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ScienceDirect
Journal of Hazardous Materials 153 (2008) 194–200
Journal of Hazardous Materials
www.elsevier.com/locate/jhazmat

Optimization of Bomaplex Red CR-L dye removal from aqueous solution by electrocoagulation using aluminum electrodes

Yalçın Şevki Yildiz *

Department of Environmental Engineering, Atatürk University, 25240 Erzurum, Turkey

Received 29 May 2007; received in revised form 30 July 2007; accepted 13 August 2007
Available online 19 August 2007

Abstract

In this paper, Taguchi method was applied to determine the optimum dye removal from aqueous solution by electrocoagulation using aluminum electrodes. An orthogonal array (OA_{16}) experimental design that allows to investigate the simultaneous variations of five parameters (initial dye concentration, initial pH of the solution, supporting electrolyte concentration, supporting electrolyte type and current density) having four levels was employed to evaluate the effects of experimental parameters. Performance measure analysis was followed by performing a variance analysis, in order to determine the optimum levels and relative magnitude of the effect of parameters. Because the desired characteristic for response has been maximum decolorization, Taguchi's 'the larger the better' performance formula was used. While the optimum conditions were found to be initial dye concentration of 100 mg/L, initial pH of the solution of 3, supporting electrolyte concentration of 0.0 mM, supporting electrolyte type of $CaCl_2$ and current density of 0.50 mA/cm^2. Under these optimum conditions, energy consumption is 0.607 kWh/kg dye, when the system evaluated also based on the energy consumptions it can be said that optimum conditions should be modified as follows: supporting electrolyte concentration of 2.5 mM; supporting electrolyte type NaCl, for 100 mg/L initial dye concentration; initial pH of the solution of 3; current density of 0.50 mA/cm^2.

Keywords: Taguchi method; Optimization; Decolorization; Electrocoagulation

1. Introduction

The large quantity of aqueous waste generated by textile industries has becomes a significant environmental problem. Dye bath effluents, in particular, are not only aesthetic pollutants by nature of their color, but may interfere with light penetration in the receiving bodies of water, thereby disturbing biological processes. Furthermore, dye effluent may contain chemicals, which are toxic, carcinogenic, mutagenic or teratogenic in various microbiologic, fish species [1]. The characteristics of wastewater from textile dyeing are high or low pH, high temperature and a high concentration of coloring material [2].

Traditional methods for dealing with textile wastewater consist of various combinations of biological, physical and chemical methods [3]. Common biological treatment processes are often ineffective in removing dyes which are highly structured polymers with low biodegradability [4]. Various physical–chemical techniques are also available for the treatment of aqueous streams to eliminate dyes such as chemical coagulation followed by sedimentation [5], adsorption which are the widely used ones, [6] but other advanced techniques are often applied, for example, UV [7,8], ozonation [9], ultrasonic decomposition or combined oxidation processes [10–12]. When chemical coagulation is used to treat dyeing wastewater, the pollution may be caused by chemical substance added at a high concentration [2]. Meanwhile, high treatment costs of these methods have stimulated, in recent years, the search for more cost-effective treatment methods.

Electrocoagulation is a process consisting of creating a floc of metallic hydroxides within the effluent to be cleaned, by electrodissolution of soluble anodes. Compared with traditional flocculation and coagulation, electrocoagulation (EC) has in theory, the advantage of removing the smallest colloidal particles: the smallest charged particles have a greater probability

* Tel.: +90 442 2314799; fax: +90 442 2314799.
E-mail address: ysevki@yahoo.com.

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doi:10.1016/j.jhazmat.2007.08.034

of being coagulated because of the electric field that sets them in motion. It has also the advantage of producing a relatively low amount of sludge. Secondary pollution may be caused by chemical substance added at a high concentration when chemical coagulation is applied to treat dyeing wastewater. Excessively added coagulants can be avoided by EC, due to the generation of the coagulants by electrooxidation of a sacrificial anode. The characteristics of EC are simple equipment and easy operation, brief reactive retention period, decreased or negligible equipment for adding chemical and decreased amount of sludge [13].

The most common electrode materials for EC are aluminum and iron. They are cheap, readily available and proven effective [14]. When aluminum is used as electrode material, the reactions are as follows.

At the cathode:

$$3H_2O + 3e^- \rightarrow (3/2)H_2(g) + 3OH^- \quad (1)$$

At the anode:

$$Al \rightarrow Al^{3+} + 3e^- \quad (2)$$

In the solution:

$$Al^{3+}(aq) + 3H_2O \rightarrow Al(OH)_3 + 3H^+(aq) \quad (3)$$

The H_2 produced as a result of the redox reaction may remove dissolved organics or any suspended materials by flotation and it can be said that the this phenomenon is one of the advantages of the EC process.

EC treatment of textile dye-containing solutions or wastewaters samples has been tested on a laboratory scale and good removal of COD, color, turbidity and dissolved solids at varying operating conditions were obtained [2,13,15–18].

There is a wide range of applications of Taguchi method, from chemistry to engineering [18–25]. The advantage of the Taguchi method on the conventional experimental design methods, in addition to keeping the experimental cost at a minimum level, is that it minimizes the variation in product response while keeping the mean response on target. Its other advantage is that the optimum working conditions determined from the laboratory work can also be reproduced in the real production environment [18].

Aim of this investigation, is to explore the decolorization by electrocoagulation from the solutions containing Bomaplex Red CR-L dye and to determine the influence of the variables such as initial dye concentration, current density, initial pH, supporting electrolyte concentration and type on decolorization process. The experiments were carried out according to Taguchi orthogonal array (OA) experimental design with two replicate and four center points.

2. Materials and methods

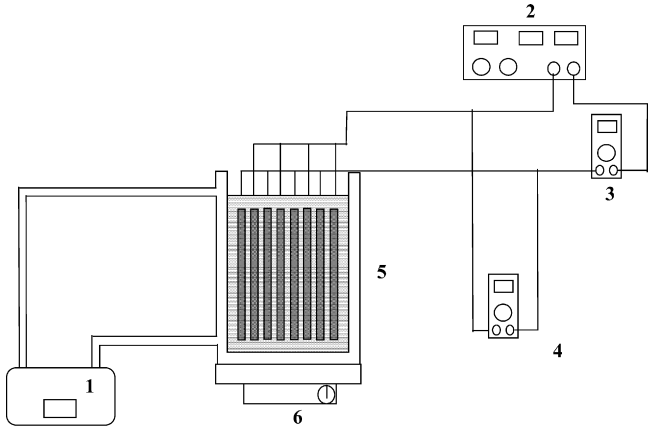
2.1. Materials

Bomaplex Red CR-L dye used in this study was supplied from a textile mill in Turkey (dye textile industry company project, Gazi Antep). Dye solutions were prepared by dissolving desired amounts of dye in 1 L of distilled water and used. All chemicals used were analytical grade and used without any further treatment. Distilled water was used in all experiments. Solutions were prepared from NaCl (Merck, 99.5%), $NaNO_3$ (Merck, 99%), Na_2SO_4 (Sigma–Aldrich, 99%) and $CaCl_2$ (Merck, >90%) used as supporting electrolyte. Treated solution was collected over a desired period of time from the reactor and collected samples were centrifuged (Runne Heidelberg) 10,000 rpm and 10 min before the analysis. The dye was analyzed spectrophotometrically. A high precision, double-beam spectrophotometer (Shimadzu UV-160A) was used to measure the absorbance of dye solution at wavelengths between 200 and 800 nm and 450 nm is chosen as the suitable wavelength in this study to measure the dye concentration in water. The initial pH was adjusted to a desired value using NaOH (Merck, 5N) or HNO_3 (Carlo Erbaa, 65%).

2.2. Experimental setup and procedure

The experimental setup is schematically shown in Fig. 1. The EC unit consists of five pair of electrodes made of plate aluminum with total area of approximately 1000 cm^2 and the gap between the electrodes is 5 mm. Electrodes were connected to a digital DC power supply (Shenzen-Mastech HY 3005-3) in monopolar mode. Two digital multimeters (Brymen Bm 201) as ampermeter and voltmeter were used to measure the current passing through the circuit and the applied potential, respectively.

The EC unit has been stirred at 150 rpm by a magnetic stirrer. (Heidolp MR 3004 S). The thermostated electrocoagulator is made of plexiglass with the volume of 800 mL. During the experiments, temperature, conductivity and pH of the solutions were measured by a multi-parameter (WTW Multiline P-4 F-Set-3). Reactor was operated in batch and galvanostatic mode.



1. Water Circulator
2. Digital D.C. Power Supply
3. Digital Ampermeter
4. Digital Voltmeter
5. Electrochemical Reactor
6. Digital Magnetic Stirrer

Fig. 1. Schematic diagram of the experimental setup.

Table 1
Variables and their values corresponding to their levels investigated in the experiments

Variables	Levels			
	1	2	3	4
A: initial dye concentration (mg/L)	100	200	400	600
B: initial pH of the solution	3	4	5	6
C: supporting electrolyte concentration (mM)	0.0	2.5	5.0	10.0
D: supporting electrolyte type	NaCl	$NaNO_3$	Na_2SO_4	$CaCl_2$
E: current density (mA/cm^2)	0.25	0.50	0.75	1.00

2.3. Statistical analysis

The variables chosen for this investigation are initial dye concentration, initial pH of the solution, supporting electrolyte concentration and type and current density. The variables investigated and their levels were summarized in Table 1. Reaction period was kept constant in 30 min for statistical analysis. In order to optimize the dye removal process, experimental parameters and their levels investigated are given in Table 1.

The experimental design, based on standard OA_{16} (5^4) orthogonal array, is conducted to change the settings of the various process parameters (Table 2).

Because it is the most suitable for the conditions being investigated, five parameters, each with four levels was considered. In order to observe the effects of noise sources (uncontrollable factors) on this process, each experimental trial was repeated twice under the same conditions at different times. Also, the order of experiments was made random in order to avoid noise sources which had not been considered initially and which could take place during an experiment and affect the results in a negative way. Performance measure analysis reflecting the variation in the response at each setting was chosen as the optimization criteria. Its analysis determines the controllable factors and their settings, which minimize the variation in process while keeping the mean response on target. By setting those factors at their optimal levels, the process can be made robust to changes in operating and environmental conditions. When the desired characteristic for the response is the larger, it is better; Taguchi recommends the use of larger is better:

$$\frac{S}{N} = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y_i^2}\right) \quad (4)$$

where S/N is performance characteristics, n the number of repetitions done for an experimental combination and Y_i is the performance value of the ith experiment. The performance value corresponding to the optimum working conditions can be predicted by utilizing the balanced characteristic of OA. For this, the following additive model may be used:

$$Y_i = m + X_i + e_i \quad (5)$$

where m is the overall mean of performance value, X_i the fixed effect of the parameter level combination used in the ith experiment and e_i is the random error in the ith experiment. Detailed description of the Taguchi method can be found in Refs. [19–23].

3. Results and discussions

3.1. Statistical analysis

Fig. 2 illustrates the variation of dye removal efficiency (η) with time. Fig. 2 is plotted using the data obtained from experi-

Table 2
Experimental variables, their levels and results of conducted experiments corresponding to L16 experimental plan

Experiment no.	Variables and their levels					Dye removal efficiency (%)		
	A	B	C	D	E	First series	Second series	Average
1	1	1	1	1	1	92.78	96.79	94.79
2	1	2	2	2	2	87.43	82.09	84.76
3	1	3	3	3	3	58.19	60.50	59.35
4	1	4	4	4	4	99.94	99.13	99.54
5	2	1	2	3	4	91.12	83.67	87.40
6	2	2	1	4	3	95.94	92.42	94.18
7	2	3	4	1	2	83.40	82.37	82.89
8	2	4	3	2	1	51.53	59.81	55.67
9	3	1	3	4	2	98.63	98.71	98.67
10	3	2	4	3	1	34.32	34.66	34.49
11	3	3	1	2	4	85.29	78.01	81.65
12	3	4	2	1	3	77.73	79.45	78.59
13	4	1	4	2	3	67.89	65.29	66.59
14	4	2	3	1	4	74.01	69.87	71.94
15	4	3	2	4	1	87.81	91.13	89.47
16	4	4	1	3	2	74.01	76.71	75.36

Table 3
Results of the analysis of variance for the dye removal efficiencies

Variables	Sum of squares (SS)	Degrees of freedom (DOF)	Mean of squares (MS)	F	Cr (%)
A: initial dye concentration (mg/L)	665.50	3	221.83	20.97	6.53
B: initial pH of the solution	1172.82	3	390.96	36.96	11.76
C: supporting electrolyte concentration (mM)	1912.52	3	637.52	60.26	19.39
D: supporting electrolyte type	4296.57	3	1432.19	135.38	43.97
E: current density (mA/cm^2)	1483.20	3	494.40	46.73	14.96
Error	169.26	16	10.58		
Total	9699.87	31			

ment 15 and similar tendencies were observed in other test runs (not shown).

Sharp increases of removal efficiencies are clearly observed initially. After the treatment period of approximately 30 min the removal efficiencies approach plateaus at 90% (the point with arrow in Fig. 2), thus all observations were performed for a reaction time of 30 min otherwise stated.

The collected data for dye removal was analyzed using the computer software package program (MINITAB Release 13.20) for the evaluation of the effect of each parameter on the optimization criteria. The results are given in Fig. 3.

The optimal level of a process parameter is the level with the highest S/N value calculated by Eq. (4). Fig. 3 shows the variation of the performance characteristics with the variables. To determine the experimental conditions for the first data point, the initial pH of the solution for that point is level 1 which is 3 for this parameter. The experiments for which initial pH of the solution level is 1 are experiments 1, 5, 9 and 13. The performance characteristics value of the first data point is thus the average of those obtained from experiments 1, 5, 9 and 13. Thus, experimental conditions for the second data point are the conditions of the experiments for which column C is 2 (experiments 2, 5, 12 and 15). The numerical value of the maximum point in each graph marked the best value of that particular parameter and was found as A1 (100 mg/L), B1 (3), C1 (0 mM), D4 ($CaCl_2$) and E2 (0.5 mA/cm^2). These parameter values provide the optimum conditions.

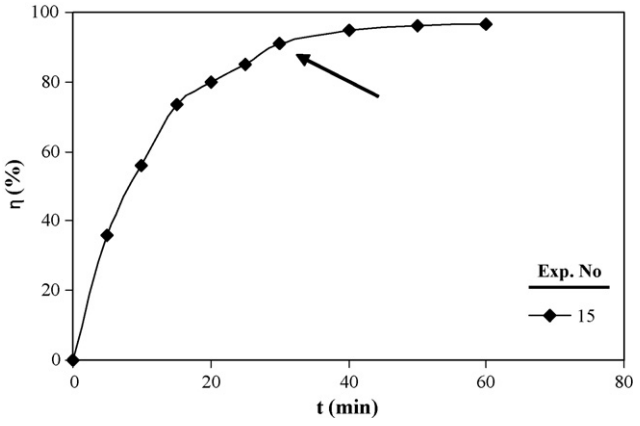


Fig. 2. Variation of dye removal efficiency with time (C_0 = 600 mg/L, pH$_i$ = 5.0, C_{SE} = 2.50 mM, $CaCl_2$ and CD = 0.25 mA/cm^2).

The optimal levels of these factors are the levels with the maximum performance measures that is, with minimum variability. It is clear from performance measures and ANOVA (Table 3) that factors supporting electrolyte type and concentration, and to a lesser degree factors current density and initial pH of the water, significantly effect the variation in the response.

According to Taguchi, the use of the F ratios in an ANOVA analysis is only helpful for the qualitative evaluation of whether factorial effects exist. For quantitative evaluation, this something that can be achieved through the use of a contribution ratio (Cr). The contribution ratio of a main factor effect is its contribution (in percentage terms) to the total variability of the experimental results [19,20]. The contribution ratio can be achieved by dividing the source's net variation by SS_{total}, which is given as follows

$$Cr_A = \frac{SS_A - DOF \times MSS_{error}}{SS_{total}} \times 100 \quad (6)$$

It is clear from the Cr column of Table 3 that the highest contributors to the variability of the experimental results are supporting electrolyte type and supporting electrolyte concentration, with the supporting electrolyte type and concentration accounting for more than 63% of total variation.

From Fig. 2, the optimal levels of these factors are initial dye concentration (A1: 100 mg/L), initial pH of the solution (B1:3), supporting electrolyte concentration (C1: 0 mM), supporting electrolyte type (D4: $CaCl_2$) and current density (D2: 0.50 mA/cm^2). If the experimental plan given in Table 3 is studied carefully, it can be seen that this combination of factor levels (1, 1, 1, 4 and 2) was not 1 of the 16 combinations tried in the experiment. This is to be expected because of the high fractionality of the experimental design used (16 out of 4^5 = 1024 possible combinations). In order to test the predicted results, confirmation experiments were carried out once at the same working conditions. Thus, some confirmation runs also including optimum working conditions were made and presented in Table 4.

If experimental results are stated in a percentage (%), such as removal efficiency before evaluating Eq. (5), the Ω transformation of percentage values should be applied first using Eq. (7) by which values of interest are also determined later by carrying out a reverse transformation by using the same equation [24]:

$$\Omega\,(\mathrm{db}) = -10 \log\left(\frac{1}{P} - 1\right) \quad (7)$$

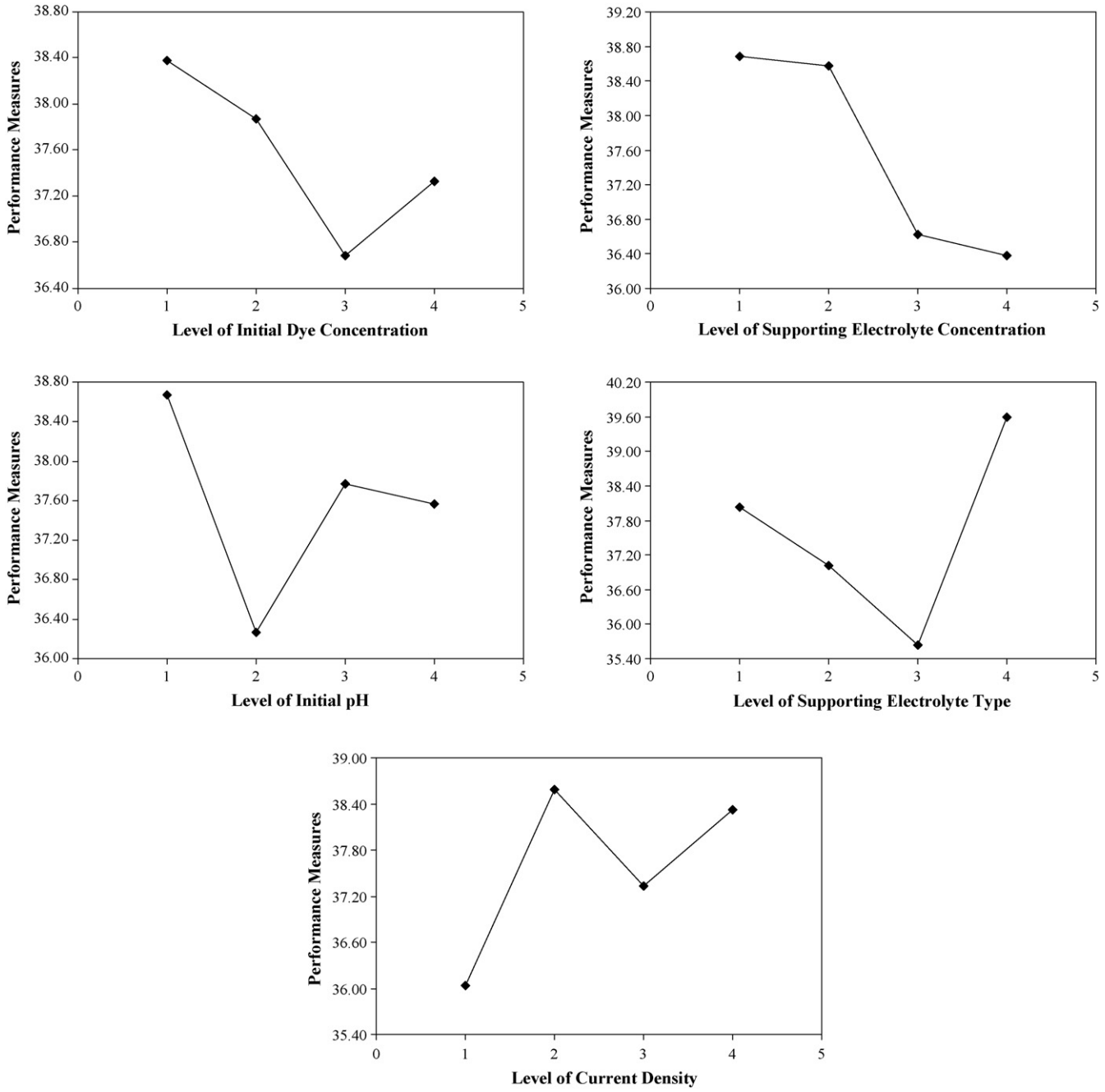


Fig. 3. Effect of parameters on optimization criteria.

Table 4
Optimum and alternative working conditions for different experimental runs observed and predicted dye removal efficiencies

Case no.	A (C_0)		B (pH$_i$)		C (SEC)		D (SET)		E (CD)		Observed	Predicted	Confidence limit
	Level	Value	Level	Value	Level	Value	Level	Value	Level	Value	–	–	–
1[a]	1	100	1	3	1	0	4	$CaCl_2$	2	0.5	99.1	99.8	90.3–100.0
2	1	100	1	3	2	2.5	1	NaCl	2	0.5	96.4	97.5	88.0–100.0
3	1	100	2	4	1	0	4	$CaCl_2$	2	0.5	93.0	99.2	89.7–100.0
4	1	100	1	3	1	0	4	$CaCl_2$	4	1.0	98.0	99.9	90.4–100.0
5	4	600	1	3	2	2.5	1	NaCl	2	0.5	83.15	89.1	79.6–98.6

[a] The parameter levels for optimum dye removal efficiency.

where Ω (db) is the decibel value of percentage value subject to omega transformation and P is the percentage of the product obtained experimentally. Since Eq. (6) is a point estimation which is calculated by using experimental data in order to determine whether the additive model is adequate or not, the confidence limits for the prediction error must be evaluated [25]. The prediction error is the difference between the observed Y_i and the predicted Y_i. The confidence limits for the prediction error are

$$\mathrm{Se} = \pm 2\sqrt{\left[\frac{1}{n_0}\right]\sigma_e^2 + \left[\frac{1}{n_r}\right]\sigma_e^2} \quad (8)$$

$$\sigma_e^2 = \frac{\text{sum of squares due to error}}{\text{degrees of freedom for error}} \quad (9)$$

$$\frac{1}{n_0} = \frac{1}{n} + \left[\frac{1}{n_{A_i}} - \frac{1}{n}\right] + \left[\frac{1}{n_{B_i}} - \frac{1}{n}\right] + \left[\frac{1}{n_{C_i}} - \frac{1}{n}\right] \cdots \quad (10)$$

where Se is the two-standard deviation confidence limit, n the number of rows in the matrix experiment, n_r the number of repetitions in the confirmation experiment and $n_A, n_B, n_C, \ldots$ are the replication numbers for the parameter levels $A_i, B_i, C_i, \ldots$. If the prediction error is outside these limits, the possibility that the additive model is not adequate should be suspected. Otherwise, the additive model can be considered to be adequate.

Also, the results in Table 4 are confidence limits of predictions. In order to test the predicted results, confirmation experiments were carried out once at the same working conditions. The fact that the removal efficiencies from the confirmation experiments are within the calculated confidence intervals calculated from Eqs. (8)–(10) (Table 4) shows that the experimental results are within ±5% in error. This case states that there is a good agreement between the predicted values and experimental values and the interactive effects between the parameters are indeed negligible. It may be concluded that the additive model is adequate for describing the dependence of the dye removal process on the various parameters [25].

Energy consumptions and removal efficiencies obtained in the experiments is the same conditions as Table 4 is presented in Fig. 4. When examining Fig. 4, system has consumed the electrical energy of 0.607 kWh/kg dye in optimum conditions (experiment #1). Essentially, experiment #2 is a good alternative when experiments have been evaluated based on removal efficiencies and energy consumptions. For example, in optimum conditions (experiment #1) while system has consumed the electrical energy of 0.607 kWh/kg dye (corresponding to removal efficiency of 99.1%) in experiment #2 energy consumption equals to 0.397 kWh/kg dye and removal efficiency is 96.4%.

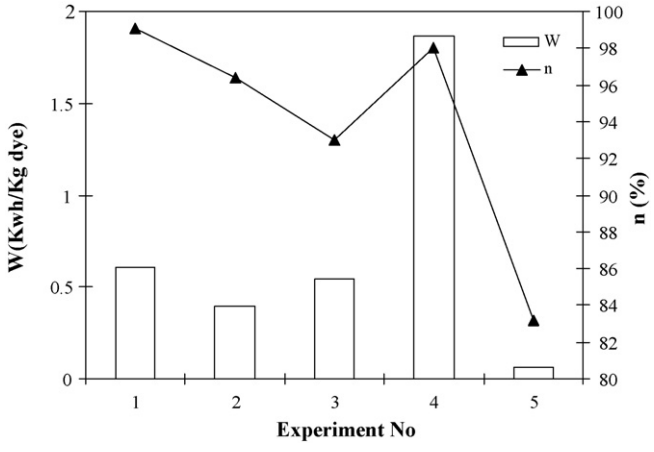


Fig. 4. Removal efficiencies and energy consumptions for optimum and alternative working conditions.

In light of the above conclusions, it can be said that energy consumptions are also evaluated together removal efficiencies. Therefore in order to get the cost-effectiveness, treatment system should be provided higher removal efficiencies and at the same time lower energy consumptions if possible.

4. Conclusions

In this paper, Taguchi method has been used to determine the optimum working conditions for the dye removal from aqueous solutions by EC. The orthogonal array, OA_{16} (4^5), technique is described for experimental design as it reduces the number of experiments required to investigate a set of parameters and to minimize time and cost while performing experiments. Experimental investigations into the parameter effects have allowed to determine the optimum configuration of design parameters for dye removal performance.

It can be said that EC is highly effective process for dye removal from aqueous solutions, due to obtained efficiencies based on decolorization are in general satisfactory levels (Table 2).

The experiments on dye removal by EC process indicated that the significant parameters were supporting electrolyte type, supporting electrolyte concentration and current density, in which supporting electrolyte type was the most effective factor in the total variation of dye removal process; initial pH and initial dye concentration had an effect at a lesser degree. The use of the F ratios in an ANOVA analysis is only helpful for the qualitative evaluation of whether factorial effects exist. For quantitative evaluation, contribution ratio (Cr) has been used. It can be concluded that supporting electrolyte type, supporting electrolyte concentration and current density are the effective parameters on dye removal by EC according to Cr values in Table 3. Cr value of supporting electrolyte type which equals to about 44% is the biggest one among the Cr values, so it is the most effective parameter on process. Therefore, detailed structural investigation should be conducted for the determination of chemical specie formed in the solution with different supporting electrolytes.

According to Taguchi's model the optimum conditions have been suggested as the first level of dye concentration (100 mg/L), first level of initial pH of the solution (3), first level of supporting electrolyte concentration (0.0 mM), fourth level of supporting electrolyte type ($CaCl_2$) and second level of current density (0.50 mA/cm^2). Under these optimum conditions, removal efficiency was 99.1% (Table 4). Additionally, some alternative conditions should be evaluated based on both removal effi-

ciencies and energy consumptions. Therefore, experiment #2 in Table 3 should be taken into consideration. Because the energy consumptions are 0.397 and 0.607 kWh/kg dye for the experiments #2 and #1, respectively.

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