Influence of the reaction medium on the properties of SBA-15/TiO₂ nanocomposites

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Keywords: nanocomposites, ordered mesoporous silica, titanium dioxide, ionic liquid. Introduction

Titanium dioxide (TiO₂) is a material of unique properties, such as high chemical stability, low cost and lack of toxicity, having a high demand for various applications, such as photocatalysis, solar cells and sunblocks. Its incorporation in ordered mesoporous silica, in particular of the SBA-15 kind, is beneficial as it results in a material with high surface area, enhanced thermal and chemical stability, as well as it prevents particle applomeration and leads to increased safety for application in cosmetics.¹⁻³

The use of ionic liquids (IL) as green solvents in organic and inorganic synthesis is extremely promising considering their negligible vapor pressure, low flammability and tunable properties. Nevertheless, reports on how IL influence TiO₂ crystalline phase are still very scarce.⁴

This work aims to study the influence of the reaction medium on the structural, morphologic and textural properties of SBA-15/TiO₂ nanocomposites.

Results and Discussion

SBA-15/TiO₂ nanomaterials were prepared with a 20% Ti molar ratio in different reaction medium (w/o IL, w/ CMITf₂N, w/ CMIBF₄, w/ or w/o isopropyl alcohol). Relative peak intensities on X-Ray Diffraction (XRD) patterns (Fig. 1 and 2) indicate that the presence of $CMIBF_4$ greatly favors TiO_2 anatase crystalline phase, while CMITF₂N slightly favors rutile crystalline phase. An anatase-phase selective material was synthesized using 0.032M of CMIBF₄ without alcohol. Selectiveness of rutile phase by CMITf₂N was most pronounced when higher concentration of IL was used and was not significantly affected by the alcohol presence.

Small Angle X-Ray Diffraction patterns (SA-XRD) (Fig. 3 and 4) revealed that nanocomposites prepared with lower IL concentration were able to maintain the ordered bidimensional hexagonal pore structure, characteristic of SBA-15, whilst the ones prepared with higher IL concentration were not. Nevertheless, high surface areas and pore volumes observed in N₂ adsorption-desorption were isotherms for the non-structured materials.

Fig. 1. XRD patterns of SBA-15/TiO2CMI 0,032M





Fig. 2. XRD patterns of

SBA-15/TiO2CMI 0,32M

Fig. 3. SA-XRD of SBA-15/TiO2CMI 0,032M



Fig. 4. SA-XRD of SBA-15/TiO2CMI 0,32M



Conclusions

The properties of SBA-15/TiO₂ are indeed influenced by the reaction medium, specifically the presence/absence of IL (CMITf₂N or CMIBF₄) and isopropyl alcohol. CMIBF4 favored anatase and an anatase-phase selective material was obtained using 0.032M of CMIBF₄ and no alcohol. CMITf₂N slightly favored rutile, however this behavior is lessened with the reduction of IL concentration, regardless of alcohol presence. Structured mesoporous materials were obtained with lower IL concentration. Next steps include: Rietveld refinement for phase quantification, further characterization and doping with lanthanides (Ce and Eu).

Acknowledgements

Thanks are due to A.C.F. Silveira and T. Germano from Instituto de Física (USP) and C.M. Fukumoto, R.M. da Silva, R. Rodrigues and S. Pinheiro from Central Analítica (UNIFESP) for XRD, SAXS and N₂ adsorption-desorption measurements, as well as CNPq/CAPES for financial support.

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