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Data Article

Data for persulfate activation by UV light to degrade theophylline in a water effluent



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ABSTRACT

The aim of this study was to degrade theophylline (TP) drug in a pharmaceutical effluent solution utilizing persulfate (PS). A simulated and a real effluent solution were used, with different conditions tested to optimize the degradation process. HPLC analyses and a modified-HPLC method were used to track concentrations of TP and PS respectively in the treatment process. Experiments were done in triplicates and treated data is presented as graphs. A detailed analyses of this study can be found in the article "Degradation of theophylline in a UV₂₅₄/PS system: matrix effect and application to a factory effluent" [1] published in Chemical Engineering Journal.

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Specifications Table

Subject	Water Science and Technology
Specific subject area	water treatment, AOPs, UV ₂₅₄ , persulfate
Type of data	Table
	Figure
How data were acquired	High performance liquid chromatography (HPLC, Agilent 1100 series)
Data format	Raw and Analyzed
Parameters for data collection	Experiments were done in triplicates in separate reactors. Room temperature was maintained throughout the reactions.
Description of data collection	HPLC coupled to DAD was used to track TP concentrations throughout the reaction. A modified-HPLC method utilizing KI was used to track PS concentrations. The average and error bars were obtained for each data point presented in the graph.
Data source location	American University of Beirut
	Beirut
	Lebanon
Data accessibility	Within the article
Related research article	Author's name
	Suha Al Hakim, Saly Jaber, Nagham Zein Eddine, Abbas Baalbaki, Antoine Ghauch*
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	Title
	Degradation of theophylline in a UV_{254}/PS system: matrix effect and application to a factory effluent [1]
	Journal
	Chemical Engineering Journal DOI https://doi.org/10.1016/j.cej.2019.122478

Value of the Data

• Data related to treatment of a pharmaceutical effluent solution using a UV/PS system is presented.

- Researchers involved in advanced oxidation processes and treatment of pharmaceuticals in water can benefit from the following data.
- Presented data can be utilized to develop a continuous treatment system for pharmaceutical effluents by taking into consideration the effects of spiking the reactor with the oxidant or the pharmaceutical. Additionally, effect of phosphates and comparison between two different oxidants provide useful information in this process.
- The homemade reactor's setup can be utilized by other interested researchers in a study where similar commercially available UV lamps are used and system cooling is required.

1. Data

Calibration curve and some properties for TP and presented in Fig. 1.

The observed rate constants, obtained for pseudo-first order fitting, for degradation of TP at different [PS]₀ is presented in Table 1.

A mini review of effects of chlorides and bicarbonates on the degradation of different organic probes in PS-based AOPs are presented in Table 2 and Table 3 respectively.

TP degradation in UV/PS system in the presence of phosphates (Fig. 2) and chloroform (Fig. 4) is studied in a simulated pharmaceutical effluent with $[TP]_0 = 10 \text{ mg } \text{L}^{-1}$ and $[PS]_0 = 0.25 \text{ mM}$. Comparison between UV and base activation of PS is studied with 10 mM phosphate buffer at pH = 11 used (Fig. 3). Additionally, H₂O₂ and PS oxidants are compared (Fig. 5).

A real effluent solution containing $[TP]_0 \approx 160 \text{ mg L}^{-1}$ is treated with simultaneous and successive additions of PS (Fig. 6a). The UV₂₅₄/PS system was studied upon spiking the reactor with varying amounts of concentrated effluent solution (Fig. 6b).

The raw data corresponding to all figures and tables can be found in the file "raw data" within this article. These data include concentrations of TP and PS and the standard deviation for their measurement.



Fig. 1. (a) Calibration curve of TP obtained using HPLC/DAD at wavelength of 270 nm, (b) LINEST output for TP calibration curve, and (c) selected TP characteristics [2].

Table 1 Degradation of [TP]0 = 10 mg L-1 at [PS]0 = 0.01-0.5 mM. kobs and linearity constant (R2) obtained for plots of $ln \frac{[TP]t}{[TP]_0}$ versus time (min) for tested conditions upon first order fitting are presented.

[TP] ppm	[PS] ₀ mM	$k_{obs} \ge 10^{-1} (min^{-1})$	R ²
10	0.01	0.04 (±0.01)	0.9675
	0.1	$0.49(\pm 0.02)$	0.9553
	0.25	1.74 (±0.06)	0.9933
	0.5	3.7 (±0.2)	0.955

2. Experimental design, materials, and methods

2.1. Experimental setup: UV reactors

A bench-scale experimental setup was used simulating the commercial UV water sterilization apparatus. Six units of a generic 1 gallon per minute UV water disinfection element (Fig. 7a) were used in the construction of the experimental setup. The commercial units utilized originally consist of a UV 11-W low-pressure mercury lamp (LPHgL) (Philips TUV 11W G11 T5, Poland), fitted in a quartz tube

Table 2

Review of the effect of chloride (Cl^{-}) presence on the degradation of several organic probes in Persulfate activated systems. (+) and (-) represent an increase or a decrease in the degradation rate constant of the oxidation reaction, respectively.

Family	Probe	Structure	PS system used	[PS] ₀ mM	[Cl ⁻] (mM)	[NaCl] (mg L ⁻¹)	Effect on k _{obs}	Main reactive radical species	Reference
Antibiotic	Florfenicol	$H_{0}C^{-K} \left(\begin{array}{c} \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	UV/PS	1	1, 10, 100	58.4, 584, 5844	_	No data	[3]
	Sulfamethazine	Se N	Heat/PS	2	5, 10	292, 584	Negligible effect	$SO_4^{\bullet-}$	[4]
		H ₂ N H			100, 200	5844, 11688	+		
	Penicillin G	H0-20	Heat/PS	0.5	0.171 0.0943	10 5.51	_	$SO_4^{\bullet-}$	[5]
Antipyretic	Acetaminophen		Fe ²⁺ /PS	0.8	0.1	5.84	Negligible effect	$SO_4^{\bullet-}$	[6]
		но			2	116.9	_		
				0 1 2 2	5, 10	292, 584	+	s0•-	[7]
			07/25	0.152	2, 10, 20	117, 584, 1169	+	30 ₄	[7]
					50, 100	2922, 5844	-		
	Antipyrine	N.	Heat/PS	1.855	18.5	1081	+	$SO_4^{\bullet-}$	[8]
		L'N- C			1850	108114	_		
Beta blocker	Atenolol	о он Ц снз	Heat/PS	0.5	1, 5, 10, 50	58.4, 292, 584, 2922	Negligible	$SO_4^{\bullet-}$	[9]
		H ₂ N CH ₃				507, 2522	cheet		
	Bisoprolol	OH H	Heat/PS	1	0.0855	5	+	$SO_4^{\bullet-}$, OH^{\bullet}	[10]
		Lo~o							

Hormone	17β-estradiol	HO HHHHHHHHHHHHHHHHHHH	UV/PS	Slow-release flow through system	0.028, 0.282, 2.82 28.2, 42.3 56.4, 564, 846, 1269	1.6, 16.5, 165 1648, 2472 3256, 32960, 49440, 74160	- + -	<i>SO</i> ^{€−} ₄	[11]
Nonsteroidal anti-inflammatory	Diclofenac	CI CI CI	UV/PS	1	25, 50 100, 200	1461, 2922 5844, 11688	+ _	No data	[12]
	Ketoprofen	CH3 OH	Heat/PS	1	3.42 34.2 342	200	+	No data	[13]
		\bigcup \bigcup \downarrow	UV/PS	0.1	3.42	2000, 20000	+		
					34.2, 342	2000, 20000	_		
			Fe ²⁺ /PS	0.5	3.42, 34.2, 342	200, 2000, 2000,	-		
Persistent organic molecules	Cyclohexanoic acid	С	UV/PS	2	14.1	824	-	ОН•	[14]
Pesticides	1,1,1-Trichloroethane	CI	Heat/PS	15	1	58.4	Negligible	No data	[15]
		CI			10, 100	584.4, 5844	_		
	Triclosan	CI OH	Heat/PS	0.155	0.15, 1, 10 20, 50	8.8, 58.4, 584.4 1169, 2922	- +	$SO_4^{\bullet-}$	[16]
	Diatrizoate	о _{у∕} он	UV/PS	12	1, 10, 100	58.4, 584.4,	Negligible	$SO_4^{\bullet-}$	[17]
		o 1/1/ o				5844	effect	×	
					500	29220	_		

Table 3

Review of the effect of bicarbonate (HCO_3^-) presence on the degradation of several organic probes in Persulfate activated systems. (+) and (-) represent an increase or a decrease in the degradation rate constant of the oxidation reaction, respectively.

Family	Probe	Structure	PS system used	[PS] ₀ mM	$[HCO_3^-]$ (mM)	Effect on kobs	Main reactive radical species	Reference
Antibiotic	Florfenicol	H ₅ C ⁻ OH N CI	UV/PS	1	1, 10, 100	_	No data	[3]
	Sulfamethazine	H _N N C H N	Heat/PS	2	5, 10, 50	+	SO4-	[4]
	Penicillin G		Heat/PS	0.5	0.0943	_	SO4-	[5]
Antipyretic	Acetaminophen	HO	UV/PS	0.132	2 10, 20, 50, 100	- +	<i>SO</i> ^{•-} ₄	[7]
	Antipyrine		Heat/PS	1.855	18.55, 92.75, 185.5, 927.5	_	SO4 ⁻	[8]
Beta blocker	Atenolol	H _N N CH ₃	Heat/PS	0.5	1, 5, 10, 50	-	<i>SO</i> ^{•-} ₄	[9]
Hormone	17β-estradiol	HO HH H	UV/PS	Slow-release flow through system	4.76 11.9, 23.8	+ -	SO4-	[11]

Nonsteroidal anti-inflammatory	Diclofenac	CI CI CI OH	UV/PS	1	25, 50, 100 and 200	+	No data	[12]
	Ketoprofen	C C C C C C C C C C C C C C C C C C C	Heat/PS	1	1, 50, 100 mM	-	No data	[13]
Persistent organic molecules Pesticide	1,1,1-Trichloroethane		Heat/PS	15	1, 10, 100	-	No data	[15]
	Triclosan	CI OH	Heat/PS	0.155	1–50	_	<i>S0</i> ^{•-} ₄	[16]
	Diatrizoate	O OH	UV/PS	12	2.5, 5, 10, 20, 40	+	SO4-	[17]



Fig. 2. (a) Effect of phosphates (0, 10 and 20 mM) on TP degradation. (b) pH in buffered and non-buffered solutions. Experimental conditions: $[TP]_0 = 10 \text{ mg } L^{-1}$, $[PS]_0 = 0.25 \text{ mM}$. Error bars are calculated as $\frac{t}{\sqrt{m}}$, where absent bars fall within the symbols.



Fig. 3. Comparison between UV and base activation of PS. Experimental conditions: $[TP]_0 = 10 \text{ mg } L^{-1}$, $[PS]_0 = 0.25 \text{ mM}$, and [phosphate buffer] = 10 mM of pH = 11 in both cases. Error bars are calculated as $\frac{ds}{\sqrt{n}}$, where absent bars fall within the symbols.

placed in a 280 mL 316 stainless steel casing that continuously intakes pre-filtered water. The UV lamps and their quartz fittings were utilized from the commercial product, and the stainless-steel casing was replaced by a 400 mL home-made borosilicate cylinder. The borosilicate used in the setup is as inert as the stainless-steel used in the commercial product, however the borosilicate allows easy handling and real-time monitoring of reactors to assure no precipitates were formed. Our developed system (Fig. 7b) utilizes a temperature-controlled water bath (20 °C) by the aid of a PolyScience MM7 chiller. UV-254 nm intensity was 3.2 mWcm⁻² \pm 0.16 measured using a radiometer (Model UVC-254 Lutron; Taipei, Taiwan) at a distance of 3cm, which is similar to the distance between the lamp and the termination part of the solution treated in the designed system. All experiments were done in triplicates, where two different experiments could be done at once.



Fig. 4. TP degradation in presence and absence of chloroform used as superoxide radical quencher. Solution is saturated with chloroform at 20 °C. Experimental conditions: $[TP]_0 = 10 \text{ mg L}^{-1}$ and $[PS]_0 = 0.25 \text{ mM}$.



Fig. 5. Comparison of oxidation effectivity of H_2O_2 and PS toward TP. (a) [TP]/[TP]₀ for three different tested systems and (b) the corresponding pH at initial (t = 0 min) and final time (t = 16 min) with the calculated k_{obs}. Experimental conditions: [TP]₀ = 10 mg L⁻¹, [PS]₀ = 0.25 mM, [H₂O₂]₀ = 0.25 mM and [PS]₀ = [H₂O₂]₀ = 0.125 mM for the case of mixed oxidants.

2.2. Experimental procedure

Stock solution of TP (0.555 mM) was prepared by dissolving 100 mg of dry TP powder in one liter of DI water, and that of Persulfate (PS) (100 mM) was prepared by dissolving 2.38 g of sodium persulfate in 100 mL of DI water. Both solutions were stored in dark at 4 °C for a maximum period of two weeks. For persulfate detection method potassium iodide solution (40 mM) was prepared by dissolving 6.64 g of KI and 5 g of NaHCO₃ in 1 L volumetric flask and left to stir overnight. Before every experiment LPHgLs (11 Watts) were stabilized for a period of 60 min. Monthly routine check of the LPHgL's UV-254 nm intensity was performed using a UVC radiometer (UVC-254 Lutron, Taiwan) to confirm that the power of the lamps is within \pm 10% the original value, otherwise the lamps were replaced by new ones. The water chiller was turned on and set at 20 °C 60 minutes ahead of all experiments. Temperature of water in reaction medium was measured several times during the experiment to make sure it is within a range of 20 \pm 2 °C. The order of addition of reagents was as follows: first Theophylline solution from stock was placed in the reactor, then DI was added, followed by additive matrix solutions (if any), finally, PS or H₂O₂, from stock solutions were added. Samples were withdrawn every 2 min using a



Fig. 6. Degradation of TP in a real pharmaceutical effluent sample in UV254/PS system. (a) PS is added either successively over 5 cycles ([PS] = 5 mM/cycle) or simultaneously at t = 0 min ([PS] = 25 mM) to TP effluent solution ($[TP]0 \approx 160 \text{ mg L-1}$). (b) Concentrated effluent solution spiked over 3 cycles of 1 h each starting at $[TP]0 \approx 160 \text{ mg L-1}$ and [PS]0 = 25 mM added at t = 0 min. The insets correspond to the average % RSE calculated for every cycle. Error bars are calculated as $\frac{ts}{\sqrt{n}}$, where absent bars fall within the symbols.



Fig. 7. (a) Generic 1 gallon per minute UV continuous water disinfection element, and (b) an exploded diagram of the experimental setup.



Fig. 8. Wastewater samples containing TP collected from a local pharmaceutical production plant. (a) Washing of 1000 L 316 SS L mixing container, (b) collecting wastewater samples in amber bottles, and (c) filter press used in the manufacturing process.

separate syringe for each reactor and placed in 2 mL HPLC vials after filtration by a 0.45 µm PTFE 13 mm disc filters. Wastewater used in experiments was pre-filtered using a 1 µm ashless glass fiber filter. For the case of real pharmaceutical factory effluent, water samples were collected from washing the reactors used in the production process (Fig. 8).

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104614.

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