## Small-angle X-ray Scattering (SAXS) for Studies of Soft, Biological and Hard Matter

## **Reidar Lund**

Dept. of Chemistry University of Oslo Email: reidar.lund@kjemi.uio.no



Bio-world

Nanomedicine

Catalysis-world

# Small-angle scattering reveals the organization of many atoms:

Polymer-world



## Structural *in situ* Studies of e.g. Biological Materials

We want to say something about this:



But often we only measure this:



## Small-angle scattering provides *in situ* information in solutions - complementary to crystallographic methods

Frozen croaker fish



## Small-angle Scattering: length scales



## **S**AXS at the Department of Chemistry @UiO (REXC)

#### Bruker "Nanostar" SAXS



#### **Optimized for weak solution scattering**

High flux (~ 8.0E7 cps), low background, quantitative background subtraction, calibrated on absolute scale

Multi-sample scans

Flow-through cell for solutions

Peltier elements for precision temperature scans



## What can we do with SAS?



-196 - 300 C

X-Y-stage for scanning SAXS

Grazing-incidence SAXSlateral structures on surfaces



## SAXS: wide range of systems in the nano-range **biology**



nanoparticles

polymers

## Hard Matter

nanoparticles



#### Porous rocks



Catalysts







## Hard Matter: nanoparticle synthesis

#### Color depends on nanoparticle size and shape



#### **Reactivity depends on nanoparticle size**



Rao et al. Chem. A 2002

Size, distribution and shape are essential. - Control of the process - control of properties

## Hard Matter: nanoparticle synthesis

Example: nucleation & growth to a final particle



## Nanoparticle synthesis: in situ observation by SAXS



Abécassis, B., Testard, F., Spalla, O., and Barboux, P. (2007) Probing in situ the Nucleation and Growth of Gold Nanoparticles by Small-Angle X-ray Scattering., *Nano Lett.* 7, 1723–1727.

## Nanoparticle synthesis: in situ observation by SAXS



σ (nm)

Time (s)

SAXS provides complete quantitative information on particle growth, size and distributions

## Soft Matter





#### **Block Copolymer Micelles**

#### Dendrimers



#### Mesoscopic soft crystals

### More Moore? - nanomaterials for storage devices

#### A hunt for smaller and smaller storage units



Reidar Lund, UC-BReidar Lund, UiO, 14 Feb. 2013

## Mesoscopic Soft Crystals: block copolymer melts



SAXS reveals ordering and detailed structure of soft crystals for nano-lithography



## Nano-carriers for drug-delivery



Self-assembly of n-alkane-Poly(ethylene oxide)

SAXS data from RECX



## Reveals detailed overall morphology + internal structure of micellar nano-carriers

with Dr. T. Zinn (UiO)



## Proteins in solution

#### Model protein: lysozyme



#### SAXS provide rapid characterisation of proteins in solution



## Which structure does the protein adapt in solution? Will determine enzymatic function

## **Structure of eEF3 and the mechanism of transfer RNA release from the E-site**

nature

Christian B. F. Andersen<sup>1\*</sup>, Thomas Becker<sup>2\*</sup>†, Michael Blau<sup>2</sup>, Monika Anand<sup>3</sup>, Mario Halic<sup>2</sup>†, Bharvi Balar<sup>3</sup>, Thorsten Mielke<sup>4</sup>, Thomas Boesen<sup>1</sup>, Jan Skov Pedersen<sup>5</sup>, Christian M. T. Spahn<sup>6</sup>, Terri Goss Kinzy<sup>3</sup>, Gregers R. Andersen<sup>1</sup> & Roland Beckmann<sup>2</sup>†

From Prof. Jan Skov Pedersen (Aarhus University)



SAXS gives direct quantitative information on molecular weight, shape, size and interactions at the nanoscale.

High-flux / low background at SAXS/RECX give good data even for weakly scattering samples

The instrument can be used for virtually any type of samples.

*In situ* studies may give unique insight into kinetic process



## Welcome to the SAXS instrument in the new REXC-lab!

## You are most welcome to discuss possible experiments and ideas with us!

**THANK YOU** 

#### **nstrumentation:** examples

#### Polymer Colloid Group @ Dept. Chem. UiO

Multi-angle Dynamic and Static Light Scattering



SANS at IFE, Kjeller



Rheometer



**Rheo-SALS** 



#### International Facilities

#### D11 at ILL, Grenoble



ID02 at ESRF, Grenoble



### **Surfactant Solutions:** kinetics of transformation



Fit model: coexistence worm-like and spherical micelles

Reidar Lund, Soft Matter, SynkNøyt 21 Oct. 2013

## **Surfactant Solutions:** kinetic pathways



Reidar Lund, Soft Matter, SynkNøyt 21 Oct. 2013

## Conclusion

- SAS and LS provide unique possibilities to investigate **nanostructures** *in situ* without perturbations.

- Together with light scattering, SAXS/SANS provides a **broad length scale**: from Å's to micrometers.

- SANS and contrast variation allow structural details to be highlighted and renders molecular transport processes visible (not shown).

- Time-resolved SANS/SAXS offers both structural and temporal resolution - **unique insight into kinetic processes.**