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Original Supercritical Water Device for Continuous Production of Nanopowders Examples of Synthesized Nano-oxides

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Metal oxide nanoparticles are usually used in a variety of applications including sensors, varistors, pigments, fillers, electrography and medical materials. Nanoparticles can be produced by several techniques such as precipitation, spray pyrolysis, thermal decomposition and hydrothermal synthesis. In particular, Supercritical Water Hydrothermal Synthesis (ScWs) is an environmentally process for the production of potentially valuable metal oxide nanoparticles (TiO₂, ZrO₂, ...) usually applied in the Nanotechnology. Currently, most scWH Syntheses are performed in batch reactors offering a low production of nanomaterials unlike continuous reactors. In this context, a continuous hydrothermal production process of oxide and hydroxide nanoparticles synthesized in supercritical conditions has been developed by the Laboratoire Interdisciplinaire Carnot de Bourgogne (ICB) since 2001.

In the suggested method employing a patented flow type tubular reactor (WO 2011010056) optimized with the help of Computational Fluid Dynamics (CFD) simulations, metal salt aqueous solution is mixed with preheated water to rapidly increase the solution temperature up to the supercritical state. The high reaction rate of synthesis and the low solubility of metal oxides in SCW conditions lead to the formation of nano-sized particles.

Synthesis of crystalline ZnO, ZrO₂, TiO₂, CeO₂, Y₂O₃ and La₂O₃ nano-particles are carried out from metallic salts (Zn(NO₃)₂, ZrO(NO₃)₂, Ce(NO₃)₃, Y(NO₃)₃, La(NO₃)₃), and organometallic (ALT) as precursors. At room temperature, metallic salt and a hydroxide aqueous solution (NaOH or KOH) are pressurized to 25-30 MPa. Then, the two reactants are rapidly heated to 550-673 K by mixing with the supercritical water (25-30 MPa, 550-773 K) in the reactor. The residence time inside the reactor is in the range from 2 to 10 s. Production of nanosize particles with uniform particle size distribution (\leq 20 nm) show highly crystalline phases identified by XRD, Raman spectroscopy and TEM observations.

Keywords: nano-oxides, continuous hydrothermal synthesis, SCW, Nanotechnology.