TiO_2-derived nanotubes treated with different acids.

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Palavras Chave: *titanate nanotubes, acid treatment.*

Abstract

The titanate phase thermal stability of hydrothermally produced nanotubes treated with different acids was compared by XRD.

Introdução

TiO_2-derived nanotubes from alkaline hydrothermal reactions (TNTs) are extensively studied for their potential applications in photocatalysis, energy conversion and water remediation by absorption of heavy metal ions. Since their discovery in 1998[1], the role of a post acid treatment has been under debate. It has been established that the acid washing replaces interlayer Na^+ - present due to the highly concentrated NaOH solution used for synthesis - with H^+, thus avoiding the crystallization of Na-titanate at temperatures higher than 650 °C. Although most studies employ different concentrations of HCl solutions, there has been evidence that the conjugated base may influence the anatase formation temperature.[3] Here we discuss the use of the 0,1 mol.L^{-1} solution of the different acids HCl, HF, H_2SO_4, H_3PO_4, HNO_3 and acetic acid (HOAc).

Titanate nanotubes were produced under 10 mol.L^{-1} NaOH solution, heated in a Teflon©-lined stainless steel autoclave for 20h at 140 ºC. After being centrifuged and washed with water, the powder was divided into 6 portions, each one treated with 50 mL of 0,1 molL^{-1} acid solution. After washing with water, the dried samples were heated for 30 min at different temperatures and analyzed by XRD after each heating step.

Resultados e Discussão

After heating at 200 °C, XRD patterns (Fig. 1) are similar to those typically obtained for hidrothermally produced TNTs. Except for HF-TNT, which shows the presence of poorly crystallized anatase. At every heating step, anatase crystal growth and crystallization takes place, making the XRD peaks of HF-TNT better defined. As shown in Fig. 2, at 400 °C all the samples but H_3PO_4-TNT consist of anatase-TiO_2, although with different levels of crystallization. The low intensity peak at ~12 ° in HNO_3- and HOAc-TNTs is usually associated with the interlayer distance between the nanotube walls. H_3PO_4-TNT was further heated up to 700 °C, but the titanate nanotube phase remained the only one present in the XRD pattern.

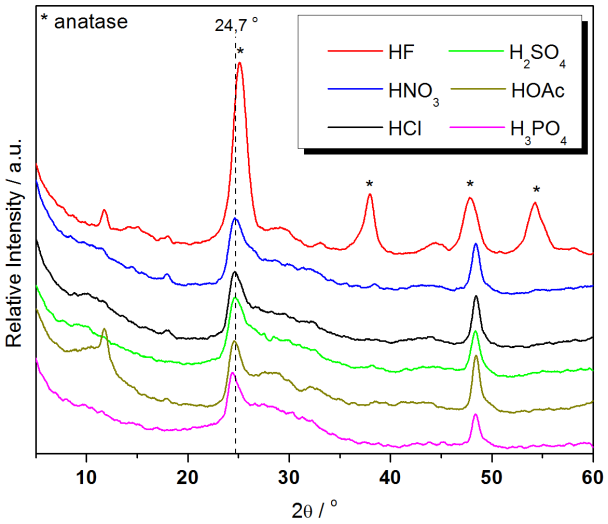

Figura 1. XRD patterns of samples after heating at 200 °C.

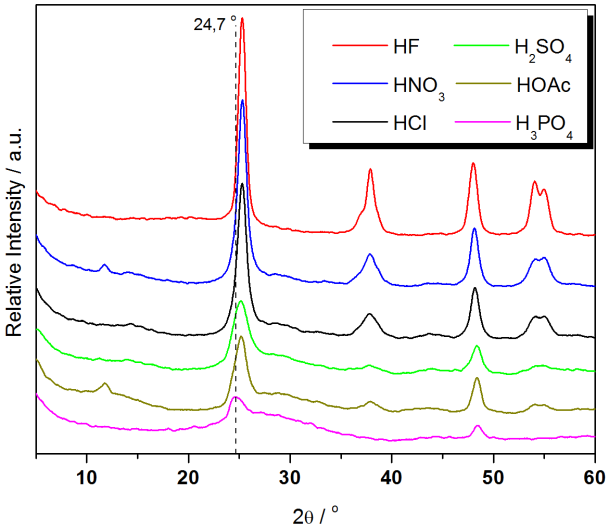

Figura 2. XRD patterns of samples after heating at 400 °C.

Conclusões

HF promotes the conversion of titanate to anatase at low temperatures, whereas H_3PO_4 restrains the phase transformation even at 700 °C. HCl and HNO_3 favors the phase change when compared to HOAc and H_2SO_4.

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[1]Kasuga, T.; Hiramatsu, M.; Hoson, A.; Sekino T. and Niihara, K. *Langmuir*. **1998**, *14*, 3160.

[2] Körösi, L; Papp, S.; Hornol, V. et al. *J. Solid State Chem*. **2012**, 192, 342