CONTINUOUS HYDROTHERMAL SYNTHESIS OF ZINC OXIDE NANO-PARTICLES UNDER SUPERCRITICAL CONDITIONS.

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Abstract

The wide-band-gap semi conductor ZnO is used in a variety of applications including sensor, varistor, pigments, fillers, electrography, medicals materials and UV light-emitting and lasing devices. ZnO nano-particles can be produced by several techniques such as precipitation, spray pyrolysis, thermal decomposition and hydrothermal synthesis. In particular, hydrothermal synthesis is well known as an environmental safe and simple process, since it does not require any organic solvents and additional processing such as calcination.

In this context, synthesis crystalline ZnO nano-particles from zinc salt ($Zn(CH_3COO)_2$, $ZnSO_4$, $Zn(NO_3)_2$) and alkali metal hydroxide (LiOH, KOH) aqueous solutions were carried out with an continuous production prototype for hydrothermal synthesis in subcritical and supercritical water developed in our group. The reactor was optimized with the help of a commercially available CFD package (FLUENT® 6.3). At room temperature, zinc salt and hydroxide aqueous solutions were pressurized to 30 MPa. Then, the two reactants were rapidly heated to 673 K by mixing with the supercritical water (30 MPa, 773 K) in a tubular reactor. Residence time is about 4 s. The reaction was finished by cooling at the exit of the reactor. Production of nanosize particles (50-100 $g.h^{-1}$) with uniform particle size distribution ($\leq$ 20 nm) showed a highly crystalline ZnO phase identified by XRD, Raman spectroscopy and TEM and SEM observations.

The aims of this project are (1) to optimize the hydrothermal process in order to obtain rapidly pure ZnO nanopowders with a high yield and controllable morphology parameters, (2) to understand the chemical process synthesis in supercritical conditions and, finally (3) to attempt to explain the variation of morphology of the obtained ZnO powder with solvents which mainly depends on the different zinc species and their environment during the hydrothermal processing.

Keywords: *ZnO, Continuous hydrothermal synthesis, Supercritical water, Nano-particles.*