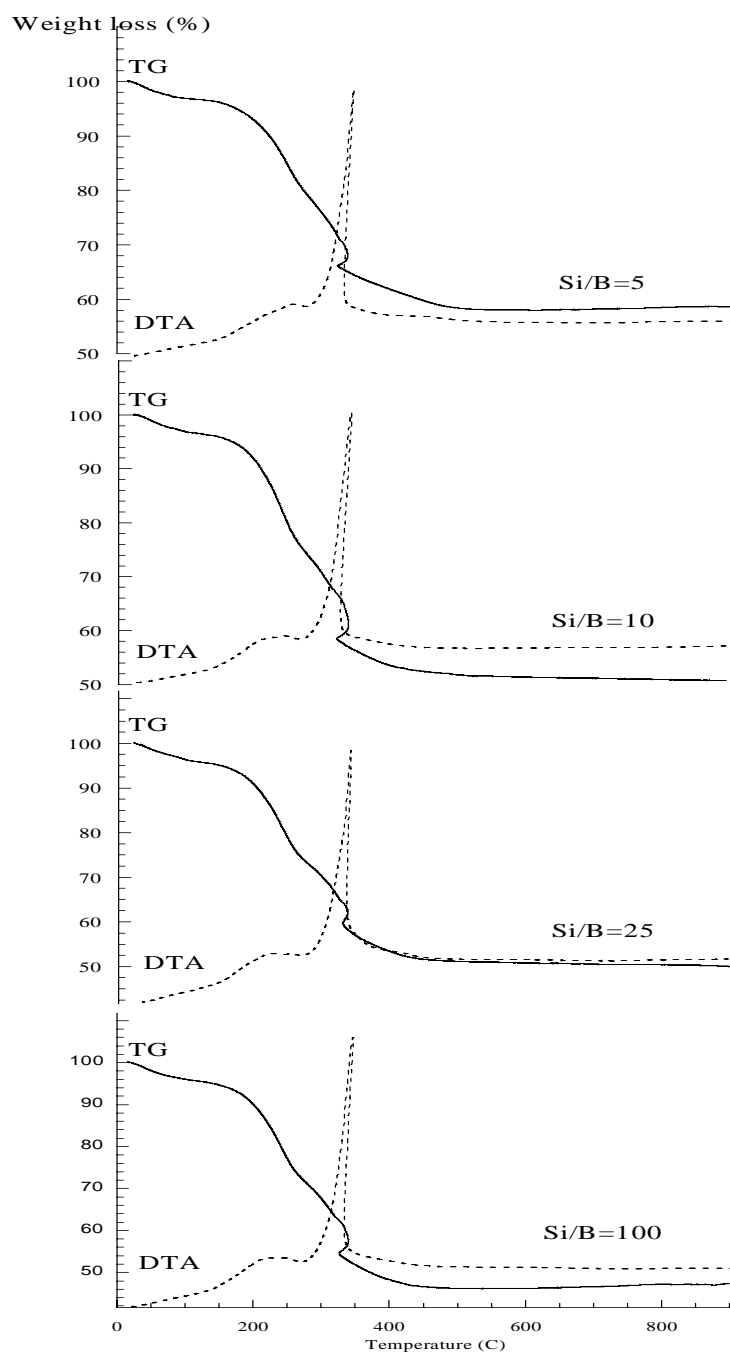


***In-situ* synthesized Pd nanoparticles supported on B-MCM-41: An efficient catalyst for nitroaromatics hydrogenation in supercritical carbon dioxide**

Maya Chatterjee,* Takayuki Ishizaka, Toshishige Suzuki, Akira Suzuki, Hajime Kawanami*

Research Center for Compact Chemical System, AIST Tohoku, 4-2-1 Nigatake, Miyagino-ku, Sendai, 983-8551, Japan, Tel.: +81-22-237-5213, FAX: +81- 22-237-5388; E-mail: c-maya@aist.go.jp; h-kawanami@aist.go.jp

Fig. 1s: Thermogravimetric analysis of Pd/B-MCM-41 of different Si/B ratio



Phase change observations

Method: Visual observation of phase behaviour of nitrobenzene in scCO₂ under the studied reaction condition was conducted separately using a 10 ml high pressure view cell fitted with a sapphire window. The cell was placed over a magnetic stirrer for stirring the content and connected to a pressure controller, to regulate the pressure inside the view cell. In addition, temperature controller was used to maintain the desired temperature of 50 °C. In the beginning, nitrobenzene was introduced into the view cell (mentioned as 'initial' in the Fig. 2s) at a constant H₂ pressure of 2 MPa while CO₂ pressure was varied in the range of 7-14 MPa and the phase behaviour of nitrobenzene-H₂-CO₂ system was monitored.

Fig.2s: Phase change observations of nitrobenzene at 50 °C and P_{H₂}= 2. MPa (a) initial (b) 8, (c) 10 and (d) 12 MPa of CO₂ pressure.

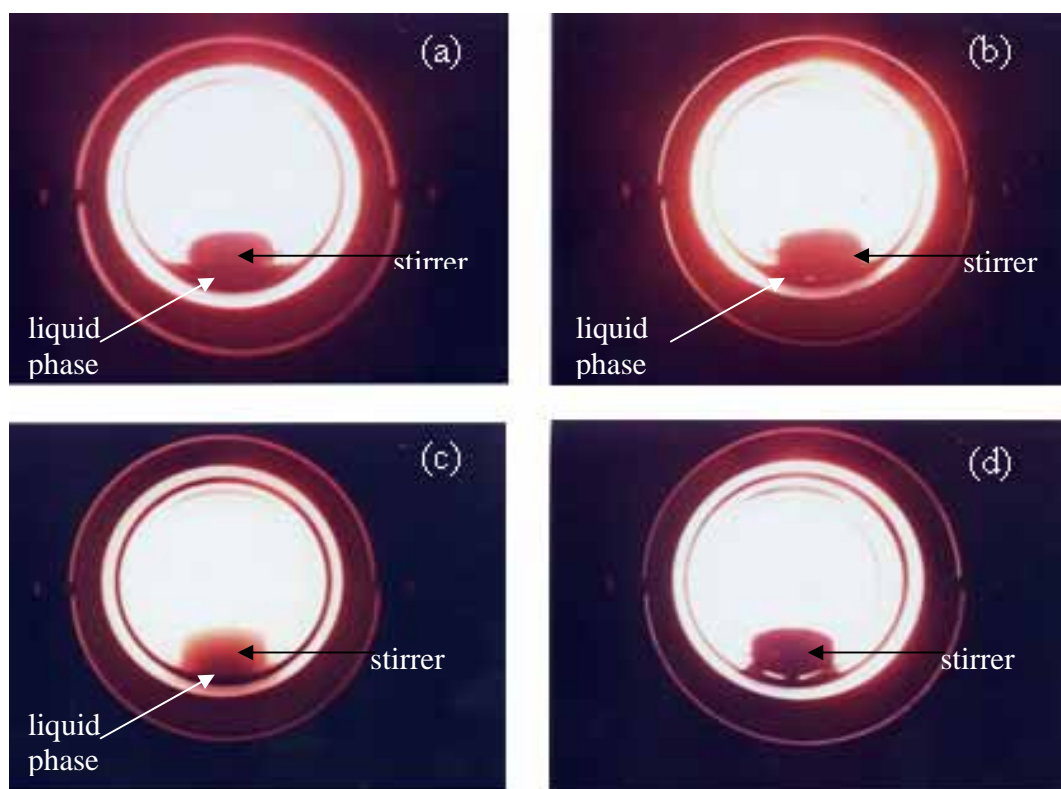


Fig. 3s: XRD pattern of Pd/Al-MCM-41 and Pd/Ga-MCM-41 (a) low angle and (b) higher angle

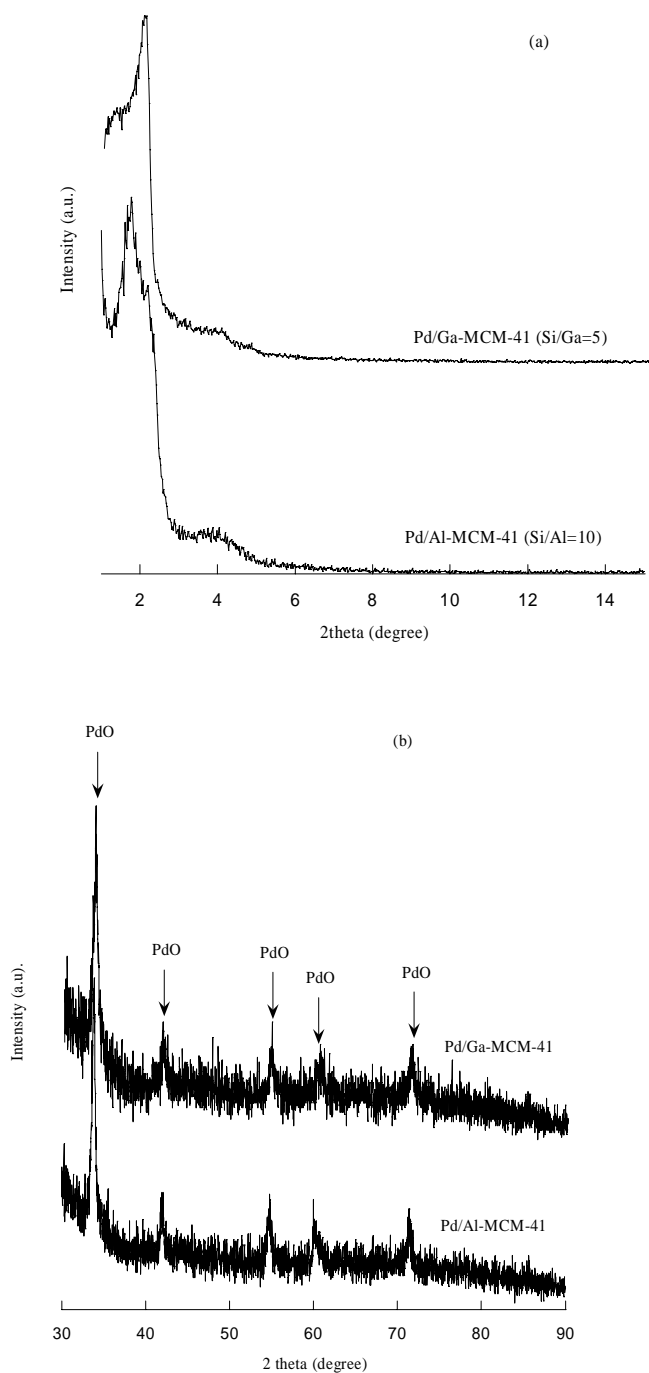


Fig. 4s: TEM image after (a) 4th and (b) 8th recycle. Before recycling the catalyst was separated from product by filtration.

