MATERIAIS 2007
Porto, 1-4 April 2007

IN-SITU XRD STUDIES OF THERMAL STABILITY OF PURE SILICA AND Ti-MCM-41 MATERIALS

N. Franco [1], E. Alves [1*], M. M. L. Ribeiro Carrott [2], C. Galacho [2], P.J.M Carrott [2]

1 Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2686-953 Sacavém, Portugal
2 Centro de Química de Évora and Departamento de Química, Universidade de Évora, 7000-671 Évora, Portugal
* corresponding author: ealves@itn.pt

Keywords: In-situ X-ray diffraction, ordered mesoporous materials, thermal stability.

Abstract

In this work we present a study on the thermal stability of siliceous and titanium containing ordered mesoporous materials with MCM-41 structure, prepared by direct synthesis at ambient temperature and pressure, as previously described [1]. Grades with different pore diameter and Si/Ti molar ratios were analyzed, in the form of compacted powder, to assess its influence on the order-disorder transition temperature. The temperature was raised up to 1000 ^{o}C under vacuum (~10^{-5} mbar), flowing N_2 (~10^{-3} mbar) atmosphere and air. An intense X-ray beam collimated by a Göbel mirror was used to follow in-situ the temperature dependence of the unit cell dimension in θ-2θ configuration. During the annealing, in steps of 100 ^{o}C, the porous parameter decreases, as well as the diffracted signal leading to its extinction at around 1000ºC. The results show that the exact temperature at which the collapse of the ordered mesopore structure occurs is dependent on the atmosphere conditions. Under vacuum, this transition is reversible and the main diffraction peak (100) of the two-dimensional hexagonal lattice appears with the same intensity and slightly shifted to higher angles after cooling to room temperature. The same result, although not so pronounced, was observed when the measurements were done in flowing nitrogen atmosphere. The samples reveal a totally different behavior when annealed in air where the transition temperature decreases and the reversible nature of the process disappears.

Reference

[1] M.M.L. Ribeiro Carrott, C. Galacho, F.L. Conceição, P.J.M. Carrott. Influence of the Synthesis Conditions on the Pore Structure and Stability of MCM-41 Materials Containing Aluminium or Titanium. *Stud. Surf. Sci. Catal.*, 160, 567-574, 2006.

Acknowledgement

The authors acknowledge financial support from FCT and FEDER (project no. POCTI/CTM/45859/2002).