SUPERCRITICAL CO_2 IMPREGNATION TO PREPARE HIGHLY DISPERSED METAL NANO-PARTICLES/POROUS SUPPORT CATALYST: COBALT/MESOPOROUS SILICA SYSTEM

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Mesoporous silica has attracted considerably much attention due to their advanced applications such as gas sorption, inorganic fillers and catalytic supports. These promising applications can be attributable to the unique properties of mesoporous silica such as large surface area and uniform distribution of pore size. Recently, the mesoporous silica containing metal nanoparticles inside its micropores has received significant interest as hybrid functional material or effective catalyst. Several methods to synthesis these composite materials have been developed, wet-impregnation, ion exchange, sol-gel methods etc. Although there have been reported some examples of successful dispersions of metal particles in pores of silica by these methods It has been pointed the difficulties in penetrating metal precursors into mesopores due to low diffusivity and high surface tension of liquid solvents.

We have applied supercritical CO_2 impregnation technique to synthesize cobalt nanoparticles in mesoporous silica using methanol as an entrainer under temperatures from 313 to 333 K, pressures from 10 to 20 MPa and methanol concentrations from 10 to 30 mol%. The Cobalt particles were highly dispersed and their loading amount in the materials was quantified by energy-dispersive X-ray spectroscopy and was revealed to reach 3.8 wt% at maximum after calcinations,,at 333 K, 20 MPa, a methanol concentration of 10 mol% and a fixed reaction time of 5 h. A repeated treatment under the optimized condition improved the Co loading up to 6.6 wt%, where the Co particles observed to be in the mesopores silica by TEM.