Characterization of Pd-Ni/SiO_2 Catalysts Prepared from Organometallic Cluster Precursor

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Jatuporn Wittayakun* and Jarunan Wongwiwattana
School of Chemistry, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima, 30000, Thailand

Tohru Mori and Yutaka Morikawa
Chemical Resource Laboratory, Tokyo Institute of Technology, Nagatsuta Campus, Yokohama, Japan

ABSTRACT

The Pd-Ni/SiO_2 catalysts were prepared by impregnation of two precursors on silica: an acetonitrile solution of $[PPh_4]_4[Pd_{13}Ni_{13}(CO)_{34}]$ (**1**) and a mixture of palladium and nickel salts. Both catalysts were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), BET surface area analysis, and temperature-programmed reduction (TPR). In addition, the metal loading was determined by ICP and the number of active sites was measured by hydrogen adsorption experiment. The cluster's carbonyl ligands were removed by heating at the 80°C for 10 minutes while the counter cations, $[PPh_4]^+$ on were removed by heating at 400°C. XRD peaks of Pd-Ni/SiO_2 prepared from the cluster shifted slightly possibly due to Pd-Ni alloy formation. From TPR experiment, it was found that the reduction temperature of Ni in the Pd-Ni/SiO_2 catalysts prepared from cluster was significantly lowered than that of the Ni/SiO_2 reference indicating that Pd and Ni were still intact. When the two metals formed Pd-Ni alloy, some Pd coordination sites were taken by Ni and lowered the hydrogen adsorption sites. As a result, Pd-Ni/SiO_2 catalysts prepared from salt solution adsorbed hydrogen better than that from cluster.