

## EXAFS analysis of Precursors for RhTe nanoparticles Evidence for Rh-Te interaction in the precursor state

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### Introduction

The palladium tellurium (PdTe) bimetallic works as the catalyst of oxidative diacetoxylation reaction of butadiene. In our previous study, PdTe nanoparticles were prepared by liquid-phase reduction and the resulting nanoparticles had homogeneous composition and size. In this study, we have applied the liquid phase reduction method to prepare rhodium tellurium (RhTe) nanoparticles. We have measured EXAFS spectra to determine the structure of RhTe. In this paper, we investigate the condition of precursors in reaction solution.

### Experimental

The EXAFS measurements were carried out at NW10A of PF-AR with 6 GeV – 50 mA. The X-rays were monochromatized with a Si(311) double crystal monochromator. The monochromatized X-ray was focused to the sample by a Pt-Coated bent cylindrical mirror. The critical energy for the total reflection of the mirror was set at 42 keV. The  $I_0$  and  $I$  signals were measured by Ar filled ionization counters. The RhTe nanoparticles were prepared by the liquid-phase reduction method. In order to investigate the condition of Rh and Te in the reaction solution, Rh and/or Te precursor and complex agent were dissolved into ion-exchanged water and the pH of the solution was fixed in 13.

### Results and discussion

Figure 1 shows Rh EXAFS oscillation in solution. In the cases of adding PAR, PAR-Citric acid and PAR-Citric acid-Te into Rh solution, several peaks were observed. The first peak appeared at 0.16 nm and corresponded to the Rh-O distance. From this result, the ligand of Rh precursor would be changed from Cl to PAR. When Te precursor was dissolved into the solution, the strength of the peak at 0.27 nm, which corresponded to Rh-Rh or Rh-Te distance, was larger than others. This result shows increase of the amount of Rh or coordination of Te to Rh. In order to investigate this phenomenon, Te EXAFS measurement was carried out.

Figure 2 shows Te EXAFS oscillation in solution. In the case of adding Te precursor and citric acid, the first peak at 0.15 nm appeared and corresponded to Te-O distance. Te will be stabilized by forming Te-citric acid complex or coordinating citric acid to Te oxide. When Rh precursor was added into the solution, the peak at 0.26

nm showed coordination of Rh to Te. It follows from this result that the growth of the peak at 0.27 nm in Fig. 1(3) due to coordination of Te to Rh.

In conclusion, Rh and Te FT-EXAFS oscillations of Rh-PAR-Citric acid-Te solution show the formation of dinuclear complex of Rh and Te. We are planning to obtain the RhTe nanoparticles on silica support by liquid-phase reduction of the solution and carry out in-situ EXAFS measurement of the reaction.

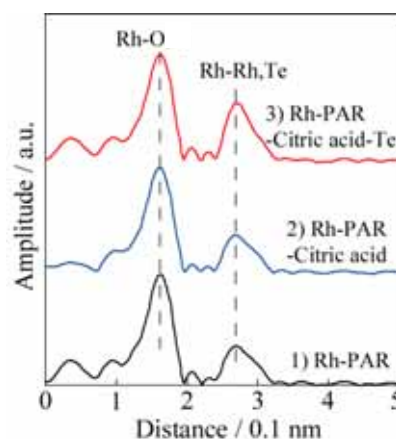


Figure 1. Rh K-edge FT-EXAFS of 1) Rh-PAR, 2) Rh-PAR-Citric acid, and 3) Rh-PAR- Citric acid-Te.

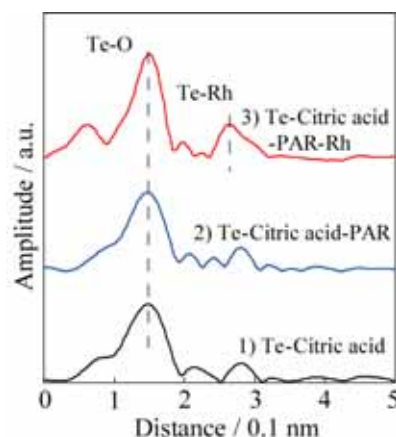


Figure 2. Te K-edge FT-EXAFS of 1) Rh-PAR, 2) Rh-PAR-Citric acid, and 3) Rh-PAR- Citric acid-Te.

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