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# Preparation of small and highly dispersed nano-sized Pt particles on $TiO_2$ by microwave heating

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## Introduction

The photocatalytic production of $H_2$ from chemical wastes to store solar energy by converting light energy into chemical energy is widely investigated. Noble metals (e.g., Pt, Pd, Ru and Rh) as co-catalyst supported photocatalysts are frequently utilized for this purpose. It is known that the metal particle size and size distribution greatly affect the catalytic activity. In order to produce $H_2$ with high efficiency, it is necessary to develop the novel technique to simply prepare small and highly dispersed metal particles supported photocatalyst, which finally lead a reduction in consumption of expensive noble metals. Recently, microwave dielectric heating has attracted a great deal of attention as a promising method for preparation of small and uniform metal nanoparticles. In this study, we offer a novel technique to prepare small and highly dispersed Pt nanoparticles on $TiO_2$ using uniform and rapid heating of the microwave heating.

## Experimental

The commercial anatase $TiO_2$ powder was suspended in an aqueous solution of $H_2PtCl_6$ in a flask. The flask was irradiated by microwave (500 W, 2450±30 MHz, MWO-1000S, Tokyo Rikakikai Co. Ltd.) for 15 min with stirring. After the filtration and washing with distilled water, the resultant powder was dried at 333 K overnight under air and then reduced by $H_2$ (20 $cm^3$ $min^{-1}$) at 473 K for 2 h, resulting in the formation of Pt-loaded-$TiO_2$ (Mw-Pt-$TiO_2$). In order to compare the conventional preparation techniques, the Pt deposition on $TiO_2$ was also performed by the photo-assisted deposition method (PAD-Pt-$TiO_2$), equilibrium adsorption method by conventional heating (EA-Pt-$TiO_2$) and the impregnation method (Imp-Pt-$TiO_2$). CO pulse adsorption was performed to measure Pt dispersion and particle size by BEL-METAL-1 (BEL Japan, Inc.). The Pt $L_{III}$-edge X-ray absorbance fine structure (XAFS) spectra of these catalysts were measured in fluorescence mode.

## Results and discussions

The dispersion and particle size of Pt metals of Mw-, PAD-, EA- and Imp-Pt-$TiO_2$ calculated by the pulsed CO adsorption measurement increased in the order of Imp < EA < PAD < Mw and Mw < PAD < EA < Imp, respectively. Figure 1 shows Pt $L_{III}$-edge XAFS spectra of these Pt supported $TiO_2$. The XANES spectra of all samples are similar to that of Pt foil, suggesting the presence of Pt in metal state. In the Fourier transforms of Pt $L_{III}$-edge EXAFS spectra, all samples exhibited a peak at approximately 2.7 Å due to the contiguous Pt–Pt bond in the metallic form nanoparticles. The peak intensity decreased in the order of Imp > EAD ≈ PAD > Mw, which is due to the smaller particle size of the Pt. The order of peak intensity gave close agreement with the order of the Pt particle size calculated by the pulsed CO adsorption measurement. In addition, it was also found that Mw-Pt-$TiO_2$ exhibited stable and high $H_2$ production activity compared with samples prepared using the conventional methods in photocatalytic $H_2$ production reaction from chemical wastes. The generation rate of $H_2$ increased with increase in the dispersion of Pt. These results indicate that Mw method is the most promising way to support small and highly dispersed Pt particles on $TiO_2$ and the small and highly dispersed Pt particles is important factor to produce $H_2$ with high efficiency in photocatalytic reaction.

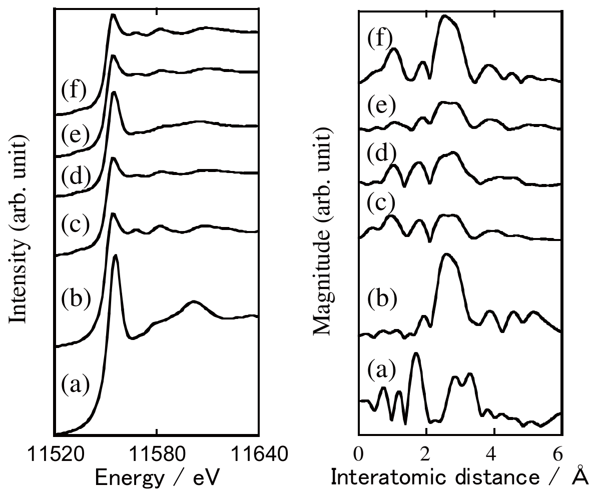


Figure 1 Pt $L_{III}$-edge XANES spectra (left) and FT-EXAFS spectra (right) of (a) $PtO_2$, (b) Pt foil, (c) Mw-Pt-$TiO_2$, (d) PAD-Pt-$TiO_2$, (e) EA-Pt-$TiO_2$ and (f) Imp-Pt-$TiO_2$.

## Conclusions

The small and highly dispersed Pt nanoparticles could be supported on $TiO_2$ by the Mw method and the Mw-Pt-$TiO_2$ exhibited high photocatalytic $H_2$ production activity from chemical wastes. The easy-to-use and energy-saving preparation of Pt nanoparticles on $TiO_2$ to efficiently produce $H_2$ by photocatalytic reaction was achieved using the Mw heating.

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