

# In-situ XAFS characterization of sprayed Mo/SiO<sub>2</sub> catalyst during propene photometathesis

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## Introduction

Information on the structure of active sites under reaction conditions is crucial for developing new catalysts. XAFS has been one of the most promising techniques to analyze the catalyst structure under *in-situ* conditions.

It was reported that the isolated molybdenum ions on silica support exhibit a higher activity for the photometathesis of propene [1]. So the bulk Mo oxide seemed to have no photocatalysis. However, we have reported that Mo oxide catalyst prepared by the spray reaction method showed photocatalytic activity toward propene photometathesis [2]. In this study, we elucidate the differences in the catalyst structures in the course of photo-catalytic reaction using *in-situ* XAFS technique.

## Experimental

Sprayed Mo/SiO<sub>2</sub> catalyst (5wt%) was prepared from MoO<sub>3</sub>, dissolved in ammonium solution, and SiO<sub>2</sub> (Aerosil #200). The above suspension was ultrasonicated and aspirated through the furnace (573 K) in air.

Mo K-edge in-situ XAFS spectra were collected at BL-10B of the Photon Factory with Si(311) channel cut monochromator. Catalysts were pressed into self-supporting disks and settled in the cell. The cell was made from quartz glass and has two acrylic windows, so the EXAFS spectra can be measured under photometathesis reaction. Irradiation was carried out by using 75 W high-pressure Hg lamp (Toshiba SHL-100UV).

## Results and discussion

Figure 1 shows the XANES spectra of Mo/SiO<sub>2</sub> catalysts. Each spectrum was collected in 10 min. Catalysts were treated under following conditions; (1) UV irradiation with N<sub>2</sub> flow, (2) UV irradiation with N<sub>2</sub> and C<sub>3</sub>H<sub>6</sub> flow and (3) O<sub>2</sub> flow with heating to 673 K. Figure 2 shows the relative intensity of 1s-4d pre-edge peak height normalized by that of pretreated sample. The intensity of this peak was gradually reduced from 1 to 0.9 under photo-irradiation (Fig. 2 (1)). By introducing C<sub>3</sub>H<sub>6</sub> under this condition, the pre-edge intensity became smaller from 0.9 to 0.83 (Fig. 2 (2)). These results suggest the conversion of Mo<sup>6+</sup>=O<sup>2-</sup> to Mo<sup>5+</sup>-O<sup>-</sup> by photo-excitation (Fig. 2 (1)) and the more change in Mo site by introducing propene under irradiation (Fig. 2 (2)). These changes in Mo site were fully recovered by oxidation at

673 K (Fig. 2 (3)). The structural change in the photo-catalytic reaction was clearly observed by this technique.

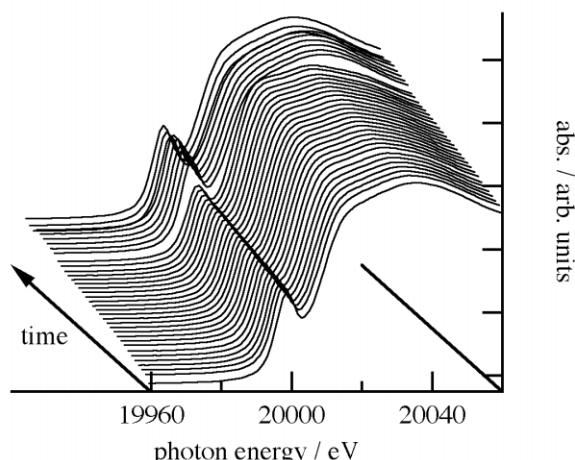


Fig. 1. XANES spectra for sprayed Mo/SiO<sub>2</sub> catalyst in the course of propene photometathesis reaction.

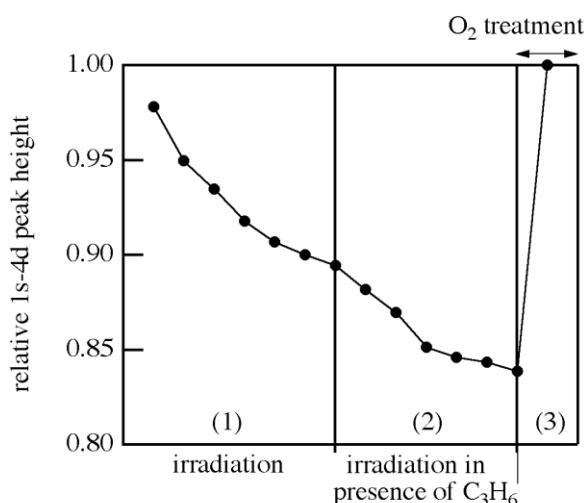


Fig. 2. Relative intensity of 1s-4d pre-edge peak height normalized by that of the pretreated sample.

## References

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- [2] H. Murayama *et al.*, *Stud. Surf. Sci. Catal.*, **132**, 793 (2001).