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In-situ X-ray Absorption Study of Oxidation State Changes of Gold in Aqueous Solutions at Different pH Conditions.

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1. 背景と研究目的

Gold is one of the premier materials for soft bioelectronics, like neural probes, owing to its biocompatibility, long-term stability and high electrical conductivity. However, the development of soft and stretchable gold-based bioelectronics is hampered by the challenges of synthesizing high aspect ratio gold nanomaterials in a scalable manner. In this context, a detailed understanding of gold coordination in solution is essential to achieve controlled reduction. The nature of the ligands coordinating Au (e.g., Cl^-, OH^-, or sulfite) directly governs its effective oxidation state, redox potential, and reduction kinetics. Precise control over Au coordination enables the decoupling of homogeneous reduction from nucleation, thereby favoring surface-confined processes such as epitaxial growth or directional deposition. Consequently, elucidating Au speciation and coordination is not merely a fundamental chemical consideration, but a key design principle for scalable synthesis routes toward high-aspect-ratio gold nanostructures with well-defined morphology and structural integrity.

2. 実験内容

The samples were measured after the addition of HCl in order to induce controlled changes in gold coordination. All measurements were performed in a flow cell under continuous agitation to ensure homogeneous mixing and to minimize beam-induced photoreduction effects. X-ray absorption scans were intentionally kept short (10 s per scan) to allow time-resolved monitoring of coordination and oxidation-state changes. For each sample, 40–60 consecutive spectra were collected and analysed to follow the evolution of gold speciation during the reaction.

3. 結果および考察

As the pH decreases upon gradual addition of HCl, the contribution from Au–OH coordination progressively diminishes. This is evidenced by systematic changes in both the near-edge and extended regions of the X-ray absorption spectra, indicating ligand exchange and destabilization of hydroxyl-coordinated Au species. Concomitantly, sulfite coordination becomes more dominant, accompanied by changes in the Au electronic structure and partial reduction at lower pH. These results demonstrate that acidification suppresses Au–OH complexation and drives the evolution of gold speciation through sulfite-mediated coordination pathways.

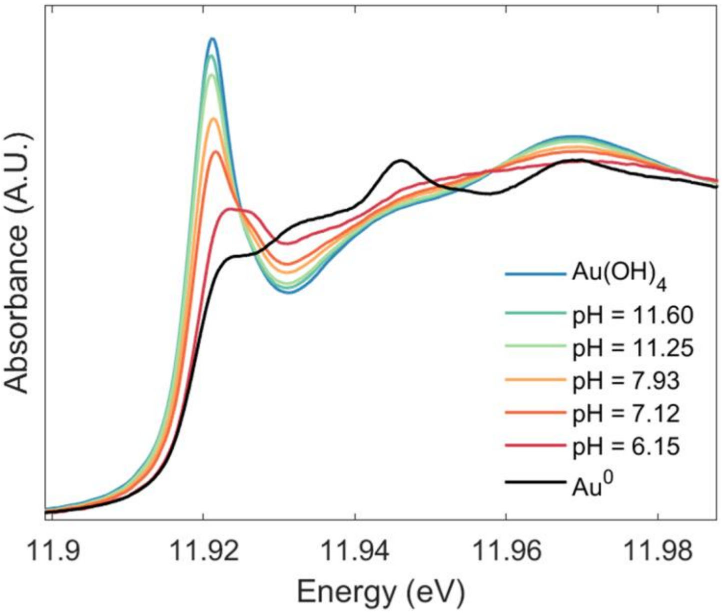

Fig 1 - XANES spectra at different pHs.