

Efficient photolytic degradation of disinfection by-products by using a high photon flux UV system: monochloroacetic acid, dichloroacetic acid and trichloroacetic acid

Ting Li, Qian Zhang, Chaolin Li, Weidang Ai and Liangchang Zhang

ABSTRACT

Photon UV irradiation is promising for organic pollutant decomposition, such as disinfection by-products (DBPs). However, due to the photostability and high water solubility, chloroacetic acids (CAAs) decomposition using routine UV photolysis is very slow. The present study employed a high photon flux UV ($3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$) system to investigate its feasibility and suitability for enhancing CAAs (MCAA, DCAA and TCAA) decomposition. The results showed that increasing UV photon flux accelerated the photolysis remarkably. Under the condition of high UV photon flux $3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$, almost complete degradation of 20 mg L^{-1} MCAA, 20 mg L^{-1} DCAA, and 20 mg L^{-1} TCAA in a mixed solution can be achieved within 50, 30 and 25 min, respectively. To the best of our knowledge, efficient photolytic degradation of CAAs in such short time has not been reported. The pseudo-first-order rate constant (k_{obs}) steadily increases with the increasing of UV intensity, indicating that the utilization of light energy is efficient. In addition, the variation of pH from 3.2 to 9.0 showed minor effect on CAAs decomposition in this present studied system. The outcome of this study would be helpful for future employment of high photon flux UV systems for those photolytic resistant pollutants' decomposition.

Key words | dichloroacetic acid, high photon flux UV irradiation, monochloroacetic acid, photolysis, trichloroacetic acid

Ting Li
Qian Zhang (corresponding author)
Chaolin Li
 Environmental Science and Engineering Research Center, Shenzhen Graduate School, Harbin Institute of Technology, Shenzhen 518055, China
 E-mail: zhangqianhiter@126.com

Ting Li
Weidang Ai
Liangchang Zhang
 Space Science and Technology Institute of South China, Shenzhen 518117, China

Qian Zhang
 Postdoctor Innovation and Practice Base, Public Service Bureau of Longgang District, Shenzhen 518117, China

Weidang Ai
Liangchang Zhang
 China Astronaut Research and Training Center, Beijing 100094, China

INTRODUCTION

Disinfection is a major stage for conventional drinking water treatment, due to its excellent performance in pathogen control to ensure drinking water safety. Disinfection by-products (DBPs) are an unintentional result of water treatment, which are generated by the reaction between the disinfectant (chlorine) and the natural organic or inorganic matter present in raw water (In-Young *et al.* 2014; Lee & Jin 2014). Chloroacetic acids (CAAs), as a class of typical DBPs, are detected in chlorinated drinking water or recycled wastewater, in which one or more halogen atoms replace hydrogen atoms from the α -carbon in

acetic acid where the electronegative halogens enhance the stabilizing negative charges of the conjugate base (Plewa *et al.* 2010). CAAs majorly include monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), and trichloroacetic acid (TCAA). These three kinds of CAAs are associated with the development of cancer in laboratory animals as well as humans, and have been restricted in drinking water treatment and recycled waste water treatment (USEPA 1998; 2000; Munakata *et al.* 2009). As a consequence, efficient approaches for onsite decomposing of CAAs generated via disinfection are of great importance

for controlling their release into drinking water and recycled water.

Because of its low biological degradability, high water solubility, very large Henry's law constants and relatively higher acidity (Lifongo *et al.* 2004; Jo *et al.* 2011), conventional treatment techniques, i.e. biological processes, granular activated charcoal or air stripping, are less applicable for CAA decomposition; its degradation requires some advanced technical treatment, such as photochemical, sonochemical, electrochemical and advanced oxidation processes (AOPs) (Lifongo *et al.* 2004; Gu *et al.* 2016). Since ultraviolet (UV) light was originally established for drinking water disinfection (Lu *et al.* 2008) and provides a simple technique for oxidation and mineralization without supplementary oxidants (Basturk & Karatas 2014), photochemical decomposition using UV irradiation has recently become attractive for organic contaminants' removal in water (Lee *et al.* 2005; Kelly & Arnold 2012; Chen & Chu 2014). Although photolytic degradation of some organic contaminants by direct UV radiation has been successfully used in drinking water treatment (Dantas *et al.* 2010; Chen & Chu 2014; Lee & Jin 2014), routine UV photolysis did not work well on CAA decomposition. In the published literatures, there was no obvious performance in CAA decomposition by low intensity UV irradiation, achieving less than 1% removal after 30-min reaction time due to its photostability (Lifongo *et al.* 2004; Toor & Mohseni 2007; Jo *et al.* 2011). Efficient UV photolysis of organic pollutants was considered to be caused by direct and indirect photolysis. Direct photolysis occurs when a molecule of the pollutant absorbs UV photons' flux and induces energy level transition, then leading to the pollutant C-X bond breakage (Xu *et al.* 2009; Kelly & Arnold 2012). Indirect photolysis occurs when UV photons' flux, interacting with water or organic matter, generates transient reactive species (e.g. OH, O₃ and H₂O₂), or other photochemically produced reactive intermediates, which subsequently oxidize the pollutant and induce its degradation (Canonica *et al.* 2008; Lee & Jin 2014). The efficiency of CAA degradation was determined by how fast the C-X bond breakage occurred and how much of the transient reactive species was generated. Poor CAA decomposition through routine UV photolysis seems restricted by the limited amount of UV photons. Hence, promoting UV photon production may be

a plausible way for efficient CAA decomposition. It was reported that UV photons can be promoted by employing higher intensity UV irradiation, which is much higher than that of low- and medium- pressure UV irradiation lamps (Zhang *et al.* 2013a, 2013b).

In this study, the feasibility and suitability of using high photon flux UV irradiation ($3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$) for enhancing CAAs (MCAA, DCAA and TCAA) decomposition was investigated. In order to fully examine overall performance of CAA decomposition by a high photon UV system, and help in offering a new way to decompose organic pollutants with strong photostability, a laboratory study using a specific high photon UV system setup to treat simulated water was carried out. The effects of experimental parameters on the pollutant decomposition, i.e. UV photon flux, CAA initial concentration, and pH, were also analyzed.

EXPERIMENTAL SECTION

Chemicals

All chemicals, including MCAA (C₂H₃ClO₂, ≥99%), DCAA (C₂H₂Cl₂O₂, ≥99%) and TCAA (C₂HCl₃O₂, ≥99%), as typical kinds of CAAs, were chosen to be the target pollutant. These pollutant solutions were prepared by the fine chemical agent obtained from Jiangsu Yonghua Fine Chemical Co., Ltd. NaOH and H₂SO₄ were obtained from commercial sources (Sigma-Aldrich and Aladdin) and used as received in this study. Ultrapure water (Millipore Milli-Q) was used for solution preparation.

Experimental setup

CAA photolysis decomposition experiments were carried out in an opened glass reactor (50 mm in diameter and 60 mm in length). A 250 W UV high pressure mercury lamp (Ushio Inc., Japan) was used as the irradiation source assembled with a reflector, a shutter, and a timer in a closed box (Zhang *et al.* 2013a, 2013b). The lamp was placed at the focal axis of the quartz reflector and an optical filter was inserted to cut off the long wavelength components ($\lambda > 425 \text{ nm}$) for avoiding heating of the aqueous

solution. UV photons flux entering the glass reactor directly without going through any cover, and can be adjusted by transmission filters with different density (Zhang *et al.* 2013a, 2013b). Different UV light photons fluxes were measured inside the reactor cylinder using the potassium ferrioxalate method (Hatchard & Parker 1956). The incident photons flux of the high pressure mercury lamp entering the photoreactor was varied from 1.57×10^3 to $3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$, which reported as a fraction of the maximum UV photon flux (I_{max}), i.e. $I = 50\% I_{max}$. I_{max} was measured at $3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$.

10 mL of testing solutions were used in the experiments, and the initial MCAA, DCAA and TCAA solution concentration falls in a typical concentration range present. In the open experimental system, there was no extraneous O_3 recourse in the experimental environment and without controlling dissolved oxygen (DO) in the testing solution during the experiment, due to their own rare influence on the CAA decomposition (date not shown). Initial solution DO was about $5 \pm 0.5 \text{ mg/L}$ and not adjusted unless specified in each experiment set. Initial solution pH was 3.2 and not adjusted unless specified. In cases investigating the influences of pH, initial solution pH was adjusted by dropwise addition of diluted NaOH or H_2SO_4 solution and varied from pH 3.2 to 9.0.

Experimental time was varied up to 40 min. A water cooling jacket was applied to maintain the solution temperature at $25 \pm 3^\circ\text{C}$ throughout the experiment, and an air cooling system was also used to control the temperature of the UV lamp (Zhang *et al.* 2013a; Gu *et al.* 2016). At the end of the experiment, the reactor was moved away from the UV lamp and the testing solution was withdrawn for immediate chemical analyses. All experiments were carried out in duplicate and repeated 5 times. The student's t-test was used to statistically evaluate the significance of a difference of two data sets. A confidence level of 95% was used to determine the critical value of the t-distribution.

Analytical methods

CAAs were analyzed in a Dionex ion chromatograph (Dionex ICS-3000). The solution pH was determined by PHS-3C digital pH-meter (Shanghai San-Xin).

Absorption spectra was determined by a UV-vis spectrophotometer (Shimadzu UV-2450). Incident UV light photons fluxes inside the reactor cylinder were measured using the potassium ferrioxalate method (Hatchard & Parker 1956).

RESULTS AND DISCUSSION

Photolytic degradation of MCAA, DCAA, TCAA and their 1/1/1 mixture

In the dark controls, three kinds of CAAs showed almost no changes during the reaction time (data not shown), hence their degradation by other processes could be negligible in the presented study. In contrast, a fast individual decomposition of MCAA, DCAA and TCAA was observed in the high photon flux irradiation system ($3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$) (Figure 1). Almost all MCAA (>95%), DCAA (>97%) and TCAA (>99%) were degraded from its initial concentration of 20 mg L^{-1} within 40 min. Meanwhile, MCAA, DCAA and TCAA arrived to be lower than MDL in 50 min, 30 min and 25 min, respectively. The decomposition kinetics of these three kinds of CAAs can be well described by the pseudo-first order model and the obtained kinetic constants (k_{obs}) of MCAA, DCAA and TCAA were 0.0666, 0.1623 and 0.2317 s^{-1} (Table 1), respectively. Their photolytic degradation rate determined in this study is in the order of $\text{TCAA} > \text{DCAA} > \text{MCAA}$.

Compared to the extremely poor performance of CAA photodegradation with less than 1% removal rate within 30 min by using low photon flux irradiation in the previous study (Wang *et al.* 2009), since high UV irradiation ($3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$) can promote great generation of UV photon (Zhang *et al.* 2013a; 2013b), subsequently induced more intensive $\cdot\text{OH}$ attack and much faster C-X bond breakage (Canonica *et al.* 2008; Lee & Jin 2014; Xu *et al.* 2009; Kelly & Arnold 2012), efficient photolytic degradation of CAAs was observed in this study. It demonstrated that the high photon flux UV irradiation system is an attractive and potential approaches for CAAs decomposition, even for other hard-to-degrade DBPs.

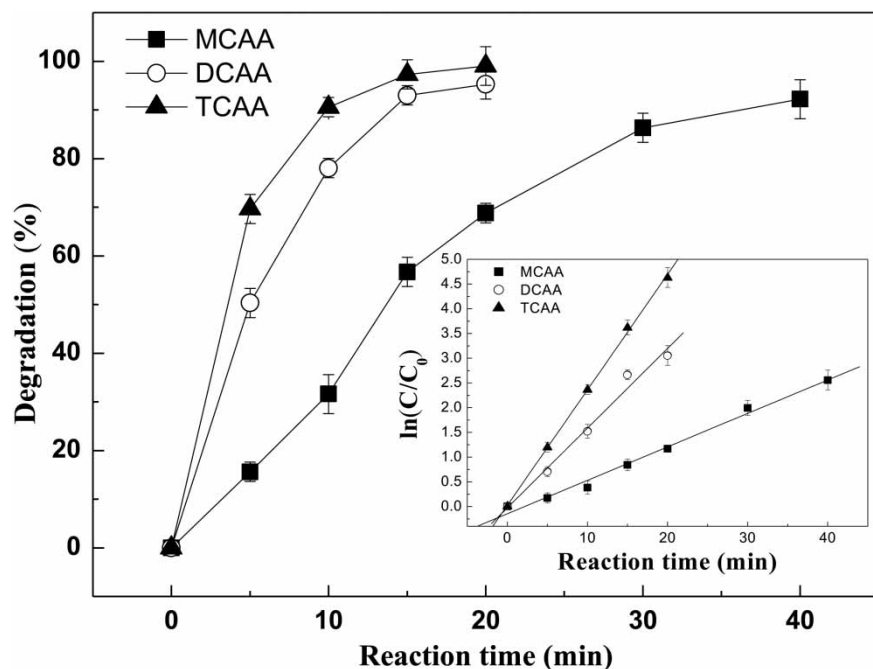


Figure 1 | Photolytic degradation of chloroacetic acids versus irradiation time separately.

As an important class of DBPs, CAAs are often formed during water chlorination, and the chosen three (MCAA, DCAA and TCAA) are usually simultaneously found to be present together in disinfected drinking water or recycled water (Munakata *et al.* 2009). Therefore, the effect of high photon flux UV on the mixture of CAA photolytic degradation was investigated, which contained MCAA, DCAA and TCAA in a 1/1/1 weight ratio. A mixture of three CAAs had the similar obvious decomposition to the previous individual results, and their decomposition also followed a pseudo-first order model well (data not shown). In order to get closer to realistic conditions, a mixture solution of 20 mg/L MCAA, 20 mg/L DCAA and

20 mg/L TCAA was used as the target pollutant in the subsequent section.

Effect of UV irradiation intensity

An experiment further investigating the effect of UV photon fluxes on the mixture solution of 20 mg/L MCAA, 20 mg/L DCAA and 20 mg/L TCAA photolytic degradation was carried out. As shown in Figure 2, when the UV photon flux increased from 1.56×10^3 to $3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$, the decomposition of MCAA, DCAA and TCAA all followed pseudo-first order kinetics. The kinetic constants were estimated from the

Table 1 | The kinetic parameters of individual and simultaneous photolysis of chloroacetic acids

	Photolysis of chloroacetic acids individually		Photolysis of the mixture of chloroacetic acids	
	Reaction rate constant (s^{-1})	Correlation coefficient R^2	Reaction rate constant (s^{-1})	Correlation coefficient R^2
MCAA	0.0666 ± 0.00269	0.9911	0.0668 ± 0.00351	0.9894
DCAA	0.1623 ± 0.00524	0.9917	0.1613 ± 0.00254	0.9844
TCAA	0.2317 ± 0.0120	0.9988	0.2334 ± 0.00857	0.9990

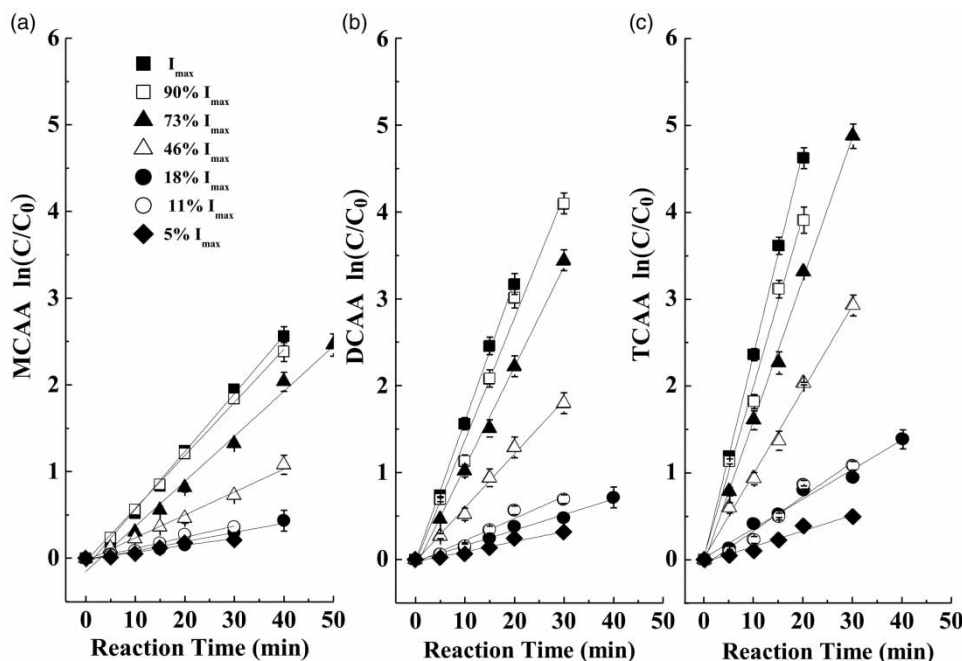


Figure 2 | The effect of different UV photon flux on the photolytic degradation of the mixture of chloroacetic acids.

slope of $\ln(C/C_0)$ versus time using a linear regression. As shown in Figure 3, k_{obs} of each MCAA, DCAA and TCAA steadily increased from 0.0152 to 0.0667 s^{-1} , from 0.0239 to 0.1617 s^{-1} , and from 0.0247 to 0.2327 s^{-1} , respectively, when UV intensity increased from $5\% I_{max}$ to $100\% I_{max}$. The linear relationship

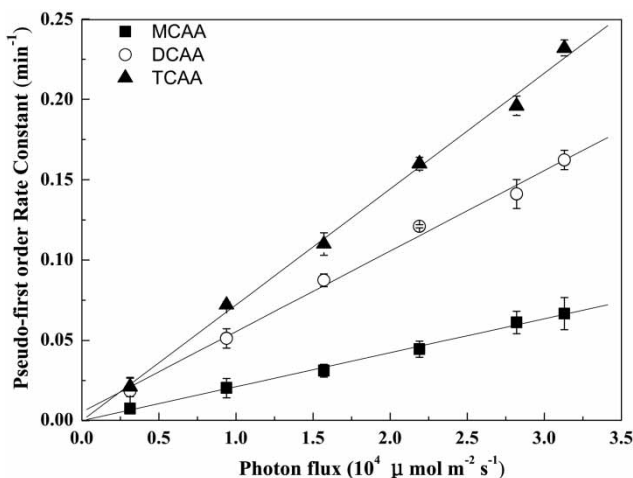


Figure 3 | The effect of photon flux on the photolytic degradation pseudo-first order constant.

between UV photon fluxes and CAA kinetic constant demonstrated that the high photon UV system also showed efficient light energy utilization. In addition, the three fitted lines of each MCAA, DCAA and TCAA decomposition approximately passed through the origin point (Figure 3), indicating that the CAA photolysis efficiency linearly increased with the UV photon flux. This also helps explain the poor degradation of CAAs when applying low intensity photon flux from 1×10 to $1 \times 10^2 \mu\text{mol m}^{-2} \text{ s}^{-1}$ in the previous study (Lifongo et al. 2004; Toor & Mohseni 2007; Wang et al. 2009; Jo et al. 2011), and announces an effective means for those photolytic resistant pollutants decomposition.

Effect of initial chloroacetic acid concentration

Besides UV irradiation, the initial concentration of the mixture of CAAs (MCAA, DCAA and TCAA) will affect their photolytic degradation. Under the condition of the UV photon flux $I = I_{max}$, the degradation performance of a mixture solution with an initial concentration range of

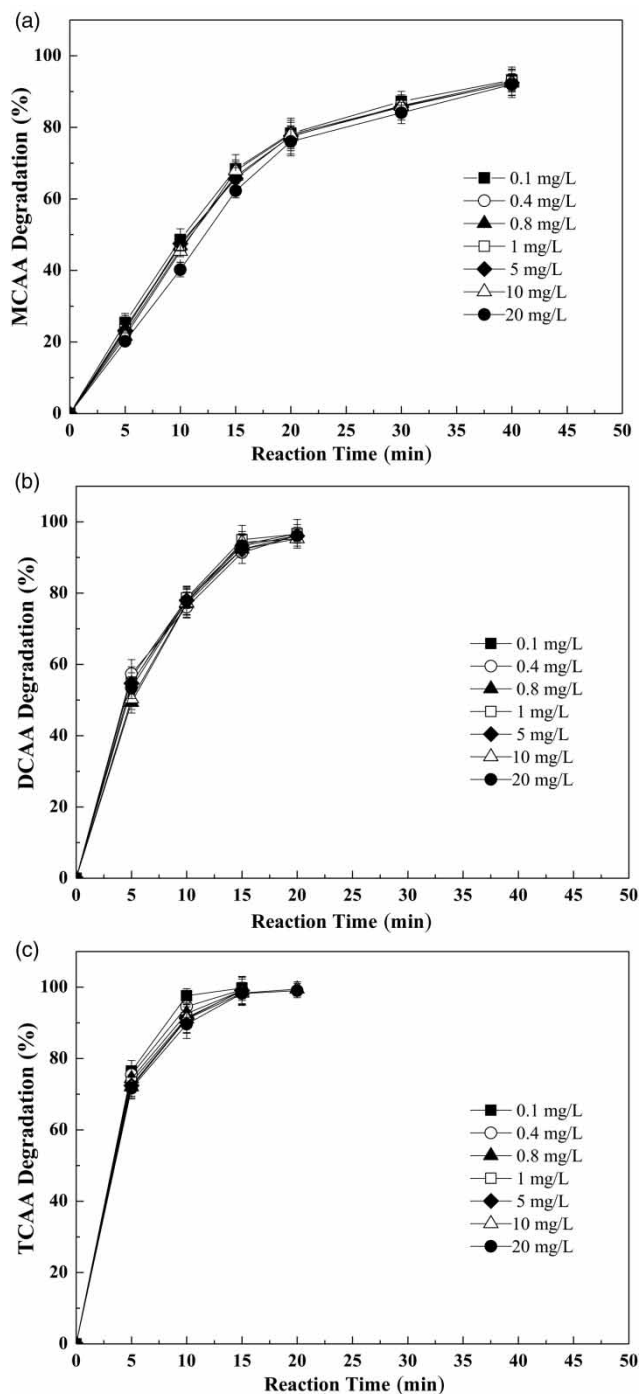


Figure 4 | The effect of chloroacetic acids initial concentration on the photolytic reaction.

0.1 ~ 20 mg L⁻¹ was investigated (Figure 4). When the initial concentration increased from 0.1 to 20 mg/L (Wang *et al.* 2009; Tang *et al.* 2012), pollutant

decomposition showed no obvious variation, and efficient degradation of each chloroacetic acid was observed. In addition, photodegradation of MCAA, DCAA and TCAA with different initial concentrations all followed a similar degradation pattern and fitted well to the first-order kinetics. The pollutant MCAA, DCAA and TCAA removal rate drastically increased in the first 20, 10 and 5-min periods, and then gradually increased to nearly 100% by the end of 40, 20 and 15-min reaction time, respectively. This obtained fast CAA degradation and intensive tolerance to a larger range of initial concentration, highlighting the powerful treatability and flexibility of high photon flux UV photolysis in the wild field application.

Effect of initial pH

The effect of initial pH ranging from 3.2 to 9.0 on the mixed solution of MCAA, DCAA and TCAA degradation using the high photon flux UV system was investigated. Among the tested pH range, the photolytic degradation of MCAA, DCAA and TCAA all fitted well to the first-order kinetics and the results are shown in Table 2. When the initial pH increased from 3.2 to 9.0, there was no obvious change in the decomposition kinetic constants, where the k_{obs} of MCAA, DCAA and TCAA ranged from 0.0647 to 0.0648 s⁻¹, from 0.1613 to 0.1619 s⁻¹ and from 0.2328 to 0.2334 s⁻¹, respectively. According to the indirect UV photolysis, the higher availability of OH⁻ would facilitate radical formation (Equations (1) and (2)) (Gonzalez *et al.* 2004; Zoschke *et al.* 2014), and then it may accelerate the pollutant decomposition. Nevertheless, alkaline pH in the range from 7 to 9 showed no obvious effect on the CAA degradation in the present high photon flux UV system. It was speculated that the great amount of photons generated in the high photon flux UV system could eliminate the impact of •OH loss by breaking the pollutant C-X bond directly through molecular electronic transition according to direct UV photolysis (Zalazar *et al.* 2007).



Table 2 | Effect of pH on the photolytic degradation pseudo-first order constant

	Reaction rate constant (s ⁻¹)			
	pH = 3.2	pH = 5	pH = 7	pH = 9
MCAA	0.0647 ± 0.00551	0.0651 ± 0.00254	0.0649 ± 0.00114	0.0647 ± 0.00085
DCAA	0.1613 ± 0.00225	0.1617 ± 0.00754	0.1616 ± 0.00214	0.1619 ± 0.00258
TCAA	0.2332 ± 0.00354	0.2329 ± 0.00547	0.2334 ± 0.00322	0.2328 ± 0.00354

CONCLUSION

As one of the efficient approaches for onsite advanced technical treatment, photon UV irradiation is promising for organic pollutant decomposition. However, due to the photostability and high water solubility, it is difficult to decompose CAAs by using routine UV irradiation photolysis. This study demonstrated the feasibility and suitability of enhancing CAA (MCAA, DCAA and TCAA) decomposition through producing a great amount of UV photons by using high photon UV flux irradiation ($3.13 \times 10^4 \mu\text{mol m}^{-2} \text{s}^{-1}$).

The pollutant of 20 mg L^{-1} MCAA, 20 mg L^{-1} DCAA and 20 mg L^{-1} TCAA in mixed solution achieved almost complete degradation (>99%) within 50 min, 30 min and 25 min, respectively. The photolysis process under high UV light intensity followed the pseudo-first-order reaction kinetics model, and the k_{obs} for all three kinds of CAAs is in the order of TCAA > DCAA > MCAA. Each k_{obs} linearly increased with the increasing of UV intensity, and the fitted line approximately passed through the origin point, indicating that the light utilization efficiency was not negatively affected by the high photon UV flux irradiation. In addition, the variation of pH from 3.2 to 9.0 showed no observable influence on CAA decomposition, which further indicated the minor effect of pH in this present studied system.

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