

Mesoporous silica organofunctionalized with *p*-phenylenediamine: synthesis and characterization

Diego Luiz Cavaretti Golinelli, Ivana Cesarino, Fernando Cruz de Moraes, Sergio Antonio Spinola Machado*

Instituto de Química de São Carlos, Universidade de São Paulo, São Carlos, SP, Brazil

The organofunctionalization of porous silica surfaces has received much attention, because they combine a high surface area and a narrow pore size distribution with the advantage of organic ligand properties. This expands the range of potential applications in catalysis, sensors, separation and opto-electric devices [1-3]. Aminosilica is a type of organic/inorganic hybrid material created by incorporation of amino groups in the silica structure via synthesis procedure. In this work a one-step synthesis of organofunctionalized cetyltrimethylammonium bromide (CTAB)-template silica, using *p*-phenylenediamine (*p*-PDA) like a modifier is presented. The mesoporous silica organofunctionalized with *p*-PDA was characterized by IR spectroscopy, thermogravimetry (TG), elemental analysis (EA) and X-ray diffraction (XRD).

Aiming to verify the organofunctionalization of the silica, the mesoporous silica was synthesized in the presence and absence of amino ligand. EA presented an increase in the nitrogen, carbon and hydrogen contents after functionalization with *p*-PDA suggested that the modifier is being incorporated to the silica matrix. Calculations pointed out to 0.18 mol of the modifier per 100 g of silica after modification procedure, on the basis of the carbon percentage. IR spectroscopy is employed for accompanying the organofunctionalization of mesoporous silica. The bands observed confirmed the presence of *p*-PDA groups bound on the silica surface.

Thermogravimetry was used for accompanying the thermal behavior of the products obtained in the organofunctionalization. The decomposition profiles in the region of the organic matter burning are different, showing that the modifier promoted changes in the silica.

The XRD pattern for the unmodified silica displayed a well-resolved pattern with a diffraction peak at 6.74° and two weak peaks at 10.1° and 13.6° with d-spacing values of 13.10, 8.73 and 6.52 Å, respectively. After organofunctionalization with *p*-PDA a considerable decrease in the XRD peaks intensity was observed, providing further evidence of functionalization occurring mainly inside the mesopore channels.

The results showed that the organofunctionalization of the mesoporous silica with *p*-phenylenediamine could be attained. Therefore, the modified silica obtained is intended to be used as solid-phase reactors in electrochemical flow system for analysis of phenols in municipal and industries waste water.

Keywords: *p*-phenylenediamine, mesoporous silica, IR spectroscopy

Work supported by CNPq (grant 151393/2009-5).

[1] I. Cesarino, E.T.G. Cavaleiro, C.M.A. Brett, *Electroanalysis* **22**, 61 (2010).

[2] H. Yang, N. Coombs, I. Sokolov, G.A. Ozin, *Nature* **381** 589 (1996).

[3] I. Cesarino, E.T.G. Cavaleiro, *Electroanalysis* **20** 2301 (2008).

*ssmach@iqsc.usp.br