02-058

Liquid crystals as pores template for sulfated zirconia ceramics

Pulcinelli, S.H.(1); Alves-rosa, M.A.(2); Moris, C.H.(1); Freitas, F.(1); Santilli, C.V.(1);
(1) UNESP/IQ; (2) IQ/UNESP;

Porous sulfated zirconia was prepared by using the sol-gel process associated to liquid crystal templates (LCT). The Zr+4:SO4-2 molar ratio and the ageing time were evaluated since the LCT gel formation to the final ceramic powders. Polarized light microscopy (PLM) and small angle X-ray diffraction (SAXD) analysis of the 8 days aged LCT wet gel samples revealed the hexagonal as the prevalent mesophase. The thermal treatment of the samples resulted in sulfated ZrO2 ceramic powders, in which infrared (FTIR) spectra exhibited bands characteristics of mono- and bi-dentate SO42- groups bonded to ZrO2 (1056-1220 cm-1). X-ray diffraction (XRD) patterns showed a mixture of monoclinic and tetragonal phases of zirconia in which the tetragonal phase rises in function of sulfate amount with crystallite sizes about up to 9 and 7.5 nm for Zr and sulfated samples, respectively. The lattice fringe distances observed by selected-area electron diffraction patterns (SAED) and high resolution TEM confirmed the mixture of tetragonal and monoclinic crystalline phases. Analysis of the region of power-low scattering from SAXD pointed that scattered objects have radius of gyration around 2 nm and the particles constitute a network of branches with fractal surface when sulfate is inserted in the zirconia structure, favoring better porous characteristics. The LCT generated higher pores size diameter (5.7 nm) in the non sulfated Zr sample. This parameter decreases (? 3 nm) as the sulfate content increases, while the surface area rises 146 m2g-1. The gel ageing process promoted the reinforcement of the pores walls structure, prevented shrinkage effects during calcination, and allowed reaching higher surface areas. Images from scanning and transmission electronic microscopies (SEM and TEM) elucidated the pores walls as platelets of irregular format and the presence of mesopores, respectively. The porous structure combined to the presence of acid catalytic sites, improved by sulfate groups at the zirconia surface, makes the materials promising for application as acid catalysts in dehydration reactions.