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Color removal from textile dyebath effluents in a zeolite fixed bed reactor: Determination of optimum process conditions using Taguchi method

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ABSTRACT

Taguchi method was applied as an experimental design to determine optimum conditions for color removal from textile dyebath house effluents in a zeolite fixed bed reactor. After the parameters were determined to treat real textile wastewater, adsorption experiments were carried out. The breakthrough curves for adsorption studies were constructed under different conditions by plotting the normalized effluent color intensity (C/C_0) versus time (min) or bed volumes (BV). The chosen experimental parameters and their ranges are: HTAB concentration (C_{htab}), 1–7.5 g L^{-1}; HTAB feeding flowrate (Q_{htab}), 0.015–0.075 L min^{-1}; textile wastewater flowrate (Q_{dye}), 0.025–0.050 L min^{-1} and zeolite bed height (H_{bed}), 25–50 cm, respectively. Mixed orthogonal array L_{16} ($4^2 \times 2^2$) for experimental plan and the larger the better response category were selected to determine the optimum conditions. The optimum conditions were found to be as follows: HTAB concentration (C_{htab}) = 1 g L^{-1}, HTAB feeding flowrate (Q_{htab}) = 0.015 L min^{-1}, textile wastewater flowrate (Q_{dye}) = 0.025 L min^{-1} and bed height (H_{bed}) = 50 cm. Under these conditions, the treated wastewater volume reached a maximum while the bed volumes (BV) were about 217. While HTAB concentration, g L^{-1} (*A*); zeolite bed height, cm (*D*) and wastewater flowrate, L min^{-1} (*C*) were found to be significant parameters, respectively, whereas, HTAB flowrate, L min^{-1} (*B*) was found to be an insignificant parameter.

1. Introduction

The textile industries discharge wastewater originating from the dyeing and finishing processes. The use of organic dyes in these industries has reached an amount of 700 000 tonnes/year while an annual world production of 30 million tonnes of textiles is present [1]. Reactive azo dyes are presently the most important compounds, constituting about 20–40% of the total dyes used for coloring [2]. Dye is the most 'difficult to treat' constituent of the textile wastewater. Reactive dyes are hardly biodegraded in an aerobic environment. Hence, their presence in wastewater is undesirable, and it is essential to remove coloring material from effluents before being discharged in the environment. This is important in regions, especially, where water resources are scarce [3–5].

Such voluminous quantities pose considerable environmental problems because reactive dyes are water-soluble and cannot be easily removed by conventional methods such as chemical coagulation/flocculation and biological methods [6]. The removal of colorants and other organic pollutants from industrial wastewater is considered an important application of the adsorption process using suitable adsorbents [7]. The presence of 4.5 million tonnes of natural zeolites of high quality, mainly those of clinoptilolite zeolite in Turkey, creates an impetus for the utilization of clinoptilolite in wastewater treatment [5]. The typical unit cell formula of natural zeolite mineral, clinoptilolite, is given as $Na_6[(AlO_2)_6(SiO_2)_{30}]\cdot 24H_2O$ [8]. Zeolite has been studied in wastewater treatment in recent years such as adsorption of ammonia [9,10], heavy metals [11–13] and reactive dyes [5,14] in batch mode and fixed bed reactors.

In the research and development stage, Taguchi method has been found effective by means of improved productivity, which brings along obtaining high quality items at low costs. Also, this method has been found applicable in a wide range of industrial fields all over world [15–17].

In this study, the color removal from real textile wastewater in a zeolite fixed bed reactor was investigated. The effect of experimental parameters such as HTAB concentration (C_{htab}), HTAB feeding flowrate (Q_{htab}), textile wastewater flowrate (Q_{dye}) and zeolite bed height (H_{bed}) on the color removal were investigated using an L_{16} ($4^2 \times 2^2$) orthogonal array. The Taguchi experimental design

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Table 1
Chemical composition of Gördes clinoptilolite

Constituent	Zeolite (% by wt)
Purity	92–96
SiO_2	70.5
CaO	2.90
K_2O	1.75
Al_2O_3	13.5
MgO	1.2
TiO	0.05
P_2O_5	0.05
Fe_2O_3	1.10
Na_2O	0.40
H_2O	4.00
LOI[a]	4.55
pH	7–7.65

[a] Loss on ignition.

method was used to determine optimum color removal conditions for maximizing the treated wastewater volume related to bed volumes (BV).

2. Materials and methods

2.1. Experimental system

The clinoptilolite sample – hereafter referred as zeolite – used in the experiments was received from Incal Mining Company in the Gördes region of Turkey. The chemical analysis of zeolite is given Table 1. In the experiments, real textile wastewater was used which was supplied from a textile factory located in Kayseri, Türkiye. In order to increase the adsorption capacity, the surface of natural zeolite was modified with a typical quaternary amine surfactant. hexadecyltrimethylammonium bromide (HTAB, $C_{19}H_{42}BrN$).

The laboratory-scale experimental set-up consists of zeolite fixed bed column, HTAB solution and real textile wastewater tanks, peristaltic pump, flowmeter, valves and treated water tank. The cylindrical plexiglas column has a diameter of 3 cm and a height of 100 cm. The particle size of clinoptilolite is 0.5–1 mm (35–18 mesh) and has bed heights of 25 and 50 cm.

A sample of 15 g HTAB was dissolved in a known volume distilled water to obtain the desired concentration of HTAB solution. Experiments were performed to determine the optimum conditions using HTAB solution for preparing modified zeolite and subsequent color removal from real textile wastewater by modified zeolite in the column system. The system is made of a fixed bed column to which HTAB solution (1–7.5 g L^{-1}) or real textile wastewater is pumped with a peristaltic pump (Seko PR1) from a tank to the top of the column at a particular flowrate.

The analysis for the cationic surfactant (HTAB) was performed by a two-phase titration technique applied to anionic surfactants using dimidium bromide and disulfine blue as indicators. This technique is based on the formation of a complex structure between anionic surfactant (sodium dodecylsulfate) and cationic reagent (HTAB). The complex is soluble in chloroform and changes from blue to pink in the presence of indicators [18,19].

The effectiveness of the modification of zeolite surface with HTAB was tested by the color removal ability of the fixed bed reactor. Color was measured by UV–vis spectrophotometer (Aquamate) at peak absorption wavelength of real textile wastewater (448 nm). Absorbance spectrum gave a peak at 448 nm between 350 and 700 wavelength as shown in Fig. 1 [20]. Adsorption performance of the zeolite bed was evaluated while bed volumes (BV) at breakthrough point ($C/C_0 < 0.1$) is maximum. The breakthrough curves were constructed by plotting the normalized effluent color intensity (C/C_0) [21] versus time and/or bed volumes (BV) which is defined as follows [14]:

$$BV = \frac{V_F}{V_R} = \frac{Q_F t}{V_R} \quad (1)$$

where V_F is the total water volume passing through the column during the adsorption process (m^3), V_R is the fixed bed volume of clinoptilolite (m^3), C_0 is influent color intensity, C is effluent color intensity, Q_F is the feed flowrate in the fixed bed ($m^3\,s^{-1}$) and t is the adsorption time (s).

2.2. Taguchi method

The Taguchi method, which is developed by Taguchi, is a complete application in design and analysis of experiments. Optimization of processes of engineering experimentation by this or suchlike methods is expressed with the concept “quality engineering”. The methods emphasize the application of engineering knowledge rather than advanced statistical techniques [22]. Taguchi’s ideas do not focus on the mathematical aspects of the design of experiments, but on the philosophy. In his methods, parts of classical methods are joined with cost considerations.

The greatest difference between the Taguchi methods and classical methods is that in Taguchi’s experiments orthogonal arrays are used to assure the reproduction of the effects of parameters. Another difference is that various types of “signal to noise” (S/N) ratios are used in a Taguchi study in order to measure variability around the target performance [23–25].

In the optimization of a process with various process performance characteristics for parameter design of Taguchi method, given steps should be followed [23,26]. First, performance characteristics are identified and process parameters that are to be estimated are selected. Second, it is decided that how many number of parameter levels there are and what kind of mutual effects are possible for the process parameters. Next, appropriate orthogonal array (OA) is selected and process parameters are assigned to the orthogonal array. After that, experiments are ready to be run based on the arrangement of the orthogonal array. Then, performance statistics are calculated. After the calculations, experimental results are analyzed with the help of performance statistics and analysis of variance (ANOVA). Following is the selection of the optimal levels of process parameters. Finally, optimal process parameters are verified through the confirmation experiment.

There are three basic S/N ratios. However, according to Fowlkes and Creveling, the variety of S/N ratios is limitless [27]. In this study, the performance statistics of “the larger the better” was used to define the optimum conditions [24,26]. “The larger the better” performance statistics was given by,

$$SNL = -10 \log \left(\frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2} \right) \quad (2)$$

where SNL is the S/N ratio or the performance characteristics as “the larger the better”, y_i is the comparison variable in experiment i for a certain combination of control factor levels, and n is the number of experiments performed for that combination.

Optimum operation conditions may have not been obtained during the entire experimental section. In such cases, the balanced characteristic OA can help predicating the performance value corresponding to the optimum operation conditions, where the following additive model may be used:

$$Y_i = \mu + X_i + e_i \quad (3)$$

where μ is the overall mean of the performance value, X_i is the fixed effect of the quantity level combination used in ith experiment, and e_i is the random error in the ith experiment [16]. Because Eq.

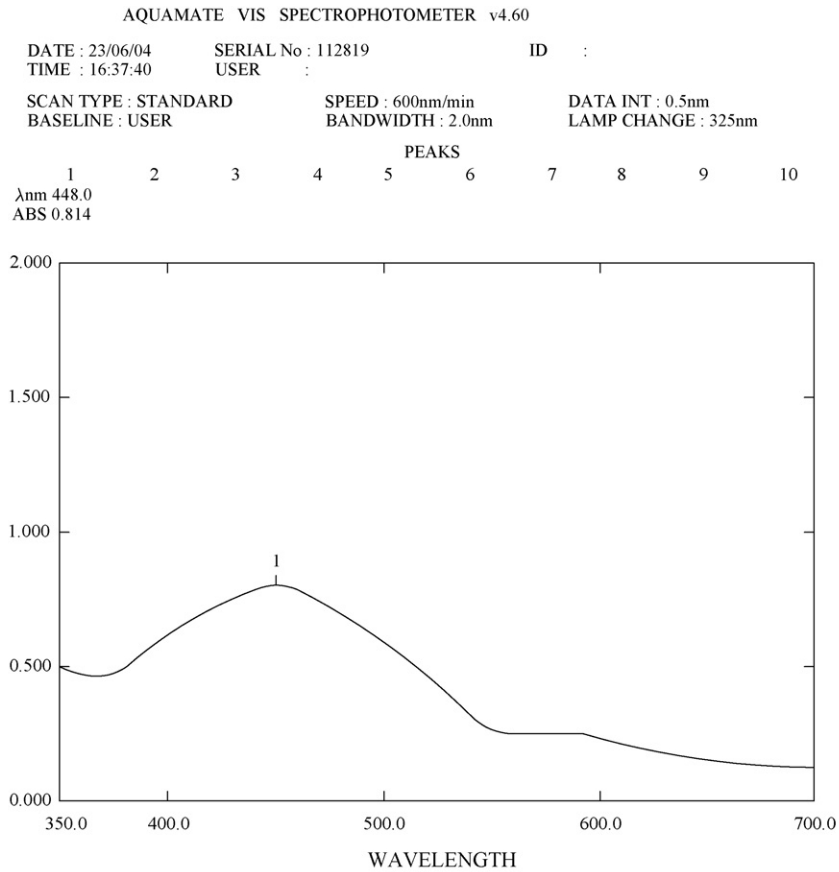

Fig. 1. UV–vis absorbance spectrum of real textile wastewater sample used in the experiments.

(3) is a point estimation, which is calculated by using experimental data in order to determine whether results of the confirmation experiments are meaningful or not, the confidence interval must be evaluated. At the selected error level, the safety interval is calculated with:

$$Y_i \pm \mathrm{CI}\left(\mathrm{CI} = \sqrt{F_\alpha(1, f_e) \times v_e \times \left[\frac{1}{n_{\mathrm{eff}}} + \frac{1}{S}\right]}\right) \tag{4}$$

where, $F_\alpha\,(1, f_e)$ is F-ratio at a confidence level of $(1-\alpha)$ against DOF 1, f_e is the error degree of freedom (DOF), n_{eff} is $(N/(1+(\text{total DOF associated in the estimate of mean})))$, N is total number of results, S is the sample size for confirmation test, v_e is error variance, CI is confidence interval [16,26].

Orthogonal arrays (OAs) were originally developed by Taguchi to control experimental error. OAs are constructed in such a way that, for each level of any factor, all levels of other factors occur equal number of times, which gives a balanced design. As compared to a full factorial design, the number of experiments in Taguchi's technique is substantially reduced. For the selection of a particular OA, the number of parameters, the number of levels and their possible interactions must be taken into consideration [28].

In this study, four parameters are considered to determine their best levels in order to optimize the color removal from the real textile dyed wastewater. These are: (1) C_{htab} (HTAB concentration), (2) Q_{htab} (HTAB feeding flowrate), (3) Q_{dye} (textile wastewater flowrate), and (4) H_{bed} (zeolite bed height). At the end of the engineering discussion on parameters's level to be used in experiments, four levels for C_{htab} and Q_{htab} parameters and two levels for the other Q_{dye} and H_{bed} parameters were selected, as shown in Table 2.

The Taguchi experimental design method was used to determine optimum color removal conditions for maximizing the treated wastewater volume (V_F) per zeolite fixed bed volume (V_R), namely bed volumes ($\mathrm{BV} = V_F/V_R$). The response parameter of these experiments is bed volumes (BV). Actually, in this situation, to have a whole idea about this response parameter, 64 ($=4\times4\times2\times2$) experiments have to be carried out in classical engineering studies. However, instead of dealing with 64 experiments, which is more costly and time consuming, Taguchi fractional orthogonal arrays were preferred to be used. Only main effects were considered, assuming that there were no reasonable interaction effects among these four parameters. Therefore, the Taguchi L16 ($4^2\times2^2$), orthogonal mixed fractional factorial array (four levels for C_{htab} and Q_{htab} parameters and two levels for the other Q_{dye} and H_{bed} parameters)

Table 2
Experimental parameters and their levels

Process parameter	Designation	Level 1	Level 2	Level 3	Level 4
HTAB concentration ($\mathrm{g\,L^{-1}}$)	*A*	1	2	3	7.5
HTAB flowrate ($\mathrm{L\,min^{-1}}$)	*B*	0.015	0.025	0.050	0.075
Wastewater flowrate ($\mathrm{L\,min^{-1}}$)	*C*	0.025	0.050		
Zeolite bed height (cm)	*D*	25	50		

Table 3
Taguchi L16 ($4^2 \times 2^2$) orthogonal array and results for bed volumes ($BV = V_F/V_R$)

Experiment no.	*A* C_{htab} ($g\,L^{-1}$)	*B* Q_{htab} ($L\,min^{-1}$)	*C* Q_{dye} ($L\,min^{-1}$)	*D* H_{bed} (cm)	Y_i BV_i
1	1	1	1	1	144
2	1	2	1	1	108
3	1	3	2	2	154
4	1	4	2	2	149
5	2	1	1	2	113
6	2	2	1	2	128
7	2	3	2	1	43
8	2	4	2	1	42
9	3	1	2	1	128
10	3	2	2	1	80
11	3	3	1	2	166
12	3	4	1	2	136
13	4	1	2	2	110
14	4	2	2	2	110
15	4	3	1	1	93
16	4	4	1	1	115

was selected in order to be able to find out the optimum levels of parameters [29]. In doing this, Minitab® 15 Statistical Software was used.

3. Results and discussion

The collected data were analyzed by using Minitab® 15 Statistical Software for the evaluation of the effect of each parameter on the optimization criteria. In order to determine the effective parameters and their confidence levels on the color removal process, an analysis of variance was performed. A statistical analysis of variance (ANOVA) was performed to see which process parameters were statistically significant. *F*-test is a tool to see which process parameters have a significant effect on the dye removal value. The *F*-value for each process parameter is simply a ratio of the mean of the squared deviations to the mean of the squared error.

The color removal from the real textile wastewater was investigated in different experimental conditions. To determine the optimum conditions for the color removal in the dyebath effluents, the effects of HTAB concentration (C_{htab}), HTAB feeding flowrate (Q_{htab}), textile wastewater flowrate (Q_{dye}), and zeolite bed height (H_{bed}) were investigated respectively. The experimental results and the reaction conditions for which the effect of parameters were investigated are given in Table 3.

The degrees of the influences of parameters on the performance statistics are given in Table 4 and Fig. 2. "The larger the better" responses are generally considered when the objective of the experiment is to maximize the response such as strength, efficiency etc. as it is in this work BV (bed volumes). The main expectation in this study is to maximize the response parameter BV as far as possible, so, as seen in Table 4 and Fig. 2, "*A*1 ($C_{htab} = 1\,g\,L^{-1}$), *B*1 ($Q_{htab} = 0.015\,L\,min^{-1}$), *C*1 ($Q_{dye} = 0.025\,L\,min^{-1}$), and *D*2 ($H_{bed} = 50$ cm)" combination is the best solution from the point of the mean of process response.

Table 4
Response table for means for "larger is better" option

Level	*A*	*B*	*C*	*D*
1	138.75	123.75	125.375	94.125
2	81.50	106.50	102.000	133.250
3	127.50	114.00		
4	107.00	110.50		
Delta	57.25	17.25	23.375	39.125
Rank	1	4	3	2

In this situation, the predicted value of the response parameter BV (μ_{BV}) can be calculated as shown below,

$$T = \frac{\sum BV}{N} = \frac{1819}{16} = 113.69$$

Predicted value for BV is

$$\mu_{BV} = A1 + B1 + C1 + D2 - 3T$$

$$\mu_{BV} = 138.750 + 123.750 + 125.375 + 133.250 - 3 \times 113.69 = 180.06$$

The purpose of the ANOVA is to investigate which process parameters significantly affect the process responses. Table 5 shows the results of the ANOVA test for bed volumes (BV). In the ANOVA, the Fischer ratio (or *F*-test) is used to determine significant process parameters. Minimum or critical values for the Fischer ratio (*F*-critical values) can be found in most of the statistics and experimental design handbooks [30].

An *F*-ratio is calculated from the experimental results and then compared to the critical value. If the *F*-ratio calculated is bigger than the F_{cr}-critical value, it is an indication that the statistical test is significant at the confidence level selected. In this study, the total degree of freedom DOF is 15 (=16 − 1). The DOF for *A* and *B* parameters are 3 (=4 − 1) and for *C* and *D* parameters are 1 (=2 − 1). From this, the DOF for the error is calculated as 7. The F_{cr}-critical value of *A* and *B* parameters for DOF of 3 and 7 at a confidence level of 95% is 4.35. The F_{cr}-critical value of *C* and *D* parameters for DOF of 1 and 7 at a confidence level of 95% is 5.9.

Table 6 shows that *p*-ratio, which is another indicator to be used, and also the comparison of the calculated *F*-ratio to the F_{cr}-critical value both indicate that HTAB concentration, $g\,L^{-1}$ (*A*); zeolite bed height, cm (*D*) and Wastewater flowrate, $L\,min^{-1}$ (*C*) are significant parameters, respectively whereas, HTAB flowrate, $L\,min^{-1}$ (*B*) is an insignificant parameter in these experiments. Again, *P*-ratio, which is defined as a ratio of the parameters' sum of square to the total sum of square, indicates the contributions of these parameters. For example, *P* (%)-value for HTAB concentration, $g\,L^{-1}$ (*A*) given in Table 5 is calculated as follows,

$$P_A = SS_A \times \frac{100}{SS_T} = 7598.7 \times \frac{100}{19195.4} = 39.58$$

Confirmation testing is a necessary and important step in the Taguchi method. Therefore, a confirmation test must be carried out

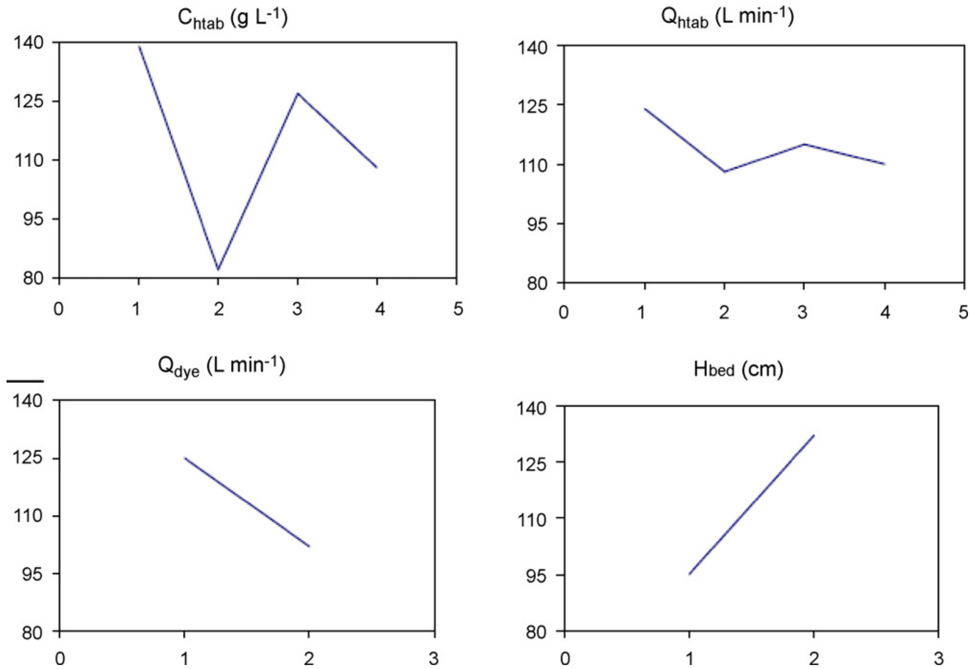

Fig. 2. The effect of each parameter on the optimization criteria of bed volumes (BV).

Table 5
Results of ANOVA for bed volumes (BV)

Source	DOF	SS	Adj SS	Variance	*F*-ratio	F_{cr}	*p*	*P* (%)
A	3	7598.7	7598.7	2532.9	6.73	4.35	0.018	39.58
B	3	652.7	652.7	217.6	0.58	4.35	0.648	3.40
C	1	2185.6	2185.6	2185.6	5.81	5.39	0.047	11.38
D	1	6123.1	6123.1	6123.1	16.26	5.39	0.005	31.90
Error	7	2635.4	2635.4	376.5				13.74
Total	15	19195.4						100.0

SS, sum of squares; DOF, degree of freedom; *p*, probability of *F* > *F*-ratio; *P*, percent contribution.

Table 6
Significant process parameters, their optimal levels and optimal predicted value for BV process response

Parameter	Optimal settings of process conditions		Significant process parameters
	Value	Level	
A, HTAB concentration ($g\,L^{-1}$)	1	1	*A*
B, HTAB flowrate ($L\,min^{-1}$)	0.015	1	
C, Wastewater flowrate ($L\,min^{-1}$)	0.025	1	*C*
D, Zeolite bed height (cm)	50	2	*D*
Predicted the BV process response	180.3		

at the end of the optimization study. A confirmation test was conducted for bed volumes (BV) without repetition at the optimum setting of the process parameters. The 95% confidence interval of the confirmation test (CI) was calculated by using Eq. (4) as follows,

$$\pm \mathrm{CI} = \sqrt{F_{\alpha}(1, f_e) \times v_e \times \left[\frac{1}{n_{\mathrm{eff}}} + \frac{1}{S}\right]} = \pm 57.33$$

So, predicted optimal range (for confirmation runs of one test) is obtained as;

$$180 - 57.33 < \mu < 180 + 57.33, \text{ which is;}$$

$$122.67 < \mu < 237.3$$

where, $f_e = 7$, $N = 16$, $F_{0.05}$: 1 and 7 (tabulated *F*-value) = 5.59, $S = 1$, $v_e = 376.5$, total DOF associated in the estimate of mean = 8, $n_{eff} = 1.78$.

The confirmation test indicates that 217 values for the bed volumes (BV) at the breakthrough point ($C/C_0 < 0.1$) is in acceptable limits. Hence, it can be concluded that the optimal levels of parameters are: *A*1 ($C_{htab} = 1\,g\,L^{-1}$), *B*1 ($Q_{htab} = 0.015\,L\,min^{-1}$), *C*1 ($Q_{dye} = 0.025\,L\,min^{-1}$), and *D*2 ($H_{bed} = 50\,cm$). Under these conditions, the volume of treated textile effluents reaches a maximum in the process of color removal from real textile wastewater.

4. Conclusions

Taguchi method is applied in color removal from textile dyebath effluents in a zeolite fixed bed reactor. The salient findings are summarized below.

The most important parameter affecting the color removal from real textile wastewater in the zeolite bed reactor is HTAB. The adsorption capacity of the HTAB modified zeolite bed shows a peak value by using HTAB concentration of 1 g L^{-1}l, but decreases with the increase of HTAB concentration from 1 g L^{-1} to 7.5 g L^{-1}. However, HTAB flowrate, L min^{-1} (*B*) is found to be an insignificant parameter. Color removal is influenced from the parameters in the following order: HTAB concentration (*A*) > zeolite bed height (*D*) > wastewater flowrate (*C*).

Confirmation test indicates that 217 values for the bed volumes (BV) at the breakthrough point ($C/C_0 < 0.1$) is in acceptable limits. Finally, the optimal levels of parameters are found as: *A*1 (C_{htab} = 1 L min^{-1}), *B*1 (Q_{htab} = 0.015 L min^{-1}), *C*1 (Q_{dye} = 0.025 L min^{-1}), and *D*2 (H_{bed} = 50 cm).

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