Physicochemical characterization of manufactured nanomaterials (TiO_2, SiO_2) used for genotoxicity testing

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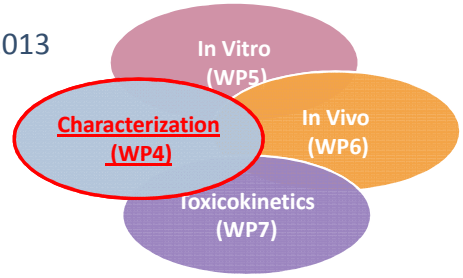
NANOGENOTOX — European Joint Action 2010-2013

- Methodology to test genotoxicity of manufactured nanomaterials (MNs) (harmonized protocols)
- Relevant and reliable data for risk assessment by public health authorities



Coordination (WP1)

Dissemination (WP2)

Evaluation (WP3)

❖**WP4** (5 institutions – 4 countries)

Raw material Powder - dispersion → **Dispersion protocols** → **Suspension in biocompatible medium**

4 TiO_2, 4 SiO_2, CNT

From JRC repository *

❖Detailed physico-chemical properties

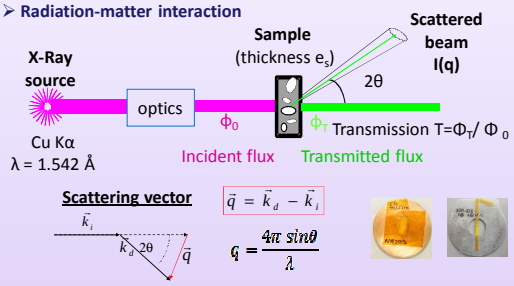
Size, shape, polydispersity, specific surface area, impurities, surface modification, suspension stability, etc.

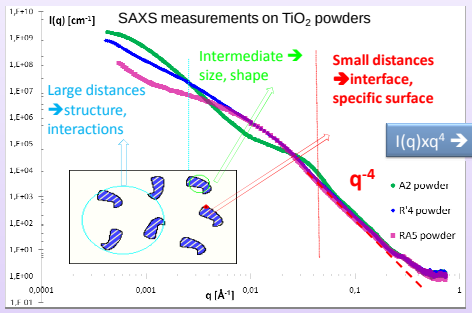
❖Characterization techniques

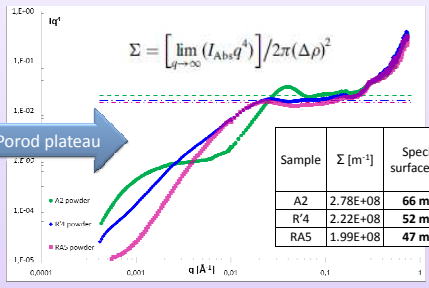
XDR, Raman, TEM, SEM, **AFM**, BET, **SAXS**, ICP-MS/ AAS, MALDI-TOF, Dustiness, **DLS**, **zetametry**, redox, etc.

Small Angle X-ray Scattering (SAXS)

➢ **Radiation-matter interaction**

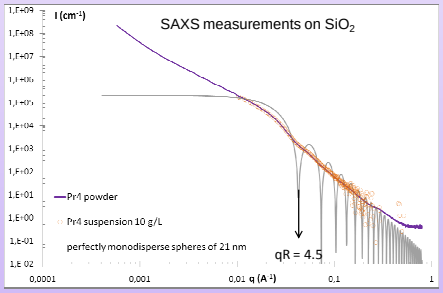




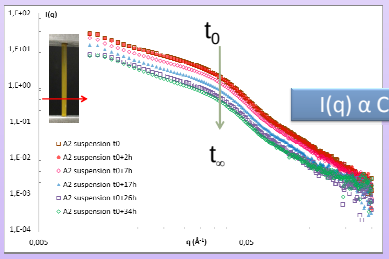


Sample	Σ [m⁻¹]	Specific surface area	Equivalent diameter for spheres
A2	2.78E+08	**66 m²/g**	**22 nm**
R'4	2.22E+08	**52 m²/g**	**27 nm**
RA5	1.99E+08	**47 m²/g**	**30 nm**

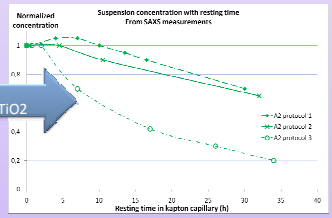
➢ Specific surface area



➢ Size
➢ Polydispersity
➢ Aggregation state
➢ Experimental data fitted by models of size distribution (nanostructure)



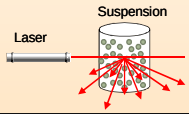
I(q) α C_{TiO2}



➢ Stability and aggregation over time

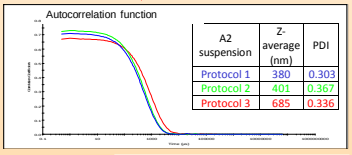
Dynamic Light Scattering (DLS) - Zetametry

❖ **Size measurement (hydrodynamic diameter)**



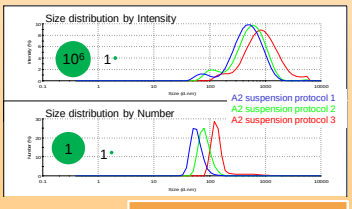
Stokes-Einstein

$$d_h = \frac{kT}{3\pi\eta D}$$



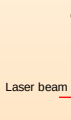
A2 suspension	Z-average (nm)	PDI
Protocol 1	380	0.303
Protocol 2	401	0.367
Protocol 3	685	0.336

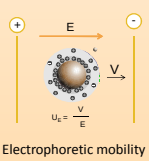
➢ Aggregation state



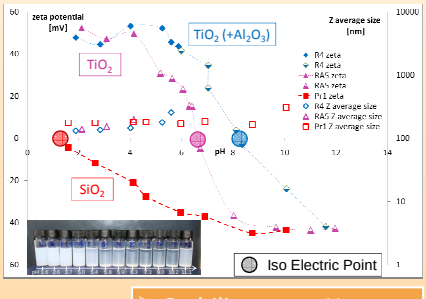
➢ Size distribution

❖ **Zeta potential measurement (surface charge)**

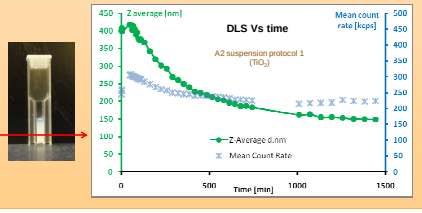




$$U_E = \frac{2\varepsilon\zeta f(\kappa a)}{3\eta}$$

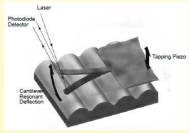


➢ Stability over pH range

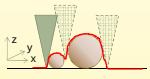


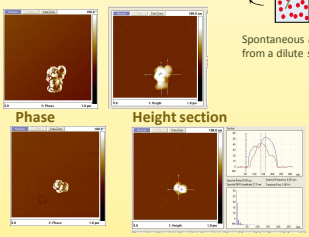
➢ Stability over time

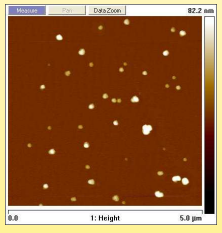
Atomic Force Microscopy (AFM)



➢ Z ⇨ accurate measurement (nm), independent of the probe
➢ x, y ⇨ convolution of the probe diameter







➢ Shape
➢ Aggregation state
➢ Height distribution

➢ Statistics (size, polydispersity)

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Executive Agency for Health and Consumers

http://www.nanogenotox.eu/

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