. The highest %TP removal efficiency is generated at a dose of 750 ppm.

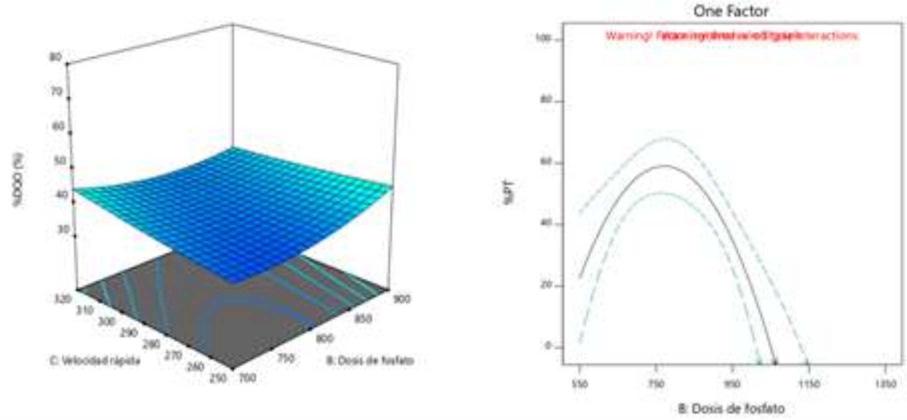

Figure 6: Plots of significant effects of phosphate dose on %COD and total phosphorus

4.3. Optimization

After obtaining the significance of the response parameters, the desirability function was proposed to optimize the multiple responses. This desirability function allows conditioning the different variables to obtain an optimal result according to the estimated adjustment models. For indicators such as % COD, % turbidity, % total phosphorus removed, the aim is to maximize the percentage, while pH and conductivity are not conditioned since they do not meet any significant model (Table **8**).

Table 8: Contaminant removal

Variables	Indicators	Condition	Result
Independent	Ferric chloride dosage	500-700	700
	Phosphate Dosage	700-900	900
	Fast stirring speed	250-320	320
	Slow stirring speed	90-100	100
	%DQO	Maximize	50.75%
Dependents	%Turbidity	Maximize	83.47%
	Total Phosphorus	Maximize	44.08%

Figure **7** shows the numerical optimum points for the response variables such as % COD, % turbidity and % total phosphorus.

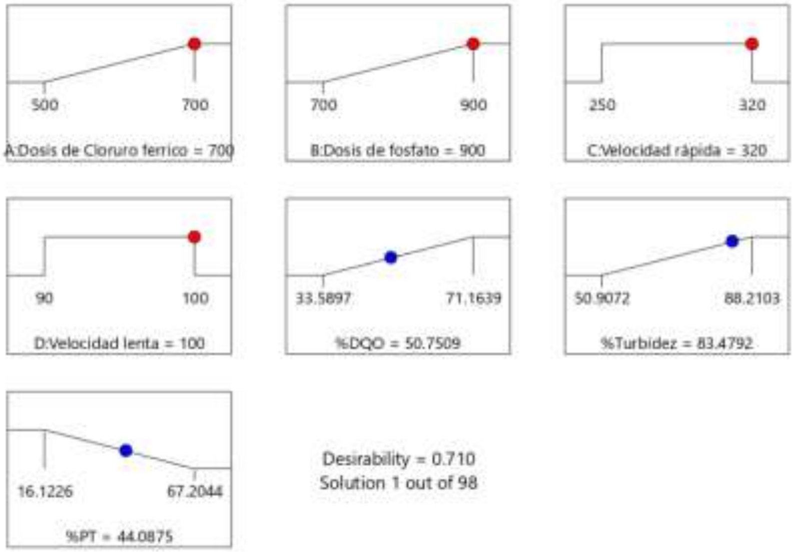

Figure 7: Desirability response plots.

5. DISCUSSION OF RESULTS

The results should be explained because the iron chloride coagulant maintains a relatively low efficiency of 49.07% in COD removal and 75.89% in turbidity removal. This observation is in agreement with the study of [9] where with a Box-Behnken surface model design using de ClFe3 they obtained COD and Turbidity efficiencies of 72.64% and 60.17%, respectively, treating slaughterhouse waters with efficiencies similar to this study. However, in the study by [10] treating by oxidation processes showed better performance was obtained for COD removal greater equal to 71.6% and turbidity of 85-100%.

[11] suggested a saturation of the medium with iron chloride to show high suspended solids removal (98%). Although, iron chloride was not used in this study, so for this research the optimum region could not be reached. Ferric chloride has been used and has been shown to be the most significant factor during treatment. Other studies of coagulation/flocculation of industrial waters such as [12] reported low efficiencies of ferric chloride (FeCl3) (less than 64%) compared to other coagulants such as aluminum sulfate (alum).

Additional advantages of ferric chloride for coagulation treatments include the removal of phosphorus compounds [13]. In this research the effect of phosphate as a flocculant was not significant. The improvement of the process was expected according to the literature reviewed the high reaction between iron and phosphorus, but the addition of ferric chloride to wastewater produces iron and chloride ions with positively charged iron ions that neutralize the negatively charged colloidal particles leading to coagulation and so the addition of phosphate on the clots does not add the necessary molecular weight to improve flocculation.

The application of statistical optimization models allowed to have a large reach of the flocculation process by dosing. Other optimization studies such as [14], who used a response surface methodology (RSM) for process optimization and maximize the treatment of slaughterhouse wastewater showed an R^2 adjustment greater than 70%. Similarly, [15] used a screening experimental design for the development of photo-Fenton process that is very effective for treating synthetic textile wastewater, obtaining 86% COD removal, with relatively high fit values. For this research the central composite model delimited an optimal working region with low R^2.

CONCLUSION

A physicochemical treatment of wastewater from the Conchucos slaughterhouse was carried out using the coagulation/flocculation process with ferric chloride and phosphate to achieve the reduction of COD, turbidity and total phosphorus. It was concluded that at least some of the effects of the operating parameters, ferric chloride and phosphate dosage are significant in the reduction of pollutants. The results show that the statistical models obtained F-values of 3.33, 4.27 and 4.16 for the percent reduction of COD, turbidity and total phosphorus, respectively. Furthermore, the statistical models developed to predict the responses were confirmed by significant probability values ($p<0.05$). On the fit of the models, an R^2 of 0.61, 0.66 and 0.67 are shown for COD, turbidity and total phosphorus percentage, respectively. The optimum conditions were found experimentally at 700 ppm of ferric chloride dose, 900 ppm of phosphate, 320 rpm fast speed and 100 rpm for a reduction of 75.46% of COD, 83.47% of turbidity and 44.08% of total phosphorus representing a desirability of 0.7.

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