

X-Ray Diffraction (XRD)

Category:

C. Particle Characterisation in and ex-situ

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Short technology description/Overview:

Maybe the most versatile and widely used characterization tool for materials science and now also for nanotechnology. XRD is based on the optical interferences when a monochromatic radiation pass through a gap that have the same length than its wavelength. As X-Ray have similar wavelengths than the interatomic lengths inside crystals (in the order of Angstroms) when pass through a crystal, this acts as a diffracting net: diffracts the x-rays with a concrete direction and intensity and a pattern of peaks is obtained.

Sample preparation and experimental procedure.

- If synthesis are performed in liquid phase, preparation of the samples is made by extracting out the solid phase of the colloids. For this purpose, destabilization of the NPs mixing the colloid with a solvent of different polarity is followed by soft centrifugation after which NPs precipitated. The supernatant is discarded, and the pellet of NPs is dried in an oven to eliminate all the moisture.
- XRD spectra are acquired using a PANalytical X'Pert diffractometer operating with a Cu K α radiation source ($\lambda=1.541\text{\AA}$) or Co K α ($\lambda=1.789\text{\AA}$).

Sample requirements for NPs below 100 nm.

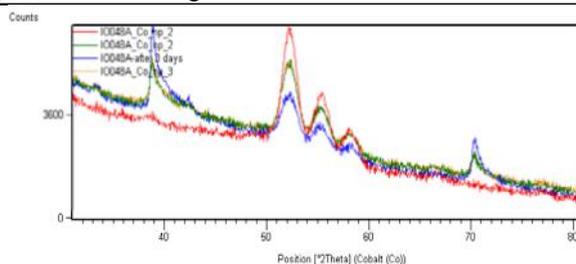
Samples for XRD consist on the NPs in powder form. The more powder, the higher the intensity of peaks.

Main Features (Equipment Capabilities):

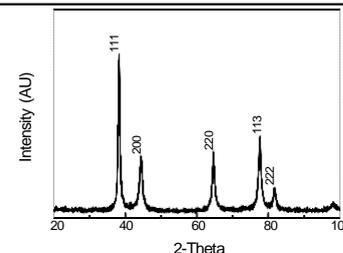
This pattern provide information about

- Composition of the NPs (and purity).
- Arrangement of the atoms inside the NP (crystalline phase).
- Size of the crystal using Scherrer's formula.
- Size, crystal face, purity, crystal defects, degree of oxidation

Typical Samples & Images:



Monitoring oxidation of Co NPs



Typical XRD spectrum for Au NPs

Any further Information: