



## The photooxidative destruction of C.I. Basic Yellow 2 using UV/S<sub>2</sub>O<sub>8</sub><sup>2-</sup> process in a rectangular continuous photoreactor

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### ABSTRACT

The photooxidative decolorization of C.I. Basic Yellow 2 (BY2), was investigated using UV radiation in the presence of peroxydisulfate (S<sub>2</sub>O<sub>8</sub><sup>2-</sup>) in a rectangular photoreactor at experimental condition. S<sub>2</sub>O<sub>8</sub><sup>2-</sup> and UV-light showed negligible effect when they were used independently. Removal efficiency of BY2 was sensitive to the operational parameters such as initial concentrations of S<sub>2</sub>O<sub>8</sub><sup>2-</sup>, BY2, light intensity, flow rate and pH. The conversion ratios of BY2 at the volumetric flow rates of 330, 500 and 650 ml/min were 84%, 79%, 51% in 30 min, respectively. Our results showed that light intensity was a beneficial parameter for dye removal. The results showed that in the presence of S<sub>2</sub>O<sub>8</sub><sup>2-</sup>, the photooxidation quantum yield obtained was higher than direct photolysis quantum yield, suggesting that photodecay of BY2 was dominated by photooxidation. The electrical energy per order (E<sub>EO</sub>) values for decolorization of BY2 solution was calculated. Results show that applying a desired peroxydisulfate concentration can reduce the E<sub>EO</sub>.

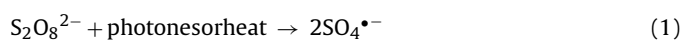
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### 1. Introduction

C.I. Basic Yellow 2 (4,4-dimethylaminobenzophenonimide) and its hydrochloride salt are used in the coloring of paper, textiles and leather [1]. International Agency for Research on Cancer included BY2 among chemicals for which there is sufficient evidence of carcinogenicity in experimental animals due to its bio-transformation to reactive species in target organs of both rats and humans [2]. The conventional methods used in sewage treatment, such as primary and secondary treatment systems, are insufficient in removing of dyeing and finishing agent, for example: adsorption and chemical coagulation processes are two common techniques of wastewater treatment. However, these methods merely transfer dyes from the liquid to the solid phase causing secondary pollution and requiring further treatment. Advanced oxidation processes (AOPs) are alternative techniques of destruction of dyes and many other organics in wastewater and effluents. These processes, generally, involve UV/H<sub>2</sub>O<sub>2</sub>, UV/O<sub>3</sub> or UV/Fenton's reagent for the oxidative degra-

dation of contaminants. Semiconductor photocatalysis is another developed AOP, which can be conveniently applied to remove different organic pollutants [3,4]. However, the potential of alternative AOPs to improve the removal performance is always of interest and the photochemical oxidant, peroxydisulfate, could be a good candidate for such a purpose.

Peroxydisulfate (S<sub>2</sub>O<sub>8</sub><sup>2-</sup>) is a strong oxidant (E<sup>0</sup> = 2.05 V) which has been used widely in the petroleum industry for the treatment of hydraulic fluids or as a reaction initiator in the petrochemical industry [4]. The peroxydisulfate is normally available as a salt associated with ammonium, sodium, or potassium. Potassium peroxydisulfate (KPS) recently was shown to be an effective disinfectant and/or oxidant for the Norwalk virus, foot and mouth disease and Coronaviridae (causing severe acute respiratory syndromes SARS). It has also been reported to be effective for degrading organics in hazardous wastewaters in acidic or basic media through direct chemical oxidation (DCO), where peroxydisulfate is used as a sacrificial reagent [5,6]. However, since the reactions of peroxydisulfate are generally slow at normal temperature, the thermal or photochemical activated decomposition of S<sub>2</sub>O<sub>8</sub><sup>2-</sup> ion to S<sub>2</sub>O<sub>8</sub><sup>2-</sup> radical has been proposed as a method of accelerating the process [6], as summarized in the following reactions:



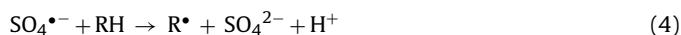
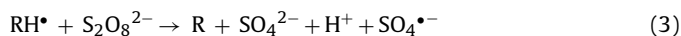
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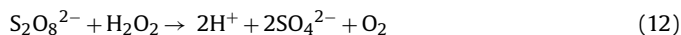
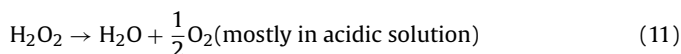
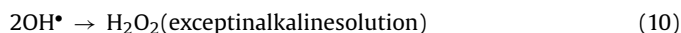
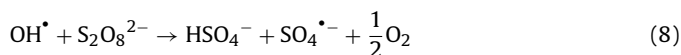
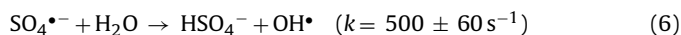
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(R is an organic material)

Also, the available oxidants in the solution and its related intermediates were indicated in the following reactions:



In the oxidation process, sulfate ions will be generated as the end-product, which leads to an increase in salt content in the effluent [6]. The  $\text{S}_2\text{O}_8^{2-}$  is practically inert and is not considered to be a pollutant; the USEPA has listed it under the secondary drinking water standards with a maximum concentration of 250 mg/l, based on aesthetic reasons [6]. Alternatively, potassium peroxydisulfate cost is much cheaper than other oxidants like hydrogen peroxide and ozone. Since peroxydisulfate will start reacting exothermally only at 100 °C [5,6], it is not recommended for use in conventional water purification processes, other factors such as different light intensity, water pH, and temperature might also affect its decomposition rate.

Previous studies have indicated that photooxidative degradation using  $\text{S}_2\text{O}_8^{2-}$  is a viable option, with complete decolorization and mineralization potentials [7–9]. However, many researchers have only used batch stirred reactor open to atmosphere with illumination from a UV lamp from above the solution or from immersion lamps. Flow bypass and light penetration are significant concerns in those types of reactor configurations.

In this study, a rectangular reactor, utilizing a small gap to improve mass transfer and UV light penetration, is used to treat a synthetic dye effluent. Important textile effluent parameters, such as the effect of UV light irradiation,  $\text{S}_2\text{O}_8^{2-}$  concentration, dye concentration, pH and light intensity were also investigated to examine their effects on the dye degradation rate. In this work with increasing light intensity (increase the number of switched lamps) and making a thin film layer of effluent on the photoreactor tray, we can decrease the time of photooxidation from 60 to 30 min in comparison with our pervious work in annular photoreactor [10]. The EE/O “Electrical Energy per Order” was calculated and showed that applying a desired peroxydisulfate concentration can reduce the  $E_{EO}$ .

## 2. Experiments and methods

### 2.1. Materials and photoreactor

The dye, C.I. Basic Yellow 2 (C.I. number = 41,000) was provided by Boyakhsaz Iran, and used without further purification (Fig. 1). Sodium hydroxide and hydrochloric acid were used to adjust pH, and all solutions were prepared in real water used in the finishing and textile factories in our region. The characteristics of the real water are presented in Table 1. Experiments were conducted using

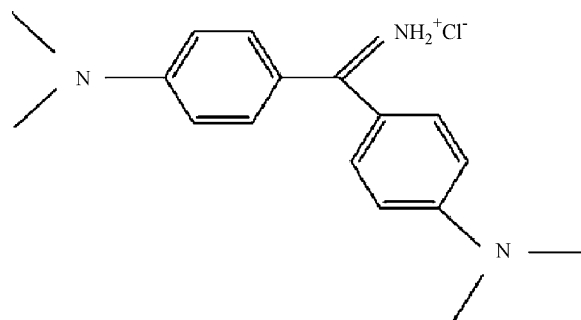


Fig. 1. Structure of C.I. Basic Yellow 2.

Table 1

Characteristics of real water.

Characteristics	Value
pH	7.1
Specific conductivity (mS/cm)	0.885
Total hardness (mg/l CaCO <sub>3</sub> )	192.5
Calcium hardness (mg/l CaCO <sub>3</sub> )	95
Carbonate hardness (mg/l CaCO <sub>3</sub> )	87

a semi industrial unit (Fig. 2) consisting of a rectangular continuous reactor. The photoreactor was constructed from rectangular stainless steel trays with two tanks at their ends. The tray was sized to accommodate a UV/C lamp (90 cm long and 30 cm wide). A holder of three ultraviolet lamps (Philips 30 W) was placed above the reactor. The distance between the single sun bed tubes was 5 cm. The solution in the reservoir was continuously stirred with a mechanistic stirrer, ensuring uniform mixing of the degrading dye solution.

### 2.2. Procedures

First of all, 4l synthetic solution containing the desired initial concentration of  $\text{S}_2\text{O}_8^{2-}$  and dye was charged to the photooxidative reactor and the agitating control system were turned on then a sample was taken as the reference. The UV light was turned on and the dye solution samples were taken at the desired time intervals. The dye solution samples were analyzed by an UV/vis spectrophotometer (WPA 2000, England) at  $\lambda_{\text{max}} = 432 \text{ nm}$  with a calibration curve based on the Beer–Lambert’s law. The operating conditions of all experimental test runs are summarized in each figure legend.

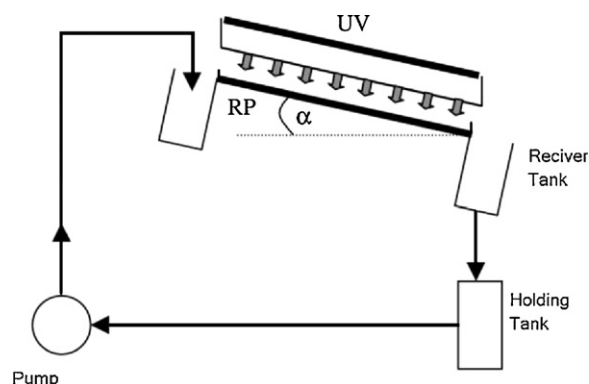
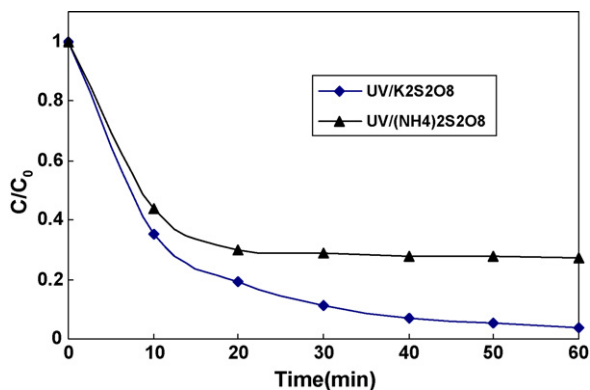


Fig. 2. Schematic representation of the continuous rectangular photoreactor.



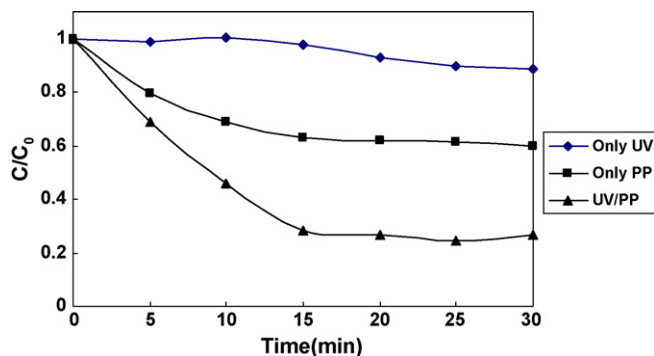
**Fig. 3.** Photooxidative decolorization of BY2 with different peroxydisulfate salt (5 mM  $S_2O_8^{2-}$ ).  $[BY_2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity = 15 W/m<sup>2</sup>, volumetric flow = 500 ml/min.

### 3. Results and discussion

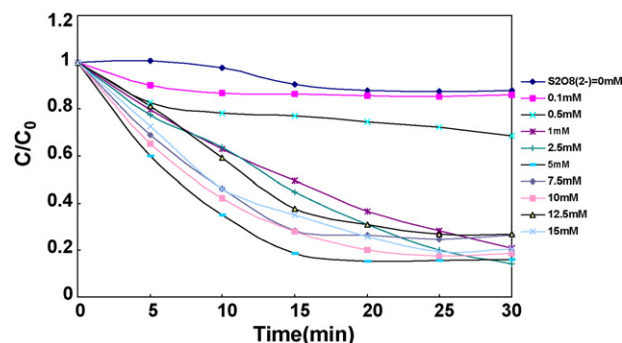
#### 3.1. Effect of UV irradiation in the presence of $S_2O_8^{2-}$

The comparative performance of using 5 mM  $K_2S_2O_8$  (KPS) and  $(NH_4)_2S_2O_8$  (APS) as the oxidant in the UV photolysis of 20 ppm BY2 decolorization at initial neutral pH is shown in Fig. 3. Although Graham et al. [5] recommended APS due to its exceptional solubility; we observed in our screening tests that KPS gave a more rapid removal of BY2 than APS at initial neutral pH. The difference in the removal efficiency is apparently due to the presence of the ammonium ion. The aqueous ammonium can undergo photo-oxidation [10] leading to nitrate and/or nitrite by the available oxidants in the solution, such as,  $S_2O_8^{2-}$  and its related intermediates  $H_2O_2$  or  $O_2$ . Furthermore, the reaction of  $NH_4^+/NH_3$  with UV/ $S_2O_8^{2-}$  process are proved to be able to convert it to nitrate under the 254 nm photolysis at a rate constant of  $0.25 \text{ mg l}^{-1} \text{ min}^{-1}$  or  $4 \times 10^{-3} \text{ mM min}^{-1}$  [11], thereby making the ammonium a competitor of BY2. In view of this, and the general unsuitability of adding ammonia to waters and wastewaters, APS is not recommended to be used in the UV/ $S_2O_8^{2-}$  oxidation process.

Fig. 4 shows the decay of BY2 vs. time for experiments carried out with UV radiation only,  $S_2O_8^{2-}$  without UV radiation and UV radiation plus  $S_2O_8^{2-}$ . There was no observable loss of the color when the irradiation was applied in the absence of  $S_2O_8^{2-}$ ; the color removal was not considerable in the absence of UV radiation. The results reveal that a considerable decrease in the concentration of the dye occurs when the sample was irradiated with UV in the



**Fig. 4.** Effect of UV radiation and peroxydisulfate in oxidative decolorization of BY2.  $[BY_2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity = 15 W/m<sup>2</sup>, volumetric flow = 500 ml/min.

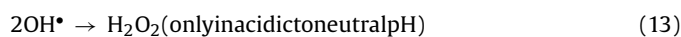


**Fig. 5.** Effect of initial concentration of peroxydisulfate in oxidative decolorization of BY2.  $[BY_2]_0 = 20$  ppm, initial pH = 6.72, light intensity = 15 W/m<sup>2</sup>, volumetric flow = 500 ml/min.

presence of  $S_2O_8^{2-}$ . This loss is due to the formation of hydroxyl radicals, which are powerful oxidizing agents.

#### 3.2. Effect of initial $S_2O_8^{2-}$ concentration

The concentration of  $S_2O_8^{2-}$  was found to be an important parameter for the degradation of BY2 in the UV/ $S_2O_8^{2-}$  system. The decay of BY2 was indicated in Fig. 5. For different  $S_2O_8^{2-}$  concentrations that increased from 0.1 to 5 mM, the photooxidative reaction rate constants increased from 0.0042 to 0.0642 min<sup>-1</sup>, respectively. This is likely because sulfate radicals and hydroxyl radicals are generated simultaneously and improved the photooxidative decolorization of BY2 [5,7]. Furthermore, when the  $S_2O_8^{2-}$  concentration increased beyond 5 mM, the increment of BY2 decay rate slowed down slightly. According to Qiao et al. [12] at higher  $S_2O_8^{2-}$  dosages, excessive generation of hydroxyl radicals (Eqs. (1) and (6)) would be recombined to form less reactive  $H_2O_2$  (Eq. (13)), which is a known quencher of  $OH^\bullet$  radical (Eq. (14)). Therefore, the rate increment of BY2 photooxidative decolorization is slightly slowed down at higher  $S_2O_8^{2-}$  dosages. However, such a recombination effect of the radical is likely not very effective due to the low steady-state concentrations of the radicals; higher decay rates of BY2 at higher  $S_2O_8^{2-}$  dosages are still expected.



#### 3.3. Effect of initial dye concentration

It is important from an application point of view to study the dependence of removal efficiency on the initial concentration of dye. Fig. 6 shows the effect of initial BY2 concentration on photooxidative decolorization efficiency. It can be seen that photooxidation efficiency decreased as initial dye concentration is increased at the same concentration of  $S_2O_8^{2-}$ . This can be postulated by considering that, the molar extinction coefficient of the dye at  $\lambda = 254$  nm is very high, so that a rise in its concentration induces an inner filter effect and hence the solution becomes more and more impermeable to UV radiation [13].

#### 3.4. Effect of the initial pH

The pH dependency on the BY2 photodegradation at 5 mM  $S_2O_8^{2-}$  is depicted in Fig. 7. Although a high degree of photooxidative decolorization by the UV/ $S_2O_8^{2-}$  process was observed, at all tested pH levels, the decolorization performance increased from low to initial neutral pH (pH 6.72) levels but decreased under basic conditions as shown in Fig. 7. This is likely due to the concentration

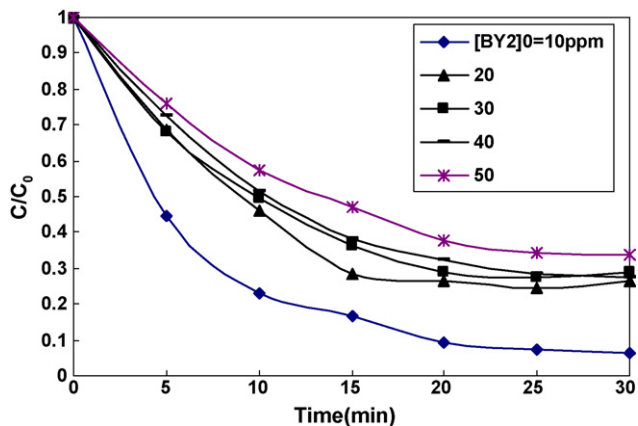


Fig. 6. Effect of initial concentration of BY2 in oxidative decolorization of dye.  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity =  $15$  W/m<sup>2</sup>, volumetric flow = 500 ml/min.

effect of active radicals in the solution. The BY2 decay increment from acidic to initial neutral pH is known due to the increase of  $OH^-$ , which induces a higher generation of  $OH^\bullet$ , and therefore the photooxidative decolorization efficiency. However, the decreasing photodecay at  $pH \geq 7$  can be explained by the following reasons: (a) the instability of  $H_2O_2$  at high pH level, (b) relatively higher amounts of  $OH^\bullet$  and  $SO_4^{\bullet-}$  were generated catalytically in alkaline conditions, which induced recombination of these two radicals (Eq. (9)), though this could be minor [5].

### 3.5. Spectral changes of BY2 during photodestruction

The changes in the absorption spectra of BY2 solutions during the photodestruction process at different irradiation times are shown in Fig. 8. The decrease of the absorption peak of BY2 at 432 nm in Fig. 8 indicated a rapid decolorization of dye. Complete decolorization of BY2 was observed after 30 min of irradiation. Also after 5 min the band intensity at 320 nm started to decrease and disappeared after 20 min. The decrease is also meaningful with respect to the carbon–nitrogen double bond of the diarylmethane dye, as the most active site for oxidative attack.

COD values have been related to the total concentration of organics in the solution and the decrease of COD reflects the degree of mineralization. It is important to measure the COD of the dye solution after the oxidative degradation in order to verify whether the dye is actually mineralized. The experiments for UV/ $S_2O_8^{2-}$  process showed that 45.5% reduction of COD occurred for 30 min of irradiation time.

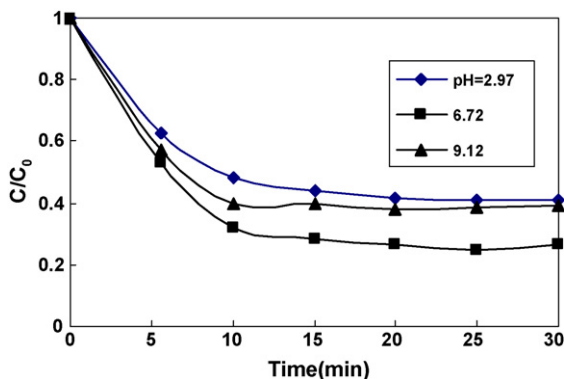


Fig. 7. Effect of pH in oxidative decolorization of BY2.  $[BY2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, light intensity =  $15$  W/m<sup>2</sup>, volumetric flow = 500 ml/min.

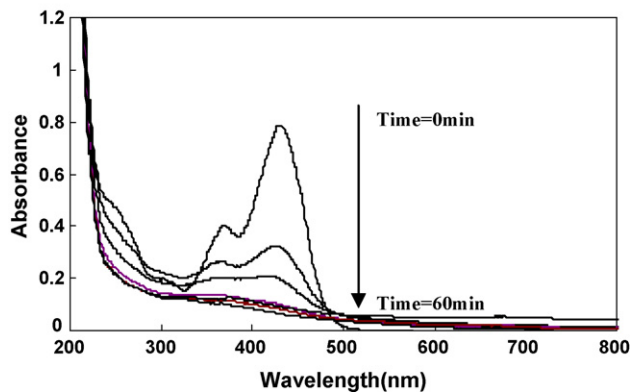


Fig. 8. Spectral changes of BY2 solution during illumination in the presence of  $S_2O_8^{2-}$ .  $[BY2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity =  $15$  W/m<sup>2</sup>, volumetric flow = 500 ml/min.

Fig. 9 shows three decreasing trends of COD, decolorization and aromatic content degradation as the UV/ $S_2O_8^{2-}$  oxidation proceeds. It appears that the UV/ $S_2O_8^{2-}$  process is more beneficial for decolorization rather than for degradation and COD removal. These results show that the measurement of 254 nm absorbance cannot exactly reflect the actual content of the solution [13].

### 3.6. Effect of volumetric flow rate on the BY2 photooxidative decolorization in an annular reactor

In commercial applications, the design of a continuous reactor is very important for a continuous operation. Investigation of residence time distribution (RTD) in a circulation reactor with the same volume BY2 with an inlet concentration of 20 ppm was done at the volumetric flow rates of 330, 500, and 650 ml/min, respectively. To investigate the effect of volumetric flow rate on the conversion ratio of BY2 due to the photodecomposition, the BY2 conversions were plotted with time at the flow rates of 330, 500, and 650 ml/min as shown in Fig. 10. The conversion ratio of BY2 increased as the flow rate decreased, which meant that the contact time of BY2 with a UV illumination was an important parameter in designing an annular photoreactor [10].

### 3.7. The effect of UV-light intensity

The influence of UV-light intensity on the decolorization of BY2 has been monitored by varying the UV-light intensities from 5.5

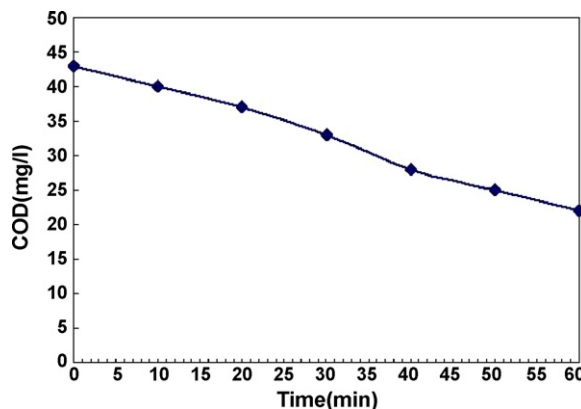
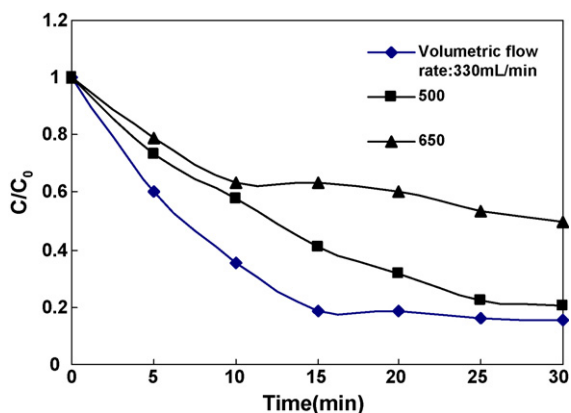


Fig. 9. Removal of COD for UV/ $S_2O_8^{2-}$  process.  $[BY2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity =  $15$  W/m<sup>2</sup>, volumetric flow = 500 ml/min.

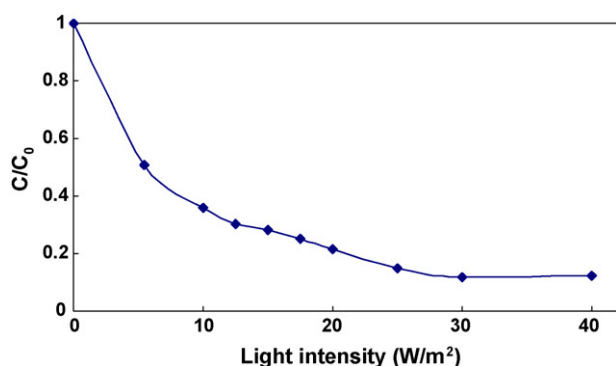


**Fig. 10.** Effect of volumetric flow rate in oxidative decolorization of BY2 in an annular photoreactor.  $[BY2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, light intensity =  $15$  W/m<sup>2</sup>.

to 40 W/m. The results are shown in Fig. 11. It appears that with increasing the light intensity the decolorization rate increases. This increase is due to the enhanced production of hydroxyl radicals. At low UV power the rate of photolysis of  $S_2O_8^{2-}$  are limited and at high UV power more hydroxyl radicals are formed, upon the photodissociation of  $S_2O_8^{2-}$ , hence decolorization rate increases [13,14].

### 3.8. Electrical energy efficiency

Science photodegradation of aqueous organic pollutant is an electric-energy-intensive process, and electric energy can represent a major fraction of the operating costs, simple figures-of-merit based on electric energy consumption can be very useful and informative. Recently, the Photochemistry commission of the International Union of Pure and Applied Chemistry (IUPAC) proposed a figure-of-merit (or more appropriately, an efficiency index, as it compares electrical efficiency of different AOPs) for UV-based AOPs. It compares electrical efficiency of different UV-based AOPs and it is a measure of the electrical efficiency of an AOP system. It is defined (for low concentration of pollutants) as the electrical energy in kilowatt hours (kWh) required bringing about the degradation of a contaminant by one order of magnitude in 1 m<sup>3</sup> of contaminated water. Considering first-order degradation kinetics, the UV dose was calculated for each of the processes using Eq. (15). The  $E_{EO}$  values were obtained from the inverse of the slope of a plot of  $\log(C_0/C)$  vs. energy dose (kWh/m<sup>3</sup>). The  $E_{EO}$  values are



**Fig. 11.** Effect of light intensity in oxidative decolorization of BY2 in photoreactor.  $[BY2]_0 = 20$  ppm,  $[S_2O_8^{2-}]_0 = 5$  mM, initial pH = 6.72, volumetric flow = 500 ml/min.

**Table 2**

$E_{EO}$  values for decolorization of BY2 (20 ppm) by UV-light ( $29.7$  W/m<sup>2</sup>) in the presence of various amounts of peroxydisulfate.

$[S_2O_8^{2-}]_0$ (mM/L)	$E_{EO}$ (kWh/m <sup>3</sup> ) (decolorization)
0.1	68.9655
0.5	28.0898
2	4.3365
5	4.2301
10	4.4822
20	5.0428

**Table 3**

Photodecay quantum yield of BY2 at 253.7 nm (the molar absorptivity of BY2 at 253.7 nm is  $8227$  mol<sup>-1</sup> l cm<sup>-1</sup> and light intensity =  $2.33 \times 10^{-6}$  Einstein l<sup>-1</sup> s<sup>-1</sup> and  $l = 1.4$  cm).

Process	Quantum yield
UV	0.00088
UV/ $S_2O_8^{2-}$	0.0093

presented in Table 2.

$$UV\text{Dose} = \frac{1000 \times \text{lamp power (kW)} \times \text{time (h)}}{\text{treated volume (l)}} \quad (15)$$

The electrical energy per order ( $E_{EO}$ ) values for decolorization of BY2 solution was calculated. Results show that applying a desired peroxydisulfate concentration can reduce the  $E_{EO}$  [15,16].

### 3.9. Comparison of the quantum yield for both direct photolysis and photooxidation

The quantum yield is a useful parameter indicating the efficiency of a photooxidation reaction, and is defined as the number of molecules being decomposed per photon absorbed [17]:

$$\varphi = \frac{\text{number of molecules reacted (or produced)}}{\text{number of photons of light absorbed}} \quad (16)$$

The quantum yield of BY2 degradation can be calculated from the observed first-order degradation rate constant where a specific form of quantum yield,  $\varphi$  based on first-order kinetics was indicated in the following [18]:

$$\varphi = \frac{k}{2.303 I_{0,\lambda} \varepsilon_\lambda l} \quad (17)$$

where  $k$  (s<sup>-1</sup>) is the pseudo-first-order rate constant,  $\varphi$  the quantum yield,  $I_{0,\lambda}$  (Einstein l<sup>-1</sup> s<sup>-1</sup>) the incident light intensity at wavelength  $\lambda$ ,  $\varepsilon_\lambda$  (cm<sup>-1</sup> mol<sup>-1</sup> l) the molar absorptivity at wavelength  $\lambda$ , and  $l$  is the cell path length (cm). The molar absorptivity,  $\varepsilon_\lambda$ , was calculated by the Beer–Lambert law from the measurement of absorbance at different BY2 concentrations.

The results of BY2 decay quantum yield through either direct photolysis or photooxidation under the illumination of UV at 253.7 nm are presented in Table 3. The results showed that in the presence of  $S_2O_8^{2-}$ , the photooxidation quantum yield was obtained higher than direct photolysis quantum yield, suggesting that photodecay of BY2 was dominated by photooxidation.

## 4. Conclusion

It is believable that UV/ $S_2O_8^{2-}$  is applicable to the existing drinking water or wastewater treatment plants, especially for those equipped with UV systems as disinfection. With the advantages of the high reactivity of UV/ $S_2O_8^{2-}$  process and the high solubility of peroxydisulfate, it is practically possible to dose the chemical solution at the upstream of wastewater to remove recalcitrant organic compounds effectively.

Using a continuous rectangular reactor configuration with a thin gap, high photooxidation efficiency was achieved, in less time than that found in previous studies. Removal efficiency of BY2 was sensitive to the initial concentrations of  $S_2O_8^{2-}$  and BY2. Alkaline and acidic pH conditions were found to not significantly effect on the dye photooxidation. The conversion ratios of BY2 at the volumetric flow rates of 330, 500 and 650 ml/min were 84%, 78% and 69% at 1 h, respectively. Our results showed that increasing the light intensity can increase decolorization efficiency. The electrical energy per order ( $E_{EO}$ ) values for decolorization of BY2 solution was calculated. Results show that applying a desired peroxydisulfate concentration can reduce the  $E_{EO}$ .

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