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Investigation of enhanced Fenton process (EFP) in color and COD removal of wastewater containing Acid Red 18 by response surface methodology: evaluation of EFP as post treatment

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ABSTRACT

The purpose of this study was to investigate the application of enhanced Fenton process (EFP) using zero-valent iron combined with ultrasonic irradiation for treatment of wastewater containing acid red 18 (AR18) by response surface methodology. According to the analysis of variance results, the model presents high R^2-value of 98.2 and 98.3% for the COD and AR18 dye removal, respectively, indicating that the model was successful. In this paper, the results of the monitoring of oxidation reduction potential and dissolved oxygen were presented to obtain the reaction time. At the 60 min of contact time, AR18 dye and COD removal efficiency raised more than 30 and 5% with the increase in Fenton reagents, respectively. Effluent without the presence of phosphate indicated 40% increment of AR18 dye removal in comparison to the influent with the presence of phosphate. Considering the EFP as post treatment of a sequencing batch reactor, the combination system improved the efficiency of high azo dye AR18 concentration wastewater treatment.

Keywords: Enhanced Fenton process; Response surface methodology (RSM); Acid Red 18 removal; COD removal

1. Introduction

Wastewaters from textile processes are characterized by high levels of color and chemical oxygen demand (COD) [1,2]. Several treatment methods including physical, chemical, and biological technologies with advantages and drawbacks have been widely investigated to remove dyes from the wastewater [3]. The Fenton process is one of the most popular advanced oxidation processes due to its ease of operation and high efficiency for the removal of dyes [2,4]. Fenton’s reagent is a mixture of hydrogen peroxide and ferrous ion, which generate hydroxyl radicals in acidic condition. The produced hydroxyl radicals can attack and destroy the structure of dyes [4,5].

It is noteworthy that the application of huge quantities of ferric salts as well as anions can be induced environmental problems in the treated

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wastewater with the increase in their concentration after the treatment in classical Fenton process [6]. Meanwhile, for obtaining of higher dye removal efficiency in the high concentration of dyeing wastewater, more ferrous ion and hydrogen peroxide were consumed in this process resulting in high reagent expenses and also generation of great amount of Fenton sludge [5]. On the other hand, the efficiency of Fenton reaction can be reduced in the presence of phosphate due to the forming of iron phosphate decreasing the amount of iron available for the reaction [7].

In view of these facts, usage of advanced techniques for the development of Fenton's reaction has been studied by other researchers [6,8]. Recently, the implementation of zero-valent iron (ZVI) was proposed from the environmental and efficient point of view [6,9]. On the other side, using ultrasonic irradiation could be also enhanced the corrosion of ZVI, releasing massive amounts of Fe^{2+} continuously and destroy a wide range of dyes [6]. In order to the deep mineralization of the dyeing wastewater, more studies have been also developed using the combination of Fenton process followed by biological systems for the removal of dye [5,7,10]. Therefore, the completely mineralization and COD removal especially for the treatment of wastewater containing azo dyes are still challenges faced by researchers in the recent years [11]. In the case of azo dye acid red 18 (AR18), the previous researches are limited to the studies accomplished using only classical Fenton processes or sequencing batch reactor (SBR) individually [11–14].

Accordingly, the objective of this article is to investigate enhanced Fenton process (EFP) for the decolorization of AR18 dye and COD removal. The effectiveness of the Fenton process was enhanced using micro ZVI along with ultrasonic irradiation. For this purpose, response surface methodology (RSM) is used to develop a mathematical correlation between two main factors of EFP (iron powder (ZVI) dose and hydrogen peroxide concentration) for the dye and COD removal. In the design and statistical evaluation of experiments, RSM can be offered for modeling and analysis of problems in which responses of interest are influenced by several variables, process optimization, and prediction of the interaction between process variables, reducing the numbers of experiments and thus the time and costs compared with results obtained from conventional process [15–17]. As far as we are concerned, no scientific work has been considered dealing with the investigation of EFP to treat azo dye AR18 wastewater along with nutrients especially phosphate using RSM so far. Furthermore, the appropriate reaction time obtained by monitoring of oxidation reduction potential (ORP) and dissolved oxygen (DO). Finally, the AR18 dye and COD removal efficiency were compared at the effluent of EFP in both of single and as post treatment of a SBR system under the lower amounts of two variables presented by RSM.

2. Materials and methods

The composition of synthetic azo dye wastewater was as follows: AR18 (500 mg/L), glucose (2,000 mg/L), Urea (214 mg/L), K_2HPO_4 (56 mg/L), KH_2PO_4 (44 mg/L), and $NaHCO_3$ (250 mg/L). The azo dye AR18 was provided from Alvan Sabet Company (Tehran, Iran). The general characteristics of AR18 are: molecular formula = $C_{20}H_{11}N_2Na_3O_{10}S_3$, molecular weight = 604.5 and $\lambda_{max} = 506$ nm. In Fenton process, the hydroxyl radical was generated by adding the hydrogen peroxide (H_2O_2, 30% w/v) and iron powder (extra pure, 99% purity, 10 μm). All the chemicals were obtained from Merck (analytical grade) and used without any further purification.

The experiments of EFP were carried out in a 400 mL batch reactor filled with 250 mL of the samples. Solution pH values were adjusted to the desired level (pH 3) using 1N H_2SO_4 or 1N NaOH. Subsequently, the appropriate amount of iron powder was added to the beaker. The solution was then irradiated with ultrasonic (SONOSWISS, SW 1H model) for 10 min. Finally the desired hydrogen peroxide concentration was added and the solution was continuously mixed at room temperature for 60 min at 200 rpm using a jar test equipment.

Before the analysis, the Fenton effluent samples were centrifuged at 3,000 rpm for 3 min. To eliminate the interference of hydrogen peroxide in COD analysis, the samples were adjusted to pH 10 and left overnight.

RSM is a statistical technique for designing experiments, building models, and evaluating the effects of several factors for desirable responses [15]. It defines the effect of independent variables, alone or in combination (the interaction between the parameters) with a minimum number of experiments in the process [15,18,19]. In this study, iron powder (ZVI) dose and hydrogen peroxide concentration for the AR18 dye and COD removal efficiency were studied with a standard RSM design (central composite design (CCD)) for EFP. CCD, which is the most widely used form of RSM, was employed to evaluate the effect of important process variables [16]. Five levels of iron dose and hydrogen peroxide concentration were investigated according to the obtained experimental data which are presented in Table 1. The independent variables are coded to the (−1, 1) interval where the low and high levels are coded as −1 and +1, respectively. The value of α for rotatability depends on the number of points in the factorial portion of the design, which is 1.414. Therefore, a total of 13 experiments according to a 2^2 full factorial CCD were conducted. The results of the experimental design were studied and analyzed by statistical software MINITAB 14. The statistical significance of the model was justified through analysis of variance (ANOVA) for polynomial model with 95% confidence level. The quality of the fit polynomial model was also expressed by the coefficient of determination, R^2.

The dye concentration was determined by measuring the absorption intensity at $\lambda_{\max} = 506$ nm using UV–vis spectrophotometer (DR 4000, HACH, USA). The concentration of phosphorus in solution was analyzed using inductively coupled plasma-optical emission spectroscopy (Agilent 700 series (Varian 735)). COD was carried out according to standard methods [20]. DO, pH, and ORP were also monitored regularly.

3. Results and discussion

3.1. Determination of EFP reaction time by monitoring of ORP and DO

In order to obtain optimum reaction time of EFP, two run of experiments including low (50 mg/L of iron dose and 300 mg/L of hydrogen peroxide concentration) and high (110 mg/L of iron dose and 700 mg/L of hydrogen peroxide concentration) levels of independent variables presented by RSM (listed in Table 1) were carried out at pH of 3. According to the literatures, pH of 3 is optimal for the Fenton reaction [21,22]. In this regard, the variations of ORP and DO values vs. reaction time were monitored for both of two run of experiments. As obvious in Fig. 1(a) and (b), the ORP values increased rapidly at the start of EFP. This is because hydroxyl radicals, which are strong oxidants, were more generated at the beginning of this process. Greater increases in ORP values also occurred for higher doses of hydrogen peroxide (Fig. 1(a)). This result is compatible with those obtained by Yu et al. [2]. Afterward, the ORP values reached slightly to around +582 mV and +562 mV demonstrated in Fig. 1(a) (high level of variables) and Fig. 1(b) (low level of variables), respectively. Then, these values dropped indicating the optimum reaction time of Fenton process in these times.

On the other hand, the increase in DO concentration can also assist in the controlling of the contact time for Fenton process. It should be noted that the increase in DO concentration may be due to the generation of oxygen in one of the reactions of Fenton process [23,24]. Therefore, the reaction time of around 15 and 90 min were chosen the optimum contact time for the AR18 dye removal in Fig. 1(a) and (b), respectively. These findings are also compatible with those presented in Fig. 1(c) and (d). As shown in Fig. 1(c) and (d), the dye removal efficiency increased rapidly at 15 and 90 min for two runs of high and low levels of experiments, respectively, and thereafter rate of removal was found to be slow and then constant. Regarding these results, monitoring of ORP and DO can help to control required reaction time for Fenton process which is in agreement with the results reported by Yu et al. [2]. Finally, the reaction time was selected 60 min for all of the experiments of EFP in this study.

3.2. Experimental results, regression model, and ANOVA

In order to study the effect of iron dose and hydrogen peroxide concentration, the experiments were done for different combinations of these parameters using statistically designed experiments in 60 min of the reaction time at pH of 3. The experimental results and the predicted values for the dye and COD removal efficiency are presented in Table 2.

Based on these results of RSM, an empirical relationship between the responses and independent variables were attained for AR18 and COD removal and expressed by the following second-order polynomial equations:

Table 1
Experimental ranges and levels of independent variables

			Range and level				
Variables	Factor	Unit	$-\alpha$	−1 (low)	0	1 (high)	α
Iron dose	X_1	mg/L	37.57	50	80	110	122.42
Hydrogen peroxide concentration	X_2	mg/L	217.15	300	500	700	782.84

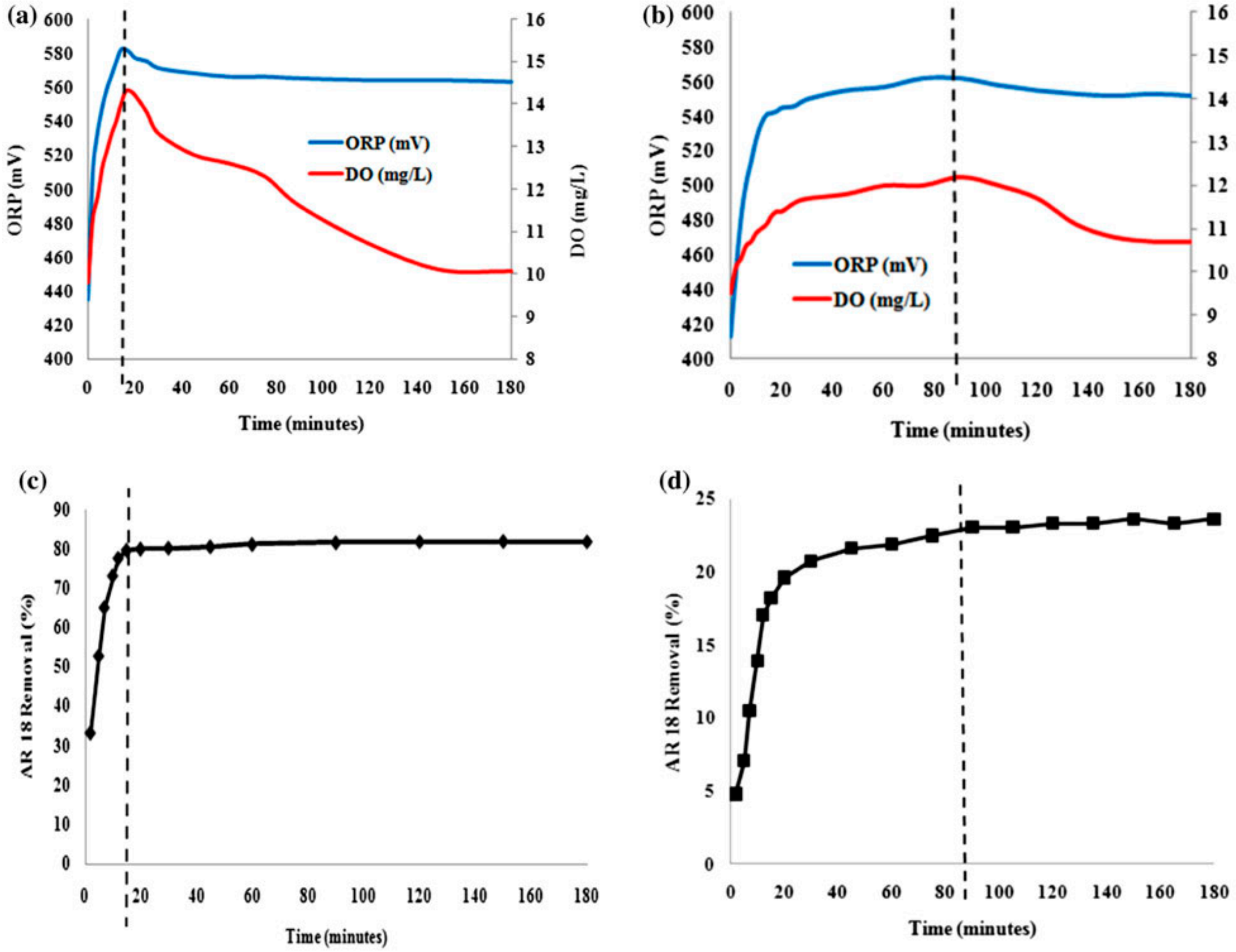

Fig. 1. ORP, DO, and AR18 dye removal profiles for (a,c) run of high (iron dose = 110 mg/L and hydrogen peroxide = 700 mg/L) and (b,d) run of low (iron dose = 50 mg/L and hydrogen peroxide = 300 mg/L) levels of variables.

$$\text{Dye removal}\,(\%) = 175.143 - 2.556x_1 - 0.372x_2 + 0.012x_1^2 + 0.000x_2^2 + 0.002x_1x_2 \tag{1}$$

$$\text{COD removal}\,(\%) = 59.655 - 0.152x_1 - 0.032x_2 + 0.0006x_1^2 + 0.000x_2^2 + 0.0002x_1x_2 \tag{2}$$

As shown in Table 3, the regression analysis of the experimental design demonstrated that the linear model terms, quadratic model terms, and interactive model term for two responses were significant ($p < 0.05$). According to the ANOVA results, the model presents high R^2-value of 98.2 and 98.3% for the COD and AR18 dye removal, respectively, which indicated that the accuracy of the polynomial model was good.

On the other hand, the adjusted R^2-value was high and close to the R^2-value that ensured a satisfactory adjustment of the polynomial model to the experimental data. Normal probability plot is a suitable graphical method for judging the normality of the residuals [18,19]. According to Fig. 2, the normality assumption was relatively satisfied as the points in the plot formed fairly straight line for the COD and dye removal.

These findings are in agreement with this fact showing that model is adequate to describe the COD and AR18 dye removal by RSM. As listed in Table 2, the predicted values obtained were also quite close to the experimental values for two responses, indicating that the model was also successful.

3.3. *Performance of sole EFP for the dye and COD removal*

As shown in Fig. 3, the addition of iron dose would improve the dye and COD removal in the presence of H_2O_2. This is because Fe^{2+} ions react with hydrogen peroxide generating more hydroxyl radicals

Table 2
Full factorial CCD matrix and results

Run order	Iron dose (mg/L) (x_1)	Hydrogen peroxide concentration (mg/L) (x_2)	Dye removal efficiency (%)		COD removal efficiency (%)	
			Experimental	Predicted	Experimental	Predicted
1	80	500	16.16	16.39	48.96	48.72
2	110	700	81.18	79.79	53.59	53.95
3	50	300	21.98	22.65	48.69	48.81
4	80	500	16.62	16.39	48.74	48.72
5	80	500	17.76	16.39	48.83	48.72
6	80	782.84	61.33	65.68	53.15	52.86
7	110	300	14.91	19.18	48.47	48.76
8	122.42	500	54.71	52.53	51.69	51.33
9	37.57	500	19.70	22.60	48.39	48.27
10	50	700	38.97	33.99	49.40	49.40
11	80	500	16.96	16.39	48.34	48.72
12	80	500	14.45	16.39	48.74	48.72
13	80	217.15	18.44	14.80	48.83	48.64

Table 3
Estimated regression coefficients of EFP

Term	Dye removal efficiency (%)			COD removal efficiency (%)		
	Coefficient	*t*-value	*p*-value	Coefficient	*t*-value	*p*-value
Constant	175.143	9.442	0.000	59.655	36.909	0.000
x_1	−2.556	−8.575	0.000	−0.152	−5.854	0.001
x_2	−0.372	−8.541	0.000	−0.032	−8.585	0.000
x_1^2	0.012	7.505	0.000	0.0006	4.401	0.003
x_2^2	0.000	8.455	0.000	0.000	8.259	0.000
x_1x_2	0.002	6.622	0.000	0.0002	6.804	0.000

which decolorize azo dye molecules [23,25]. However, increase in iron dose had no significant effect on COD removal. As clear in Fig. 3, AR18 dye and COD removal efficiency raised about 30 and 5% with the increase in iron powder dose, respectively. As illustrated in Fig. 3, the removal of dye and COD also increased about 40 and 5% with the increasing hydrogen peroxide concentration, respectively. This is due to the oxidation power of Fenton reaction which was improved with increasing hydroxyl radical amount attained from the decomposition of increasing hydrogen peroxide [25,26]. Regarding these findings, the removal of AR 18 dye in comparison with the COD removal had great difference in the Fenton process which is one important point to be considered. Therefore, the maximum dye and COD removal obtained about 81 and 53%, respectively, indicating higher removal efficiency of AR18 removal in comparison with COD removal. In agreement with this finding, less than 15% of the COD removal and around 80–90% of the azo dye Reactive Black B removal has been reported by Yu et al. [2]. Furthermore, Gökkuş and Oğuz reported that the dye removal (99%) was higher than the COD removal (58%) in aqueous solutions containing acid red 266 by Fenton process [27]. The difference of the dye and COD removal mechanisms by Fenton process presented fact that the dye removal was not synchronous with COD removal [28]. It is notable that the decolorization of azo dye is a fast process under the Fenton process but the degradation of aromatic amines forming by the cleaving of azo groups in this process is difficult. These results are compatible with those obtained by Sun et al. [25]. Therefore, COD can be removed only when the organic compounds (aromatic amines) are completely mineralized [28].

On the other hand, the presence of phosphate (placed in the wastewater as a nutrient) in the

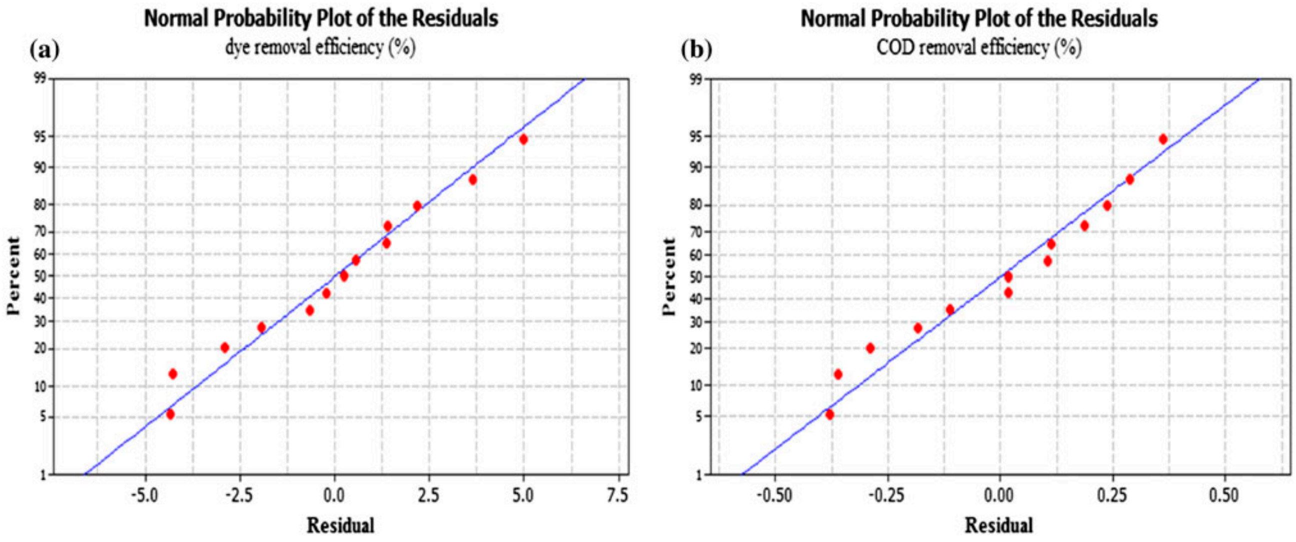

Fig. 2. Normal probability plot.

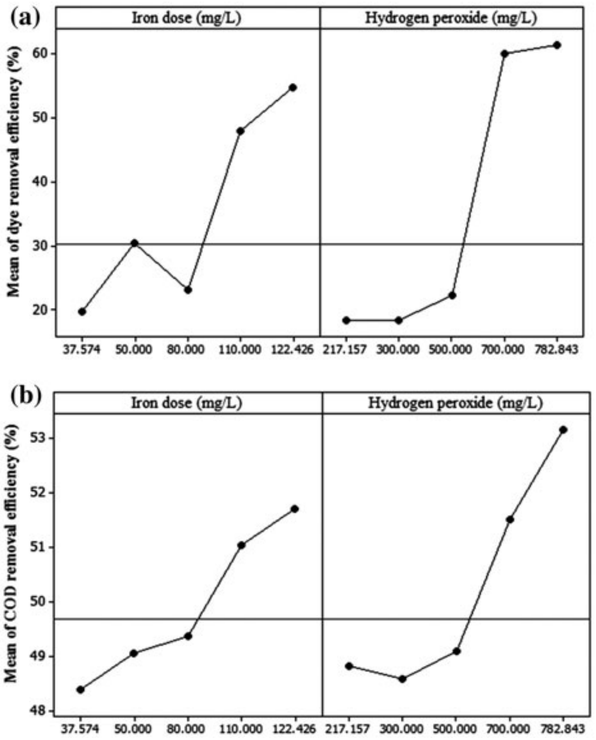

Fig. 3. Main effects of iron dose and hydrogen peroxide on (a) dye removal efficiency and (b) COD removal efficiency.

solution negatively affects the Fenton process by forming and precipitation of iron phosphate decreasing the amount of iron available for the reaction [7]. These results are also shown in the equation below:

$$Fe^0 + 2H^+ \longrightarrow Fe^{2+} + H_2 \tag{3}$$

$$Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + {}^{\cdot}OH + HO^- \tag{4}$$

$$Fe^{2+} + HO^- \longrightarrow Fe(OH)_2 \tag{5}$$

$$KH_2PO_4 \longrightarrow K^+ + H_2PO_4^- \tag{6}$$

$$H_2PO_4^- + H^+ \rightarrow H_3PO_4 \tag{7}$$

$$H_3PO_4 + Fe(OH)_2 \longrightarrow Fe_2(PO_4)_3 \tag{8}$$

Iron (II) phosphate is produced according to specific reactions and reduced the efficiency of Fenton process. Iron (II) is converted to iron (III) by reaction with hydrogen peroxide. The hydroxyl ion is generated too. The iron (II) ions also react with hydroxyl ions to produce $Fe\ (OH)_2$. Potassium dihydrogen phosphate (KH_2PO_4), as a main phosphate sources in the effluent of wastewater, completely dissociates into dihydrogen phosphate anions and potassium cations. Hydrogen ions and dihydrogen phosphate anions recombine to form phosphoric acid molecules. Iron (II) phosphate, $Fe_3\ (PO_4)_2$, can be formed by reacting one mole of phosphoric acid with one mole of Iron (II) hydroxide.

In order to investigate the effect of phosphate on Fenton reaction, the AR18 removal efficiency obtained under these conditions of 500 mg/L of hydrogen peroxide concentration, 80 mg/L of iron dose, reaction time of 60 min, pH of 3, and synthetic wastewater without nutrient (only 500 mg/L of AR18 dye concentration). The experimental result indicated that the

AR18 dye removal efficiency increased about 40% in comparison with the presence of phosphate in influent. In this study, it should be noted that the high percentage of decolorization (81%) presented in Table 2 also requires more consumption of iron and hydrogen peroxide resulting in more treatment expenses. According to obtaining high efficiency of Fenton reaction and reduction of Fenton reagents consumption, elimination of phosphate ion from wastewater could be considered using a pretreatment system and then applied Fenton process for wastewater containing AR18 removal. In agreement with these result, to effectively apply the photoFenton process, phosphate ion removal has been proposed by García-Montaño et al. for the treatment of wastewater containing Cibacron Red FN-R [29]. To investigate the above-mentioned problems, the performance of EFP was evaluated as post treatment of a SBR system (with high phosphorous removal efficiency) in high concentration of AR18 for decolorization and COD removal.

3.4. COD and the dye removal in EFP as post treatment of SBR system

In this part of experiments, EFP was carried out at the effluent of a SBR reactor which is operated for 60 d

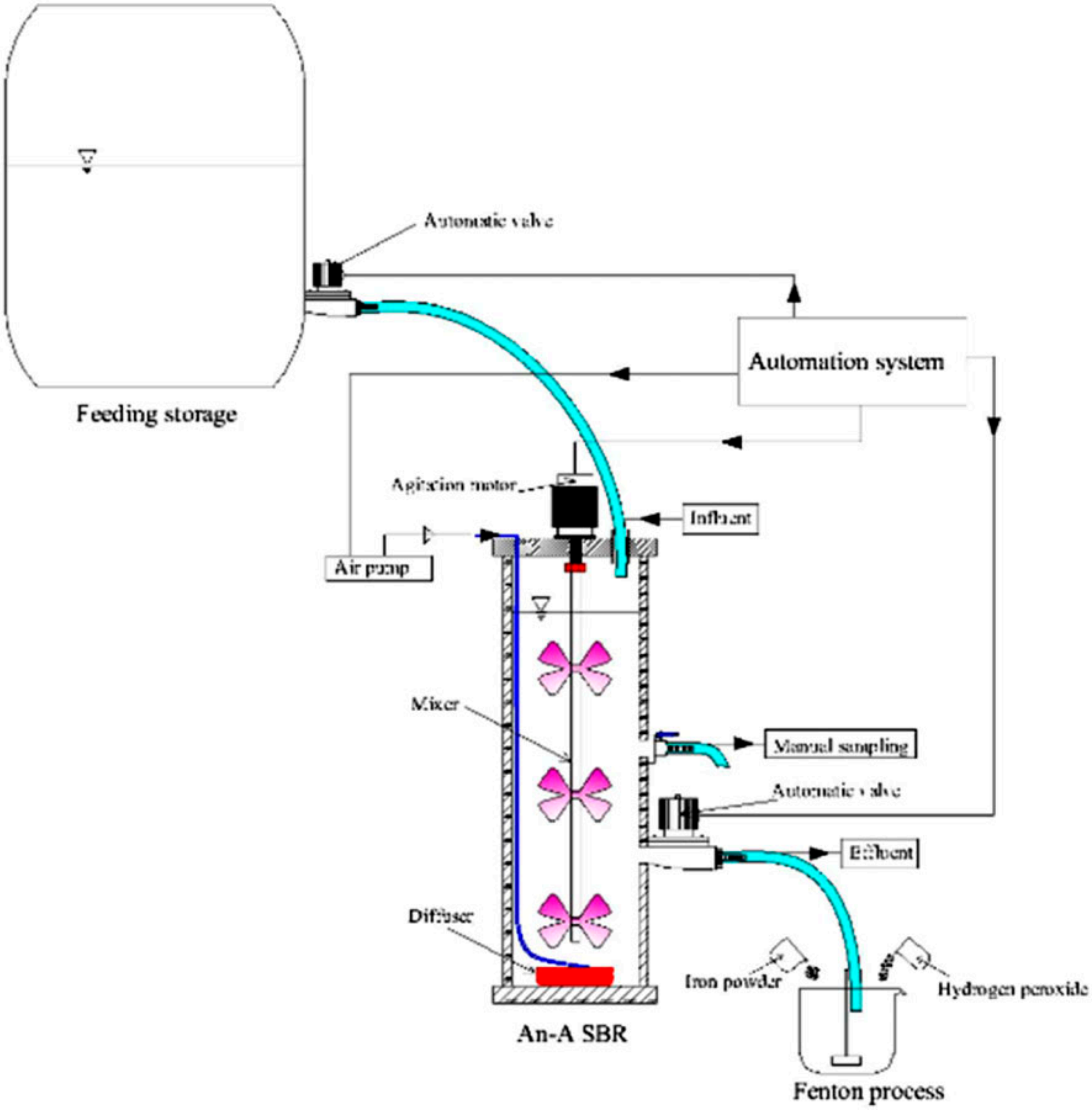

Fig. 4. Schematic of the combined treatment system (SBR + Fenton) used in this study.

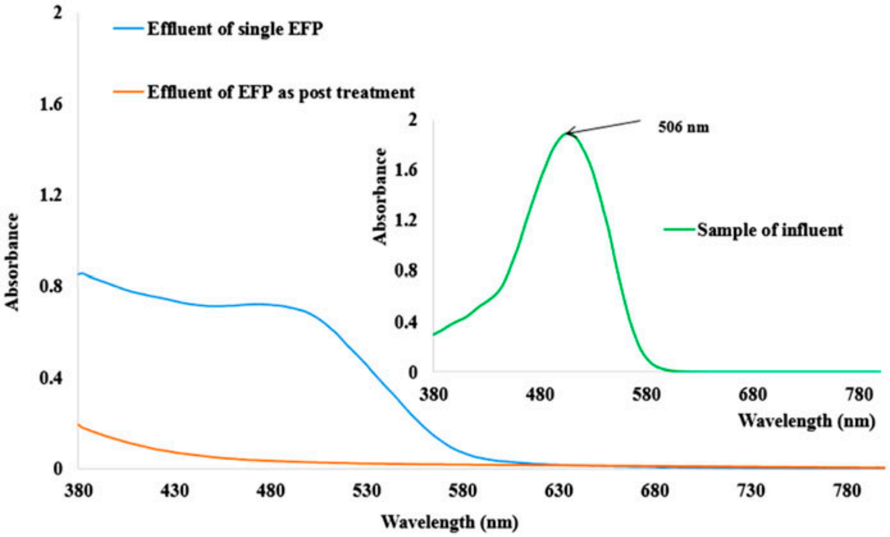

Fig. 5. Change of UV–vis spectrum (diluted 10 times).

in 24 h batch cycles with hydraulic retention time of 2 d and sludge retention time of 18 d in our laboratory. A 24 h operation cycle of reactor consisted of five phases including filling (10 min), reaction (16 h anaerobic and 7 h aerobic phases), settling (40 min), draw (5 min), and idle (15 min) which was controlled by an electrical automatic system. After the acclimation, the reactor was fed with 500 mg/L of azo dye AR18. The characteristics of the treated effluent of SBR were determined from the steady-state data obtained during the last 30 d of the operation period. More than 94% of decolorization of AR18 and 91% of COD removal occurred in SBR. The schematic diagram of the combined system (SBR + EFP) is illustrated in Fig. 4.

EFP was examined as single process and post treatment of the SBR effluent under these conditions: 217.15 mg/L (6.39 mMol) of hydrogen peroxide, 37.57 mg/L (0.67 mMol) of iron powder (lower amounts of two variables presented by RSM), pH of 3 and reaction time of 60 min. The COD and AR18 removal efficiency at the end of single EFP were more than 49 and 25% resulting in the concentration of COD and AR18 about 1,148 and 373 mg/L, respectively. However, EFP removed more than 86 and 64% of remained AR18 and COD concentration at the effluent of SBR, respectively. Indeed, the high dye concentration of 500 mg/L in the influent was reached to less than 5 mg/L of AR18 using Fenton process as post treatment indicating almost complete decolorization of AR18. This outcome is compatible with Fig. 5 of UV–vis analysis. The disappearance of the absorbance peak at 506 nm confirmed the complete decolorization and better mineralization of AR18 in the effluent of EFP as post treatment of SBR. This finding is compatible with those obtained by García-Montaño et al. that photo Fenton process has been considered as a chemical post-treatments of the anaerobic process for the complete dye byproducts mineralization [29]. Furthermore, the lower COD concentration of 98 mg/L obtained at the end of EFP as post treatment which is proved due to the higher removal of phosphorous (87%) in SBR. Considering the findings of this study, application of combined SBR and EFP can be suggested as an efficient system to enhance the efficiency of dye and COD removal in dye-containing wastewater.

4. Conclusions

In this study, EFP using ZVI along with ultrasonic irradiation was applied for the COD and acid red (AR18) dye removal using RSM as an experimental design. Based on RSM results, the predicted values obtained were quite close to the experimental values, indicating that the accuracy of the polynomial model was good. This finding is in agreement with the high R^2-value of 98.2 and 98.3% for the COD and AR18 dye removal, respectively. Monitoring of ORP and DO values controlled reaction time of Fenton process in this study and obtained 60 min of suitable time for reaction of EFP. In contrast with the significant increment of the dye removal efficiency (about 40%) with the increasing Fenton reagents dose, the COD removal efficiency (about 5%) had no remarkable variations in the 60 min of reaction time of EFP. According to investigation of phosphate effect on Fenton reaction, effluent without the presence of phosphate indicated 40% increment of AR18 dye removal in comparison to the influent with the presence of phosphate. Removal of more than 49% COD and 25% AR18 for treatment of 500 mg/L concentration wastewater was achieved using single EFP under the 6.39 mMol of hydrogen peroxide and 0.67 mMol of iron powder. On the other hand, the high dye concentration of 500 mg/L in the influent was reached to less than 5 mg/L of AR18 using EFP as post treatment indicating almost complete decolorization, while the concentration of AR18 obtained about 373 mg/L in the effluent by single EFP. Investigation of different biological systems as pretreatment of Fenton process, sludge management, and disposal as well as economic issues can be suggested as future studies.

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