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Mineralization of AV dye in water by solar Fenton-like process

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ABSTRACT

In this study, treatment of alizarin violet 3B (AV) dye in water by a artificial solar irradiation assisted Fenton-like process was investigated. Optimized reaction conditions based on response surface methodology (RSM) were established under a $[H_2O_2]_0$/COD ratio of 1.75, a $[H_2O_2]_0/[Fe^{3+}]_0$ molar ratio of 15 and a reaction time of 15 min in which the COD and TOC degradation efficiency of AV would be more than 90%. As compared to conventional Fenton-like process, the solar Fenton-like process with an irradiation of 500 W/m^2 increased the mineralization reaction rate by a factor of 6.9, and the mineralization efficiency was increased from 65% to 92% as the temperature of water was raised from 15 to 35 °C due to solar heat irradiation. Accordingly, solar energy consisting of light energy and heat irradiation showed a hybrid promoting effect on the Fenton-like reaction.

Keywords: Mineralization; Artificial solar irradiation; Fenton-like process; RSM; Alizarin violet 3B; TOC

1. Introduction

Wastewaters from textile and dye industries are generally highly colored with considerable amount of auxiliary chemicals. Direct discharge of textile industry wastewater into the receiving media causes serious environmental pollution by imparting intensive color and toxicity to aquatic environment [1]. Over 100,000 different types of dyes are commercially available and 700,000 tons are produced yearly all over the world. Nearly 50% of these dyes are azo-type dyes [2]. Due to the complex aromatic structure and stability of the azo-dyes, conventional biological treatments are ineffective for degradation and mineralization of dye molecules [3]. A promising alternative for treatment of dye wastewater is Fenton-related process. Fenton-like process usually uses H_2O_2 and ferric salts to generate hydroxyl radicals. Nevertheless, Fenton's reaction cannot completely mineralize organic pollutants since normally less than 50% of the organic carbon of solution can be converted to CO_2 [4]. When a Fenton process uses ultraviolet radiation, visible light or a combination of both, it is known as the photo-Fenton process. The photo-Fenton process has several advantages, mainly the increase of degradation rate and less sludge generation [5]. Basically, the photo-Fenton-like process starts with the reduction of Fe^{3+} through the following reactions. When an irradiation ($\lambda < 450$ nm) is involved, the added Fe^{3+} or Fe^{3+} generated by Fenton's reaction (Eq. (1)) is continuously reduced to Fe^{2+} as described by Eqs. (2) and (3) [6]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^- \tag{1}$$

$$Fe^{3+} + H_2O \rightarrow Fe(OH)^{2+} + H^+ \tag{2}$$

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$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + {}^{\bullet}OH \qquad (3)$$

Hydroxyl radicals can be generated through the use of solar energy as well. Although the photo-Fenton-like process using natural solar energy as an irradiation source has attracted great interest in recent years [6], the effect of solar photo-enhancement involving light energy and heat irradiation on the degradation of dye wastewater are still not clear nowadays. Furthermore, parameters including initial dosage of Fenton-like reagents ($[H_2O_2]_0$ and $[Fe^{3+}]_0$) and reaction time were also played an important role. To understand intensively the effect of different combination of parameters, response surface methodology (RSM) provides elaborative vision over various combinations of parameters. RSM is essentially a particular set of mathematical and statistical methods for designing experiments, building models, evaluating the effects of variables, and searching optimum conditions of variables to predict targeted responses [8].

In this study, RSM was employed to assess the individual and interactive effect of critical process parameters ($[H_2O_2]_0$/COD, $[H_2O_2]_0/[Fe^{3+}]_0$ and reaction time) on treatment performance in terms of COD and TOC degradation efficiency. Moreover, the effect of solar irradiation on Fenton-like oxidation of alizarin violet 3B (AV) in water was investigated.

2. Materials and methods

2.1. Materials

AV ($C_{21}H_{14}NO_6SNa$) with a purity of 68% were purchased from the Chroma Co. (Japan) and used without further purification. The chemical properties of AV are shown in Table 1. As shown in Table 1, the peaks of 253, 276 and 567 nm are the characteristic and relatively higher absorbance peaks of the UV–VIS absorption spectrum for AV dye solution. An initial AV concentration of 100 mg/L was prepared for all experimental runs. Hydrogen peroxide (H_2O_2) with a concentration of 30% was supplied by Panreac Co., EU. Analytical-grade $Fe_2(SO_4)_3$ (Panreac Co., EU) with a purity of 75 % was used as the source of ferric ion. All other chemicals used in this study were analytical grade and used as received.

2.2. Procedures

All experiments were carried out in a batch mode. A 1-L glass beaker containing 200 mL of AV solution was used and maintained at a preset temperature in a water bath during the experiments. The initial pH of the solution was adjusted to pH 3.0 ± 0.1 using 0.1 M H_2SO_4. A 3 factors * 3 levels RSM experimental design with three replicates at center point as shown in Table 2 was applied. The prepared dye solution was placed into the photoreactor and irradiated by an intensity of 500

Table 1
Chemical characteristics of alizarin violet 3B

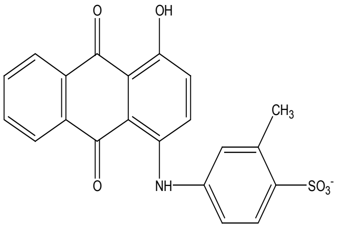
Dye	Chemical structure	Relative λ_{max} (nm)	MW, g/mol	Category
Alizarin violet 3B ($C_{21}H_{14}NO_6SNa$) (C.I. No: 60730)		253, 276, 567	431.4	Anthraquinone

Table 2
Experimental range and levels of the process independent variables in this study

Independent variable	Factor	Range and level		
		–1	0	+1
g H_2O_2/g COD	X_1	1.0	1.5	2.0
$[H_2O_2]_0/[Fe^{3+}]_0$	X_2	10	30	50
Reaction time, min	X_3	5	10	15

W/m^2 artificial solar light provided by an ATLAS Suntest CPS+ solar simulator (ATLAS Co., USA) with a spectral distribution resembling the solar spectrum (300–800 nm). During the experiments, the pH of the solution was monitored using a pH meter (SP-701LI 120, Suntex Co., Taiwan) equipped with a glass electrode. Samples were withdrawn from the reactor at preset time intervals (5, 10, 15 min), measured instantly the residual H_2O_2 concentration according to the method reported by Seller [9] which used the reagent – potassium titania oxalate ($K_2TiO(C_2O_4)_2{\cdot}2H_2O$) reacting with H_2O_2 to form Ti^{4+} yellow complex solution and then measured the absorbance of solution at 400 nm with a UV–VIS spectrophotometer (U-2001, Hitachi Co., Japan). In addition, iron as Fe^{2+} ion determined by Fe(II)/1,10-phenanthroline complex at 510 nm [10] using a Hitachi (Japan) U-2001 spectrophotometer, and then quenched with proper amount of sodium hydrogen sulfite to avoid further reactions and interference of COD analysis. Basically, a 0.8 mM of $NaHSO_3$ was added per mM of residual H_2O_2 in the solution according to a calibration curve developed by our lab. The remaining samples were then stored at 4 °C for the following COD, TOC, and IC analysis. In addition, two blank experiments including photolysis reaction and the AV oxidation with H_2O_2 only under the experimental conditions of this study were performed. The result indicated that the mineralization efficiency of AV solution was less than 1% and 2%, respectively, which implied that the mineralization of AV solution due to other effects was slight in this study.

2.3. Analysis

2.3.1. Chemical oxygen demand (COD) and total organic carbon (TOC) measurement

COD of solution via a DR 4000 photometer (HACH Co., USA) by using a $K_2Cr_2O_7$ as the reacting reagent was measured in order to understand the changes on degree of oxidation for AV wastewater. TOC of solution was measured by using a Shimadzu VCPH analyzer (Shimadzu Co., Japan) in order to know the amount of AV molecules degraded to CO_2 during oxidation.

2.3.2. Ion chromatography (IC) analysis

To quantify the degradation product – oxalate which may affect the formation of Fe^{2+}, an ion-chromatographic system (Dionex ICS-1000) equipped with an IonPac AS12A column (*L*: 200 mm, ID: 4.0 mm) was used. Aqueous solutions of 2.7 mM Na_2CO_3 and 0.3 mM $NaHCO_3$ were used as eluents. Analysis were performed under a flow rate of 1.5 mL/min and operated on an isocratic mode. Under the operation conditions, the retention time of oxalate was 11.0 ± 0.1 min.

3. Results and discussion

3.1. Effect of $[H_2O_2]_0$/COD and $[H_2O_2]_0/[Fe^{3+}]_0$ on AV dye degradation efficiency

Fig. 1 displays the 2D contour plots for percent COD and TOC removal efficiencies as a function of $[H_2O_2]_0$/COD and $[H_2O_2]_0/[Fe^{3+}]_0$. It was found that under a $[H_2O_2]_0/[Fe^{3+}]_0$ molar ratio of about 30, at least a 59% of COD removal and a 38% of TOC removal were obtained. Furthermore, the COD or TOC removal efficiencies were improved significantly as increasing the dosage of H_2O_2. For example, increasing the applied $[H_2O_2]_0$/COD from 1 to 2, significantly enhanced the COD and TOC removal

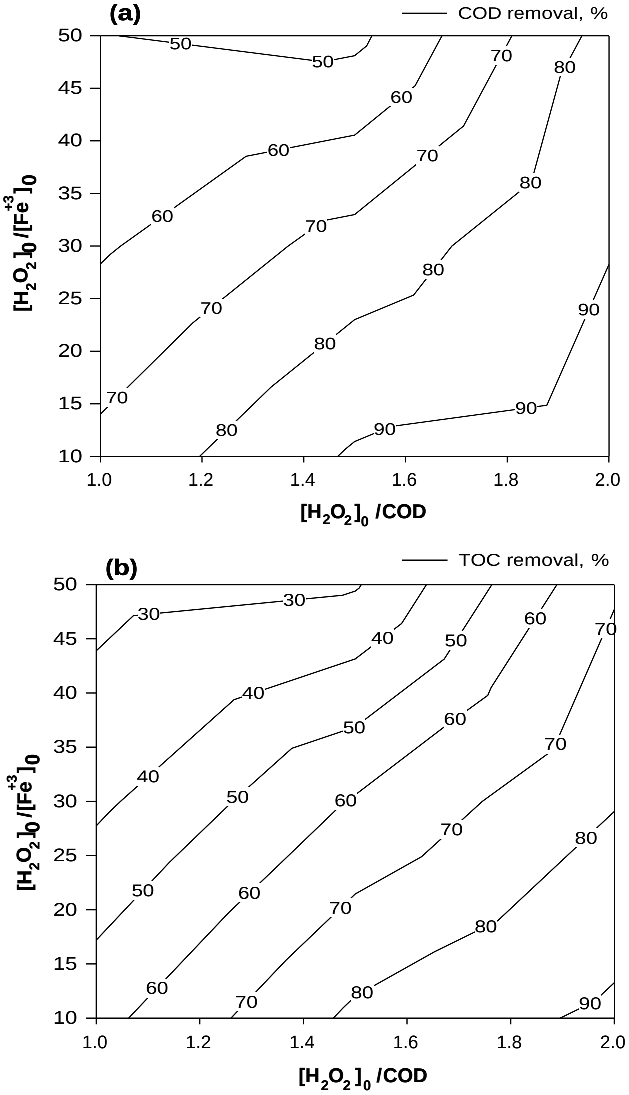


Fig. 1. Contour plots for (a) COD and (b) TOC removal efficiency of AV dye in water as a function of $[H_2O_2]_0$/COD and $[H_2O_2]_0/[Fe^{3+}]_0$. (Reaction time: 15 min, light intensity: 500 W/m^2, water temp.: 25 °C.)

efficiencies to 95% and 91%, respectively but with a H_2O_2/Fe^{3+} molar relation of 10. This is possibly due to the fact that an increase in H_2O_2 concentration could produce a higher amount of ·OH responsible for AV destruction. However, an excess of hydrogen peroxide may reduce he amount of radicals available for destroying dye and producing the scavenger effect as described by Eq. (4) [11,12], and then decrease dye degradation efficiency.

$$\cdot OH + H_2O_2 \rightarrow \cdot OOH + H_2O \qquad (4)$$

Moreover, as shown in Fig. 2, increasing the $[H_2O_2]_0$/$[Fe^{3+}]_0$ ratio showed a negative effect on the removal of COD and TOC at a fixed $[H_2O_2]_0$/COD ratio. For example, Increasing the molar ratio of $[H_2O_2]_0/[Fe^{3+}]_0$ from 10 to 50 (i.e. X_2 was from –1 to 1) would lead to a reduction in COD and TOC removal efficiency from 72% and 57% to 50% and 27%, respectively. This is due to the fact that ferric ions in solution play an important role in Fe^{2+} formation, and Fe^{2+} can quickly react with H_2O_2 to produce ·OH radicals as expressed by Eq. (1). A lower concentration of ferric ion may result in a less amount of Fe^{2+} formation for catalyzing H_2O_2 decomposition.

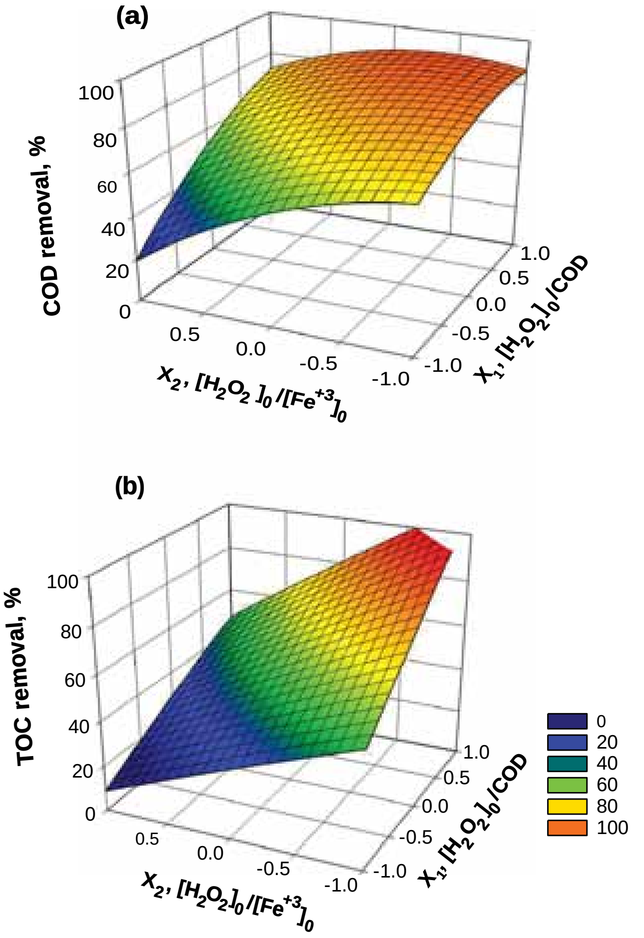


Fig. 2. Response surface for (a) COD and (b) TOC removal efficiency of AV dye in water as a function of initial $[H_2O_2]_0$/$[Fe^{3+}]_0$ and $[H_2O_2]_0$/COD. (Reaction time: 15 min, light intensity: 500 W/m^2, water temp.: 25 °C.)

3.2. *Optimization of solar photo Fenton-like treatment of AV dye in water*

On the basis of RSM application, the empirical relationship between the dye removal efficiency (*Y*) and independent variables are listed in Table 3. The regression model had a high value of coefficient of determination ($r^2 > 0.98$). This implies that the process efficiency could be predicted well by the polynomial regression equations under the conditions studied.

Based on the model equations, the optimal values of the process parameters were established as a $[H_2O_2]_0$/COD of 1.75, a $[H_2O_2]_0/[Fe^{3+}]_0$ molar ratio of 15 and a reaction time of 15 min in which at least a 90% COD and TOC degradation of AV in water would be achieved. In order to confirm the optimum conditions of AV degradation, a five replicates and *t*-test analysis were performed. The results showed that the average COD and TOC removal efficiency were 91.33% and 90.08%, respectively, and the 95% of confidence intervals for COD and TOC removal efficiency were 90.73–91.93% and 89.51–90.66%, respectively. Consequently, the credibility of the optimal conditions obtained in this study was acceptable.

3.3. *Effect of artificial solar light intensity*

Under the optimum dosage of Fenton-like's reagents, the effect of artificial solar light intensity on COD and TOC removal efficiency of AV in water was examined. As shown in Fig. 3, a 62% of COD and a 23% of TOC removal of AV were observed in Fenton-like process, in contrast to a 91% of COD and a 89% of TOC removal in solar Fenton-like process with a artificial solar light irradiation of 500 W/m^2 was achieved. This result implied that the generation of ·OH radicals may be comparatively slower in Fenton-like process [13]. In solar Fenton-like process, solar light irradiation could be used for the photolysis of H_2O_2 and the reduction of Fe^{3+}. Therefore, the higher the solar light irradiation, the higher the H_2O_2 consumption of in solution was. Moreover, the dyes in solution could absorb visible light irradiation and be degraded through photosensitization mechanism in which dye molecules were excited by Eq. (5) and then the excited dye molecules (dye*) released electrons into Fe^{3+} ions (Eq. (6)), resulting in the formation of Fe^{2+} ions and then promoting the proceeding of Eq. (1) [14].

Table 3
Polynomial regression equation of AV degradation on the basis of COD and TOC removal

Polynomial regression equation	r^2
$^{a}Y_{\text{COD}} = 78.6 + 18X_1 + 15.1X_2 + 14.2X_3 - 6.7X_1^{\,2} - 9.2X_2^{\,2} - 15.6X_3^{\,2} + 8.5X_1X_2 - 0.45X_1X_3 - 0.4X_2X_3$	0.981
$^{b}Y_{\text{TOC}} = 47.7 + 13X_1 - 18.9X_2 + 16.6X_3 - 1.2X_1^{\,2} - 0.9X_2^{\,2} - 6X_3^{\,2} - 6.1X_1X_2 + 12.2X_1X_3 - 8.4X_2X_3$	0.994

$^{a}Y_{\text{COD}}$: the COD removal efficiency of AV wastewater.
$^{b}Y_{\text{TOC}}$: the TOC removal efficiency of AV wastewater.

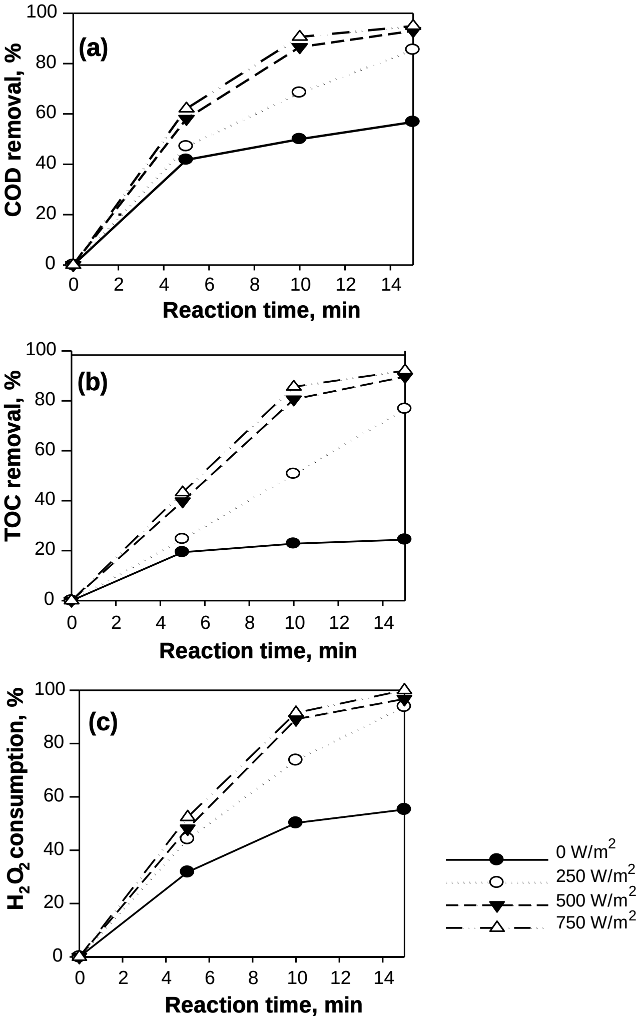


Fig. 3. Effect of solar light intensity on (a) COD, (b) TOC removal efficiency and (c) H_2O_2 consumption of AV dye in water. (Water temp.: 25 °C, 1.75 g H_2O_2/g COD, $[H_2O_2]_0$/$[Fe^{3+}]_0$ = 15).

$$\text{dye} + h\nu \text{ (visible light)} \rightarrow \text{dye*} \tag{5}$$

$$\text{dye*} + \text{Fe}^{3+} \rightarrow \text{dye}^{+} + \text{Fe}^{2+} \tag{6}$$

In addition, carboxyl intermediates such as oxalic acid were produced in solution (Fig. 4), which may further form ferric carboxylate complexes (Eq. (7)) during the reaction, leading to another reduction pathway of ferric ions – photodecarboxylation occurred by the aid of solar irradiation as expressed by Eq. (8), and drove the carboxyl intermediates to be further mineralized [15].

$$\text{Fe}^{3+} + 3\text{C}_2\text{O}_4^{2-} \rightarrow \text{Fe(C}_2\text{O}_4)_3^{3-} \tag{7}$$

$$\text{Fe(C}_2\text{O}_4)_3^{3-} + h\nu \xrightarrow{e<4\square\square\square\square} \text{Fe}^{2+} + 3\text{CO}_2 + 3\text{CO}_2^{\bullet-} \tag{8}$$

According to Eq. (1), the presence of more ferrous ions in solution could catalyze effectively more H_2O_2 molecules in solution, leading to additional ·OH radicals generation to degrade AV. Hence, it was found that the consumption of H_2O_2 was almost completed within 15 min of reaction under solar irradiation, while there was about 45% of H_2O_2 remained in solution in Fenton-like process. Moreover, the Fenton-like process could not perform photodecarboxylation due to lack of light irradiation. Consequently, as shown in Fig. 4, oxalate was continuously accumulated in Fenton-like process while the produced oxalate was mineralized through Eqs. (7) and (8) in solar Fenton-like process. The COD and TOC removal efficiencies of dye wastewater were therefore much lower in Fenton-like process. Increasing solar light irradiation from 500 W/m² to 750 W/m² did not impart a considerable enhanced effect, indicating that proper irradiation was enough for incessant reduction of Fe^{3+}

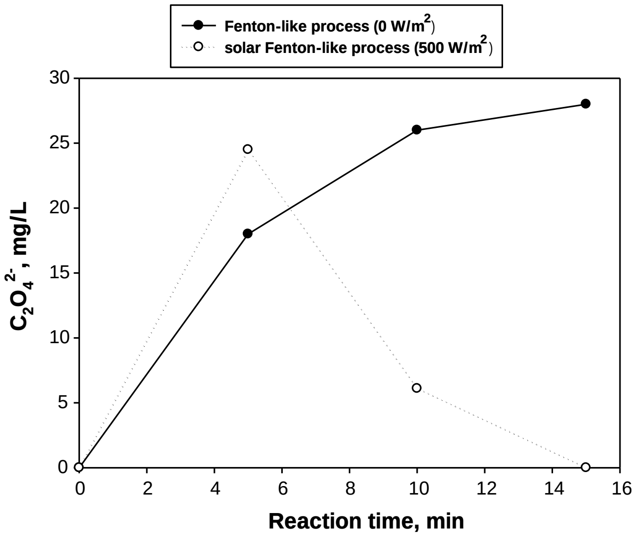


Fig. 4. Oxalate formation during degradation of AV dye with treatment of different process. (Water temp.: 25 °C).

ions through the mechanisms of direct photoreduction, photosensitization, and photodecarboxylation, and then persistent promotion of ·OH production.

3.4. *Effect of solar heat irradiation*

Effect of solar heat irradiation in terms of water temperature on the COD and TOC removal efficiency of AV dye in water was shown in Fig. 5. As shown in Fig. 5, raising water temperature showed a positive impact on the COD and TOC removal efficiencies of AV dye in water. The COD and TOC degradation efficiency of AV in water within 15 min could be increased from 78% and 65% to 96% and 92%, respectively as water temperature was raised from 15 to 35 °C due to solar heat irradiation. This is due to a higher water temperature increased the reaction rate between hydrogen peroxide and any form of ferrous/ferric ion, leading to increase the rate of generation of oxidizing species such as ·OH radical [16,17]. However, it was concluded in the study of Zapata et al. [17] that solar photo-Fenton process should be designed for operating at temperatures below 45 °C to avoid significant loss of iron and a decrease in efficiency.

To quantitatively analyze the effect of solar heat irradiation on solar Fenton-like oxidation of AV, a pseudo-first order model expressed by Eq. (9), which was generally used for the kinetic analysis of photo-Fenton process under conditions with a low initial concentration of pollutant in solution, was applied to obtain the rate constants

$$-\frac{dC}{dt} = kC \tag{9}$$

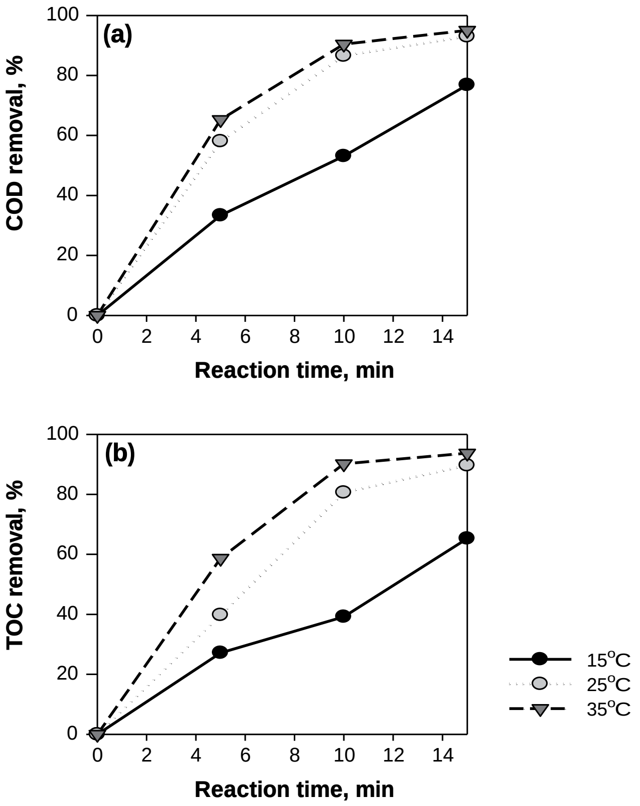


Fig. 5. Effect of water temperature on (a) COD and (b) TOC removal efficiency of AV dye in water (light intensity: 500 W/m^2, 1.75 g H_2O_2/g COD, $[H_2O_2]_0/[Fe^{3+}]_0 = 15$).

In which C is the concentration of COD or TOC in solution at time t, and k is the pseudo-first order rate constant. It was found that both the COD and TOC removal efficiencies of AV wastewater under various process temperatures followed the pseudo-first order reaction kinetics as indicated by a high correlation coefficient ($r^2 > 0.96$) (Table 4).

It was obvious that a significant enhancement for the oxidation rate of AV was achieved by increasing water temperature as indicated by a higher rate constant and a shorter half-life. In other words, the higher the solar heat irradiation, the faster the degradation rate of AV was. Particularly, the mineralization rate of AV increased more than two times by increasing water temperature from 15 to 25 °C, accompanied with a much shorter half-life for AV degradation. The temperature dependence of rate constants was further simulated with an Arrhenius equation (Eq. (10)):

$$\ln k = \ln A_0 - \frac{E_a}{RT} \tag{10}$$

where k is the reaction rate constant (min^{-1}), A_0 is the frequency factor (min^{-1}), E_a is the energy of activation

Table 4
Pseudo-first order rate constant of COD removal and TOC removal for AV wastewater at different water temperature

Process[a]	k_{COD}[b], min^{-1}	$t_{1/2}$[d], min	r^{2e}	k_{TOC}[c], min^{-1}	$t_{1/2}$[d], min	r^{2e}
15°C	0.0901	7.69	0.970	0.0640	10.83	0.967
25°C	0.1838	3.77	0.990	0.1513	4.58	0.989
35°C	0.2107	3.29	0.985	0.1973	3.51	0.984

[a] Operation condition: $[AV]_0$ = 100 mg/L, 1.75 g H_2O_2/g COD, $[H_2O_2]_0/[Fe^{+3}]_0$=15.
[b] k_{COD}: pseudo-first order rate constant based on the degradation of COD in solution.
[c] k_{TOC}: pseudo-first order rate constant based on the degradation of TOC in solution.
[d] $t_{1/2}$: ln 2/k.
[e] r^2: coefficient of correlation.

(kJ mol^{-1}), R is the universal gas constant (8.314 J/mol·K) and T is the absolute temperature (K). The result shown in Fig. 6 implied that the value of k was positively related to water temperature. According to the Arrhenius equation, the reaction activation energy could be calculated, and the value was found to be 7.5 kcal/ mol. The activation energy value obtained in this study is comparable to the result reported by Chen and Zhu [18]. In general, the reaction activation energy of ordinary thermal reactions is usually between 14.2 kcal/mol and 59.5 kcal/mol. Accordingly, it implied that the solar Fenton-like system required a much lower activation energy.

4. Conclusions

RSM could be used successfully in optimizing the treatment performance of solar Fenton-like process. Optimized reaction conditions based on the application of RSM were established as a $[H_2O_2]_0$/COD ratio of 1.75, a $[H_2O_2]_0/[Fe^{3+}]_0$ molar ratio of 15 and a reaction time of 15 min for more than a 90% of COD and TOC removals of AV in water. Also, experimental findings have revealed that Fenton-like oxidation assisted by solar irradiation is an effective treatment method. Increasing solar light irradiation could enhance the reduction of Fe^{3+} ions through the mechanisms of direct photoreduction, photosensitization, and photodecarboxylation, and then persistent promotion of ·OH production. Furthermore, water's temperature could be increased due to the effect of solar heat irradiation, which renders additional positive effect on the performance of solar Fenton-like process.

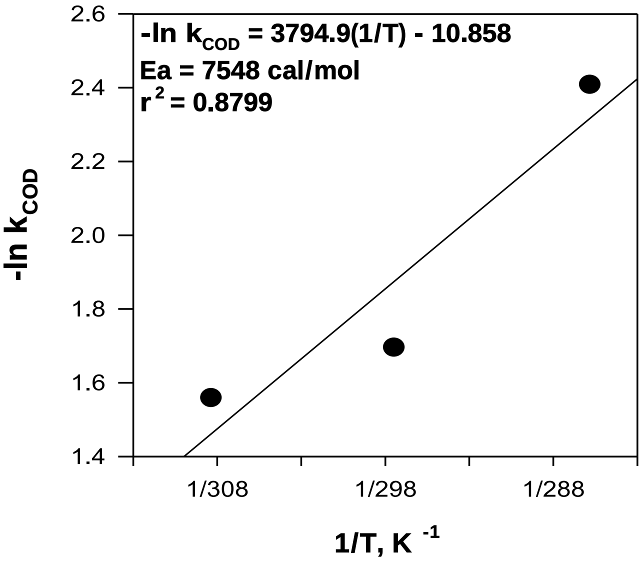


Fig. 6. Effect of water temperature on the degradation rate of AV dye in water (light intensity: 500 W/m^2).

Acknowledgements

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