

## **Direct Formation of Thermally Stabilized Amorphous Mesoporous Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> Nanocomposites by Hydrolysis of Aqueous Iron (III) Nitrate in Sols of Spherical Silica Particles**

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Nanocomposite materials containing 10% and 20% iron oxide/silica, Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> (w/w), were prepared by direct hydrolysis of aqueous iron (III) nitrate solution in sols of freshly prepared spherical silica particles (Stöber particles) present in their mother liquors. This was followed by aging, drying, calcination up to 600 °C through two different ramp rates, and then isothermal calcinations at 600 °C for 3 h. The calcined and the uncalcined (dried at 120 °C) composites were characterized by thermogravimetric analysis, differential scanning calorimetry, Fourier transform infrared spectroscopy, X-ray diffraction (XRD), N<sub>2</sub> adsorption/desorption techniques, and scanning electron microscopy as required. XRD patterns of the calcined composites showed no line broadening at any *d*-spacing positions of iron oxide phases, thereby reflecting the amorphous nature of Fe<sub>2</sub>O<sub>3</sub> in the composite. The calcined composites showed nitrogen adsorption isotherms characterizing type IV isotherms with high surface area. Moreover, surface area increased with the increasing of the iron oxide ratio and lowering of the calcination ramp rate. Results indicated that iron oxide particles were dispersed on the exterior of silica particles as isolated and/or aggregated nanoparticles. The formation of the title composite was discussed in terms of the hydrolysis and condensation mechanisms of the inorganic Fe(III) precursor in the silica sols. Thereby, fast nucleation and limited growth of hydrous iron oxide led to the formation of nanoparticles that spread interactively on the hydroxylated surface of spherical silica particles. Therefore, a nanostructured composite of amorphous nanoparticles of iron oxide (as a shell) spreading on the surface of silica particles (as a core) was formed. This morphology limited the aggregation of Fe<sub>2</sub>O<sub>3</sub> nanoparticles, prevented silica particle coalescence at high temperatures, and enhanced thermal stability.