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THE TEMPLATE-GUIDED SYNTHESIS OF SILICA AND ORGANOSILICATE NANOTUBES OBTAINED BY THE SOL-GEL PROCESS

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Abstract – Silica nanotubes derived from tetraethylorthosilicate (TEOS) and organossilicate nanotubes derived from methyltrietoxysilane (MTES) and vinyltrietoxysilane (VTES) were synthesized using ammonium tartarate and ammonium citrate as templates, via the sol-gel process. The products were characterized by TGA, FTIR and XRD. The morphology of the materials was examined by optical and scanning electron microscopy (SEM). It was observed the formation of tubular morphologies and some spherical micro particles that coexisted in the materials.

The synthesis of novel porous materials has recently attracted much attention because of the potential applications of these materials in catalysis, separation science, and nanotechnology [1]. Although, there are a number of reports on the synthesis strategies of the nanotubular structures of oxide materials in the literature, the templating method has many more advantages over the templateless methods, such as facile fabrication, various compositions of materials, and uniform sizes of the formed nanotubes. Therefore, template-directed methods can allow us to have manufacturing ability to produce monodisperse 1-D nanotubular-structured materials with high yields. The template-guided synthesis of nanotubes was pioneered by Martin and co-workers [2]. Their methods can be used to produce nanotubes from a variety of materials over a wide range of size scales. The sol-gel process has so far been one of the most attractive ways of synthesizing these porous materials because of the mild synthesis conditions required [3].

In this study we have performed the synthesis and characterization of hybrid organic–inorganic silica nanotubes prepared by controlled hydrolysis of tetraethylorthosilicate (TEOS), methyltrietoxysilane (MTES) and and vinyltrietoxysilane (VTES) in the presence of ammonium DL-tartarate and ammonium citrate crystals as the structural directing agent. The syntheses were carried out using a modified procedure described in literature [3].

In a typical synthesis, the organosilicate precursor (TEOS, MTES or VTES) was dissolved in anidrous ethanol and hydrolyzed with stoichiometric amount of water. Afterwards, the DL-tartaric acid or citric acid dissolved in solution of NH_4OH (28 wt%) was dropwised to form a gel. The gel was then aged at room temperature for a specific time, which depended on the precursor used, and then dried in the oven at 75ºC. The products were calcined under static air at different temperatures up to 1000ºC. The characterization was performed by a series of analytical techniques such as TGA, FTIR and XRD. SEM analysis of the ammonium salt templates, showed the ability of this material to form crystals with pillar-like morphologies, and also ability to direct the growing of organosilicate nanotubes, even if some spherical micro particles coexisted in the materials. TG-DTA and FTIR results indicate that the hydrolysis/condensation reactions were complete after the thermal treatment. Our experimental procedure enabled us to prepare some organosilicate materials derived from VTES and MTES, which produced silicon oxycarbide glasses (SiOC) after pyrolysis at 1000°C. Results based on XRD and FTIR agreed with the formation of an amorphous network in the Si-O-C system.

The control of synthetic conditions has been observed to be essential for the formation of silica and organosilicate nanotubes. Factors such as time of hydrolysis and the template structure have a direct relationship with the morphologies of the resulting materials. Then we can concluded that this positive template method can be used to prepare silica and organosilicate nanotubes starting from TEOS and MTES or VTES.

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