ELECTRICAL PROCESSING OF BIOLOGICAL OBJECTS AND FOOD PRODUCTS

Removal of Direct Dyes from Textile Wastewater by Means of Combined Methods

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INTRODUCTION

Wastewater discharge by textile factories in natural water is impossible. Being of poor quality with a low biological index (the biochemical consumption of oxygen (BCO)/the chemical consumption of oxygen (CCO) < 0.3), such wastewater is toxic for water organisms.

To remove textile dyes from wastewater, various methods are used: neutralization and coagulation [1-2]; electrochemical oxidation [3-5]; and catalytic oxidation by means of hydrogen peroxide [6-9], ozone, or chlorine [10-12].

Coagulation methods are effective when the textile dyes' concentration reaches up to 150-250 mg/l and more and the value of the chemical consumption of oxygen (CCO) reaches up to $8000-10000 \text{ mgO}_2/1$. These methods lead to the effective decoloration of the dyes (the effect of decoloration is 90-97%); however, the degree of reduction and mineralization due to the molecular oxidation of the dye is sufficiently low (57-62%). The use of an aluminum coagulant in the reactions of neutralization and coagulation allows one to destroy all the dye molecules by means of colloidal aluminum hydroxide, which is afterward removed at sedimentation. However, these methods lead to a lot of sediment quantity, that needs further treatment. Thus, on the one hand, the use of coagulation methods adds to this process because of the great expense for chemicals. On the other hand, due to this process, a great sediment quantity that requires treatment and disinfection is generated. The use of only oxidation methods is not perfect too. According to these methods, the dye molecules are oxidized to more simple organic matter, which is further removed by activated carbon [13]. Oxidation methods can be used for only some polluted wastewater purification. Moreover, because of the quickly lost adsorption capacity of activated carbon, whose regeneration is an unsolved problem, these methods are expensive.

Thereby, first of all, it is necessary to remove the dye molecules by means of an aluminum coagulant and

then to realize combustion for their final destruction. Moreover, it is necessary at the primary stage of the purification to use electrocoagulation in the presence of electrogenerated aluminum, because of its chemical activity, compared with the coagulant, generated due to the hydrolysis of aluminum salts [14]. Due to the electrochemical treatment, one part of the dye molecules is decolored and the other is oxidized to elementary substances [15].

This work is devoted to the study of the removal process of textile dyes from model solutions at their electrochemical and catalytic treatment. Also, we investigated the dependence of the separation efficiency of water on the electrogenerated coagulant quantity, the initial concentration and nature of the dyes, the oxidizer consumption (H_2O_2), and the process duration.

EXPERIMENTAL

The electrochemical research was performed on model solutions prepared from direct dyes: "direct blue" (DBI) and "direct brown" (DBr) with addition of a salt cake for the electroconductivity maintenance [16].

The concentration of the dyes was varied in the range of 50–200 mg/l, because these concentrations occur in real-life environments. Model solutions were processed in an electrochemical cell with dissoluble electrodes of aluminum. The area of the electrodes was 8 dm², the distance between the electrodes was 4-5 mm, and the anodic current density was 0.5 A/dm². The quantity of energized current was determined by calculation depending on the required dose of aluminum hydroxide. After the electrocoagulation, the pH of each sample was varied from 5.0 to 5.1 by means of a hydrochloric acid solution or aluminum hydroxide; this mixture was precipitated for 2 hours. The solid phase was separated by means of decantation, and the samples were filtered. In the derived solution, by means of measuring the optical density at a suitable wave length (DB1 – 590 nm; DBr – 490 nm) using an SF-46 spectrophotometer, the residual concentration of the dye was determined.

	C ma/l	"Dir	ect Blue" dye (DBl)	"Direct Brown" dye (DBr)							
no.	C _{Al³⁺} , mg/l	C _{res} , mg/l	EDR, %	$m(Al^{3+})/m_{dye}$	C _{res} , mg/l	EDR, %	$m(Al^{3+})/m_{dye}$					
	$C_0 = 50 \text{ mg/l}$											
1	4.32	1.55	97.0	0.09	2.10	96.6	0.09					
2	5.40	1.25	97.5	0.11	1.38	97.4	0.11					
3	6.48	1.05	97.9	0.13	0.60	98.9	0.13					
4	7.56	0.55	98.8	0.15	0.40	99.2	0.15					
	I	I	$C_0 = 1$	00 mg/l		I	I					
1	4.32	31.42	68.6	0.06	35.60	64.4	0.07					
2	5.40 29.57 70.4		0.08	30.10	69.1	0.08						
3	6.48	15.56	84.4	0.08	14.50	85.5	0.07					
4	7.56	6.15	93.8	0.08	6.10	93.9	0.08					
5	8.64	5.25	94.7	0.09	5.20	94.8	0.09					
6	9.72	1.30	98.7	0.10	1.35	98.6	0.10					
7	10.80	10.80 0.55 99.4		0.11	0.55	99.4	0.11					
	I	I	$C_0 = 2$	00 mg/l		I	1					
1	4.32	92.30	53.8	0.04	94.20	52.9	0.04					
2	5.40	84.70 57.6		0.05	86.10	56.9	0.05					
3	6.48	42.60	78.7	0.04	43.20	78.4	0.04					
4	7.56	18.47	90.8	0.04	19.05	90.5	0.04					
5	8.64	7.68	96.2	0.04	8.10	95.9	0.04					
6	9.72	6.18	96.9	0.05	7.20	96.4	0.05					
7	10.80	3.72	98.1	0.05	3.91	98.0	0.05					
8	12.96	2.75	98.6	0.06	2.75	98.6	0.06					
9	15.12	1.15	99.4	0.08	1.20	99.4	0.08					

Table 1. Dependence of the effect of the dyes removal (DBl and DBr) on the quantity of the electrogenerated coagulant and on the initial dyes' concentration. pH = 5.0-5.1, I = 4.0 A

The catalytic oxidation of the dye molecules was realized as follows. In order to get the concentration in range from 3×10^{-3} to 5×10^{-3} M, different amounts of hydrogen peroxide solution were added to 500 ml of the model dye solution. The concentration of the iron ions (II) was in range from 1×10^{-4} to 7×10^{-4} M. By means of a sulphuric acid solution, the pH was reduce to 2.0-2.5. The solutions were intermixed by means of a magnetic stirrer for 5, 10, 20, 40, and 60 minutes. After the oxidation of the dye molecules, the samples were analyzed for the residual concentration of the dyes. Also, by means of the index of the chemical consumption of the oxygen (CCO) determined by suitable methods [17], the intermediate products of the oxidation were analyzed. Based on the experimental data, the effect of the dye removal (EDR) and the degree of the dye mineralization (DDM) were calculated as follows:

EDR (%) =
$$(C_0 - C_{res}) \times 100/C_0$$
,
DDM (%) = $(CCO_0 - CCO_{res}) \times 100/CCO_0$,

where C_0 is the initial dye concentration in the model solution, mg/l; C_{res} is the residual dye concentration in the def-

ecated solutions, mg/l; CCO_0 is the chemical consumption of oxygen in the model solution, mgO₂/l; and CCO_{res} is the chemical consumption of the oxygen in the oxidized solutions, mgO₂/l.

RESULTS AND DISCUSSION

The decoloration of the model dyes occurred due to the neutralization and coagulation of the associated dye molecules, which occurs owing to the hydrogen bonds between them and their size increase due to the electrocoagulation. After the sediment precipitation, the residual concentration of the dyes decreases, while the quantity of the electrogenerated coagulant increases. This regularity is observed at an increase of the initial dye concentration too (Table 1).

However, according to the experimental data, the initial concentration of the direct dyes increases together with the quantity of the electrogenerated coagulant needed for the removal of the dyes before their decoloration. On the other hand, together with the increase of the initial concentration of the dyes, a decrease of the specific consumption of the electrogen-

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		"Direct I	$CCO_0 = 41 \text{ mgO}_2$	"Direct Brown" dye ($CCO_0 = 35 \text{ mgO}_2/l$)					
no.	t, min	without boiling		at boiling for 10 min		without boiling		at boiling for 10 min	
		CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %
				[Fe ²⁺]	$= 1 \times 10^{-4}$	М			
1	5	20.62	49.7	1.25	96.4	13.75	60.7	3.12	91.1
2	10	13.12	68.0	1.25	96.4	12.50	64.3	2.50	92.6
3	20	12.55	69.5	0.62	98.5	11.25	67.9	1.87	94.7
4	40	6.87	82.2	0.62	98.5	10.00	71.4	1.87	94.7
5	60	6.85	83.3	0.62	98.5	9.98	71.5	1.87	94.7
			1	[Fe ²⁺]	$= 3 \times 10^{-4}$	М	I	l	1
1	5	13.37	68.0	1.25	96.4	10.62	69.7	1.25	96.4
2	10	12.50	69.5	1.25	96.4	9.37	73.2	1.25	96.4
3	20	10.78	73.7	0.62	98.5	8.75	75.0	1.25	96.4
4	40	6.87	82.2	0.62	98.5	7.50	78.6	1.25	96.4
5	60	6.80	83.4	0.62	98.5	7.35	79.0	1.25	96.4
				[Fe ²⁺]	$= 5 \times 10^{-4}$	М	1		1
1	5	12.50	69.5	0.62	98.5	17.50	50.0	8.75	75.0
2	10	11.25	72.6	0.62	98.5	16.25	53.5	8.75	75.0
3	20	10.00	75.5	0.62	98.5	15.62	55.4	8.75	75.0
4	40	8.75	78.7	0.62	98.5	15.00	57.1	1.25	96.4
5	60	8.90	78.3	0.62	98.5	14.50	58.6	1.25	96.4
				[Fe ²⁺]	$= 7 \times 10^{-4}$	М			•
1	5	13.75	68.0	0.62	98.5	16.25	53.6	10.00	71.4
2	10	12.50	69.5	0.62	98.5	15.00	57.1	9.37	73.2
3	20	10.78	73.7	0.62	98.5	13.75	60.7	9.37	73.2
4	40	10.00	75.6	0.62	98.5	12.50	64.3	9.37	73.2
5	60	9.95	75.7	0.62	98.5	12.50	64.3	9.37	73.2

Table 2. Dependence of the mineralization degree (MDD) of the DBI and DBr dyes on the catalytic oxidation time at different concentrations of iron ions (II). pH = 2.5, $[H_2O_2] = 3 \times 10^{-3}$ M

erated coagulant (for removal of 1 mg of dye) occurred. Thus, the removal process of the dyes becomes more effective. This regularity is observed both for the DB1 and DBr. The decrease of the specific consumption of the electrogenerated coagulant is caused by the following. At the increase of the initial concentration of the direct dyes, due to the appearance of hydrogen bonds, the generation of large associates is observed. Hence, the surface charge of the particles decreases and a lower quantity of aluminum hydroxide is needed for their neutralization and coagulation. Therefore, it is more efficient, at first, to add so much electrogenerated coagulant that the residual concentration of the dyes equals 18–20 mg/l. In this case, the quantity of the generated sediment and of the spent coagulant decreases by two times.

For the next decrease of the dye concentration to the standards for open waters, the catalytic method of oxidation with the presence of hydrogen peroxide is suggested. Due to this, the dye molecules are decomposed in harmless carbonic acid and water.

As a result, the process of the removal of the dyes from the solutions by means of their catalytic oxidation with application of the hydrogen peroxide in the presence of iron ions (II) was investigated. The results are presented in Tables 2–4.

As is evident from Table 2, at different concentrations of iron ions (II), the mineralization of the dyes increases together with the increase of the oxidation time (without boiling). At that, the maximal degree of mineralization (78.5% in DBr and 82.2% in DBl) is observed at a concentration of the iron ions equaled 3×10^{-4} M. The increase of the temperature of the reaction (at boiling for 10 min) leads to an increase of the mineralization degree both for the DBl and DBr. At that, the maximal degree of mineralization (at the same concentration of iron ions (II)) reaches 98.49% in the DBl and 96.43% in the DBr. The value reaches the maximal

		"Direct Blue" dye (CCO $_0 = 41 \text{ mgO}_2/\text{I}$)				"Direct Brown" dye ($CCO_0 = 35 \text{ mgO}_2/l$)				
no.	t, min	without boiling		at boiling for 10 min		without boiling		at boiling for 10 min		
		CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	
$[H_2O_2]_0 = 2 \times 10^{-3} M$										
1	5	10.62	74.1	4.37	89.3	14.37	58.9	7.50	78.5	
2	10	10.00	75.6	4.37	89.3	14.37	58.9	6.87	80.4	
3	20	10.00	75.6	4.37	89.3	12.50	64.3	7.50	78.6	
4	40	8.75	78.7	4.37	89.3	11.87	66.1	7.50	78.6	
$[H_2O_2]_0 = 3 \times 10^{-3} M$										
1	5	12.50	69.5	0.62	98.5	10.62	69.7	1.25	96.4	
2	10	11.25	72.6	0.62	98.5	9.87	73.2	1.25	96.4	
3	20	10.00	75.6	0.62	98.5	8.75	75.0	1.25	96.4	
4	40	8.75	78.7	0.62	98.5	7.50	78.6	1.25	96.4	
	1		1	[H ₂ O ₂]	$_{0} = 5 \times 10^{-1}$	³ M	I	I	I	
1	5	16.87	58.8	0.62	98.6	23.12	33.9	5.00	85.7	
2	10	15.00	63.4	0.62	98.5	21.25	39.1	5.00	85.7	
3	20	13.12	68.0	0.62	98.5	20.00	42.9	5.00	85.7	
4	40	10.00	75.6	0.62	98.5	18.75	46.4	5.00	85.7	
$[H_2O_2]_0 = 7 \times 10^{-3} M$										
1	5	24.37	40.6	6.25	84.6	24.2	29.7	5.00	85.7	
2	10	22.50	45.1	6.25	84.6	21.70	38.0	5.00	85.7	
3	20	20.62	49.7	6.25	84.6	21.1	39.7	5.00	85.7	
4	40	18.75	54.3	6.25	84.6	19.2	45.0	5.00	85.7	

Table 3. Dependence of the mineralization degree of the dyes (MDD) on the oxidation time and the hydrogen peroxide. pH = 2.5; $[Fe^{2+}]_0 = 3 \times 10^{-4} \text{ M}$

mineralization degree at $t = 100^{\circ}$ C, indicating the maximal oxidation of the dyes. This value differs from the mineralization degree revealed after the oxidation of the dyes at their mixing for 5, 10, 20, 40, and 60 minutes at $t = 20^{\circ}$ C. The difference in the mineralization degree of the DBI and DBr indicates the different stability of these dyes regarding oxidizers. The DBr is more stable in regards to oxidation, and the value of its mineralization differs from such at DBI of 1.0–1.5% only.

Further, we investigated the process of the catalytic oxidation of the direct dyes depending on the concentration of hydrogen peroxide. According to Table 3, an increase of the concentration of the hydrogen peroxide to 3×10^{-3} M, at first, leads to an increase of the mineralization degree, and then it decreases for both of the dyes.

As in the case of using iron ions (II), the mineralization degree of the dyes increases together with the oxidation time at all the concentrations of the hydrogen peroxide. The maximal mineralization degree is 78.6– 78.7% (at H_2O_2 equal to 2×10^{-3} – 3×10^{-3} M and an oxidation time of 40 min).

The decrease of the mineralization degree of the dyes together with an increase of the hydrogen peroxide concentration may be caused by the appearance of a hydrogen peroxide excess due to part of the newly generating hydroxyl radicals being consumed as follows:

$$Fe^{2+} + HO \longrightarrow Fe^{3+} + HO;$$

$$H_2O_2 + HO \longrightarrow H_2O + HO_2.$$

Hence, at an increase of the hydrogen peroxide concentration, the mineralization degree of dyes decreases, because the concentration of the active hydroxyl radicals decreases too. By using the optimal concentration of hydrogen peroxide and iron ions, the concentration of the dyes due to the oxidation of them can be decreased (at their initial concentration up to 50 mg/l) to the maximum permissible concentration (MPC) according to value of the CCO_{res}.

Thereby, we investigated the process of the removal and mineralization of direct dyes depending on their initial concentration (Table 4). It is evident that, at an increase of the initial concentration of the dyes, the degree of their mineralization decreases a little and the value of CCO_{res} is more than such provided for wastewater addition to nature. Furthermore, the DBr oxidation (with the increase of its initial concentration) occurs ineffectively because of the great residual concentration of it remaining in the solutions.

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	<i>t</i> , min	"Direct I	Blue" dye ($CCO_0 = 41 \text{ mgO}_2$	/1)	"Direct Brown" dye (CCO ₀ = $35 \text{ mgO}_2/l$)				
no.		without boiling		at boiling for 10 min		without boiling		at boiling for 10 min		
		CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	CCO _{res} , mgO ₂ /l	MDD, %	
$C_0 = 50 \text{ mg/l}$										
1	5	8.75	78.7	0.625	98.5	10.62	69.7	1.25	96.4	
2	10	7.50	81.7	0.625	98.5	9.87	73.2	1.25	96.4	
3	20	5.00	87.8	0.625	98.5	8.75	75.0	1.25	96.4	
4	40	4.75	88.4	0.625	98.5	7.50	78.6	1.25	96.4	
$C_0 = 100 \text{ mg/l}$										
1	5	31.25	56.9	5.00	93.1	80.00	0.0	52.50	34.4	
2	10	25.00	65.5	5.00	93.1	80.00	0.0	47.50	40.6	
3	20	14.375	80.3	5.00	93.1	72.50	9.4	42.50	46.9	
4	40	13.75	81.0	5.00	93.1	45.00	43.7	37.50	53.1	
$C_0 = 150 \text{ mg/l}$										
1	5	35.00	65.8	4.37	95.7	105.00	0.0	75.00	28.6	
2	10	28.125	72.6	4.37	95.7	105.00	0.0	75.00	28.6	
3	20	23.75	76.8	4.37	95.7	105.00	0.0	70.00	33.3	
4	40	19.375	81.1	4.37	95.7	105.00	0.0	67.50	35.7	

Table 4. Dependence of the mineralization degree of the dyes (MDD) on the oxidation time and their initial concentration. pH = 2.5; $[Fe^{2+}]_0 = 3 \times 10^{-4} \text{ M}$

Thus, it was shown that the concentration of the dyes can be decreased to the MPC at the optimal parameters of the catalytic oxidation at the initial concentrations up to 50 mg/l.

To purify more concentrated solutions and wastewater to the MPC, it is necessary to combine electrocoagulation and catalytic oxidation by means of hydrogen peroxide.

CONCLUSIONS

1. It has been determined that the effect of the direct dyes' removal from the model solutions by means of electrogenerated coagulants depends on their initial concentration. Due to the increase of the dye concentration from 50 to 200 mg/l, the specific coagulant consumption decreases. Hence, it is more effective to remove these dues by means of electrogenerated coagulants to residual concentrations equaled to 18.0–20.0 mg/l.

2. It is more effective and economical to remove the direct dyes by means of electrogenerated coagulation from more concentrated model solutions and textile wastewater (when the initial dye concentration is more than 200 mg/l).

3. At the use of catalytic oxidation, direct dyes can be removed from less concentrated solutions (the dye concentrations must not be more than 50 mg/l).

4. It has been determined that the mineralization degree for both dyes depending on the concentration of

the iron ions (II) increases together with the increase of the catalytic oxidation time from 5 to 60 min.

5. At the increase of the concentration of the iron ions (II), the degree of the dyes mineralization firstly increases, reaches a peak at the ion concentration of $(Fe^{2+}) = 3 \times 10^{-4}$ M, and then finally decreases.

6. It has been determined that the model solutions and textile wastewaters containing more than 200 mg/l of dyes can be purified due to combining of the electrocoagulation and catalytic oxidation by hydrogen peroxide. At that, the degree of purification and of mineralization reaches 97-98%, and the quantity of the spent electrogenerated coagulant and of the sediment decreases by two times.

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