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Application of coagulants in pretreatment of fish wastewater using factorial design

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ABSTRACT

The effect of aluminum sulfate and ferric chloride on the coagulation of the effluent from the fish industry was investigated. Jar test experiments were used to evaluate the effect of parameters such as pH, type of coagulation and coagulant dose. A complete 3^2×2 factorial design was used where the independent variables were: type of coagulant (aluminum sulfate and ferric chloride), coagulant concentration (50 mg L^{-1}; 300 mg L^{-1}; 550 mg L^{-1}) and pH of the effluent (6.0, 7.0, 8.0). The responses were the removal of volatile solids, suspended solids, turbidity and chemical oxygen demand (COD). The studied variables were statistically significant ($p \leq 0.05$) for all responses. The exception was volatile solids considering coagulant concentration. A significant and negative effect for the variable type of coagulant was observed when it was considered COD and turbidity removal. The best condition was using ferric chloride in the concentration of 550 mg L^{-1} at pH 8.0, whose results indicated maximum efficiency removals of 86, 96, 89 and 60% for COD, turbidity, suspended solids and volatile solids, respectively.

Keywords: Coagulants; Aluminum sulfate; Ferric chloride; Fish effluent

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1. Introduction

The treatment of industrial effluents involves processes for the removal of impurities generated in the manufacturing of products. The design of a treatment system is dependent upon several factors: the flow rate and wastewater characteristics, type of product, degree of purity required by regulatory agencies, capital requirements, sophistication of management required and available land space [1,2]. In this way, the variability of the wastewater from a fish processing industry on both flow rate and composition (change of species that are being processed, unsteady operation of the plant, washing, etc.) definitely makes it difficult or inhibits the process of treatment using conventional biological processes [3,4].

Sedimentation aided by coagulation/floculation is a physicochemical method used as a pre-treatment before the chemical or biological process in order to remove suspended solids, foams, turbidity and organic matter [5]. It is a process by which small particles are bonded to each other, forming flocs by the addition of appropriate chemical products that neutralize or reduce the negative charge on the particles, whereas similar electric charges on small particles in wastewater cause the particles to naturally repel one another and hold the small, colloidal particles apart keeping them in suspension. Flocculation is the procedure of bringing the microfloc particles together to form large agglomerations by physical mixture [6,7].

In the treatment of water and effluents, several chemicals have been conventionally used as coagulants. Coagulants commonly used in wastewater treatment are aluminum and iron salts such as aluminum sulfate or ferric chloride [6,8,9]. The determination of the dosage of clotting necessary for the treatment of an effluent is analytically difficult since there are complex relationships between the chemical coagulant and the several compo-

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nents present in the effluents to be treated. Therefore, an equipment known as jar test is used to obtain the most efficient and economical dose of coagulant for a certain stirring speed and duration [6,9].

It was recognized that employing response surface methodology (RSM) can significantly minimize the number of experiments, evaluate mutual interactions between multiple variables, and determine the optimal process conditions. RSM is a group of techniques used to evaluate relationships between one or more measured responses and a number of quantitative independent variables that may have important effects on the measured responses [10].

The objective of this study was to investigate the feasibility of treating fish effluent by chemical coagulation. The changes in the water quality after coagulation were evaluated by monitoring the parameters COD, turbidity, suspended solids and volatile solids. The effects of coagulation conditions (type of coagulant, coagulant concentration and pH) were successively assessed using a complete factorial design.

2. Methods

2.1. Wastewater

Wastewater of the fish processing industry originated from the secondary treatment of an anaerobic pilot reactor (dimensions: 3 m height, 2.5 m diameter, effective volume 13,000 L) was used in the experiments. Wastewater samples were collected and transported in polyethylene bottles to the Laboratory of Biotechnology at Fundação Universidade Federal do Rio Grande (FURG), Brazil. The samples were stored at 4°C once received at the laboratory. The investigated parameters included chemical oxygen demand (COD), turbidity and suspended and volatile solids according to the standard procedures described in the standard methods for the examination of water and wastewater [11]. The turbidity was measured with a calibrated Termo Orion, model Aquafast II. The average raw wastewater characteristics measured in the plant during a period of 12 months are 1561 mg L^{-1} (COD), 119 NTU (turbidity), 264 mg L^{-1} (suspended solids) and 975 mg L^{-1} (volatile solids).

2.2. Biomass flocculation

Experiments on 1 L samples were performed using a Milan JT 101 jar test apparatus with six jars (cups of 2000 mL capacity, height of 19 cm and width of 12.5 cm), and the flocculation and efficiency of solids, COD and turbidity removal were evaluated in presence of $FeCl_3$ and $Al_2(SO_4)_3$.

During the treatment each sample was stirred rapidly (110 rpm) for 30 s to obtain complete mixing of the coagulant with the effluent to maximize the destabilization of colloidal particles and initiate coagulation, followed by one 10 s mixing period at 50 rpm to increase contact between coagulating particles and to facilitate the development of large flocs. The suspension was maintained at rest for the observation of flocs sedimentation and the quality of the clarified liquid and the characteristics of the chemically treated effluents were determined after 20 min settlement. Samples for analysis were taken by a suction device allowing the withdrawal of accurate amounts of 100 mL from jars for complete analysis.

2.3. Experimental design

A complete $3^2 \times 2$ factorial design for two independent variables (coagulant concentration and pH) at three levels and one independent variable (type of coagulant) at two levels each was carried out to evaluate the effect of these variables on turbidity, COD and solids removal. Based on this design, eighteen treatments were tested with replicas to provide additional degrees of freedom for error estimating and to improve the estimates of effects. The independent variables and their levels are presented in Table 1. According to the responses from the experimental design, the effects of each variable were calculated and the interactions between them determined. Through multiple regression analysis the empirical models expressed as Eq. (1) were generated. Response surfaces were then obtained through fitting the empirical models in order to understand the overall effect from the type of coagulant, coagulant concentration and pH on the removal of COD, turbidity and solids.

$$\begin{aligned} Y = {} & \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 \\ & + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \end{aligned} \tag{1}$$

X_1, X_2 and X_3 are the coded levels of the independent variables; β is the regression coefficient (β_0: intercept; β_1, β_2, β_3: linear; β_{12}, β_{13}, β_{23}: interaction and β_{11}, β_{22}, β_{33}: quadratic coefficients) accounting for each main, quadratic and interaction effect and Y is the predicted response for the dependent variable.

4. Results and discussion

4.1. Full factorial design

The results for the removal of COD, turbidity, suspended solids (SS) and volatile solids (VS) after using ferric chloride and alum compared to the raw effluent are presented in Table 1. The results indicate that the best removals for the studied parameters occurred in

Table 1
Experimental matrix design and results obtained for each of the response variables studied

Treatment	Coded levels and real values			Results			
	X_1	X_2	X_3	E-COD	E-Turbidity	E-SS	E-VS
1	−1 ($FeCl_3$)	−1 (50)	−1 (6,0)	63	64	14	13
2	−1 ($FeCl_3$)	0 (300)	−1 (6,0)	79	96	80	37
3	−1 ($FeCl_3$)	+1 (550)	−1 (6,0)	67	—	7	25
4	−1 ($FeCl_3$)	−1 (50)	0 (7,0)	74	68	25	44
5	−1 ($FeCl_3$)	0 (300)	0 (7,0)	67	89	50	9
6	−1 ($FeCl_3$)	+1 (550)	0 (7,0)	84	97	89	41
7	−1 ($FeCl_3$)	−1 (50)	+1 (8,0)	54	3	42	33
8	−1 ($FeCl_3$)	0 (300)	+1 (8,0)	72	88	88	35
9	−1 ($FeCl_3$)	+1 (550)	+1 (8,0)	86	96	89	60
10	+1 ($Al_2(SO_4)_3$)	−1 (50)	−1 (6,0)	44	22	31	11
11	+1 ($Al_2(SO_4)_3$)	0 (300)	−1 (6,0)	57	90	90	8
12	+1 ($Al_2(SO_4)_3$)	+1 (550)	−1 (6,0)	58	56	70	3
13	+1 ($Al_2(SO_4)_3$)	−1 (50)	0 (7,0)	36	12	24	44
14	+1 ($Al_2(SO_4)_3$)	0 (300)	0 (7,0)	50	85	58	32
15	+1 ($Al_2(SO_4)_3$)	+1 (550)	0 (7,0)	64	96	79	39
16	+1 ($Al_2(SO_4)_3$)	−1 (50)	+1 (8,0)	40	4	22	27
17	+1 ($Al_2(SO_4)_3$)	0 (300)	+1 (8,0)	57	—	68	10
18	+1 ($Al_2(SO_4)_3$)	+1 (550)	+1 (8,0)	60	94	73	9

Note: X_1: type of coagulant X_2: coagulant concentration (mg L^{-1}); X_3: pH; E-COD: chemical oxygen demand removal efficiency; E-SS: suspended solids removal efficiency; E-VS: volatile solids removal efficiency.

experiments 6 (ferric chloride, 550 mg L^{-1}, pH 7.0) and 9 (ferric chloride, 550 mg L^{-1}, pH 8.0) and the maximum removals occurred for COD, turbidity and suspended solids. Nunez et al. [12] investigated the use of coagulation/flocculation to remove organic matter from slaughterhouse wastewater by adding ferric and aluminum salts reporting a maximum COD removal efficiency of 45–75%. These results are inferior to those obtained in our work, which ranged from 84 to 86% when the best conditions were used. Al-Malack et al. [13] investigated the feasibility of treating polymeric industrial wastewater by sedimentation and chemical coagulation. They reported that ferric chloride produced the best result in terms of turbidity removal, similar to that founded in this work, since, in general the greatest removal efficiencies occurred with this coagulant.

The effect estimates for each variable, as well as the interactions between them or COD, turbidity and solids reduction were determined (Table 2). The main effects as well as their interactions showed significant influence ($p \leq 0.05$) in COD, turbidity and solids removal. An exception was the interaction type of coagulant and pH for COD removal. These results agree with Moraes et al. [14], who, analyzing the use of two inorganic coagulants (ferric chloride and aluminum sulfate) in the treatment of fish effluent, observed that for solids and turbidity removal, both of them as well as the effluent pH showed significant influence. It was also observed that coagulant concentration presented a significant effect and the increase in the concentration of this variable led to an increase in turbidity and suspended solids removal and a decrease in COD removal. That corresponds to say that passing from the low (−1) to high (+1) level a reduction of around 17.99% of COD, 65.08% of turbidity and 43.98% of suspended solids occurs. This fact is in concordance with the results presented in Table 1, which showed the largest reductions in the concentrations of 300 and 550 mg/L.

There were statistically significant differences and a negative influence between ferric chloride and aluminum sulfate for COD and turbidity removal. That corresponds to say that passing from the low (−1) to high (+1) level a reduction of around 18.19% of COD and 12.89% of turbidity occurs. This is in agreement with the results reported by Al-Malack et al. [13] and Ebeling et al. [6], who observed that ferric chloride is more efficient as a coagulant than aluminum sulfate. An increase in pH from 6.0 to 8.0 exhibited a positive influence, increase of COD, suspended and volatile solids removal. An exception is the turbidity removal, where the increase in the pH of the effluent led to a decrease of the turbidity reduction. According Al-Mutairi et al. [9] higher pH values may produce negatively charged organic contaminants on which adsorption will be electrostatically hindered.

The results obtained in Table 2 also indicate that all independent variables were shown to be significant and the increase of these variables led to an increase in

Table 2
Main effects and interaction analysis for COD, turbidity and solid removal in fish effluent

Factor	COD			Turbidity			Suspended Solids			Volatile Solids		
	Effect	Std err	p value	Effect	Std err	p value	Effect	Std err	p value	Effect	Std err	p value
Interc.	6446	38	0	7745	401	0	6507	52	0	2780	301	0
X_1	−1819	75	0	−1289	807	0	1037	100	0	417	582	0
X_2	1799	86	v0	−6508	890	0	4398	115	0	134	668	59
X_3	184	86	45	−435	961	0	1693	115	0	1637	668	0
X_1X_2	294	71	0	337	798	0	1516	96	0	709	556	0
X_1X_3	72	71	328	1051	759	0	−1342	96	0	516	556	0
X_2X_3	508	76	0	1617	817	0	584	102	0	218	593	1

Notes: X_1: type of coagulant; X_2: coagulant dose; X_3: pH; X_1X_2: interaction type of coagulant and concentration; X_1X_3: interaction type of coagulant and pH; X_2X_3: interaction concentration and pH.
Interc.: Intercept; Effect: The increase or decrease percentage (positive or negative effect) on the values answers variables (dependent variables) in the pass of the upper or lower level of the studied parameters (independent variables) according to the experimental design; Std error: theoretical standard deviation of all sample means of size *n* drawn from a population and depends on both the population variance (sigma) and the sample size (*n*); *p* value: represents the probability of error that is involved in accepting our observed result as valid.

suspended solid removal. In the study carried out by Al-Mutairi et al. [9], the removal of suspended solids ranged from 98% to 99%; aluminum sulfate was used alone in the range of 100–1000 mg L^{-1}, with pH in the range of 4.0–9.0. In addition, Ata and Jameson [15] stated that more than 98% of the suspended solids can be removed from industrial wastewaters with the use of coagulation/flocculation. In our work the maximum value (90%) for suspended solids in fish effluent was reached with high values of pH and coagulant concentration. The removal rates of suspended materials observed in the experiments are beneficial when a subsequent stage of biological treatment exists to facilitate the degradation of dissolved material by microorganisms.

For the tested samples, the main and interaction effects observed for suspended solids were higher than those for volatile solids. The coagulation is an important physicochemical step in industrial wastewater treatment to reduce the suspended and colloidal materials therefore, the low effect comparing to suspended solids and the non-significant effect for coagulant dose is considered normal. Furthermore, the volatile solids can be removed in a subsequent step (biological unit) present in the wastewater treatment plant.

4.2. Model fitting

A model fitting was accomplished for the experimental design as shown in Table 1. The independent and dependent variables were fitted to the second order model equation and examined in terms of the goodness of fit. The linear and interaction terms of the model, not significantly different from zero (p >0.05), were excluded from Eq. (1). β_{33} and β_{13}, the coefficients for the COD removal, were non-significant. Therefore, these coefficients were dropped from the model and then a new ANOVA was performed and the mathematical model was refitted by multiple linear regression. The results are listed in Table 3. Eqs. (2)–(5) represent the models generated for the responses turbidity, COD and solids removals using the ferric chloride and aluminum sulfate coagulants, considering the significant effects and interaction of the factors in study in the codified form. It can be noted that the coefficient of determination (R^2), which is the fraction of variation of the observed response values explained by the model, was higher than 0.80 for the turbidity and COD removal, indicating that more than 80% of the variability in the response could be explained by the models. The statistical significance of the models were confirmed by the Fisher's *F*-test which determines if the regression equation is statistically signi-ficant by the values of $F_{\text{calculated}}$ and F_{critical}. According to Box et al. [10], so that a regression is not only statistically significant but also useful for predictive purposes, the ratio between the mean square for regression and mean square for residual ($\text{MS}_{\text{regression}}/\text{MS}_{\text{residual}}$, which corre-sponds to $F_{\text{calculated}}$), must be at least three times greater than the value of F_{critical} since its calculated *F* value is higher than the critical *F* value for turbidity removal ($F = 43.13 > F_{8,24,0,05} = 2.36$) and COD removal ($F = 36.13 > F_{5,30,0,05} = 2.53$). The data for the independent variables turbidity (R^2 = 0835) and COD (R^2 = 0868) and reason $F_{\text{calculated}}/\ F_{\text{critical}}$ were 18.27 and 14.28, indicating that these models are also predictive. Based on these results, the models can be utilized to generate response surfaces for the analysis of the variable effects on pollutants removal.

Table 3
Models of the responses in the fish effluent using the coagulant ferric chloride and aluminum sulfate

Model	Eq.
Turbidity = $110.12 - 0.2598X_1 + 58.09X_2 - 0.5765X_2^2 + 3.9591X_3 - 0.0426X_3^2 + 0.006X_1X_2 + 0.002X_1X_3 + 0.0031X_2X_3$ ($R^2 = 0.8686$)	(2)
COD = $74.978 - 0.20893X_1 + 11.10823X_2 - 0.11009X_2^2 - 0.02906X_3 + 0.00057X_1X_2 + 0.00098X_2X_3$ ($R^2 = 0.8351$)	(3)
SS = $68.17 + 0.08504X_1 + 45.82551X_2 - 0.45596X_2^2 + 5.7499X_3 - 0.05511X_3^2 + 0.00291X_1X_2 - 0.00258X_1X_3 + 0.00112X_2X_3$ ($R^2 = 0.7421$)	(4)
VS = $30.05502 - 0.07684X_1 - 9.39855X_2 + 0.09323X_2^2 + 18.37668X_3 - 0.18287X_3^2 + 0.00136X_1X_2 + 0.00099X_1X_3 + 0.00042X_2X_3$ ($R^2 = 0.3761$)	(5)

Notes: X_1: type of coagulant; X_2: coagulant concentration; X_3: pH; R^2: coefficient of determination.

Figs. 1–4 express the response surfaces obtained through the models for COD and turbidity [Eqs. (2) and (3)]. The surface methodology was used to evaluate relationships between the measured responses (COD and turbidity) and the independent variables (type of coagulant, coagulant concentration and pH) that may have important effects on the measured responses [10]. Variables giving quadratic and interaction terms with the largest absolute coefficients in the fitted models were chosen for the axes of the response surface plots to account for curvature of the surfaces. The greatest coefficients (β_2) in the fitted models were obtained for turbidity and COD removal (Table 3), which revealed the high sensitivity of the turbidity and organic matter to removal by different doses of coagulant.

Fig. 1 shows that an increase in coagulant concentration led to an increase in COD reduction. When type of coagulation is considered, applying ferric chloride the optimum coagulant concentration can be found around the higher doses of the experimental region studied (that is, from 50 to 550 mg L^{-1}). These results are in agreement with Nuñez et al. [12], who applied the coagulants ferric chloride and aluminum sulfate to the treatment of bovine slaughterhouse effluent. Their results showed COD reductions of 75% and 45% respectively. Similar results have been reported applying the process of coagulation–flocculation as treatment in mixed industrial–domestic wastewater where a reduction of 69.5% for organic matter was obtained in the presence of 200 mg L^{-1} ferric chloride at pH 6.0 [5]. Comparative studies between ferric chloride and aluminum sulfate demonstrate that the ferric chloride enables the production of decanted water of better quality in relation to the parameters of reduction of COD and turbidity [16]. This observation can be confirmed by the data registered in Table 2 as well as in Figs. 1–4, where the COD and turbidity reduction was up to 86% and 96% respectively.

Fig. 2 shows the dependence of the COD removal (variable y_1) on x_1 (coagulant concentration) and x_2 (pH) for $FeCl_3$. The COD removal increased as the coagulant concentration increased to its central level (300 mg L^{-1}). Thereafter, COD decreases as the coagulant concentration increases toward its high level, i.e. 550 mg L^{-1}. Besides, there is no change in COD removal as the pH increases toward its high level.

Fig. 3 shows the dependence of the turbidity removal on X_2 and X_3 for the coagulant ferric chloride. This increases with coagulant concentration to its central level (300 mg L^{-1}). Thereafter, turbidity decreases as the coagulant dose increases toward its high level, i.e. 550 mg L^{-1}. Also, in Fig. 4 an increase in the turbidity reduction with high coagulant concentration was observed, regardless the type of coagulant. The best turbidity removal was observed using coagulant concentrations between 300 and 550 mg L^{-1}. Al-Mutairi et al. [9] observed an increase in the turbidity removal as the coagulant dosage increases, and the best concentration for turbidity removal found by these authors occurred in an alum dosage 200 mg L^{-1}. However, their initial concentration was around 26 NTU, while in our work this concentration was about 119 NTU, where a reduction in turbidity of up to 80% was observed using ferric chloride in the concentrations of 300 and 550 mg L^{-1}.

Delgado et al. [17], using ferric chloride as coagulant in the treatment of refrigerating effluent, reached efficiencies which oscillated in the range of 60–75% for turbidity reduction, applying dosages that varied between 50 and 500 mg L^{-1} in the sedimentation process.

The largest removal efficiencies were observed for experiments 6 and 9. There were no significant difference at a probability level of 0.05 among the average values from the removal efficiencies for COD ($p = 1.0$), turbidity ($p = 0.16$) and suspended solids ($p = 1.0$). This enables treatment 9 to be chosen as the best condition once in agreement with Hornes et al. [18] the average values of pH in the fish effluent lies in the range from 8.5 to 9.0. According to Mittal [19] and Nuñez et al. [12], the units of coagulation/flocculation can achieve chemical oxygen demand (COD) reductions ranging from 32% to 90%. In the best considered condition (treatment 9), reductions of

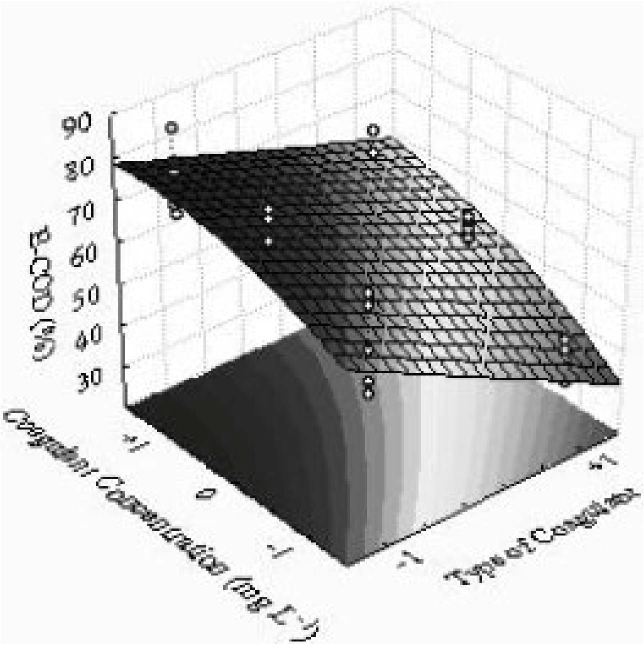

Fig. 1. Second-order response surface plot in the E-COD (Y_i) for the fish effluent treatment by coagulation/flocculation. Dependence of Y_i on the type of coagulant (X_1) and coagulant concentration (X_2) is shown pH, X_3 = 7.0.

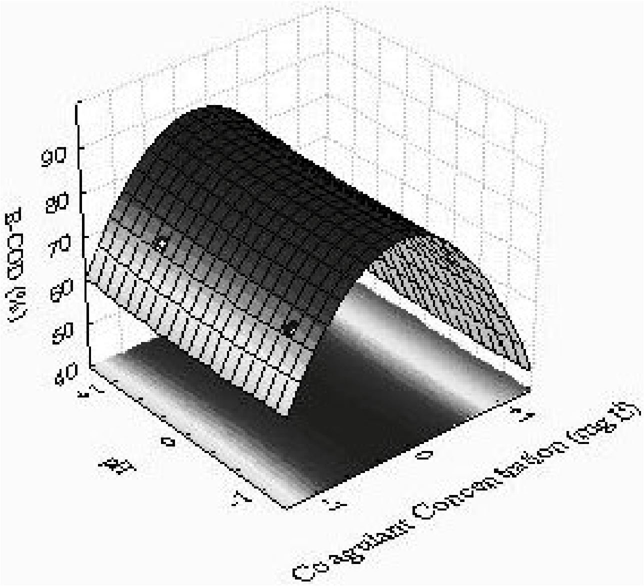

Fig. 2. Second-order response surface plot in the E-COD (Y_i) for the fish effluent treatment by coagulation/flocculation. Dependence of Y_i on the coagulant concentration (X_2) and pH (X_3) is shown type of coagulant, X_1 = $FeCl_3$.

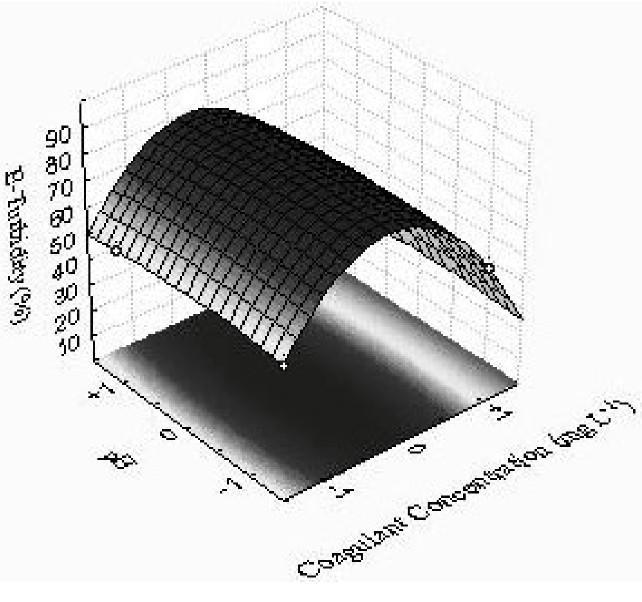

Fig. 3. Second-order response surface plot in the E-turbidity (Y_i) for the fish effluent treatment by coagulation/flocculation. Dependence of Y_i on the coagulant concentration (X_2) and pH (X_3) is shown type of coagulant, X_1 = $FeCl_3$.

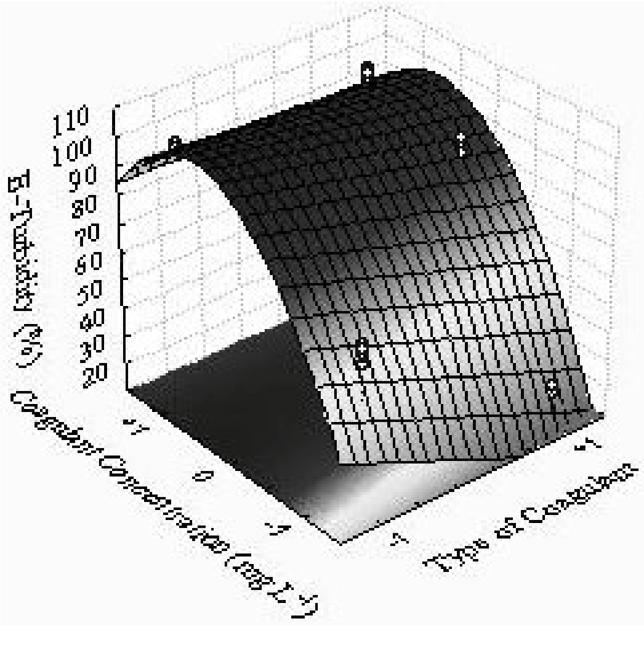

Fig. 4. Second order response surface plot in the E-turbidity (Y_i) for the fish effluent treatment by coagulation/flocculation. Dependence of Y_i on the type of coagulant (X_1) and coagulant concentration (X_2) is shown pH, X_3 = 7.0.

respectively 86%, 96%, 89% and 60% for COD, turbidity, SS and VS (Table 1) are registered. It is important to point out that soluble or filtered COD includes the portion due to dissolved particles (totally soluble) as well as the one due to the presence of colloidal particles [20]. This way, the high removal efficiency obtained due to the coagulant addition—mainly the ferric chloride—proves the coagulation process to be efficient not only in the reduction of suspended material, but also in the soluble and colloidal matter.

5. Conclusions

Under the experimental conditions it is possible to conclude that the type of coagulant, concentration and effluent pH significantly influence ($p \leq 0.05$) the reduction of COD, turbidity and SS. The optimum conditions for COD (86%), turbidity (96%), suspend solids (89%) and volatile solids (60%) efficiency removal were utilizing ferric chloride 550 mg L^{-1} and pH 8.0. The removal efficiencies of the studied parameters are quite satisfactory proving the efficiency of the coagulation/flocculation process in the primary treatment of effluents from the fishing industry.

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