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Efficient optimization and mineralization of UV absorbers: A comparative investigation with Fenton and UV/H_2O_2

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Abstract: UV absorbers developed for finishing of textile materials play a significant role in protection against UV radiations but their discharge in wastewater during processing and laundry action also retain serious concern to living species due to their recalcitrant nature. The current study examined the mineralization and degradation of two vinylsulfone and nitrogen (N-) containing UV absorber compounds (1a, 2a) via two effective Fenton and UV/H_2O_2 oxidation. The results showed that both the Fenton and UV/H_2O_2 processes mineralized the synthesized UV absorbers effectively; however the mineralization process with Fenton oxidation was more effective than the UV/H_2O_2. The mineralization of synthesized UV absorbers was affected by process parameters (dosage of Fe^{2+} and H_2O_2, pH and reaction time). Under attained optimum conditions of Fenton oxidation, dose of Fe^{2+} (15 mg/L), H_2O_2 (500 mg/L), pH (3.0) and contact time (120 minutes), 75.43 and 77.54% of Chemical Oxygen Demand removal was achieved for 1a and 2a, respectively. Whereas, the optimum conditions of UV/H_2O_2 process were H_2O_2 (700 mg/L), pH(3.0) and irradiation time (200 minutes) that brought 54.33 and 57.65% COD removal in case of 1a and 2a, respectively. The results indicated that the Fenton oxidation can be successfully employed for the mineralization of triazine based UV absorbers.

Keywords: UV Absorbers; Oxidation; Mineralization; COD removal; Triazine.

1 Introduction

A number of different synthetic dyes, finishes and detergents have been employed frequently in several industries including textile [1-2], leather [3], automobiles [4], food and beverage [5], pharmaceutical [6], furniture [7], paper industry [8] and many more. They are preferred over naturally existing dyes and finishes due to their availability in various attractive shades, good wash and light fastness [9] and superior properties too [10-12]. However, considerable health issues are connected with them due to their recalcitrant nature [13-14], high molecular weight and complex structures [2].

Consequently, a huge amount of these chemicals can be found in industrial effluents, changing the taste, odor and color of water [15], and reacting with many heavy metals to form stable complexes [16]. Therefore, industrial waste comprises of huge quantities of organic pollutants. They represent high chemical oxygen demand (COD)/ total organic carbon (TOC) values [13] along with low biodegradability of industrial effluents [1]. Hence, their discharge into water bodies without any prior treatment causes serious health issues for aquatic and terrestrial life [17] due to their toxic and carcinogenic properties [18].

UV absorbers are excessively utilized in sunscreens [19] and on clothing [10] to protect us from the damages of UV radiations. Many organic UV absorbers incorporated in sunscreens, such as 2-phenylbenzimdazole-5-sulfonic acid (PBSA) [20] and monochlorotriazine based UV absorbers [10], to combat with UV light. The presence of SO_3H enhanced the photostablity of PBSA due to extended conjugation, but due to their high molecular weight and complex structure, their presence in effluents also generates high toxicity to biosphere [20].

Many physical, biological and chemical treatment methods are reported in literature, dealing with pollutants in industrial wastewater, such as precipitation, adsorption [21], membrane separation [22], treatment with bacteria [23] and oxidation [24]. All these methods convert pollutants in industrial effluents to smaller toxic

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molecules, resulting in incomplete mineralization [25]. Oxidation of organic pollutants by advance oxidation processes (AOPs) have been utilized to degrade a large variety of organic contaminates like dyes [26], pesticides [27] and pharmaceuticals [28].

The above processes produce hydroxyl radicals that are highly reactive with electrical potential 2.8 V and the organic substances are completely mineralized [13]. The combination of H_2O_2 with Fe^{2+} and UV radiation is characterized by a reduced cost and a high efficiency for removing non-biodegradable organic contaminants from water [24, 29]. The decomposition of hydrogen peroxide with Fe^{2+} and UV light generate reactive species such as OH^- and $^\bullet OH$ [30-32] (equation 1-3).

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

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

$$H_2O_2 + h\nu \rightarrow 2{}^\bullet OH \tag{3}$$

In the current investigation, two new reactive hetrofunctional vinyl sulfone and nitrogen (N-) containing organic UV absorber compounds (1a, 2a) that were designed and synthesized in our previous studies [33] were selected for mineralization. The degradation study of these UV absorbers has been not reported yet. The comparative degradation study was performed by Fenton and UV/H_2O_2 processes. The influencing parameters of processes were optimized to improve the degradation process.

2 Experimental

2.1 Chemicals and reagents

Both UV absorbers (1a-2a) were synthesized in the laboratory and their structures were confirmed with spectroscopic techniques. Structures of these UV absorbers are shown in Figure 1. H_2O_2 (30% solution w/w), $FeSO_4.7H_2O$ (99%), Na_2SO_3, H_2SO_4 (1M) and NaOH (1M) were of analytical grade and purchased from Sigma Aldrich. All of the solutions were made in distilled water.

2.2 Fenton procedure

UV absorber solution (500 ppm) was prepared in distilled water and pH adjusted using a digital pH meter with the help of NaOH (1 M) and H_2SO_4 (1M). The experiments were conducted in a conical flask (500 mL) containing 100 mL of UV absorber solution. The desired amount of $FeSO_4.7H_2O$ and H_2O_2 was added into the flask at the start of reaction. The conical flasks were covered by aluminum foil and placed on a water bath shaker (100 rpm) at room temperature (25±1°C) in Fenton process. To evaluate the effect of time, the sample (2 mL) was withdrawn periodically and the reactive species were quenched by Na_2SO_3 (0.5 mL) and the COD removal (%) of UV absorber solution was analyzed. To examine the possible maximum removal of UV absorber, the effect of different experimental parameters such as pH (2.0, 3.0, 5.0, 7.0 and 9.0), contact time (40, 80, 120, 160 and 200 min), H_2O_2 dose (100, 300, 500, 700, 900 mg/L) and $FeSO_4$ concentration (5, 10, 15, 20, 25 mg/L) were also assessed [30].

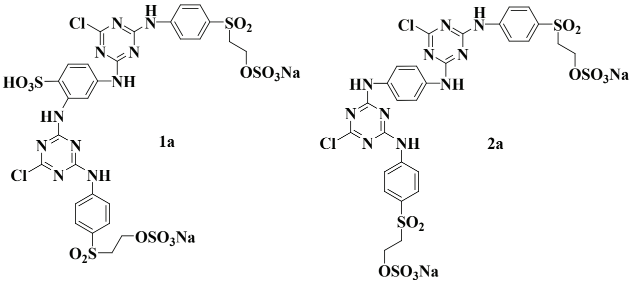

Figure 1: Structures of UV absorbers (1a-2a) selected for degradation.

2.3 UV/H_2O_2

A UV absorber solution (500 ppm) was prepared and adjusted to the pH of solution using a digital pH meter. A photo-reactor emitting at 254 nm was utilized for this experiment. An aqueous solution of UV absorber (100 mL) was placed under UV lamp and the desired amount of hydrogen peroxide (30%) was added at the start of the reaction and stirred at 100 rpm speed. To evaluate the effect of time, the sample was withdrawn periodically and the COD removal (%) of UV absorber was analyzed. To examine the possible maximum removal of UV absorber by UV/H_2O_2, the effect of different experimental parameters such as pH (2.0, 3.0, 5.0, 7.0 and 9.0), contact time (40, 80, 120, 160 and 200 min) and H_2O_2 dose (100, 300, 500, 700, 900 mg/L) were also measured [24].

2.4 Analytical methods

The concentration of UV absorbers (1a and 2a) was measured by calculating their absorbance at their λ_{max} 292 nm and 282 nm, respectively using Perkin Elmer Lambda (CE-7200) UV/Vis Spectrophotometer [20]. The pH of the solution was measured with the use of a digital pH meter (Hanna Instruments, Europe) [32]. The COD of solution was determined via the colorimetric method using equation 4 [30].

$$\%\mathrm{COD} = \frac{COD_0 - COD_t}{COD_0} \times 100 \tag{4}$$

Where, COD_0 and COD_t are the chemical oxygen demand of UV absorber before and after degradation respectively. Ethical approval: The conducted research is not related to either human or animals use.

3 Results and discussion

3.1 Generation of reactive species by Fe^{2+}/UV irradiation

The generation of reactive oxygen species (OH^-, $^\bullet OH$) by the decomposition of hydrogen peroxide by Fe (II) or UV irradiations plays a central role in degradation of organic contaminants in water [34-35]. As the generation of reactive oxygen species is enhanced, it ultimately improves the degradation process [36]. For the optimization of influencing factors of the Fenton and UV/H_2O_2 processes, a number of experiments were conducted and results are discussed here.

3.2 Fenton Oxidation

3.2.1 Effect of pH on COD removal

The efficiency of the oxidative process of organic toxins has been highly affected by pH change in Fenton oxidation [24,37]. But, there is no such consistency reported regarding the optimum pH, and it varies from one case to another. In the current study, the pH of aqueous solutions of UV absorbers were set at pH 2.0, 3.0, 5.0, 7.0 and 9.0 under constant H_2O_2 dosage of 100 mg/L, Fe^{2+} dosage of 5 mg/L and contact time of 120 minutes. Figure 2 (A) demonstrated that the maximum COD removal efficiency (%) was attained at pH of 3.0 in case of both the UV absorbers (**1a-2a**). With an increase in pH above 3.0, the COD removal efficiency of Fenton process was reduced. When the pH value increased from 2.0 to 3.0, the mineralization of absorbers proportionally increased, and then inversely decreased after pH 3.0. Such behavior of the Fenton process can be explained by a decline in the amount of free ions in solution due to precipitation of iron or reaction of $^\bullet OH$ with one another to reduce the quantity of free hydroxyl radical at pH > 4.0. The pH < 3.0 causes the creation of $[Fe^{2+}(H_2O)]^{2+}$ in medium, which slowly reacts with hydrogen peroxide and generates fewer hydroxyl radicals. Kinetically, H_2O_2 decomposition follows pseudo-first order kinetics [38].

3.2.2 Effect of H_2O_2 concentration COD removal

For the optimization of H_2O_2 dosage, the experiments were performed by varying the concentration of hydrogen peroxide (100, 300, 500, 700 and 900 mg/L) under constant pH of 3.0, Fe^{2+} dosage 5 mg/L and contact time 120 minutes. Figure 2(B) explained the effect of hydrogen peroxide concentration on the COD removal efficiency (%) of UV absorbers (**1a-2a**). The results confirmed that maximum COD removal efficiency (%) was achieved with 500 mg/L of hydrogen peroxide in Fenton oxidation. After the optimum level had been reached, no further improvement in COD removal efficiency (%) was achieved. Excess H_2O_2 produced per-hydroxy radical; having less oxidation potential and starts working as hydroxyl radical scavenger [39].

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

3.2.3 Effect of Fe^{2+} dosage on the COD removal

Figure 2(C) demonstrated that the maximum COD removal efficiency (%) was attained with 15 mg/L of iron dosage for UV absorbers (**1a-2a**). It was found that the COD removal efficiency of absorbers have a similar changing pattern with an increase of Fe^{2+} dose. The COD removal was increased considerably as the concentration of iron increased but after a certain level, the COD removal was decreased. As the dose of iron rises, the COD removal efficiency improves. After 15 mg/L, addition of Fe^{2+} is unproductive. Higher dose of Fe(II) after optimum level would generate other side and competitive reactions, decreases the concentration of free radicals [(reaction (6), (7), (8) and (9)]. Figure 3 shows the absorbance of aqueous solutions of UV absorbers prior and following the degradation via Fenton oxidation.

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + HO_2^\bullet \tag{6}$$

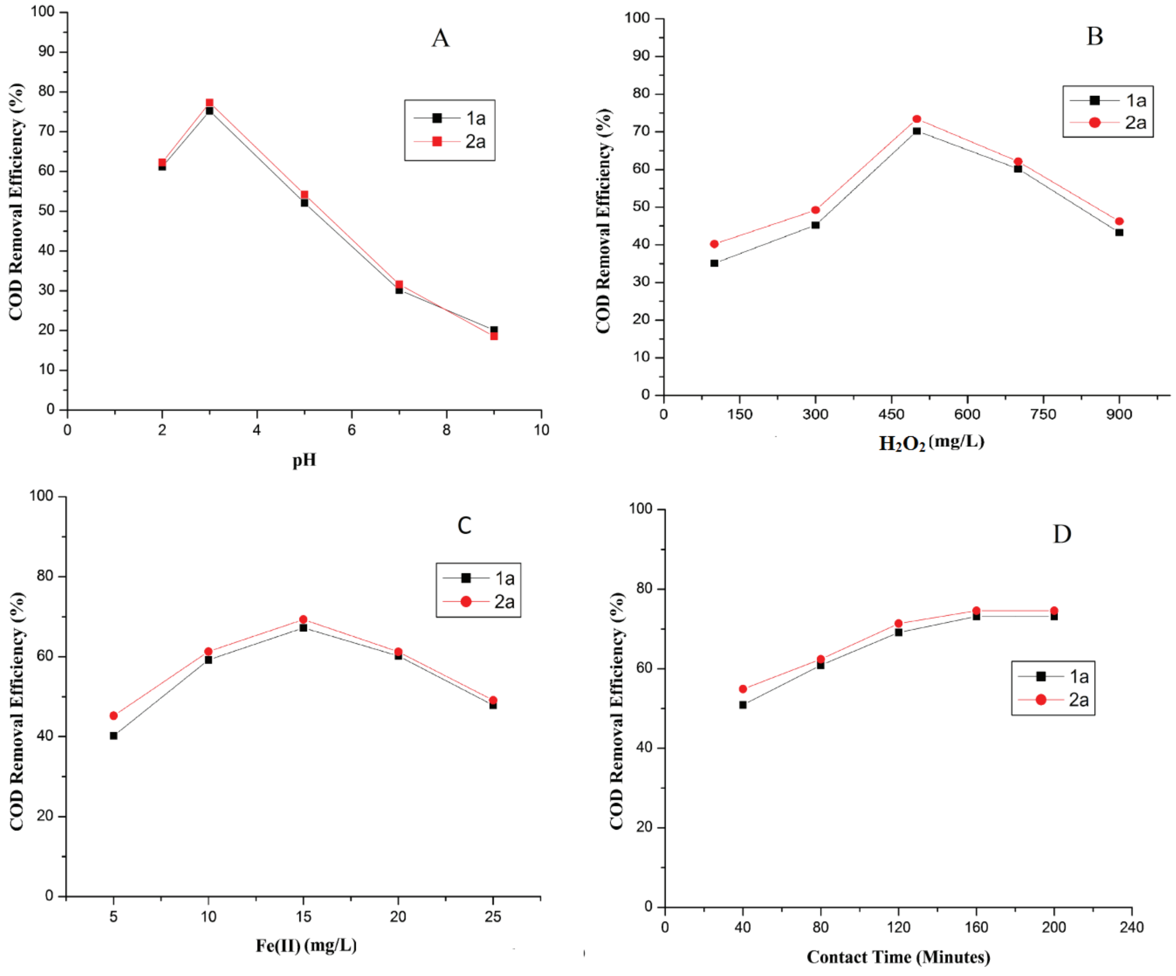

Figure 2: Effect of pH (A), H_2O_2 (B), Fe^{2+}(C) and contact time (D) on COD removal efficiency of UV absorbers (1a-2a) in Fenton oxidation.

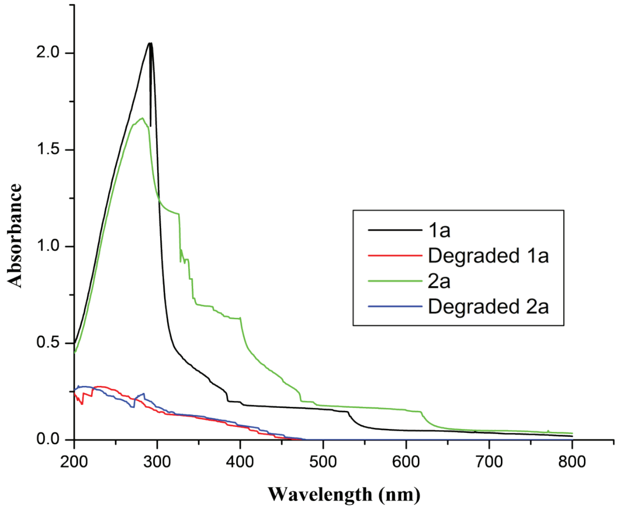

Figure 3: UV-Vis spectra of absorbers in aqueous solution (500 ppm) before and after degradation in Fenton oxidation.

$$^{\bullet}OH + H_2O_2 \rightarrow HO^{\bullet} + H_2O \tag{7}$$

$$Fe^{3+} + HO_2^{\bullet} \rightarrow Fe^{2+} + H^+ + O_2 \tag{8}$$

$$Fe^{2+} + HO_2^{\bullet} \rightarrow Fe^{3+} + HO_2^- \tag{9}$$

3.2.4 Effect of reaction time on the COD removal

Figure 2(D) demonstrated that maximum COD removal efficiency (%) was attained at 80 minutes in Fenton process. After the optimum level of time duration, no further improvement in COD removal efficiency (%) was achieved. As the reaction time increased, the improvement in COD removal (%) increased linearly, and then it became constant. This can be explained by the fact that most of the hydrogen peroxide reacted with Fe (II) in the beginning of the reaction. The rate of the reaction was high at start

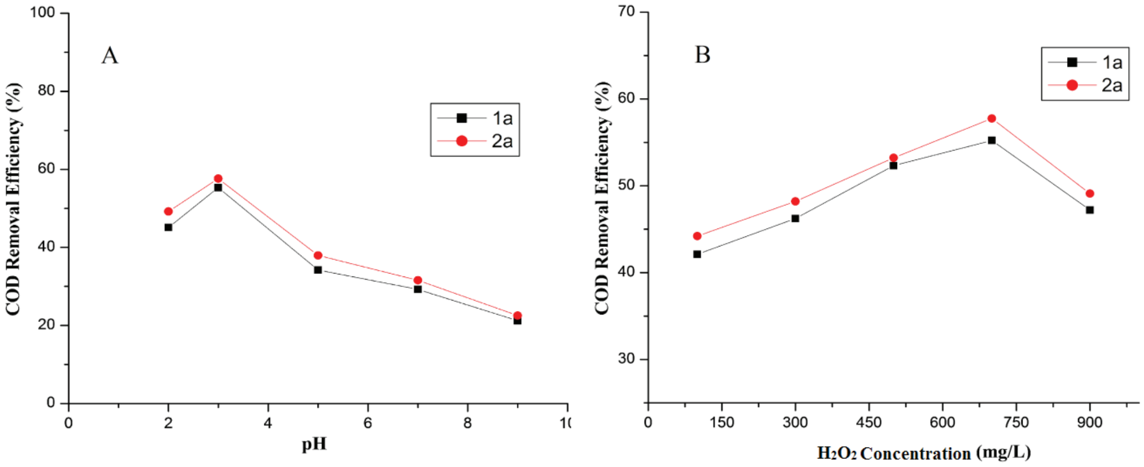

Figure 4: Effect of pH (A) and concentration of H_2O_2 (B) COD removal efficiency of UV absorbers (1a-2a) in UV/H_2O_2.

of the reaction than it increased slowly and became constant. A study on the effect of time for the treatment of landfill leachate in terms of COD removal efficiency (%) revealed that organic pollutants were speedily degraded in the first 20 minutes, after which any further increase in time duration became insignificant. Generation of a layer of foam on the surface of solution was observed as the oxidation proceeded due to formation of carbon dioxide [40].

3.3 UV/H_2O_2 Oxidation

3.3.1 Effect of pH on COD removal

With the UV irradiation time of 200 minutes and concentration of H_2O_2 100 mg/L, the effect of pH including 2.0, 3.0, 5.0, 7.0 and 9.0 on the mineralization of synthesized UV absorbers is shown in Figure 4(A). When the pH value increased from 2.0 to 3.0, the mineralization of UV absorbers increased and then linearly decreased. A pH of 3.0 achieved the maximum mineralization of UV absorbers 1a and 2a which were 55.34 and 57.68 respectively. At a pH of 9.0, the COD removal percentage of 1a and 2a were 21.23 and 18.54 respectively.

3.3.2 Effect of H_2O_2 concentration

In the experiments using a constant irradiation time of 200 minutes and pH of 3.0, hydrogen peroxide dosage were set at 100, 300, 500, 700 and 900 mg/L. The effect of H_2O_2 concentration on mineralization of UV absorbers is shown in Figure 4(B). The maximum COD removal of both the UV absorbers achieved at a 700 mg/L of H_2O_2. Particularly, the removals were 55.23 and 57.67 % for UV absorber 1a and 2a respectively.

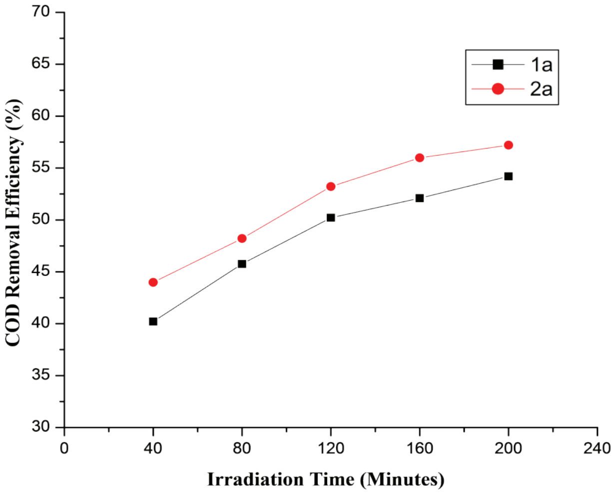

Figure 5: Effect of irradiation time on COD removal efficiency of UV absorbers (1a-2a) in UV/H_2O_2 process.

3.3.3 Effect of irradiation time on the COD removal

Preliminary experiments were performed by varying UV irradiation times to 40, 80, 120, 160 and 200 minutes under constant dosage of H_2O_2 (100 mg/L) and pH of 3.0. Figure 5 shows the effect of UV irradiation time on mineralization of target UV absorbers (**1a** and **2a**). It is evident from the figure that mineralization increases gradually with time. The COD removal (%) of UV absorbers (1a and 2a) was 54.2 and 57.32 respectively at the irradiation time of 200

Table 1: optimum condition for Fenton and UV/H_2O_2 oxidation for mineralization of selected UV absorbers (1a-2a).

Sr. No.	Selected parameters	Fenton Oxidation	UV/H_2O_2 Oxidation
1	pH	3.0	3.0
2	H_2O_2	500 mg/L	700 mg/L
3	Fe^{2+}	15 mg/L	-
4	Reaction Time	120 minutes	200 minutes
5	COD Removal (%)	75-77%	54-57%

minutes. Abdelraheem et al. (2015) mineralized the UV absorber and found that due to strong photostablity of UV absorber, its degradation appeared after 285 minutes of irradiation time. This result showed that degradation of these compounds was due to attack of hydroxyl radicals that were produce through decomposition of H_2O_2 by UV radiations.

4 Comparison of Fenton and UV/H_2O_2 oxidation

Comparison of Fenton and UV/H_2O_2 oxidation based on COD removal percentage (%) shows that, ferrous ions catalyzed the production of $^{\bullet}OH$ from hydrogen peroxide during Fenton oxidation. The generation of hydroxyl free radicals increases as the concentration of ferrous ions increases up to a optimum level, which propagated the oxidation of UV absorbers. However, the generations of hydroxyl free radicals in UV/H_2O_2 is less due to the presence of photostable organic UV absorbers, ultimately it reduces the efficiency of UV/H_2O_2 oxidation. Table 1 shows that economically Fenton oxidation is better for mineralization of selected triazine based UV absorbers.

5 Conclusion

In this study, two new triazine based UV absorbers designed for cotton fabric were degraded by applying two advance oxidation processes (Fenton oxidation and UV/H_2O_2). The results indicated that Fenton oxidation was more efficient towards mineralization of both the UV absorbers than UV/ H_2O_2 and results were in agreement with previous literature [20, 30, 35, 40]. Under attained optimum conditions of Fenton oxidation, dose of Fe^{2+} (15 mg/L), H_2O_2 (500 mg/L), pH (3.0) and contact time (120 minutes), 75.43% and 77.54% of COD removal were achieved for 1a and 2a, respectively. Whereas, the optimum conditions of UV/H_2O_2 process were H_2O_2 (700 mg/L), pH (3.0) and irradiation time (200 minutes) which brought 54.33 and 57.65%. It is concluded that synthesized organic triazine based UV absorbers can efficiently be mineralized and degraded via AOPs.

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Conflict of interest: There is no conflict of interest.

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