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研究課題名(和文) Electron Scattering and Electron Angular Anisotropy in Water and Aqueous Solution

研究課題名(英文) Electron Scattering and Electron Angular Anisotropy in Water and Aqueous Solution

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研究成果の概要(和文)：液体の光電子分光を用いて溶液表面の分子分布と配向が研究されてきたが、分光法の検出深さが決める電子散乱プロセスは明らかでない。本研究は広いエネルギー範囲で溶液中の電子散乱を系統的に測定し、電子散乱と検出深さの関係とスペクトルへの影響を調べた。その結果、電子の運動エネルギーは16 eV以下になると、振動励起の散乱プロセスが支配的になり、スペクトル特徴の劣化によって正確な情報を得られないと判明した。一方、界面活性物質の場合は16 eV以下でも溶媒との非弾性散乱が少ないため、低エネルギー光源を用いても測定可能と分かった。また、電子の角度分布から界面活性物質の深さを原子レベルの分解能での測定に成功した。

研究成果の学術的意義や社会的意義

The distribution of solutes at a solution's surface is important for atmospheric processes and chemistry. This project advanced our understanding of electron scattering in liquids, which enables to extract depth and surface orientation of molecules with high precision via photoelectron spectroscopy.

研究成果の概要(英文)：Liquid-jet photoelectron spectroscopy is routinely used to study the surface propensity and orientation of solutes in aqueous solutions, which relies on the inelastic scattering of the photoelectron within the solvent. However, our understanding of electron scattering in liquids is lacking, which hinders the interpretation of spectra and extraction of depth information. This project succeeded in mapping the electron scattering over a wide range of energies for the first time. It was revealed that vibrational scattering dominates below kinetic energies of ~16 eV, which strongly affects the photoelectron spectrum unless the solute is surface-active. This is important for low-energy experiments, such as laser-based studies. Furthermore, a method of interpreting the angular anisotropy of photoelectrons from molecules with site-specify was developed, which makes it possible to determine a solute's depth and orientation on the solution surface with near atomic precision.

研究分野：物理化学

キーワード：electron spectroscopy electron scattering liquid water aqueous solution angular anisotropy

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1. 研究開始当初の背景

It is of crucial importance to understand the liquid-gas interface of aqueous solutions in the context of chemistry, atmospheric and environmental processes as well as biological systems. Here, the propensity, charge state and surface orientation (e.g., which functional group is exposed) of surface molecules is an important factor in interfacial reactions. The development of the liquid-microjet (LJ) technique enabled the use of the surface-sensitive photoelectron spectroscopy (PES) for studying volatile liquids such as aqueous solutions. PES is utilized to directly measure the chemical properties of species at a solution's surface, and extract the surface propensities and orientations of solute molecules. The former is inferred from signal-intensity measurements, which is a function of the average depth from which photoelectrons can escape as well as a given solute's number density. The molecular orientation is inferred by measuring relative signal intensities from chemically distinct molecular sites or from angular distribution measurements, which exploit the fact that the photoelectron angular distribution (PAD) is inevitably altered by the photoelectron's traversal through the solvent. These methods rely on the photoelectron's scattering behavior on the solvent (water) molecules. Inelastic electron scattering diminishes the PES signal intensity as a function of depth into the solution, from which a solute distribution may be constructed. Furthermore, all scattering processes, including elastic scattering, modify the photoelectron's flight path and thus the PAD, which can be a complementary measure of depth but also molecular orientation. Electron scattering depends on the kinetic energy of the outgoing photoelectron, which in turn can be tuned by changing the photon energy used for ionizing the sample. This is called depth probing (see Fig. 1) and has been widely applied to aqueous solution.

However, at the beginning of the project, the scattering probabilities and scattering mechanisms of electrons on their way through the liquid, specifically liquid water, remained largely unexplored, and average scattering lengths, especially at low kinetic energies, were under debate. The project goal was to systematically study electron scattering and its effect on the PES signal and PADs of solutes and the solvent alike. A further aim of the project was to develop novel PE spectroscopic tools for a detailed measurement of surface-active solutes.

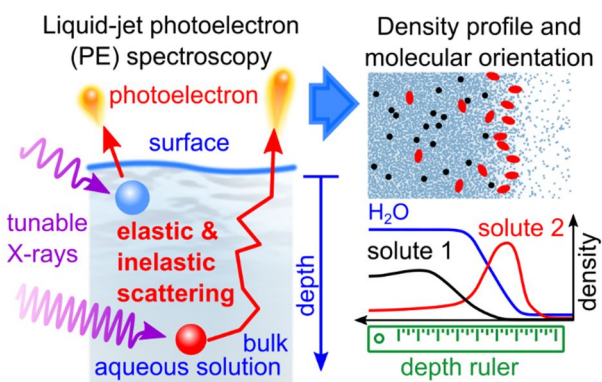


Figure 1: Sketch the measurement process (left), and subsequent extraction of depth and orientation information (right) for solutes in aqueous solution from liquid-jet PES measurements.

2. 研究の目的

The goals of this project were to solve the following problems, which were holding back the study of solute depth distributions with photoelectron spectroscopy:

(1) A systematic study of electron scattering in liquid water over a wide range of kinetic energies, from only a few eV (UV) to keV (soft X-rays), to understand different scattering regimes. High kinetic energies are dominated by electronic scattering processes, such as impact ionization. At low energies, on the other hand, the energy is insufficient for electronic scattering and vibrational scattering processes dominate instead. Both contribute to the average length (mean free path) an electron can travel before scattering occurs. Accurate electron mean free paths for liquid water and aqueous solution are an indispensable foundation of any depth-probing technique.

(2) Quantification of changes in a solute's electron angular anisotropy (i.e., PADs) under the influence of electron scattering. Since scattering changes the photoelectron's flight path and thus modifies any initial (genuine) angular distribution, this information can be exploited to study the detailed depth distribution of the solute. The measurement of PADs is a promising complementary tool to study depth of a solute molecule or even the relative depths of different molecular sites, thus revealing molecular orientation. This requires a good understanding of the effects of electron scattering on PADs in aqueous solution.

(3) Finally, the project aimed to develop the necessary experimental and theoretical tools for a successful quantitative interpretation of PES data. One aspect is theory, i.e., the correct simulation of electron scattering, which is a crucial aid for the interpretation of experimental results. Furthermore, the quantification of subtle scattering effects, such as energy changes on the order of 100 meV from vibrational scattering, requires accurate energy-calibration schemes to be further developed and external influences to be quantified.

3 . 研究の方法

The project requires access to both radiation tunable over a wide range of photon energies, to study scattering as a function of the electron's kinetic energy, as well as tunable light polarization, to study the angular dependency of the electron signal. Both features are available at modern synchrotron facilities. Furthermore, a modern liquid-jet photoelectron spectroscopy apparatus must be employed to be able to measure volatile aqueous solutions, which however is not available in Japan. For this reason, the project was carried out in international collaboration, most prominently with groups in Germany and France.

Most measurements were carried out at the Petra III synchrotron facility (DESY Hamburg) as well as the SOLEIL synchrotron facility (Paris) using state-of-the-art LJ-PES setups at the respective facility. For the low energy regimes, additionally a lab-based UV source was employed. The latter did not have the strict time restrictions of the synchrotron facilities, which made lab measurements best suited for studying a wide range of sample conditions. The liquid samples were usually biased against the setup (ground), both to separate signal contributions from the vapor phase surrounding the quickly evaporating liquid beam from the signal of interest of the solution, as well as enabling accurate energy referencing which was developed in the previous Kaken project.

4 . 研究成果

This project yielded many important results in the field of LJ-PES beyond the focus on electron scattering, but here I summarize the results most relevant to the main goals.

(1) For the first time, a comprehensive study across an energy range of close to zero eV up to 1 keV has been successfully performed; see Figure 2. These experiments have revealed that electron scattering in liquid water has two distinct regimes: non-electronic electron scattering mechanisms, most prominently vibrational scattering, dominate below ~16 eV kinetic energy, and electronic mechanisms such as impact excitation and ionization being the sole contributor at higher energies. The former leads to a significant deterioration of PES signal features and hinders the interpretation of spectra, such as the extraction of solute signal intensities and (binding) energies. This data is an important benchmark for PES studies on aqueous solutions. Yet, we found a distinct difference between bulk solutes and surface-active solutes; an example for the latter are organic molecules with hydrophobic groups. If solutes reside mainly at the surface, photoelectrons do not traverse a thick liquid layer before being expelled, and are thus much less affected by electron scattering.

The magnitude of the effect and the precise photon-energy onset of the electron scattering for surface-active layers was studied on prototypical molecular species such as phenol or methylbenzylamine in solution, with the result that the effect of electron scattering on the solvent is roughly one order of magnitude lower than for a comparable bulk species. Here, the energy loss from scattering is on the order of only one vibrational excitation event. This result is very relevant for PES studies using low-energy light sources, e.g., for laser-based experiments, where surface-active species are being studied most often.

At higher energies, up to keV (X-ray) energies, electron scattering is dominated by electronic processes, which, in the case of liquid water, lead to a minimum loss in energy of several eV thanks to the large energy gap for excitation and ionization in water. The PES signal of scattered electrons is thus well separated from the originating feature, and only minimally impacts the interpretation of spectra. Furthermore, the signal contributions from the electronically scattered electrons can contain valuable details about the scattering target, which may be exploited to study the effect of energy deposition by fast and slow electrons in liquid water and solutions. Results were published in *Chemical Science* and *Accounts of Chemical Research*. A further report, which details the quantitative description of high-energy scattering in liquid water is in preparation.

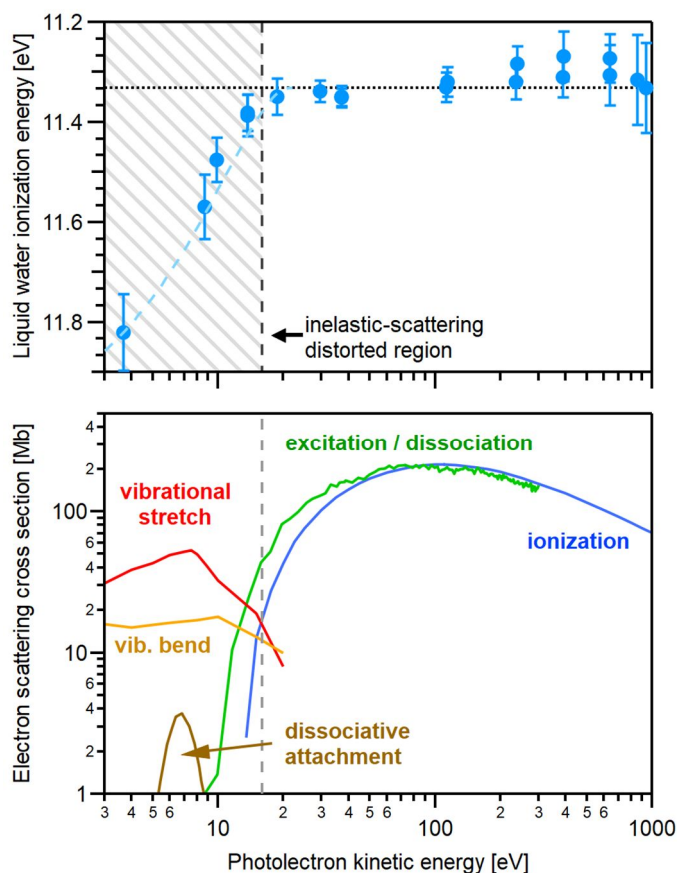


Figure 2: (top) The ionization energy of liquid water appears to change below an electron kinetic energy of ~ 16 eV, which is, however, just an artefact of electron scattering. **(bottom)** Overview of various channels for electron scattering on water (gas phase). The observed ~ 16 eV onset coincides with a distinctive switch from vibrational to electronic scattering mechanisms.

(2) In order to study how molecular PADs are altered by electron scattering and how this can be exploited to gain depth information, several prototypical surface-active molecules were studied. The study revealed that it is possible to obtain depth information on an atomic length scale from core-level PADs, which enables site-selective probing of a molecule. One instructive example is perfluoro-pentanoate (PFP), which is known to orientate perpendicular to the solution surface via repulsion of its hydrophobic chain. The molecule has four carbon sites distinguishable via chemical shift and a solvated functional group containing oxygen; see Figure 3. The photoelectron angular distributions of each molecular site (core-level peak) were measured by rotating the light polarization vector. The resulting PAD, expressed in the anisotropy factor β , was then compared with the gas-phase value, and from this the anisotropy reduction R_β was determined. Reductions in anisotropy are directly related to the amount of scattering the photoelectron experienced. With the help of molecular dynamics simulations this reduction can be quantified and assigned to molecular sites. This yields a precise depth scale on the order of \AA ($=0.1$ nm), and thus the precise depth of the molecule into the solution could be determined. In short, we demonstrated that core-level PADs can be utilized for depth profiling with excellent sensitivity and chemical specificity,

which promises a great leap forward for determining quantitative solute depth profiles and solute propensities at a solutions surface. These results are published in *Physical Review Letters* and *Accounts of Chemical Research*.

(3) The project also helped to drive several technological advances in the field of LJ-PES. For example, one major drawback of the cylindrical liquid microjet, employed so far PES experiments, is its curved surface. The curvature averages out takeoff angles of the photoelectrons, which complicates interpretation of depth profiles and especially the angular anisotropy information. A novel jet design, featuring a flat surface (a so-called flat jet), was introduced and tested for the first time for the use with PES; these results are published in the *Journal of Chemical Physics*. This new design, thanks to a defined surface normal, promises to enable studies of the solution surface as a function of surface orientation.

Furthermore, the quantification of crucial surface properties, such as the solution's surface potential barrier (via the measurement of the work-function), was enabled by controlling any detrimental sample charge-up, which inevitably happens with the flowing liquid jet. Such charge up alters the photoelectron's kinetic energy, which in turn usually prevents the accurate energy referencing necessary to determine subtle changes in work function. By overcoming this limitation, we were able to measure changes of the solution's work function as a function of solute concentration for the first time. Results were published in *Chemical Science*.

Overall, the project has expanded the scope and applicability of PES to extract precise solute depth profiles as well as deepened the understanding of electron scattering phenomena in aqueous solution.

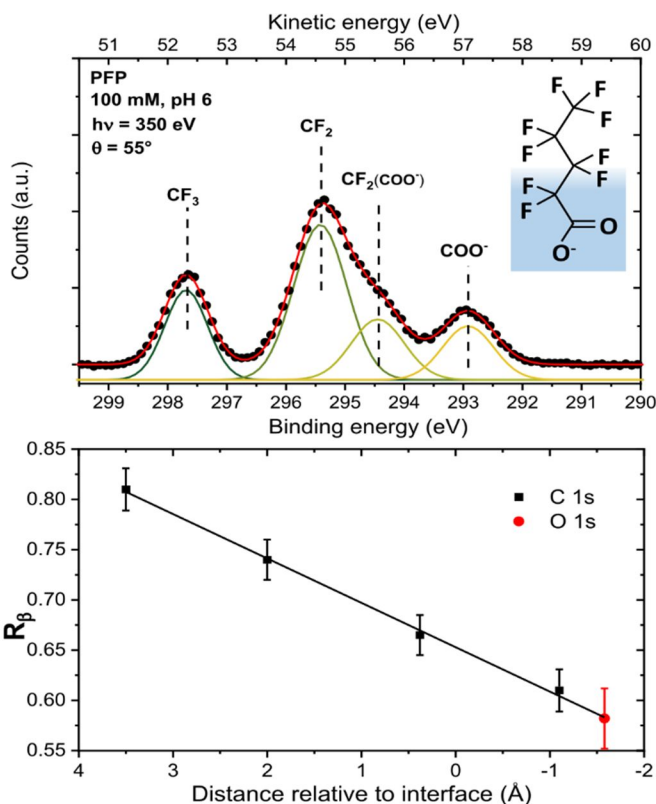


Figure 3: (top) Carbon 1s PES of PFP. Four chemically distinct molecular sites can be identified (compare with the molecular sketch in the inset). **(bottom)** Change in the PAD of PFP in the liquid vs. gas phase R_{β} . A larger reduction of R_{β} implies more scattering and thus depth into the solution.

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〔図書〕 計0件

〔産業財産権〕

〔その他〕

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6. 研究組織

	氏名 (ローマ字氏名) (研究者番号)	所属研究機関・部局・職 (機関番号)	備考
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7. 科研費を使用して開催した国際研究集会

〔国際研究集会〕 計0件

8. 本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関
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