Soft X-ray absorption spectroscopy of cyclodextrin compounds including a noble metal atom

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Introduction

An environmentally friendly method for gold recovery is recently proposed as simple as mixing $KAuBr_4$ and cyclodextrins [1]. A 1:2 ratio of $KAuBr_4$ and α-CD forms gold-containing nanowires that precipitate immediately, whereas β- and γ-CD do not. The different binding positions of K to CD is considered to be determine whether nanowire formation occurs or not by the results of crystallographic analysis [1]. To investigate the difference in the binding of K to each CD in aqueous solution, soft X-ray absorption spectroscopy of the K2p absorption edge was performed at the beamline BL-6 of HiSOR.

Experiment

A mixture of $KAuBr_4$ aq. (2 mM) and CD aq. (4 mM) was prepared and the measurements were carried out on drop-dried films. The solution was dropped onto a stainless plate and vacuum dried. This process was repeated several times to obtain a K-containing thin film with sufficient concentration to be used for drain current measurement. The sample was placed in a vacuum chamber, and synchrotron radiation was incident at an angle of 45° to the normal of the sample. The obtained signals were normalized by incident light intensities.

Results and Discussion

First, solid samples of KCl was measured for energy calibration (Figure 1). The obtained spectrum shows two well-resolves peaks for each 2*p* spin-orbit component. Crystal-field splitting, T_{2g} and E_g, in crystals with cubic symmetry is observed in each 2*p* peak. The spectrum is good agreement with the previous study [2] and the energy axis is calibrated at $2p_{3/2} \rightarrow 3d$ (296.461 eV) and $2p_{1/2} \rightarrow 3d$ (299.141 eV) peaks. Figure 1 also shows the spectra of $KAuBr_4$ and a 1:2 mixture of $KAuBr_4$ and α-, β-, and γ-CD in the films.

The spectrum of $KAuBr_4$ has a similar shape to that of KCl. Although the respective peak widths are broadened, the peak energies of $2p_{3/2}$ and $2p_{1/2}$ are almost the same. On the other hand, in the spectrum of the cyclodextrin mixture, the respective spin-orbit peaks become broader. The difference between the α-, β-, and γ-CD spectra is small. This may be due to the formation of bonds between various sites of cyclodextrins and K in the liquid, which disrupted the crystalline field around K.

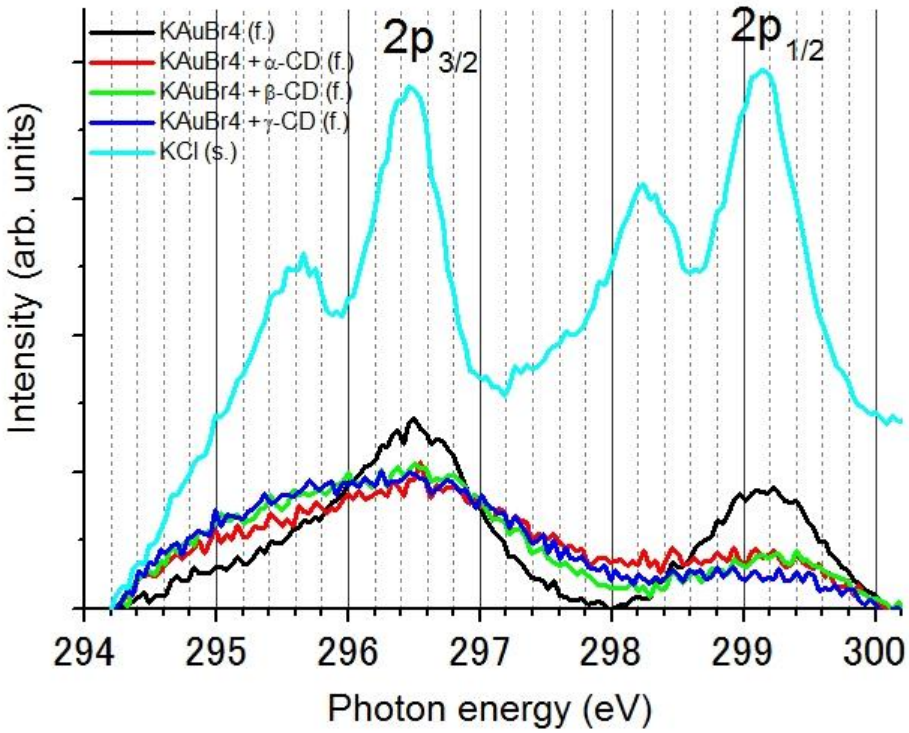

Figure 1 XAS spectra of KCl(solid), $KAuBr_4$(film), $KAuBr_4$+α-CD(film), +β-CD(film), and +γ-CD (film) at the K2p edge.

REFERENCES

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