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Dye removal from synthetic and dye bath wastewater by electrocoagulation method and isotherms

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ABSTRACT

In this study, treatment of wastewaters containing Setazol Black TNN, which is a reactive diazo dyestuff that is prevalently used in dyeing natural fibers in the textile industry, by the electrocoagulation method was examined. Experiments investigating the effectiveness of Al and Fe electrodes in treatment of real wastewaters collected from the dye bath of a yard dyeing facility and synthetic wastewaters prepared with Setazol Black TNN used in this bath were carried out by determining the optimum treatment conditions. The optimum current, pH and initial dye concentration in the synthetic wastewaters for the Al electrodes were 8 ampere (A), 7.57 and 50 mg/L, respectively, while these for the Fe electrodes were 8A, 9 and 100 mg/L, respectively. The optimum current, pH and thinning rate were seen in the dye bath wastewaters for the Al electrodes as 8A, 8 and 1/60 and for the Fe electrodes as 8A, 9 and 1/30, respectively. While carrying out the experiments using these experimental conditions, color and Chemical Oxygen Demand (COD) removal from the synthetic wastewaters and the dye bath wastewaters with long-running treatments was investigated. As a result of the treatment experiments, removal efficiencies of 96% and 57% respectively for color and COD for the Al electrodes and 99% and 61% for the Fe electrodes in the synthetic wastewaters, 87% and 55% for the Al electrodes and 89% and 55% for the Fe electrodes in the dye bath wastewaters were obtained. With this study, it was aimed to reveal not only the position and effectiveness of the treatment process for wastewaters containing intense color but also the effects of chemicals used in dye baths on removal efficiency. Moreover, it was determined that the Langmuir isotherm model was more convenient for the experimental data in the process. In the last part of the study, a correlation analysis was carried out between the color and COD removal efficiencies. Besides, the efficiencies for the use of Al and Fe electrodes respectively for both color and COD removal were compared, and comments were made for different groups by one-way ANOVA..

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1. Introduction

Wastewater constitutes the greatest environmental problem in the textile industry. Washing wastewater, process wastewater, cooling waters and rainwater are the main components of wastewater flows [1,2]. Process wastewaters in the sector originate to a significant extent from dyeing processes [2]. Wastewaters emerging from dyeing operations contain assistive chemicals such as residual colorants, inorganic salts, alkalis and surface-active substances and various byproducts [1-5]. The main group of pollutants in this wastewater flow is usually synthetic dyestuffs which have a high molecular weight, complex chemical structure and low biological solubility [1,2,6]. Reactive dyestuffs which contain a reactive group in their structure constitute the most significant and greatest dyestuff class for the sector [1,7]. Reactive dyes are water-soluble anionic chemicals that react with functional groups and have high durability for wet processes such as washing and rubbing which can be rapidly covalently bonded to textile fibers in the presence of alkaline conditions [1,7,8]. Dyeing with these dyes is the result of covalent bond formation between the carbon atom of the coloring molecule and the sulfur, nitrogen or oxygen atom of the thiol, amino or hydroxyl group in the polymer creating the fiber [7,9]. Reactive dyestuff molecules contain four significant structural classes as the chromogen, water-solubilizing, reactive and bridging groups. Chromogens from these groups give the molecule its color and have a direct effect on the properties of the dye such as light fastness. For reactive dye molecules to be able to bond with textile fibers, they need to be water-

soluble. For this purpose, reactive dyes contain ionic groups such as one or more sulfonates that provide solubility in an aqueous environment. Reactive groups are some of the most fundamental structural groups of reactive dyes. These structures are specific groups that react with the textile fiber and provide bonding by establishing chemical bonds. Bridging groups are structures that are mostly constituted by amino groups which are used to bond the chromogen part of the dye molecule and the reactive group [9].

Dyes are also chemically named based on the chromogen groups in their structure which are unsaturated functional groups and create color by absorbing light in the ultraviolet region [6,10]. One of the most important chromophore groups used in dyes is the azo group, and dyes containing this chromophore structure are known as azo dyes. Approximately 80% of azo dyes, which constitute about 60-70% of all dyes that are produced, are used in the textile industry. Azo dyes are mostly aromatic but sometimes aliphatic or aliphatic-aromatic compounds that contain one or more azo groups (N=N) in their chromophore structures. The most important azo dyes are monoazo dyes that contain a single azo group in their structure [10-12]. Azo dyes are a large class of synthetic dyes that can provide color in a dense and broad tone range, can be synthesized relatively easily and with low costs with desired properties such as high light and washing fastness and contain more than 2000 chemical substances with high water-solubility [10,12,13].

The properties of dyes that are the reasons for their commercial preference make these dyes a great problem in the environmental sense. Reactive dyes, which constitute the largest dye class based on application methods, are the most environmentally problematic dye class which consumes the most water per unit fiber and leads to the discharge of most salts, alkalis, etc. per unit fiber among all dyes [5]. Azo dyes which constitute the most significant reactive dyes are those that are resistant to degradation. These dyes usually cannot be removed with classical treatment methods and are found with unchanged chemical structures in the output waters of treatment facilities. Azo dyes, which increase their concentration in nature by accumulation due to these properties, are also known as toxic chemicals that have mutagenic and carcinogenic effects. Removal of dyes, especially those containing an azo group in their chromophore structures, carries great importance especially because of their potential harms on human and environmental health [12,14,15].

This study examined the treatability of wastewaters belonging to the most intense pollutant-containing process of the textile industry, which is accepted as one of the most pollutant sectors all over the world, with the electrocoagulation method. In the experiments conducted with two different electrode materials as Al and Fe and two different wastewaters as synthetic wastewater (SW) and dye bath wastewater (DBW), it was firstly aimed to determine the parameters effective on the treatment process that was used. After determining the optimum treatment conditions with 30-minute short-term experiments, 300-minute long-term experiments were carried out using the current and pH values at which the highest removal efficiency was achieved. This way, the efficiency of the electrocoagulation method was examined not only in terms of color but also about COD for the SW and the DBW containing concentrated dyestuffs and additional chemicals.

2. Materials and methods

2.1. Experimental procedure

The study was carried out with authentic wastewaters taken from the yarn dyeing bath of a facility that operates in the fields of yarn dyeing, fabric dyeing and fabric printing in the borders of the province of Tekirdağ, which is one of the most significant provinces of Turkey in terms of the textile industry, and synthetic wastewaters. Setazol Black TNN is a reactive diazo dye that is mostly used in dyeing natural fibers. The chemical structure of the dye whose chemical name is tetrasodium 4-amino-5-hydroxy-3,6-di [[4-[[2-(sulfonate oxy) ethyl] sulfonyl] phenyl] azo] naphthalene-2,7-disulfonate is given in Figure 1.

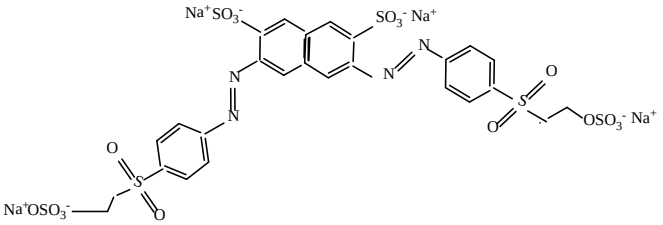

Figure 1. *Chemical structure of Setazol Black TNN.*

At yarn dyeing baths, based on the recipes prepared according to the type of the yarn and the dyestuff to be used, the fiber is prepared for the dye, bonding of the dye with the fiber is made easier, and impurities in the medium are eliminated. Table 1 presented the characteristic properties of SW and DBW.

Table 1. *Electrocoagulation reactor input values of the samples*

Parameter	Synthetic Wastewater (SW)	Dye Bath Wastewater (DBW)
COD (mg/L)	1400	3050
pH	7.70	11.34
Conductivity (mS/cm)	2.94	118.3

The electrocoagulation reactor in which the treatment study is carried out had a volume of approximately 3 L. Al and Fe electrode groups consisting of 14 parallel plates were used in the reactor. In these electrode groups, electrodes, each 2 mm thick, were placed 3 mm apart.

2.2. Analyses

In the study, color, measurements were made at three different wavelengths as 436, 525 and 620 nm with the help of a spectrophotometer (Aquamate Thermospectronic/US) [16]. COD analysis was carried out in a thermoreactor (Spectroquant TR-620/Germany) by using the method titled 5220 C "Close Reflux, Titrimetric Method" [17]. given in the Standard Methods. All experiments were conducted at room temperature, and the pH values of the specimens were adjusted by using 0.1 N H_2SO_4 and 0.1 N NaOH.

3. Results and discussion

At the first stage of the two-stage study, the color removal efficiencies of the Al and Fe electrodes for SW and DBW were examined. First of all, the optimum experimental conditions for the parameters as current, pH and initial dye concentration which are effective on the process yield for these two wastewaters and two electrode materials were determined. At the second stage of the study, using these optimum experimental conditions, the long-run treatment of these DBW and SW was examined by monitoring color and COD removal. This way, it was aimed to determine the dye removal efficiency of the electrocoagulation method and the effects of additional chemicals used at dyeing baths on removal efficiency.

3.1. Effect of current

When the reaction time is kept constant, the current that is applied becomes directly effective on the amounts of ions leaving the electrodes. Based on metal ions, the current controls important operational parameters such as the coagulant production rate, reaction speed, duration, size of the gas bubbles that are produced and size of the flocs. This way, it directs the progression of the process and determines the removal efficiency and operational cost of electrocoagulation [18-20]. The experiments where the optimum current was examined for the Al and Fe electrodes were carried out at room temperature (21°C) under 3, 5, 8 and 10 ampere current at a dye concentration of 50 mg/L and natural pH (7.57) for SW and with specimens diluted by 1/60 at natural pH (9.32) for DBW (Figure 2a-b).

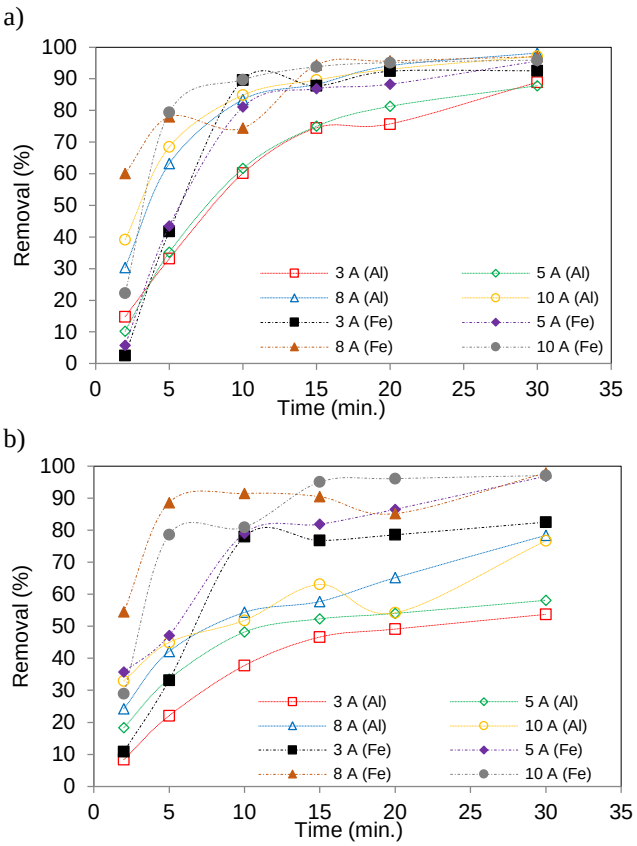

Figure 2. *Effect of current on removal efficiency of (a) SW, (b) DBW.*

Under 4 different currents varying in the range of 3-10 A, the removal efficiencies were obtained as 98% for the Al electrodes and 97% for the Fe electrodes in SW, while they were obtained as 78% for the Al electrodes and 98% for the Fe electrodes in DBW. Based on the results, the optimum current for both electrode types and both wastewater types was determined as 8 A. The aluminum and iron ions that emerge with the electrical current passing through the electrolyte during the electrocoagulation process form monomeric and polymeric hydroxyl complexes. Increased current passing through the system increases the quantity of ions that emerge, and in relation to this, it increases the formation of these complex compounds with high oxidative potential. These compounds, which are also excellent coagulants, lead to the formation of large floc networks that have a high pollutant removal capacity [20-22].

3.2. Effect of pH

pH is one of the important operational parameters that are effective on the performance of all electrochemical processes, especially electrocoagulation. This is because, at suitable pH values, metal ions may form metal hydroxides that absorb and precipitate dissolved pollutants and species that coagulate in a

broad spectrum with destabilized and aggregated suspended particles. In the electrocoagulation method, the pollutant removal efficiency varies to a large extent based on the structure and quantities of the monomeric and polymeric compounds that are formed throughout the process. These monomeric, polymeric and oligomeric aluminum and iron species are transformed with complex and precipitation kinetics into insoluble amorphous $Al(OH)_3$ and $Fe(OH)_3$ based on the pH of the medium [18,23]. The complex mechanism that allows the formation of metal hydroxide flocs is expressed as follows in Eq. (1) [24].

$$M^{+3} \rightarrow \text{monomeric kinds} \rightarrow \text{polymeric species} \rightarrow \text{amorphous } M(OH)_3 \quad (1)$$

Dye removal mechanisms that are usually defined as precipitation for low pH values and adsorption for higher pH values are summarized as follows in Eqs. (2-5) [20,25-27].

Precipitation

$$\text{Dye} + \text{monomeric Al/Fe} \rightarrow [\text{dye-monomeric Al/Fe}]_s \quad (2)$$

$$\text{Dye} + \text{polymeric Al/Fe} \rightarrow [\text{dye-polymeric Al/Fe}]_s \quad (3)$$

Adsorption

$$\text{Dye} + Al(OH)_{n(s)}/\ Fe_n(OH)_{n(s)} \rightarrow \text{Sludge} \quad (4)$$

$$[\text{Dye} + \text{polymeric Al/Fe}]_s + Al(OH)_{n(s)}/\ Fe_n(OH)_{n(s)} \rightarrow \text{Sludge} \quad (5)$$

Electrocoagulation is defined as a process with a buffer effect that has the capacity of balancing pH. In addition to this, process efficiency is explained by the formation of ionic species that do not precipitate or have high solubility which gain dominance in the medium based on pH [23,28].

The experiments where the optimum pH value was investigated were carried out at a dye concentration of 50 mg/L for SW, a dilution rate of 1/60 for DBW, at a current of 8 A and room temperature at pH values of 3, 5, 7, 8, 9 and natural pH for the Al electrodes and 3, 5, 7, 8, 9, 10 and natural pH for the Fe electrodes (Figure 3a-b).

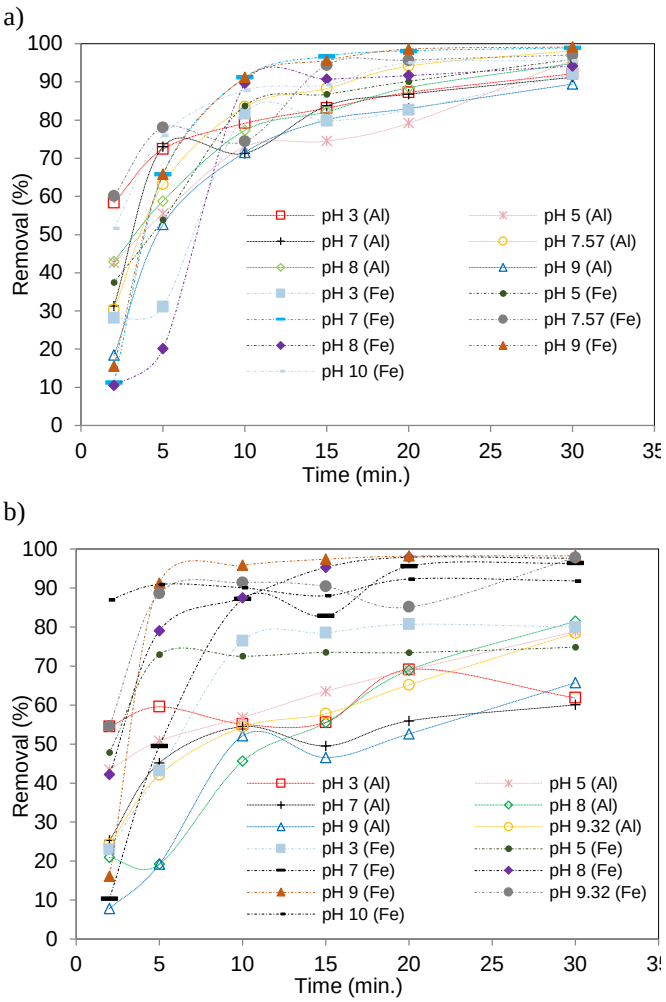

Figure 3. *Effect of pH on removal efficiency of (a) SW, (b) DBW.*

At the studied pH values, the removal efficiencies were obtained as 98% for the Al electrodes and 99% for the Fe electrodes in SW and 82% for the Al electrodes and 98% for the Fe electrodes in DBW. The optimum pH value was determined as the natural pH in SW and 8 in DBW for the Al electrodes, while it was determined as 9 for both wastewater types for the Fe electrodes.

3.3. Effect of initial dye concentration

The initial dye concentration, which provides a significant driving force to overcome the mass transfer resistance of the dissolved substances between the solid and liquid phase, constitutes one of the operational conditions that affect the removal efficiency in electrocoagulation, as in most other treatment methods [29]. Under the same current conditions and same electrolysis time, the same quantity of flocs is formed at different initial dye concentrations. The concentration of metal ions produced during the process shows a limiting effect for floc formation and is accepted as a limit factor. As the initial dye concentration increases, the quantity of flocs produced falls insufficient for adsorption and

sedimentation of all dye molecules. While electrocoagulation is generally a process with high efficiency in dye removal, studies have shown that, as the initial dye concentration increases, the removal efficiency decreases, and for the same efficiency to be achieved, longer treatment times are needed for higher concentrations [18,26,27,30].

The effects of the initial dye concentration were tested at a current of 8 A and room temperature at 5 different initial SW dye concentrations as 50, 100, 150, 200 and 300 mg/L at the natural pH (7.57) for the Al electrodes and at pH 9 for the Fe electrodes, while these effects were tested at 5 different DBW dilution ratios as 1/60, 1/30, 1/20, 1/15 and 1/10 at a pH of 8 for the Al electrodes and 9 for the Fe electrodes (Figure 4a-b).

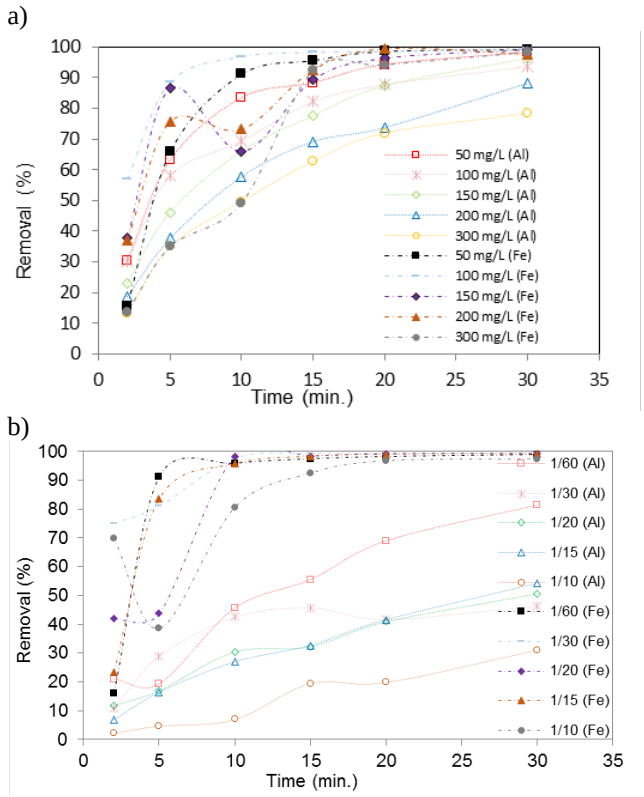

Figure 4. *Effect of the initial dye concentration on removal efficiency of (a) SW, (b) DBW.*

In the experiments carried out with different dye concentrations/dilution ratios, the removal efficiencies were obtained as 98% for the Al electrodes and 99.5% for the Fe electrodes in SW and as 82% for the Al electrodes and 99% for the Fe electrodes in DBW. Based on these results, the optimum initial dye concentration in SW was determined as 50 mg/L for the Al electrodes and 100 mg/L for the Fe electrodes. The optimum dilution ratio in DBW was determined as 1/60 for the Al electrodes and 1/30 for the Fe electrodes. Considering the results, it is seen that the dye concentration and removal efficiency changed in inverse proportion to each other, which was in agreement with the information in the literature. However, it was seen that, in the experiments with the Fe electrodes, the highest removal efficiency was obtained with the second lowest dye concentration. This may be explained by that iron electrodes provide color to the wastewater.

At the second stage of the study, long-run treatments of SW and DBW were carried out without dilution and by using the optimum experimental conditions determined at the first stage. Color and COD removal efficiencies were examined in the experiments where Al and Fe electrodes were used. The treatment conditions were determined as a current of 8 A, the natural pH of SW for the Al electrodes and pH 9 for the Fe electrodes in SW experiments, while the conditions were determined as a current of 8 A, pH 8 for the Al electrodes and pH 9 for the Fe electrodes in the treatment of DBW (Figure 5a-b).

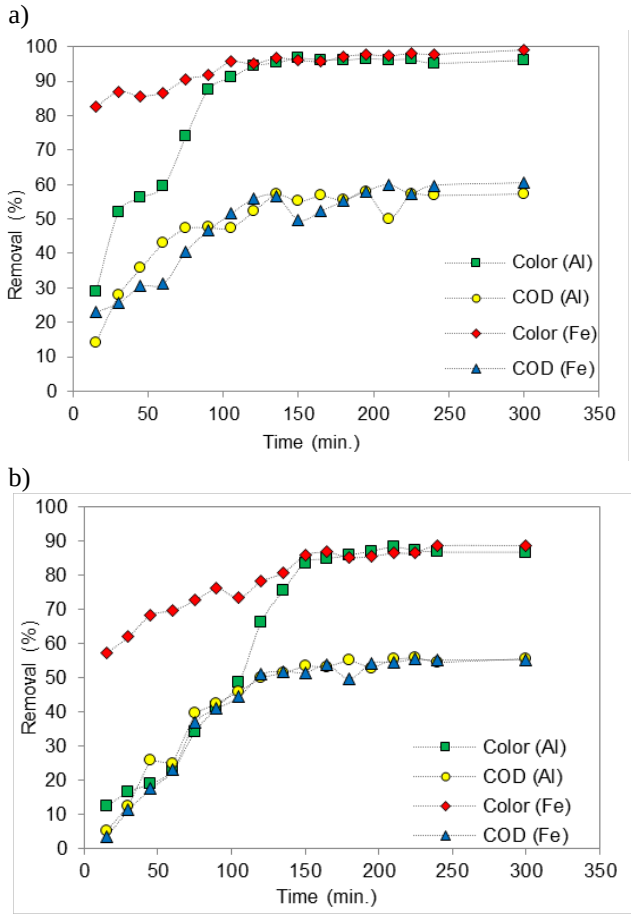

Figure 5. *Color and COD removal efficiency in (a) SW, (b) DBW.*

The color and COD removal efficiencies were found respectively as 96% and 57% with the Al electrodes and 99% and 61% for the Fe electrodes as a result of SW experiments lasting for 5 hours. Only after the 120th-135th minutes, the color and COD removal efficiencies reached the ranges of 94-95% and 52-57% for the Al electrodes and 95-97% and 56-

57% for the Fe electrodes respectively. As a result of the DBW treatment experiments, the color and COD removal efficiencies were found respectively as 87% and 55% for the Al electrodes and 89% and 55% for the Fe electrodes. In the experiments, after the 120th-135th minutes, the color and COD removal efficiencies reached the ranges of 66-76% and 50-51% for the Al electrodes and 78-81% and 51-52% for the Fe electrodes.

3.4. Isotherms

Various isotherms, especially the Freundlich isotherm (FI) and Langmuir isotherm (LI), have been developed to reveal the progression of adsorption processes. These isotherms may also be used to explain the reciprocal interactions between metal hydroxide and dye molecules in electrocoagulation. The linear FI and LI were given in Eqs. (6-7) below.

$$lnq_e = lnK_f + \left(\frac{1}{n}\right) lnC_e \quad (6)$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (7)$$

In the study, by calculating FI and LI for different dye concentrations, their suitability with the experimental results was investigated. In the isotherms, qe refers to the amount of adsorbed dye per unit adsorbent (mg/g), C_e is the dye concentration at equilibrium (mg/L), n is the adsorption intensity, K_f is Freundlich isotherm adsorption coefficient, q_m is the maximum dye amount adsorbed by unit adsorbent (mg/g), and K_L is Langmuir isotherm constant regarding adsorption energy (L/mg) [31-33]. The constants calculated from the isotherm curves in the study are given in Table 2.

Table 2. *Langmuir and Freundlich isotherm constants*

		Langmuir isotherm (LI)			Freundlich isotherm (FI)		
SW		qm	K_L	R^2	K_f	n	R^2
	Al	555.556	0.146	0.983	126.474	2.793	0.901
	Fe	303.030	0.429	0.942	80.002	1.877	0.844
DBW		qm	K_L	R^2	K_f	n	R^2
	Al	238.095	0.035	0.901	43.692	3.251	0.697
	Fe	285.741	0.385	0.920	78.668	1.730	0.777

The equilibrium constant as an important parameter determining the type of adsorption in Langmuir isotherm is calculated as shown in Eq. (8).

$$R_L = \frac{1}{1 + K_L C_o} \quad (8)$$

This constant shown as R_L is a dimensionless value calculated based on Langmuir isotherm constant (K_L) and the initial dye concentration (C_0). In the cases were this constant is found as $R_L>1$, $R_L=1$, $0<R_L<1$ or $R_L=0$, the adsorption process is defined respectively as unfavorable, linear, favorable or irreversible [32,33]. The change in the R_L values in SW and DBW with the Al and Fe electrodes based on different dye concentrations is shown in Figure 6.

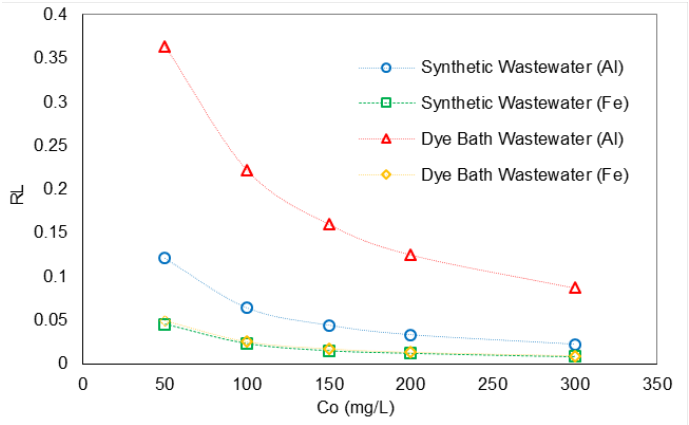

Figure 6. *Change of R_L value according to initial dye concentration.*

As seen in the results, the correlation coefficients for LI in SW were 0.983 and 0.942 respectively for the Al and Fe electrodes, while these were respectively 0.901 and 0.920 in DBW. Accordingly, the process was more compatible with LI, suggesting that the adsorption of dye molecules occurred on a homogenous surface as a single molecular layer than FI suggesting that it occurred on the regions with different adsorption energy of a heterogenous surface in multiple molecular layers [31,34].

3.5. Statistical Analysis

In this study, the statistical analysis of COD and color removal from SW and DBW by Al and Fe electrodes was performed using "IBM SPSS Statistics 22", and the results were given in Table 3.

Table 3. *Correlation analysis*

		SW (Al)		SW (Fe)		DBW (Al)		DBW (Fe)	
		Color	COD	Color	COD	Color	COD	Color	COD
Color	Pearson Correlation	1	0.956**	1	0.979**	1	0.930**	1	0.954**
	Sig. (2-tailed)		0.000		0.000		0.000		0.000
	N	17	17	17	17	17	17	17	17
COD	Pearson Correlation	0.956**	1	0.979**	1	0.930**	1	0.954**	1
	Sig. (2-tailed)	0.000		0.000		0.000		0.000	
	N	17	17	17	17	17	17	17	17

** Correlation is significant at the 0.01 level (2-tailed).

In terms of correlation analysis, as expressed in Table 3, there was a positive relationship between the variables (color and COD) at the 0.01 significance level. The homogeneity of the variance was firstly analyzed for color removal, and a heterogeneous variance distribution was reached since the result was not homogeneous (Levene's: 23.552; $p < 0.001$). As a result of the one-way analysis of variance (ANOVA), according to sig. $p < 0.05$, there was a statistically significant difference between SW (Al), SW(Fe), DBW (Al) and DBW (Fe). Post hoc analysis was performed to find out in which groups this difference occurred, and the results were presented in Table 4.

Table 4. *Multiple comparisons dependent variable: color*

					95% Confidence Interval	
(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
SW (Al)	SW (Fe)	-10.698	5.222	0.290	-26.123	4.726
	DBW(Al)	22.471	8.816	0.095	-2.434	47.377
	DBW (Fe)	4.486	5.594	0.966	-11.630	20.603
SW (Fe)	SW (Al)	10.698	5.222	0.290	-4.726	26.123
	DBW(Al)	33.171*	7.324	0.002	11.386	54.955
	DBW (Fe)	15.185*	2.684	0.000	7.505	22.865
DBW(Al)	SW (Al)	-22.472	8.816	0.095	-47.377	2.434
	SW (Fe)	-33.171*	7.324	0.002	-54.955	-11.386
	DBW (Fe)	-17.985	7.593	0.159	-40.211	4.241
DBW (Fe)	SW (Al)	-4.486	5.594	0.966	-20.603	11.630
	SW (Fe)	-15.185*	2.684	0.000	-22.865	-7.505
	DBW(Al)	17.985	7.593	0.159	-4.241	40.211

* The mean difference is significant at the 0.05 level.

According to the analysis results, there were significant differences between the group SW-Fe and DBW-Al and between SW-Fe and DBW-Fe in terms of wastewater mean value for the color. When the COD removal efficiencies were

examined in the experiments, firstly, the homogeneity of the variances was analyzed, and homogeneous variance distributions were used (Levene's: 1.207; sig.=0.314 i.e. p>0.001). As a result of the one-way ANOVA, according to sig. p>0.05, there was no statistically significant difference between SW (Al), SW(Fe), DBW (Al) and DBW (Fe).

4. Conclusion

This study was carried out with DBW collected from a yarn dyeing bath where Setazol Black TNN, which is a reactive diazo dye, was used and SW prepared with the same dye. As a result of the experiments, the optimum current and pH for both electrode materials and the optimum initial dye concentration for SW and the optimum dilution ratio for DBW were determined. Considering the results, it was seen that the optimum values for SW and DBW were mostly the same, and in the pH determination experiments with the Al electrodes, the results were close as pH 7.57 and pH 8.

In the study, the treatment of DBW without dilution and SW prepared with the same color value as these wastewaters was studied as a 5-hour treatment by using the determined optimum experimental conditions and monitoring color and COD removal efficiencies. As a result of the long-run treatment experiments, treatment efficiencies of 96% and 57% for respectively color and COD for the Al electrodes and 99% and 61% for the Fe electrodes in SW, 87% and 55% for the Al electrodes and 89% and 55% for the Fe electrodes in DBW were obtained. Moreover, it was observed that, beginning with the 120th-135th minutes, in color and COD removal, respectively, removal efficiencies in the ranges of 94-95% and 52-57% for the Al electrodes and 95-97% and 56-57% for the Fe electrodes in SW, 66-76% and 50-51% for the Al electrodes and 78-81% and 51-52% for the Fe electrodes in DBW were reached. This study showed that, based on the results on the dye used here, the electrocoagulation method may be used as a treatment step for wastewaters containing intense concentrations of color. Moreover, the suitability of the study for FI and LI, which are the two most significant adsorption isotherm models, was also examined. It was determined that the experimental data were more suitable for LI model for both wastewater types and the Al and Fe electrodes. Finally, a correlation analysis was performed between color and COD for the Al and Fe electrodes and SW and DBW types. Besides, the yield ratios for the usage of Al and Fe electrodes respectively for both color and COD were compared and comments were made for different groups by one-way ANOVA. One-Way ANOVA test was performed using IBM SPSS Statistics 22 to test whether there was a significant difference between different electrode (Al and Fe) and wastewater types (SW and DBW) for color and COD removal. According to the correlation analysis, there is a positive relationship between the variables (color and COD) on a significance level of 0.01. In one-way ANOVA tests performed separately for color and COD, since the significance for the color was < 0.05, there was a significant difference between the variables, while for COD, there was no significant difference between the variables because the significance was > 0.05.

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