287j Polymer Coating of Submicron Particles from Sas Process and Characterization Using Tem-Eels

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Coating of submicron particles with polymer using Supercritical Anti-Solvent (SAS) process was investigated and the encapsulations were characterized by Transmission Electron Microscopy (TEM)–Electron Energy Loss Spectroscopy (EELS) analysis using LEO 922 TEM equipped with Omega Energy Filter (EF) to demonstrate the capability of EF-TEM in characterizing multi-component composites. In this research work, the spherical SiO2 particles (SO-E2, 400-600 nm from Admatechs) were encapsulated with Eudragit (Rohm GmbH, E100) using a supercritical CO2 processing system. Eudragit is a family of polymer widely used in food and drug industry. It has been used extensively as encapsulating materials in drug formulation. Because it is alkaline soluble and therefore resistant to stomach acids, the active substance is not released in the stomach, but in the intestine, where it becomes activated. In the experiment, the suspension of silica particles in an acetone–eudragit solution was sprayed through a co-axial ultrasonic nozzle into supercritical CO2 environment, where the polymers nucleate and encapsulate the silica particles. The co-axial flows of solution suspension with the antisolvent, together with the ultrasonic vibration greatly enhance the mutual mixing. Evenly coated silica particles were obtained. In this work, silica encapsulations is a challenge in characterization due mainly to multi-components/elements involvement. TEM is able to resolve the composites structure, but, it is generally incapable of distinguishing the multi-components/elements comprising the material. X-ray microanalysis (EDS or WDS) has been employed in elemental analysis in TEM investigations. However, microanalysis scanning capability (STEM) is needed. Besides, X-ray analysis is not efficient for light elements, such as, C, N, F, etc. Currently, EELS provides superior capability in identifying and quantifying light elements under nanometer scales. Other examples of TEM-EELS characterization of nano-scale particle mixtures are also presented.

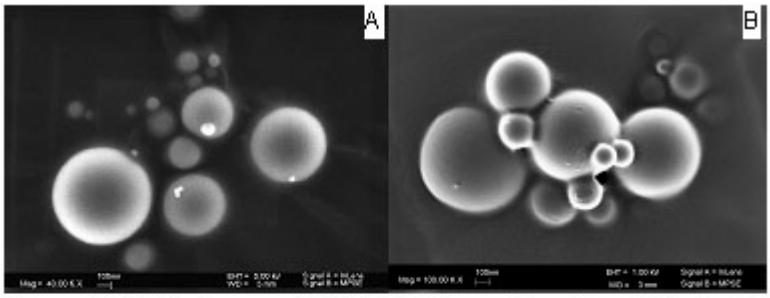

Figure 1. SEM images of Original SiO_2 particles (A) and Eudragit encapsulated SiO_2 particles (B).

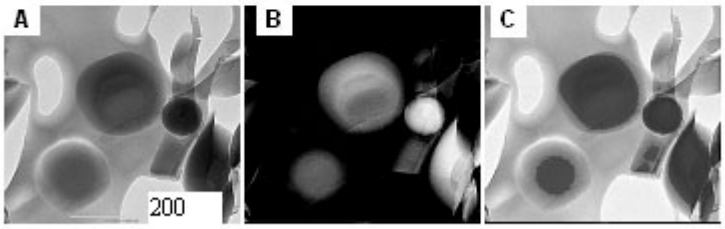

Figure 2. TEM images of Eudragit encapsulated SiO_2 particles: A: zero loss; B: Si distribution; C: superimposition of A and B.