



Optimizing potassium ferrate for textile wastewater treatment by RSM

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Abstract

Background: Application of potassium ferrate is a chemical oxidation approach used for water and wastewater treatment. The aim of this study is to apply central composite design (CCD) and response surface methodology (RSM) to optimize potassium ferrate consumption in the treatment of wastewater from carpet industries.

Methods: Samples in this experimental study were collected from wastewater, originating from a carpet factory. Wastewater sampling was carried out monthly for a period of two seasons. Ferrate oxidation experiments were conducted by means of a conventional jar-test apparatus. The time and speed for mixing were set with an automatic controller. Parameters of study were measured based on given methodologies in Standard method for examining water and wastewater. CCD and RSM were applied to optimize the operating variables including potassium ferrate dosage and pH.

Results: Results showed that potassium ferrate concentration (A), pH (B), their interactions (AB) and quadratic effects (A² and B²) were significant in the removal of COD, turbidity, color and TSS from carpet industries effluents. At an optimum point (COD: 160 mg/L of potassium ferrate and pH 4, turbidity: 165 mg/L of potassium ferrate and pH 4, color and TSS: pH 4.5 and 150 mg/L of potassium ferrate) removal efficiencies for COD, turbidity, color and TSS were 86, 86, 87 and 89%, respectively.

Conclusion: Potassium ferrate has a significant impact on pollutants decomposition and the removal of color from wastewater produced in carpet industries. This process can be employed for the pretreatment or post treatment of wastewaters containing refractory organic pollutants. CCD and RSM are suitable tools for experimental design.

Keywords: Advanced treatment, carpet wastewater, Statistical experiment design, Response Surface Methodology.

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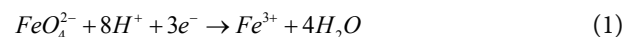
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Introduction

A large amount of wastewater produced in textile industries emerges from color containing units with a wide range of contaminants that must be treated before being discharged into the environment. Wastewater originating from color-containing effluents embodies a high COD, large fluctuations, high pH and temperature (1). Moreover, in many cases, these effluents are toxic and lead to ecosystem damage by producing some hazardous by-products. Application of potassium ferrate as an advanced approach is appropriate for the treatment of such types of wastewater (2,3).

Potassium ferrate (K₂FeO₄) is a ferrous salt (iron salt) containing an uncommon ion with oxidation valence +6. The ion (FeO₄)²⁻ is a strong oxidant and its oxidizing potential in acidic and alkaline conditions is 2.2 and 0.72 V

respectively. As potassium ferrate is reduced to Fe³⁺ ions or ferric hydroxide during oxidation process, it is capable of conducting coagulation. As a result, potassium ferrate has two functions in water and wastewater treatment: oxidation and coagulation. Hence, it is recommended for the removal of environmental contaminants. This chemical compound is a green chemical (4,5) and its reaction in aquatic environment is shown in Eq. (1).



Ferrate (VI) has been successfully utilized in water and wastewater treatment (6). Currently, some researches has been carried out regarding the removal of pharmaceutical contaminants and micro pollutants utilizing ferrate (VI) and the results showed the effective impact of this com-



pound in decomposition of such contaminants in aquatic environment.

The limitations of classical methods include high time consumption due to numerous experiments and high cost of consumables that can be omitted by statistical experimental designs. Furthermore, in this approach, it is impossible to examine the interactions among variables (7). Response surface methodology (RSM) is an appropriate method that can resolve these limitations. In this study, RSM was employed to optimize variables influencing the elimination of chemical oxygen demand (COD), color, turbidity and TSS from wastewater. RSM was derived from statistical and mathematical technique. It can be used to study the effect of several factors at three or five level as well as their influence on each other (8). In this study, central composite design (CCD) was utilized for experimental design. It is an effective design that does not need a greater number of experimental runs (9). Moreover, experimental designs such as CCD model provides valuable data on direct, pair wise interaction, and curvilinear variable effects (7). This study uses CCD and RSM to optimize potassium ferrate consumption in the treatment of wastewater from textile industry.

Methods

Experimental setup and procedure

Samples were collected from a carpet factory's effluents. The colors utilized in this industry were of azo and anthraquinone types. Sampling was carried out monthly and during summer and fall. The samples were transferred to the laboratory of Qazvin University of Medical Sciences in less than 48 hours at 4°C. The conductivity and pH of the samples were measured with a Multi WTW 340i instrument (Germany). Total organic carbon (TOC) content of the samples was determined employing an ANATEC TOC analyzer (model ANATEC II Australia). Total solids (TS), total suspended solids (TSS), total volatile solids (TVS), total dissolved solids (TDS), turbidity, alkalinity, acidity and COD were determined based on standard methods for the examination of water and wastewater (10).

Ferrate oxidation experiments were conducted by means of a conventional jar-test (zag-shimi, Iran) apparatus with impellers equipped with six of 1.5 cm × 4.5 cm rectangular blades. The time and speed for mixing were set with an automatic controller. Jar testing steps include rapid mix with the mixing speed of 200 rpm for one minute, slow mix with the mixing speed of 40 rpm for 20 minutes and settlement for 60 minutes. Potassium ferrate (97%) was provided by Sigma-Aldrich Corporation (USA). Other chemicals were of analytical grade and purchased from Merck (Germany). In order to adjust pH, 0.1M sulfuric acid and 0.05 L of sodium hydroxide solution was used. A total of 30 preliminary experiments were carried out to determine pH range and the amount of ferrate needed, based on literature reviews (3). The active ferrate solution (1 g/L) was prepared by adding the solid form of K_2FeO_4 to 0.005 M Na_2HPO_4 solution and 0.001M borate buffer at pH 9.

Experimental design and model development

In this study, determination of optimum conditions (pH and potassium ferrate consumption) for the removal of COD, color, turbidity and TSS was carried out employing CCD/RSM procedures. Design Expert software (Stat-Ease Inc., Minneapolis, USA) was applied for designing, statistical modeling and optimization. Model statistics computed were F-value, Lack of fit, coefficient of determination (R^2), and coefficient of variation (CV). Generally, a good model must be significant based on *P* value and F value as opposed to the lack of fit (insignificant).

The independent variables were concentration of potassium ferrate (mg/L) and pH denoted by A and B respectively. Furthermore, the removal efficiencies of COD, turbidity, color and TSS were considered as response variables that are denoted by Y_1 , Y_2 , Y_3 and Y_4 , respectively. Performance of the procedure was evaluated by assessing the efficiency of COD, turbidity, color and TSS removals. The variables were coded according to Eq. (2):

$$x_i = \frac{x_i - x_0}{\Delta x} \quad i=1,2,3,\dots,k \quad (2)$$

Where x_i is the coded value of the independent variable, x_0 is the value of x_i at the center point and Δx is the difference of variable values. The maximum, central and minimum values (+1, 0, -1) of each variable was specified based on CCD. In this study, CCD included 2^k fractional factorial points ($k=2$), $2k$ axial points and 5 center points. Thus, 13 experiments including 4 factorial points, and 4 axial points were carried out. In order to evaluate the pure error, 5 additional experiments were carried out at the center point. Following the analysis of experimental data, optimal conditions were graphically determined. Determination of optimum conditions for several parameters was carried out by overlaying of plots called graphical optimization. Graphical optimization reveals the area of feasible response values in the factor space and the regions that do fit the optimization criteria would be shaded (7,8).

To confirm the results obtained from experimental data, two additional experiments were carried out in optimum conditions and the results were compared. System behavior was described by Eq. (3) which is an experimental quadratic polynomial model.

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{\substack{i=1 \\ i \leq j}}^k \sum_{j=1}^k \beta_{ij} x_i x_j + \dots + e \quad (3)$$

Where Y , i , j , β , e and k represent the response variable, linear constant, quadratic constant, regression constant, random error as well as the number of parameters, respectively.

The minimum and maximum range of variables was investigated along with the full experimental plan, with respect to their values in actual and coded form as listed in Table 1.

Results

The results of qualitative analysis of the wastewater are shown in Table 2.

Table 1. Independent variables and limit level for response surface study

Variables	Unit	Coded symbols	Levels				
			-1	-0.5	0	+0.5	+1
Doses of potassium ferrate	mg/L	A	50	75	150	175	200
pH	-	B	3	3.75	4.5	5.25	6

Based on the 13 experimental results, the CCD for the experimental variables is shown in Table 3.

According to the obtained results, the highest removal efficiencies for COD, turbidity, color and TSS were 90%, 87%, 89.6% and 91%, respectively.

The results obtained from analysis of variance (ANOVA) with regards to the removal of COD, turbidity, color and TSS utilizing potassium ferrate are shown in Table 4. As shown in Table 4, all the terms in the statistical quadratic model were significant ($P \leq 0.0001$). Lack of fit test for COD, turbidity, color and TSS removal ($P = 0.4560, 0.7661, 0.4735, 0.7152$, respectively) were not significant. The variables R^2 (coefficient of determination), $A.R^2$ (adjusted R^2), $A.P$ (adequate precision) and $C.V$ (coefficient of variance) are shown in Table 4. R^2 and $A.R^2$ were found to be 0.96, 0.99, 0.99 and 0.98, 0.94, 0.97, 0.98 and 0.97 for the removal of COD, turbidity, color and TSS respectively. The obtained values of AP and $C.V$ for the mentioned parameter were 19.21, 30.13, 30.32 and 22.3, 3.4, 2.05, 3.4 and 3.83 respectively.

The ANOVA results for each response (Table 4) indicated that the linear of two independent variables (A and B),

Table 2. Characteristics of raw wastewater from the carpet factory

Parameters	Mean \pm SD
pH	6.54 \pm 1.2
EC ($\mu S/Cm$)	950 \pm 20
TOC (mg/L)	700 \pm 30
COD (mg/L)	1600 \pm 50
TSS (mg/L)	280 \pm 20
TDS (mg/L)	900 \pm 30
TVS (mg/L)	850 \pm 40
Turbidity (NTU)	554 \pm 15
Alkalinity (mg/L as $CaCO_3$)	180 \pm 30
Acidity (mg/L as $CaCO_3$)	40 \pm 3.5

Table 3. CCD for the experimental variables

No.	Run	pH	Potassium ferrate (mg/L)	Removal efficiency (%)			
				COD	Turbidity	Color	TSS
1	4	6	200	75	75	55	50.4
2	10	4.5	150	85	84	83.4	90.1
3	1	3	50	75	71	49	44
4	11	4.5	150	90	87	85.7	90
5	12	4.5	150	87	87	88	83
6	13	4.5	150	83	83	89.6	91
7	2	6	50	50	52	35	45
8	8	4.5	175	83.9	85	85.5	85.2
9	9	4.5	150	85	84	88.4	87.4
10	3	3	200	65	67.5	54.3	46.3
11	5	3.75	150	80	82	78.5	86
12	6	5.25	150	82	82	80	77.8
13	7	4.5	75	81.5	80	77.8	78

Table 4. ANOVA results for the removal of COD, turbidity, color and TSS using potassium ferrate

Source	Sum of squares	df	Mean squares	F-value	P value
COD removal (%)					
Model	1330.18	5	266.04	36.91	<0.0001
A	43.56	1	43.56	6.04	0.0436
B	58.32	1	58.32	8.09	0.0249
AB	306.25	1	306.25	42.49	0.0003
A ²	41.08	1	41.08	5.7	0.0483
B ²	9.31	1	9.31	1.29	0.2931
Residual	50.45	7	7.21		
Lack of fit	22.45	3	7.48	1.07	0.4560
Pure error	28	4	7		
Turbidity removal (%)					
Model	1167.81	5	233.56	90.24	<0.0001
A	29.39	1	29.39	11.35	0.0119
B	107.56	1	107.56	41.56	0.0004
AB	175.56	1	175.56	67.83	<0.0001
A ²	25.57	1	25.57	9.88	0.0163
B ²	16.55	1	16.55	6.4	0.0393
Residual	18.21	7	2.59		
Lack of fit	4.12	3	1.37	0.39	0.7661
Pure error	14	4	3.5		
Color removal (%)					
Model	3916.35	5	783.27	127.7	<0.0001
A	35	1	35	5.71	0.0483
B	188.83	1	188.83	30.79	0.0009
AB	54.02	1	54.02	8.81	<0.0209
A ²	138.64	1	138.64	22.6	0.0021
B ²	49.47	1	49.47	8.07	0.0250
Residual	42.93	7	6.13		
Lack of fit	18.57	3	6.19	1.02	0.4735
Pure error	24.37	4	6.09		
TSS removal (%)					
Model	3717.15	5	743.43	90.55	<0.0001
A	54.43	1	54.43	6.63	0.0368
B	179.24	1	179.24	21.83	0.0023
AB	49	1	49	5.97	<0.0446
A ²	70.6	1	70.6	8.6	0.0219
B ²	97.69	1	97.69	11.9	0.0107
Residual	57.47	7	8.21		
Lack of fit	15.15	3	5.05	0.48	0.7152
Pure error	42.32	4	10.58		
Other statistical parameters					
	R²	Adj. R²	Mean \pm SD	AP	CV
COD	0.96	0.94	78.65 \pm 2.68	19.21	3.4
Turbidity	0.99	0.97	78.4 \pm 1.61	30.13	2.05
Color	0.99	0.98	73.1 \pm 2.48	30.32	3.4
TSS	0.98	0.97	74.78 \pm 2.87	22.3	3.83

quadratic effects (A^2 and B^2) and their interactions (AB) were significant ($P \leq 0.0001$) in the removal of COD, turbidity, color and TSS. As presented in Table 4, the Fisher coefficient (F-value) for the removal of COD, turbidity, color and TSS were significant.

The final quadratic equations for the removal of the study parameters were obtained as shown in Eq. (4) to (7).

$$\text{COD removal (\%)} Y_1 = 85.2 - 3.11A + 3.6B + 8.75AB - 12.98A^2 - 6.18B^2 \quad (4)$$

$$\text{Turbidity removal (\%)} Y_2 = 84.82 - 2.56A + 4.9B + 6.62AB - 10.24A^2 - 8.24B^2 \quad (5)$$

$$\text{Color removal (\%)} Y_3 = 86.27 - 2.79A + 6.48B + 3.67AB - 23.84A^2 + 14.24B^2 \quad (6)$$

$$\text{TSS removal (\%)} Y_4 = 87.6 - 3.48A + 6.31B - 3.5AB - 17.01A^2 - 20.01B^2 \quad (7)$$

The model equations were employed to generate 3D contour plots. The 3D contour plots for the effective factors in eliminating COD, turbidity, color and TSS are plotted in Figure 1. As shown in the plots, all responses have an optimum removal point. With respect to Figure 1A and 1B, the optimum pH for the removal of COD and turbidity was 4, while it was 4.5 for color and TSS removal (Figure 1C and 1D). Also, the optimum dose of potassium ferrate for the removal of COD and turbidity were obtained as 160 and 165 mg/L, respectively, while it was 150 mg/L for color and TSS removal. In this condition, removal efficiencies for COD, turbidity, color and TSS were 86%, 86%, 87% and 89%, respectively.

By overlaying four 3D plots, the optimum removal efficiencies for COD, turbidity, color and TSS were obtained as 84%, 85%, 85% and 87%, respectively. In this condition,

the dose of potassium ferrate and pH value was obtained as 160 mg/L and 4.3 respectively after overlaying four 3D plots (Figure 2).

Based on the results obtained from the final quadratic model, to confirm the reliability of the model, two additional laboratory experiments were carried out utilizing the optimum conditions suggested by the Design-Expert software as shown in Table 5.

The optimum conditions were obtained at pH 4.3 and 160 mg/L potassium ferrate concentration. At optimum conditions, COD, turbidity, color and TSS efficiency were obtained 84.5, 85, 85.3 and 88.3% respectively.

Discussion

The quality of quadratic polynomial model fit was expressed by R^2 and $Adj.R^2$. R^2 should be close to 1, as this

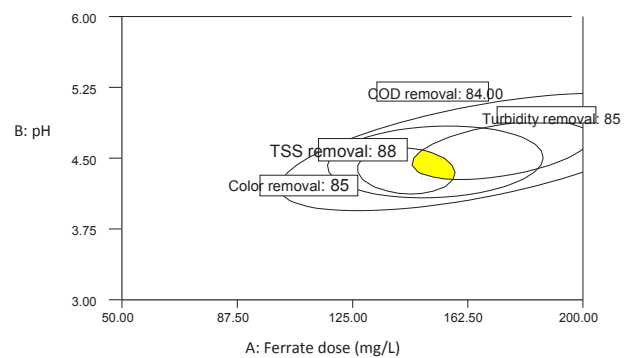


Figure 2. The overlaying plots for the removal of COD, turbidity, color and TSS

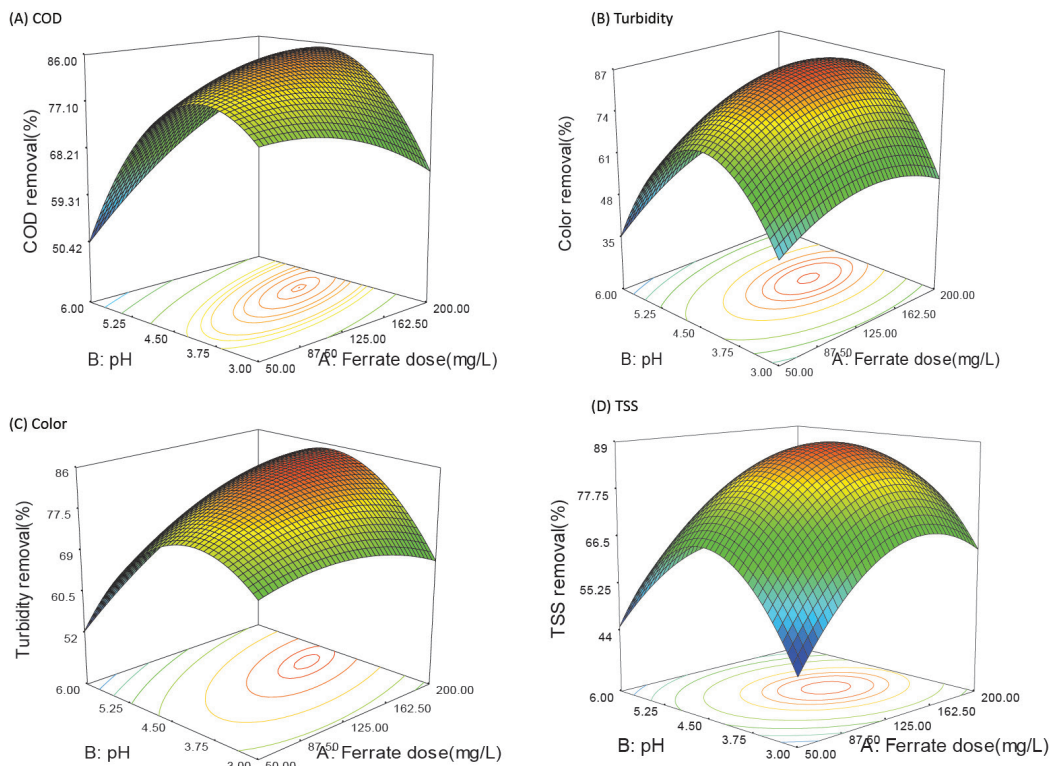


Figure 1. The 3D contour plots for the removal of COD, turbidity, color and TSS by potassium ferrate.

Table 5. Verification of experimental results at optimum conditions

Optimum condition (pH 4.3 and ferrate dose 160 mg/L)	COD removal efficiency (%)	Turbidity removal efficiency (%)	Color removal efficiency (%)	TSS efficiency (%)
Experimental results	83%	84.1%	84.5%	88%
Model response	84.5%	85%	85.3%	88.3%
Error	1.5	0.9	0.7	0.3
Standard deviation	0.7	0.43	0.3	0.2

would show better correlation between the predicted and experimental values. A high R^2 coefficient ensures a satisfactory adjustment of the quadratic model to the experimental data. As a result, an optimal agreement with the adjusted R^2 is important (8,9). In this research, in all cases, R^2 coefficient was high. The high values of R^2 suggest the high agreement of the obtained experimental data, with the data evaluated by the model. So, it can be said that the quadratic model has a good fitness for the data. Adequate precision is a measure of the range in predicted response relative to its associated error, in other words, a signal-to-noise ratio. Its desired value is 4 or more (11,12). In this study, AP values for the removal of COD, turbidity, color and TSS were greater than 4 indicating the existence of adequate signal and the high capability of this model in predicting the results. Coefficient of variance (CV) estimates the ratio of the standard error to the mean value of the observed responses (as a percentage) and also describes the reproducibility of models. Normally, we can consider a model to be reproducible when its CV is not greater than 10% (9). CV was within the acceptable range of 10% which proves the reproducibility of the model and high precision in the results. All of these terms indicated a good fitting when there is high correlation between the observed and predicted values. The lack of fit (LOF) F-test describes the variation of data around the fitted model. If the model does not fit the data well, the lack of fit test will be significant (8). Lack of fit test for COD, turbidity, color and TSS responses were not significant, thus it shows a good fitting of the data to the model.

The response surface plots were approximately symmetrical in shape with circular contours. All response plots showed clear peaks, indicating that optimal conditions for the maximum responses depend on the dose potassium ferrate and pH in the design space. Initial concentration of ferrate and initial pH value has a significant influence in the oxidative ability of the ferrate ion. With respect to Figure 1, the interaction effects of ferrate dose and pH on responses, demonstrate concentric closed curves. The centers represent the optimum conditions, indicating that experimental design has been well carried out. Furthermore, the experimental results which were obtained from the optimum conditions, showed the precision of the resulting data and models.

Previous studies have reported that potassium ferrate can also eliminate a range of metals (e.g., Fe^{2+} , Fe^{3+} , Mn^{2+} , and Cu^{2+}) and toxic heavy metals (e.g., Pb^{2+} , Cd^{2+} , Cr^{3+} , and Hg^{2+}) to a low level at a dose range of 10–100 mg/L as K_2FeO_4 (13, 14).

The results of a study carried out on the application of

70 mg/L potassium ferrate for the treatment of color-contained textile wastewater, indicated that high removal efficiencies of COD, color and turbidity were 91%, 50% and 93%, respectively. Except the color removal efficiency, other variables were consistent with current study (15). This can be due to different characteristics of textile wastewater.

Some researchers employed encapsulated potassium ferrate (VI) for the removal of DNBP (dinitro butylphenol). The optimal conditions regarding the degradation of DNBP were pH 6.5 and encapsulated concentration of 1.2 g/L. They also reported that encapsulated potassium ferrate (VI) was very effective in COD reduction as well as the decolorization of real wastewater from the manufacturing of DNBP. In addition, they stated that the removal of DNBP increased with encapsulated Fe (VI) concentration. The DNBP removal increased with corresponding efficiency from 68% to 93% when the encapsulated Fe (VI) dosage increases from 0.4 to 1.2 g/L (16). High consumption of ferrate for the degradation of DNBP may be as a result of complex structure of this compound.

The results of a study showed the highest removal efficiencies for Ibuprofen and Ciprofloxacin by potassium ferrate in pH 4 and 5-6, respectively (17). Moreover, another research reported complete removal of flurbiprofen from aqueous solutions by ferrate in pH 4 (18), which were in agreement with our study.

The results of a research carried out on the removal of bisphenol compounds by ferrate (VI) indicated that when pH increases from 3 to 5, bisphenol removal efficiency by ferrate (VI) would increase from 85.1% to 95.2% (19). Our results were in line with those of the mentioned study.

A study carried out in relation to the removal of turbidity and COD in the batch reactor with ferrate dose of 15 mg/L for secondary wastewater treatment, showed a significant improvement with respect to the removal of suspended solids (70.8%) and turbidity (78%) (20). The low consumption of potassium ferrate when compared to the findings of our study may be as a result of low concentration of organic matters in secondary effluent.

A report obtained on the comparative performance of potassium ferrate (VI) and ferric sulphate for the removal of COD, color and turbidity in sewage treatment, showed that potassium ferrate (VI) could reduce COD and organic contaminants in comparison with ferric sulphate for the same doses utilized. Furthermore, the volume of sludge produced by potassium ferrate (VI) was much less than ferric sulphate, which ought to make subsequent treatment of sludge easier.

Conclusion

Potassium ferrate is an effective compound for the treatment of wastewater from carpet factories. However, the limitations of the ferrate application are related to the instability of its solutions along with the complicated preparation and storage of this compound.

CCD of RSM can be utilized to determine the significant variables and optimum condition for textile wastewater treatment with respect to COD, TSS, turbidity, and color removal. Potassium ferrate concentration (A), pH (B), their interactions (AB) and quadratic effects (A^2 and B^2) were significant in the removal of COD, turbidity, color and TSS from carpet industries effluents. The statistical approach has been confirmed to be a powerful tool in studying textile wastewater treatment utilizing potassium ferrate.

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Ethical issues

The authors certify that all data collected during the study is as stated in this manuscript, and no data from the study has been or will be published separately elsewhere.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

All authors contributed equally to data collection, and article approval.

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