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Treatability of raw textile wastewater using Fenton process and its comparison with chemical coagulation

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ABSTRACT

The textile industry has significant effects on the environment due to the intensive water consumption and the use of toxic raw materials. Every succeeding day, water quality to receive water environments is deteriorating due to the high amount of wastewater generated by the textile sector. In this study, the performance of the Fenton Process has been optimized to minimize the damage of acrylic yarn dye-house wastewater to the environment regarding operating conditions, Fe(II) and H_2O_2 dosages, pH and reaction time using the response surface methodology method over chemical oxygen demand (COD), colour and total organic carbon (TOC) measurement parameters. The results were compared with the results of chemical coagulation. The most effective parameter of the Fenton Process was determined as Fe(II) dosage for this kind of wastewater sample. As a result of the optimization, efficiencies by 82.8%, 96.2%, and 75.6% were obtained for COD, colour and TOC removal, respectively, by means of the Fenton process. The total treatment cost was determined as 1.216 € per 1 m^3 textile wastewater. According to the experimental results, both processes were found to be effective for colour removal, whereas chemical coagulation was found to be insufficient for COD removal. When the two processes were compared in terms of sludge amount, the Fenton process was found to produce less sludge by 1.60 kg sludge/kg COD.

Keywords: Textile wastewater; Acrylic yarn dye; Fenton process; Chemical coagulation

1. Introduction

The industrial activities are very important in terms of polluting sources. The textile industry is the primary sector for the prevention of industrial pollution and the protection of water resources. The textile industry has a significant added value in the export and employment of the developing countries. It requires the necessary work to be carried out in this regard. Textile industry wastewater generally contains high chemical oxygen demand (COD), strong colour and low biodegradable pollutants [1]. Many subdivisions of this industry produce wastewater in high volumes containing complex pollutants.

Particularly in the pre-treatment (washing) and colouring processes (dying), significant amounts of water is consumed. Most of the azo group dyes commonly used in the colouring process are known to produce toxic effects when are given to the receiving water environments [2]. Because the wastewater of the textile industry has very variable characters, and this makes it necessary to consider each production plant separately instead of choosing a certain method for treatment methods. Conventional treatment methods include

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biological processes, physical-chemical processes, adsorption and chemical oxidations [3–5].

The advance oxidation processes (AOPs) which are the most preferred treatment methods, especially for colour removal, is the preferred treatment method in domestic wastewater and industrial wastewater treatment. The use of AOPs in the treatment of industrial wastewater is still a current issue by being highly effective in the disintegration of organic pollutants and colours. In AOP methods, reactive oxidant species are produced and provided for the treatment. The Fenton process oxides these species by the oxidation power of $^{\bullet}OH$ radical ($^{\bullet}OH$: 2.80 V), which is known as the strongest oxidant by following fluorine gas (F2: 3.06 V) [6] and is generated by the disintegration of hydrogen peroxide in general [7]. When the H_2O_2 and Fe(II) ion are used together at low pH (pH ≈ 3,0), the OH radical and a complex reaction chain are formed [8–10] as in the following.

$$Fe^{+2} + H_2O_2 \rightarrow Fe^{+3} + OH^- + {}^{\bullet}OH \tag{1}$$

$$Fe^{+3} + H_2O_2 \rightarrow Fe^{+2} + {}^{\bullet}OOH + H^+ \tag{2}$$

$${}^{\bullet}OH + H_2O_2 \rightarrow {}^{\bullet}O_2H + H_2O \tag{3}$$

$${}^{\bullet}OOH + Fe^{+3} \rightarrow Fe^{+2} + O_2 + H^+ \tag{4}$$

$${}^{\bullet}HO_2 \leftrightarrow H^+ + {}^{\bullet}O_2^- \tag{5}$$

$$Org. + {}^{\bullet}OH \rightarrow {}^{\bullet}Org. \rightarrow \ldots \rightarrow CO_2 + H_2O \tag{6}$$

In the treatment of textile industry wastewater, especially biological treatment methods [11], chemical and physicochemical methods [12–14], and membrane technologies [15] are used for colour and COD removal. Various methods, such as ozonation [16], chlorination [17], UV and Photo-Fenton processes [18] have been applied as oxidation processes. Advanced oxidation processes have been extensively tested as more environmentally friendly approaches in the textile industry [14,19–22]. Gilpavas et.al. [23] were conducted a study with industrial textile wastewater by comparing chemical coagulation and Fenton process. They showed that chemical coagulation can only remove 48%, whereas the following Fenton process can only remove 74% COD in 90 min of reaction time. In Girsales' study, it is reported that the biodegradability can be enhanced by 36% in dye bath effluents degradation by the Fenton process [24].

In this study, we aimed to determine the success of the Fenton process in the treatment of acrylic yarn dyeing wastewater through COD and colour removals. We also aimed to optimize the operating conditions Fe(II) and H_2O_2 dosages, pH and reaction time using the response surface methodology (RSM) method and to show the performance of the Fenton process comparing the results obtained with the chemical coagulation results.

2. Experimental

2.1. Wastewater

The wastewater used in the experimental studies was supplied by an acrylic yarn dye-house (AYD). The viscose acrylic yarns were dyed before being subjected to the pre-treatment process. Dye bath wastewater and bath washing waters were deposited in a balancing pool and treated chemically. Experimental studies were conducted with wastewater taken separately from acrylic paints as the dye bath wastewater represents the facility wastewater. Various contaminant parameters of dyeing bath waters are given in Table 1. Basic colouring agents were used in the acrylic dye bath. The chemical structure of the dispergator, in which AYD is used, is alkylpolyglycolether and non-ionic.

2.2. Experimental study

Experimental works were carried out by taking 250 ml of dye bath water sample per well with 500 ml effective volume. The hydrogen peroxide remaining after the reaction causes a positive entry in COD analyzes. Thus, hydrogen peroxide, which remains as a result of Fenton reactions, was known to cause interventions in COD analyzes. The removal of COD, which is equivalent to the remaining H_2O_2 from COD, and the determination of the H_2O_2 amount were provided through the iodometric method, which is the most commonly used method to prevent this intervention [6,25]. Talinli and Anderson stated that the concentration of H_2O_2 below 20 mg/L in the sample is sufficient for COD intervention [26]. Hence, whether the remaining H_2O_2 is higher than 20 mg/L in the samples filtered were ascertained using peroxide kits. In cases where the 20 mg/L value was exceeded, the amount of remaining H_2O_2 was determined using potassium permanganate. The COD equivalent of the amount of remaining hydrogen peroxide was calculated using the equation given in the literature [25] and the actual COD value was determined by subtracting it from the measured COD value. All analysis were conducted according to the Standard Methods, COD, colour and total organic carbon (TOC) analysis's were performed under SM 5220C, SM 2120 E and 5310B.

2.3. Optimization method

In this study, D-optimal design method, which is provided by a computer algorithm called the RSM, was used

Table 1
Characteristics of dyehouse wastewater

COD, mg/L	1.550
TOC, mg/L	551
Color	
λ_{436}	0.182
λ_{525}	0.149
λ_{620}	0.135
Abs	0.466
pH	4.32
Conductivity, μs/cm	2.150

to optimize the data obtained from the experiment. It is a design that reveals the relationship between the dependent variable and the set of independent variables in an appropriate manner [27]. The following second-degree polynomial regression model was used to see the effects of the variables and find the optimum conditions (Eq. (7)).

$$y = a_0 + \sum a_i x_i + \sum a_{ij} x_i x_j + \sum a_{ii} x_i^2 \tag{7}$$

Here, y shows response variables, a_0 shows fixed variables, a_i, a_{ii} and a_{ij} show linear coefficients, and x_i, x_j show independent variables. The variables were encoded in accordance with the following equation:

$$\alpha = \frac{x_i - x_0}{\Delta x} \tag{8}$$

Here $\propto$ is the code value of the independent variables, x_i shows the actual value, x_0 gives the actual value at the midpoint, and Dx shows the change in x_i variable. A five-level four-factor central composite design (CCD) was applied for the Fenton process. Current density (x_1), initial pH (x_2) and reaction time (x_3) were the selected independent variables, and the levels of the variables were coded as –2, –1, 0, +1 and +2. Codded factors belong to the Fenton processes are given in Table 2, respectively.

3. Results and discussions

3.1. Fenton process optimization

In the Fenton experiments, Fe dosage, H_2O_2 dosage, pH and time parameters that affected the process were examined. Preliminary study data (COD removal efficiency) was used in the determination of the levels (values range) of these factors to be used in the statistical model.

Generally, the COD load from dying agents and other chemicals varies between 1,000–1,200 mg/L. In the experiments, when the duration was increased from 30 to 120 min in wastewater, COD removal was observed to be increased by approximately 50%. Thus, the time interval to be examined for the duration factor in the statistical model was determined as 30–150 min.

In the second region of Table 3, a common level for pH was sought. In here, the highest level of remission is 3.2 as can be seen from Table 3. Hence, the pH range to be used in this study was determined as 2.8–3.6. In the third part of the table, dosages of iron and peroxide were tried to be determined together since there was a stoichiometric relationship between them. When the results were examined, the iron dosage was determined as 800 mg/L, and the hydrogen proximity dosage was determined as 1,600 mg/L, and this showed that a high efficiency could be obtained. Although COD removal by 75% as the optimum conditions was achieved in all experiments, it is expected that a higher efficiency than this value is likely to be obtained at the end of the optimization and that these efficiencies could be reached with lower concentrations. The matrix prepared in the direction of the obtained data is given in Table 2. Created CCD and obtained results are given in Table 4.

Table 3
Preliminary studies carried out to determine operating conditions with the Fenton process in AYD wastewater and dye house

Time (min)	Fe dosage (mg L^{-1})	H_2O_2 (mg L^{-1})	pH	COD Removal (%)
30	300	1,000	3	31
60	300	1,000	3	36
90	300	1,000	3	42
120	300	1,000	3	43
180	300	1,000	3	43
255	300	1,000	3	44
360	300	1,000	3	44
30	350	400	2.2	44.05
30	350	400	3.2	46.88
30	350	400	4.2	44.60
30	350	400	5.2	41.22
30	350	400	6.2	37.54
30	50	400	3.2	29.03
30	150	400	3.2	34.00
30	250	400	3.2	41.11
30	350	400	3.2	46.88
30	450	400	3.2	49.78
30	550	400	3.2	52.67
30	200	1,600	3.2	55.23
30	600	1,600	3.2	66.56
30	1,000	1,600	3.2	75.19
30	1,400	1,600	3.2	76.89
30	1,800	1,600	3.2	75.56
30	2,000	1,600	3.2	75.78

Table 2
Working matrix for the treatment through Fenton

Independed variable (process variable)	Factor	Response surface method levels				
	X_j	$\alpha = -2$	–1	0	+1	$\alpha = +2$
Fe dosage (mg L^{-1})	X_1	200	500	800	1,100	1,400
H_2O_2 dosage (mg L^{-1})	X_2	1,200	1,400	1,600	1,800	2,000
Initial pH	X_3	2.8	3	3.2	3.4	3.6
Reaction time (min)	X_4	30	60	90	120	150

Table 4
Results of RSM performed with Fenton experiment

No	Fe dosage	H_2O_2 dosage	Initial pH	Reaction Time	COD Removal (%)	Color Removal (%)	TOC Removal (%)
	x_1	x_2	x_3	x_4	y_1	y_2	y_3
1	–1	–1	–1	–1	70.37	95.42	60.14
2	1	–1	–1	–1	77.06	96.25	64.86
3	–1	1	–1	–1	69.56	94.16	59.18
4	1	1	–1	–1	79.95	95.68	71.44
5	–1	–1	1	–1	67.6	92.65	56.54
6	1	–1	1	–1	76.83	95.36	63.92
7	–1	1	1	–1	66.48	94.38	56.62
8	1	1	1	–1	77.76	96.54	61.98
9	–1	–1	–1	1	73.71	95.46	63.32
10	1	–1	–1	1	82.7	96.97	74.28
11	–1	1	–1	1	72.89	95.97	62.04
12	1	1	–1	1	82.64	97.62	74.97
13	–1	–1	1	1	71.4	95.25	59.95
14	1	–1	1	1	78.81	95.89	67.57
15	–1	1	1	1	70.49	97.65	59.04
16	1	1	1	1	79.77	96.32	68.31
17	–2	0	0	0	61.54	90.76	42.39
18	2	0	0	0	80.97	96.02	68.95
19	0	–2	0	0	79.07	95.89	64.02
20	0	2	0	0	78.97	98.05	65.67
21	0	0	–2	0	74.96	97.19	68.02
22	0	0	2	0	75.83	96.11	65.11
23	0	0	0	–2	72.73	96.15	63.44
24	0	0	0	2	81.86	95.89	72.34
25	0	0	0	0	80.14	96.17	71.37
26	0	0	0	0	78.8	95.46	69.54
27	0	0	0	0	80.97	96.1	68.27
28	0	0	0	0	81.16	95.46	69.81
29	0	0	0	0	80.04	96.45	69.24
30	0	0	0	0	80.2	96.35	68.2

Based on the results in Table 4, the analysis of variance (ANOVA) results of COD, colour and TOC removal efficiencies are given in Table 5, which suggests that each of the efficiency could have a high significance. Accordingly, the equations obtained by selecting the parameters effective as confidence interval $p > 0.05$ are as follows:

$$y_1 = 80.218 + 4.662x_1 + 1.878x_4 - 2.385x_1^2 - 1.35x_3^2 - 0.875x_4^2 \quad (9)$$

$$y_2 = 96 + 0.842x_1 - 0.658x_1^2 \quad (10)$$

$$y_3 = 69.405 + 5.151x_1 + 2.192x_4 - 3.389x_1^2 \quad (11)$$

Pareto analysis was also shown by Fig. 1. Pareto analysis gives information about the effectiveness of independent parameter [28]. It was prepared for COD results. When the result from the equation and Pareto graphic are compared, Fe dosage (x_1) was found to be the most important parameter among the independent variables.

The iron dosage in the COD removal, the initial pH and reaction time were found to be effective in the TOC removal, whereas the iron dosage alone was found to be effective in the colour removal. A similar situation could be seen in Fig. 2 which shows that COD and TOC removals by the Fenton process were found as 80% and 75%, respectively.

The codes obtained by the optimization made with the aid of MathCad through the equations obtained in Table 6 and independent variables are given in Table 6. Based on these optimized values, COD, Colour and TOC can be removal by 82.8%, 96.2%, and 75.6%, respectively.

In the work carried out with the optimum operating conditions obtained in Table 6, iron dosage (1,093 mg/L), hydrogen peroxide dosage (1,600 mg/L), initial pH (3.2) and reaction time (150 min) were obtained as 84.6% COD,

Table 5
Fenton process ANOVA results

Depended variable	Coded	Multi–R^2	R^2	Significance
COD	y_1	0.985	0.970	6.59E-09
Color removal	y_2	0.902	0.813	2.66E-03
TOC removal	y_3	0.975	0.950	3.27E-07

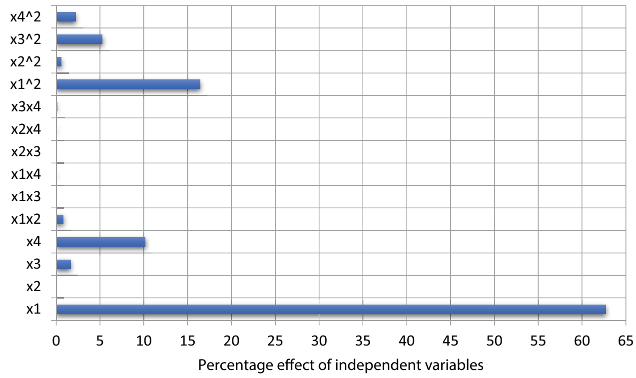

Fig. 1. Pareto graphic analysis.

98.6% colour and 78.6% TOC. RSM results are within 95% confidence interval. These values are similar when compared with literature data; colour, COD and TOC removal efficiencies according to the studies in the literature were obtained as 90%–98%, 70%–85% and 60%–80%, respectively [2,3,28–31]

The primary cost sources on the Fenton process are the cost of Fe and H_2O_2 dosages. When the cost for optimum conditions was calculated, it was found that the requirement for Fe dosage is 0.026 €/m^3 wastewater, 1.19 € for hydrogen peroxide and the total treatment cost was determined as 1.216 €/m^3 textile wastewater.

3.2. Comparison with chemical coagulation

To compare the chemical coagulation process with the Fenton process, two different coagulants, which were $Al_2(SO_4)_3$ and $FeSO_4$, were used in the coagulation experiments. To determine optimum pH in chemical coagulation experiments, the optimum pH ranges in the literature were used for the coagulant species that were reported, and the pC-pH diagrams were used for $Fe(OH)_2$, $Fe(OH)_3$ and $Al_2(SO_4)_3$ [32]. The chemical coagulation experiments were carried out in the pH ranges of 6.5–7 and 8.5–9.0 for $Al_2(SO_4)_3$ and $FeSO_4$, respectively, with 1,100 mg/L of coagulant dose which was closer to the value obtained in the

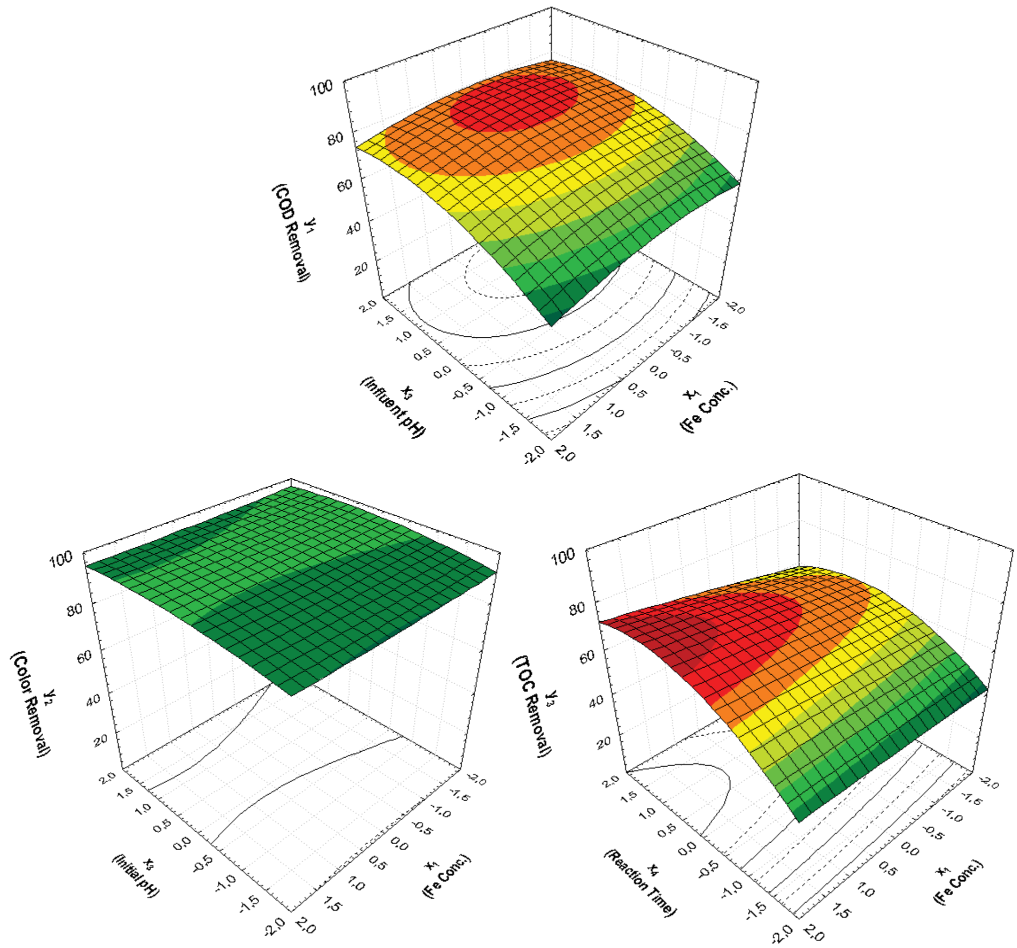

Fig. 2. COD, Color and TOC removal by Fenton process.

Table 6
Fenton process optimization

Independent factors	Coded levels	Real values
Fe dosage (mg L^{-1})	0.977	1,093
H_2O_2 dosage (mg L^{-1}	0	1,600
Initial pH	0	3.2
Reaction time (min)	2	150

Table 7
Comparison of recovery efficiencies of chemical coagulation and Fenton processes

Process	COD removal (%)	TOC removal (%)	Color removal (%)
Chem coagulation (Al)	33.4	30.9	75.6
Chem coagulation (Fe)	39.7	38.1	87.9
Fenton	84.6	78.6	98.6

Table 8
Sludge formation of chemical coagulation and Fenton processes

Process	Chemical coagulation	Fenton
Sludge (organic), %	34.36	27.47
Sludge (inorganic), %	65.65	72.53
Sludge/COD, kg/kg	3.44	1.60

Fenton process optimization. The maximum efficiencies that are obtained and their comparison with the Fenton process are given in Table 7. The obtained values are similar when compared to literature [31].

When two coagulants were compared for chemical coagulation, Fe(II) was found to be more effective, and chemical coagulation was found to be insufficient for both COD and colour removal. This result was also obtained by Gilpavas et al. [23], while chemical coagulation can be effective on COD removal by 48%, Fenton process efficiency was higher as 74% [23]. The amounts of sludge formed by chemical coagulation and Fenton processes were also compared and are given in Table 8.

The findings showed that less sludge formation was observed through Fenton process. As a result of the removal of organic substances by oxidation, more stable and less amount of sludge was observed.

4. Conclusion

The most effective parameter in the process optimization and Fenton process was found to be iron concentration, and the best removal was achieved with 1,093 mg/L Fe(II) concentration, 1,600 mg/L H_2O_2 concentration, pH 3.2 and 150 min treatment. When these systems are applied, 84.6% COD, 98.60% colour and 78.60% TOC removal could be achieved. The findings suggest that very high removal efficiencies could be achieved through the Fenton process in terms of these pollutant parameters. To show the effectiveness of the Fenton process, the chemical coagulation study was performed according to the optimum results obtained from the Fenton process results, and removal by 39.7%, 38.1%, and 87.9% were obtained for COD, TOC and colour, respectively. Chemical coagulation was found to be insufficient for both COD and colour removal. The obtained efficiencies suggest that the contaminant parameters could be easily lowered below the discharge limits by the Fenton process. Unlike the coagulation mechanism of the Fenton process, this situation also depends on the high oxidation capacity based on OH radicals. Thus, Fenton-type advanced oxidation processes are thought to be more effective in the treatment of AYD wastewater.

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