Structural and textural studies of NiO impregnation in mesoporous ZrO2-CeO2 for catalysis applications.

21a. Reunião da Associação Brasileira de Cristalografia, 2013, Goiânia. Anais da 21a. Reunião da Associação Brasileira de Cristalografia, 2013.

Structural and textural studies of NiO impregnation in mesoporous ZrO_2-CeO_2 for catalysis applications

R. Bacani[a], A. D. S. Bruno Costa[a], T. S. Martins[b], M. C. A. Fantini[a]

[a]*Instituto de Física, Universidade de São Paulo – São Paulo, Brasil.*
[b]*Instituto de Ciências Ambientais, Químicas e Farmacêuticas, UNIFESP, São Paulo, Brasil.*

The synthesis of zirconia-based ordered mesoporous structures for catalytic applications is a research area under development. These systems are also potential candidates as anodes in intermediate temperature solid oxide fuel cells (it-SOFC) due to an enhancement on their surface area [1-4]. The structural features of mesoporous zirconia-ceria materials in combination with oxygen storage/release capacity (OSC) are crucial for various catalytic reactions. The direct use of hydrocarbons as fuel for the SOFC (instead of pure H_2), without the necessity of reforming and purification reactors can improve global efficiency of these systems [4].

The X-ray diffraction data showed that ZrO_2-x%CeO_2 samples with x>50 are formed by a larger fraction of the cubic phase (spatial group Fm3m), while for x<50 the major crystalline structure is the tetragonal phase (spatial group $P4_2/nmc$). The crystallite size of the cubic phase increases with increase in ceria content. The tetragonal crystallite size decreases when ceria content increases. After impregnation, the Rietveld analysis showed a NiO content around 60wt.% for all samples. The lattice parameters for the ZrO_2 tetragonal phase are lower for higher ZrO_2 contents, while for all samples the cubic NiO and CeO_2 parameters do not present changes. The calculated densities are higher for higher ceria content, as expected. The crystallite size of NiO are similar (~20nm) for all samples and 55nm for the NiO standard.

Nitrogen adsorption experiments revealed a broader particle size distribution for higher CeO_2 content. The superficial area values were around $35m^2/g$ for all samples, the average pore diameter and pore volumes were higher when increasing ceria content. After NiO impregnation the particle size distribution was the same for all samples, with two pore sizes, the first around 3nm and a broader peak around 10nm. The superficial area increased to approximately $45m^2/g$ for all samples, and the pore volume was also higher after impregnation and increased when ceria content increased.

These results point up that the impregnation of NiO improves the textural characteristics of the pristine material. The complementary TEM/EDS images present a homogeneous coating of NiO particles over the ZrO_2-x%CeO_2 support, showing that these samples are excellent for catalysis applications.

[1] D. Y. Zhao, J. Feng, Q. Huo, N. Melosh, G. H. Fredrickson, B. F. Chmelka, G. D. Stucky, Science 279, 548-552 (1998).
[2] C. Yu, Y. Yu, D. Zhao, Chem. Comm. 575-576 (2000).
[3] A. Trovarelli, M. Boaro, E. Rocchini, C. de Leitenburg, G. Dolcetti, J. Alloys Compd. 323-324 (2001) 584-591.
[4] S. Larrondo, M. A. Vidal, B. Irigoyen, A. F. Craievich, D. G. Lamas, I. O. Fábregas, et al. Catal. Today 107–108 (2005) 53-59.

Acknowledgments: CNPq and FAPESP.