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Nonaqueous Synthesis of metal oxide nanoparticles

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Abstract – Nonaqueous synthesis routes to metal oxide nanoparticles are a valuable alternative to the aqueous sol-gel processes, offering advantages such as high crystallinity at low temperatures and ability to control the crystal growth without the use of surfactants. In this work the synthesis of metal oxide species, such as SnO_2 , ZrO_2 and $Sn:In_2O_3$ (ITO), is presented by a surfactant-free method, within some reaction system such as metal halides – benzyl alcohol, metal alkoxides – benzyl alcohol and metal acetylcetonates – benzyl alcohol showing the versatility of this approach. The characterization of the products (XRD, HRTEM) confirms the desired characteristics promised by the synthesis method.

Although the term "nanomaterials" represents a large variety of materials in the domain of nanometers, oxide nanoparticles have been considered as one of few core materials in nanoscience and nanotechnology. In addition to the size effect, nanoparticles represent the most popular morphology of the nanoscale world and are ideal manipulable building blocks to construct larger devices, structures and systems following the so-called "bottom-up" approach in nanotechnology¹. In this way, nonaqueous synthesis routes to metal oxide nanoparticles are a valuable alternative to the known aqueous sol-gel process, offering advantages such as high cristallinity at low temperatures and ability to control the crystal growth without addition of surfactants². This work presents an overview of the method by the synthesis of two well-known oxides, SnO_2 and ZrO_2 , and a doped oxide, Sn:In₂O₃ (ITO). The experimental procedure consists in a one pot reaction, initiated by the addition of an organometallic precursor (metal alkoxides, acetates, acetylacetonates) into on alcoholic solution following by solvothermal treatment at temperature range from 150°C to 300°C². All the products presented a well crystallized structure (Fig. 1), with small particle size, revealed by XRD peak width. The reaction of zirconium isopropoxide with benzyl alcohol at 290 °C resulted in zirconia nanocrystals with diameters of 4-6 nm (Fig. 2a), while the reaction of tin(IV) choride with benzyl alcohol at 100°C resulted in SnO₂ nanocrystals with diameters of 3-6 nm (Fig. 2 b)³. In the case of ITO, the precursors tin *ter*t-butoxide and indium acetylacetonate were reacted in benzyl alcohol at 200°C, yelding ITO nanoparticles in the size range of 5-12 nm (Fig. 2c). The solvothermal reaction between the organometallic compounds above in benzyl alcohol provides a comfortable nonaqueous route to crystalline metal oxides nanoparticles with uniform spherical shape and sizes in the range of 3-12 nm.





Figure 2: HRTEM images for the three synthesized oxides: a) ZrO_2 ; b) SnO_2 ; c) ITO.

References

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