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Optimization of the treatment of a real textile wastewater by coagulation–flocculation processes using central composite design

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ABSTRACT

The treatment of real textile wastewater by coagulation–flocculation (CF) using ferric chloride as coagulant and polymer as flocculant was optimized with the central composite design (CCD) based on response surfaces methodology. The independent variables considered were pH, coagulant, and flocculant dose. Their effects on the treatment were evaluated by the analysis of variance. The models are validated by the comparison between the predicted and experimental values, with a coefficient of determination reached a value above 93%, for all responses; removal efficiency of chemical oxygen demand (COD), biological oxygen demand (BOD₅), turbidity, and color. The graphical representations of the models in the space of the variables enable us to determine the optimal conditions, which are pH = 8.1, a dose of FeCl₃ = 0.8 g/L and a dose of flocculant = 2.6 mL/L. Under these conditions, the removal efficiency of COD, turbidity, color, and BOD₅ achieved 95%, 92%, 96%, and 60% respectively.

Keywords: Real textile wastewater; Coagulation–flocculation; optimization; Central composite design; Surface response methodology; Turbidity; COD; BOD₅; Color

1. Introduction

The textile industry is a big producer of pollution because of the big volume of water consumed and generated in dyeing operations, printing, and finishing [1]. Moreover, the composition of their effluents rich with organic and mineral matter (dye, heavy metals surfactant, and recalcitrant compounds) can affect aquatic life, environment, and humans [2]. For this reason, this wastewater should be treated before its evacuation or reuse. However,

the treatment of this wastewater is very complex; because it is characterized by intense color, high values of chemical oxygen demand (COD), turbidity, biological oxygen demand (BOD₅), toxicity, suspended matter, etc. [3].

The treatment of textile wastewater is becoming a big environmental concern. Thus, various techniques have been tested in this field such as Fenton reagent, advanced oxidation, wet oxidation, granular activated carbons, adsorption, filtration, and biological treatment [4,5]. However, those methods are not practical in terms of cost manipulation

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and sometimes they produce a toxic sludge. Coagulation–flocculation is considered one of the most attractive and favorable techniques because of its low cost, easy operation, and high efficiency [6].

The main purpose of coagulation is to destabilize the particles in suspension and facilitate their agglomeration. In practice, this process is characterized by the injection and dispersion of coagulants. The flocculation aims to promote with a slow mixture, the contacts between the destabilized particles. These particles form a floc eventually removed by decantation [7]. Influenced by many factors like pH, the dose of coagulant, etc., coagulation–flocculation can be optimized by controlling these factors, their effects and interactions on the treatment of wastewater [8].

Many researches have been done in this field previously, but most of them were conducted with synthetic dyes to simulate real textile wastewater (RTW) [9,10], the optimization of the coagulation–flocculation process with chloride ferric and polymer for the treatment of RTW have not been studied yet with responses surface methodology (RSM). This statistical method is a group of mathematical and statistical techniques that are based on the fit of empirical models to the experimental data obtained with experimental design [9].

The aim of this study is the evaluation of the effect of pH, coagulant, and flocculant dose as independent variables on the treatment of RTW by the CF process; using central composite design (CCD) beside optimizing these operating conditions with response surface methodology, for attending a maximum elimination rate of the polluting load presented by COD, BOD₅, turbidity, and color.

2. Materials and methods

2.1. Samples

The effluent was taken from textile industry (production of jeans). The jeans are made mainly of cotton, which is then transformed into large denim fabrics; this factory is located in Ain Sbaa, Casablanca, Morocco. After Printing, the washing step is a finishing stage that is necessary to eliminate the excess of colorants that has not been fixed. The sampling was done at this point in the process. The samples were collected in 50 L polypropylene carboy with a frequency of 3 L every 2 h for 24 h, transported immediately to the laboratory and stored in a refrigerator at 4°C before proceeding for the analysis and treatment. Effluent characterization is shown in Table 4.

2.2. Chemicals and materials

The experimental set-up used for the coagulation–flocculation experiments at a laboratory scale is composed of a Jar-test device (Jar Test Flocculator FC-6S Velp Scientific) with a six-place. Jar test experiments consisted of three stages: stirring the RTW at 300 rpm for 10 min after adding ferric chloride (40%), the stirring is reduced to 30 rpm for 20 min after adding polymer (polyacrylamide Himoloc DR3000). Then Samples were taken from the supernatant and analyzed after being settled for 24 h.

Jar test was employed for optimizing pH, coagulant, and flocculant dose, based on the highest percentage removal of

COD, BOD₅, turbidity, and color. The equation for removal efficiency is shown below, where C_i and C_f are the initial and final concentrations of each parameter of wastewater [11].

$$\text{Removal (\%)} = \left[\frac{(C_i - C_f)}{C_i} \right] \times 100 \quad (1)$$

2.2.1. Coagulant FeCl₃ 40%

The coagulant used is ferric chloride 40%. The characteristics of this coagulant are shown in Table 1.

2.2.2. Flocculant Himoloc DR3000

The flocculant used is an anionic polymer 0.3%; its trade name is Himoloc DR3000. The characteristics of this flocculant are shown in Table 2.

2.3. Analytical methods

The physicochemical analysis (pH, conductivity, turbidity, COD, Color) was carried out to the Standard Methods of the AFNOR 1999. Turbidity was measured by nephelometry using a laboratory turbidimeter (HI 93703 microprocessor turbidity meter) and expressed in NTU (nephelometric turbidity unit). The pH was determined by a pH meter model 6209 and the conductivity was determined by a laboratory conductimeter.

2.4. Central composite design

The optimization of textile wastewater treatment by the coagulation–flocculation process is achieved using the RSM (response surface methodology). This methodology is based on a rotatable central composite design, where there are axial points, factorial points and central points. Moreover, the variance between predicted response at any point depends only on the distance between this point and the central point of the design [12]. This design is employed to study the empirical relationships between four responses (COD, BOD₅, turbidity, and color removal efficiency) and three factors: pH, FeCl₃, and flocculant dose.

Table 1
Characteristics of ferric chloride

Determination	Data
Chemical formula	FeCl ₃
Appearance	Dark brown
Ferric chloride, wt. %	39.0–41.0
Fe (III), wt. %	13.4–14.2
Fe (II), % mass fraction of iron (III) content	<2.5
Manganese	<0.4
Insoluble matter	<0.2
Density to 20°C, kg/dm ³	1.400–1.440
pH at 20°C	<1
Melting/freezing point, °C	12
Boiling point, °C	106

To determine the appropriate parameters and experimental domain preliminary tests were performed. Table 3 shows the levels attributed to each variable [13].

The analysis of variance method and *p*-value lower than 0.05 was considered significant in surface response analysis investigated data. The optimal values of the operation parameters were determined by the three-dimensional response surface and the contour plots [13,14].

To make an efficient prediction and to have a descriptive quality in the whole experimental domain chosen, with a minimum number of experiments, the design of the plan requires 16 experiments:

$$2^k + 2k + N_0 = 8 + 6 + 2 = 16 \tag{2}$$

where *k* is the number of independent variables; *N*₀ is the number of repetitions of the central points.

3. Results and discussion

The physicochemical characteristics of textile wastewater are shown in Table 4. This effluent is characterized by high turbidity, loaded with organic matter represented by COD and BOD₅. With a low biodegradability which makes the physicochemical treatment most appropriate.

The 16 experiments proposed by JMP were performed. The *i*th line of this matrix defines the experimental conditions of the *i*th experiment [14]. To minimize the error due to the experiments, the plan is carried out with a randomized order; the results obtained are subsequently grouped in Table 5. The principle of the exploitation of the results is based on the analysis of variance of the model, the computation of the estimates of the coefficients for the factors and their interactions, and determining their significance based on specific statistical tests [8].

Table 2
Characteristics of polymer

Parameter	Unites	Value
Appearance	–	Milky
Density, g/cm ³		1.2
Viscosity, cp		<600
pH	–	3.0–4.1
Cationicity molecular	%	35 high weight

Table 3
Parameters, experimental range, and level of independent variables

Natural variables (<i>x</i> _{<i>i</i>})	Coded variables <i>X</i> ₁ , <i>X</i> ₂ , <i>X</i> ₃ *				
	–α	–1	0	1	+α
<i>x</i> ₁ = pH	7.1	7.1	8.1	9.1	9.1
<i>x</i> ₂ = [FeCl ₃], g/L	0.48	0.48	0.64	0.8	0.8
<i>x</i> ₃ = [Floc], mL/L	1.6	1.6	2.6	3.6	3.6

$$*\alpha = 1.00, X_i = \frac{x_i - x_0}{\Delta x_i}$$

3.1. Variance analysis

Variance analysis allows us to see if the variables selected for the modeling have a significant effect on the response [14]. According to analysis of variance (Table 6), the *F*_{statistics} values for all regression of responses (8.7138, 12.1908, 9.9536, and 9.2321), for the removal efficiency of COD, BOD₅, turbidity, and color respectively) are higher than *F*_{th} (*F*_{0.01}(9.6) = 7.98). The large value of *F* indicates that the variation in the response can be explained by the regression equation.

The associated *P*-value confirms the interpretation determined by Fisher test. A value of *P* lower than 0.05 indicates that the model is considered to be statistically significant. The *P* values for all of the regressions are lower than 0.05. Consequently, these results mean that there is no statistical difference between the experimental values and the estimated values of the models [15].

3.2. Graphical study of the effect of the factors

The graphs depicted in Fig. 1 describe the effect of the three factors on the four responses. At first, it is noted that the pH reflects a positive effect on the rate of removal of COD, BOD₅, and color. On the other hand, above a pH equal to 8.1 the effect becomes negative. For turbidity, the pH has a negative effect until it exceeds 8.1 it starts to react positively to the response [16].

The second factor, which is the dose ferric chloride, has a weak effect on the removal of COD, color, and turbidity. For the elimination of BOD₅, the effect of FeCl₃ is negative, but above 0.64 g/L, the effect becomes positive.

The dose of the flocculant has a low effect on turbidity. Whereas, for the removal rate of COD, BOD₅, and color the third factor has a positive effect up to a dose value of 2.6 mL/L the effect becomes negative [16].

3.3. Statistical study of factors effects

The main effects of the three studied variables and their interactions are shown in Table 7. Each coefficient is associated with the statistics values of “*F*_{experimental}” and *p*-value.

The Fisher test is used to determine the importance and the significance of factors effects and interactions between the variables on the responses studied. Generally, the largest amplitude of *F* corresponds to the smallest *p*-value [14].

Table 4
Characterization of the textile effluent before treatment

Parameter	Value
pH	8.02
Conductivity, ms/cm ³	2.6
Turbidity, NTU	132
BOD ₅ , mg O ₂ /L	124
Colouration	1.547
COD, mg O ₂ /L	1,150
DCO/BOD ₅	9.27

Table 5
Experimental data for responses according to central composite design

Random order	Actual order	pH	[FeCl ₃] (g/L)	[Floc] (mL/L)	BOD ₅ (%)	COD (%)	Turbidity (%)	Color (%)
18	1	-1	-1	-1	38	73.4	93.11	75.05
14	2	-1	-1	1	54	83.5	94.64	17.8
22	3	-1	1	-1	47	76.6	94.91	28.16
25	4	-1	1	1	50	84.8	93.16	59.68
15	5	1	-1	-1	46	87	93.48	92.25
28	6	1	-1	1	50	82.83	98.46	77.72
5	7	1	1	-1	58	84.8	99.24	72.94
24	8	1	1	1	46	85.56	93.92	96.83
29	9	-α	0	0	41	81.5	94.63	72.9
19	10	+α	0	0	46	89.21	97.30	98.11
9	11	0	-α	0	67	86.7	91.7	95.75
12	12	0	+α	0	67	92.25	93.18	96.58
2	13	0	0	-α	46	87.3	90.84	88.54
13	14	0	0	+α	48	92.1	90.9	90.83
8	15	0	0	0	58	90.8	91.53	92.79
7	16	0	0	0	63	95.44	91.97	89.21

Table 6
Variance analysis

Source	Degree of freedom	S _r ²	Sum of squares	Mean square	F _{statistics}	Prob. > F	Response
Model	9		90.41273	10.0459	8.7138	0.0080*	Turbidity (%)
Residual	6	1.1528743	6.917246	1.1529			
Total	15		97.329976				
Model	9		1,100.5841	122.287	12.1908	0.0033	BOD ₅ (%)
Residual	6	10.031130	60.1868	10.031			
Total	15		1,160.7708				
Model	9		458.43837	50.9376	9.9536	0.0056	COD (%)
Residual	6	5.1175282	30.70517	5.1175			
Total	15		489.14354				
Model	9		8,156.9697	906.33	9.2321	0.0068	Color (%)
Residual	6	98.172111	589.0327	98.172			
Total	15		8746.0024				

F_{statistics}: Experimental Fisher factor.

*significant to 1% ($F_{0.01}(9,6) = 7.98$).

S_r²: The variance of the experimental error.

We note in Table 7 a significant effect of pH and [FeCl₃] × [Floc] interaction and quadratic pH × pH interaction on the efficiency of elimination of turbidity, with F_{statistic} values larger than critical Fisher value ($F_{th} = 5.99$).

For the estimation of the coefficients on the removal efficiency of BOD₅. It is found that only the interactions pH × [Floc] and [FeCl₃] × [Floc], as well as the quadratic interactions pH × pH, [FeCl₃] × [FeCl₃], and [Floc] × [Floc] have a significant effect for a probability equal to 99% and F_{statistic} values higher than F_{th} (5.99) [13].

For a probability of 95%, the two factors pH and FeCl₃ concentration, pH × [Floc] interaction and quadratic interaction

of pH × pH are significant on the removal efficiency of COD, with F_{statistic} values significantly higher than F_{th} (5.99).

Furthermore, it is noted that for a 95% probability, only the pH factor, the [FeCl₃] × [Floc] interaction and the quadratic interaction of pH × pH have a significant effect on the removal efficiency of (color%), with high values of F_{statistic} [16].

3.4. Modelization

To describe the variation of the responses according to influencing factors, we choose to use a polynomial model

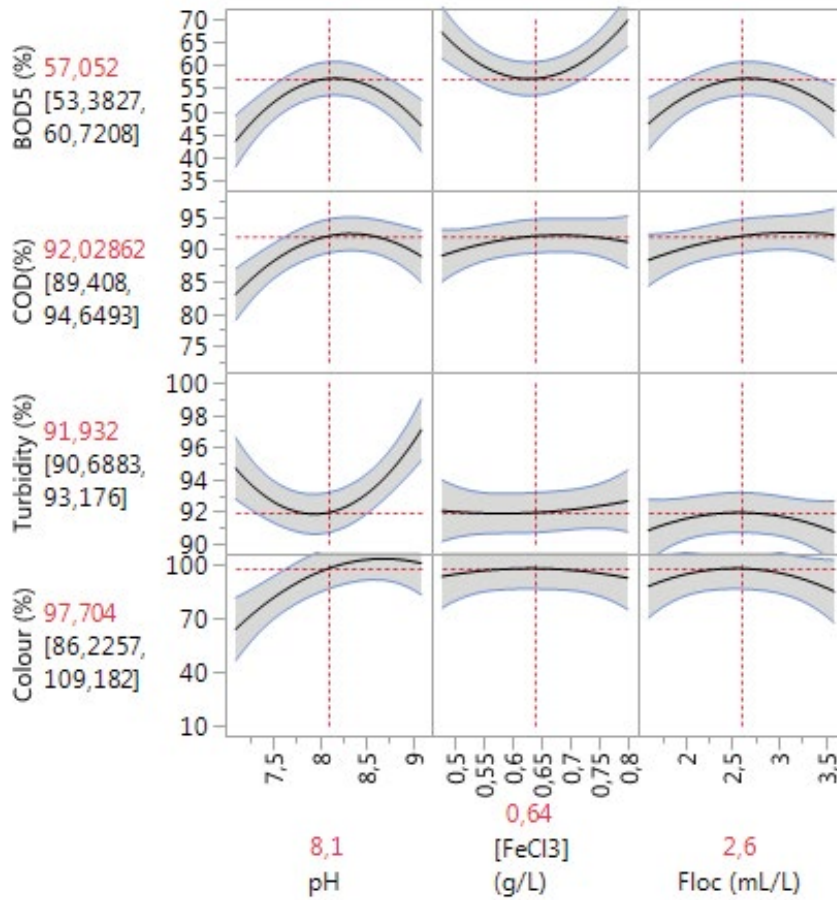


Fig. 1. Main effects plot of parameters on the responses.

that will be descriptive and representative of the model, hence, the need for the choice of a second-order model:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (3)$$

With:

- Y: Value of the calculated response,
- X_i: Value of the coded variable "i"
- b_i: Coefficient of the variable X_i model,
- b_{ii}: Coefficient of the model of the square variable X_i²,
- b_{ij}: Coefficient of the interaction model between X_i and X_j.

This model has 10 terms:

- Constant term = 1
- Linear term = 3
- Square term = 3
- Rectangle term = 3

The mathematical model only takes into account factors having a p-value < 0.05. This model is written as follows:

- removal rate of BOD₅%:

$$\text{BOD}_5\% = 57.051724 - 3.5416 \text{ pH} \times [\text{Floc}] - 3.708333 [\text{FeCl}_3] \times [\text{Floc}] - 11.91092 \text{ pH} \times \text{pH} + 11.33908 [\text{FeCl}_3] \times [\text{FeCl}_3] - 8.41092 [\text{Floc}] \times [\text{Floc}] \quad (4)$$

- removal rate of COD%:

$$\text{COD}\% = 92.028621 + 2.96 \text{ pH} + 1.969 [\text{Floc}] - 2.71375 \text{ pH} \times [\text{Floc}] - 6.107931 \text{ pH} \times \text{pH} \quad (5)$$

- removal rate of turbidity%:

$$\text{Turbidity}\% = 91.932172 + 1.1947 \text{ pH} - 1.697125 [\text{FeCl}_3] \times [\text{Floc}] - 3.9417414 \text{pH} \times \text{pH} \quad (6)$$

- removal rate of color%:

$$\text{Color}\% = 97.703793 + 18.42 \text{ pH} + 15.89875 [\text{FeCl}_3] \times [\text{Floc}] - 15.55069 \text{ pH} \times \text{pH} \quad (7)$$

3.5. Validation of model

The correlation between predicted and experimental values is shown in Fig. 2, characterized by points clouds whose alignment is close to a straight line [9]. The condition of normality of the residues is thus well-respected for

Table 7
Result of multiple linear regression of removal efficiency of all responses

Model term	Turbidity (%)			BOD ₅ (%)			COD (%)			Color (%)		
	Estimation	F _{statistic}	Prob. > F	Estimation	F _{statistic}	Prob. > F	Estimation	F _{statistic}	Prob. > F	Estimation	F _{statistic}	Prob. > F
Constant	91.932172	–	<.0001	57.051724	–	<.0001	92.028621	–	<.0001	97.703793	–	<.0001
pH	1.1947	12.3804*	0.0125	1.6833333	2.8248 ^{NS}	0.1438	2.96	17.1208**	0.0061	18.426	34.5839 ^{NS}	0.0011
[FeCl ₃]	0.3017	0.7895 ^{NS}	0.4084	1.3333333	1.7723 ^{NS}	0.2314	1.058	2.1873 ^{NS}	0.1896	–0.438	0.0195 ^{NS}	0.8934
[Floc]	–0.0497	0.0214 ^{NS}	0.8884	1.3833333	1.9077 ^{NS}	0.2165	1.969	7.5758*	0.0332	–1.408	0.2019 ^{NS}	0.6689
pH × [FeCl ₃]	0.112125	0.0872 ^{NS}	0.7777	0.4166667	0.1385 ^{NS}	0.7226	–0.49625	0.385 ^{NS}	0.5578	0.60125	0.0295 ^{NS}	0.8694
pH × [Floc]	–0.014625	0.0015 ^{NS}	0.9705	–3.541667	10.0036*	0.0195	–2.71375	11.5125*	0.0146	4.38625	1.5678 ^{NS}	0.2571
[FeCl ₃] × [Floc]	–1.697125	19.9865**	0.0042	–3.708333	10.9672*	0.0162	0.37875	0.2243 ^{NS}	0.6526	15.89875	20.5981**	0.0039
pH × pH	3.9417414	35.5304***	0.001	–11.91092	37.286***	0.0009	–6.107931	19.2191**	0.0046	–15.55069	6.4941*	0.0436
[FeCl ₃] × [FeCl ₃]	0.4167414	0.3972 ^{NS}	0.5518	11.33908	33.7918**	0.0011	–1.987931	2.0359 ^{NS}	0.2035	–4.89069	0.6423 ^{NS}	0.4534
[Floc] × [Floc]	–1.153259	3.0414 ^{NS}	0.1318	–8.41092	18.5927**	0.005	–1.762931	1.6011 ^{NS}	0.2527	–11.37069	3.721 ^{NS}	0.1117

***significant to 0.1% ($F_{0.001}(1.6) = 35.51$);

**significant to 1% ($F_{0.01}(1.6) = 13.75$);

*significant to 5% ($F_{0.05}(1.6) = 5.99$);

NS: not significant.

the model. Indeed, in this model, the value of R^2 was evaluated as 0.93, 0.95, 0.94, and 0.93 indicating that 93%, 95%, 94%, the model for turbidity could explain 93% of the variability in the response, COD, BOD₅, color efficiency removal, respectively. We conclude from these results, that there is no statistically significant difference between experimental and predicted values [14].

3.6. Optimization

The responses surfaces and two-dimensional (2D) contour plots (Figs. 3 and 4) are obtained using STATISTICA software. The principle of these curves is based on the study of the variation of the two factors having a very significant effect on the response, keeping the other factors in a fixed value.

The 3D surfaces and 2D contour plots are graphical representations of the regression equation for the optimization of reaction conditions and are the most useful approach in revealing the conditions of the reaction system [14,16].

The optimization of the coagulation–flocculation process gave appreciable results. Concerning the turbidity removal rate, a value about 100% is obtained for a pH of 9.1, with a flocculant concentration of 2.6 mL/L and a concentration of FeCl₃ varying between 0.48 and 0.8 g/L [10].

The BOD₅ abatement rate can reach values higher than 50%, provided that the pH is equal to 8.1, with a flocculant concentration of 2.6 mL/L, and a concentration equal to 0.64 g/L of FeCl₃ dose. These operating conditions allow for obtaining a COD reduction rate higher than 92% [11].

The color removal rate can be up to 100% provided the pH is above 7, and the flocculant concentration is set at 2.6 mL/L [16,17].

3.7. Experimental verification

To verify the optimal values obtained from the response surface and contour plot for the four responses (Figs. 3 and 4), experiments are done in the optimal conditions (Table 8). From these results, we can say that at pH 8.1 with a dose of FeCl₃ equal to 0.8 g/L and a dose of flocculant equal to 2.6 mL/L, the optimal elimination of turbidity, color, COD, and BOD₅ are 93.18%, 96.58%, 95.44%, and 67%; respectively.

4. Conclusion

The coagulation–flocculation technique has proven to be an effective method for the treatment of textile wastewater. A CCD was used to describe the process by a model of second order, by controlling the principal factors affecting the treatment, namely initial pH, the dose of Ferric chloride and the dose of flocculant. The analysis of variance tests indicate a high significance of the models chosen with high values of the Fisher report. Elsewhere, the results of model validation show that the model proposed agrees with experimental results with high values of the coefficient of determination R^2 . The RSM shows an optimal condition for efficient treatment, at a pH equal to 8 and a dose of FeCl₃ equal to 0.8 g/L and flocculant with a dose of 2.6 mL/L. The removal of turbidity, BOD₅, COD, and color, respectively, can reach 95%, 60%, 92%, and 96% of treatment efficiency.

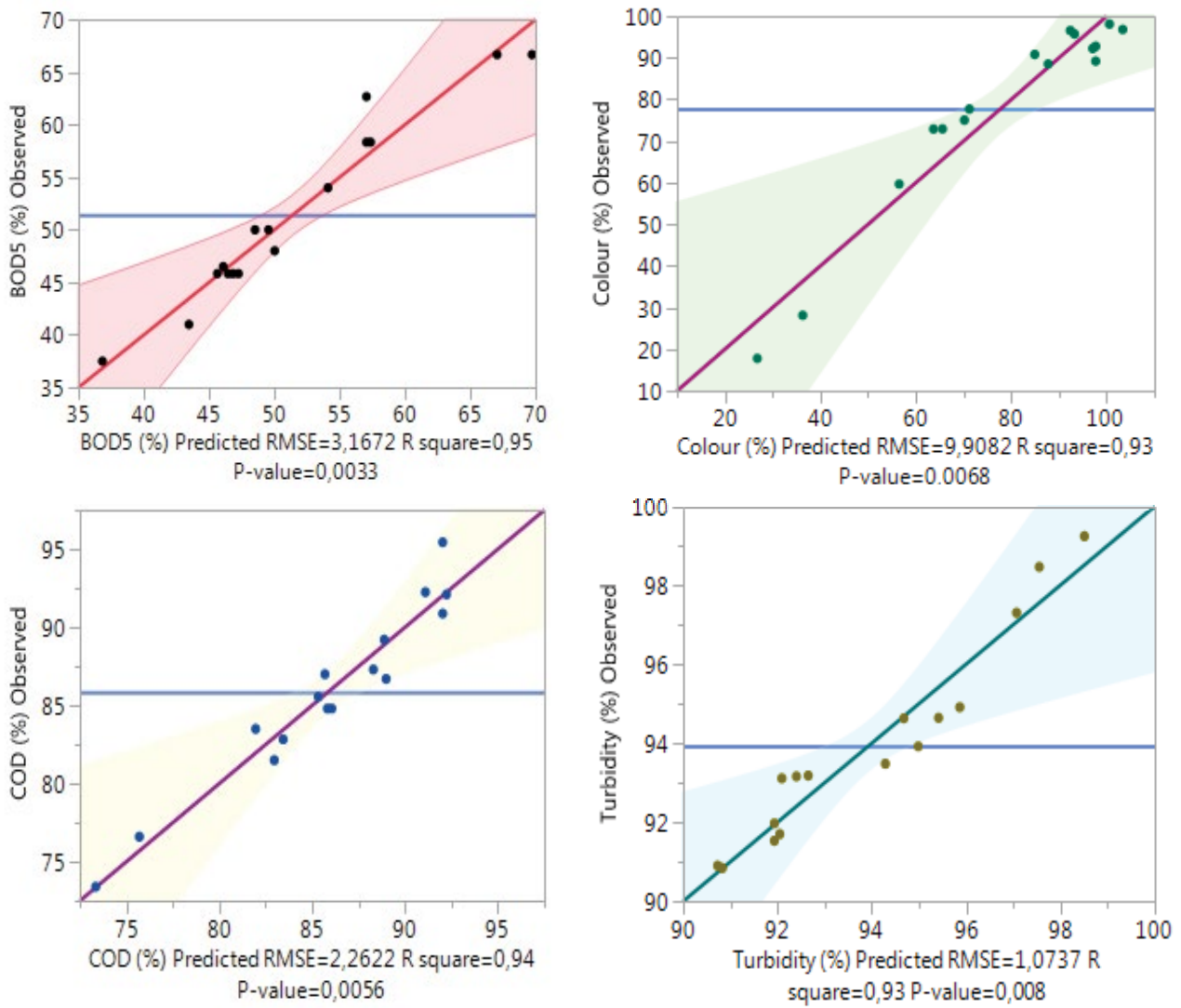


Fig. 2. Correlation between experimental and predicted values for the responses (removal efficiency of BOD₅ (%), COD (%), turbidity (%), and color (%)).

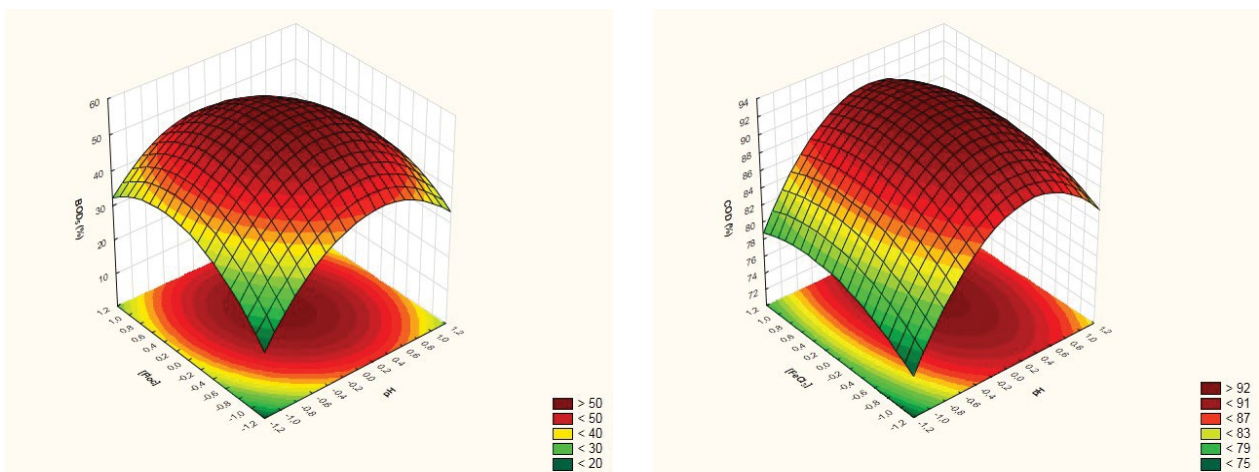


Fig. 3. Response surface and contour plot for BOD₅ (%) and COD (%).

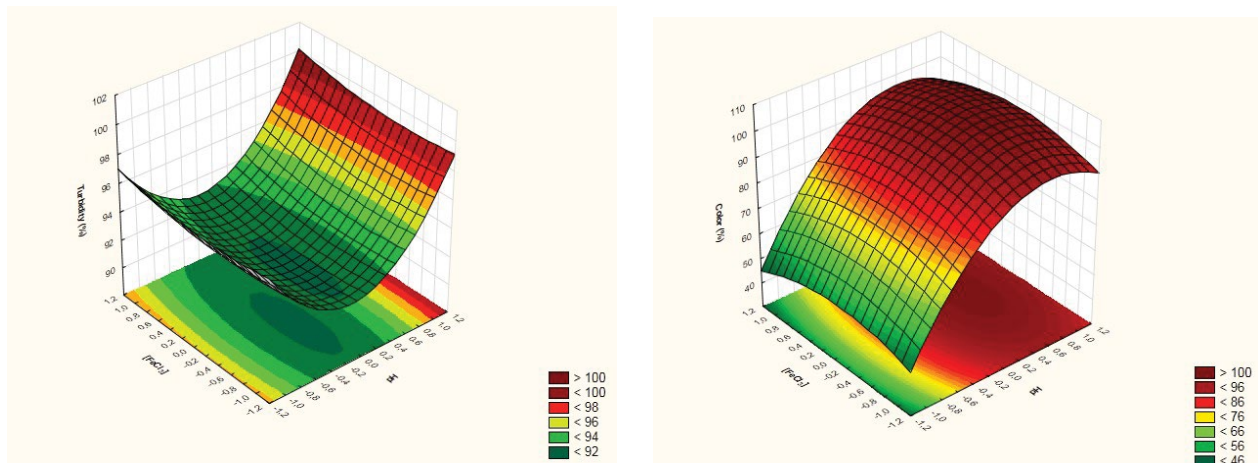


Fig. 4. Response surface and contour plot for turbidity (%) and color (%).

Table 8

Optimal conditions for an optimal treatment (the predicted and the experimental values)

Parameters	Optimum values	
	Predicted values	Experimental values
$X_1 = \text{pH}$	7.5–9.0	8.1
$X_2 = [\text{FeCl}_3, \text{g/L}]$	0.64–0.80	0.8
$X_3 = [\text{Floc mL/L}]$	2.6	2.6

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