Characterization of nanomaterials (pristine or in dispersion)

Category:
C. Nanomaterial characterisation in situ / ex situ

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Short technology description/Overview:

Field Emission Gun SEM
(FEG-SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern and producing information about its surface topography and composition. The JSM-7500F offers the highest resolution at the lowest kV of any SEM available, achieving a resolution of 1.4 nm at 1 kV. The JSM-7500F provides in-lens performance (1.0nm at 15kV) but can handle samples up to 200mm in diameter x 10mm in height. The user interface is designed to facilitate operations. Particle size distributions can also be determined (SmileView software). The FEG-SEM is coupled to an EDX detector (JED-2300) for measuring the elemental chemical composition of nanomaterials.

Analytical centrifugation
A technique for measuring particles in the range of 10 nm to 40 microns. The analyzer measures particle size distributions using centrifugal sedimentation within an optically clear spinning disc that is filled with fluid. Sedimentation is stabilized by a density gradient within the fluid, and accuracy of measured sizes is insured through the use of a known size calibration standard before each test.
The concentration of particles at each size is determined by continuously measuring the turbidity of the fluid near the outside edge of the rotating disc. Weight, surface and number distribution can be obtained and the contribution (interferences) from the dispersing media can be removed. For a majority of applications, analysis times are in the range of 3 to 15 minutes per sample.

PIXE
Particle-Induced X-ray Emission (PIXE) is a technique based on excitation of the electronic levels of the atoms, by means of an ion beam, producing X-ray emission. These X-rays are characteristic and proportional to every element, thus allowing an easy identification and quantification of the elemental composition of the measured target (both the nanomaterial and its containing matrix).
PIXE possesses no restrictions of nanomaterial size; it offers a low background noise when compared to EDX, achieving ppm-levels of sensitivity; it can measure unmodified nanomaterials: no need for radiolabelling / radioactivation / fluorescence modifications to the nanomaterial that can potentially alter the response in the target media. Big volume samples can be measured for statistical significance. Nanomaterials can be analyzed in either dispersion or solid form. Analysis times are in the range of 2 to 5 minutes per sample.

Main Features (Equipment Capabilities):

FEG-SEM (Jeol JSM 7500f): Characterization of the raw nanomaterial

- Imaging modes: SEI (secondary electron image), LEI (low secondary electron image), LABe (backscatter electron image). LABe: Eliminates charging effects, allows low kV backscattered electron imaging and provides more surface detail & compositional contrast.
- GB (gentle beam) mode - provides extreme images at very low accelerating voltage, suitable for non-conducting samples. Field emission gun and acceleration voltage: 100 V to 30 kV (100 V steps). EDX coupled for elemental composition determination.
- Type of samples: inorganic nanomaterials, low conductive nanomaterials, nanopowders, nanomaterial dispersions (dried), biological samples (after conditioning).
- The structure, morphology and elemental composition of samples can be obtained in one run.
- Limitations: insulating materials should be covered with thin films (Au, Ag).

Analytical centrifugation (Disc centrifuge 24000): Characterization of the nanomaterial dispersions

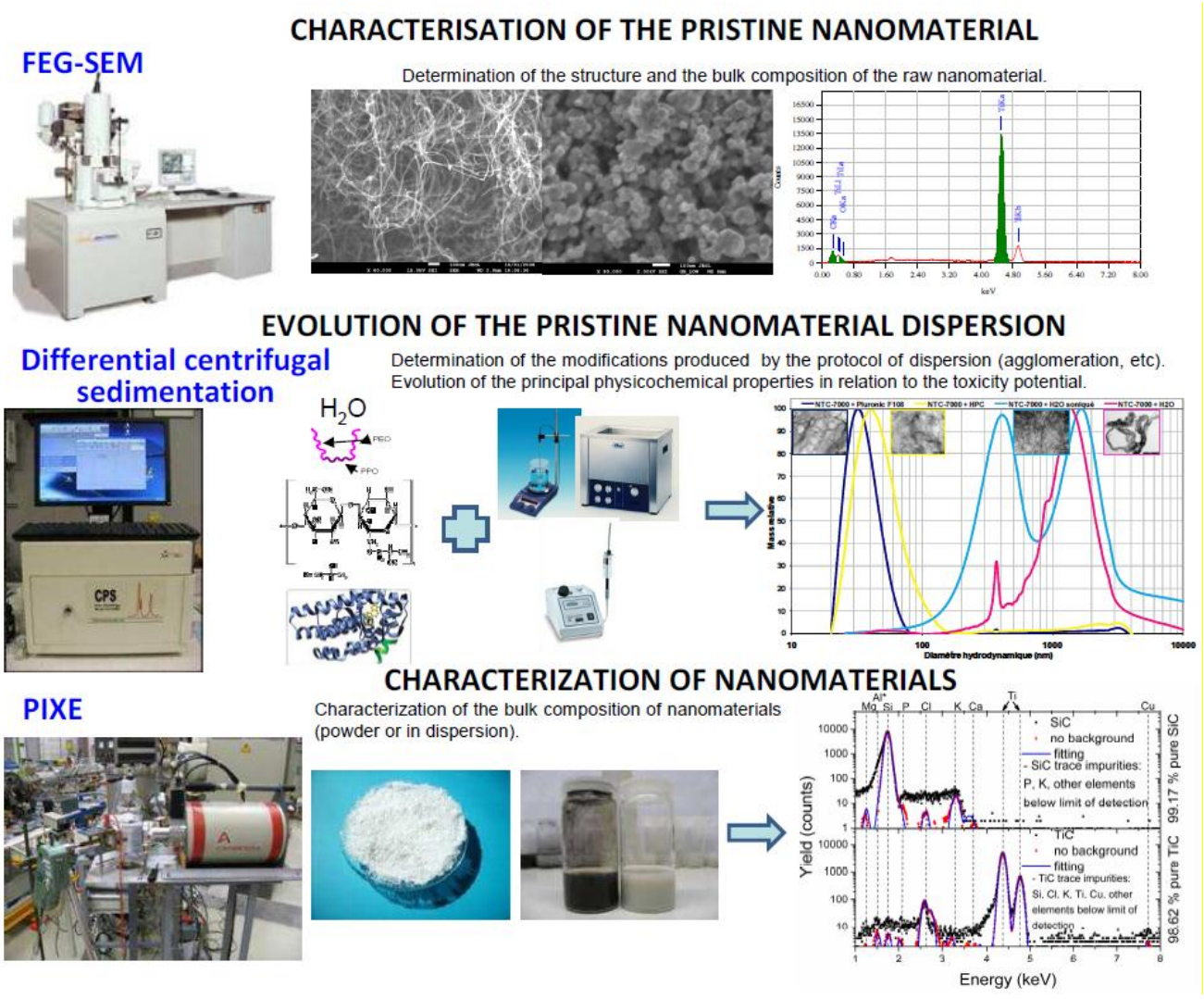
- Extremely high resolution compared to methods based on light scattering or particle counting, with clean separation of narrow peaks that differ in size by as little as 2%.
- Very high sensitivity, with a minimum detection limit below 10^{-8} gram active sample for narrow peaks. Highly reproducible results; the typical 95% confidence range for reported peak sizes in replicate analyses is +/-1% or better.

- Measurement of particles of virtually any density, including those equal to or less than the density of the fluid in which they are suspended. Validation from the IRMM (Institute for Reference Materials and Measurements, JRC, Geel, Belgium).
- Instrument suited to study the kinetics of nanomaterial dispersions in several kinds of dispersants, culture media, etc. Sedimentation of the nanomaterials (instability dispersion) is not a drawback for the technique.
- Limitations: nanomaterial density higher than water.

PIXE (Ion beam accelerator): Bulk characterization of nanomaterials (pristine and in dispersions)

- Multi-elemental analysis (any element with an atomic number higher than Z=10).
- Solid/liquid samples: emulsions, dispersions, radiolabeled/radioactivated/fluorescent nanomaterials, etc.
- Fast, highly sensitive and high volume measurements.
- Limitations: The nanomaterial elemental composition should not be the same as the matrix (or be in very low amounts). Example: SiO_X nanoparticles in silica dispersion.

Typical Samples & Images:



Any further information:

- Effects of the dispersion methods in Pluronic F108 on the size and the surface composition of MWCNT and their implications in toxicology assessment. Mejia J, Tichelaar F, Saout C, Toussaint O, Masereel B, Mekhalif Z, Lucas S & Delhalle J. (2011) *J Nanoparticle Res*, 13: 655-667.
- Development of a PIXE analysis method for the determination of the biopersistence of SiC and TiC nanoparticles in rat lungs. Lozano O, Mejia J, Masereel B, Toussaint O, Lison D, Lucas S. (2011). Nanotoxicology (doi:10.3109/17435390.2011.572301).
- Cytotoxicity of multi-walled carbon nanotubes in three skin cellular models: effects of sonication, dispersive agents and corneous layer of reconstructed epidermis. Vankoningsloo S, Piret JP, Saout C, Noël F, Mejia J, Zouboulis C, Delhalle J, Lucas S & Toussaint O. (2010). *Nanotoxicology*, 4: 84-97.