

PRODUCTION OF METAL OXIDE NANOPARTICLES BY SUPERCRITICAL EMULSION REACTION

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A technique is proposed for the production of tungsten dioxide nanoparticles in which supercritical CO₂ plays a double role acting, as antisolvent and as reactant. A continuous phase containing n-heptane or cyclohexane and AOT surfactant is first prepared. A solution of sodium tungstate dihydrate in water is then added to the above mixture, to form a clear reverse micellar solution. Tungsten dioxide nanoparticles are synthesized in sodium bis (2-ethylhexyl) sulfosuccinate (AOT) reverse micelles by reaction with CO₂ and their depositions has been investigated. Precipitated nanoparticles are collected on the filter together with the sodium carbonate, the residual surfactant and water. Mostly of the residual components are eliminated during the washing step by continuously purging of CO₂.

Process parameters affecting nanoparticles size are studied such as pressure, water and surfactant concentration. After the depressurization of the chamber, different recovery methods of the precipitated nanoparticles are explored such us ultrafiltration or separation by ultrasonic probe. The nanoparticles are characterized by Field Emission Electron Microscopy (FESEM) and Energy Dispersive X-Ray Analysis (EDX). The specimen for the SEM imaging is prepared by depositing a few drops of the diluted nanoparticles suspension onto a carbon tape, followed by solvent evaporation under ambient condition. The results show that the dimensions can be easily controlled by tuning the pressure and nanoparticles can be obtained smaller than 50 nm.