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Pore hierarchy control in SiO2/Nb2O5 monolithic composites obtained by Spinodal decomposition

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Niobium pentoxide and silica have synergic properties for making composites. Good catalytic and adsorbent properties are achieved due to acidic sites of niobium pentoxide particles, enhanced by the silica high specific surface area (SSA). At this work we controlled the textural properties of SiO2/Nb2O5 monoliths using a soluble polymer, PEO 10000, to induce a phase segregation during the gelification of the sol (spinodal decomposition). The influences of the polymer concentration and temperature annealing in the porosity, SSA, pore size distribution (nitrogen gas adsorption), morphology of particles and pores (TEM and SEM), chemical composition (EELS) and crystallinity (XRD) were studied. Monoliths with PEO at 1% and 5% (m/m) showed hybrid isotherms of type IV and II as they have meso and macropores. PEO at 3% lead to hybrid isotherms of type I and IV. Composites without PEO showed isotherms of type IV and SSA of 482 $m^2\,g^{-1}$ The highest SSA was achieved for 3% PEO, annealed at 500 °C (766 $m^2\,g^{-1}$) and in all composites the SSA reduces with increasing the annealing temperature from 500 to 900 °C as the sintering of the silica matrix occurs by viscous flow. Pore size distribution curve also showed that composites without PEO are mesoporous only; meanwhile the PEO addition creates macroporosity, explaining the changes in isotherms types. The microscopy showed an interconnected porous network, with well dispersed Nb2O5, whose crystalline phase depends on annealing temperature, and particles size increases with increasing the temperature. The monoliths obtained with 1 and 5% PEO were completely transparent and showed a more pronounced shrinkage rate during drying and annealing than the white opaque 3% PEO monolith. It was possible to tailor, in one step synthesis, the textural properties of monolithic xerogel SiO2/Nb2O5, leading to well dispersed Nb2O5 particles in the silica matrix, with high SSA.