

## Degradation of organic micropollutant by vacuum ultraviolet process: a kinetics study

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**Abstract**—Vacuum ultraviolet (VUV) process has been regarded as a novel advanced oxidation process for its unique way of generating HO<sup>•</sup> via direct photolysis of water. This study investigates the kinetics of the VUV process for degradation of a typical pesticide, glyphosate (GLY), by using a photoreactor equipped with a precision sampler. The precision sampler enables us to take samples at distances ranging from 0.1 to 1.5 cm to the irradiation source so that GLY degradation by VUV in different parts of the reaction volume can be evaluated. The results of experiments on H<sub>2</sub>O<sub>2</sub> production from VUV photolysis of water confirmed that there existed an extremely uneven distribution of VUV flux and HO<sup>•</sup> generated in the VUV process. Enhancing mass transfer by agitation could significantly increase GLY degradation efficiency by improving the distribution of HO<sup>•</sup>. The initial concentrations of water and GLY governed the reaction kinetics of GLY degradation in the VUV process. As the solution pH increased from 5.0 to 9.0, the overall degradation efficiency of GLY decreased from 97.6% to 77.8%. Depending on their molar absorption coefficients, inorganic anions affected adversely on the degradation of GLY by VUV in the order of NO<sub>3</sub><sup>-</sup> > HCO<sub>3</sub><sup>-</sup> (alkalinity) > Cl<sup>-</sup> > SO<sub>4</sub><sup>2-</sup>. As a strong HO<sup>•</sup> scavenger, natural organic matter shows a much more adverse impact on GLY degradation than that of inorganic anions. Based on the experimental results, this study has provided insights into the kinetics of VUV process for pollutant degradation.

Keywords: Vacuum Ultraviolet, Glyphosate, Degradation Kinetics, Flux Distribution

### INTRODUCTION

Advanced oxidation processes (AOPs) have been intensively studied for the degradation of refractory organic micropollutants that conventional water treatment processes can hardly remove [1,2]. The AOPs are characterized by the generation of hydroxyl radical (HO<sup>•</sup>), which has a high redox potential of 2.59 V [3] and can degrade most of the pollutants that can be possibly found in water.

As a novel AOP, the vacuum ultraviolet (VUV) process has been applied in the production of ultrapure water, conversion of organic materials and water treatment, specifically the degradation of refractory organic micropollutants [4]. It has the advantages of efficient degradation of pollutants, no addition of chemical agents, simple reaction system and convenient operation and maintenance [5]. Unlike other AOPs, the VUV process has a unique way of generating HO<sup>•</sup> via photolysis of water (Eqs. (1) and (2)), which makes the VUV process a clean and green technology for water treatment. Because the generated H<sup>•</sup> and e<sub>aq</sub><sup>-</sup> (Eqs. (1) and (2)) can readily be scavenged by dissolved oxygen in water, they contribute little to micropollutant degradation in the VUV process [6]. Therefore, the target pollutants with low concentration are actually degraded by reaction with the generated HO<sup>•</sup> [7]. Many efforts have been made to make good use of the generated HO<sup>•</sup> for pollutant degradation [8-10]. For example, Dobrovic et al. [9] examined the effect of hy-

drodynamic conditions on the degradation of natural organic matter (NOM) by VUV and found that increasing Reynolds number could significantly improve the mass transfer of HO<sup>•</sup>, resulting in a much higher degradation rate of NOM. Li et al. [10] reported that the addition of free chlorine remarkably increased the degradation rate of methylene blue by VUV and attributed the synergistic mechanism to the effective use of HO<sup>•</sup> for pollutant removal through formation of longer-lived secondary radicals (e.g., ClO<sup>•</sup>). Since water has an excessively high concentration (55.6 M), its high absorption coefficient causes the uneven distribution of VUV flux, which could decrease by 90% pathing through 0.55 cm thickness of water (i.e., irradiated region) [11,12]. In this irradiated region, the generated HO<sup>•</sup> reacts with the target pollutants, but a large amount of HO<sup>•</sup> would recombine with H<sup>•</sup> to form water due to the short life-time of HO<sup>•</sup> [13-15], which significantly reduces the efficacy of the VUV process. To summarize, the efficacy of the VUV process depends largely on utilization efficiency of the generated HO<sup>•</sup>.



With a relatively low molar absorption coefficient of 0.032 M<sup>-1</sup> cm<sup>-1</sup>, water has an absorption coefficient of 1.8 cm<sup>-1</sup> (the product of molar absorption coefficient and concentration) at 185 nm [16], which exerts great influence on the pollutant degradation by VUV. However, the pollutants can still exert comparable influence on the VUV process because they usually have much larger molar absorption coefficients (>1,000 M<sup>-1</sup> cm<sup>-1</sup>), although with a relatively low

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concentration. Consequently, the initial concentrations of water and pollutants govern their absorption ratios of VUV, which strongly affects the degradation reaction kinetics in VUV process. However, there are contradictory results reported regarding the effect of initial pollutant concentration on its degradation by VUV. For example, Imoberdorf and Mohseni [17] reported that the degradation rate of 2,4-D in ultrapure water was independent of the initial concentration of the contaminant, while Wu et al. [18] found that the degradation kinetics of dimethoate by VUV was strongly dependent on the initial concentration of the contaminant. Other factors, such as solution pH, alkalinity and water components, could also have a great impact on the degradation of pollutants by VUV and are also worthy of further investigation [4].

Although many papers have been published regarding the degradation of organic pollutants by VUV process, little work has been done on the relationship between the distribution of VUV flux and the pollutant degradation kinetics. Therefore, based on the photoreactor equipped with a precision sampler, the main aim of this study is to investigate the effects of distribution of VUV flux and HO<sup>•</sup> formed on the degradation of a typical pesticide, Glyphosate (GLY), by VUV process. The effects of initial concentration of water and pollutant on the degradation of GLY are closely examined. Other effects, such as solution pH and water components on the GLY degradation by VUV, will also be evaluated.

## MATERIALS AND METHODS

### 1. Chemicals

Glyphosate (GLY) with a purity of >99% was obtained from Sigma-Aldrich. H<sub>2</sub>O<sub>2</sub> (30%) was from Jin Shan Hua Shi Co., Ltd., Chengdu, China. Other chemical reagents of at least analytical grade were used without further purification. All solutions were prepared with deionized (DI) water.

### 2. Experimental Setup and Reaction Conditions

The experiments were carried out in a conventional annular batch photoreactor. The inner diameter and height of the photoreactor are 8.0 and 38 cm, respectively. A low-pressure mercury lamp (8 W) was used as the VUV irradiation source. The lamp is enveloped in a quartz sleeve to avoid direct contact with water. The outside diameter of the quartz sleeve is 4.8 cm, so the actual irradiation water layer thickness is 1.6 cm, which ensures that all the VUV is absorbed by the aqueous solution. To take samples at the exact position in the volume, a precision sampler was designed. The length of the sampler needle inserted into the reactor is accurately controlled by a vernier caliper so that samples can be taken at a distance interval of 0.2 cm from the quartz sleeve to the reactor wall. The reactor is placed on a magnetic agitator, which has a digital display to control the rotary speed.

### 3. Analytical Methods

The H<sub>2</sub>O<sub>2</sub> concentration was analyzed by a photometric method with a low detection limit of 3 μg L<sup>-1</sup> [19]. GLY was detected by ultra-performance liquid chromatography-tandem mass spectrometry (Xevo TQD MS/MS, Waters, USA). The injection volume was 10 μL. The mobile phase was a mixture of 0.2% formic acid in water (50%) and methanol (50%) at a flow rate of 0.3 mL min<sup>-1</sup>. The MS/MS detector was operated under the following conditions:

negative electrospray ionization mode, capillary voltage 3.5 kV, cone voltage 15 V, cone gas flow rate 50 L h<sup>-1</sup>, desolvation gas flow rate 550 L h<sup>-1</sup>, desolvation temperature 500 °C. The quantitative precursor and product ions were 168 and 124 (*m/z*), respectively, with a collision energy of 15 V.

## RESULTS AND DISCUSSION

### 1. Distribution of VUV Flux

As water itself can be broken up after absorbing VUV, it usually starts with Eqs. (1) and (2) resulting in the formation of HO<sup>•</sup> in the VUV process for degradation of pollutants. Therefore, water is the main reactant in the VUV process, besides acting as a solvent as in the cases of many other AOPs. Since water has a high absorption coefficient, the distribution of VUV flux in the photoreactor is dramatically uneven. In an annular photoreactor, Eq. (3) can be used to model the VUV flux distribution in water [20]:

$$F = \frac{I_0}{2\pi(d+R_s)L} 10^{-A_{185}d} \quad (3)$$

where *F* (einstein s<sup>-1</sup>) is the VUV flux at a specific position; *I*<sub>0</sub> is the total VUV intensity of the lamp; *A*<sub>185</sub> is the absorption coefficient of water at 185 nm; *d* (cm) is the distance from the position to quartz sleeve; *R*<sub>s</sub> (2.4 cm) is the radius of the quartz sleeve; *L* is the length of the lamp. The modeling result (Fig. 1, black line) shows an exponentially decreasing tendency for the distribution of VUV flux. *F*/*F*<sub>0</sub> decreases from 100% at the quartz sleeve to less than 1% within a distance of 1 cm to the quartz sleeve.

Since HO<sup>•</sup> is extremely reactive and has a short lifetime of 2.3 × 10<sup>-9</sup> s [21], it will readily be depleted by Eqs. (4) and (5) or by reacting with other substances in water. Therefore, the in-situ generated HO<sup>•</sup> will not diffuse far away from where it is generated, which causes the same uneven distribution of HO<sup>•</sup> [20]. As it is difficult to directly detect HO<sup>•</sup> in water, the formation of H<sub>2</sub>O<sub>2</sub> (Eq. (5)), which is stable and easy to detect, provides a good method for evaluation of the behavior of HO<sup>•</sup>. According to our previous study [22], the formation of H<sub>2</sub>O<sub>2</sub> via Eq. (5) follows pseudo-zero-order

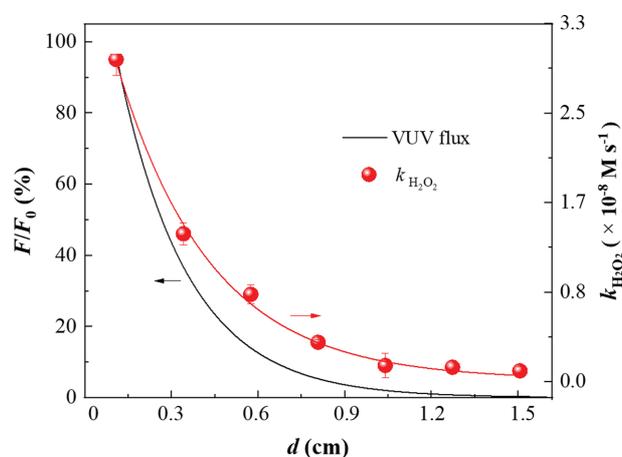
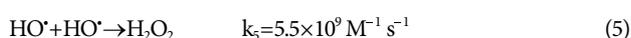
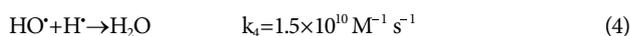


Fig. 1. Distribution of VUV flux and H<sub>2</sub>O<sub>2</sub> production rate as a function of distance to the quartz sleeve without agitation (*F*<sub>0</sub> referring to the highest value at *d*=0).

reaction kinetics (Eq. (6)), and its pseudo-zero-order production rate constant ( $k_{H_2O_2}$ ,  $\mu\text{M min}^{-1}$ ) is proportional to VUV flux, which has been considered as a green and simple method to determine the VUV flux. Fig. 1 shows that  $k_{H_2O_2}$  also decreased exponentially as a function of distance to the quartz sleeve without agitation, which confirmed the uneven distribution of VUV flux and  $\text{HO}^*$  generated by VUV photolysis of water in the VUV process. However, the distribution of  $k_{H_2O_2}$  (red line) showed a positive deviation from VUV flux distribution (black line) due to the diffusion of  $\text{H}_2\text{O}_2$ . Consequently, the method [22] we previously developed for the determination of VUV flux based on the  $\text{H}_2\text{O}_2$  production rate is only applicable to determining the averaged VUV flux of the whole volume for a batch reactor after the solution is well mixed.



$$\frac{dc_{H_2O_2}}{dt} = k_{H_2O_2} \quad (6)$$

## 2. Effect of Agitation on GLY Degradation by VUV

As the low-pressure mercury lamp emits both 185 nm VUV and 254 nm UV at the same time, the UV usually interferes with the VUV process for pollutant degradation. However, the absorption spectrum of GLY at wavelength from 190 to 600 nm (Fig. S1) shows that little UV at 254 nm was absorbed by GLY. Therefore, the GLY degradation by direct UV photolysis could be neglected and that by direct VUV photolysis and/or indirect VUV photolysis (i.e.,  $\text{HO}^*$  oxidation) should be the main cause. Fig. 2(a) shows that the uneven distribution of VUV flux led to the remarkable heterogeneity of GLY degradation by VUV without agitation. When the GLY degradation was fitted with pseudo first-order reaction kinetics (Eq. (7)), the degradation rate constant ( $k_{GLY}$ ,  $\text{s}^{-1}$ ) decreased from  $5.5 \times 10^{-2} \text{ s}^{-1}$  at  $d=0.1 \text{ cm}$  to  $2.6 \times 10^{-3} \text{ s}^{-1}$  at  $d=1.5 \text{ cm}$ . As  $\text{HO}^*$  had the same uneven distribution as that of VUV flux [20], GLY degradation by direct VUV photolysis and/or indirect VUV photolysis would cause the same heterogeneity of GLY degradation by VUV process. The highest  $k_{GLY}$  in the vicinity of the quartz sleeve was attributed to the highest VUV flux. When the distance was higher than 1 cm (i.e., the VUV dark zone), the decreasing of GLY concentration was caused by diffusion of GLY. Therefore, the mass transfer resistance of GLY became a major factor affecting the efficiency of GLY degradation by VUV, and one simple way to reduce the resistance is agitation. Compared with Fig. 2(a), Fig. 2(b) shows that the distribution of  $k_{GLY}$  was much more uniform at a rotary speed of 1,000 rpm. The  $k_{GLY}$  decreased from  $3.6 \times 10^{-2} \text{ s}^{-1}$  at  $d=0.1 \text{ cm}$  to  $1.5 \times 10^{-2} \text{ s}^{-1}$  at  $d=1.5 \text{ cm}$ . To quantitatively determine the improvement of overall degradation efficiency ( $\eta$ , %) caused by agitation, the reaction volume was separated into eight sections with the same thickness of 0.2 cm (Fig. S2). The overall degradation efficiency was determined by averaging the degradation efficiencies in all sections (Eq. (8)). Fig. 2(c) shows that  $\eta$  at a rotary speed of 1,000 rpm reached 68.7% after 50 s reaction, while that was only 36.7% without agitation. It suggests that enhancement of mass transfer by agitation could significantly increase pollutant degradation efficiency by improving the distribution of  $\text{HO}^*$  in VUV process.

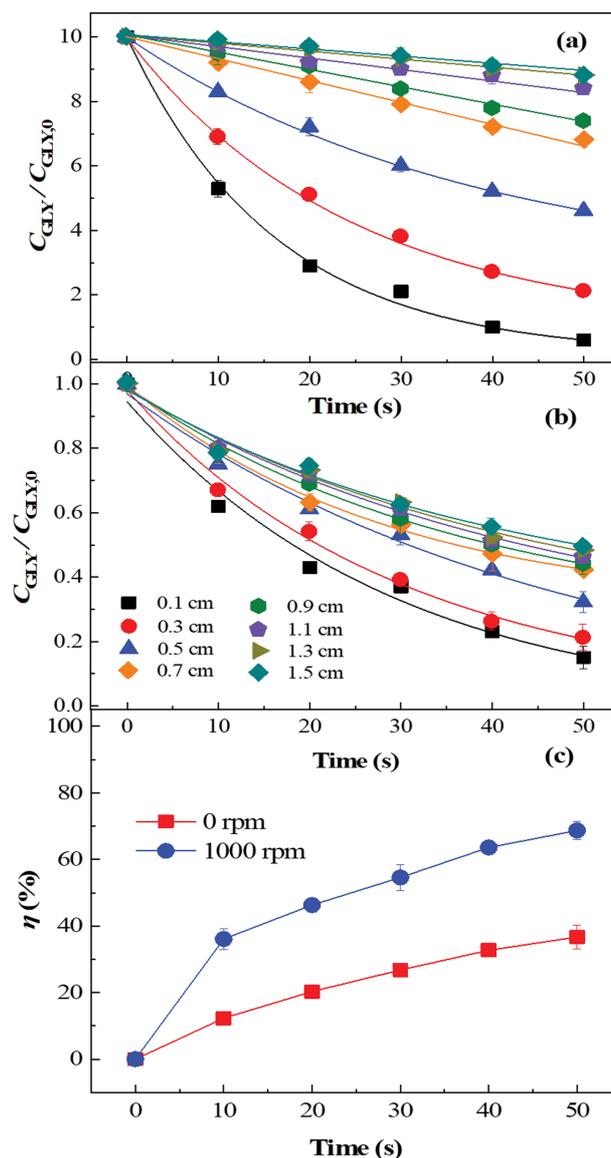


Fig. 2. Degradation of GLY by VUV at different distances as a function of time at rotary speeds of (a) 0 rpm and (b) 1,000 rpm, and (c) improvement of overall degradation efficiency by agitation ( $C_{GLY,0}=10 \text{ mg L}^{-1}$ ).

$$\frac{dc_{GLY}}{dt} = k_{GLY}c_{GLY} \quad (7)$$

$$\eta = 1 - \frac{\sum_{i=1}^8 (1 - \eta_i) V_i}{V} \quad (8)$$

## 3. Effect of Initial GLY Concentration

GLY at high initial concentration will compete with water for VUV and governs its absorption ratio of VUV. As the absorbance at 185 nm could not be directly detected by a common commercial spectrophotometer that usually has a detection limit of 190 nm, the molar absorption coefficient of GLY at 185 nm was determined to be  $2,190 \text{ M}^{-1} \text{ cm}^{-1}$  by extrapolating the absorption line (Fig. S1). The VUV absorption ratio of water and GLY as a function of GLY

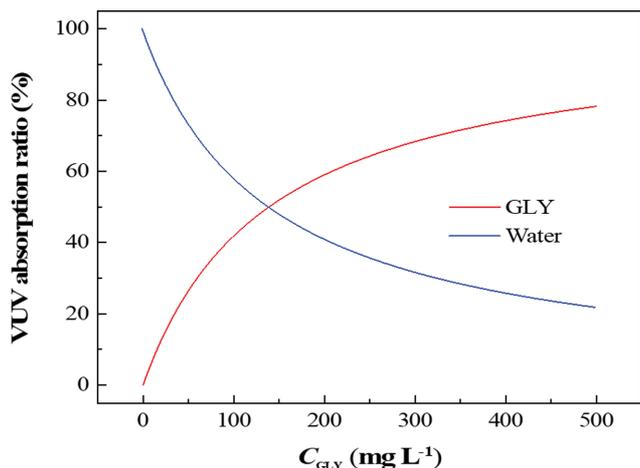


Fig. 3. VUV absorption ratios of GLY and water as a function of initial concentration of GLY.

concentration is shown in Fig. 3. GLY starts to absorb an appreciable amount of VUV ( $>5\%$ ) with its concentration over  $7.4 \text{ mg L}^{-1}$ , where direct VUV photolysis of GLY must be considered. When its initial concentration reaches  $138 \text{ mg L}^{-1}$ , GLY absorbs an equal amount of VUV with that of water ( $50\%$  for each).

To reduce the influence of diffusion, the effect of initial concentration on GLY degradation by VUV was evaluated without agitation. Fig. 4 shows that the initial concentration of GLY had a significant impact on the reaction kinetics of GLY degradation by VUV. When GLY had a low initial concentration of  $1.0 \text{ mg L}^{-1}$ , its degradation followed pseudo-first-order kinetics at all distances to the quartz sleeve. When GLY had a moderate initial concentration of  $10 \text{ mg L}^{-1}$ , its degradation followed pseudo-first-order kinetics at distance of  $\leq 0.5 \text{ cm}$  and followed pseudo-zero-order kinetics at distance of  $>0.5 \text{ cm}$ . When GLY had a high initial concentration of  $100 \text{ mg L}^{-1}$ , its degradation followed pseudo-zero-order kinetics at all distances to the quartz sleeve. Whether the GLY degradation could be fitted with pseudo-first-order or pseudo-zero-order was controlled by the ratio of VUV flux and GLY concentration for direct VUV photolysis (Eq. (9)) or by the ratio of the concentration of generated  $\text{HO}^\bullet$  and GLY for indirect VUV photolysis (Eq. (10)). Both Eqs. (9) and (10) originate from Eq. (7) where  $k_{\text{GLY}}$  equals to  $\Phi F$  for direct VUV photolysis (Eq. (9)) and equals to  $k_{\text{GLY},\text{HO}^\bullet} c_{\text{HO}^\bullet}$  for indirect VUV photolysis (Eq. (10)). When  $c_{\text{GLY}}$  was relatively low, e.g.  $1.0 \text{ mg L}^{-1}$  (Fig. 4(a)) or when the VUV flux was high at  $d < 0.5 \text{ cm}$  with a moderate GLY concentration of  $10 \text{ mg L}^{-1}$  (Fig. 4(b)), namely high ratio of VUV flux and GLY concentration, the degradation of GLY followed pseudo-first-order reaction kinetics because  $c_{\text{GLY}}$  must be regarded as a variate, which is in agreement with many previous studies [12,13]. However, when the initial concentration of GLY was high, e.g.  $100 \text{ mg L}^{-1}$  (Fig. 4(c)) or when the VUV flux was low at  $d > 0.5 \text{ cm}$  with a moderate GLY concentration of  $10 \text{ mg L}^{-1}$  (Fig. 4(b)), namely low ratio of VUV flux and GLY concentration,  $c_{\text{GLY}}$  had a negligible decrement and could be regarded as a constant coefficient. Accordingly, Eqs. (9) and (10) could be simplified to Eq. (6) with  $\Phi F c_{\text{GLY}}$  and  $k_{\text{GLY},\text{HO}^\bullet} c_{\text{HO}^\bullet} c_{\text{GLY}}$  being a constant as a whole. This is the reason why the GLY degradation fol-

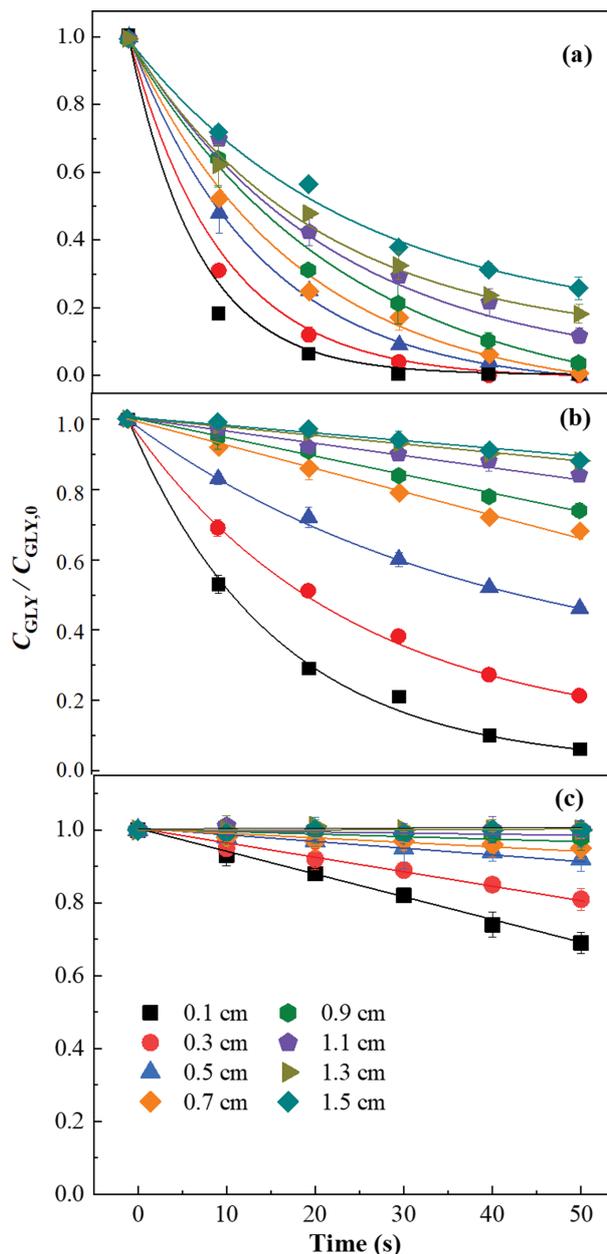


Fig. 4. Degradation reaction kinetics of GLY by VUV at different initial concentrations of (a)  $1.0 \text{ mg L}^{-1}$  and (b)  $10 \text{ mg L}^{-1}$  (data repeated here for clarity) and (c)  $100 \text{ mg L}^{-1}$ .

lowed pseudo-zero-order reaction kinetics with high initial concentration or at far distance from the irradiation source.

$$\frac{dc_{\text{GLY}}}{dt} = \Phi F c_{\text{GLY}} \quad (9)$$

$$\frac{dc_{\text{GLY}}}{dt} = k_{\text{GLY},\text{HO}^\bullet} c_{\text{HO}^\bullet} c_{\text{GLY}} \quad (10)$$

where  $k_{\text{GLY},\text{HO}^\bullet}$  is the second-order rate constant of GLY with  $\text{HO}^\bullet$  and  $c_{\text{HO}^\bullet}$  is the equilibrium concentration of  $\text{HO}^\bullet$ .

#### 4. Effect of Solution pH

Solution pH has a strong impact on the overall degradation effi-

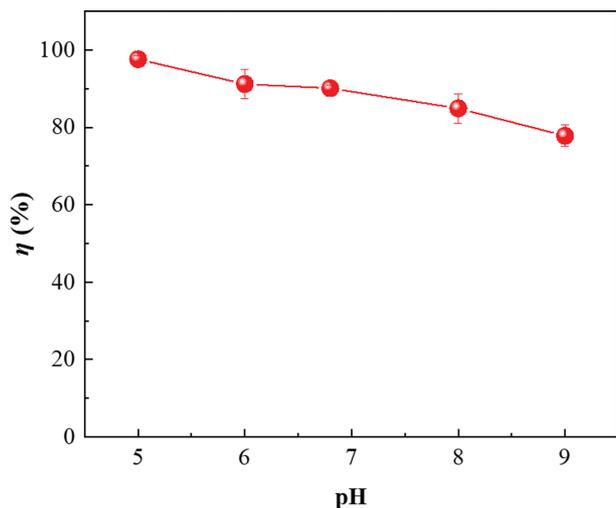


Fig. 5. Effect of solution pH on the overall degradation efficiency of GLY by VUV at a rotary speed of 1,000 rpm ( $C_{GLY,0}=10 \text{ mg L}^{-1}$ ).

ciency of GLY by VUV. Fig. 5 shows that the overall degradation efficiency of GLY decreased from 97.6% to 77.8% after a reaction time of 2 min when the solution pH increased from 5.0 to 9.0. This is in good agreement with Huang et al. [23] who found that 4-*tert*-octylphenol degraded much faster under acidic conditions than under alkaline conditions in VUV process. This is because high pH value would reduce both the redox potential and the formation rate of  $\text{HO}^\bullet$  [22]. Koppenol and Liebman [3] reported that the redox potential of  $\text{HO}^\bullet$  decreased from 2.59 V at pH=0 to 2.18 V at pH=7.0 and  $\text{HO}^\bullet$  would further dissociate to  $\text{O}^\bullet$  with a much lower redox potential (Eq. (11)) under alkaline conditions.



### 5. Effect of Water Components

Inorganic anions such as  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$  and  $\text{HCO}_3^-$  (alkalinity), and NOM are ubiquitous in natural waters and they exert an impact on the VUV process for pollutant degradation [4]. As Fig. 6(a) shows, when the initial concentration of  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$  and  $\text{HCO}_3^-$  increased from 0 to 250  $\text{mg L}^{-1}$ , the degradation efficiency of GLY decreased from 90.1% to 90.0%, 82.6%, 68.9% and 79.2%, respectively. The impact of anions on the GLY degradation was mainly caused by the competitive absorption of VUV by these anions. For example,  $\text{SO}_4^{2-}$  had little impact on the GLY degradation by VUV for the reason that  $\text{SO}_4^{2-}$  has a negligibly low molar absorption coefficient of  $146 \text{ M}^{-1} \text{ cm}^{-1}$  at 185 nm and hardly reacts with  $\text{HO}^\bullet$  [24].  $\text{NO}_3^-$  showed the most adverse impact on the GLY degradation because it has the highest molar absorption coefficient for VUV among the selected anions [24]. However, besides absorbing VUV, NOM is also a strong scavenger for  $\text{HO}^\bullet$  [25,26]. For this reason, the degradation efficiency of GLY decreased significantly from 90.1% to 61.2% after the addition of NOM with a much lower concentration of 2.5  $\text{mg L}^{-1}$  (Fig. 6(b)).

### 6. Discussion of the Optimization of Reactor Dimension

Since there is an extremely uneven distribution of VUV flux and therefore the formed  $\text{HO}^\bullet$ , the optimization of reactor dimension for VUV process is largely dependent on these characteristics. To

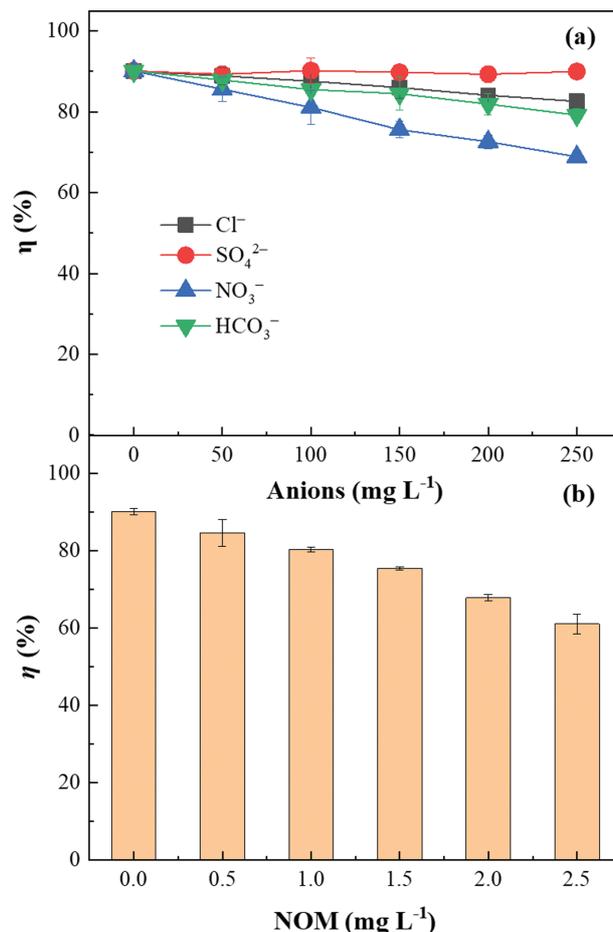


Fig. 6. Effects of inorganic anions (a) and NOM (b) on the overall degradation efficiency of GLY by VUV at a rotary speed of 1,000 rpm ( $C_{GLY,0}=10 \text{ mg L}^{-1}$ ).

optimize the reactor dimension, VUV flux and the formed  $\text{HO}^\bullet$  should be utilized to the most. The previous results indicate that the utilization ratios of VUV and  $\text{HO}^\bullet$  have a positive correlation with the uniformity of degradation rates in each section of the reactor (Fig. S2) for the reason that higher uniformity of degradation rates would result in higher total pollutant degradation efficiency (Fig. 2). Therefore, the precision sampler developed in this study provides a good tool to optimize the reactor dimension by determining the uniformity of degradation rates in each section of the reactor. For example, the optical path length should be no more than 1 cm when there is no mixing of solution, as in the case of a flow-through reactor because there is literally no VUV irradiation or  $\text{HO}^\bullet$  outside that range (Fig. 1). The path length could be much more than 1 cm when there are good mixing conditions, such as efficient agitation or installation of mixer to increase mass transfer. Other factors, such as initial pollutant concentration, solution pH and inorganic anions and NOM, which strongly affect the pollutant degradation as discussed above, must also be taken into consideration to optimize the reactor dimension. To summarize, the uniformity of degradation rates could be used as an indicator for the optimization of reactor dimension after all important factors have been considered.

## CONCLUSIONS

This study investigated the kinetics of the VUV process for degradation of GLY by VUV process. The results confirmed that there existed an extremely uneven distribution of VUV flux and HO<sup>•</sup> generated in the VUV process. Enhancing mass transfer by agitation could significantly increase GLY degradation efficiency by improving the distribution of HO<sup>•</sup>. The initial concentration of water and GLY governed the reaction kinetics of GLY degradation in the VUV process. Increasing solution pH would inhibit GLY degradation, and inorganic anions affected adversely on the degradation of GLY by VUV in the order of NO<sub>3</sub><sup>-</sup>>HCO<sub>3</sub><sup>-</sup> (alkalinity)>Cl<sup>-</sup>>SO<sub>4</sub><sup>2-</sup>. As a strong HO<sup>•</sup> scavenger, NOM had the most adverse impact on the GLY degradation. Based on the experimental results, this study has provided insights into the kinetics of VUV process for pollutant degradation.

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## SUPPORTING INFORMATION

Additional information as noted in the text. This information is available via the Internet at <http://www.springer.com/chemistry/journal/11814>.

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## Supporting Information

### Degradation of organic micropollutant by vacuum ultraviolet process: a kinetics study

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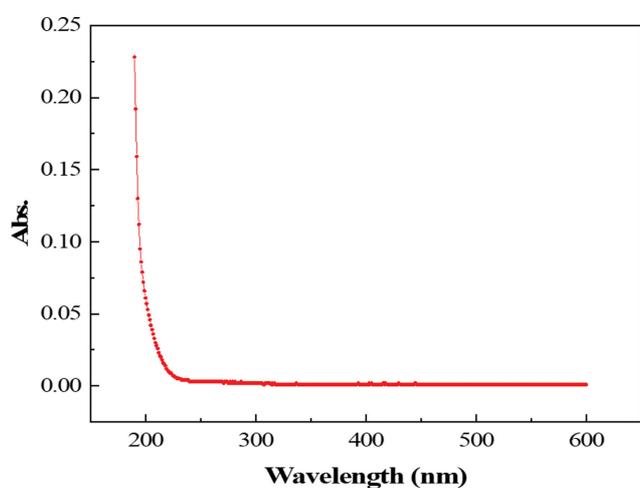


Fig. S1. Absorbance of GLY at wavelength from 190 to 600 nm.

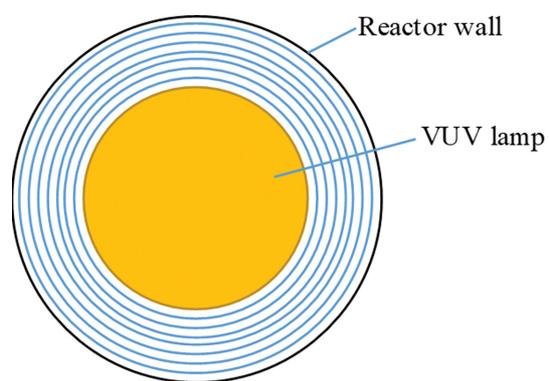


Fig. S2. Schematic diagram of separating the reaction volume into eight sections.