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O_3-assisted UV-Fenton treatment of refining reverse osmosis water: optimization of process conditions by response surface methodology

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ABSTRACT

In this study, the O_3-assisted UV-Fenton process was used to treat the reverse osmosis concentrate of refining wastewater. Five factors were investigated in the single factor experiments, and the results showed that the chemical oxygen demand (COD) removal rate could reach 92.0% at the initial pH of 3, H_2O_2 concentration of 680 mg/L, Fe^{2+} dosage of 400 mg/L, O_3 consumption of 15 mg/L and ultraviolet (UV) fluence of 540 mJ/cm^2. Based on the results of single factor experiments, the first four factors (initial pH, H_2O_2 concentration, Fe^{2+} dosage and O_3 dosage) were selected as the independent variables and optimized by the response surface approach. According to the response surface experiment results, the quadratic model was established. Through the modeling and analysis of variance, the most significant interactive influence was observed between pH and O_3 dosage. The maximum COD removal of 93.2% in the verifying test was obtained under the optimum conditions such as the pH of 2.5, H_2O_2 dosage of 826 mg/L, Fe^{2+} dosage of 300 mg/L, ozone dosage of 17 mg/L and UV fluence of 540 mJ/cm^2.

Keywords: Advanced oxidation; Ozonation; Response surface methodology; Reverse osmosis concentrate; UV-Fenton

1. Introduction

Reverse osmosis (RO) membrane technology can be applied to the reclamation of the secondary sewage effluent. In the RO process, the concentrate water is generated as a by-product that contains high salinity and bio-refractory organic pollutants [1]. Industrial wastewater with high organic concentration usually was treated by physico-chemical methods in combination with the secondary biochemical process. Although the biodegradable organics could be removed in the biochemical phase, it was hard to dispose the RO concentrate water because of the high salinity and degradation-resistant characteristics.

Numerous studies have shown that it was difficult to achieve the ideal treatment effect by the traditional biological methods [2–4] and the chemical oxidation method was more suitable for dealing with RO concentrate water. Currently, the treatment techniques for the RO concentrate are focused on coagulation, adsorption, chemical oxidation, etc. [5–8]. In recent years, the advanced oxidation processes (AOPs) have become a hotspot for the organic wastewater treatment because the excited reactive free radicals especially the highly active hydroxyl radicals (·OH), could improve the wastewater biodegradability by oxidizing the organic pollutants to easily degradable intermediate products or the mineral compounds directly [9]. The AOPs included ozonation, Fenton technology, photocatalysis or photo-oxidation, electro-oxidation and sonolysis. Pophali et al. [10] used ozone to purify the tannery wastewater, and in this way the sewage biodegradability could be improved and the organic matter could be removed simultaneously. Westerhoff et al. [11] adopted O_3/H_2O_2 to treat the RO concentrate

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and achieved a 75.0% removal of DOC, but high dosages of hydrogen peroxide (0.7 mol H_2O_2/mol O_3) and ozone (1000 mg/L) were necessary. Hurwitz et al. [12] employed the ultraviolet chemical method (UVC) and the electro-chemical method to treat the RO concentrate, and the removal rates of DOC after 5 hours of operation only reached 25.0 and 35.0%, respectively. The UVC in conjunction with the electro-chemical method could remove more than 80.0% of DOC. The UVC/H_2O_2 also was used to treat the RO concentrate [13]. As the Fenton reaction could produce hydroxyl radicals, many researchers combined the Fenton process with other AOPs. Cortez et al. [14] treated the mature landfill leachate with the Fenton process and other AOPs, the biodegradability of the wastewater was improved. The oxidation effects of Fenton and photo-Fenton processes in the paint industrial wastewater treatment were studied by Oliveira et al. [15] and the results showed that the photo-Fenton process exhibited better COD and TOC removal efficiencies than the Fenton process. James et al. [16] used the electro-Fenton process to treat thin film transistor-liquid crystal display (TFT-LCD) wastewater, and the COD removal of 79.0% could be reached under the optimal conditions (pH 1.95, Fe^{2+} loading of 5.125 mM and H_2O_2 loading of 325 mM). Zhou et al. [17] investigated photocatalytic oxidation, sonolysis, ozonation, H_2O_2 oxidation and their combinations for removing organics from the RO concentrate and finally achieved 95.0% removal of the organics using an integrated method of $FeCl_3$ coagulation plus photocatalysis.

Although good results were obtained from these methods, a variety of combined methods were better than the single treatment method for the treatment effects. But if several methods were used at the same time, the influences of every factor and the interaction between factors on the experiment needed to be concerned. In this study, the UV-Fenton process was combined with ozonation for the treatment of refining reverse osmosis water. Single factor experiments and response surface experiments were carried out in order to investigate the influencing factors in the treatment process. The corresponding model was established to ensure the optimal conditions and to achieve a desired treatment effect.

2. Materials and methods

The wastewater samples were obtained from the RO concentrated drainage of a petrochemical wastewater treatment plant located in Shandong, China. Characteristics of the wastewater were presented in Table 1.

Table 1
Characteristics of the wastewater sample

Parameters	Unit	Value
pH		8–9
COD	mg/L	308–320
Chromaticity		45
Ammonia–nitrogen	mg/L	28
DO	mg/L	6.07
Conductivity	µS/cm	5.37
TDS	mg/L	2512

The COD, chromaticity and ammonia-nitrogen were measured by the AHPA [18] standard method. The pH was measured by a pH meter (phs-3C, INESA). Dissolved oxygen (DO) was measured by a dissolved oxygen meter (HQ-30D, LDO101 probe, Hach). Conductivity and total dissolved solids (TDS) were measured by conductivity and TDS integrated meter (DDBJ-350, INESA).

The experimental apparatus mainly consisted of a cylindrical reactor (ID: 60 mm; H: 600 mm) (Fig. 1), which had a UV-LED light (Cnlight corporation, China) with an output wavelength of 255 nm and the power of 40W. A litre of wastewater was treated in each experiment and all experiments were performed at the room temperature of 25.0°C. Single factor experiments were repeated for three times.

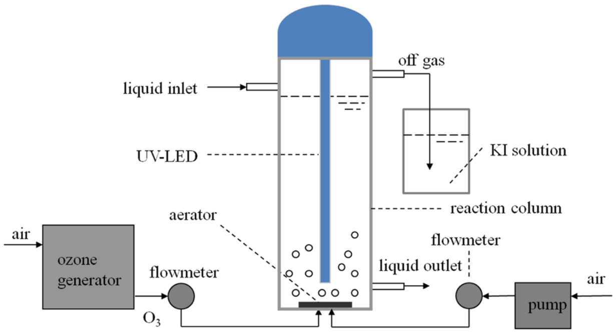

Fig. 1. Diagram of the experimental setup.

Ozone was produced by an ozone generator (sw-002, Qingdao VISTER) with air as oxygen source. The ozone and air mixture were pumped into the reactor through a titanium aerator (micropore diameter: 0.22–100 µm) and the mixture gas flow rate was 0.5–1.0 L/min. Unconsumed ozone was absorbed by KI solution for the measure of residual ozone. The input ozone concentration and residual ozone was measured by iodometric method [19] and the input ozone concentration was about 30 mg/L. The ozone dosage was calculated according to the following equation:

$$O_{3\ \text{dosage}} = \frac{c_{\text{ozone}} \cdot Q \cdot t - O_{3\ \text{residual}}}{V} \tag{1}$$

where c_{ozone}, Q, t, $O_{3\ \text{residual}}$ and V are input ozone concentration (mg/L), mixture gas flow rate (L/s), ozone aeration time (s) and volume of RO concentrate sample (L), respectively.

Catalytic oxidation was carried out in the following steps. The pH of wastewater in a 2 L glass beaker was adjusted to the set value by the addition of sulphuric acid (H_2SO_4, 2 M) and sodium hydroxide (NaOH, 1 M). Then the solution was poured into the reactor and $FeSO_4{\cdot}7H_2O$ was added. The solution was slowly mixed with H_2O_2 when the $FeSO_4{\cdot}7H_2O$ was dissolved entirely and started timing. Finally, aeration was carried out on the solution under the UV-LED light and aeration rate was 100 L/h.

The Fenton reaction was considered finished after 2 h of operation and stopped by adjusting the pH value to 10 using the sodium hydroxide (NaOH, 1 M). Reaction mixture was filtrated using a 0.45 µm filter and the supernatant was recovered for the measurement of COD and ammonia-nitrogen. But only the COD removal as the discussion object because of the removal efficiency of ammonia-nitrogen (0–5%) was not significant.

3. Results and discussion

In a series of experiments, different parameters were varied to achieve a better removal rate of COD. The optimal conditions for RO concentrate water treatment were obtained by single factor tests and response surface methodology. The significant operating variables were chosen as initial pH, H_2O_2 dosage, Fe^{2+} dosage, ozone dosage and UV fluence.

3.1. Single factor experiments

3.1.1. Effect of the initial pH value

In the series of single factor experiments, the initial pH value of the sample was varied in the range of 2–6 given that H_2O_2 is unstable and loses its oxidizing potential in alkaline solutions [20]. The values of other factors were set as follows. H_2O_2 dosage was 680 mg/L, Fe^{2+} dosage was 600 mg/L, UV fluence was 540 mJ/cm^2 and ozone concentration was 10 mg/L. The impacts of pH on the COD removal are presented in Fig. 2a. The figure shows that the COD removal increased with the pH increasing when the pH value was less than 3, the maximum COD removal of 83.0% was achieved at pH 3, and then the removal rate decreased gradually. Based on the principle of UV-Fenton oxidation reaction [21], H_2O_2 produces the hydroxyl radical resulted from the catalysis of Fe^{2+} in the acidic environment. But if the pH value is less than 2, the peroxide will be converted to $H_3O_2^+$ because of the high concentration of H^+ (Eq. (2)) and Fe^{2+} will be transformed into $[Fe(H_2O)]^{2+}$ which would react more slowly with H_2O_2 [22]. The reaction of hydrogen peroxide and ferrous ions was hindered in both cases. On the other hand, the hydroxyl radical will be reduced with the increase of the pH because of the deactivation of Fe^{2+} and the formation of Fe^{3+} [23]. So the proper pH value was optimized to be 3.

$$H_2O_2 + H^+ \rightarrow H_3O_2^+ \tag{2}$$

3.1.2. Effect of H_2O_2 dosage

The H_2O_2 dosage is one of the most important factors for effective Fenton treatment. In this set of tests, the H_2O_2 was dosed in the range of 340–1700 mg/L, the pH was adjusted to 3, Fe^{2+} dosage was 600 mg/L, ozone concentration was 10 mg/L and UV fluence was 540 mJ/cm^2. The effect of H_2O_2 on the COD removal is presented in Fig. 2b. It shows that the COD removal rate increased at the beginning and then decreased with the increase of H_2O_2 concentration, and the maximum COD removal was 84.4 % at the H_2O_2 dosage of 680 mg/L. Hydrogen peroxide reacted with the ferrous ions to produce a mass of hydroxyl radicals and ferric ions (Eq. (3)) at the startup of operation. When the dosage of hydrogen peroxide was more than the optimum dosage, it reacted with ferric ions to produce hydroperoxyl radicals (·OOH) and ferrous ions (Eq. (4)). Likewise, the excessive hydrogen peroxide would consume hydroxyl radicals (Eq. (5)). Due to the stronger oxidation capability of ·OH than ·OOH, the COD removal rate declined even more H_2O_2 was dosed than the optimum dosage. Huseh et al. [20] also found the same phenomenon for using Fenton and Fenton-like reagent for disposing azo dyes. The reactions involved during Fenton oxidation were represented as the following three equations [24].

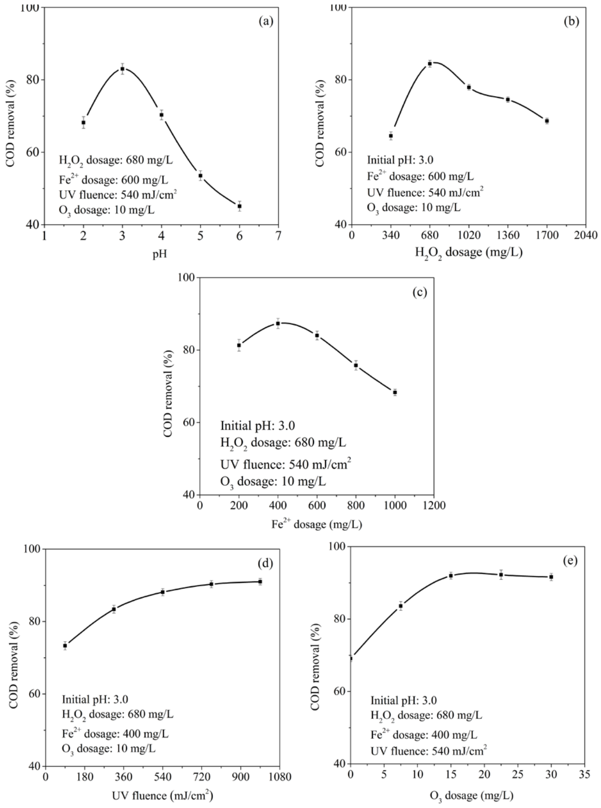

Fig. 2. Effects of (a) initial pH, (b) hydrogen peroxide dosage, (c) ferrous dosage, (d) UV fluence and (e) ozone dosage on the COD reduction for RO concentrate water.

$$H_2O_2 + Fe^{2+} \rightarrow \cdot OH + OH^- + Fe^{3+} \tag{3}$$

$$H_2O_2 + Fe^{3+} \rightarrow H^+ + Fe^{2+} + \cdot OOH \tag{4}$$

$$H_2O_2 + \cdot OH \rightarrow \cdot OOH + H_2O \tag{5}$$

3.1.3. Effect of Fe^{2+} dosage

The dosage of ferrous ions was varied in the range of 200–1000 mg/L. The pH and H_2O_2 dosage were maintained at the respective observed optimum (3 and 680 mg/L), ozone concentration was 10 mg/L and UV fluence was 540 mJ/cm^2. Fig. 2c depicts the effect of the amount of Fe^{2+} on the COD removal. The results shows that the COD removal efficiency increased with increasing Fe^{2+} dosage until a concentration of 400 mg/L and the maximum COD removal efficiency was 87.4%. In the Fenton reaction, there is an appropriate ratio between the dosages of ferrous ions and hydrogen peroxide [25]. Different sources of wastewater have different ratios and the appropriate mass ratio is about 1:1.7 (Fe^{2+}:H_2O_2) in the series of single factor experiments. A continual increase of COD removal was observed with the increment of ferrous dosage because of chemical oxidation and coagulation effects. When the ferrous dosage exceeded the matching ratio, the degradation efficiency of pollutants would reduce because of the reaction between Fe^{2+} and ·OH (Eq. (6)) [26].

$$Fe^{2+} + \cdot OH \rightarrow Fe^{3+} + OH^- \tag{6}$$

3.1.4. Effect of UV fluence

As the UV fluence could facilitate the Fenton oxidation reaction, the UV fluence was varied in the range of 90 to 990 mJ/cm^2 in the experiments at the initial pH of 3, H_2O_2 dosage of 680 mg/L, Fe^{2+} dosage of 400 mg/L and ozone concentration of 10 mg/L. Fig. 2d shows the variation of COD removal rate with the change of UV fluence. The COD of reverse osmosis water decreased gradually over time and the removal rate reached 88.1% at 540 mJ/cm^2. After that, the rate of descent became very slowly. In theory, the greater the UV fluence, the better the degradation efficiency of refractory organics as illustrated in the Eqs. (7) and (8) [26]. In the meantime, ozone would decompose into oxygen atoms under the UV light and oxygen atoms would react with water to produce hydroxyl radicals [27].

$$H_2O_2 + [UV] \rightarrow 2 \cdot OH \tag{7}$$

$$RH + \cdot OH \rightarrow R + H_2O \tag{8}$$

For industrial applications, the processing cost is one of the main considerations. As the COD removal rate obtained at the 990 mJ/cm^2 of operation was only slightly higher than that at 540 mJ/cm^2 of operation, from an economic perspective, 540 mJ/cm^2 of UV fluence was an appropriate choice. Therefore, this factor was not chosen as an optimization variable in the response surface method.

3.1.5. Effect of O_3 dosage

In the treatment process, the ozone dosage affected the COD removal efficiency. Fig. 2e depicts the effect of the ozone dosage on the removal efficiency for COD with the initial pH of 3, H_2O_2 dosage of 680 mg/L, Fe^{2+} dosage of 400 mg/L and UV fluence of 540 mJ/cm^2. The scope of the ozone dosage in this set of experiments varied from 0 to 25 mg/L. The COD removal was 69.1% without the use of ozone, while it increased with the increase of ozone dosage and to a peak of 92.0% at the ozone concentration of 15 mg/L, and then kept nearly constant with further ozone dosed. It was because that UV could enhance the activity of organic molecules and made it easier to break down by the ozone. Another reason was that the dissolved ozone could react with radical initiators to provide hydroperoxyl radicals for organic degradation [28]. These two effects made the rapid degradation of COD when the ozone dosage was less than 15 mg/L. When the ozone dosage went above the threshold, it was difficult to degrade the rest of the organic matter and the removal rate tended to be stable.

3.2. Statistical analysis and optimization

Single factor experiments can only examine the influence of a single variable while other factors are of constant values and it was unable to understand the interaction among various factors. Therefore, on the basis of single factor experiments, central composite design (CCD) combined with response surface methodology (RSM) was applied for experimental design and process optimization. Scopes of variables were shown in Table 2.

According to the CCD, a total of 30 experiments are performed and the results were given in Table 3.

Based on the results of experiments, the regression analysis was carried out using the Design Expert 8.0 Software. Fitting of the data to various model (linear, 2 factorial, quadratic and cubic) and model fit summary showed that the COD removal rate was most suitable with a second order polynomial model. So the regression equation (based on the coded values) for the removal rate of COD was given as:

$$\begin{aligned}\%\text{COD removal} &= +91.03 + 2.72A + 0.36B - 2.25C \\ &+2.07D - 1.41AB + 0.18AC - 1.02AD \\ &+0.93BC + 1.45BD - 2.21CD - 2.13A^2 \\ &-0.33B^2 - 1.46C^2 - 2.96D^2\end{aligned} \tag{9}$$

where A, B, C and D are H_2O_2 dosage (mg/L), Fe^{2+} dosage (mg/L), pH and O_3 dosage (mg/L), respectively.

The variance analysis results of the quadratic polynomial model are shown in Table 4.

According to the results of ANOVA, the model F-value of 26.01 implies that the model is significant. Based on the F-value, the most significant impact factor is H_2O_2 dosage for the COD removal, and then followed by pH, O_3 dosage and Fe^{2+} dosage. The lack of fit F-value (2.94) indicates that the lack of fit was not significant relative to the pure error. In general, the P-value less than 0.05 means that a factor is significant. In this case, the model terms of A, C, D, AB, AD, BC, BD, CD, A^2, C^2 and D^2 are significant. The H_2O_2 dosage,

Table 2
The level and scope of variables

Factor	Levels				
	–2	–1	0	1	2
pH	2	2.5	3	3.5	4
H_2O_2 dosage (mg/L)	340	510	680	850	1020
Fe^{2+} dosage (mg/L)	200	300	400	500	600
O_3 dosage (mg/L)	5	10	15	20	25

Table 3
Summary of optimization results

Run	pH	H_2O_2 dosage (mg/L)	Fe^{2+} dosage (mg/L)	O_3 dosage (mg/L)	COD removal rate (%)
1	3.50	850	300	20.00	83.00
2	3.00	680	400	5.00	76.00
3	3.00	680	400	15.00	91.00
4	4.00	680	400	15.00	80.50
5	3.00	680	400	15.00	92.50
6	3.00	680	600	15.00	89.03
7	3.50	510	500	20.00	86.61
8	3.00	680	400	15.00	91.23
9	2.50	850	500	10.00	80.95
10	2.50	510	300	20.00	88.20
11	3.00	680	400	15.00	91.20
12	3.50	510	300	20.00	75.31
13	2.50	850	500	20.00	83.61
14	3.00	1020	400	15.00	88.90
15	2.00	680	400	15.00	91.00
16	2.50	850	300	10.00	86.66
17	2.50	850	500	20.00	94.00
18	1.50	510	500	20.00	90.59
19	2.00	680	400	25.00	82.00
20	2.50	850	500	10.00	85.10
21	2.00	680	400	15.00	89.51
22	2.00	680	200	15.00	90.00
23	2.00	680	400	15.00	89.21
24	1.50	510	500	10.00	79.27
25	1.00	340	400	15.00	75.73
26	1.50	510	300	10.00	76.58
27	2.50	850	300	10.00	88.54
28	1.50	510	300	10.00	78.82
29	2.50	850	300	20.00	91.00
30	1.50	510	500	10.00	79.01

Fe^{2+} dosage, pH and O_3 dosage interactively affected the removal of COD. In order to show the interaction between the four parameters more clearly, the response surfaces are plotted and shown in Fig. 3. The figure illustrates the effect of the interaction of two factors on the COD removal while other two factors were kept at constant values. Other than the circle contour line (Fig. 3b), the contour lines between any two factors are oval in shape (Fig. 3a, 3c–f). This phenomenon means that the interactions between any other two factors are significant except the interaction between H_2O_2 and pH. This is in agreement with the results of ANOVA. The coefficients of determination R^2 is 0.9604 and R_{adj}^2 is 0.9235, indicating that the experimental results fitted well with the model. The model could be used to predict

Table 4
Analysis of variance (ANOVA)

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	925.63	14	66.12	26.01	<0.0001
A	177.13	1	177.13	69.67	<0.0001
B	3.14	1	3.14	1.23	0.2840
C	121.14	1	121.14	47.65	<0.0001
D	103.33	1	103.33	40.65	<0.0001
AB	31.70	1	31.70	12.47	0.0030
AC	0.50	1	0.50	0.20	0.6625
AD	16.52	1	16.52	6.50	0.0222
BC	13.95	1	13.95	5.49	0.0334
BD	33.52	1	33.52	13.19	0.0025
CD	78.50	1	78.50	30.88	<0.0001
A2	124.85	1	124.85	49.11	<0.0001
B2	3.03	1	3.03	1.19	0.2923
C2	58.45	1	58.45	22.99	0.0002
D2	240.49	1	240.49	94.60	<0.0001
Residual	38.13	15	2.54		
Lack of fit	32.59	10	3.26	2.94	0.1230
Pure error	5.55	5	1.11		
Corrected total	963.77	29			

Note: R^2 =0.9604 R_{adj}^2 =0.9235

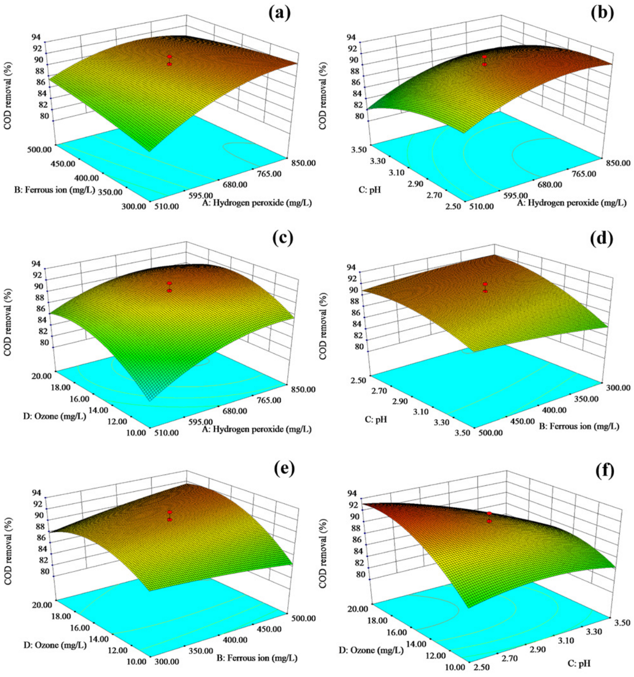

Fig. 3. Response surface and contour plot for COD removal.

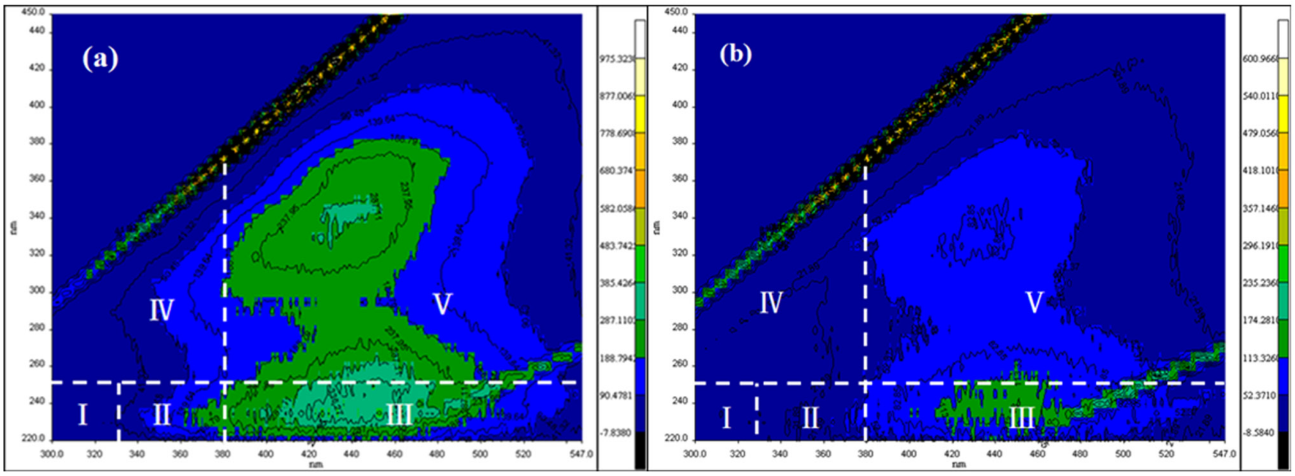

Fig. 4. EEM spectra of (a) untreated RO concentrate, (b) the effluent under the optimal conditions.

and optimize the reaction process. The maximum COD removal of prediction is 94.2% when the pH of 2.5, H_2O_2 dosage of 826 mg/L, Fe^{2+} dosage of 300 mg/L, ozone dosage of 16.69 mg/L and UV fluence of 540 mJ/cm^2. And the maximum COD removal of 93.2% in the verifying test was obtained under the optimum conditions such as the pH of 2.5, H_2O_2 dosage of 826 mg/L, Fe^{2+} dosage of 300 mg/L, ozone dosage of 17 mg/L and UV fluence of 540 mJ/cm^2.

3.3. Fluorescence EEM spectra

Fluorescence excitation-emission matrix (EEM) spectra can be used to detect the organic species in wastewater [29]. We used the fluorescence EEM to characterize dissolved organic matter (DOM) in the RO concentrate before and after the treatments. Chen et al. [30] delineated EEMs into five regions: I (aromatic protein I); II (aromatic protein II); III (fulvic acid-like); IV (soluble microbial products); V (humic acid-like). The EEM spectra were shown in Fig. 4.

Y. Qi et al. [31] and Estrada-Arriaga et al. [32] have used GC-MS to analyze COD components of petrochemical wastewater. GC–MS analysis demonstrated that the identified organic compounds consisted of hydrocarbons, benzene, phenol, ketone, nitriles and ester derivates, etc. These substance fluorescence peak are between region IV and V (λ_{ex} = 200–350 nm; λ_{em} = 300–400 nm), this result was not quite consistent with our result. That was probably because the RO concentrate water was drainage of a petrochemical wastewater treatment plant which has been pre-treated by biological method. These substances were consumed by microbes in biodegradation process and produced humic-like substances then affected the site of fluorescence peaks.

Fig. 4a indicated that the RO concentrate water contained immense amounts of fluorescent dissolved organic matter (FDOM), especially in regions III (fulvic acid-like) and V (humic acid-like). After the treatment, the FDOM in the effluent had a significant decrease in the fluorescence (Fig. 4b), showing the advanced oxidation process could make the breakdown of the conjugated bonds and aromatic rings of the humic-like substances [6]. The fluorescence EEM spectra matched up with the COD removal rate

4. Conclusion

The results of the single factor experiments showed that the COD of RO concentrate water could be removed through the UV-Fenton process in conjunction with ozone oxidation. The COD removal efficiency could reach a maximum of 92.0% with the initial pH of 3, H_2O_2 dosage of 680 mg/L, Fe^{2+} dosage of 400 mg/L, UV fluence of 540 mJ/ cm^2 and ozone dosage of 15 mg/L.

A quadratic model about the COD removal rate and the four factors were defined. And the optimal conditions were predicted to be the initial pH of 2.5, H_2O_2 dosage of 826 mg/L, Fe^{2+} dosage of 300 mg/L, ozone dosage of 16.69 mg/L and UV fluence of 540 mJ/cm^2. Under these optimized conditions, the COD removal efficiency in the verifying test (93.2%) was very close to the result of prediction (94.2%).

Acknowledgments

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