

Nanopowder Synthesis and Metrology

The development and testing of protocols and powders for reliable nanoscale characterization .

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Nanotechnology

Our research

❖ Emergence of new materials

▪ Accurate measurement of **material properties** (size, particle size distribution, surface, composition...)

Develop detailed protocols for most common characterization methods at the nanoscale.

Characterization methods at the nanoscale

❖ Environmental impact
❖ Health impact

Detection of nanoparticles
 ▪ in the environment (air, sea, food...)
 ▪ in human body

Produce reference materials, cheaper, and in higher amounts, to be able to test and improve characterization methods.

Development of detailed protocols

List of protocols developed for most common characterization techniques used in powder technology

These protocols were developed in the COST Action, to get more reliable and reproducible data, particularly at the nanoscale. Instrument specific BUT basis for host-lab protocol development.

They are freely available on the LTP website: <http://ltp.epfl.ch> / Research / Powder Characterization / Nanometrology

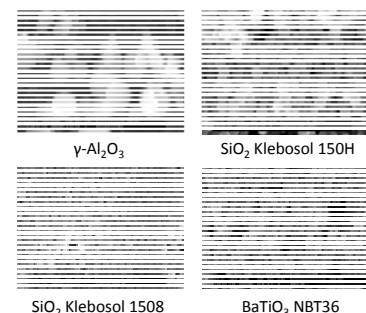
Measurement	Method	Acronym
Microscopy	Scanning Electron Microscopy	SEM
	Transmission Electron microscopy	TEM
Particle size distribution (PSD)	X-Ray Disc Centrifuge	XDC
	Centrifugal Particle Sizer (light absorption/scattering)	CPS
	Laser diffraction	LD
	Dynamic light scattering	PCS
Specific surface area	Nitrogen adsorption, BET method	SSA
Pore size distribution-volume, specific surface area	Nitrogen adsorption-desorption, BET method	ASAP
Density	He pycnometry	pycno
Zeta potential	Phase Analysis Light Scattering	Zeta PALS
	Electroacoustics	Acousto
Phase identification, crystallite size	Powder X-Ray Diffraction	XRD
Temperature behavior	Thermogravimetry	TGA

Testing of protocols: use of internal powders

Four internal reference powders (well-defined in composition, size, morphology) were used to develop and test the protocols.

Depending on the quality of the powder, results obtained between methods and instruments can be different.

Reference powders	LD Dv ₅₀ [µm]	XDC Dv ₅₀ [µm]	PCS Dv ₅₀ [µm]	CPS Dv ₅₀ [µm]	Zeta potential at pH 6 [mV]	SSA [m ² /g]	D _{BET} [µm]
γ-Al ₂ O ₃ Nanotek	0.210	0.061	0.055	0.099	13.8±1.9	39.7±0.3	0.044
SiO ₂ Klebosol 150H	0.060	0.053	0.082	0.083	-9.2±1.8	49.0±0.4	0.056
SiO ₂ Klebosol 1508	0.090	0.039	0.056	0.056	-9.7±3.2	77.8±0.2	0.035
BaTiO ₃ ORG NBT36	0.190	0.164	0.130	0.162	-5.6±1.4	10.8±0.1	0.096



➔ Always describe which method is used when giving a result on size or size distribution of a powder.

▪ BaTiO₃: high quality powder → very reproducible results, with a very small standard deviation even with significant variations in the aliquot concentration.
 ▪ γ-Al₂O₃: lower quality (bimodal, or agglomerates) → differences between methods and instruments.

➔ Need of optimum sample preparation and image analysis to help choose the most accurate method for characterising the PSD.

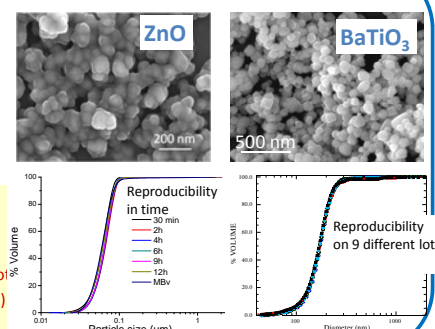
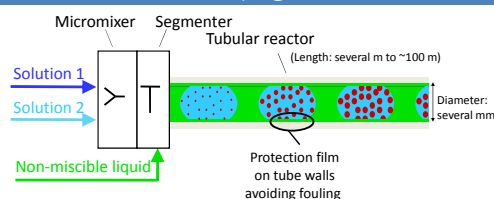
The use of measurement protocols proved to be necessary for **reliability** and **reproducibility** of measurements. They constitute a strong basis and are necessary for comparison of results obtained on different instruments or from different users. These protocols were tested on a series of powders (TiO₂, BaTiO₃, hydroxyapatite Ca₁₀(PO₄)₆(OH)₂, and LaNiO₃) within the framework of the **COST action 539** and allowed synthesis routes to be better evaluated and optimised. [http:// www.cost539.cms-bg.net/](http://www.cost539.cms-bg.net/)

Production of Internal Reference Powders with the SFTR (Segmented Flow Tubular Reactor)

There is a need of reliable "reference" powders, in larger amounts and cheaper than the Standard Reference Materials generally available. In this context, the SFTR (Segmented Flow Tubular reactor) is a powerful tool which allows the continuous production of **kg-size batches powders**. The SFTR has proved its **versatility** and its **robustness** through the preparation of different products (BaTiO₃, ZnO, copper, calcium and cobalt oxalates) with a high **powder quality** (chemical and phase composition, particle size, and shape) and **reproducibility**.

- Particles created in limited time period
- Controlled ageing conditions
- Independent micro reactor
- ❖ No fouling within whole reactor
- ❖ Scale-up production by multiplication of reactor units

- Controlled particle size & narrow particle size distribution
- Controlled particle morphology
- ❖ Excellent reproducibility from lot to lot
- ❖ Transfer from lab to production (pilot)



Acknowledgements : Swiss COST Office for funding and action support.