



Research Article

COMPARISON OF CHEMICAL AND ELECTROCHEMICAL TREATMENT PROCESSES FOR WOOL YARN DYEING EFFLUENTS

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ABSTRACT

Textile industry is one of the sectors that consume plenty of water and this situation increases the threat of contamination on the receiving water environments. In this study, treatment method performances of chemical coagulation, electrocoagulation, Fenton and electrofenton for wool yarn dyeing effluents were investigated by considering COD and TOC parameters and; advantages and disadvantages of each process were evaluated. Response surface methodology (RSM) was applied to optimize Fenton process and, in order to achieve COD and TOC removed by 90.5% 77.4%, respectively, 143 minutes of reaction time was found to be required. Optimum pH for Fenton process was determined via RSM as 3.28 pH. The required Fe dosage and H₂O₂ dosage was found to be 1135 mg/L and 1822 mg/L, respectively. When Fenton, electrofenton, coagulation and electrocoagulation processes were compared in terms of COD removal, the most effective process is found to be Fenton process and 88%, 77%, 30% and 42% of COD removals were observed, respectively. When the amount of sludge formed per treated COD is compared, it was observed that the least sludge was obtained via electrofenton process in term kg sludge/kg COD, where the maximum sludge was obtained in chemical coagulation process with 73% higher than electrofenton.

Keywords: Textile wastewater, advance oxidation processes, chemical coagulation, electrocoagulation, electrofenton, fenton.

1. INTRODUCTION

Industrial activities have an important place among pollution sources. In terms of industrial pollution prevention and protection of water resources, the textile industry is one of the sectors that need to be addressed. The fact that the textile industry has an important position in the export and employment of the developing countries makes the situation even more precise.

Textile industry is one of the most water consuming industries [1]. Textile industry wastewaters are characterized by high concentration of COD and colour, high pH and low biodegradability. Particularly in pre-treatment (washing) and colouring processes (dyeing) a significant amount of water is consumed [1]. The fact that the wastewater of the textile industry is variable makes it difficult to standardize in treatment methods and requires that each production

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facility be handled separately[2] . Conventional treatment methods include; biological processes [3], physical-chemical processes [4], adsorption [5] and chemical oxidation [6,7]. Because of inhibition to bacteria growth, biological processes such as activated sludge cannot be used effectively.

The electrocoagulation process (EC) provides less sludge formation than chemical coagulation, and it became a common treatment method for colour removal from textile wastewater. The electrocoagulation process, which is a more advanced model of chemical coagulation, has been frequently used in this field [8]. In addition to the lesser sludge formation compared to the classical chemical coagulation (CC) process, coagulant material formation can be caused by using electrodes as waste iron and aluminum plates. Thus, the pollution caused by the dissolution of anions such as sulfate and chloride from the coagulant chemical is prevented. Moreover, since the amount of coagulant entering the water increases with time in EC process, higher efficiency can be obtained when it is compared to CC process [9].

Among the advanced oxidation methods, the Fenton process is generally used to oxidize non-biodegradable and toxic characteristic substances. After the Fenton process, the non-biodegradable parts can be converted into biodegradable end products. The electrofenton process (EF) is a more advanced model of the classical Fenton process and is presented into the water through iron electrodes. In the electrofenton process, the direct reaction of the iron to the water with the peroxide given directly to the water will have a lower yield than the classical Fenton process in which the peroxide is directly involved. In EF process, coagulant Fe^{+2} is dissolved over time to water and peroxide is given only at the beginning of the reaction; it is predicted that EF process will have a lower efficiency than the classical Fenton process[8]. In addition, less sludge formation than the other chemical treatment methods also provides a significant advantage to this process.

The aim of this work is to treat the wastewater of the dyehouse, which is a subcontractor of the textile industry, with chemical coagulation, electrocoagulation, Fenton and electrofenton methods, to determine the COD and TOC removal efficiencies, to determine the amount of sludge formed. For this purpose, the independent variables affecting the treatment efficiency for the Fenton is optimized using response surface methodology and it is compared with chemical coagulation, electrocoagulation and electrofenton processes were investigated.

2 MATERIALS AND METHOD

2.1. Wastewater and Experimental Set-up

Yarn Dye effluents were obtained from wool yarn dye house of a local textile factory. Experimental studies were carried out with the wool yarn wastewater which its properties were given in Table 1. The chemical used during the experiments were in technical grade.

Table 1. Properties of the acrylic yarn dye wastewaters

COD mg/L	TOC mg/L	Colour Pt-Co	pH	Conductivity µs/cm
1.140	470	0,314	4,37	1.860

Sample volume was 250 ml which placed in Plexiglas reactor with size of 6.5 cm x 6.5 cm x 18 cm. In electrocoagulation and electrofenton processes, Fe electrodes were used with dimension of 5cm x 18 cm. While one anode and one cathode were used in electrofenton process with 6.0 cm interval, 4 electrodes were used in electrocoagulation studies with total anode area of 101 cm² and 1.5cm intervals. The reactors are mixed under 50 rpm. After treatment, the pH adjusted to pH 7 and, the effluent left for settlement for 30 minutes. The analyses are done from the samples taken from supernatant. The selected independent parameters are mentioned in Section 2.3.

2.2. Analytical Methods

All analyses were conducted according to Standard Methods. COD is applied based on SM 5220 C by using Hach-Lange brand DRB 200 thermoreactor and, for dried sludge SM 2540 is applied. Hach-Lange brand IL550 TOC/TN equipment is operated for TOC analysis according to SM 5310 method. Hach-Lange brand HQ40D is used for pH and conductivity measurement in accordance with SM 2510. Colour measurements are done with Hach-Lange DR-5000. For Fenton and electrofenton processes, to prevent intervention of H₂O₂ to COD analysis, the remained hydrogen peroxide was determined by Permanganate index [10].

2.3. Experimental Design

Response surface methodology was applied to optimize the obtained data. The second-degree polynomial regression model was used for optimization as given in the following equation.

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

where; y is the response variables, a₀ is a constant, a_i, a_{ii} and a_{ij} are the linear, quadratic and interaction coefficients of independent variables. x_i, x_j are the independent variables. Variables have been coded by the following equation.

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

where; α gives the code values of the independent variables, x_i is the real value, x₀ is the real value in the medium point and Δx is the change in x_i variable.

A five-level four-factor central composite design was chosen for more representative results with more independent parameters to optimize the Fenton process. Fe dosage (x₁), H₂O₂ dosage (x₂), Initial pH (x₃) and reaction time (x₄) were the selected as independent variables, and the levels of the variables were coded as -2, -1, 0, +1 and +2. Coded factors belong to the Fenton processes are given in Table 2.

Table 1. Working matrix for the treatment through Fenton

Independent Variable (process variable)	Factor	Response Surface Method Levels				
		α = -2	-1	0	+1	α = +2
Fe dosage (mg/l)	X ₁	200	500	800	1,100	1,400
H ₂ O ₂ dosage (mg/l)	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

3 RESULTS AND DISCUSSIONS

3.1. Fenton Proses Optimization

Preliminary studies have been conducted to determine the levels of the factors (Fe dosage, H₂O₂ dosage, pH and duration) to be used in the statistical model. The change in COD removal efficiency in the pH range of 2.2 to 6.2 was investigated in order to see the effect of different pH values (Table 3). It can be observed from Table 3 that the highest yield is obtained at pH 3.2. To this purpose, 2.7 to 3.9 range was selected as the pH range in the response surface method (RSM) to determine the optimum pH value. In the preliminary study conducted to determine the iron dosage and the range of H₂O₂ dosages to be used in RSM, pH 3.2 value was applied. The iron

dosage range was chosen between 50-550 mg/L, and 2 different H₂O₂ doses were selected to be as 400 mg/L and 1,600 mg/L.

In order to determine the iron and H₂O₂ dosage intervals to be used in the model, two sets of experiments were carried out considering the optimum pH value (pH = 3.2) obtained in the preliminary study. In the first experimental set, 400 mg/L H₂O₂ was kept constant and the iron dosage was changed between 50-550 mg/L. The highest COD removal was achieved with a dose of 450 mg/L iron at 53.67%. In the second experimental set, the COD removal efficiencies to be obtained for higher dosages were investigated. For this purpose, the molar ratio of Fe/ H₂O₂ and H₂O₂ and the iron dosages were increased by keeping the first experimental set. Obtained COD removal efficiencies are given in Table 3. Due to the significant increase in the COD removal related to the increase in dosage used, high H₂O₂ and iron dosages were employed in the experimental sets which were formed depending on the statistical model. The H₂O₂ range used in the model was 1200 - 2000 mg/L and the iron dosage was 200 - 1400 mg/L.

Table 2. Preliminary studies carried out to determine operating conditions with the Fenton process

Time (min)	Fe dosage (mg/L)	H ₂ O ₂ (mg/L)	pH	COD Removal (%)
30	350	400	2.2	38.15
30	350	400	3.2	46.12
30	350	400	4.2	43.75
30	350	400	5.2	42.85
30	350	400	6.2	36.23
30	50	400	3.2	28.83
30	150	400	3.2	39.12
30	250	400	3.2	42.86
30	350	400	3.2	46.12
30	450	400	3.2	53.67
30	550	400	3.2	45.92
30	200	1600	3.2	62.73
30	600	1600	3.2	77.42
30	1000	1600	3.2	82.22
30	1400	1600	3.2	83.84
30	1800	1600	3.2	83.56
30	2000	1600	3.2	77.37

The studies in which the Fenton process is applied for the treatment of similar wastewater reports that the reaction time is kept over 90 minutes in order to have high removal efficiency [11-13]. Thus, the reaction time was chosen between 30 to 150 minutes in order to investigate higher process efficiency as well as lower cost and time. Based on the data obtained, five-level four-factor central composite design for Fenton process of woolen yarn dye bath wastewater was created. COD and TOC removal efficiencies as experimental responses are given in Table 4.

Table 4. Results of RSM performed with Fenton experiment

No	Fe dosage	H ₂ O ₂ dosage	Initial pH	Reaction Time	COD Removal (%)	TOC Removal (%)	Total Dried Sludge (mg/L)
	x ₁	x ₂	x ₃	x ₄	y ₁	y ₂	y ₃
1	-1	-1	-1	-1	79.36	67.9	913
2	1	-1	-1	-1	86.97	72.56	2061
3	-1	1	-1	-1	77.14	65.54	980
4	1	1	-1	-1	87.51	75.63	1938
5	-1	-1	1	-1	76.32	62.7	1081
6	1	-1	1	-1	85.72	71.54	2078
7	-1	1	1	-1	76.81	68.24	855
8	1	1	1	-1	85.72	74.06	2000
9	-1	-1	-1	1	79.45	67.36	961
10	1	-1	-1	1	88.92	73.72	2009
11	-1	1	-1	1	79.05	70.97	893
12	1	1	-1	1	87.25	76.23	1966
13	-1	-1	1	1	78.53	68.5	1033
14	1	-1	1	1	87.47	72.46	2164
15	-1	1	1	1	77.41	70.63	954
16	1	1	1	1	87.56	79.66	2119
17	-2	0	0	0	70.05	58.23	401
18	2	0	0	0	88.64	73.32	2646
19	0	-2	0	0	86.23	69.38	1495
20	0	2	0	0	87.41	73.37	1490
21	0	0	-2	0	77.52	71.19	1553
22	0	0	2	0	78.03	70.33	1261
23	0	0	0	-2	82.52	72.63	1465
24	0	0	0	2	90.22	79.88	1279
25	0	0	0	0	85.15	74.56	1474
26	0	0	0	0	86.68	72.98	1367
27	0	0	0	0	86.22	75.42	1367
28	0	0	0	0	86.97	74.12	1406
29	0	0	0	0	84.91	74.21	1333
30	0	0	0	0	85.19	74.69	1348

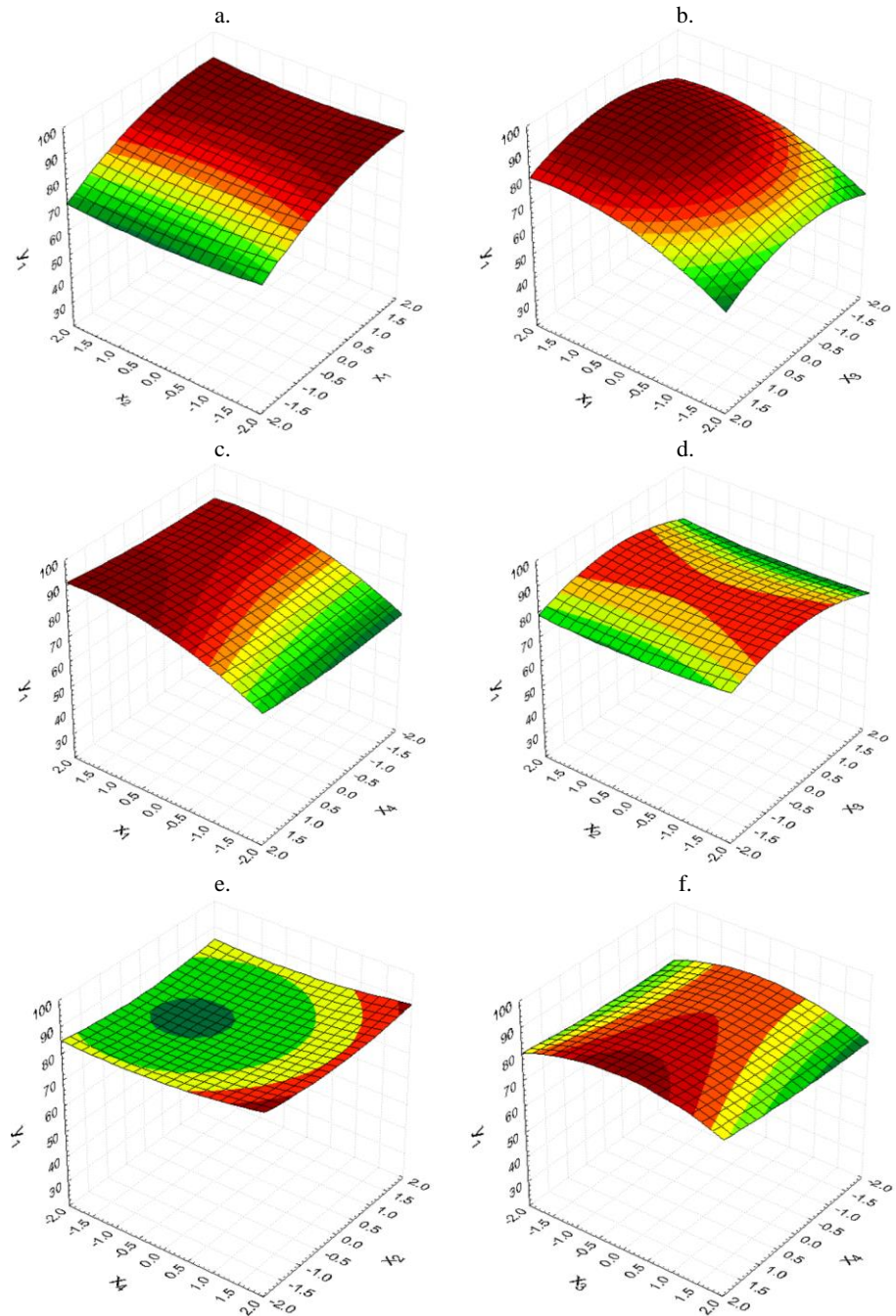


Figure 1. COD Removal Efficiency depending on independent variables. Fe Dosage (x_1), H_2O_2 dosage (x_2), initial pH (x_3) reaction time (x_4)

Figure 1.a shows the effect of COD removal efficiency on Fe Dosage (x_1) and H_2O_2 dosage (x_2). When the Fe dosage is below 500 mg/L (on the position of x_1 : -1.0), the removal efficiency can be observed to be less than 80%. In addition, when two independent variables are compared, it can be observed that Fe Dosage (x_1) has a higher effect and the effect of H_2O_2 dosage (x_2) is very low that it can be disregarded. Figure 1.b shows the effect of Fe Dosage (x_1) and initial pH (x_3). As shown in Figure 1.a. the dosage of Fe (x_1) below 500 mg/L is also shown in Figure 1.b where the removal efficiency is less than 80%. When the effect of initial pH is observed, while the removal efficiency can be seen to be higher between x_3 : pH3-pH3.4 at $-1.0 < \alpha < +1.0$. the effect is said to be great. In addition, it was found that the removal efficiency could exceed 85% over Fe dosage of 800 mg/L. Figure 1.c shows the effect of Fe Dosage (x_1) and reaction time (x_4). Here, the effect of reaction time can be seen to be less than Fe. It was observed that the removal efficiency was similar in the first 75 minutes (x_4 : -0.5) and there was a small increase in the removal efficiency after 75 minutes. Figure 1.d and Figure 1.e show the comparison of H_2O_2 dosage (x_2) with initial pH (x_3) and reaction time (x_4). It was observed that only the initial pH was effective and the effective range of pH was between pH3 – pH 3.4. Figure 1.f shows the comparison of the effects of initial pH (x_3) and reaction time (x_4). According to this, the effect of the initial pH is much higher than the reaction time and it is noted that there is a slight increase in removal efficiency in periods longer than 75 minutes.

Figure 2.a, Figure 2.b and Figure 2.c show the comparison of the dosage of Fe (x_1) on the TOC removal efficiency according to H_2O_2 dosage (x_2), initial pH (x_3) and reaction time (x_4), respectively. Among the independent variables, the most effective one can be seen to be the Fe dosage (x_1). When the used on the Fe dosage of 650 mg/L (x_1 : +0.5), it was noted that the efficiency of TOC removal was over 70%. It was observed that the effect of the selected pH range on TOC removal could be ignored (Figure 2.b). Similar to the data obtained in COD removal, when the reaction time was over 75 minutes, it was observed that the TOC removal efficiency increased slightly and it could be over 75% (Figure 2.c).

Figure 2.d and Figure 2.e show the comparison of the effects of the initial pH (x_3) and the reaction time (x_4), respectively with H_2O_2 dosage (x_2). It was found that the effect of pH could be ignored. It was also noted that the effects of H_2O_2 dosage (x_2) and reaction time (x_4) were similar, and that the removal efficiency could exceed 75% over 1500 mg/L with H_2O_2 dosage and 90 min reaction time. In Figure 2.f show the effects of initial pH (x_3) and reaction time (x_4) on TOC removal. It was seen that the effect of the initial pH could be ignored in regard to the reaction time and it was found that the removal could be over 75% over 90 minutes.

In Figure 3, effect of Fe Dosage (x_1), H_2O_2 dosage (x_2), initial pH (x_3) and reaction time (x_4) on produced dried sludge concentration can be seen. Fe Dosage (x_1) was the only parameter affecting the amount of sludge produced.

In the light of the data obtained, ANOVA analyzes were performed and compared with the obtained graphs. Regression coefficient obtained according to ANOVA results and relevance are shown in Table 5. Relevance values are quite high and regression coefficients are significant similarly. This indicates the suitability of the model. Coefficients which are effective on COD, TOC and dried sludge concentration were determined as $P > 0.05$. The equations obtained by the effective coefficients obtained according to this situation are given in Table 5.

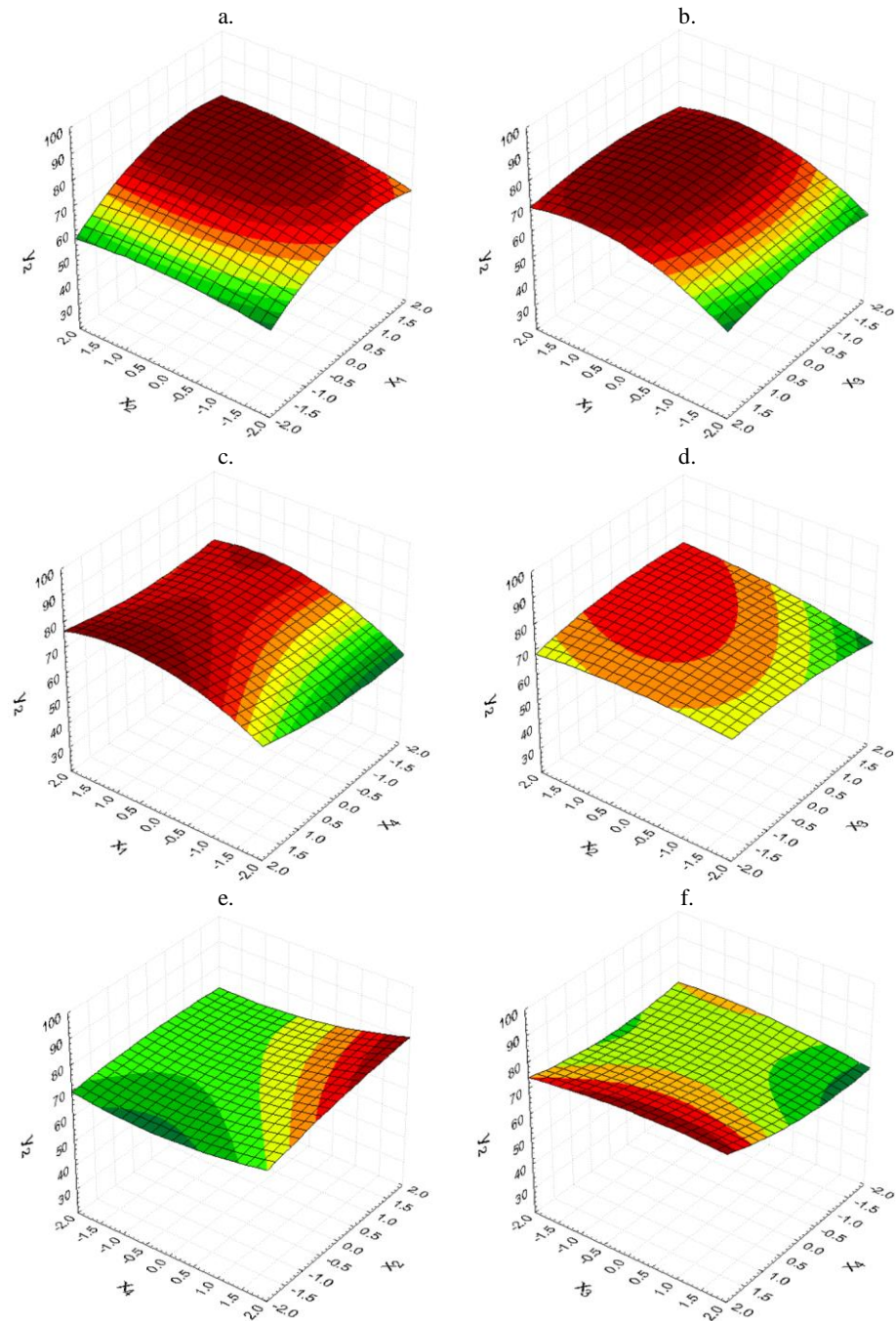


Figure 2. TOC Removal Efficiency depending on independent variables. Fe Dosage (x_1), H_2O_2 dosage (x_2), initial pH (x_3) reaction time (x_4)

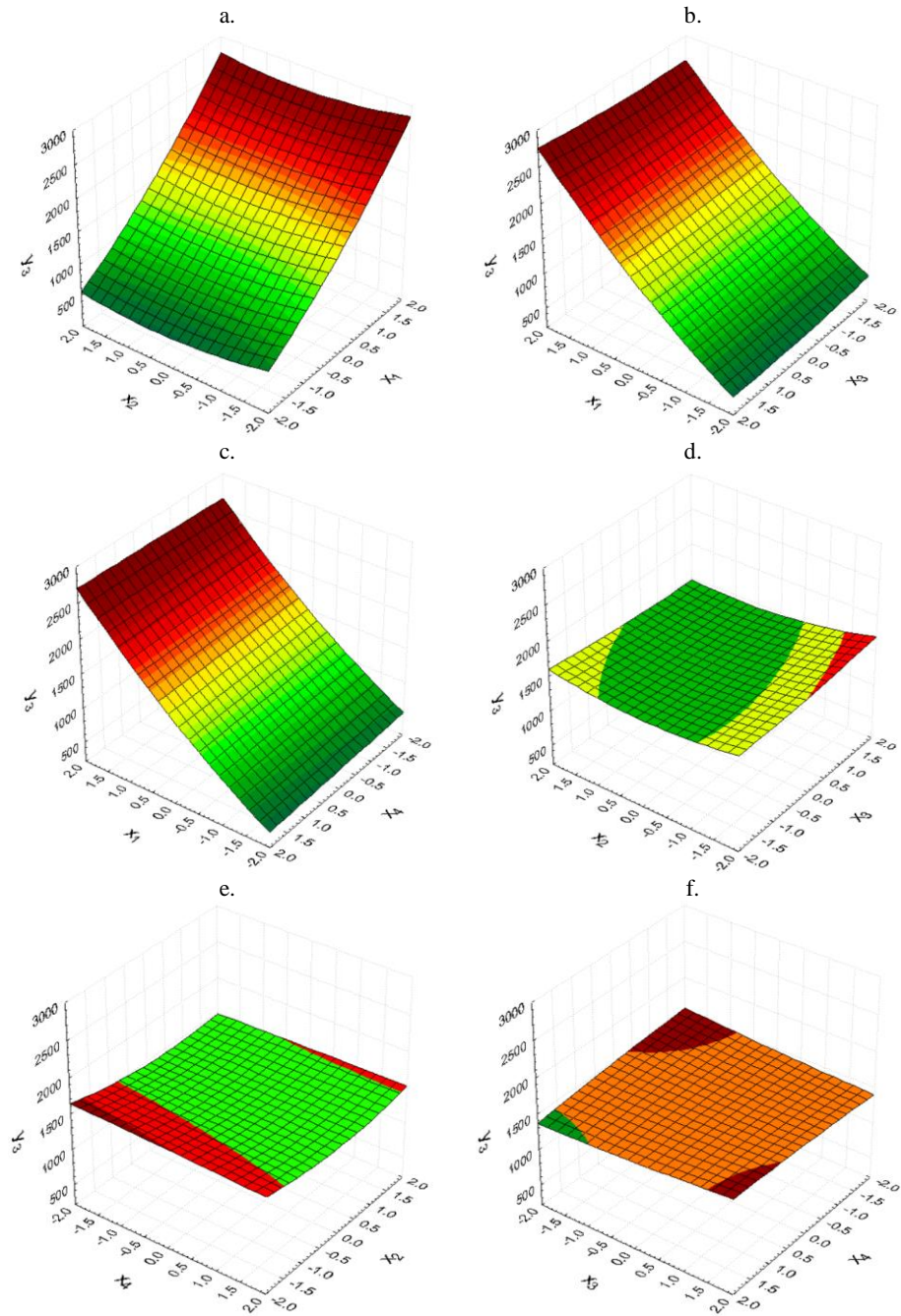


Figure 3. Produced Dired Sludge Concentration depending on independent variables. Fe Dosage (x_1), H_2O_2 dosage (x_2), initial pH (x_3) reaction time (x_4)

Table 5. ANOVA results and obtained equations by regression

Depended Variables	Coded	R ²	Relevance	Equation
COD Removal	y ₁	0.986	3.61 E-09	$y_1 = 85.853 + 4.593x_1 + 1.062x_4 - 1.628x_1^2 - 2.02x_3^2$
TOC Removal	y ₂	0.982	3.28 E-08	$y_2 = 74.33 + 3.508x_1 + 1.342x_2 + 1.494x_4 + 0.66x_2x_3 - 2.128x_1^2 - 0.728x_2^2 - 0.882x_3^2 - 0.492x_4^2$
Dried Sludge	y ₃	0.991	1.93 E-010	$y_3 = 1328 + 548x_1 + 43.8x_1^2$

Information can be obtained about the effective parameters and their effects with the equations given in Table 5. From the equation y₁, the parameters effective in the removal of COD show that Fe dosage (x₁), initial pH (x₃) and reaction time (x₄) are effective. Fe dosage and reaction time appears to be directly effective. According to the coefficients, the most effective parameter can be said to be the dosage of Fe. Similarly, this situation is also obtained from the graphs. The parameters that are effective in removing TOC are effective in all parameters selected from y₂ equation. It is also possible to obtain the information that the most effective parameter of the coefficients is the Fe dosage (x₁) and that the removal of all parameters increases with a slight increase. This is also confirmed by graphics. It can be observed from the y₃ equation given for the dried sludge concentration that the only effective parameter is the Fe dosage (x₁).

The MathCad program was employed to find the optimum condition and the values for the independent variables were calculated for both the COD and TOC removal cases and are given in Table 6. Although the H₂O₂ dosage for COD removal was not determined to be effective, the H₂O₂ dosage to be reacted was 1822 mg/L, as it was important for the removal of TOC. Similarly, it was found that the effect of the selected initial pH range was not effective in COD removal. By applying the variables given in Table 6. COD and TOC removal can be achieved by 90.5% and 77.4% respectively. The yields obtained in the control experiments are 90.1% and 77.9% and they are within the confidence interval.

Table 6. Fenton process optimization

Independent Factors	Coded Levels	Real Values
Fe dosage (mg/l)	1.118	1135 mg/L
H ₂ O ₂ dosage (mg/l)	1.11	1822 mg/L
Initial pH	0.415	pH 3.28
Reaction Time (min)	1.757	143 min

In the study carried out for similar wastewater, Fenton process is optimized for iron dosage and hydrogen peroxide and calculated as 1200 - 1680 mg/L for iron dosage and approximately 3000 mg/L for hydrogen peroxide [12,13]. Obtained results are more enhanced on hydrogen peroxide concentration basis according to literature.

3.2. Comparison with Different Processes

Coagulation, electrocoagulation and electrofenton processes have been investigated in order to investigate the purity of thread dyeing wastewater with different processes. For this purpose, the study in the range of 200-1600 mg/L Fe dosage was carried out. Fe(Cl)₃ was used for chemical coagulation, and iron electrodes were used in electrocoagulation and electrofenton processes, and the current required to be dissolved was calculated in accord with Faraday's law. The study was carried out in 250ml volume and the reaction time was selected as 30 minutes. 1200 mg/L H₂O₂ concentration was added for electrofenton and Fenton processes. The obtained results are given in Figure 4.

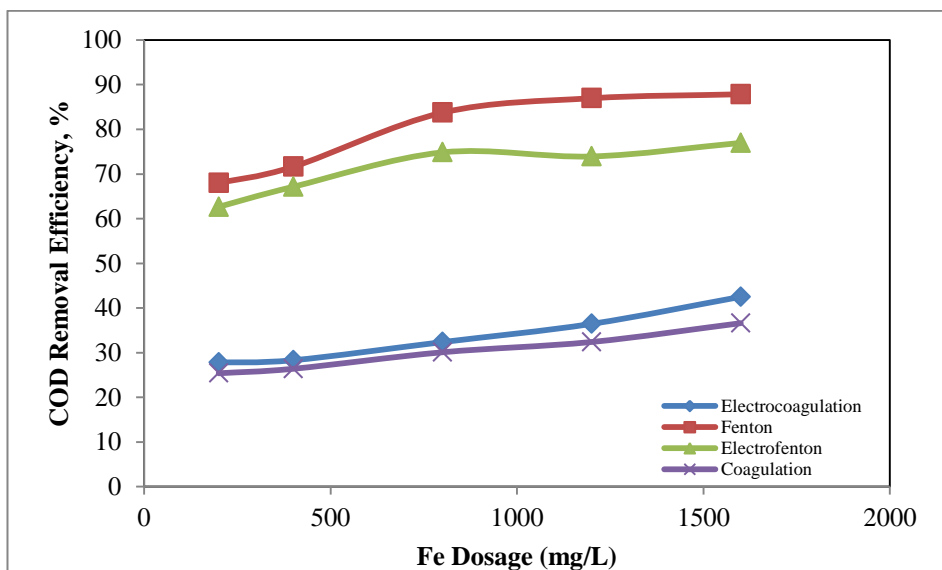


Figure 4. Comparison of treatment processes regarding to COD Removal Efficiency

Figure 4 shows the Fenton, electrofenton, electrocoagulation and coagulation processes according to COD removal efficiency. With the Fenton process, COD removal of 88% can be achieved and COD can be removed with an electrofenton process of 77%. In addition, chemical coagulation is known to be effective in the treatment of textile wastewater, but the effect of coagulation on the treatment of wooly dyehouse wastewater has been found to be only near 30%. It was found that the electrocoagulation process was more effective than coagulation and up to 42% COD removal was reached. The study conducted by Gong et al. (2014), acrylic manufacturing wastewaters are treated with electrocoagulation and, it is observed that the high removal efficiencies can be reached at high reaction time as 100 minutes [14]. When the reaction time is 30 minutes, COD removal efficiencies obtained approximately 20%, similarly to this study. According to the results from the studies for textile wastewater treatment by Fenton in literature, COD and TOC removal efficiencies were as 70-85% and 60-80%, respectively [15-20]. In addition, COD removal efficiencies by chemical coagulation process were 40-50% [9]. The obtained results are similar with literature.

The difference between Fenton and electrofenton processes can be explained as follows. The iron dissolved in the electrofenton process passes over time to water. The amount of solute will change to x in water at time t , while it will pass up to $2x$ in $2t$ time. Furthermore, the peroxide required in the electrofenton reaction is given first. Since peroxide itself is also oxidant, it reacts with the pollutant and exhausted. Since there is no hydrogen peroxide left in the water at the end of the electrofenton period, it is natural that the removal efficiency is slightly lower than the Fenton process. In the study conducted by Ilhan F. et al. (2017), they stated that the H_2O_2 dosage increased the efficiency of the removal to a certain extent in the electrofenton process at different times [8].

The difference between electrocoagulation and coagulation seen in Figure 4 can be explained as follows. In the electrocoagulation process, the iron passing through the water by electrodisolution increases with time. This means that the coagulant dissolves over time to water. In chemical coagulation, the coagulant is only given at the beginning of the reaction. It is expected that the dissolved coagulant in EC process is more efficient, because of being given to

water over time. This results the possibility of coagulants to coagulate with pollutants during the process reaction time [21].

Figure 5 shows sludge formations. one of the most important disadvantages of treatment methods. Fe coagulant was calculated to be the same amount and used in 4 processes. The sludge formation when the Fe dose is 1200 mg/L is 7.1, 4.9, 2.19 and 1.61 kg sludge/kg COD for CC, EC, Fenton and EF respectively. It is also found in the literature that sludge formations are higher in classical methods than in electrochemical processes [21,22]. In this study, sludge formation was investigated in 4 methods and, it has been seen that the study of electrocoagulation and electrofenton is relatively limited in the literature. As seen in Figure 5, electrochemical methods have more positive results compared to classical methods in terms of both yield and sludge formation.

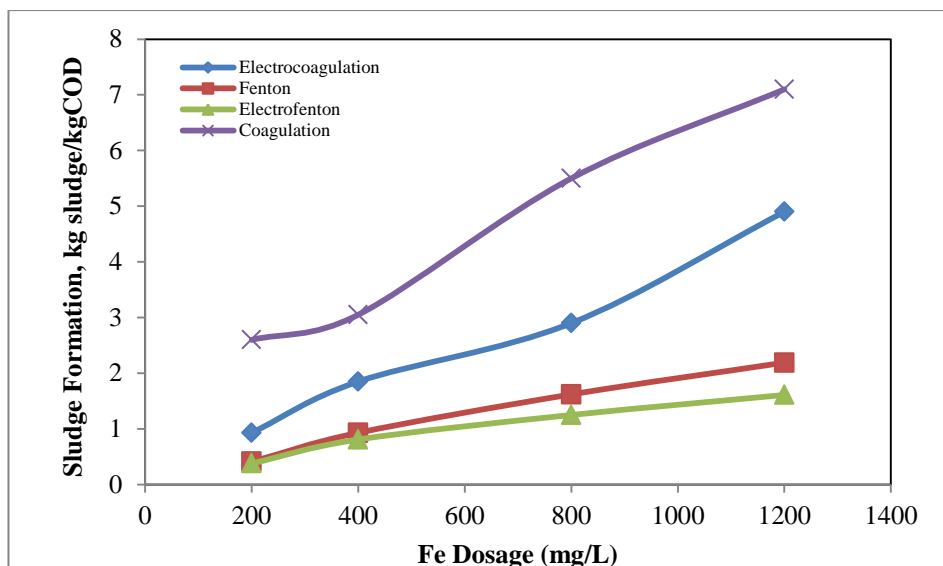


Figure 5. Comparison of treatment processes regarding to sludge formation

4. CONCLUSION

In present study, treatment of woollen yarn dyeing effluents by chemical and electrochemical processes were compared according to COD and TOC removal efficiencies. When Fenton proses was optimized by experimental design, the most effective parameter was determined as Fe dosage on both removal of COD and TOC. While H₂O₂ concentration is not effective in COD removal, it was found to be second effective variable for TOC removal. All selected independent parameters were determined as effective in TOC efficiency. In addition, Fe dosage was observed as the only effective parameter in sludge formation on Fenton process. Highest removal efficiencies of COD and TOC were calculated from the equation as 90.5% and 77.4%. In order to achieve these efficiencies, 1135 mg/L Fe dosage, 1822 mg/L H₂O₂ concentration, pH 3.28 and 143 minutes is required. To compare different treatment technologies, electrocoagulation, coagulation, electrofenton and Fenton processes were studied on COD removal efficiency basis. Within these processes, Fenton process was found to be the most effective process, while electrofenton, electrocoagulation and chemical coagulation is following it, respectively. The reason why the electrofenton process is partially less effective than the Fenton process is explained as the fact that

the added H₂O₂ concentration is given at once. Since it reacts with the pollutants in the reactor without reacting with iron; the Fenton reaction cannot occur in the later stages of the electrofenton process. To prevent this, the investigation on the effect of total required H₂O₂ added at different time intervals should be studied. Electrofenton process can be enhanced by applying H₂O₂ in more than one time, and this application will produce less sludge with higher removal efficiencies than Fenton process. It will be an appropriate process for woollen yarn dye effluents.

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