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Employing the Taguchi method to obtain the optimum conditions of coagulation–flocculation process in tannery wastewater treatment

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ABSTRACT

In this study a series of jar test experiments, designed using Taguchi method, were carried out to determine the optimum conditions for tannery wastewater treatment, and the effects of type and dose of coagulant, type and dose of coagulant aid and pH on the efficiency of coagulation–flocculation process were evaluated. The goal parameters to find the optimum conditions were each of chemical oxygen demand (COD), chromium concentration, total dissolved solids (TDS) and turbidity of the wastewater, or all together, which were used to track the efficiency of the treatment process.

Coagulant type in COD removal, pH in chromium removal, coagulant aid type in TDS removal and coagulant aid dose in turbidity removal were the most effective factors. The optimum conditions that were obtained for the treatment of the wastewater were: 800 ppm $FeCl_3$ as coagulant, 600 ppm Na_2CO_3 as coagulant aid and pH 7.5 for COD removal; 1600 ppm $FeCl_3$ as coagulant, 100 ppm CaO as coagulant aid and pH 7.5 for chromium removal; 1600 ppm $FeCl_3$ as coagulant, pH 9 and 300 ppm Na_2SiO_3 as coagulant aid for TDS removal; and 800 ppm PAC (polyaluminum chloride) as coagulant, pH 7.5 and 600 ppm Na_2CO_3 as coagulant aid for turbidity removal. The optimum conditions found for COD removal, were selected as optimum conditions for the cases where the removal of aforementioned parameters, all together, is treatment goal.

1. Introduction

The leather tannery industry is an important activity in many developing countries, "it has been estimated that about 18 billion ft^2 of leather are made annually around the world with a trade value estimated to be approximately US$ 70 billion" [1].

This industry uses the large quantities of water and, in turn, produces the large quantities of liquid effluents [2]. Tabriz, the capital city of east Azarbaijan province in north west of Iran, has approximately 320 tanneries with an estimated wastewater discharge of 3.5–4 million L/day that are released, mostly without treatment, into the environment and cause serious environmental damages. There are similar problems in other industrial areas with large number of tanneries in Iran.

In leather tanning, a number of processes are performed using a large number of chemicals such as surfactants, acids, dyes, natural or synthetic tanning agents, sulfonated oils, salts, etc., to convert animal skin into an unalterable and incorruptible product [3]. Considering the application of these low biodegradable chemicals, tannery wastewaters cause serious environmental problems. Some authorities consider the tannery wastewater as one of the ten most harmful threats for the environment [4]. It uses about 30–40 L of water per kilogram of hide [5]. The large volume of produced effluent in addition to the high concentrations of COD, chromium and total suspended and dissolved solids (TSS, TDS), cause many problems for tanneries. Wastewater treatment companies have considered aforementioned parameters as major ones in the treatment of tannery wastewaters [6].

The various wastewater treatment methods such as coagulation–flocculation [6], advanced oxidation processes [7,8], biological treatment [9], ozonation [10] and activated carbon adsorption [11] have been applied for the treatment of tannery wastewaters. Coagulation is the most widespread process in wastewater treatment. "Coagulation is usually achieved through the addition of inorganic

Abbreviations: A_i, the sum of all observations of level i of factor A; ANOVA, analysis of variance; BOD, biochemical oxygen demand (mg L^{-1}); COD, chemical oxygen demand (mg L^{-1}); DOF, the degree of freedom; F, associated F-test of significance; k_A, the number of levels of factor A; N, the number of all experiments; n, the number of repetition done for an experiment; n_{Ai}, the number of all observations at level i of factor A; OAs, orthogonal arrays; P, the percent of contribution of each factor on the response; PAC, polyaluminum chloride; ppm, parts per million; S/N, the ratio of signal to noise; SS, sum of squares/suspended solids; SS_A, sum of squares of factor A; SS_T, sum of squares for all of factors; T, the sum of all observations; TCOD, total chemical oxygen demand (mg L^{-1}); TDS, total dissolved solids (mg L^{-1}); TSS, total suspended solids (mg L^{-1}); y_i, the performance value of ith experiment.

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coagulants such as aluminum or iron-based salts, and/or synthetic organic polymers commonly known as polyelectrolytes" [12]. This process forms the precipitating flocs, which adsorb the pollutants. The pollutants are removed when the flocs are precipitated. This method is used for the removal of metals, treatment of toxic wastes, removal of turbidity and suspended solids and control of color. And also it is suitable to enhance the effectiveness of subsequent treatment and to provide treatment with lower cost [12].

This process is not always perfect and may result in small flocs when coagulation takes place at too low temperatures, or fragile flocs which break up when subjected to physical forces [13]. Flocculation is necessary to overcome these problems and to improve the coagulation process to obtain a good-quality effluent and rapid sedimentation of the flocs. So various materials, as coagulant aids, can be used to affect on coagulation or to increase flocs density and thereby, to improve sedimentation [13]. Coagulant aids are different ionic, polymeric or other materials, which can enhance the flocs quality via various mechanisms. Hence, the selection of suitable coagulant aid is essential to achieve the coagulation process with high efficiency.

The effect of inorganic coagulants dose can be explained by using the "coagulation zone" concept. Zone I: not enough coagulant is present for the destabilization of the colloids. Zone II: added coagulant is sufficient, and in weakly acidic or nearly neutral condition, the dissolved positively charged ions are adsorbed onto the colloidal particles thus destabilizing the suspension (via decreasing the negative surface charge of the colloidal particles). Zone III: the excess concentration of coagulant can cause charge reversal and re-stabilization of particles. Zone IV: in neutral or basic environment, the high concentration of coagulant cause oversaturation with metal hydroxide precipitations which entrap the colloidal particles and produces very effective sweep coagulation [14].

Some studies have been performed on the treatment of tannery wastewaters by using coagulation method. Garrote et al. [15], used two cycles of the treatment by alkaline $FeCl_3$ and $Ca(OH)_2$ to treat tannery wastewater. They obtained a colorless and odorless effluent with 87% COD removal. Song et al. [6], worked on the treatment of tannery wastewater by using chemical coagulation. They studied the influence of pH and coagulant dosage on the coagulation process to optimize the conditions to achieve the best removal of COD, SS (suspended solids) and chromium. In the best conditions, the removal range of total COD, chromium and SS were 30–37%, 74–99% and 38–46%, respectively. Haydar and Aziz [16] also reported 99.7% turbidity removal, 96.3% TSS removal, 48.3% TCOD (total chemical oxygen demand) removal and 99.7% chromium removal by using 100 mg/L $Al_2(SO_4)_3$ with 5 mg/L of cationic polymer C-496.

Table 1
Sample values for physico-chemical characteristics of studied tannery wastewater after sedimentation.

Parameter	Concentration
pH	7.5 ± 0.2
COD	3800 ± 5 mg L^{-1}
Total Cr	22 ± 2.5 mg L^{-1}
TSS	573 ± 50 mg L^{-1}
TDS	21,010 ± 100 mg L^{-1}
Cl^-	7100 ± 50 mg L^{-1}
Sulfate	600 ± 50 mg L^{-1}
Conduction	22.6 ± 0.05 mS cm^{-1}
Turbidity	56 ± 1 NTU
Color	Dark green 780 ± 1 mg L^{-1} Pt

Taguchi method which established by Dr. Genichi Taguchi [17], "utilizes orthogonal arrays (OAs) from experimental design theory to study a large number of variables with a small number of experiments. Using OAs significantly reduces the number of experimental configurations to be studied" [18]. Tippett discovered OAs in 1934, however, Taguchi simplified their use by providing tabulated sets of standard OAs and corresponding linear graphs to fit specific projects [18]. In Taguchi method the experimental response quality is expressed as the ratio of desired factor (signal) compared to uncountable factors (noise) [19].

The present paper is studying the treatment of a tannery wastewater using coagulation–flocculation process and is an attempt to: (1) determine the optimum conditions for the removal of COD, chromium, TDS and turbidity of tannery wastewater, each as a treatment goal, (2) determine the optimum conditions for the removal of COD, chromium, TDS and turbidity of tannery wastewater, all together, as treatment goal and (3) determine the influence of each investigated factor including type and dose of coagulant, pH, and type and dose of coagulant aid, on the removal efficiency. Using an "experimental design method" makes different the present work from the literature. "Experimental design method" optimizes the factors which are effective on the removal of goal parameters and reduces the number of experiments that are necessary to find the optimized conditions.

2. Experimental

2.1. Materials and methods

The tannery effluent that was used in this work obtained from the wastewater collection tank of tannery wastewater treatment

Table 2
The studied factors and their levels according to the L_{16} (4^5) experimental set.

Exp. no.	Control factors				
	A (coagulant type)	*B* (pH)	*C* (coagulant dose, ppm)	*D* (coagulant aid type)	*E* (coagulant aid dose, ppm)
1	$Al_2(SO_4)_3$	5	400	CaO	100
2	$Al_2(SO_4)_3$	7.5	800	$CaCO_3$	300
3	$Al_2(SO_4)_3$	9	1200	Na_2SiO_3	600
4	$Al_2(SO_4)_3$	11	1600	Na_2CO_3	800
5	$FeSO_4$	5	800	Na_2SiO_3	800
6	$FeSO_4$	7.5	400	Na_2CO_3	600
7	$FeSO_4$	9	1600	CaO	300
8	$FeSO_4$	11	1200	$CaCO_3$	100
9	$FeCl_3$	5	1200	Na_2CO_3	300
10	$FeCl_3$	7.5	1600	Na_2SiO_3	100
11	$FeCl_3$	9	400	$CaCO_3$	800
12	$FeCl_3$	11	800	CaO	600
13	PACL	5	1600	$CaCO_3$	600
14	PACL	7.5	1200	CaO	800
15	PACL	9	800	Na_2CO_3	100
16	PACL	11	400	Na_2SiO_3	300

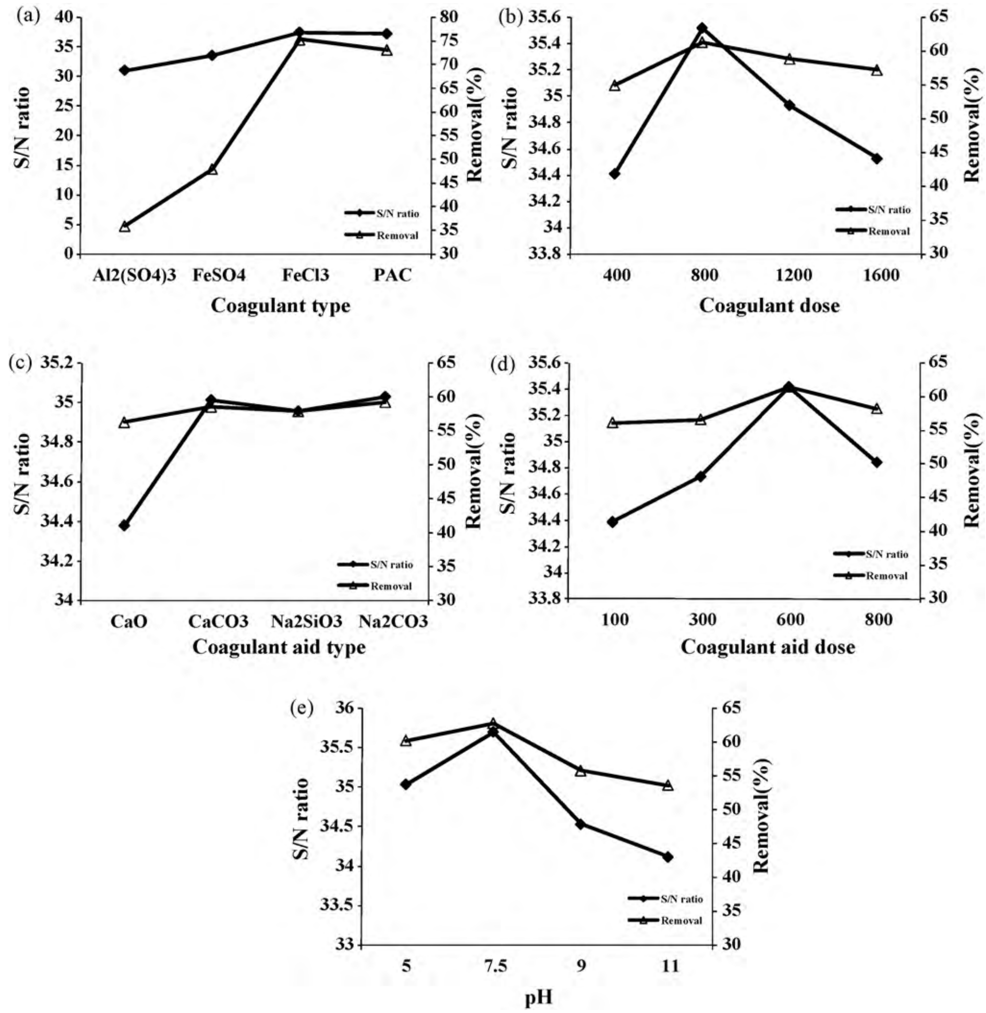

Fig. 1. The effect of experimental parameters on the S/N ratio and measured removal percent in the removal of COD.

plant located in Mayan-Tabriz (Northwest of Iran). Samples were collected in polyethylene bottles. The samples were preserved by acidification to pH ≤ 2 using sulfuric acid (H_2SO_4). The concentrations of chromium, TDS, TSS, chloride, sulfate and nitrate in the wastewater were measured according to standard methods [20]. COD, turbidity and color of the wastewater were analyzed by a Palintest 7700 (UK) photometer. The PerkinElmer spectrophotometer (Germany), model 550 SE, also was used in this study. All of the used chemicals were analytical grade and purchased from Merck Co. (Germany).

2.2. Jar tests

The coagulation–flocculation experiments were carried out in a jar test apparatus (OSK-Japan). The wastewater after a well mixing was allowed to settle for 3 h [21] and the supernatant was transferred to a clean container. The main physico-chemical characteristics of the supernatant are shown in Table 1.

Precise doses of coagulants including $Al_2(SO4)_3$, $FeSO_4$, $FeCl_3$ and PACL, and coagulant aids including CaO, Na_2CO_3, $CaCO_3$ and Na_2SiO_3, were added to 800-mL jars containing 200 mL of settled wastewater with specified pH values. A series of jar tests were carried out as follows: first rapid mixing stage carried out on jars at 100 rpm for 2 min and then slow mixing stage carried out at 30 rpm for 20 min and finally the solutions were settled in 250-mL graduated cylinders for 30 min [6,21]. The produced supernatants were used for the measurement of remained COD, Cr, TDS and turbidity.

2.3. Experimental design

Factors such as type and dose of coagulant, pH, and type and dose of coagulant aid influence the coagulation–flocculation process. In addition to these factors, stirring speed, stirring time, settling time, and temperature are effective, too. However, in this work the last ones considered constant and the effect of first ones investigated. Orthogonal array of L_{16} (4^5) type that is a standard plan of Taguchi method, was used in experimental design. The tests were conducted at average room temperature of 20 °C. The investigated factors and their levels in this study are shown in Table 2.

The S/N ratio was selected as the optimization criterion. The S/N ratios for "the larger the better" situations were calculated for COD, Cr and TDS removal (experimental results were entered in Taguchi method as the percent of removal) by the following equation [22]:

$$S/N = -10 \log \left(\frac{1}{n} \sum \frac{1}{y_i^2} \right) \quad (1)$$

Eq. (2) which is used to evaluate the S/N ratios for "the smaller the better" situations [22], were used in turbidity removal (experimen-

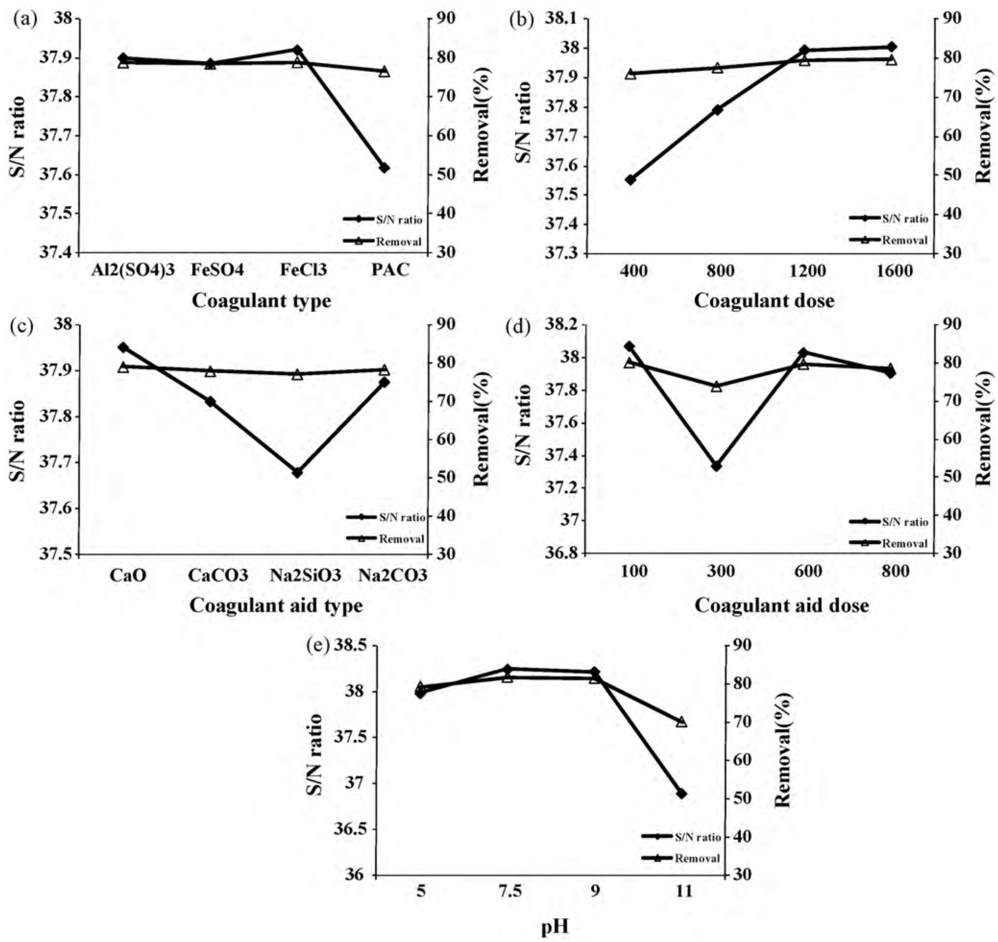

Fig. 2. The effect of experimental parameters on the S/N ratio and measured removal percent in the removal of chromium.

tal results were entered in Taguchi method as the value of residual turbidity).

$$\mathrm{S/N} = -10\ \log\left(\frac{1}{n}\sum y_i^2\right) \tag{2}$$

n is the number of repetitions for an experiment and y_i is the performance value of ith experiment [22]. In both cases, the larger S/N ratio implies the better achievement of treatment goal.

The optimization of the factors which are effective on the coagulation–flocculation process using traditional techniques, is a terrible task, which consumes considerable time and cost. But the Taguchi method helps to solve such an optimization problem by minimum number of experiments.

2.4. ANOVA analysis

ANOVA is used to estimate error variance and to determine the relative importance of various factors. It indicates the effect of each investigated factor on the optimization criterion. ANOVA also demonstrates whether the observed variation in the response is due to the alteration of level adjustments or experimental standard errors. In ANOVA analysis, the values of sum of squares, degree of freedom (DOF), mean square (variance) and associated F-test of significance (F) were calculated. Sum of squares (SS) of factor A is calculated as follows:

$$SS_A = \left[\sum_{i=1}^{k_A}\left(\frac{A_i^2}{n_{A_i}}\right)\right] - \frac{T^2}{N} \tag{3}$$

k_A is the number of levels for factor A ($k_A = 4$ for all factors in this study), n_{Ai} is the number of all observations at level i of factor A ($n_{Ai} = 4$ in this study), A_i is the sum of all observations of level i of factor A, N is the number of all experiments and T is the sum of all observations. P, is the percent of contribution of each factor on the response ($P_A = (SS_A/SS_T) \times 100$) where SS_T is the sum of squares for all factors [18].

3. Results and discussion

The resulted S/N ratios and their corresponding measured removal percents of COD, chromium, TDS and turbidity of the wastewater are shown in Figs. 1–4 for the experimental conditions proposed by Taguchi method. The S/N ratios show the effect of each level of each factor on the response, independently. It is calculated by averaging the S/N ratio values of all the experiments where that level of that factor has been used. For instance, one of the levels of factor D (coagulant aid type) is CaO. According to Table 2, experiments corresponding to this level of coagulant aid type are numbers 1, 7, 12 and 14. Consequently, the S/N ratio value of this level of coagulant aid type (CaO), is equal to the average of the S/N values obtained from those experiments. The measured removal percents (Figs. 1–4) have been obtained using a similar manner.

3.1. Optimized conditions for COD removal

Fig. 1 shows that $FeCl_3$ as coagulant, results in maximum COD removal while Al_2SO_4 has the minimum efficiency. 7.5 is the

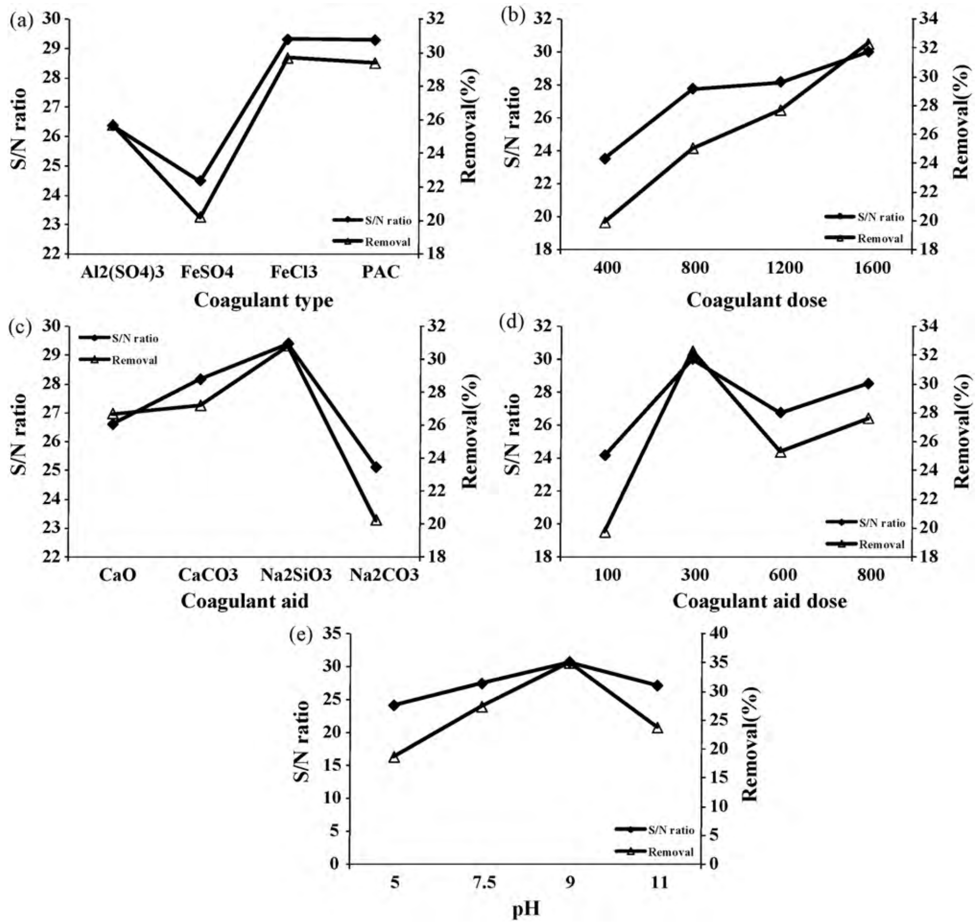

Fig. 3. The effect of experimental parameters on the S/N ratio and measured removal percent in the removal of TDS.

best among pH values and the best COD removal percentage was obtained by Na_2CO_3 as coagulant aid.

The quality of raw tannery wastewater affects on the efficiency of coagulation process [6]. The optimum dose of coagulant is dependent on the amount of parameters such as TSS, TDS and concentration of pollutants (Cr, S^{2-}, N, etc.), so it is better determined by jar test [6]. In this work, the best levels of the doses of coagulant and coagulant aid were determined equal to 800 and 600 ppm, respectively, to have the maximum COD removal efficiency. Considering the "zone" concept introduced in the Introduction section, it was observed that coagulant zone shifted from zone II to zone III when coagulant dose increased over 800 ppm and consequently COD removal percent decreased.

3.2. Optimized conditions for chromium removal

Fig. 2 shows that $FeCl_3$ has the best efficiency in chromium removal compared with other studied coagulants. According to the figure, pH 7.5 and 9 are more effective in chromium removal than other studied pH values. High pH values, lead to the formation of insoluble $Cr(OH)_3$ which is removed as chromium sludge. Therefore, coagulation should be operated at alkaline pH range to achieve maximum chromium removal [6]. The obtained results for the effect of pH on the removal efficiency of chromium are in good agreement with previous studies [6].

According to the results, when CaO was used as coagulant aid, the chromium removal was maximum. The effect of CaO on the removal of Cr(III), as the major constituent of the total chromium, is as follows:

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad (4)$$

$$2Cr^{3+} + 3Ca(OH)_2 \rightarrow 2Cr(OH)_3 + 3Ca^{2+} \quad (5)$$

Coagulant aids are used to achieve the optimum conditions for coagulation and flocculation process. They are used for faster floc formation and to produce denser and stronger flocs, decrease the coagulant dose, broaden the proper pH range, and improve the removal of turbidity and other impurities [12]. In this study, CaO was found as the most effective coagulant aid. CaO is an alkaline coagulant aid. Alkaline coagulant aids must be added to the waters without sufficient natural alkalinity which is necessary to react with acidic metallic coagulants to produce good flocs [12].

The optimum level of coagulant dose for chromium removal was determined equal to 1600 ppm. Alum ($Al_2(SO_4)_3$) or ferric chloride ($FeCl_3$) coagulants, in relatively high concentrations, react with hydroxides [23] to form $Al(OH)_3$ or $Fe(OH)_3$ sediments, respectively. The colloidal particles are entrapped in these sediments either during floc formation or just after. This type of coagulation by enmeshment of colloids in flocs is commonly called sweep coagulation [12]. Therefore, with an increasing in coagulant dose, the effect of sweep coagulation on chromium removal is increased. Also, the best level of coagulant aid dose was obtained equal to 100 ppm.

3.3. Optimized conditions for TDS removal

Fig. 3 shows the efficiency of the different levels of studied factors on TDS removal. It can be seen from the figure that $FeCl_3$

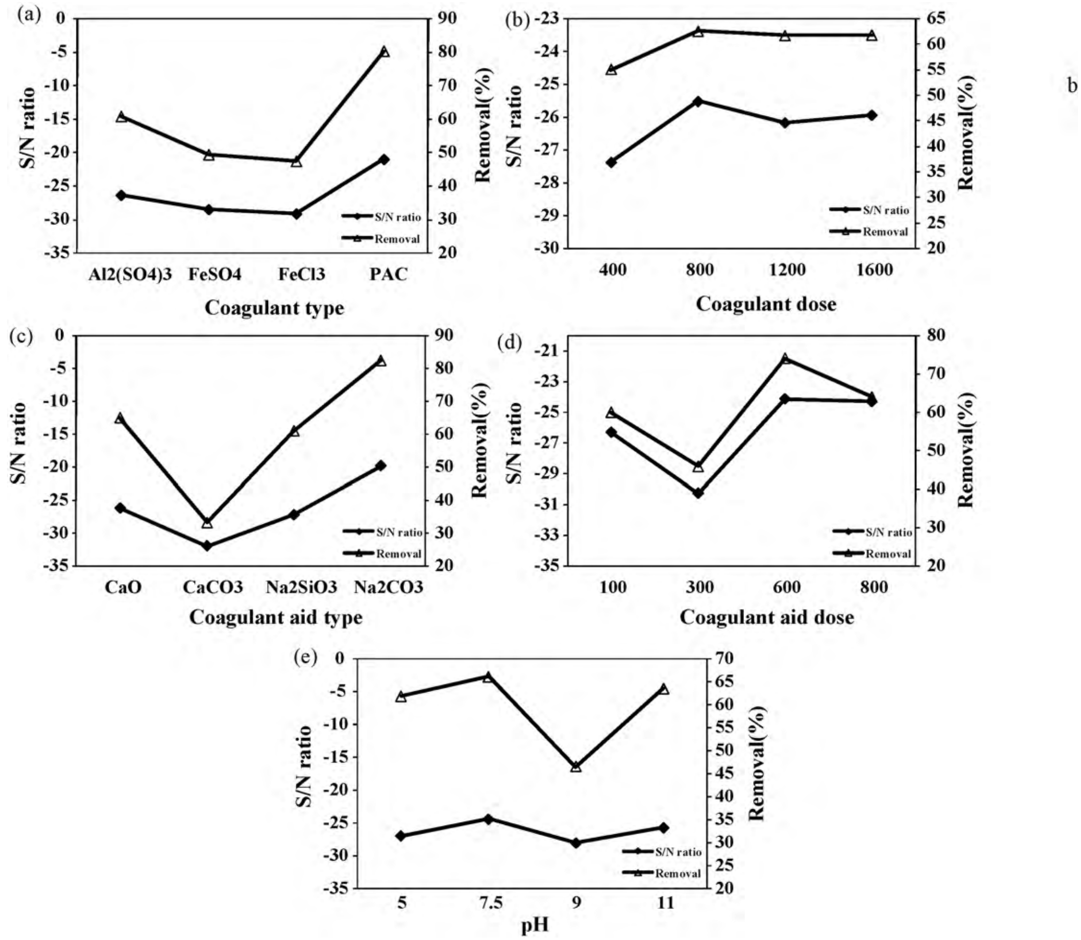

Fig. 4. The effect of experimental parameters on the S/N ratio and measured removal percent in the removal of turbidity (the S/N ration was calculated according to the value of residual turbidity).

has more efficiency than the other coagulants, and increasing the coagulant dose increases TDS removal which may be due to an increasing in sweep coagulation. The optimum pH for TDS removal was obtained equal to 9. Also, Na_2SiO_3 was found as the best coagulant aid type with 300 ppm as the best dose for TDS removal.

3.4. Optimized conditions for turbidity removal

The optimization of conditions for the removal of turbidity was done using residual turbidity as treatment goal. Fig. 4 shows that $Al_2(SO_4)_3$ and PAC coagulants had more efficiency than iron-based salts in turbidity removal. Samples treated by $Al_2(SO_4)_3$ and PAC were significantly lighter in color than those treated by iron-based salts. It was observed that at pH 9 and 11 the color of the samples turned to black when iron-based salts were used. Considering the results, PAC was the best coagulant for turbidity removal and maximum removal efficiency was obtained at pH 7.5. Also, Na_2CO_3 was the best coagulant aid to remove the turbidity and the best levels for coagulant dose and coagulant aid dose were 800 and 600 ppm, respectively.

3.5. ANOVA results

ANOVA results of the data obtained from jar tests have been presented in Table 3. The last section in Table 3 shows the percent of contribution (*P*, %) of each factor to response. The percent of contribution shows the influence of one factor on the total observed variance in the experiments. A higher value of the percent of contribution means that the factor contributes more to response. It can be observed that coagulant type has the most contribution (90.712%), among the factors, on COD removal.

According to Table 3 the factors, which have the most influence on the removal of chromium, are pH and coagulant aid dose, respectively. Also in TDS and turbidity removal, the effects of coagulant dose and coagulant aid type are higher than the other factors, respectively.

The optimum conditions found in Figs. 1–4 for each of COD, chromium, TDS and turbidity removal are given in Table 4. In addition, the current grand average of performance, the value of contribution of each factor and the expected S/N results at optimized conditions, determined by Taguchi method, are shown in this table.

3.6. Choosing the optimum conditions considering all pollution parameters as treatment goal

In the past sections, we reported the optimum conditions considering each of COD, chromium, TDS and turbidity as treatment goal (Table 4). The effect of each of these optimum conditions on the removal of studied pollution parameters was as follows.

In the optimum conditions for the removal of COD, 82.6% COD removal, 81% chromium removal, 36% TDS removal and 85.9% turbidity removal, and using the optimum conditions for the removal of chromium, 72.5% COD removal, 84.7% chromium removal, 32.2% TDS removal and 76% turbidity removal were achieved. By using the optimum conditions for the removal of TDS, 73.3% COD removal,

Table 3
Analysis of variance (ANOVA) of the data resulted from experimental design.

Factor	DOF				Sum of squares				Variance				Pure Sum				Percent, *P* (%)			
	COD removal	Cr removal	TDS removal	Turbidity removal	COD removal	Cr removal	TDS removal	Turbidity removal	COD removal	Cr removal	TDS removal	Turbidity removal	COD removal	Cr removal	TDS removal	Turbidity removal	COD removal	Cr removal	TDS removal	Turbidity removal
Coagulant type	3	3	3	3	117.0	0.3	67.3	165.4	39.0	0.1	22.5	55.2	117.0	0.3	165.4	67.3	90.7	3.5	18.9	27.5
Coagulant dose	3	3	3	3	3	0.5	90.2	7.7	1	0.2	30.1	2.6	3	0.6	7.7	90.2	2.3	7.4	25.2	1.3
Coagulant aid type	3	3	3	3	1.2	0.2	38.3	301.4	0.4	0.1	12.9	100.5	1.2	0.2	301.4	38.3	0.9	2.2	10.7	50
Coagulant aid dose	3	3	3	3	2.2	1.4	75.6	97.7	0.7	0.5	25.2	32.6	2.2	1.4	97.7	75.5	1.7	19.0	21.2	16.2
pH	3	3	3	3	5.6	5	85.5	29.9	1.9	1.7	28.5	10	5.6	5	29.9	85.5	4.4	67.9	24	5
Other/error	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total	15	15	15	15	129.0	7.4	356.9	602.1									100.0%	100.0%	100.0%	100.0%

Table 4
Optimum conditions and contribution of each factor in the removal of pollution parameters according to Taguchi method.

Column/factor	COD removal		Cr removal		TDS removal		Turbidity removal	
	[a]Level	[b]Contribution	Level	Contribution	Level	Contribution	Level	Contribution
Coagulant type	$FeCl_3$	2.7	$FeCl_3$	0.1	$FeCl_3$	2	PAC	5.3
Coagulant dose	800 ppm	0.7	1600 ppm	0.2	1600 ppm	2.6	800 ppm	0.7
Coagulant aid type	Na_2CO_3	0.2	CaO	0.1	Na_2SiO_3	2.0	Na_2CO_3	6.5
Coagulant aid dose	600 ppm	0.6	100 ppm	0.2	300 ppm	2.7	600 ppm	2.1
pH	7.5	0.9	7.5	0.4	9	3.3	7.5	1.9
Total contribution from all factors	5		1.0		12.6		16.5	
Current grand average of performance (S/N)	34.8		37.8		27.4		−26.2	
Expected result at optimum conditions (S/N)	39.8		38.9		40		−9.7	

[a] In this table, the “Level” is the amount or the compound that is the optimum one, among the examined ones, in the removal of a pollution parameter such as COD.

[b] The extent in which a factor influences on the removal of a parameter such as COD. Some factors have more influence on the removal of pollution parameters than the others.

76.7% chromium removal, 39.7% TDS removal and 32.6% turbidity removal, and in the optimum conditions for the removal of turbidity, 75.3% COD removal, 73.2% chromium removal, 32.6% TDS removal and 91.2% turbidity removal were resulted.

According to the data reported above, it can be seen that the conditions, which are optimum for COD removal, can cause acceptable removal percents for other studied parameters, i.e. chromium, TDS and turbidity. Therefore, these conditions can be considered as optimum for the removal of all studied pollution parameters, as treatment goal, in this work.

4. Conclusion

The results of this study, designed by Taguchi method, showed that coagulation–flocculation process is able to remove COD, chromium, TDS and turbidity of tannery wastewater. In addition, it can be concluded that Taguchi experimental design method is suitable to find the optimum conditions of tannery wastewater treatment and reduces the cost and the time, which are necessary to find the optimum conditions. Taguchi method also can be used to determine the importance of each of studied factors in wastewater treatment.

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