

Synthesis of zirconia nanoparticles from Sri Lankan zircon sand

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Zirconia (ZrO_2) nanoparticles are very important for the production of a wide-range of technological devices and products such as solid oxide fuel cells, oxygen sensors, abrasion-resistant gloves, heating elements, high-temperature refractory materials and ceramic prostheses. The major source of zirconia is zircon ($ZrSiO_4$) sand, a silicate mineral of zirconium. There is a vast amount of zircon sand available in coastal areas of Sri Lanka. The extraction process of zirconia from Sri Lankan zircon sand had previously been studied at the Department of Physics, University of Peradeniya by Karunaratne et.al., 1993. The present study focuses on modification of previous extraction process to synthesize nanometer sized zirconia from Sri Lankan zircon sand.

In this study, zircon sand was fused with sodium hydroxide and the fused mass was dissolved in distilled water. The obtained residue was then dissolved in concentrated hydrochloric acid to produce zirconium oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$). Diluted sodium hydroxide solution was added dropwise to the diluted zirconium oxychloride octahydrate solution and different colloid stabilization methods such as use of surfactants (Triton X-100, cetyltrimethylammonium chloride, sodium dodecyl sulfate) and *in-situ* polymerization of methyl methacrylate (MMA) were used to inhibit particle agglomeration. Precipitated hydroxide of zirconium was separated by centrifugation and decomposed at $650^\circ C$ to obtain nanometer sized zirconia. X-ray diffraction and x-ray fluorescence results confirm that the final product comprises ZrO_2 . The dynamic light scattering particle size analysis (DLSPSA) and scanning electron microscopy (SEM) reveal that the ZrO_2 product is in nanometer scale.

The smallest average particle size obtained for the zirconia nanoparticles was about 40 nm with the use of *in-situ* polymerization of MMA. Further, these nanoparticles appeared to be spherical in SEM images and approximately monodispersed according to DLSPSA. The products prepared using surfactants of SDS, CTAC and Triton x-100 were moderately polydispersed zirconia particles with the average particle sizes of 311 nm, 473 nm, and 498 nm respectively. The yield of the extraction process was about 83 % at a purity of 97 % zirconia. The major impurity in the final product was found to be 3 % of hafnia (HfO_2), which is present in zircon sand as an impurity. The separation of hafnia is not required for most of the practical applications of zirconia nanoparticles.