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Central composite design for the optimization of Basic Red V degradation in aqueous solution using Fenton reaction

A. Jaafar[a,*], A. Driouich[b], Z. Lakbaibi[b], H. Ben El Ayouchia[c], K. Azzaoui[d], A. Boussaoud[a], S. Jodeh[e,*]

[a]*Laboratory of Process, Signals, Industrial Systems and Computer Science, Superior School of Technology, Cadi Ayyad University, Dar Si-Aïssa road, 46000, BP 89, Safi, Morocco, Tel. +212-660-202-754; emails: jaafarfstgm@gmail.com (A. Jaafar), a.boussaoud@uca.ma (A. Boussaoud)*

[b]*Laboratory of chemical engineering and environment, Faculty of Sciences and Technology, Hassan II University, Mohammedia, Morocco, emails: anasdriouich23@gmail.com (A. Driouich), zouhairshaim@gmail.com (Z. Lakbaibi)*

[c]*Laboratory of Analytical and Molecular Chemistry, Polydisciplinary Faculty of Safi, Cadi Ayyad University, Morocco, email: belayou@gmail.com*

[d]*Laboratory of Solid Minerals and Analytical Chemistry, Faculty of Sciences, Mohamed Premier University, P.O. Box 717, 60000, Oujda, Morocco, email: k.azzaoui@yahoo.com*

[e]*Department of Chemistry, An-Najah National University, P.O. Box 7, Nablus, Palestinian, Tel. +970-599590-498; email: sjodeh@hotmail.com*

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ABSTRACT

Basic Red V (BR) is a stable dye used in different areas like textile industry. Fenton reaction is often used to decompose stable substances in wastewater. In this study, experimental design method (Central composite design) was used to investigate the effect of four factors on degradation of BR, that are pH, H_2O_2, Fe^{2+} and BR concentration. The response surface was determined by JMP and STATISTICA programs. The optimal reacting conditions were determined using central composite design, and it was found to be initial pH = 4.00, $[H_2O_2]$ = 54 mg L^{-1}, $[Fe^{2+}]$ = 27.50 mg L^{-1} for [BR] = 18 mg L^{-1} at ambient temperature. Under optimal conditions, 85% degradation efficiency of dye in aqueous solution was achieved after 30 min of reaction time.

Keywords: Fenton reagent; Degradation efficiency; Basic Red V; Hydrogen peroxide; Ferrous ion; Central composite design

1. Introduction

Water is one of the most essential requirements for all life on earth and is considered as very important resource for human civilization. Safe source for pure and affordable water is certainly one of the basic human goals and is identified as a major global challenge for the 21st century [1].

The release of coloured wastewater into the environment is a dramatic source of aesthetic pollution affecting human health [2]. Therefore, dyes released into the environment mainly in the form of wastewater effluents by textile, leather and printing industries cause severe ecological problems [3]. These compounds have a great variety of colours, chemical structures and are recalcitrant to microbial attack. In fact, decontamination of polluted water is become one of the most important environmental matter in the recent decade [4].

However, the application of some advanced oxidation processes such as Fenton process [5], fluidized bed solar photo Fenton [6], solar photocatalytic treatment using solar-powered photocatalytic reactors [7] and modified Fenton

* Corresponding authors.

process using zero valent iron (ZVI, Fe^0) as a catalyst [8] was an effectiveness method to treat water contaminated with organic pollutants.

Fenton reaction is a practical advanced oxidation process, used for treating wastewater containing a Basic Red V (BR) dye [9,10]. The Fenton process (Fe^{2+}/H_2O_2) Eq. (1) is a homogeneous catalytic oxidation process that uses a mixture of H_2O_2 and Fe^{2+}; the reaction between dissolved Fe^{2+} and H_2O_2 leads to the oxidation of Fe^{2+} to Fe^{3+} and the production of hydroxyl radicals ($^{\bullet}OH$) [11]. Producing hydroxyl radicals will degrade organic compounds into CO_2, H_2O and inorganic ions, or biodegradable compounds [12].

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + {}^{\bullet}OH + H_2O \qquad k_1 = 76\ M^{-1}s^{-1} \quad (1)$$

BR is an important colouring agent for its aqueous solution in deep red [13]. It is often used as linsey-woolsey colouring agent, biological stain and acid-base indicator. Therefore, BR is also an important composition in dyeing wastewater.

In this study, the response surface methodology based on central composite design was used to optimize the effect of BR, hydrogen peroxide concentration, ferrous ion concentration and initial pH as independent variables on the BR degradation [14]. The purpose is to optimize the process on the one hand, and the parameters on the other hand of the process. The traditional method remains limited because it does not consider all possible combinations. For that, a statistical design like response surface methodology was investigated. This statistical method consists of a group of mathematical and statistical techniques that are based on the fit of empirical models to the experimental data obtained in relation to experimental design [15,16].

2. Materials and methods

2.1. Reagents

BR of analytical grade was purchased from REACTIFS RAL, France. It has molecular formula $C_{15}H_{17}ClN_4$ (FW = 288.8 g mol^{-1}) with color index Number 50040. The structure of the BR is shown in Fig. 1.

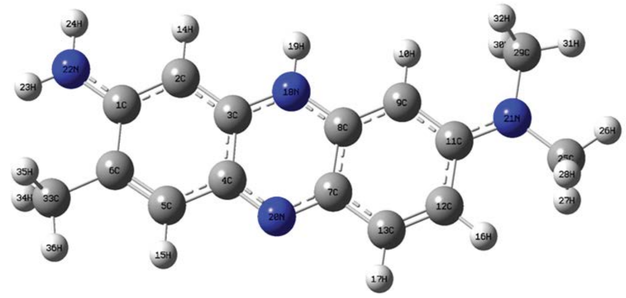

Fig. 1. Chemical structure of Basic Red V.

The chemicals used for AOP study are ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), hydrogen peroxide (30% V/V). The pH was adjusted with sodium hydroxide and sulfuric acid. All these chemicals are purchased by LobaChemie, India. All reagents were used without further purification. Distilled water was used throughout this study.

2.2. Experimental procedures

Decolorization of dye was carried out with a UV-vis spectrophotometer (Model Rayleigh UV-1800). Dye solution pH values were adjusted to the desired level using dilute sulfuric acid and sodium hydroxide, which were measured by a pH meter (Model Knick).

All experiments were carried out in 500 mL beakers and stirred by a magnetic stirrer at ambient temperature.

The effect of pH was studied by varying the pH from 2 to 6 while maintaining [BR], [Fe^{2+}] and [H_2O_2] constant at 45.00, 20.00, and 25.50 mg L^{-1}, respectively. The effect of [Fe^{2+}] was studied by varying [Fe^{2+}] from 2.50 to 52.50 mg L^{-1}, while maintaining [BR], [H_2O_2] and pH constant at 45.00, 25.50 mg L^{-1}, and 3, respectively. The effect of [H_2O_2] was studied by varying [H_2O_2] from 2.50 to 52.50 mg L^{-1}, while maintaining, [BR], [Fe^{2+}] and pH constant at 45.00, 20.00 mg L^{-1}, and 3 respectively. The effect of [BR] was studied by varying [BR] from 4.50 to 58.5 mg L^{-1}, while maintaining, [Fe^{2+}], [H_2O_2] and pH constant at 20.00, 25.50 mg L^{-1}, and 3 respectively.

Each experimental run was performed by taking an appropriate amount of stock dye solution followed by the addition of ferrous ion solution. The reactions were initiated by adding hydrogen peroxide to the beaker. Samples were taken out from the beaker using a pipette and were immediately analyzed after 30 min of reaction time.

2.3. Analytical methods

The UV-vis spectra of dye were recorded from 200 to 800 nm using a UV-vis spectrophotometer (Rayleigh UV-1800) with a spectrometric quartz cell (1 cm path length). The maximum absorbance wavelength (λ_{max}) of BR could be found at 530 nm from the spectra.

Therefore, the concentration of the dye in the reaction mixture at different reaction times was determined by measuring the absorption intensity at λ_{max} = 530 nm and from a calibration curve. The decolorization efficiency of BR was defined in Eq. (2):

$$\text{Degradation efficiency}\left(\%\right) = \left(1 - \frac{\left[BR\right]_t}{\left[BR\right]_0}\right) \times 100 \qquad (2)$$

where $[BR]_0$ is the initial concentration of BR and $[BR]_t$ is the concentration of BR at reaction time t.

2.4. Central composite design

Statistical analysis was performed using the JMP software [17,18]. Data was analysed by the analysis of variance (ANOVA), and *p*-value lower then 0.05 was considered significant in surface response analysis. The optimal values of the operation parameters were estimated by the three-dimensional (3D) response surface and the contour plots analysis of the independent variables (pH, [BR], [H_2O_2] and [Fe^{2+}]) and the dependent variable (Y%). Range and levels of independent variables are listed in Table 1.

Table 1
Optimization of parameters, experimental range and level of independent variables, on Fenton degradation of Basic Red V

Natural variables (X_j)	$-\alpha^*$	–1	0	1	$+\alpha^*$
[BR] (mg L^{-1})	4.50	18	31.50	45	58.50
[Fe^{2+}] (mg L^{-1})	2.50	15	27.50	40	52.50
[H_2O_2] (mg L^{-1})	2.50	15	27.50	40	52.50
pH	2	3	4	5	6

$*\alpha = 2.00,\ X_i = \dfrac{x_i - x_0}{\Delta x_i}$

In order to obtain a good descriptive quality of the model and to allow a reliable prediction in the whole of the experimental domain considered for a reasonable number of manipulations. The realization of the central composite design requires a number of tests equal to 31 (Eq. (3)):

$$2^k + 2k + N_0 = 16 + 8 + 7 = 31 \quad (3)$$

where k is the number of variables and N_0 is the number of center point replicates. Table 2 brings together the set of 31 tests, defining the matrix of experiments for the various factors considered. The i^{th} line of this matrix defines the experimental conditions of the i^{th} experiment [19].

All the operating conditions are randomized, and the results obtained after experimentation are grouped together in Table 2. The principle of the exploitation of the results is based on the analysis of the variance of the regression, the calculation of the estimates of the coefficients of the model, their significance in relation to the experimental error and the calculation of the residuals [19].

3. Results and discussion

3.1. Variance analysis

ANOVA allows us to see if the variables selected for the modeling have a significant effect on the response, the results of the analysis of the variance are grouped in Table 3.

One can notice that the ANOVA gave a rather high value of F (experimental Fisher factor), with a very low value of p-value. From these results it can be seen that there is no statistically significant difference between measurements [15].

The ANOVA table also shows one for the residual error, which measures the variation of the unaccompanied response data by the model. The shape of the model chosen to explain the relationship between the factors and the response model is correct [16].

Moreover, the polynomial model considered by the ANOVA for yield is significant and adequate to represent the relationship between the selected responses and the variables having a significant influence. The yield is represented by 95% of the responses are taken by the model.

3.2. Graphical study of the factors effect

The effects of the factors on the degradation efficiency are shown in Fig. 2. It was clear that the removal efficiency varies with BR concentration, Fe^{2+} concentration, H_2O_2 concentration and pH value.

Table 2
Design and experimental results

Randomized order	Actual order	[BR]	[Fe^{2+}]	[H_2O_2]	pH	Efficiency (%)
18	1	–1	–1	–1	–1	96
14	2	–1	–1	–1	1	16
22	3	–1	–1	1	–1	95
25	4	–1	–1	1	1	43
15	5	–1	1	–1	–1	92.3
28	6	–1	1	–1	1	24.1
5	7	–1	1	1	–1	98
24	8	–1	1	1	1	85
29	9	1	–1	–1	–1	78
19	10	1	–1	–1	1	17.16
9	11	1	–1	1	–1	72
12	12	1	–1	1	1	12.12
2	13	1	1	–1	–1	64
13	14	1	1	–1	1	9.3
8	15	1	1	1	–1	84.96
7	16	1	1	1	1	28.23
30	17	–2	0	0	0	86.17
11	18	2	0	0	0	42.51
26	19	0	–2	0	0	66.49
6	20	0	2	0	0	86.45
31	21	0	0	–2	0	45.2
4	22	0	0	2	0	88.27
20	23	0	0	0	–2	88
16	24	0	0	0	2	15.24
1	25	0	0	0	0	90.27
3	26	0	0	0	0	91.1
10	27	0	0	0	0	89.8
17	28	0	0	0	0	90.38
21	29	0	0	0	0	90.23
23	30	0	0	0	0	90.76
27	31	0	0	0	0	90.87

From the Fig. 2 we can say that Fe^{2+} concentration has no significant effect on the degradation efficiency; The BR concentration and the pH value have a negative effect on the response, which decreases with the increase of the two these factors. However, the concentration of the H_2O_2 has a positive effect on degradation efficiency.

3.3. Statistical study of factors effects

The main effects of the four variables studied and their interactions are shown in Table 4. Each coefficient is associated with the values of student "t" and p-value.

The relative importance of the main effects and their interactions was also observed on the Pareto chart as shown in Table 4. The values that exceed the reference line are considered significant values and those which do not are considered insignificant [20]. According to the Table 4, H_2O_2

Table 3
Variance analysis (ANOVA)

Source	Degree of freedom	Sum of squares	Mean square	F-ratio	Prob. > F
Model	14	27,102.981	1,935.93	20.3338	<0.0001*
Residual	16	1,523.316	95.21		
Total	30	28,626.297			
R squared	0.908739				
R adjusted squared	0.828886				
Root of the mean squared error	12.96279				
Average response	66.38968				
Weighted sums	31				

$F_{statistics}$: Experimental Fisher factor
*Significant to 0.1% ($F_{0.001}$ (14.16) = 5.27)

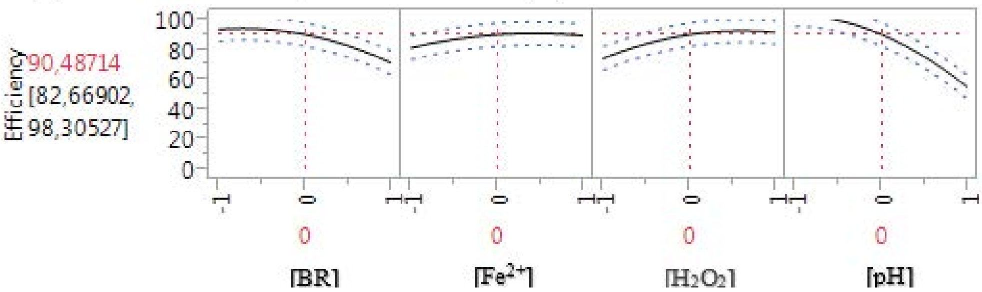

Fig. 2. Main effects plot of parameters on the BR degradation efficiency.

Table 4
Paretto diagram

Terme	Estimation	Erreur standard	t-ratio	t-ratio	Prob. > |t|
pH	–24.61958	1.99172	–12.36		<0.0001*
pH × pH	–10.98231	1.82467	–6.02		<0.0001*
[BR]	–11.28958	1.99172	–5.67		<0.0001*
$[H_2O_2]$	8.64958	1.991725	4.34		0.0005*
[BR] × [BR]	–7.80230	1.82467	–4.28		0.0006*
$[H_2O_2]$ × $[H_2O_2]$	–7.20355	1.82467	–3.95		0.0012*
$[Fe^{2+}]$ × $[Fe^{2+}]$	–4.76980	1.82467	–2.61		0.0188*
$[Fe^{2+}]$ × $[H_2O_2]$	5.7206	2.43935	2.35		0.0322*
$[H_2O_2]$ × pH	5.13312	2.43935	2.10		0.0515
$[Fe^{2+}]$	4.02208	1.99172	2.02		0.0605
[BR] × $[H_2O_2]$	–3.98437	2.43935	–1.63		0.1219
$[Fe^{2+}]$ × pH	3.75562	2.43935	1.54		0.1432
[BR] × $[Fe^{2+}]$	–2.63687	2.43935	–1.08		0.2957
[BR] × pH	–1.18437	2.43935	–0.49		0.6339

concentration is positively significant. The BR concentration, pH and pH-pH interaction are negatively significant. The quadratic interaction of BR concentration, H_2O_2 concentration, and the Fe^{2+} are lowly significant on the response.

3.4. Modelization

For the study of the improvement and the optimization of the process (the BR degradation), it is necessary to use a model polynomial of degree at least 2 [21].

Among the plans allowing the use of a polynomial model of the degree 2, before we chose a centered composite plane which makes it possible to establish the equation of the following model:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2$$

With:

- Y: The value of the calculated response.
- X_i: The value of the coded variable "i".
- b_i: The coefficient of the variable X_i model.
- b_{ii}: The coefficient of the model of the square variable X_i^2.
- b_{ij}: The coefficient of the interaction model between X_i and X_j.

This model has 15 terms:

- Constant term = 1
- Linear term = 4
- Square term = 4
- Rectangle term = 6

The mathematical model only takes into account factors having p-value < 0.05, according to the Table 5, these factors are: [BR], [H_2O_2] and pH, the interaction of [Fe^{2+}] × [H_2O_2], and the square interactions of [BR] × [BR], [H_2O_2] × [H_2O_2] and [pH] × [pH]

The Fisher test and the Student test are the basis of the statistical analysis. The Fisher test is used to determine the importance of each of the interactions between the variables, which may indicate the reasons for the interactions between the variables. Generally, the largest amplitude of F corresponds to the smallest p-value, the corresponding coefficient is therefore significant [22].

From Table 5 it can be noted that except for Fe^{2+} ions all factors have an influence on the response. For interactions between two factors, only the interaction between Fe^{2+} and H_2O_2 influences the degradation efficiency. Finally, we can say that all quadratic interactions have a significant effect on the response.

The mathematical model is written as follows:

Degradation efficiency % = 9,048,714 – 112,896 [BR] + 8,649,583 [H_2O_2] – 246,196 pH + 5,720,625 [Fe^{2+}] × [H_2O_2] –780,231 [BR] × [BR] –720,356 [H_2O_2] × [H_2O_2] – 109,823 [pH] × [pH]

3.5. Validation of model

The construction of this graph (Fig. 3) gives points clouds whose alignment is close to a straight line [23]. The condition of normality of the residues is thus well respected for the model. Indeed, in this model, the value

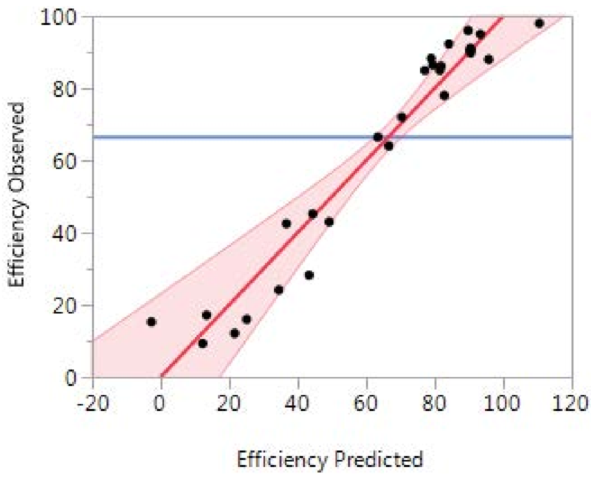

Fig. 3. Experimental values vs. predicted values (RMSE = 9,7574; R square = 0,95; P-value < 0001).

Table 5
Result of multiple linear regression of efficiency response

Model term	Estimation of coefficient	Sum of squares	F-ratio	Prob. > F	Significance
Constant	90.4871	–	–	–	***
[BR]	–11.2896	3,058.913	32.1290	<0.0001*	***
[Fe^{2+}]	4.0220	388.252	4.0780	0.0605	NS
[H_2O_2]	8.6495	1,795.567	18.8596	0.0005*	***
pH	–24.6196	14,546.973	152.7927	<0.0001*	***
[BR] × [Fe^{2+}]	–2.6368	111.250	1.1685	0.2957	NS
[BR] × [H_2O_2]	–3.9843	254.004	2.6679	0.1219	NS
[Fe^{2+}] × [H_2O_2]	5.7206	523.609	5.4997	0.0322*	*
[BR] × pH	-1.1843	22.444	0.2357	0.6339	NS
[Fe^{2+}] × pH	3.7556	225.676	2.3704	0.1432	NS
[H_2O_2] × pH	5.1331	421.584	4.4281	0.0515	NS
[BR] × [BR]	–7.8023	1,740.794	18.2843	0.0006*	***
[Fe^{2+}] × [Fe^{2+}]	–4.7698	650.583	6.8333	0.0188*	*
[H_2O_2] × [H_2O_2]	–7.2035	1,483.868	15.5857	0.0012*	**
pH × pH	–10.9823	3,448.963	36.2258	<0.0001*	***

***: significant to 0,1% ($F_{0.001}$ (1,16) = 16.12)
**: significant to 1% ($F_{0.01}$ (1,16) = 8.53)
*: significant to 5% ($F_{0.05}$ (1,16) = 4.49)
NS: not significant

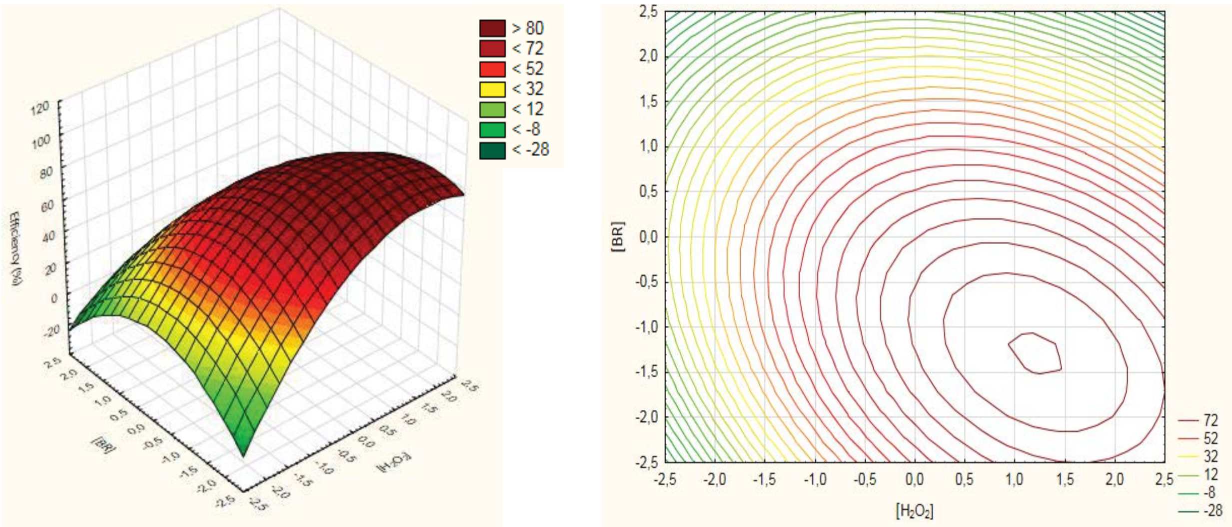

Fig. 4. Response surface and contour plot as a function of BR concentration and H_2O_2 concentration.

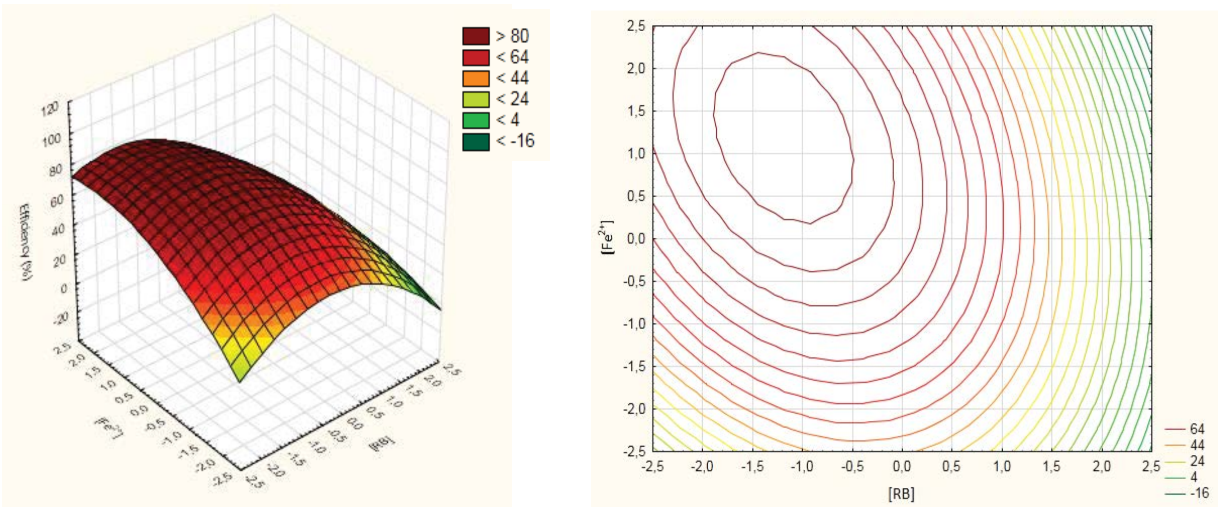

Fig. 5. Response surface and contour plot as a function of Fe^{2+} concentration and RB concentration.

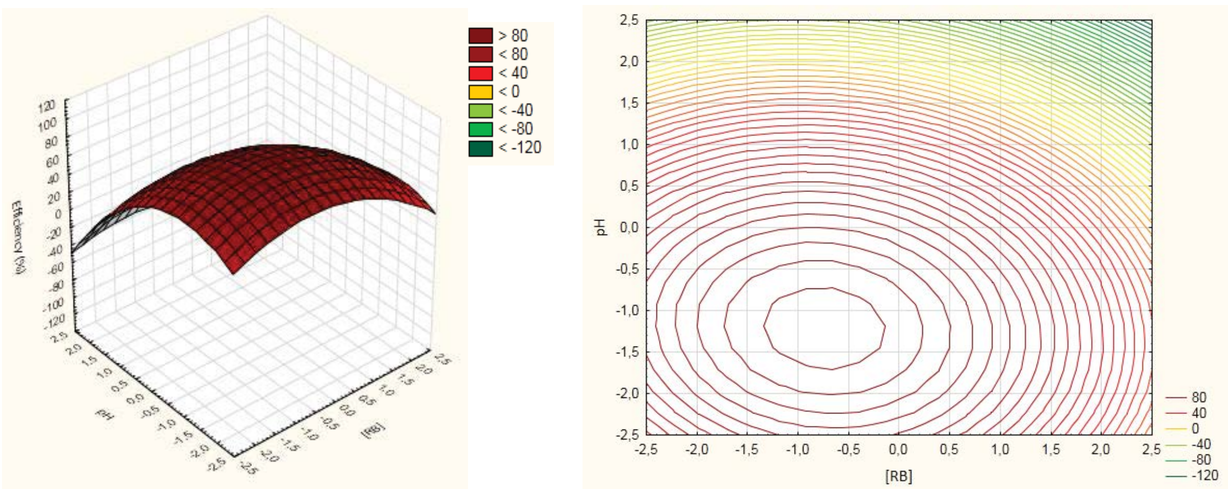

Fig. 6. Response surface and contour plot as a function of pH concentration and RB concentration.

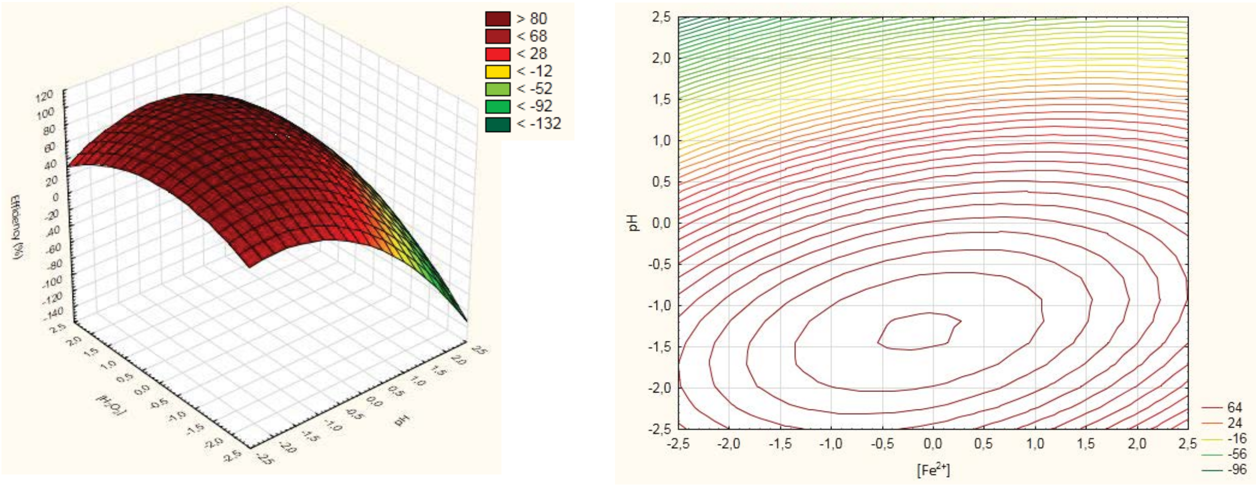

Fig. 7. Response surface and contour plot as a function of pH and Fe^{2+} concentration.

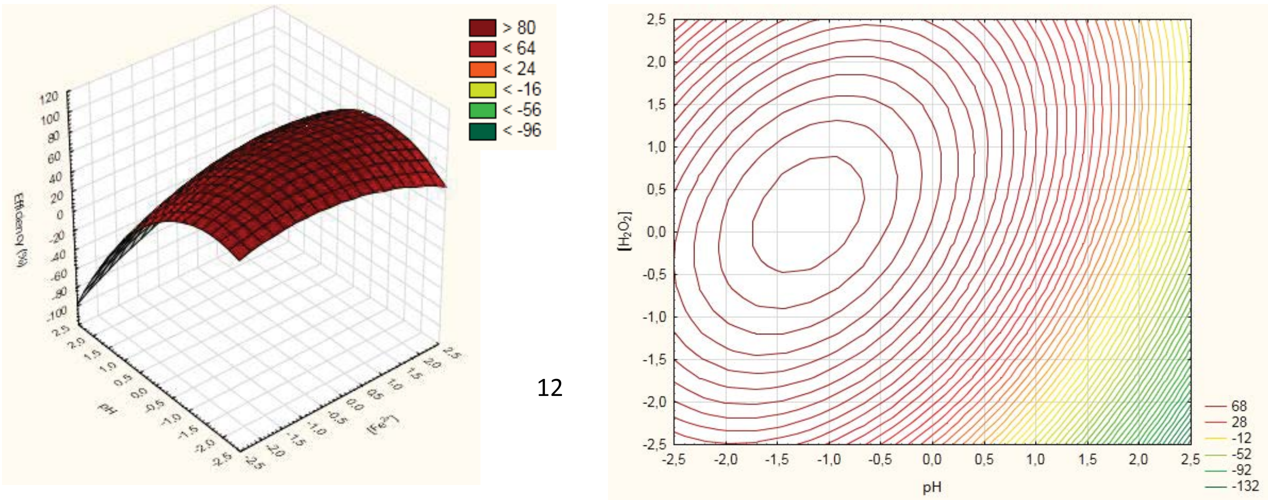

Fig. 8. Response surface and contour plot as a function of H_2O_2 concentration and pH.

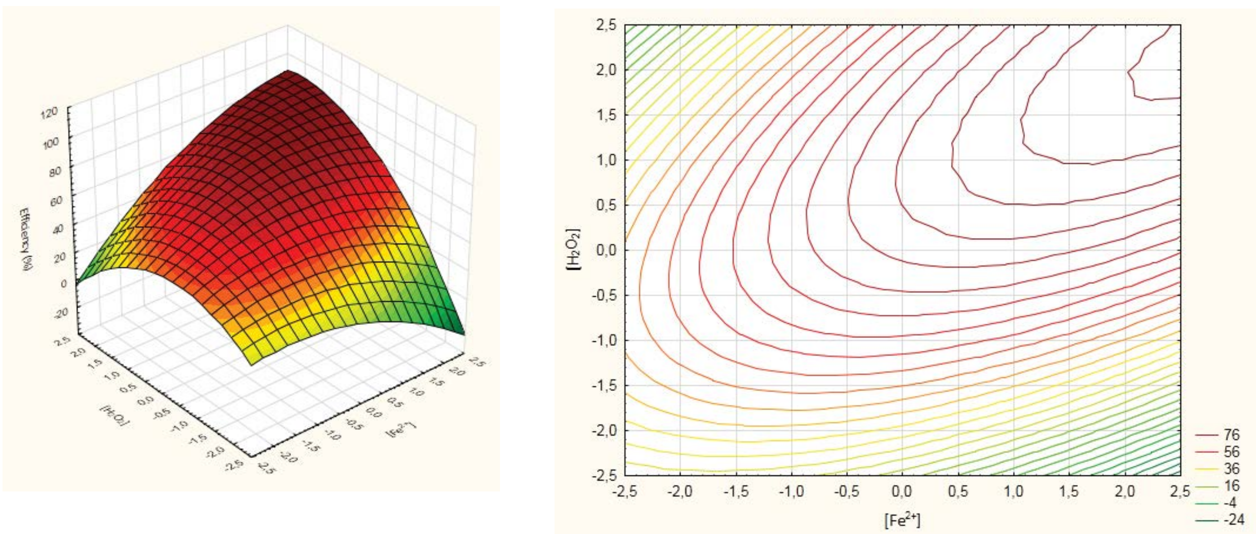

Fig. 9. Response surface and contour plot as a function of pH and Fe^{2+} concentration.

of R^2 was evaluated as 0.95 indicating that 95% of the variability in the response could be explained by the model. This indicated that the prediction of experimental data is satisfactory.

3.6. Optimization

The statistical software was used to produce 3D response surfaces and two-dimensional (2D) contour plots (Figs. 5–9). The 3D surfaces and 2D contour plots are graphical representations of the regression equation for the optimization of reaction conditions and are the most useful approach in revealing the conditions of the reaction system [19]. In such plots, the response functions of two factors are presented while all other factors are at the fixed levels. Hence, the optimum conditions for BR degradation are:

- The concentration of Fe^{2+} ions is 27.50 mg L^{-1}
- The pH is 4
- The concentration of BR is 18 mg L^{-1}
- The concentration of H_2O_2 is 54 mg L^{-1}

Under these conditions, the estimated value of efficiency degradation is 85%.

4. Conclusion

The Fenton process has proven to be an effective method for the treatment of aqueous solution contaminated with a BR dye. In order to optimize treatment efficiency, a central composite design has been used to model the treatment process, by controlling the most influential factors namely initial pH, the concentration of hydrogen peroxide, the concentration of ferrous ions and the concentration of BR. The optimal operation parameters for the Fenton oxidation of BR were 54 mg L^{-1} [H_2O_2], 27.50 mg L^{-1} [Fe^{2+}] for 18 mg L^{-1} [BR] at an initial pH of 4.00 with ambient temperature. Under these conditions, 85% degradation efficiency of BR dye in aqueous solution was achieved after 30 min of reaction time.

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