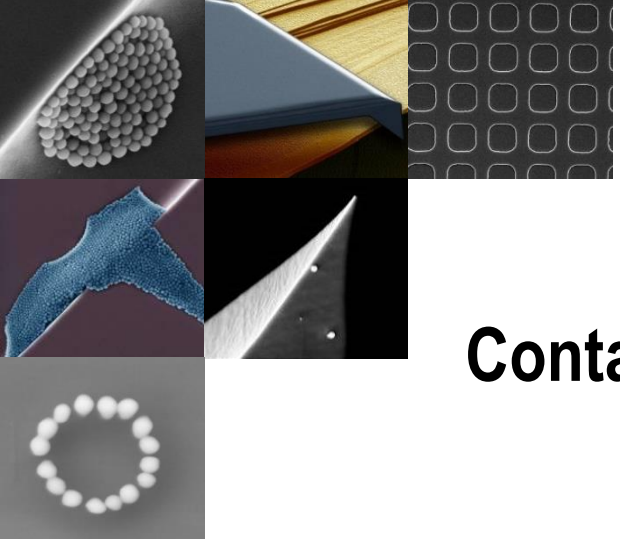
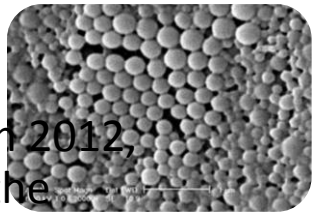


Contact less In situ Nano Particle Size monitoring using fiber remote Dynamic Light Scattering

Dr Sylvain Boj– Cordouan Technologies

Prague- September, 2015

The advent of NMs & NPs : a new era in science and industry



- Promise of major technologic, economic and societal impacts

- The global market of nanoparticles in life sciences was estimated at $22B in 2012, $25B in2013 and reached approximately $30B in 2014 - a 19% CAGR over the previous year.

-> From 2014 to 2019, revenues are projected to increase to $80B at a 22% CAGR.

- NPs and NMs already in the field : cosmetics, batteries, paints, inks, food, medicines, advanced coatings, aerospace, etc….. And it is just the beginning!





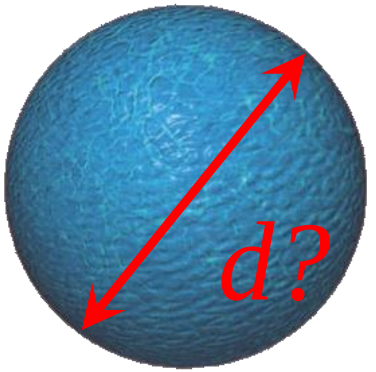
- **Booming demand requires to scale up production installations**

- **Absolute need for New monitoring tools to migrate NPs from R&D labs to pilot plant and mass production (incoming material control, process control, quality control, etc)!**

For NPs, size matters!

Properties related to NP size

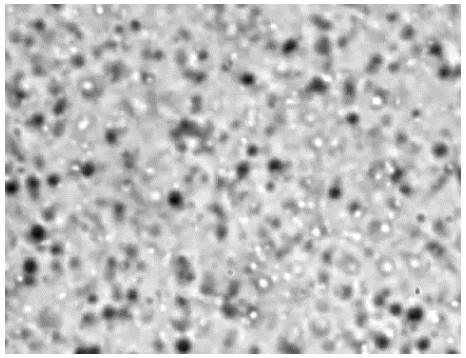
- Specific surface of the particles (catalyzer)
- Ability to penetrate membranes or interact with surface
- Aggregation and stability of suspensions
- Functionnalisation and self assembly capabilities
- Optical, mechanical and electrical properties, etc,



Many mature characterization techniques for particle size:

- Electronic Microscopy (TEM), SAXS, AFM
- Electrozone Coulter counter
- Mass sensing: Differential Centrifugal Sedimentation, resonant mass detection
- Optical : Particle tracking, Laser Diffraction, **Dynamic Light Scattering (DLS)**

DLS principle: optically probe the Brownian motion



Assumption: particles = hard spheres without interactions

time

$$< X^2(t) > \sim 2\,D\,t$$

Diffusion coefficient

L. BACHELIER (1901)

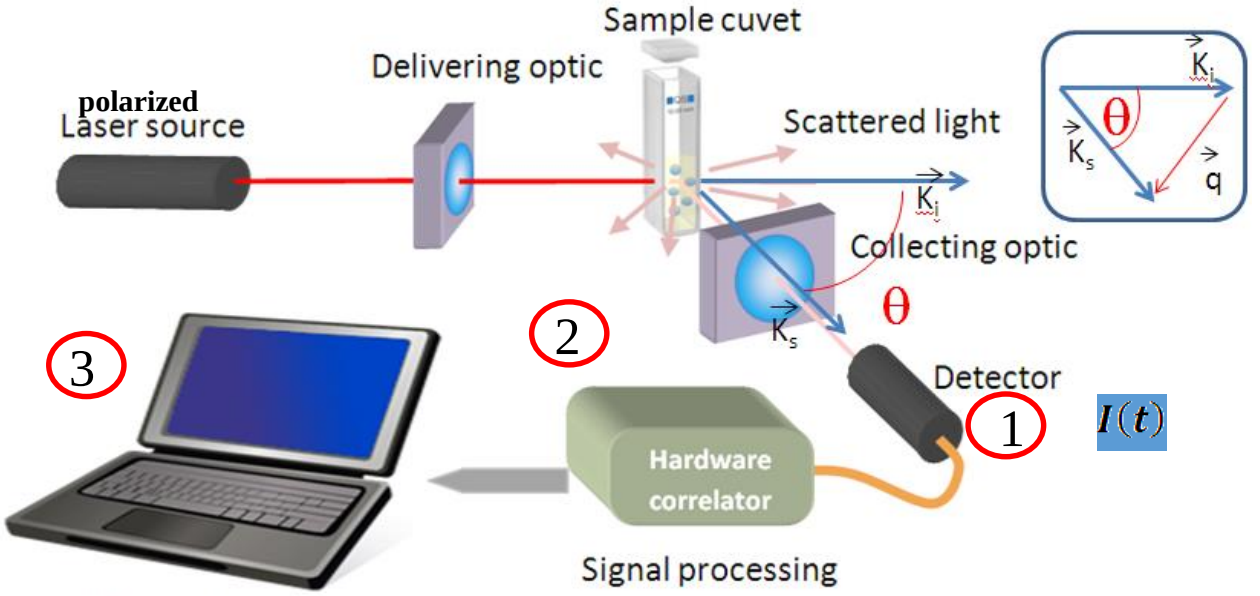
EINSTEIN (1905)

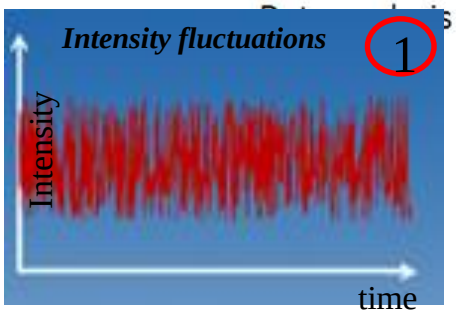
Boltzmann

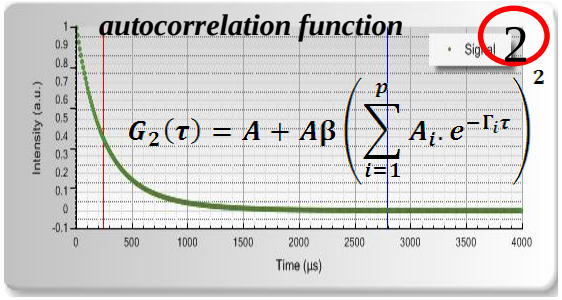
Temperature

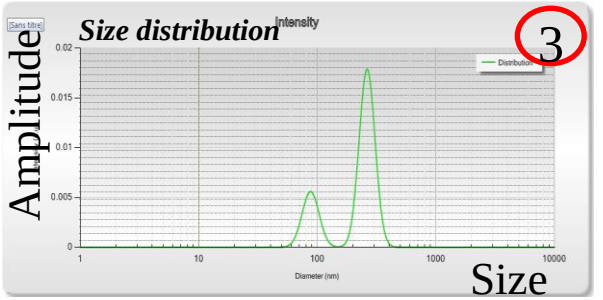
$$\emptyset_H = \frac{K_B T}{3\pi\eta D}$$

Viscosity



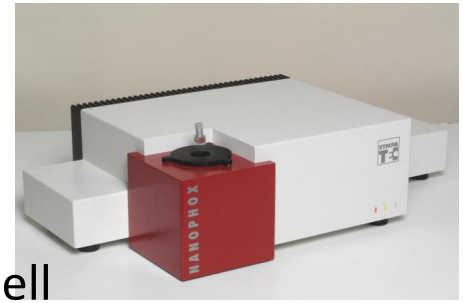




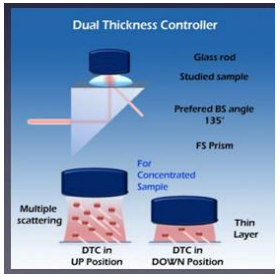


DLS equipments until today

Disposable cell



Embedded cell



- Mature and standardized method (ISO 13321 (1996) & ISO 22412 (2008)
- Bench top configuration: solutions dedicated to laboratory analysis
- Requires batch sampling: bring the sample to the measurement!
- Need sample preparation: filtering, diluting,
- Time consuming
- Risk of contamination or sample degradation

➢ **Need for a new approach for process monitoring!**

A change of paradigm:"bring your measurement to your process!"

No more batch sampling

Remote DLS probe

Features:

- In situ & Non invasive
- No need for batch sampling
- Small footprint
- Adjustable working distance /scattering angle
- Alignment laser for easy installation
- High accuracy remote temperature sensor
- Easy maintenance
- Ideal for measurements in hermetic environments (glass capillaries, reactors, autoclave, Gloves box, etc)

Example 1

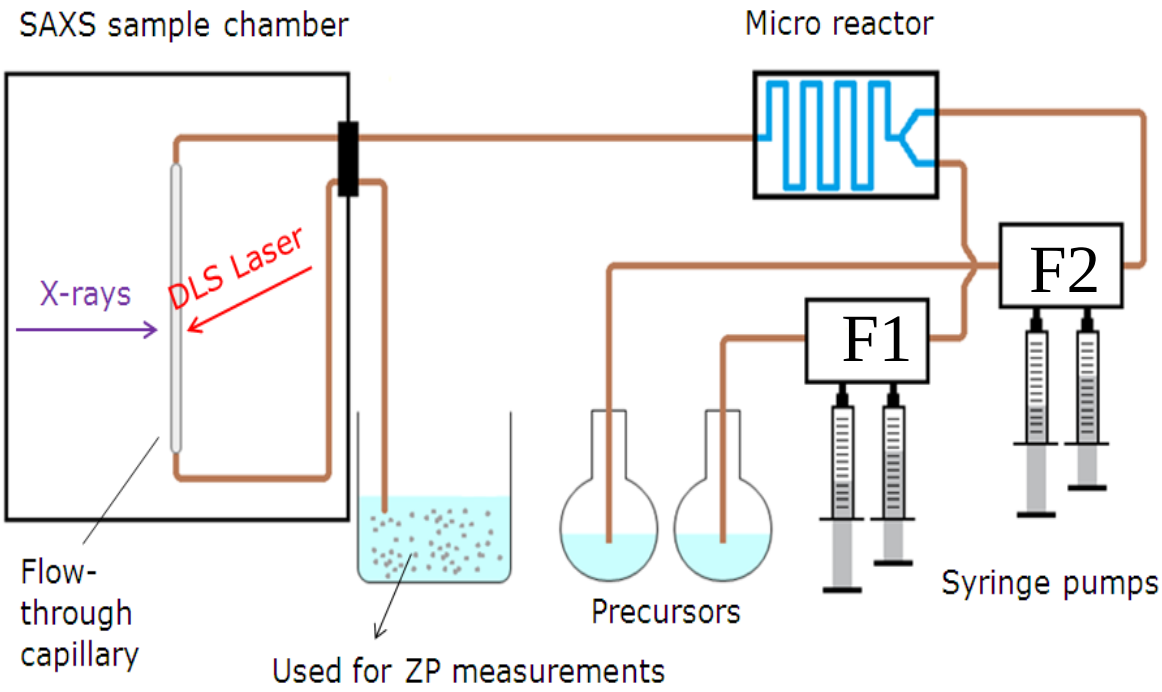
Combined Remote DLS & High flux SAXS for NPs synthesis monitoring

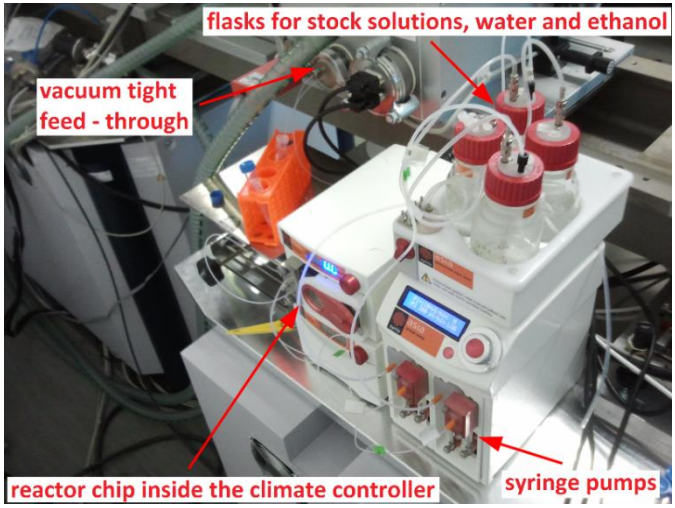
SNOW CONTROL FP7 Project

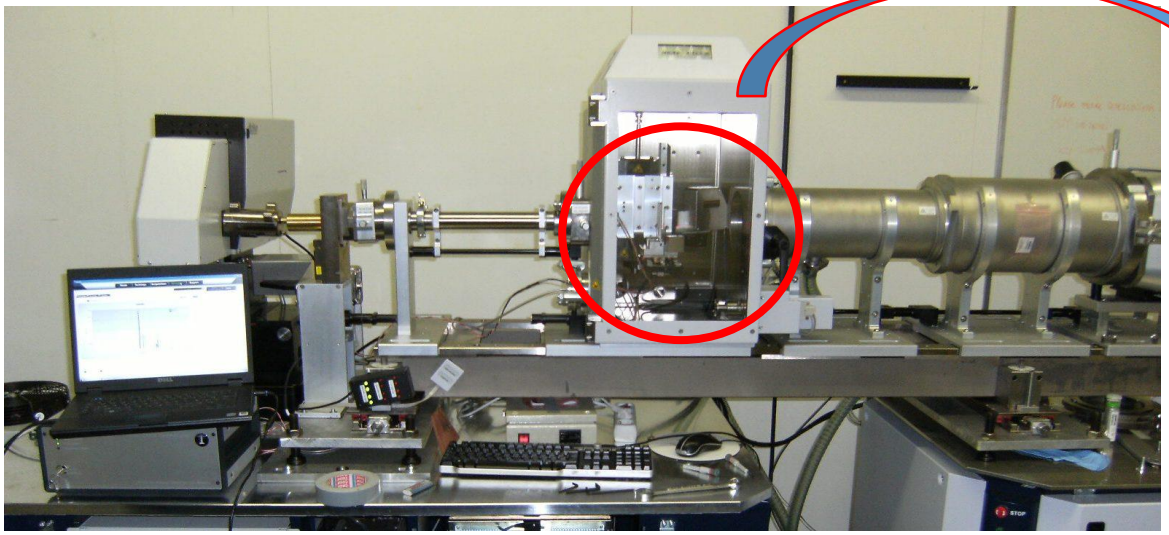
(https://fys.kuleuven.be/apps/snowcontrol/dissemination.php)

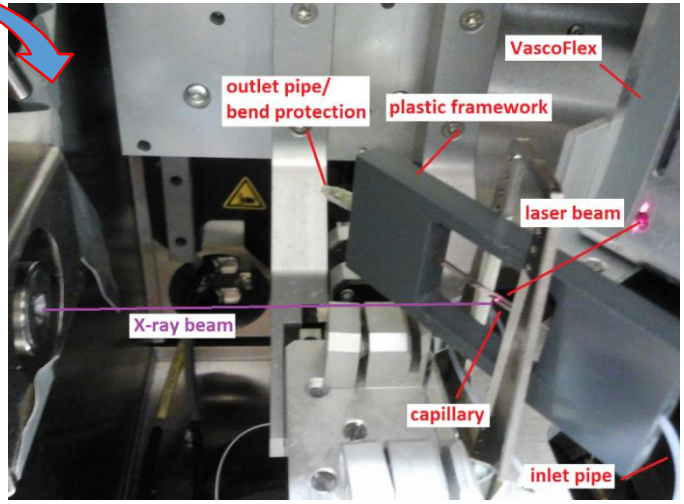
Collaboration with: Bruker gmbh, University of Leuven, IBM Zurich, DCA, Chemstream

Combined Remote DLS & High flux SAXS





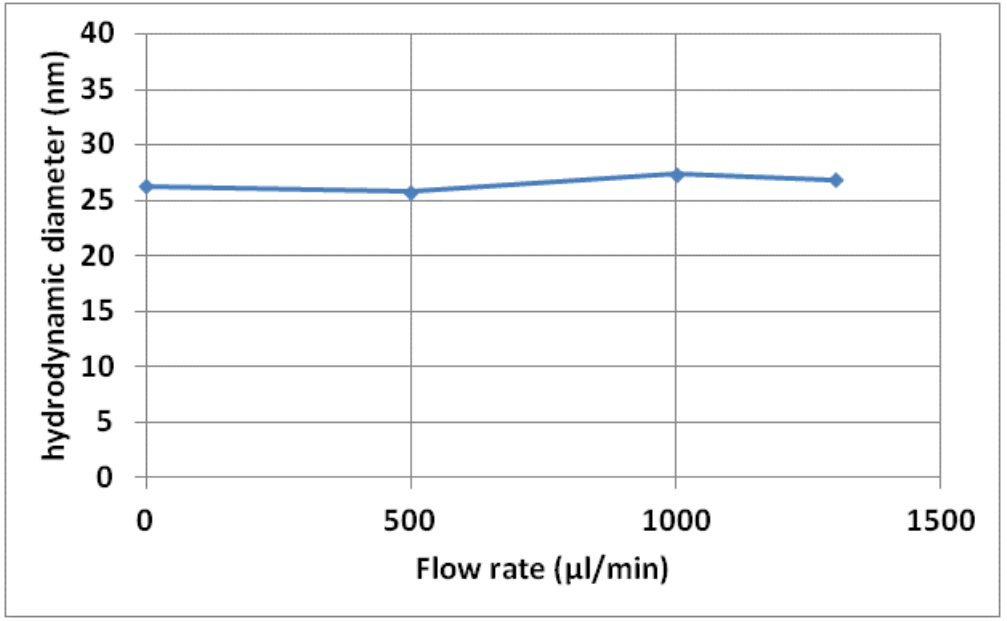




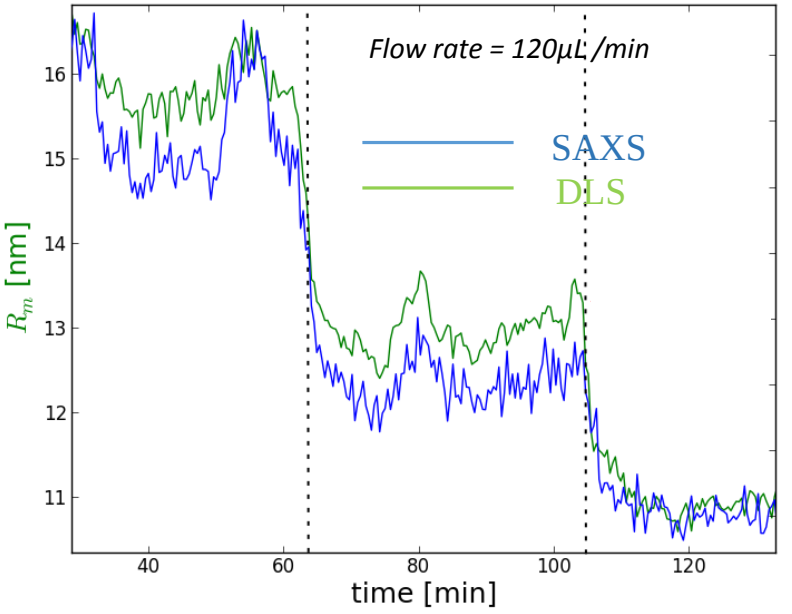
On line SiO2 NPs synthesis monitoring

Hydrolysis –condensation method : TEOS in Ethanol (F1) + NH3 in H2O (F2)

Impact of flow rate (F1+F2)



Impact on precursors mixing ratio (F1/F2)



- **Consistent results between SAXS and DLS measurements**
- **Allow to track and tune synthesis process in an accurate way**

Combining SAXS and DLS for simultaneous measurements and time-resolved monitoring of nanoparticle synthesis

A. Schwamberger & al, Nuclear Instruments and Methods in Physics Research B 343 (2015) 116–122

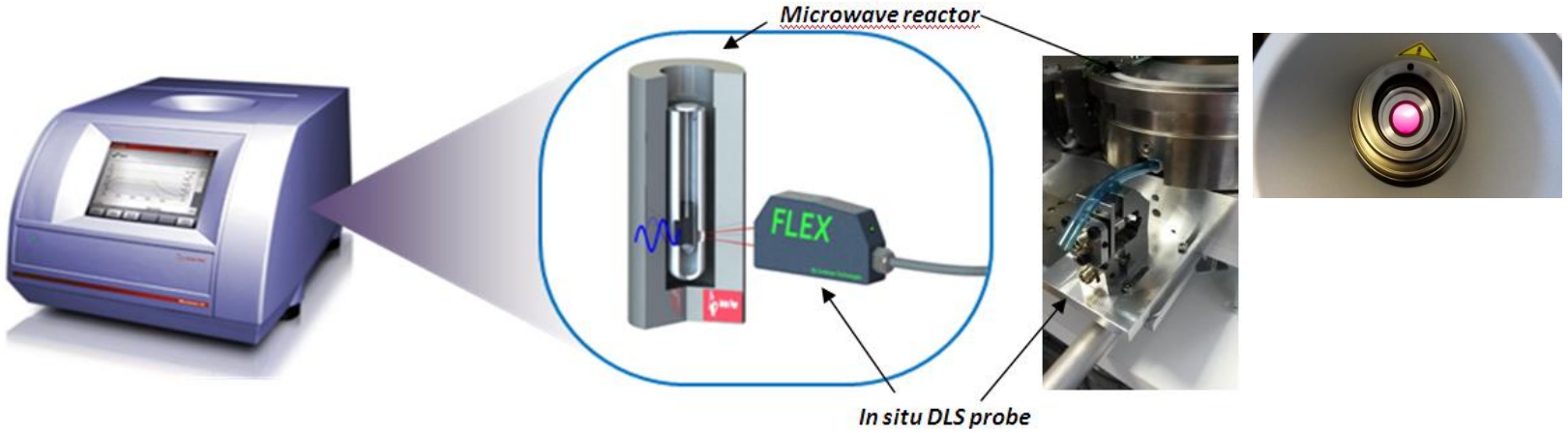
Example 2

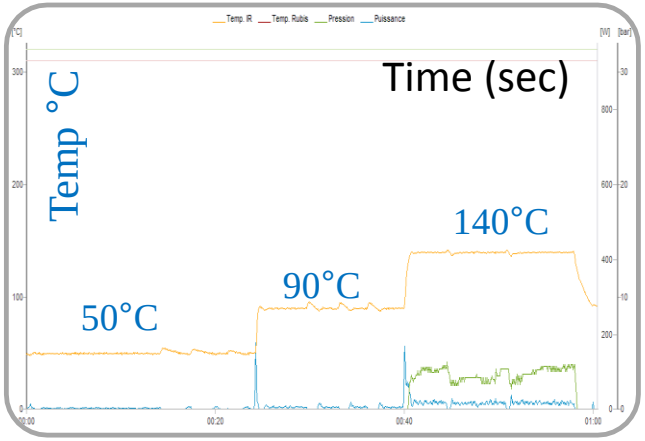
In situ kinetics monitoring of Microwave assisted NPs synthesis

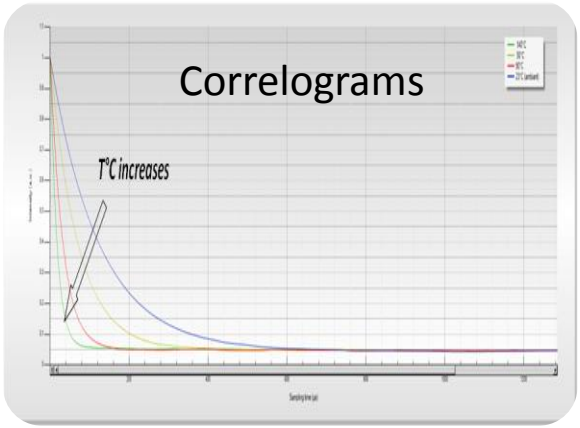
Collaboration with Anton Paar

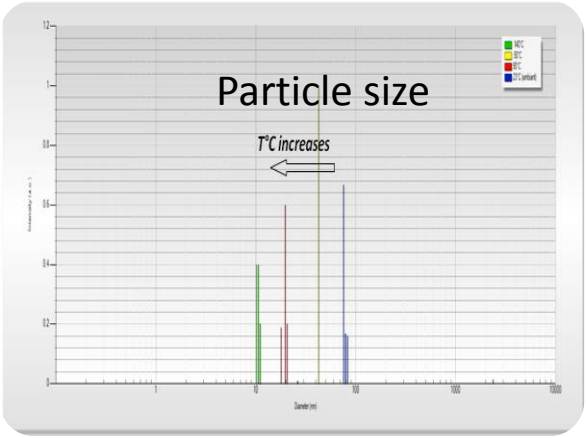
In situ monitoring of Microwave assisted NPs synthesis

The idea: replace camera monitoring by DLS probe!









- **Very consistent and reproducible results**
- **1st demonstration ever done opening up new possibility on NP synthesis monitoring**

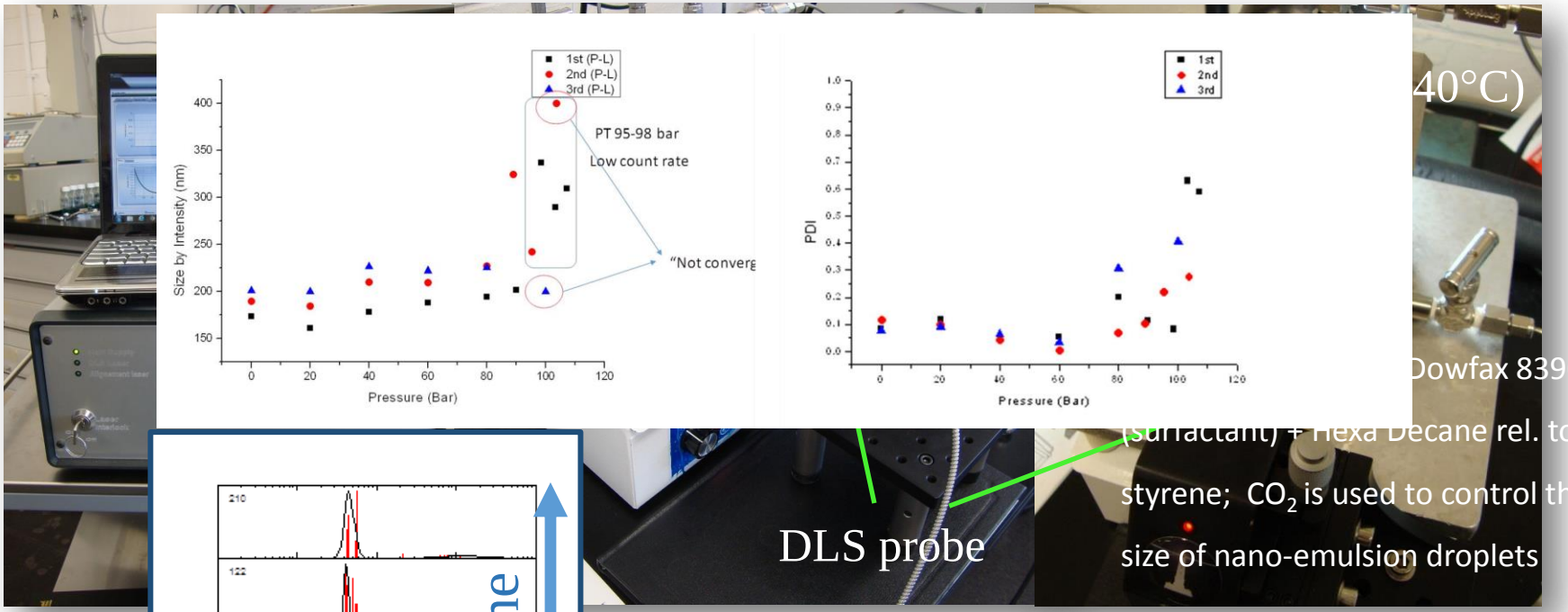
Example 3

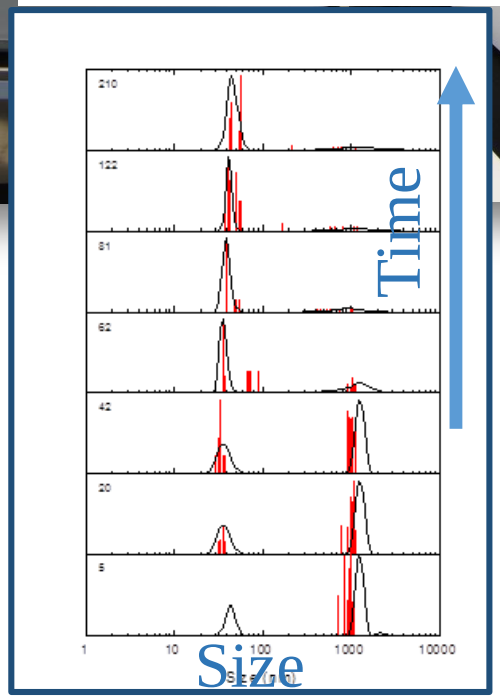
Polymer Nano Emulsion & NP synthesis monitoring in supercritical CO_2 reactor

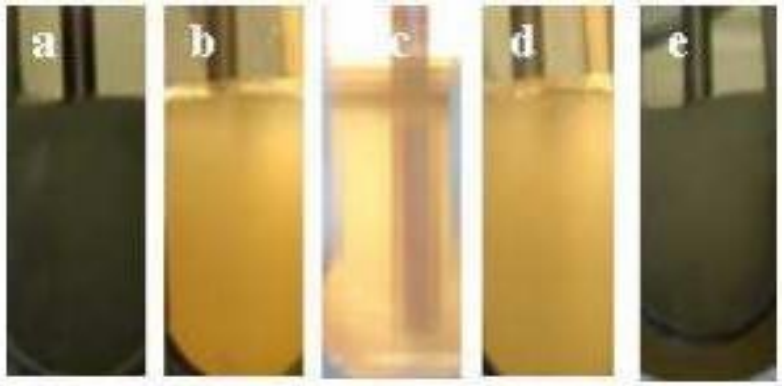
Research project at University of New South Wales (Australia)

Measurement inside SC CO_2 synthesis reactor

- Use DLS measurements to correlate turbidity variation with particle size
- Implement accurate control of the size of monomer droplets/NP

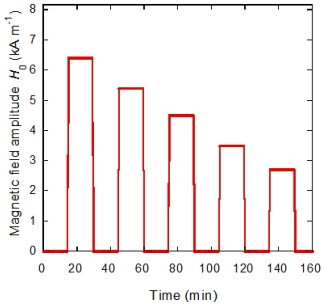


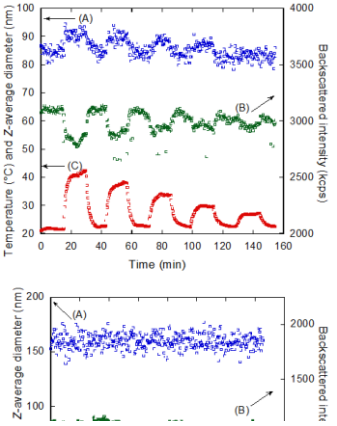




Other examples of use

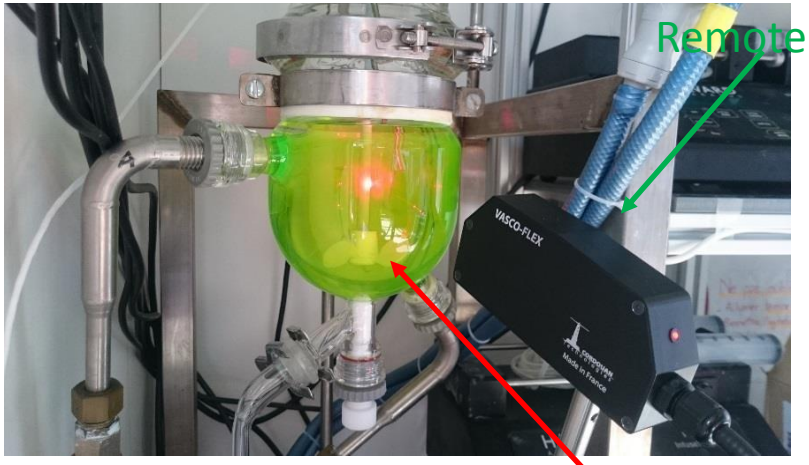
Magnetic Hyperthermia experiment on NPs for Bio med applications

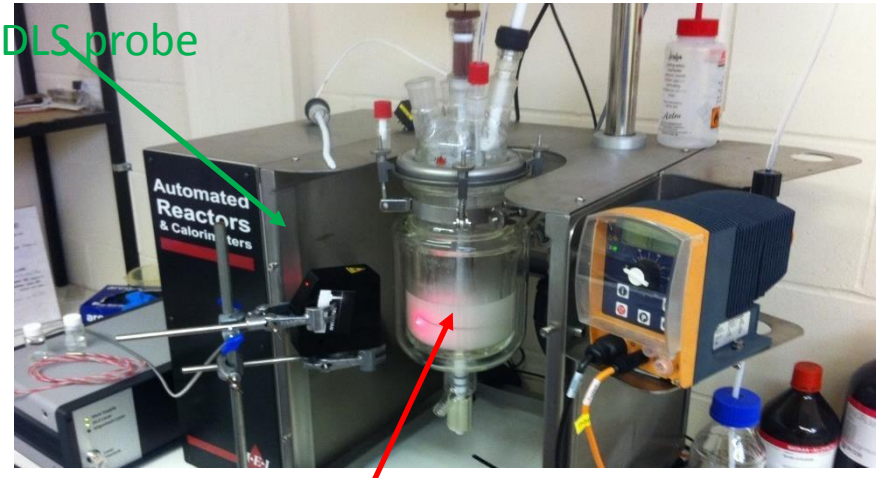




Polymer-grafted iron oxide nanoparticles as thermosensitive MRI contrast agents and magnetic nano-heaters, Gauvin Hemery & al, , Journal of Physics D: Applied Physics, to be published

Remote DLS coupled to commercial synthesis reactors

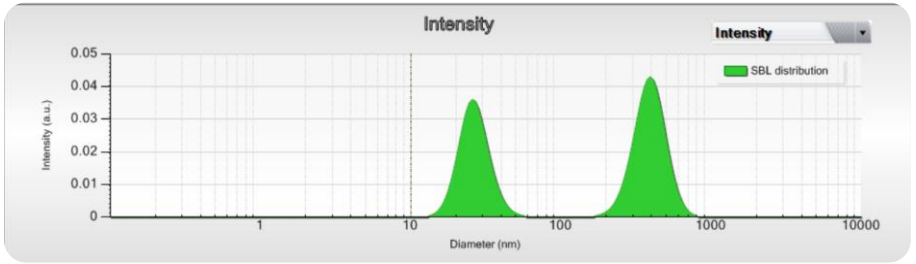




Conclusion and perspectives

- Remote in situ DLS is opening up new field of application for particle size measurements
- Concept already demonstrated in the field into various environments
- Extending application fields: (coupling to micro/milli fluidic reactor, continuous flow reactor)
- And DLS is not just about particle size: local Temperature probe, Nano-rheology probe, precipitation/ xtalization study, etc

And it's just the beginning of the story……



Thank you for your attention !

Sylvain Boj– COO

33 600 Pessac
FRANCE

www.cordouan-tech.com