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## Performance of the Persulfate/UV-C Process for the Treatment of Dimethyl Phthalate from Aquatic Environments

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### Abstract

Phthalate esters (PAEs) are used as plasticizers to impart flexibility and resilience to plastic products. In recent years, PAEs are a controversial issue because many phthalates are suspected to be mutagens, hepatotoxic agents and endocrine disruptors, and can lead to adverse effects on organisms even in a low concentration. Recently, sulfate ( $\text{SO}_4^{\bullet-}$ ) radical based advanced oxidation processes have attracted great scientific interest due to their high efficiency in the degradation and mineralization of recalcitrant and/or toxic organic pollutants. In the present study aqueous dimethyl phthalate (DMP;  $100 \text{ mg L}^{-1}$ ), being selected as a model PAE, was treated by the persulfate (PS)/UV-C process at pH 3 and varying PS concentrations (0-60 mM). DMP and TOC abatements increased with increasing PS concentrations from 5 to 40 mM. Further increase in the initial PS concentration, however, reduced both the rate and extent of DMP and TOC removals. The highest pseudo-first-order abatement rate coefficient and electrical energy per order (EE/O) values obtained for DMP treatment with PS/UV-C oxidation were found as  $0.4493 \text{ min}^{-1}$  and  $1.79 \text{ kWh m}^{-3} \text{ order}^{-1}$ , respectively, for PS = 30 mM, pH = 3, DMP =  $100 \text{ mg L}^{-1}$ . The second-order reaction rate coefficient for DMP with  $\text{SO}_4^{\bullet-}$  was determined as  $1.47 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$  by the application of competition kinetics using phenol as the probe compound. Within the scope of the present study, aqueous DMP was also subjected to peroxymonosulfate (PMS)/UV-C and hydrogen peroxide (HP)/UV-C treatments. The performance of PS/UV-C treatment was found to be higher than that of PMS/UV-C and HP/UV-C treatments both in terms of DMP and TOC abatement rates at an initial oxidant concentration of 5 mM.

**Keywords:** Dimethyl phthalate, Endocrine disrupting compound, Reaction kinetics, Sulfate radical.

### Introduction

Phthalate esters (PAEs) are ubiquitous pollutants in the environment, due to their widespread use for around 50 years. The phthalates are mainly used as additives in plastics, especially in plasticized polyvinyl chloride (PVC), as well as in the production of paints and varnishes, adhesives, lubricants and cosmetics. The most widely used representative PAEs is bis(2-ethylhexyl) phthalate (DEHP) (Clara et al., 2010), which is the dominant plasticizer used in PVC production. Another common PAE is Benzyl butyl phthalate (BBP), which is also used in the manufacture of foamed PVC. The PAEs with relatively short alkyl chains such as dimethyl phthalate (DMP), diethyl phthalate (DEP), and di-n-butyl phthalate (DBP) are used as solvents in perfumes and pesticides (Oliver et al., 2005). Since PAEs are not covalently bonded to the polymeric matrix they are easily leached

into the environment during the process of plastic aging and decomposition (Dargnat et al., 2009). In addition, these pollutants are refractory to biodegradation in the environment by microorganisms, leading to their widespread presence in the environment (Abdel daiem et al., 2012). Therefore, due to the high consumption, the continuous release to the environment and their resistance to microbial decomposition, many PAEs are found to be ubiquitously present in the environment, including rainwater/storm-water, surface water, sediments, and soils (Kolpin et al., 2002; Fierens et al., 2012; Kang et al., 2012). PAEs are known to be hazardous chemicals to human health since they have been associated to birth defects, organ damage, infertility and cancer. They are also known to be among the endocrine disrupting compounds present in water (Venkata et al., 2007).

PAEs cannot be removed effectively by conventional water/wastewater treatment processes. PAEs with long alkyl-chains are poorly degraded and some of them are considered recalcitrant to biological degradation (Bauer et al., 1998). It is necessary to develop alternative treatment technologies that can effectively remove PAEs from waters and wastewaters. Advanced oxidation processes (AOPs) using highly reactive radicals such as hydroxyl radicals ( $\text{HO}^\bullet$ ) are applied for the treatment of PAEs (Xua et al., 2007; Zhou et al., 2007; Yuan et al., 2008; Ölmez-Hancı et al., 2009). Sulfate radical ( $\text{SO}_4^{\bullet-}$ )-based AOPs, in which persulfate (PS) or peroxymonosulfate (PMS) are used as oxidants, came forth recently for the degradation of toxic and/or refractory pollutants. Previous studies showed that the  $\text{SO}_4^{\bullet-}$  can be generated by the activation of PS or PMS using transition metal ions, thermal energy, UV-C irradiation or other activators (Criquet and Leitner, 2009). Activation of PS under UV-C radiation results in the formation of two  $\text{SO}_4^{\bullet-}$  (Equation (1)) through the homolytic cleavage of the peroxide (-O-O-) bond (Baxendale and Wilson, 1957; Mark et al., 1990; Anipsitakis and Dionysiou, 2004);



$\text{SO}_4^{\bullet-}$  is very reactive due to its high reduction potential ( $E_0 = 2.5\text{-}3.1$  eV), leading to a great potential for degrading organic compounds in wastewater (Neta et al., 1988). Therefore, the PS/UV-C process has been confirmed to be effective in degrading organic contaminants (Anipsitakis and Dionysiou, 2004; He et al., 2013; Yang et al., 2016).

This study was undertaken to investigate the treatability of DMP, which was selected as the target PAE because of its frequent detection in the aquatic environment, by the PS/UV-C process. The effect of initial PS concentration on DMP and its total organic carbon (TOC) removal rates was explored. The second-order reaction rate coefficient of DMP with  $\text{SO}_4^{\bullet-}$  was determined by employing competitive kinetics by using phenol as the reference compound. Finally, treatment performance of the PS/UV-C process was compared with the PMS/UV-C and hydrogen peroxide (HP)/UV-C oxidation processes (two known UV-driven AOPs) both in terms of DMP and TOC abatements.

## Materials and Methods

### Materials

DMP (Sigma-Aldrich, Germany;  $\text{C}_{10}\text{H}_{10}\text{O}_4$ ; purity: >99%) and phenol (Merck, Germany;  $\text{C}_6\text{H}_6\text{O}$ ; purity: >99%) were used as received. Potassium persulfate (PS,  $\text{K}_2\text{S}_2\text{O}_8$ , >99.5%) and potassium peroxymonosulfate (PMS,  $2\text{KHSO}_5 \cdot 3\text{KHSO}_4 \cdot 3\text{K}_2\text{SO}_4$  available as Oxone<sup>®</sup>,  $\geq 99.5\%$ ) were purchased from Sigma-Aldrich, (Germany),  $\text{H}_2\text{O}_2$  (HP, 35%, w/w) from Merck (Germany). Aqueous DMP solutions were prepared in distilled water (Arium 61316RO, Sartorius AG, Germany), whereas for the preparation of the high performance liquid chromatography (HPLC) mobile phase and standard HPLC calibration solutions doubly distilled water (Arium 611UV system, Sartorius AG, Germany) was used. All other reagents and solvents were of at least analytical grade and obtained from Merck (Germany), Fluka (USA) or Sigma-Aldrich (USA).

### Experimental Procedures

PS/UV-C, PMS/UV-C and HP/UV-C treatment experiments were conducted in a 1.9 L capacity, cylindrical stainless-steel photoreactor (length = 95 cm; diameter = 6 cm) at room temperature ( $25 \pm 2^\circ\text{C}$ ). The UV-C photoreactor setup featuring the UV-C light source ( $\lambda_{\text{max}} = 253.7$  nm) and the procedure of a typical experimental run were previously described elsewhere in more detail (Ölmez-Hancı et al., 2011). The batch-operated photoreactor was mixed by means of a peristaltic pump at a flow rate of  $80 \text{ mL min}^{-1}$ . Samples were taken at regular time intervals for up to 60 min and analyzed for DMP, TOC and pH. The second-order rate coefficient for the reaction of DMP with  $\text{SO}_4^{\bullet-}$  was determined by employing the competitive kinetics h. For this purpose a reference pollutant (phenol) for which the second-order rate coefficient with  $\text{SO}_4^{\bullet-}$  is known, was added to the reaction solution at equimolar concentration (0.515 mM) of the model pollutant (DMP;  $100 \text{ mg L}^{-1}$ , i.e. 0.515 mM).

### Analytical Procedures

DMP concentrations were measured with high-performance liquid chromatography (HPLC, Agilent 1100 Series, USA) equipped with a Diode-Array Detector (DAD) (G1315A, Agilent Series) on an Atlantis C18 ( $3.9 \times 150$  mm,  $5 \mu\text{m}$ , Waters) column. The mobile phase was

acetonitrile-water (40:60, v:v) at a flow rate of 1 mL min<sup>-1</sup>. Quantitative analysis was monitored at 220 nm. The column temperature was set at 30°C during the measurements. The quantification limit for DMP was 1 µg/mL, using a 20µL injection volume. The mobile phase used for phenol quantification consisted of water-methanol-acetic acid (79.2:19.8:1.0; v:v:v) at a flow rate of 0.8 mL min<sup>-1</sup>. Detection was performed at 270 nm. The column temperature and injection volume were set as 30°C and 40 µL, respectively. The quantification limit for phenol was determined as 1.5 mg L<sup>-1</sup> for the method described above. TOC was monitored on a Shimadzu V<sub>CPN</sub> model carbon analyzer. The detection and quantification limits were 50 µg L<sup>-1</sup> and 0.5 mg L<sup>-1</sup>, respectively. The pH was monitored using an Orion 720A+ model pH-meter. All measurements were done in duplicate and arithmetic averages were taken throughout the data analyses and calculations.

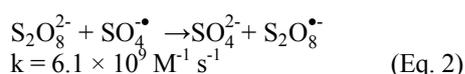
## Results and Discussion

### Effect of Initial Persulfate Concentration

In order to obtain the optimum PS concentration to treat 100 mg L<sup>-1</sup> aqueous DMP solutions by employing the PS/UV-C treatment process, a set of experiments was conducted at varying PS concentrations (0-60 mM) and at pH 3. Figure 1 delineates DMP (a) and TOC (b) removal efficiencies obtained during 60 min PMS/UV-C oxidation. In addition to the PS/UV-C process, the degradation of DMP with PS oxidation (in the absence of UV-C light) and UV-C photolysis (in the absence of PS) were also examined and the obtained results included in Figure 1 (a) and (b). Experimental results conducted with direct UV-C photolysis resulted in 14% DMP abatement at pH 3 implying that the self-photolytic degradation of DMP was not significant under UV-C irradiation. Practically no removal (≤5%) was obtained by the oxidation of DMP at 30 mM initial PS concentration. TOC removal efficiencies for PS oxidation and UV-C photolysis were also practically negligible within this time period (≤5%).

As it is evident in Figure 1, PS addition greatly enhanced DMP and TOC abatements as compared to UV-C photolysis which demonstrated that SO<sub>4</sub><sup>•-</sup> being generated by UV-C photolysis of PS was mainly responsible for the degradation and mineralization of DMP during PS/UV-C treatment. As can be seen from

Figure 1 (a), DMP removal was always complete at all studied PS concentrations. Complete DMP removals were achieved with the PS/UV-C process after 40 min (5 mM PS), 30 min (10 mM PS), 15 min (20, 30 and 40 mM PS) and 20 min (60 mM PS). The DMP degradation and mineralization increased gradually as the initial concentration of PS increased from 5 mM to 30 mM. For example, the removal of DMP at 5 min increased from 35% with 5 mM to 83% with 30 mM PS at initial pH 3. However, further increase in PS concentration to 40 and 60 mM, decreased the degradation rate of DMP. This is thought to be a consequence of the well-known free radical scavenging effect of excessive PS concentrations (Criquet and Leitner, 2009);



The optimum PS concentration for DMP degradation was 30 mM. Increasing the initial PS concentration also had a positive effect on TOC removal efficiencies; overall TOC removals increased from 38% to 100% when 5 mM and 60 mM PS was used, respectively.

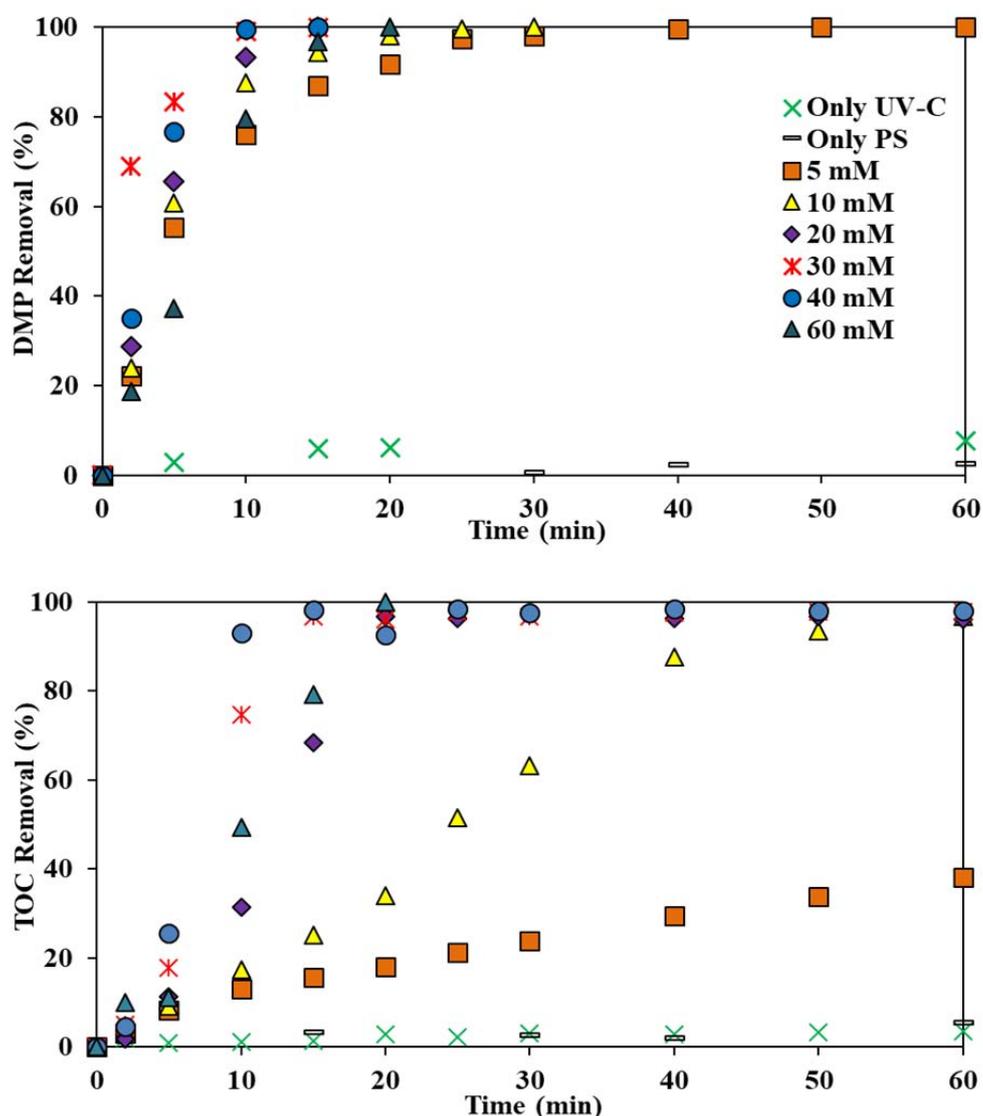
The degradation of DMP with the PS/UV-C process followed pseudo-first-order kinetics ( $d[\text{DMP}] / dt = k \times [\text{DMP}]$ ) with respect to DMP concentration. Results of the PS/UV-C treatment experiments indicated that the highest DMP removal rate coefficient ( $k_{\text{DMP}} = 0.4493 \text{ min}^{-1}$ ) was achieved at a PS concentration of 30 mM within the studied PS concentration range (Table 1). Further increase of the PS concentration reduced the DMP removal rates to  $0.2957 \text{ min}^{-1}$  and  $0.2261 \text{ min}^{-1}$  for 40 mM and 60 mM PS concentrations, respectively.

**Table 1.** Effect of initial PS concentration on DMP removal rates (DMP = 100 mg L<sup>-1</sup>; TOC = 60 mg L<sup>-1</sup>; pH = 3).

Initial PS (mM)	$k_{\text{DMP}} (\text{min}^{-1})$
5	0.0715
10	0.2165
20	0.2740
30	0.4493
40	0.2957
60	0.2261

Besides kinetics, a more comprehensive way to compare the effectiveness of UV-based technologies has been adopted by the

Photochemistry Commission of the International Union of Pure Chemistry; namely the electrical energy per order (EE/O; Bolton et al., 2001).



**Fig 1.** Effect of initial PS concentration on DMP (a) and TOC (b) removal efficiencies obtained during PS/UV-C treatment (DMP = 100 mg L<sup>-1</sup>; TOC = 60 mg L<sup>-1</sup>; pH = 3).

EE/O measures the electrical energy (kWh) required to reduce the concentration of a contaminant by an order of magnitude in a 1 m<sup>3</sup> water or wastewater and is described by the following equation:

$$EE/O = 38.4 \times P / (V \times k) \quad (\text{Eq. 3})$$

where P is the lamp power (kW), V is the reactor volume (L), and k is the pseudo-first-order rate constant of the compound degradation (min<sup>-1</sup>).

The EE/O values for DMP (100 mg L<sup>-1</sup>) degradation with PS/UV-C treatment at various initial PS concentrations were calculated and shown in Figure 2 with the smallest EE/O value representing the more energy efficient treatment condition. For the optimum initial PS concentration of 30 mM, the minimum EE/O value for DMP degradation was calculated as 1.79 kWh m<sup>-3</sup> order<sup>-1</sup>. From Figure 2 it can be

concluded that applying an optimum PS concentration can minimize the EE/O value.

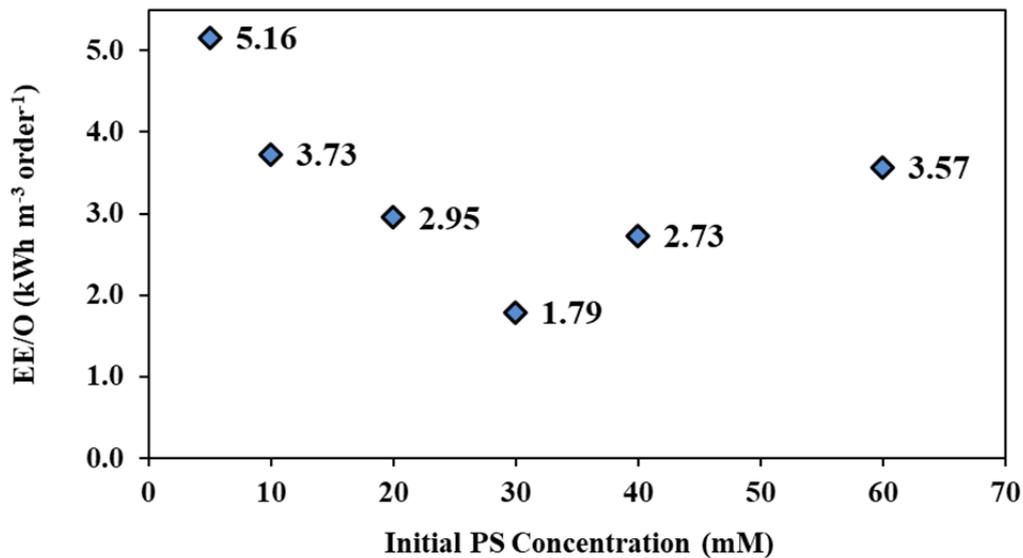
*Determination of the second-order reaction rate constant of DMP with SO<sub>4</sub><sup>•-</sup>*

The competition kinetic studies were carried out to determine the second-order reaction rate constant of DMP with SO<sub>4</sub><sup>•-</sup> using phenol whose second-order reaction rate coefficient with SO<sub>4</sub><sup>•-</sup> is known ( $k_{\text{SO}_4^{\bullet-}, \text{phenol}} = 8.8 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$ ) as the reference compound (Lindsey and Tarr, 2000; Liang and Su, 2009). The following expression

was applied for the estimation of the second-order reaction rate coefficient;

$$\ln [\text{DMP}]_o / [\text{DMP}]_t = k_{\text{SO}_4^{\bullet-}, \text{DMP}} / k_{\text{SO}_4^{\bullet-}, \text{phenol}} \times \ln [\text{phenol}]_o / [\text{phenol}]_t \quad (\text{Eq. 4})$$

where  $k_{\text{SO}_4^{\bullet-}, \text{DMP}}$  and  $k_{\text{SO}_4^{\bullet-}, \text{phenol}}$  are second-order reaction rate constants (in  $\text{M}^{-1} \text{ s}^{-1}$ ) for the reactions between the DMP (target compound) and phenol (reference compound) with SO<sub>4</sub><sup>•-</sup>, respectively.



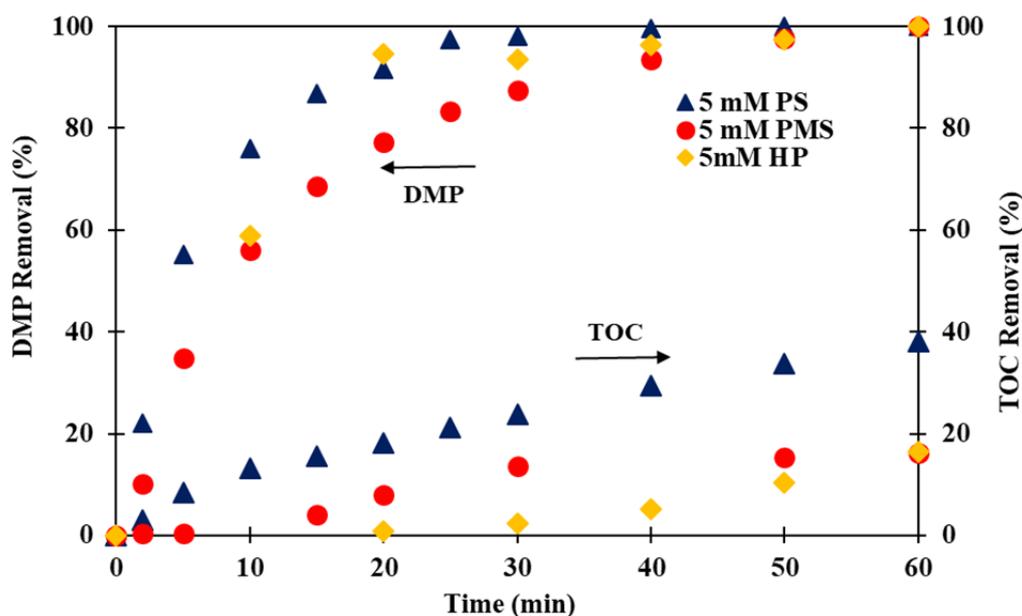
**Fig 2.** The calculated EE/O values for DMP degradation by the PS/UV-C treatment (DMP = 100 mg L<sup>-1</sup>; TOC = 60 mg L<sup>-1</sup>; pH = 3)

The second-order rate constant between DMP and SO<sub>4</sub><sup>•-</sup> was calculated as  $1.47 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$  by using the first-order rate constants determined for DMP (0.0192 min<sup>-1</sup>) and phenol (0.1147 min<sup>-1</sup>) as well as  $k_{\text{SO}_4^{\bullet-}, \text{phenol}} (= 8.8 \times 10^9 \text{ M}^{-1} \text{ s}^{-1})$ .

*Comparison of PS/UV-C with PMS/UV-C and HP/UV-C treatment processes*

To compare the performance of PS/UV-C with PMS/UV-C and HP/UV-C processes, the experiments were carried out at initial DMP concentration of 100 mg L<sup>-1</sup> and pH 3, with an initial oxidant concentration of 5 mM. The degradation efficiency of DMP with the PS/UV-C process was 76% after 10 min, which was much higher than 59% and 56% obtained for PMS/UV-C and HP/UV-C treatments, respectively (Figure 3).

DMP was completely degraded after 40 min treatment with the PS/UV-C process, whereas 100% DMP removal was realized for PMS/UV-C and HP/UV-C treatments after 60 min. As it is evident from Figure 3, after 60 min treatment, the highest TOC removal efficiency was obtained as 38% for PS/UV-C oxidation, whereas only 27% TOC removal was achieved during PMS/UV-C and HP/UV-C treatments. The  $k_{\text{DMP}}$  values of the investigated treatment processes exhibited the following decreasing order; PS/UV-C (0.1566 min<sup>-1</sup>) > HP/UV-C (0.0765 min<sup>-1</sup>) ≈ PMS/UV-C (0.0715 min<sup>-1</sup>) at an initial oxidant concentration of 5 mM, indicating that the PS/UV-C treatment process was superior over HP/UV-C and PMS/UV-C treatments on the basis of DMP removal kinetics and TOC removal efficiencies.



**Fig 3.** Comparison of DMP and TOC removal efficiencies obtained during PS/UV-C, PMS/UV-C and HP/UV-C treatments (DMP = 100 mg L<sup>-1</sup>; TOC = 60 mg L<sup>-1</sup>; PS = PMS = HP = 5 mM; pH = 3).

### Conclusions

In the present study, the treatment performance of the persulfate (PS)/UV-C process for the degradation of dimethyl phthalate (DMP), being selected as a model phthalate ester and micropollutant, was evaluated at an initial DMP concentration of 100 mg L<sup>-1</sup> (0.515 mM) and an initial pH of 3. The effect of initial PS concentration on DMP and total organic carbon (TOC) removals rates was examined. The following conclusions could be drawn from the present work;

- The PS/UV-C process was found to be effective in the degradation and mineralization of DMP. DMP degradation was rapid and complete within 10-40 min treatment time depending on the initial PS oxidant concentration (0-60 mM). Increasing the initial PS concentration resulted in a significant enhancement of TOC removal; over 90% TOC removals could be achieved at elevated PS concentrations ( $\geq 10$  mM).
- Pseudo-first-order DMP degradation rate constants increased with increasing the initial PS concentration and decreased above optimum value. The highest pseudo-first-order DMP degradation rate constant was found as 0.4495 min<sup>-1</sup> for 30 mM PS.

- The EE/O for complete DMP degradation was calculated as 1.79 kWh m<sup>-3</sup> order<sup>-1</sup> for the optimum initial PS concentration of 30 mM.
- The results of the competitive kinetics experiments, where phenol was used as the probe reference compound, showed that DMP reacted rapidly with SO<sub>4</sub><sup>•-</sup>, with the second-order rate constant of 1.47 × 10<sup>9</sup> M<sup>-1</sup> s<sup>-1</sup>.
- The PS/UV-C process was found to be more effective for not only DMP removal but also DMP mineralization than the PMS/UV-C and HP/UV-C processes at an initial oxidant concentration of 5 mM and at pH 3.

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