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Photoreduction of the uranyl ion in aqueous solution
II. Alcohols in acid solutions

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Abstract

Photoreduction of uranyl(VI) ion (UO_2^{2+}) in acid solutions has been studied with a steady-state and time-resolved exciting light, where methanol (MeOH), ethanol (EtOH) and iso-propanol (iso-PrOH) were used as quenchers (RCH_2OH) and $HClO_4$, H_2SO_4 and H_3PO_4 as acid solvents (S). The quantum yields, $\phi(U^{4+})$ of resultant U^{4+} and the rate constants, k_q, for the quenching of excited UO_2^{2+} ($^*UO_2^{2+}$) by RCH_2OH were determined.

The k_q values increased in the order of MeOH, EtOH and iso-PrOH corresponding to the increase of $\phi(U^{4+})$ at [RCH_2OH] up to 1.0 mol dm^{-3}, while at the higher [RCH_2OH] the $\phi(U^{4+})$ reached the same maximum less than unity regardless of the kinds of RCH_2OH. The photoreduction with 1.5 mol EtOH dm^{-3} in the acid solutions showed that the $\phi(U^{4+})$ increased in the order of $HClO_4$, H_2SO_4 and H_3PO_4 in the whole range of [S]. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Uranyl ion; Acid solutions; Alcohols; Photoreduction; Photo-emission; Quenching

1. Introduction

In order to clarify photoreduction of uranyl(VI) ion (UO_2^{2+}), we have studied that with ethanol (EtOH) in H_2SO_4 solutions, the mechanism of which was proposed and confirmed experimentally [1]. The mechanism indicated that quenching of photo-excited UO_2^{2+} ($^*UO_2^{2+}$) by EtOH and successive reactions of uranyl ions are involved in the photoreduction: chemical and physical quenching after an encounter complex $^*[UO_2^{2+} \cdots \mathrm{EtOH}]$ between $^*UO_2^{2+}$ and EtOH was formed; a reaction of UO_2^{2+} with α-hydroxylalkyl radical (α-EtR: CH_3CHOH) formed from EtOH.

The quenching of $^*UO_2^{2+}$ by organic reducing quenchers has been considered to be the rate-determining step in the photoreduction [2–9]. Quantum yields, $\phi(U^{4+})$, of the resultant U^{4+} and rate constants, k_q, for the quenching have been correlated with chemical and physical properties of $^*UO_2^{2+}$ and the quenchers to discuss whether the quenching takes place through electron transfer or H-abstraction and whether $^*UO_2^{2+}$ reacts with the quenchers directly or indirectly via water molecules solvating $^*UO_2^{2+}$.

While the above discussion has been made with several organic quenchers in a particular solution, different $\phi(U^{4+})$ values have been obtained in the photoreduction with a particular quencher in various solutions [10–15]. However, it is still not clear by which of the quenchers the quenching and photoreduction could be described, and few studies have provided any information on how the environment of $^*UO_2^{2+}$ would affect the quenching.

In the present experiments, the photoreduction of UO_2^{2+} with alcohols (RCH_2OH) in acid solutions was studied with a steady-state and time-resolved exciting light, and the $\phi(U^{4+})$ and k_q for the quenching by RCH_2OH were determined: methanol (MeOH), ethanol (EtOH) and iso-propanol (iso-PrOH) were used as RCH_2OH; $HClO_4$, H_2SO_4 and H_3PO_4 as acid solvents (S). At first, experiments were done in H_2SO_4–RCH_2OH systems to discuss effects of varieties of quenchers on the photoreduction. Finally, we made experiments on the photoreduction with EtOH in acid solutions to discuss effects of the environment of $^*UO_2^{2+}$.

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2. Experimental [1]

2.1. Chemicals

Stock solutions of 0.5 mol $UO_2{}^{2+}$ dm^{-3} were prepared from trioxouranium (JAERI-U2) and concentrated acid solvents, and then diluted by double-distilled water, acid solvents and alcohols to prepare sample solutions with their specific concentrations. Potassium ferrioxalate taken as an actinometer in photoreduction experiments was prepared according to Parker's procedure [16,30]. Both the alcohols and solvents were of reagent grade.

2.2. Photoreduction

Monochromatic light of 407 ± 6 nm from a 500 W mercury lamp (USH-500D, Ushio) was used as an exciting light for the photoreduction, which can be absorbed by $UO_2{}^{2+}$ but not by U^{4+} in acid solutions. 50 mmol $UO_2{}^{2+}$ dm^{-3} solutions as the sample and 20 mmol ferrioxalate ion dm^{-3} in 50 mmol H_2SO_4 dm^{-3} [16,30] as the actinometer were irradiated simultaneously in quartz cells with a 5 cm path-length where the photons could be absorbed completely in each solution.

Each concentration of U^{4+} and Fe^{2+} formed in the sample and actinometer was measured coulometrically in flow systems with column electrodes of glassy carbon fibers [17]. The number of photons absorbed in the sample was evaluated from that absorbed in the actinometer, which was evaluated from $\phi(Fe^{2+}, 405\,nm) = 1.13$ [16,30]. The photochemical yield of U^{4+} increased linearly with increasing number of absorbed photons as shown in the previous paper [1]. Thus the quantum yield $\phi(U^{4+})$ was obtained by a least square linear fitting leading to the experimental error less than 5%. Where not specified, all experiments were carried out under magnetic stirring and degassing the sample by Ar or N_2 gas at room temperature.

2.3. Photo-emission from $^*UO_2{}^{2+}$

Decays of the photo-emission from $^*UO_2{}^{2+}$ were observed by a spectrometric multichannel analyzer (SMA) with a pulsed Nd:YAG-laser ($\lambda_{ex.} = 266\,nm$), the intensity, pulse-width and repetition were 1 mJ/pulse, 15 ns and 10 Hz, respectively [18]. The time and spectral resolution were a little longer than 15 ns and 0.5 nm, respectively. The emission spectra were obtained by a spectrofluorimeter (F-4500, Hitachi). Since there were no differences in the photo-emissions in the presence and absence of O_2, each sample in 1 cm × 1 cm quartz cells was irradiated without degassing.

Lifetimes, τ, of $^*UO_2{}^{2+}$ were measured from the decay of the photo-emission, Stern–Volmer constants, K_{SV}, for the quenching of $^*UO_2{}^{2+}$ obtained on the basis of the Stern–Volmer equation and consequently the rate constants k_q determined.

3. Results and discussion

3.1. Proposed mechanism for photoreduction of $UO_2{}^{2+}$

From observations of ESR spectra of alcohol radicals (RCHOH) in $UO_2{}^{2+}$–RCH_2OH matrix at 77 K under photo-irradiation [19–21], we could propose the mechanism of the photoreduction of $UO_2{}^{2+}$ with RCH_2OH in acid solutions, as previously proposed [1], as follows:

$$UO_2{}^{2+} + h\nu \rightarrow {}^*UO_2{}^{2+} \tag{1}$$

$$^*UO_2{}^{2+} \rightarrow UO_2{}^{2+}, \quad k_0 \tag{2}$$

$$^*UO_2{}^{2+} \rightarrow UO_2{}^{2+} + h\nu', \quad k_F \tag{3}$$

$$^*UO_2{}^{2+} + RCH_2OH \rightarrow {}^*[UO_2{}^{2+} \cdots RCH_2OH], \quad k_q \tag{4}$$

$$^*[UO_2{}^{2+} \cdots RCH_2OH] \rightarrow UO_2{}^{+} + RCHOH + H^+, \quad k_c \tag{5}$$

$$^*[UO_2{}^{2+} \cdots RCH_2OH] \rightarrow UO_2{}^{2+} + RCH_2OH, \quad k_p \tag{6}$$

where $^*[UO_2{}^{2+} \cdots RCH_2OH]$ indicates an imaginary cage immediately leading to the alternative quenching of $^*UO_2{}^{2+}$: the chemical quenching (5) which forms $UO_2{}^{+}$ and RCHOH; the physical quenching (6) which is thermallized into $UO_2{}^{2+}$ and RCH_2OH. A pair of the resultant $UO_2{}^{+}$ can disproportionate quantitatively in the present deaerated acid solutions [22,23]. The RCHOH is considered to be formed by abstracting H atom from α-CH bond of RCH_2OH, and is ready to reduce $UO_2{}^{2+}$ into $UO_2{}^{+}$ in $UO_2{}^{2+}$–RCH_2OH systems [24,25].

$$2UO_2{}^{+} + 4H^+ \rightarrow U^{4+} + UO_2{}^{2+} + 2H_2O \tag{7}$$

$$RCHOH + UO_2{}^{2+} \rightarrow RCHO + UO_2{}^{+} + H^+ \tag{8}$$

Thermodynamically reduction potentials $E^0((RCHO, H^+)/RCHOH)$ (vs. NHE, pH 0) for MeOH (−1.18 V), EtOH (−1.25 V), iso-PrOH (−1.39 V) [26] and $E^0(UO_2{}^{2+}/UO_2{}^{+}) = +0.16\,V$ [27] allow the reaction (8) to take place.

On the basis of the above mechanism, the yield, $\phi(U^{4+})$, of resultant U^{4+} can be expressed by rate constants for the above reactions:

$$\phi(U^{4+}) = \frac{k_q[RCH_2OH]}{k_0 + k_F + k_q[RCH_2OH]} \frac{k_c}{k_c + k_p} \tag{9}$$

where K_{SV} and τ_0 are Stern–Volmer constant ($=k_q\tau_0$) for the quenching of $^*UO_2{}^{2+}$ by RCH_2OH and lifetime of $^*UO_2{}^{2+}$ in the absence of RCH_2OH, respectively. Thus, the experimentally obtained $\phi(U^{4+})$ gives the fraction $k_c/(k_c + k_p)$ and the constant k_q (K_{SV}) explicitly.

3.2. Photoreduction with alcohols in 0.5 mol H_2SO_4 dm^{-3}

In order to discuss the effects of varieties of quenchers, the photoreduction with RCH_2OH in a specific solution of H_2SO_4 was studied on the basis of $\phi(U^{4+})$ and k_q.

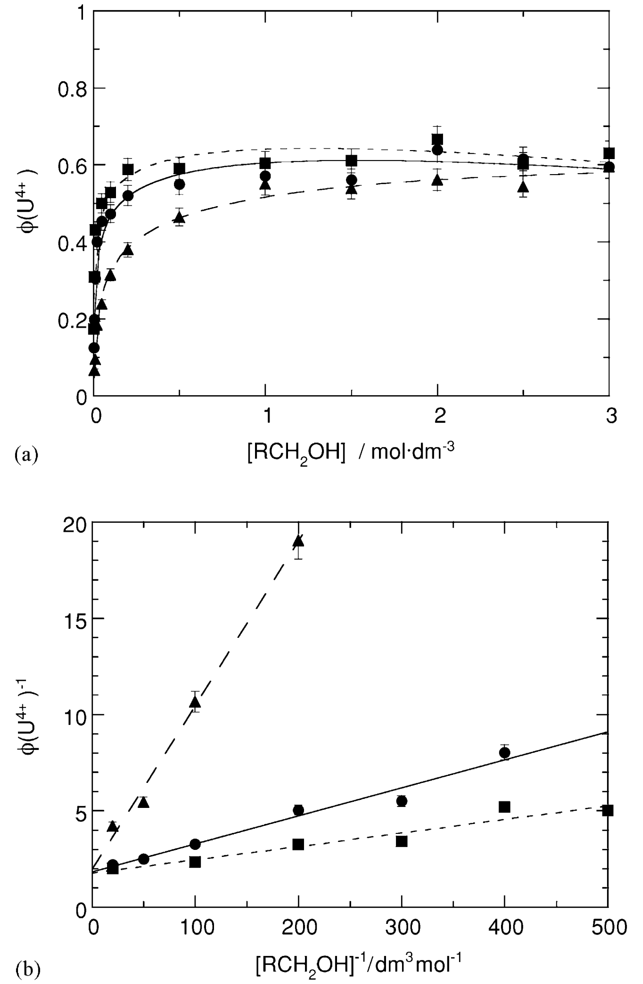

Fig. 1. Photoreduction of UO_2^{2+} with RCH_2OH in 0.5 mol H_2SO_4 dm^{-3}. (a) Dependence of $\phi(U^{4+})$ on $[RCH_2OH]$. (b) Double reciprocal plots of $\phi(U^{4+})^{-1}$ vs. $[RCH_2OH]^{-1}$: EtOH (●); MeOH (▲); iso-PrOH (■). The solution was irradiated with 407 ± 6 nm exciting light at 20 ± 2 °C under deaerated condition (1 Einstein: 6.022×10^{23} photons).

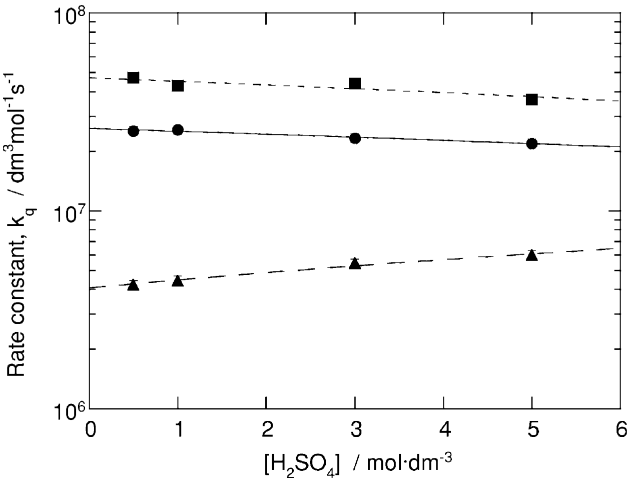

Fig. 2. Rate constants for the quenching (k_q) of $^*UO_2^{2+}$ by RCH_2OH: EtOH (●); MeOH (▲); iso-PrOH (■). The solution was irradiated with 266 nm laser pulses at 20 °C under aerated condition.

Fig. 1(a) shows $\phi(U^{4+})$ for the photoreduction with RCH_2OH in deaerated 0.5 mol H_2SO_4 dm^{-3}. The $\phi(U^{4+})$ at $[RCH_2OH]$ up to 1.0 mol dm^{-3} increased in the order of MeOH, EtOH and iso-PrOH and then was of a tendency to reach the same maximum regardless of the kinds of RCH_2OH. The double reciprocal plots of $\phi(U^{4+})^{-1}$ vs. $[RCH_2OH]^{-1}$ are shown in Fig. 1(b) from which $k_c/(k_c + k_p)$ and k_q can be derived. The k_q in H_2SO_4 solutions was also estimated from the photo-emission experiments as shown in Fig. 2, where each of the k_q for MeOH, EtOH and iso-PrOH was almost constant in the $[H_2SO_4]$ range. These results were summarized together with some parameters in Table 1. The $k_c/(k_c + k_p)$ and k_q derived from Fig. 1(b) are found to be well consistent with the experimentally obtained $\phi(U^{4+})$ and k_q, respectively. Although in preliminary experiments $\phi(U^{4+})$ for *tert*-butanol (*tert*-BtOH) as the quencher was less than one-tenth of that for the present RCH_2OH, the data for *tert*-BtOH was also included in Table 1 and compared with that for RCH_2OH.

According to the above proposed mechanism, k_q value can provide quenching ability of RCH_2OH before an encounter complex $^*[UO_2^{2+} \cdots RCH_2OH]$ was formed, where RCH_2OH would coordinate to $^*UO_2^{2+}$ through one effective encounter after hundreds to thousands of their diffusive collisions to give the complex. Then $k_c/(k_p+k_c)$ value indicates branching ratio into the chemical and physical quenching on the complex. Finally, the magnitude of $\phi(U^{4+})$ can be determined by the overall quenching processes of $^*UO_2^{2+}$

Table 1
The quenching parameters in H_2SO_4–RCH_2OH systems[a]

RCH_2OH	$k_c/(k_c + k_p)$[b]	k_q ($dm^3\,mol^{-1}\,s^{-1}$)[b]	$\phi(U^{4+})$[c]	k_q ($dm^3\,mol^{-1}\,s^{-1}$)[c]	D(CH) (kJ mol^{-1})	I_p (eV)	$\Sigma\sigma^*$[d]	k_{OH} ($dm^3\,mol^{-1}\,s^{-1}$)[e]
MeOH	0.51	4.8×10^6	0.54	4.0×10^6	393	11.0	+0.98	1.0×10^9
EtOH	0.55	2.7×10^7	0.54	2.5×10^7	380.5	10.6	+0.49	1.9×10^9
iso-PrOH	0.57	5.3×10^7	0.56	5.0×10^7	368	10.2	0.00	1.9×10^9
tert-BtOH[f]	–	–	–	1.2×10^6	405.5	9.7	–	6.0×10^8

[a] Errors for experimental results shown in this table are within 5%.

[b] These values are derived from Fig. 1(b) and Eq. (9).

[c] Experimentally obtained $\phi(U^{4+})$ for 1.5 mol RCH_2OH dm^{-3} in Fig. 1(a) and k_q for 0.5 mol H_2SO_4 dm^{-3} in Fig. 2.

[d] Sum of the substituent constants reported by Matsushima and Sakuraba [9].

[e] Reported rate constants for the reaction of OH with RCH_2OH [29].

[f] For comparison, the parameters for *tert*-butanol (*tert*-BtOH) are shown.

by RCH_2OH because the subsequent reactions such as the reaction of UO_2^{2+} with RCHOH and disproportionation of UO_2^{+} progresses quantitatively in the present solutions. Thus, the following stepwise discussions could be made on the basis of the above experimental results and the proposed mechanism:

1. The k_q, which corresponds to the $\phi(U^{4+})$ at $[RCH_2OH]$ up to 1.0 mol dm^{-3}, increased in the order of MeOH, EeOH and iso-PrOH. The k_q and $\phi(U^{4+})$ have been discussed in terms of Taft σ^* [9] as an indicator of the polarity of RCH_2OH, dissociation energies, D(CH), of C–H bond and ionization energies I_p as those of the oxidation shown in Table 1. These suggest that both more effective coordination of RCH_2OH to $^*UO_2^{2+}$ and the more probable paths of their reactants into the products should be taken into account in the interactions between $^*UO_2^{2+}$ and RCH_2OH. In the coordination, configuration among α-CH and OH bonds of RCH_2OH and linear O=U=O bond of the $^*UO_2^{2+}$, which has been discussed in some studies [6,21], should be more important for the quenching. On the other hand, H-abstraction from RCH_2OH is more likely to explain the oxidation of RCH_2OH, i.e. the reduction of $^*UO_2^{2+}$ because, as in Table 1, k_q and $\phi(U^{4+})$ increase with decreasing D(CH), while much smaller $\phi(U^{4+})$ was obtained for *tert*-BtOH with lower I_p. Concerning the photoreduction, the reaction behavior of $^*UO_2^{2+}$ has been found to be similar to those of OH radical [28] and excited carbonyl compounds [29], which would abstract H atom from an α-CH bond of RCH_2OH.
2. Each of the k_q for MeOH, EtOH and iso-PrOH was almost constant in the $[H_2SO_4]$ range, i.e. independent of the environment of $^*UO_2^{2+}$ as observed in H_2O/D_2O [6]. These facts suggest that the surrounding solvent and water molecules as well as the solvating water molecules would not likely affect the coordination of RCH_2OH to $^*UO_2^{2+}$ as discussed previously [1].
3. $\phi(U^{4+})$ at $[RCH_2OH]$ higher than 1.0 mol dm^{-3} reached the same maximum less than unity regardless of the kinds of RCH_2OH. The $\phi(U^{4+})$ values seem to support the physical quenching with probability comparable to that of the chemical quenching. This also suggests that once the complex formed branching ratio into the chemical and physical quenchings, of which $k_c/(k_c+k_p)$ indicates, it would not be affected by the kinds of RCH_2OH in the complex. Further, the $\phi(U^{4+})$ at [EtOH] higher than 0.5 mol dm^{-3} in 3.0 mol H_2SO_4 dm^{-3} [1] was larger than that in 0.5 mol H_2SO_4 dm^{-3} regardless of the constant k_q. These suggest that $k_c/(k_c+k_p)$ would depend only on the environments of the UO_2^{2+} species as discussed below.

3.3. Photoreduction with 1.5 mol ethanol dm^{-3} in acid solutions

In order to discuss the effects of the environment of $^*UO_2^{2+}$ as mentioned above, the photoreduction with a particular quencher of 1.5 mol EtOH dm^{-3} in acid solutions was studied in which the quenching of $^*UO_2^{2+}$ by EtOH is considered to be predominant ($k_0+k_F \ll k_q$ [EtOH]) among the deactivation processes of $^*UO_2^{2+}$ and $\phi(U^{4+})$ which could give $k_c/(k_c+k_p)$ value directly. $HClO_4$, H_2SO_4 and H_3PO_4 were taken as the acid solvents, which themselves were not photo-decomposed to produce by-products and could not quench $^*UO_2^{2+}$, while nitrate ion in nitric acid solution [15] and chlorate ion in hydrochloric acid solution would produce their oxidizing (NO_3 and Cl radicals) and reducing (HNO_2) species. In the case of H_3PO_4, the $\phi(U^{4+})$ was obtained at $[H_3PO_4]$ higher than 3.0 mol dm^{-3} because the resultant U^{4+} exists mainly as neutral phosphate $U(PO_4)_4$ at lower $[H_3PO_4]$ which would be precipitated.

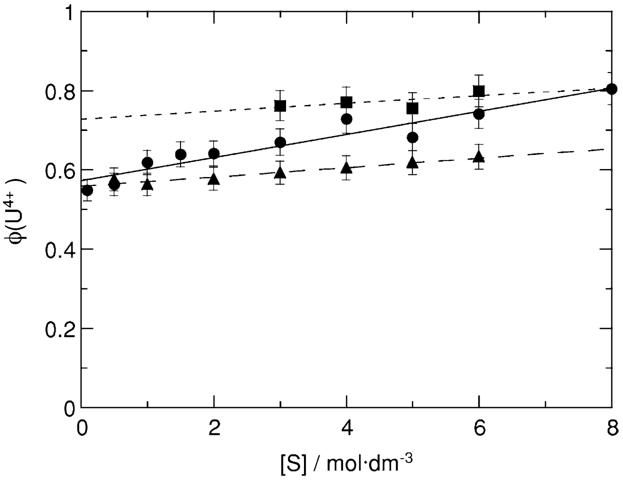

Fig. 3. Photoreduction of UO_2^{2+} in acid solutions: H_2SO_4 (●); $HClO_4$ (▲); H_3PO_4 (■). The solution containing 1.5 mol EtOH dm^{-3} was irradiated with 407 ± 6 nm exciting light at 20 ± 2 °C under deaerated condition.

Fig. 3 shows the $\phi(U^{4+})$ in the deaerated acid solutions. The $\phi(U^{4+})$ in the same acid solution increased with concentration [S] of the acid solvent linearly for H_2SO_4 and slightly for $HClO_4$ and H_3PO_4. Its value at the constant [S] increased in the order of $HClO_4$, H_2SO_4 and H_3PO_4 in the whole range of [S]. These can be explained when water molecules not only solvating but also surrounding $^*UO_2^{2+}$ are assumed to take part in the physical quenching of reaction (6) after a complex $^*[UO_2^{2+} \cdots RCH_2OH]$ is formed, in which an OH oscillator of RCH_2OH encountered with $^*UO_2^{2+}$ would combine with those of their water molecules and then the complex would be deactivated into UO_2^{2+} and RCH_2OH by vibration of their oscillators propagating from the RCH_2OH to the water. As [S] increases, the vibration propagation would be repressed because of decrease in numbers of the solvating and surrounding water molecules, and consequently, the rate constant, k_p, of the physical quenching would decrease and $\phi(U^{4+})$ equal to the $k_c/(k_c+k_p)$ value would increase.

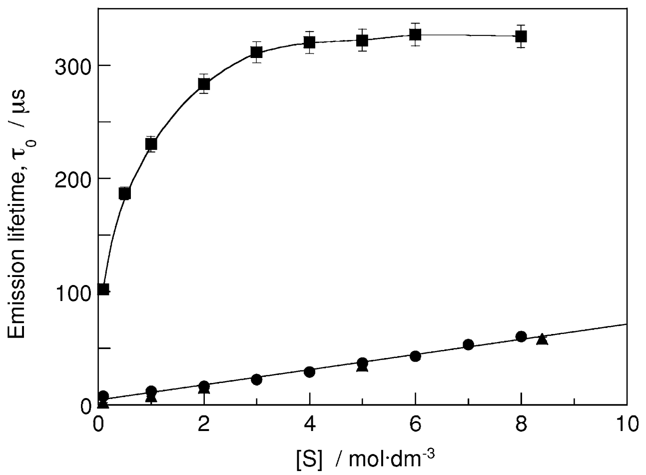

Fig. 4. Lifetimes of $^*UO_2^{2+}$ in acid solutions [18]: H_2SO_4 (●); $HClO_4$ (▲); H_3PO_4 (■). $[UO_2^{2+}] = 2.13 \times 10^{-6}$ mol dm^{-3}. The solution was irradiated with 266 nm laser pulses at 20 °C under aerated condition.

Similar tendency has been reported in lifetimes τ_0 of $^*UO_2^{2+}$ ($\tau_0^{-1} = k_0 + k_F$) in the acid solutions [18] as shown in Fig. 4, which can be explained by the above mechanism. In this case, the non-radiative decay of $^*UO_2^{2+}$ is considered to be due to vibrational relaxation propagating from the solvating water molecules to the surrounding. The non-radiative decay has been considered to be due to the vibrational relaxation of only the solvating water molecules, while this cannot explain linear increases in the τ_0 in $HClO_4$ and H_2SO_4 solutions.

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