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Photochemical degradation of dimethyl phthalate by Fenton reagent

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Abstract

The photochemical degradation of dimethyl phthalate (DMP) in water was investigated under a variety of reaction conditions using hydrogen peroxide and Fe^{2+} (Fenton reagent) as oxidant. All experiments were conducted under artificial UV light (high pressure Hg lamp). Fenton reagent vigorously enhanced the photo-degradation rate of dimethyl phthalate. The experimental results showed that the optimum conditions were obtained at pH 3 for the UV/H_2O_2/Fe^{2+} system, with original H_2O_2 and Fe^{2+} concentrations of 5.00×10^{-4} and 1.67×10^{-4} M, respectively. The photo-degradation percentage of dimethyl phthalate within 120 min was as high as 81% under the optimum conditions. Therefore, the process of photo-Fenton was an effective treatment process under acidic condition and produced a higher photo-degradation percentage of dimethyl phthalate during a very short radiation time.

Keywords: Dimethyl phthalate (DMP); Photo-degradation; Fenton reagent; Fe(II); H_2O_2; Hydroxyl radical

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1. Introduction

In recent years, various photochemically advanced oxidation processes (AOPs) such as O_3/UV [1], UV/H_2O_2 [1,2], TiO_2/UV [3–5] and Fenton/UV [1–3,6–13], have been successfully utilized to degrade most of the organic compounds present in polluted water. The reason for the utilizations of AOPs is mainly due to the inability of biological processes to treat highly contaminated and toxic wastewater. In AOP, the hydroxyl radicals are generated in solution and are responsible for the oxidation and mineralization of the organic pollutants to water and carbon dioxide [2,14].

Fenton reagent, one of AOPs, mixed by hydrogen peroxide and iron salts, is an oxidant with highly oxidative effect. The primary reactions of representative Fenton process are [15–17]:

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + {}^{\bullet}OH + H_2O \quad (k_1 = 58\ \mathrm{mol^{-1}\ dm^3\ s^{-1}}) \tag{1}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + {}^{\bullet}OOH + H^+ \quad (k_2 = 0.02\ \mathrm{mol^{-1}\ dm^3\ s^{-1}}) \tag{2}$$

where ${}^{\bullet}OH$ is the hydroxyl radical and ${}^{\bullet}OOH$ is the superoxide radical.

Owing to hydroxyl radicals' high oxidation potential ($E_0 = -2.80$ V) [18], they can oxidize almost all the organic substances and mineralize them to carbon dioxide and water [1–3,6–18]. Thus, Fenton treatment has attracted much interest for the destruction of toxic organic compounds in wastewater. In recent years, many studies have shown that the decomposition of various organic pollutants using hydrogen peroxide as an oxidants under UV illumination have been proven to be very effective [1,2,6–12]. Phenol [1], *p*-chlorophenol [2], polyvinyl alcohol [6], dichloroacetic acid [7], 2,4-dichlorophenol [7], malachite green [8], benzothiazole [9], diclofenac [10], nitrobenzene [11], nitrophenols [12] and many other aromatic compounds are degraded much more rapidly under the UV illumination of the Fe^{2+}/H_2O_2 or Fe^{3+}/H_2O_2 systems than in the dark reactions.

Phthalate acid esters (PAEs) are a class of chemical compounds most widely used in the industrial production. Their major applications are as plasticizers for polyvinyl chloride (PVC) resins, adhesives, and cellulose film coating (about 85% of the whole production) [19]. Minor applications are in cosmetics, insect repellent, insecticide carries and propellants [20]. The current worldwide yield of phthalate acid esters has reached 2.7 million metric tonnes per year [21]. The large production and utilization caused a significant environmental diffusion of these compounds. In 1984, it was estimated that at least 23,000 Mg of PAEs yearly was

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released into the environment [22], and therefore the United States Environmental Protection Agency (USEPA) and several other countries had classified the most commonly occurring PAEs as 'priority pollutants' [22].

The short-chained esters such as dimethyl phthalate (DMP) are among the most frequently identified PAEs in diverse environmental samples including surface marine waters [23–27], freshwaters [19,27–29] and sediments [23,27,30]. These esters in various environments mainly come from the discharge of wastewater with these pollutants, and leaching and volatilization from plastic products during their usage and after disposal [21,31]. Esters are found to accumulate in the environment and to be toxic to a variety of aquatic organisms, which are at the base food chain in both marine and fresh water environments [31]. Since phthalate esters are becoming a class of concerning water pollutants, which are difficult to biologically and photo-chemically degrade [31], there is a strong need to look for effective treatment processes for such pollutants. Therefore, we have first undertaken a detailed kinetic study on the photo-degradation of a representative phthalate ester (DMP) by Fenton reagent.

This work examined the photo-degradation of DMP under irradiation by UV light. It was found that the Fenton degradation of DMP could be greatly accelerated by UV irradiation. This provides an effective approach to the treatment of DMP polluted waters using UV light.

2. Experimental

2.1. Materials

Dimethyl phthalate, hydrogen peroxide (30% w/w), $FeSO_4{\cdot}7H_2O$ and Na_2SO_3, purchased from Shanghai Chemical Reagent Co., China, were of analytical grade and used without further purification. Acetonitrile was HPLC grade and also purchased from Shanghai Chemical Reagent Co., China. Deionized water was used throughout the study. The initial concentration of DMP in all experiments was $10\,mg\,l^{-1}$. In the experiments, the higher concentration of H_2O_2 was determined by a potassium permanganate method. A given volume of higher concentration H_2O_2 solution was added into the solution of DMP. The pH value of solution was 3.0 (if not mentioned differently), which was adjusted using NaOH and H_2SO_4 solution. The change of pH was within 0.2 in the solution during irradiation. Therefore, pH buffer solution was not used in the experimental processes.

2.2. Device

The photo-degradation reaction of DMP in aqueous solution during the Fenton-treatment was performed in SGY1 Model Multifunction Photochemistry Reactor (Nanjing Sidongke Instrument Co. Ltd., China). A 150 W high pressure Hg lamp (Institute of Electric Light Source of Beijing, China) as a UV light source was positioned within a cylindrical quartz vessel. The cylindrical quartz vessel with water circulation was used to cool the lamp. Quartz glass tubes (20 ml) were utilized as reaction vessels. An iron disk holding 30 reaction tubes was placed inside the reactor and tubes were positioned approximately 4 cm away from the UV lamp.

2.3. Photo-degradation procedures

The sample tubes were periodically removed from the batch during the course of the reactions. The dark reaction was conducted by wrapping the tubes with aluminum foil to prevent exposure to light. One drop of 10% aqueous solution of Na_2SO_3 was added to each sample to quench the reaction. One hundred and twenty minutes was selected as the total treatment time. All experiments were carried out at $20 \pm 1\,^{\circ}C$.

2.4. Analytical methods

At given irradiation time intervals, the samples taken out were analyzed by HPLC experiments. The HPLC experiments were carried out on both devices: a Hewlett-Packard, series 1050 with UV detection at 219 nm and a Waters chromatograph 540 with a Waters photodiode array detector 990. For analytical purpose, Touzart and Matignon column kromasil C-18, 5 μm, 100 Å, 150 mm × 4.6 mm was used. As a mobile phase, mixture of acetonitrile with H_2O (ACN/H_2O 40/60 v/v) was utilized. The flow rate was $1\,ml\,min^{-1}$. All samples were immediately analyzed to avoid further reactions. Then, changes in DMP concentrations with irradiation time were determined.

3. Results and discussion

3.1. Effect of UV/Fenton system

From Fig. 1, it can be found that the photo-degradation of DMP could not occur in aqueous solution only with Fe^{2+} or H_2O_2. Although hydroxyl radical could be formed in H_2O_2 solution under the radiation according to Eq. (3) [32],

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

the degradation of DMP still did not occur (curve b of Fig. 1). This might be because the amount of $^{\bullet}OH$ formed from Eq. (3) was very little. The point proves that DMP is extremely inert and difficult to be degraded.

Moreover, from Fig. 1 it also can be seen that the degradation of DMP occurred in the solution with Fe^{2+} and H_2O_2 (i.e. Fenton reagent) in dark (curve c of Fig. 1). The reason is mainly catalytic effect of ferrous salt. From Eqs. (1) and (2), it is apparent that the decomposition of H_2O_2 by iron ions is through the interaction between Fe^{2+} and Fe^{3+}. Because the reaction rate of Eq. (1) was faster than that of

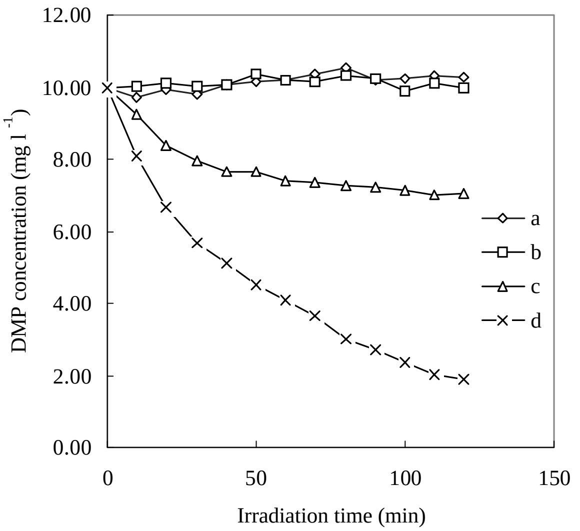

Fig. 1. Photo-degradation of DMP (10 mg l^{-1}) at pH 3.0 under different conditions. (a) Solution with Fe^{2+} (1.67×10^{-4} mol l^{-1}) under UV light irradiation; (b) solution with H_2O_2 (5.00×10^{-4} mol l^{-1}) under UV light irradiation; (c) solution with H_2O_2 (5.00×10^{-4} mol l^{-1}) and Fe^{2+} (1.67×10^{-4} mol l^{-1}) in dark; (d) solution with H_2O_2 (5.00×10^{-4} mol l^{-1}) and Fe^{2+} (1.67×10^{-4} mol l^{-1}) under UV light irradiation.

Eq. (2), Fe^{2+} was rapidly oxidized to Fe^{3+} in the reaction process. Therefore, the concentration of hydroxyl radical in the solution rapidly increased and could oxidize DMP. In addition, from Eq. (2) it could be seen that Fe^{2+} could be re-generated from the reduction of Fe^{3+}, leading to the continuous generation of hydroxyl radical.

Finally, Fig. 1 showed that the rate of degradation of DMP in the H_2O_2/Fe^{2+}/UV system (curve d of Fig. 1) was much faster than that in the H_2O_2/Fe^{2+}/dark system (curve c of Fig. 1). At the same time, the degradation percentage of DMP in the H_2O_2/Fe^{2+}/UV system during 120 min was over twofold that of DMP in the H_2O_2/Fe^{2+}/dark system (Table 1). In combination of thermal process and UV irradiation, the oxidation power of Fenton reagent was significantly increased due mainly to the photo-reduction of Fe(III) to Fe(II), which could react with H_2O_2 establishing a cycle mechanism of generating additional hydroxyl radials (Eq. (4)) [33–35].

$$Fe^{3+} + H_2O + h\nu \rightarrow {}^{\bullet}OH + Fe^{2+} + H^+ \qquad (4)$$

Furthermore, the effect of UV light was also attributed to the direct hydroxyl radical formation and regeneration of Fe^{2+} from the photolysis of the complex $Fe(OH)^{2+}$ in solution. It was known that the exiting form of ferrous iron was connected with the acidity of solution. At about pH 3.00, a part of ferrous iron would exist as the form of $Fe(OH)^{2+}$, whose photolysis under UV illumination could directly produce ${}^{\bullet}OH$ radical and Fe^{2+}, as indicated below [36]:

$$Fe(OH)^{2+} + h\nu \rightarrow {}^{\bullet}OH + Fe^{2+} \qquad (5)$$

Therefore, higher degradation rate and percentage of DMP came out in the UV/Fenton system.

Table 1
Photo-degradation percentage of DMP at pH 3.0 under different conditions within 120 min

Conditions	Fe^{2+}/UV	H_2O_2/UV	Fe^{2+}/H_2O_2/dark	Fe^{2+}/H_2O_2/UV
Degradation percentage (%)	0	0	30	81

3.2. *Effect of original H_2O_2 concentration on the photo-degradation of dimethyl phthalate*

Since the primary factor contributing to the chemical costs of Fenton reagent is the cost of H_2O_2, it is important to minimize the amount of H_2O_2 required, especially for treating large volumes of wastewater with pollutants such as DMP. Therefore, the impact of the change in H_2O_2 concentration on Fenton oxidation of DMP was investigated. In these experiments, the original concentration of H_2O_2 was changed from 0 to 8.33×10^{-4} mol l^{-1}, but the concentration of Fe^{2+} kept constant. The photo-degradation states of dimenthyl phthalate under different original concentrations of H_2O_2 were shown in Fig. 2 and the degradation percentages during 120 min were presented in Table 2.

From Fig. 2 it could found that photolysis of DMP did not occur in the absence of H_2O_2, but by the addition of H_2O_2, DMP began to be degraded and the photo-degradation rate increased with increasing H_2O_2 concentration. In addition, Table 2 showed that the photo-degradation percentage of DMP varied from 0 to 81% when the concentration of H_2O_2 varied from 0 to 5.00×10^{-4} mol l^{-1}. This proved that the effect of increasing H_2O_2 concentration is first

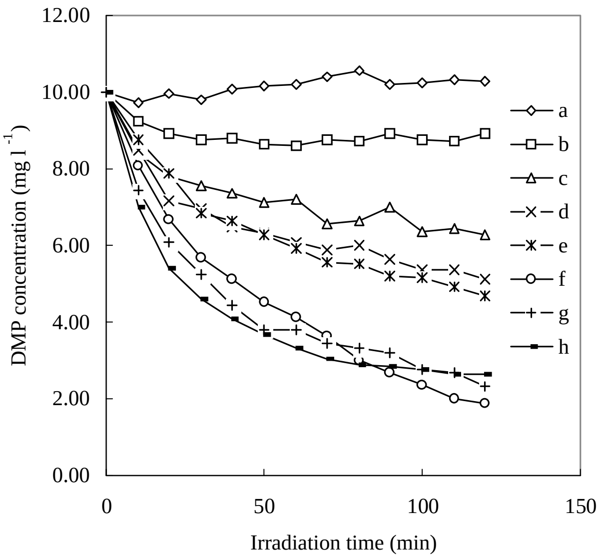

Fig. 2. Photo-degradation of DMP (10 mg l^{-1}) in the solution with Fe^{2+} (1.67×10^{-4} mol l^{-1}) at pH 3.0 under UV light irradiation at different original H_2O_2 concentrations. (a) 0 mol l^{-1}; (b) 6.67×10^{-5} mol l^{-1}; (c) 1.67×10^{-4} mol l^{-1}; (d) 2.33×10^{-4} mol l^{-1}; (e) 3.33×10^{-4} mol l^{-1}; (f) 5.00×10^{-4} mol l^{-1}; (g) 6.67×10^{-4} mol l^{-1}; (h) 8.33×10^{-4} mol l^{-1}.

Table 2
Photo-degradation of DMP in solution with Fe^{2+} (1.67×10^{-4} mol l^{-1}) at pH 3.0 under UV light irradiation at different original H_2O_2 concentrations within 120 min

H_2O_2 concentration (10^{-4} mol l^{-1})	0	0.67	1.67	2.33	3.33	5.00	6.67	8.33
Degradation percentage (%)	0	11	37	49	53	81	77	74

positive for the degradation of DMP because the oxidation power of Fenton reagent was improved with increasing hydroxyl radical amount in solution from the decomposition of increasing H_2O_2. However, with the continuous increase in the original concentration of H_2O_2, especially beyond 5.00×10^{-4} mol l^{-1}, the photo-degradation rate of dimethyl phthalate began to decrease. In the oxidation process of Fenton reagent, hydroxyl radicals were mainly responsible for the degradation of DMP. However, in the excess of hydrogen peroxide and hydroxyl radicals, competitive reactions take place. Hydroxyl radicals are prone to recombination or to reaction according to the following schemes [37–40].

$$^{\bullet}OH + H_2O_2 \rightarrow HO_2^{\bullet} + H_2O \tag{6}$$

$$HO_2^{\bullet} + H_2O_2 \rightarrow {}^{\bullet}OH + H_2O + O_2 \tag{7}$$

$$2HO_2^{\bullet} \rightarrow 2H_2O_2 + O_2 \tag{8}$$

$$HO_2^{\bullet} + {}^{\bullet}OH \rightarrow H_2O + O_2 \tag{9}$$

$$2^{\bullet}OH \rightarrow H_2O_2 \tag{10}$$

From the above Eqs. (6), (9) and (10), it can be found that a part of hydroxyl radicals would be consumed before oxidizing DMP. Therefore, an inhibitory effect for the degradation was produced and the DMP degradation rate and percentage decreased when the concentration of H_2O_2 was higher than 5.00×10^{-4} mol l^{-1}.

The results of Fig. 2 and Table 2 point out the negative effect of a defect and an excess of H_2O_2 on the degradation of DMP. The concentration of the reagent must be enough for the formation of a considerable amount of hydroxyl radicals, but a high concentration is detrimental.

3.3. Effect of original Fe^{2+} concentration on the photo-degradation of dimethyl phthalate

Fe^{2+} acts as photo-catalyst in the photo-degradation reaction of DMP and the original concentration of Fe^{2+} can influence the photo-degradation rate and percentage of DMP. In this study, various original concentrations of Fe^{2+} were investigated to obtain an optimal original concentration of Fe^{2+}. The degradation trends and percentages of DMP with various Fe^{2+} concentrations were showed in Fig. 3 and Table 3, respectively.

From the experimental results shown in Fig. 3 and Table 3, it can be seen that maximum photo-degradation rate and percentage of dimethyl phthalate appeared as the concentration of Fe^{2+} was 1.67×10^{-4} mol l^{-1}. However, the photo-degradation rate and percentage of dimethyl phthalate began to decrease when the concentration of Fe^{2+} was higher or lower than 1.67×10^{-4} mol l^{-1}. It was known that the Fe^{2+} had a catalytic decomposition effect on H_2O_2. When Fe^{2+} concentration increased, the catalytic effect also accordingly increased. When the concentration of Fe^{2+} was higher, a great amount of Fe^{3+} from the process of H_2O_2 decomposition by Fe^{2+} was easy to exit in the form of $Fe(OH)^{2+}$ in acidic environment. Because $Fe(OH)^{2+}$ had a strong absorption for UV light from 290 to 400 nm, the strength of UV light would decrease [41]. Therefore, the decrease in the photo-degradation rate and percentage of dimethyl phthalate would occur.

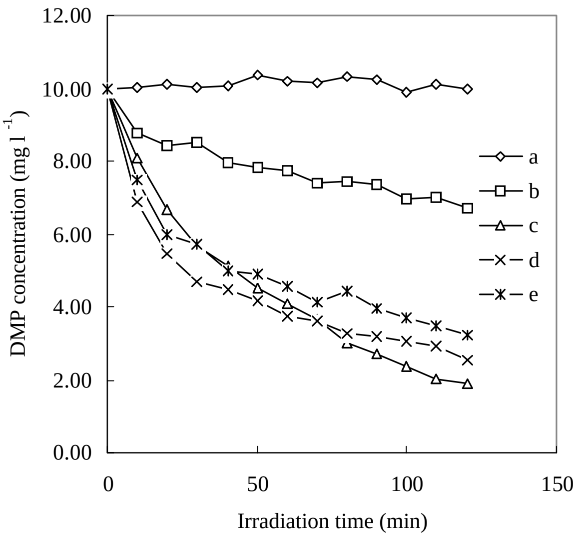

Fig. 3. Photo-degradation of DMP (10 mg l^{-1}) in the solution with H_2O_2 (5.00×10^{-4} mol l^{-1}) at pH 3.0 under UV light irradiation at different original Fe^{2+} concentrations. (a) 0 mol l^{-1}; (b) 8.33×10^{-5} mol l^{-1}; (c) 1.67×10^{-4} mol l^{-1}; (d) 2.50×10^{-4} mol l^{-1}; (e) 3.33×10^{-4} mol l^{-1}.

3.4. Effect of pH on the photo-degradation of dimethyl phthalate

The effect of pH on the photo-degradation of dimethyl phthalate is illustrated in Fig. 4 and the photo-degradation percentage of DMP during 120 min was presented in Table 4.

The pH value can affect the oxidation efficiency of organic matters through directly influencing the generation of hydroxyl radicals. The photo-Fenton reaction is strongly affected by the acidity of solutions. Fig. 4 and Table 4 show that a maximum degradation of 81% was observed in the

Table 3
Photo-degradation of DMP in the solution with H_2O_2 (5.00×10^{-4} mol l^{-1}) at pH 3.0 under UV light irradiation at different original Fe^{2+} concentrations within 120 min

Fe^{2+} concentration (10^{-4} mol l^{-1})	0	0.83	1.67	2.50	3.33
Degradation percentage (%)	0	33	81	75	68

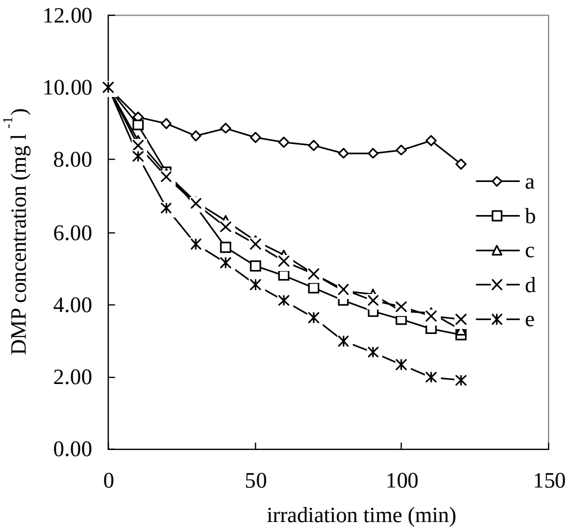

Fig. 4. Photo-degradation of DMP (10 mg l^{-1}) with Fe^{2+} (1.67×10^{-4} mol l^{-1}) and H_2O_2 (5.00×10^{-4} mol l^{-1}) under UV light irradiation at different pH values. (a) 2.0; (b) 4.0; (c) 5.0; (d) 6.00; (e) 3.0.

Table 4
Photo-degradation of DMP in the solution with Fe^{2+} (1.67×10^{-4} mol l^{-1}) and H_2O_2 (5.00×10^{-4} mol l^{-1}) under UV light irradiation at different pH values within 120 min

pH	2.0	3.0	4.0	5.0	6.0
Degradation percentage (%)	21	81	68	67	64

system $UV/H_2O_2/Fe^{2+}$ at pH 3. The degradation decreased at pH values higher than 4, because iron precipitated as hydroxide, which resulted in a reduction in the transmission of the radiation [42]. Additionally, the oxidation potential of hydroxyl radical was known to decrease with increasing pH [43]. At pH 2, DMP was degraded very slowly by Fenton reagent (curve a of Fig. 4) and the degradation percentage was only 1/4 of that at pH 3 (Table 4). At low pH, hydrogen peroxide can stay stable, probably because it solvates a proton to form an oxonium ion (H_3O^+). An oxonium ion makes hydrogen peroxide electrophilic to enhance its stability and presumably to reduce substantially the reactivity with ferrous ion [42]. Therefore, amount of hydroxyl radicals from Eq. (1), would decrease at pH 2 and then low degradation rate and percentage of DMP were reasonably expected.

4. Conclusions

The photo-oxidation of dimenthyl phthalate via Fenton reagents was reported in this study. The results showed that the degradation rate of dimethyl phthalate was greatly accelerated by the photo-Fenton process and the combination of homogeneous systems of $UV/H_2O_2/Fe^{2+}$ produced the highest photochemical degradation percentage (81%) for dimethyl phthalate. The degradation rate and percentage were influenced by the pH of solution and the amount of hydrogen peroxide and iron salt. The optimum conditions for the photo-degradation of dimethyl phthalate in the $UV/H_2O_2/Fe^{2+}$ system were observed at pH 3.0, with an original Fe^{2+} concentration of 1.67×10^{-4} mol l^{-1} and an original H_2O_2 concentration of 5.00×10^{-4} mol l^{-1}.

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