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Synthesis of Colloidal Particles of Poly(2-vinyl pyridine)-coated Palladium and Platinum in Organic Solutions under the High Temperatures and High Pressures

Masafumi HARADA*[1], Masaki UEJI[2], and Yoshifumi KIMURA[3]
[1]Department of Clothing Environmental Science, Faculty of Human Life and Environment, Nara Women's University, Nara 630-8506, Japan
[2]Department of Chemistry, Graduate School of Science, Kyoto University, Kyoto 606-8502, Japan
[3]International Innovation Center, Kyoto University, Kyoto 606-8501, Japan

Introduction

Palladium and platinum are most commonly used in the catalysis field, and preparation of small metal particles is required to achieve the high catalytic activity. In this study we present the synthetic approaches of monometallic Pd, monometallic Pt, and bimetallic Pd/Pt particles by using chemical reductions of palladium acetylacetonate ($Pd(acac)_2$) and platinum acetylacetonate ($Pt(acac)_2$) in the presence of poly(2-vinyl pyridine) (P2VP) in the mixture solution of toluene and 1-propanol under high temperatures and high pressures. For the structural analysis of the particles, EXAFS measurements have been carried out. By changing reactants and solvents from previous study [1, 2], we expect the variety of the regulation of the particle size and distribution.

Experimental

The colloidal dispersions of Pd, Pt, and Pd/Pt particles were synthesized by the following procedure. In the case of Pd particles, the solutions of a 1:1 mixture of $Pd(acac)_2$ toluene solution and P2VP 1-propanol solution were introduced into a high-temperature and high-pressure reactor in a few seconds (25 MPa, 473-623 K) by HPLC pump. The concentration of $Pd(acac)_2$ and P2VP is 7.5 mM and 7.5 g/L in the reactant solutions (volume ratio = 1:1 of toluene:1-propanol), respectively. The colloidal dispersions of Pt and Pd/Pt particles were prepared by the same method. EXAFS measurements of Pd-K and Pt-L_3 edge of the colloidal dispersions were carried out in a transmission mode at BL-NW10A and 7C, respectively. The obtained colloidal dispersions of metal particles were concentrated ([M]=30mM) for each metallic element. The colloidal dispersions were then poured into glass cells for EXAFS measurements. In order to estimate the precise coordination number of atoms around an absorbing atom, measurement of Pd, Pt, and Pd/Pt(1/1) alloy foils was performed as a reference compound.

Results and Discussion

Figs. 1(a) and 1(b) show the Fourier-transforms for the concentrated colloidal dispersions of Pd and Pt particles prepared at various temperatures (473K, 533K, 573K, and 623K) and 25 MPa, as well as the reactant solution and foil of each elemental component, respectively. In the case of Pd-K edge, the peak of the reactant $Pd(acac)_2$ solution between 1 and 2 Å is assigned to the Pd-O bond, and it completely vanishes when the colloidal dispersions of Pd particles are produced above 473K. All the Pd colloidal dispersions show the same peak position as Pd foil, whose peak is assigned to the Pd-Pd bond. As shown in Fig. 1(b), the Pt samples produced below 573K still have a peak between 1 and 2 Å, which is assigned to the Pt-O bond of the reactant $Pt(acac)_2$ solution. The sample produced at 623K has main peak and its shoulder peak between 2 and 3 Å, which is observed due to the Pt-Pt bond of Pt foil. In addition, EXAFS results indicate that bimetallic Pd/Pt particles were not synthesized at 473-623 K and 25 MPa, mainly due to the difference of decomposition rate of $Pd(acac)_2$ and $Pt(acac)_2$.

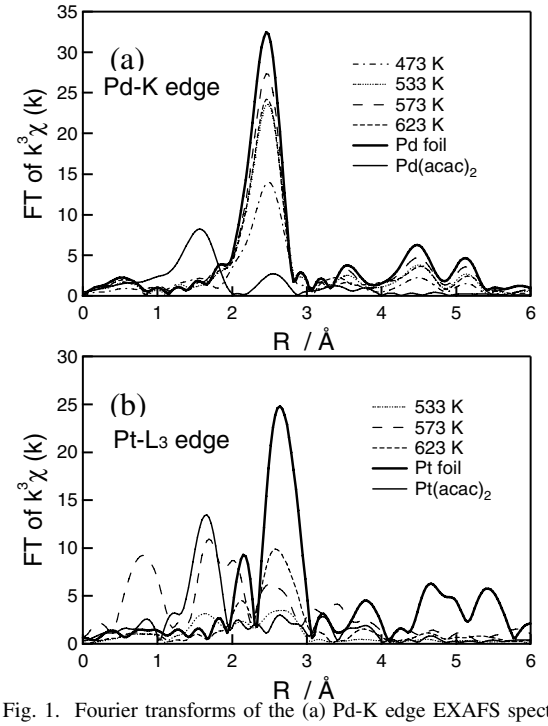

Fig. 1. Fourier transforms of the (a) Pd-K edge EXAFS spectra of the concentrated Pd colloidal dispersions and (b) Pt-L_3 edge EXAFS spectra of the concentrated Pt colloidal dispersions.

References

[1] Y. Kimura et al., *Colloids and Surfaces A: Physicochem. Eng. Aspects* **231**, 131 (2003).
[2] M. Harada et al., *J. Colloid Interface Sci.* **292**, 113 (2005).

* harada@cc.nara-wu.ac.jp