NanoInnovation 2019 - June 11-14, Rome

Applicazioni tecnologiche della diffrazione con raggi-X

Una piattaforma SAXS/GISAXS/WAXS

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SAPIENZA
UNIVERSITÀ DI ROMA

NanoInnovation 2019 - June 11-14, Rome

- **SAXSLab Sapienza: a large university equipment**

- **SAXS/GISAXS/WAXS (Small/Grazing Incidence Small/Wide Angle X-Ray Scattering) platform for structural characterization of solid and solution systems of meso and nanoscopic phases**

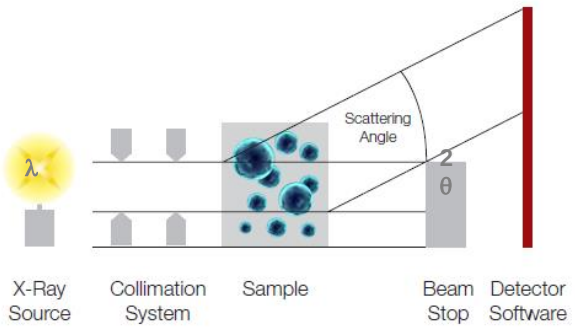
DESCRIPTION OF EQUIPMENT

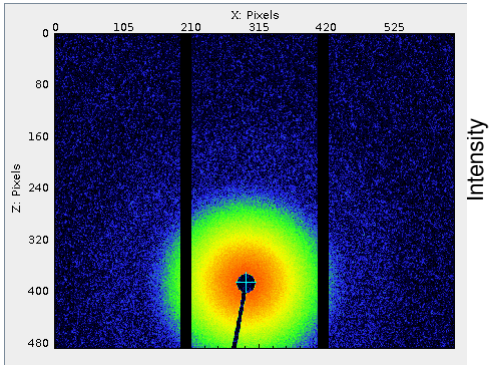
Xeuss 2.0 – Q-Xoom @ *SAXSLab Sapienza*

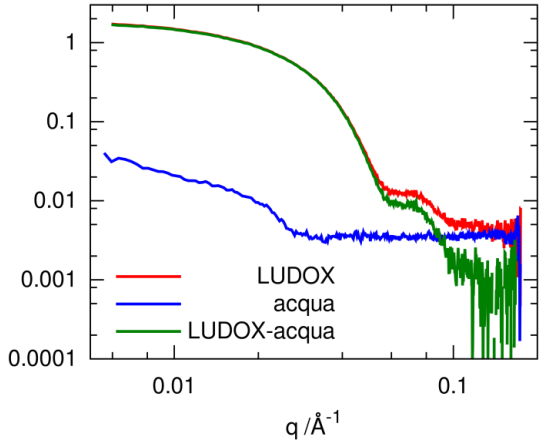
A large university equipment

What is the technique SAXS?

- The technique is based on the diffusion phenomena of X-rays from the electrons contained in the sample and on the interference between the diffused waves







Any technique based on the diffusion follows a law of reciprocity :

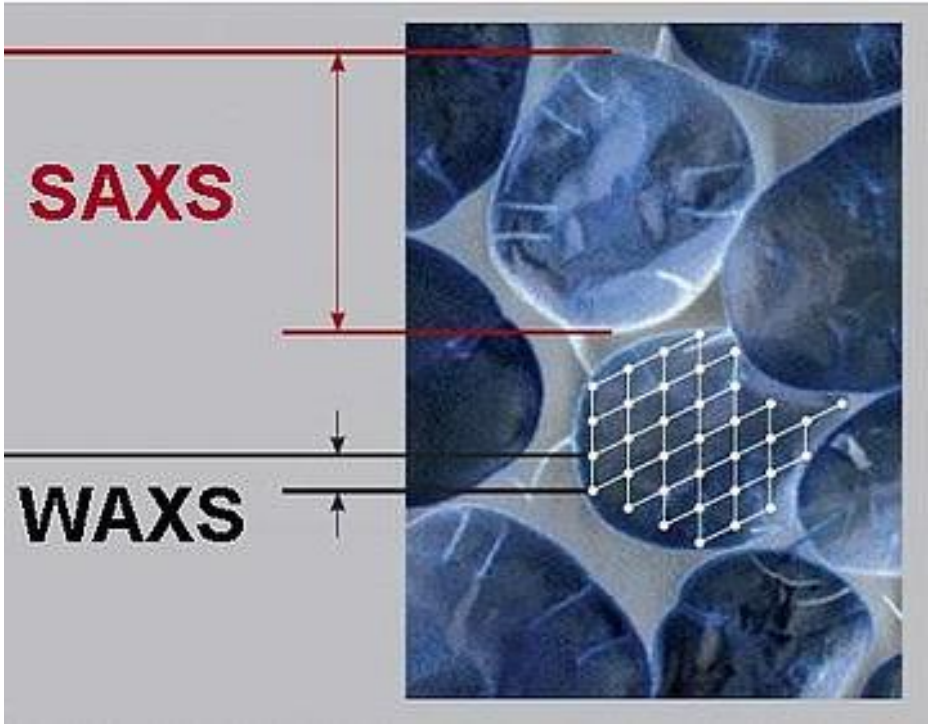
for wavelengths equal, **larger is the size** of the irradiated object, **smaller will be the angles** with respect to the direction of the incident ray to which one observes a scattered intensity different from zero.

What is the technique SAXS?

Small Angle: The angular range of measurement is limited to a few degrees <5˚

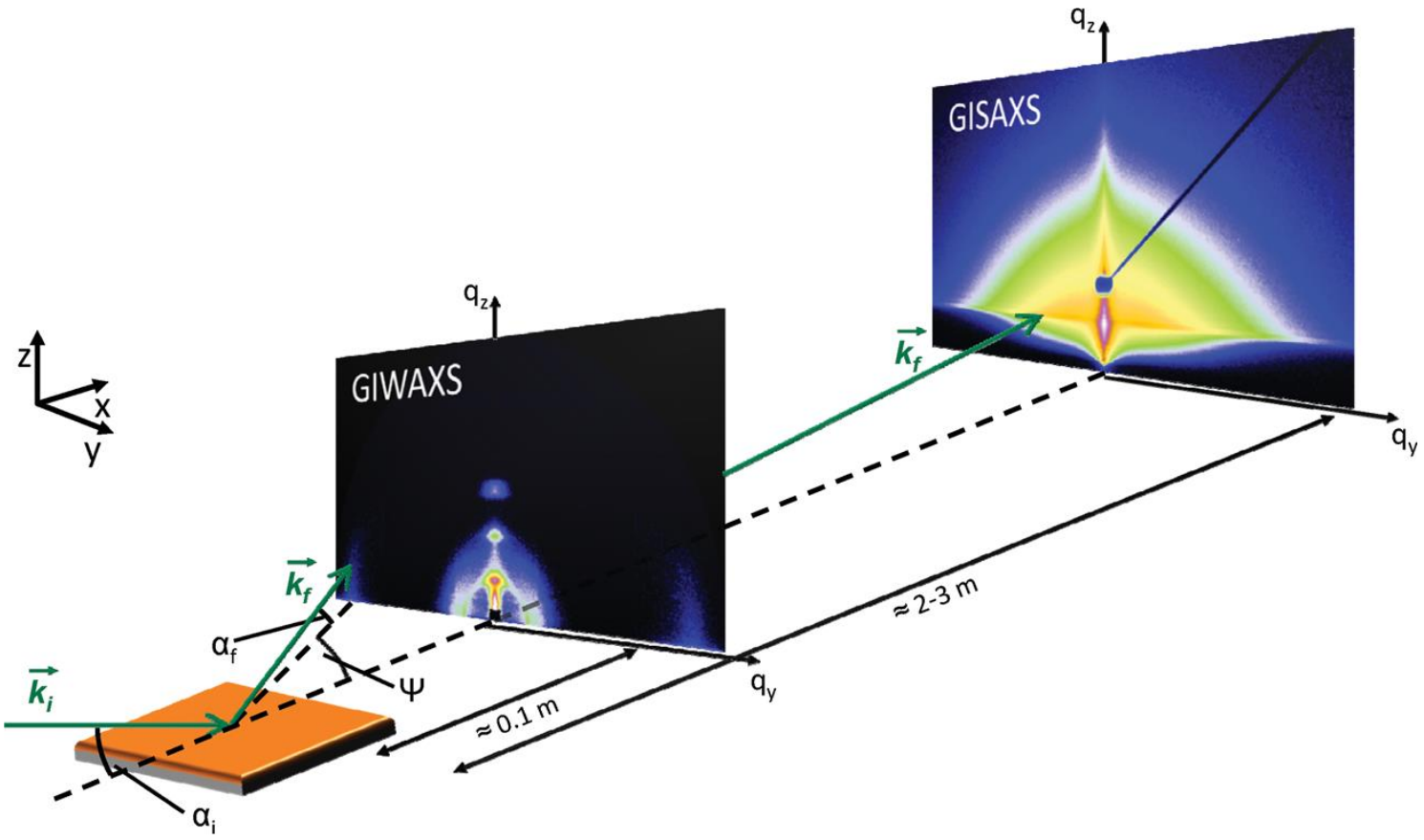
Provides structural information on the inhomogeneity of the electronic density of colloidal dimensions (1-100 nm), while does not solve the atomic details

Wide Angle: angles of measure up to 60˚ : distances features < 1 nm



GISAXS/GIWAXS technique

The study of surfaces at grazing angles



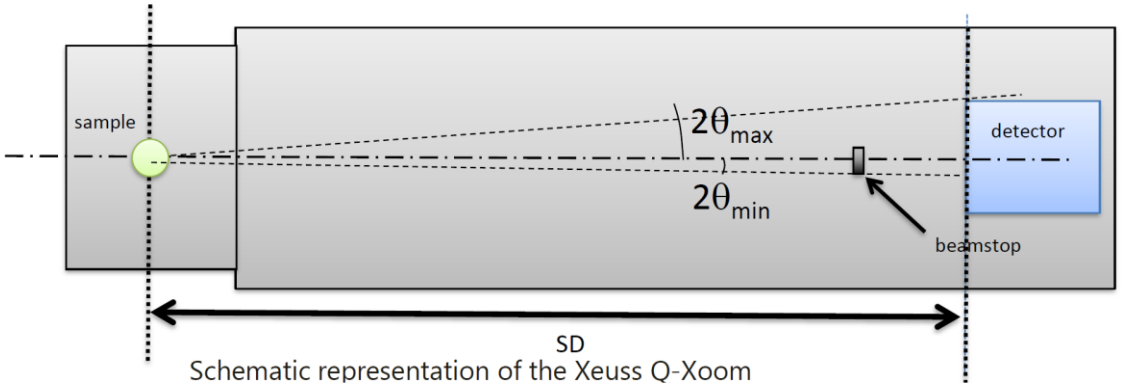
Müller-Buschbaum, Adv. Mater. (2014) 26, 7692

DESCRIPTION OF EQUIPMENT

Xeuss 2.0 – Q-Xoom @ SAXSLab Sapienza

DESCRIPTION OF EQUIPMENT

Detector 2D for SAXS (Dectris - Pilatus 300K) – a variable distance sample-detector

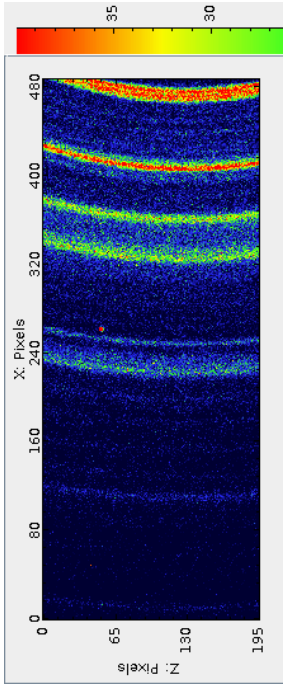


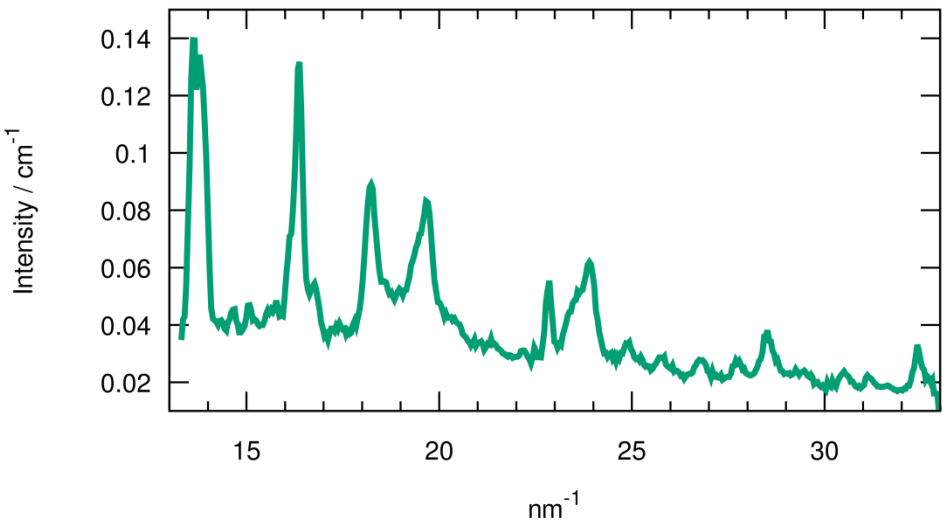
Schematic representation of the Xeuss Q-Xoom

SD [mm]	q_{min}[nm^{-1}]	q_{max} [nm^{-1}]	Max Characteristic D [nm]	Min Characteristic D [nm]
2500	0.06	0.8	125	7.5
1000	0.12	2.1	51	3
500	0.25	4.1	25	1.5
300	0.41	6.8	15	0.9
2500 – High Res	0.033	0.8	190	7.5

DESCRIPTION OF EQUIPMENT

A second detector for WAXS (Dectris - Pilatus 100K) – with fixed distance sample-detector

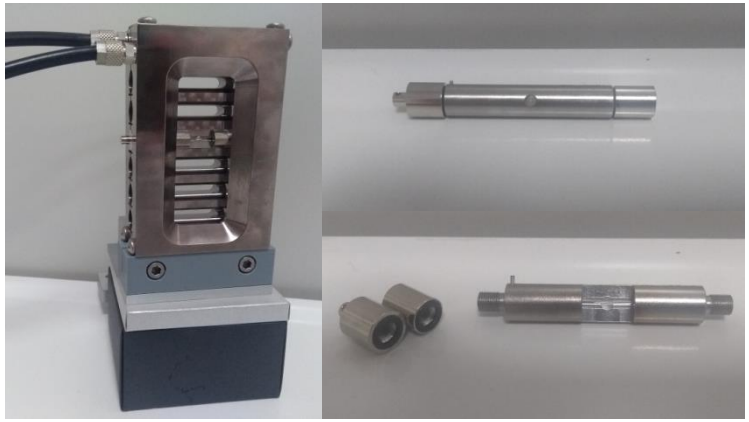




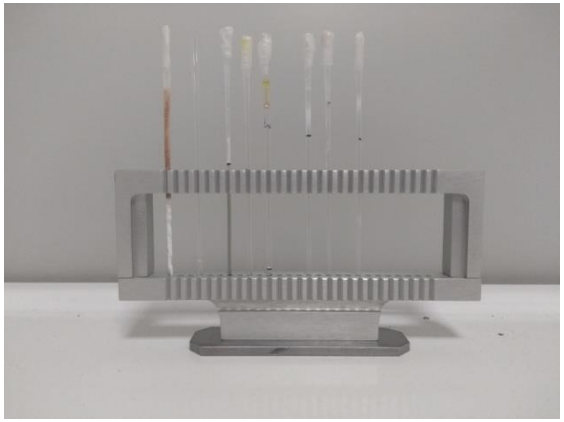
SD [mm]	q_{min}[nm^{-1}]	q_{max} [nm^{-1}]	Max Characteristic D [nm]	Min Characteristic D [nm]
162.2	13.5	32.5	0.47	0.19

DESCRIPTION OF EQUIPMENT

Sampling for measurements in trasmission: liquids, gels, solids



Vacuum-tight refillable capillaries for liquids

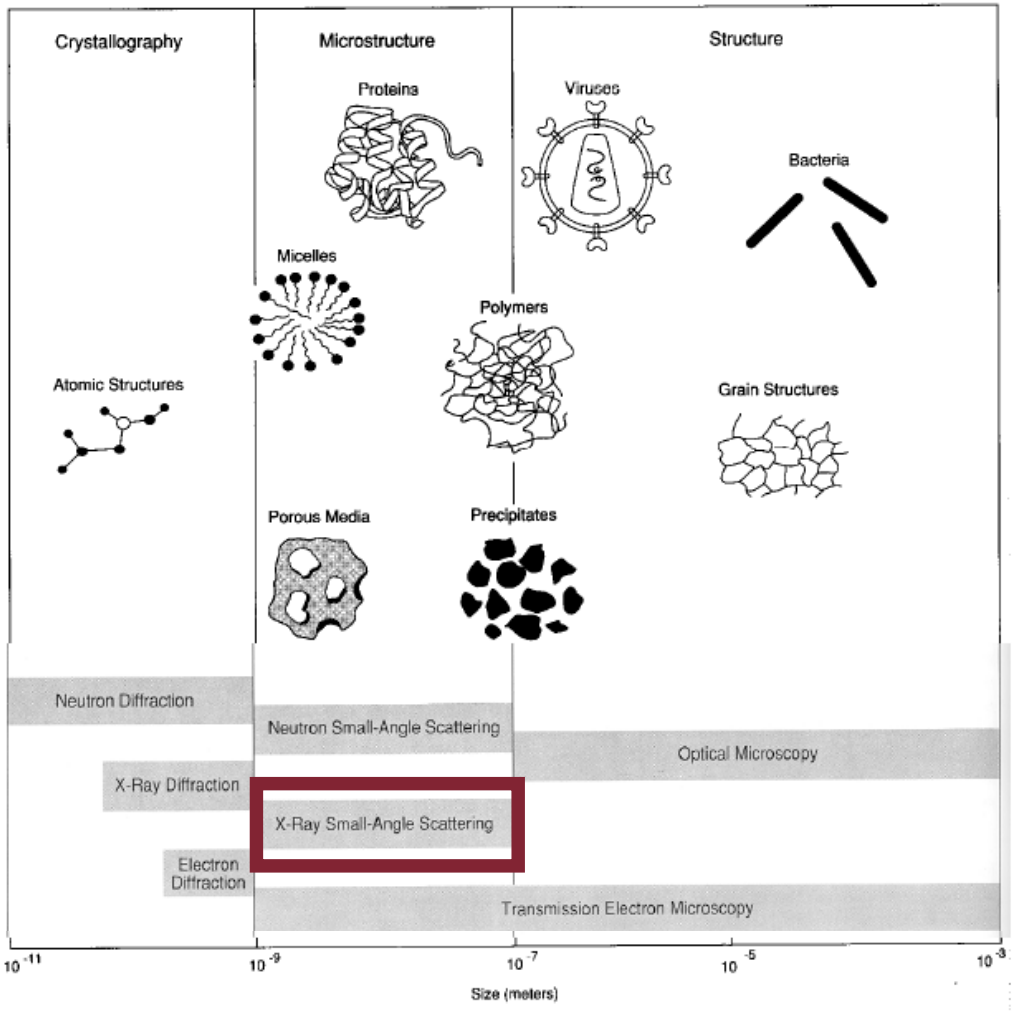


Disposable capillaries holder

Powder holder

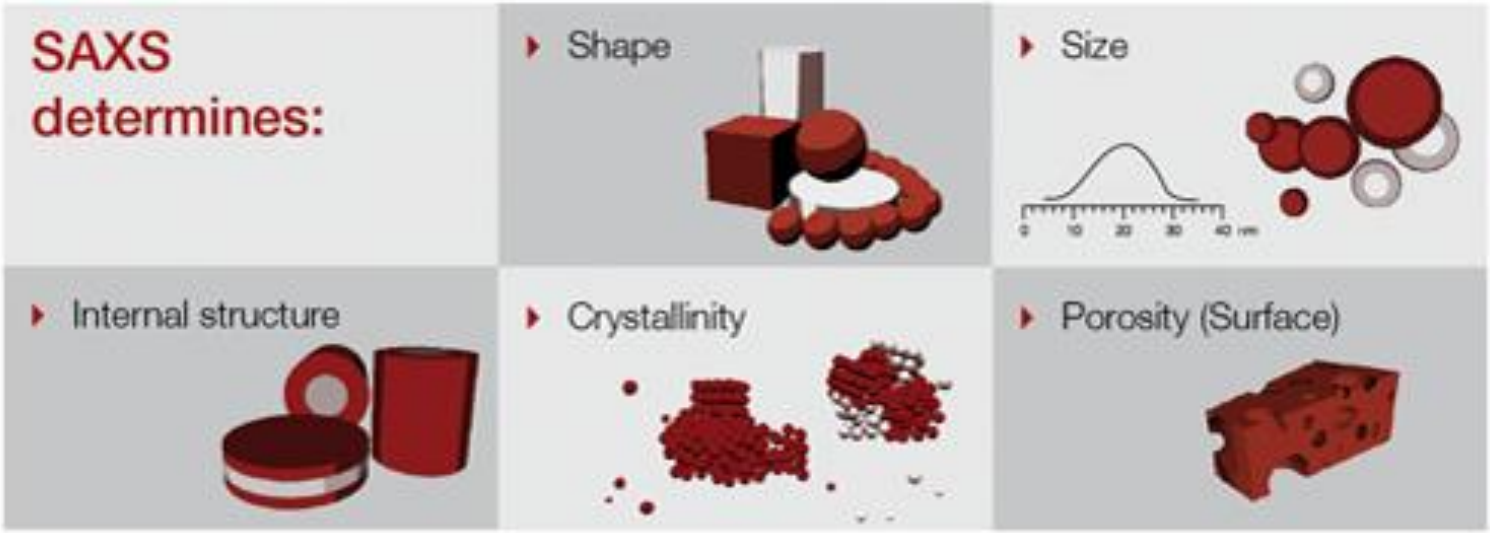
Gel-powder holder

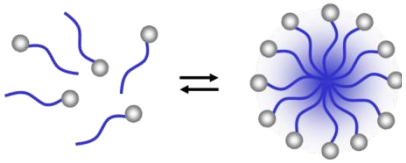
SAXS/WAXS - applications

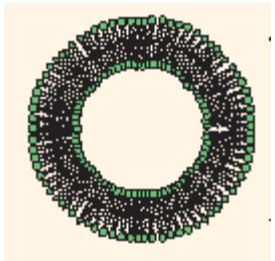


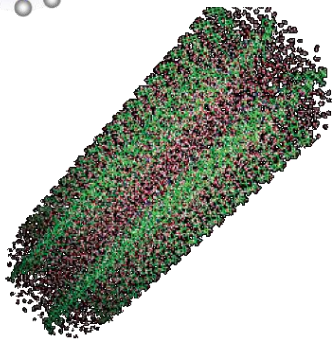
Small Angle X-ray Scattering is a technique aimed at determining the structure of matter in terms of **inhomogeneities (electron density) of the order 1-100 nm**

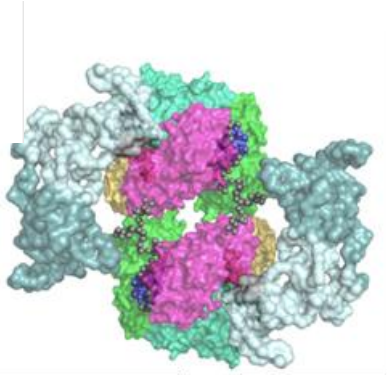
SAXS/WAXS - applications

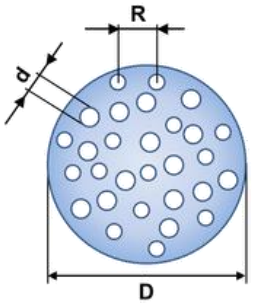








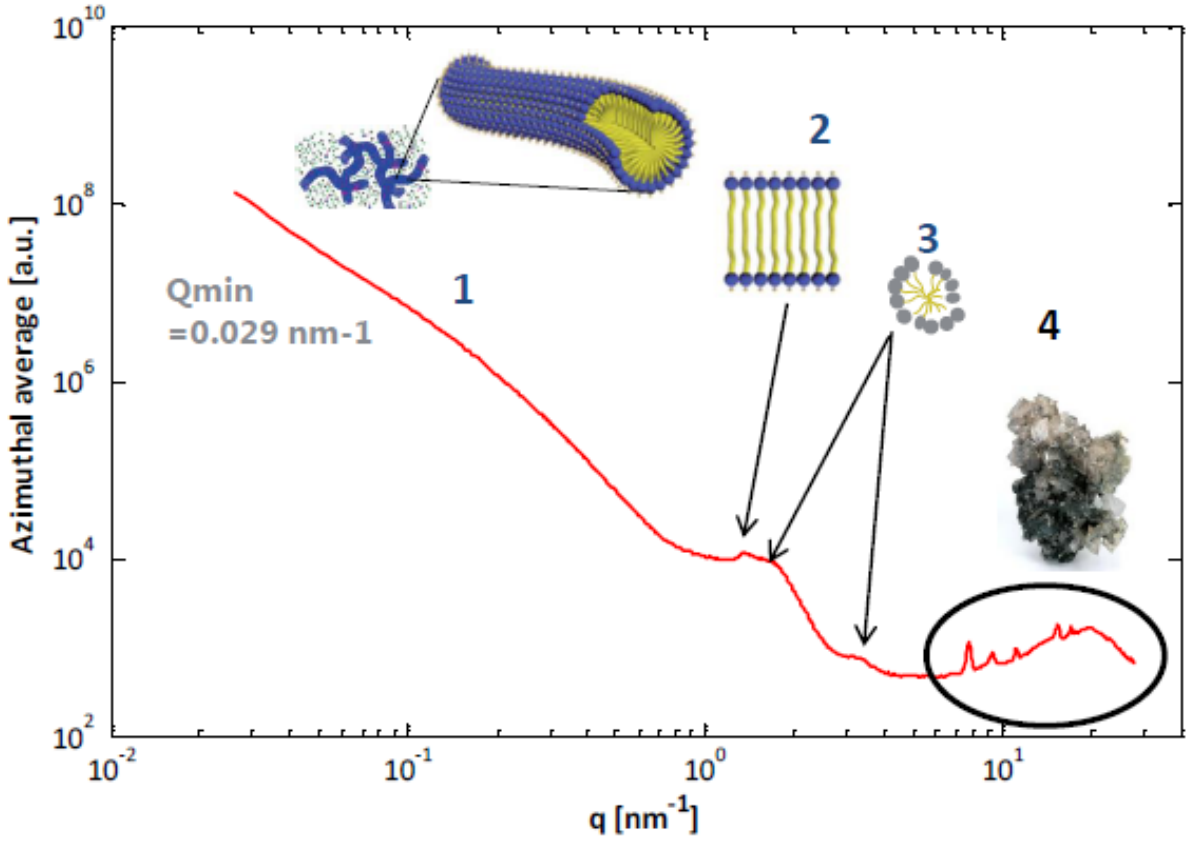




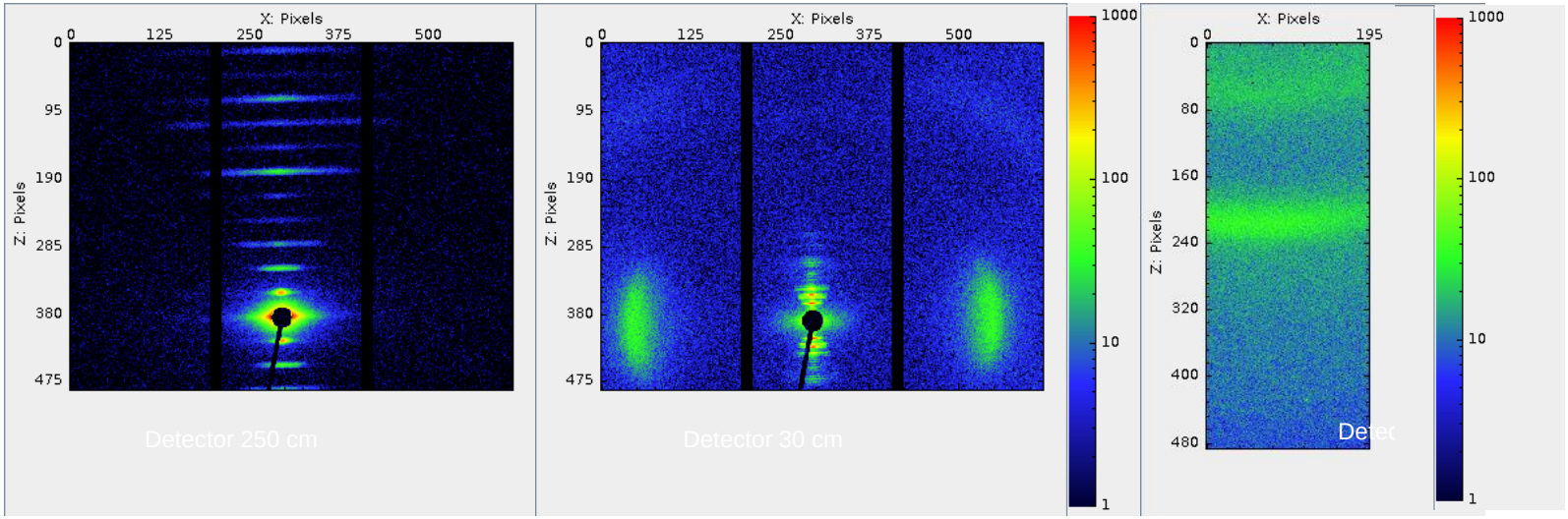
Soft Matter: Self-assembling systems

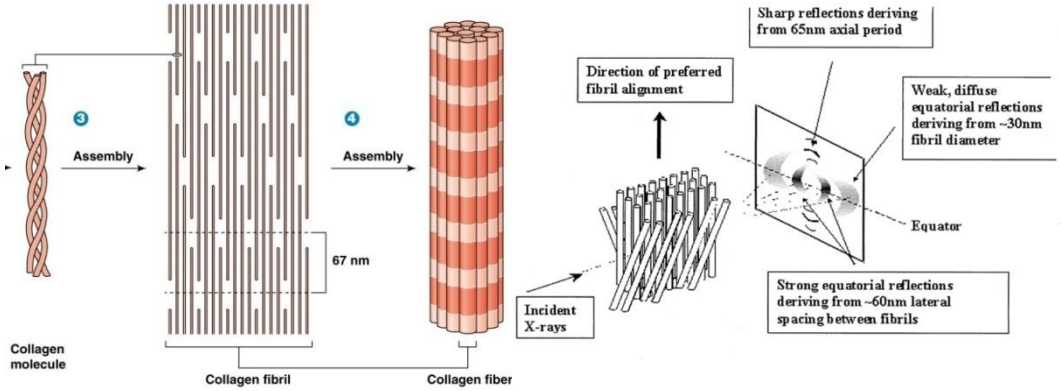
Shampoo: a complex system encountered in everyday life

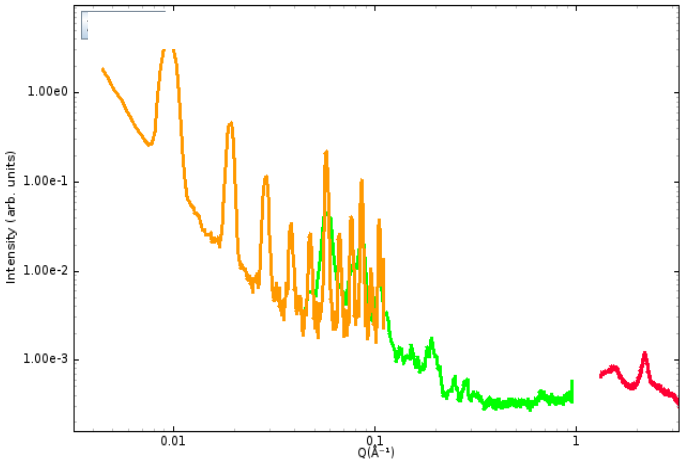
Its spectra SAXS and WAXS show hierarchical structures
They vary with the composition and with the temperature



Hard Matter: Fibres of collagen

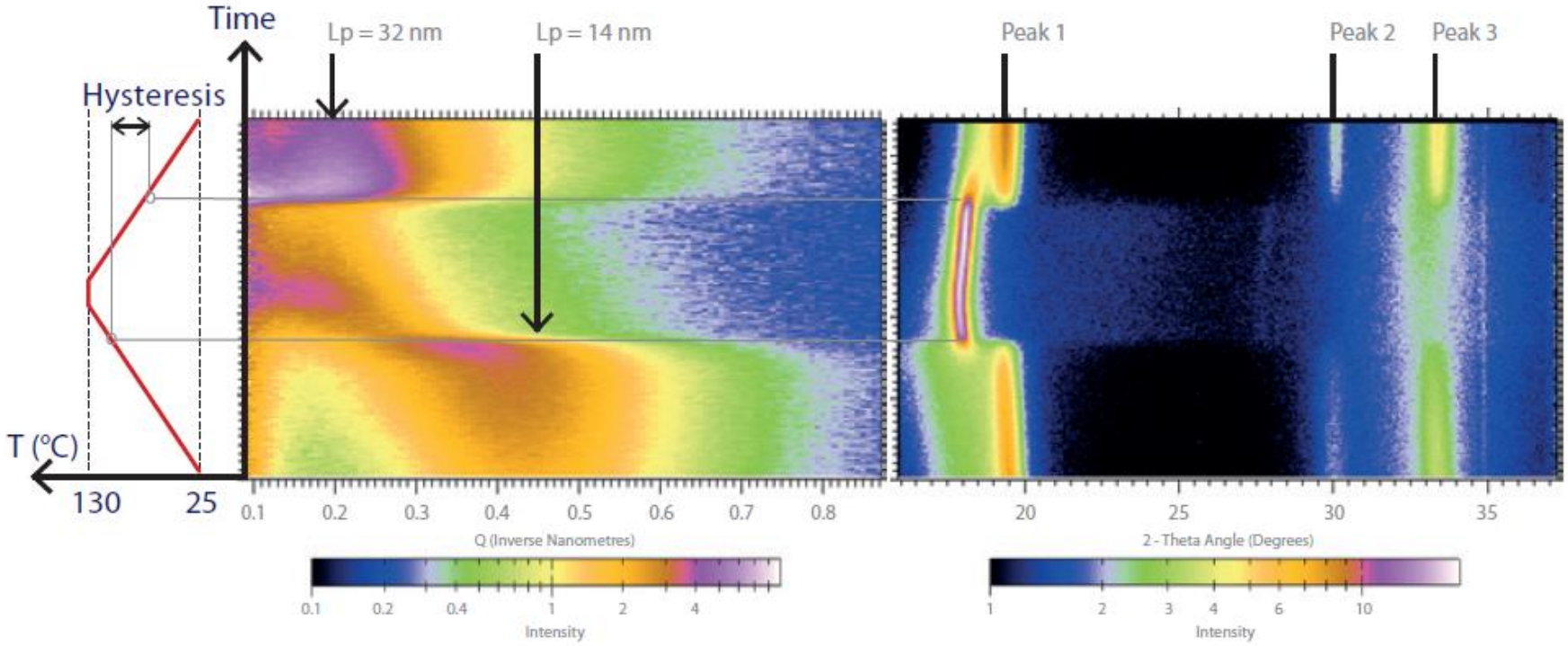






Hard Matter: Phase trasformations in a polymer

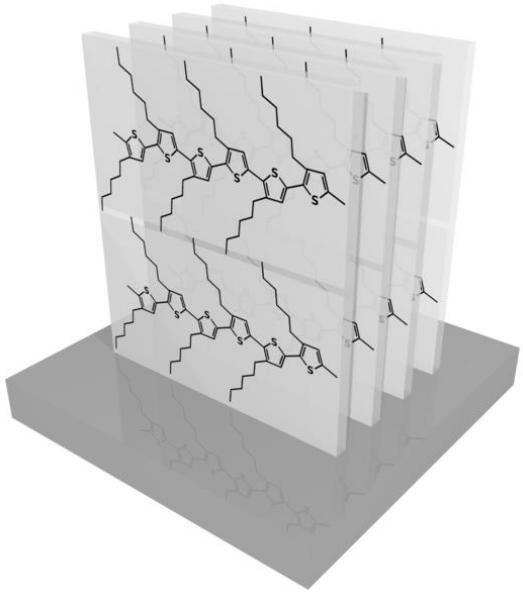
Thermal process for a semi-crystalline polymer
Simultaneous spectra SAXS and WAXS

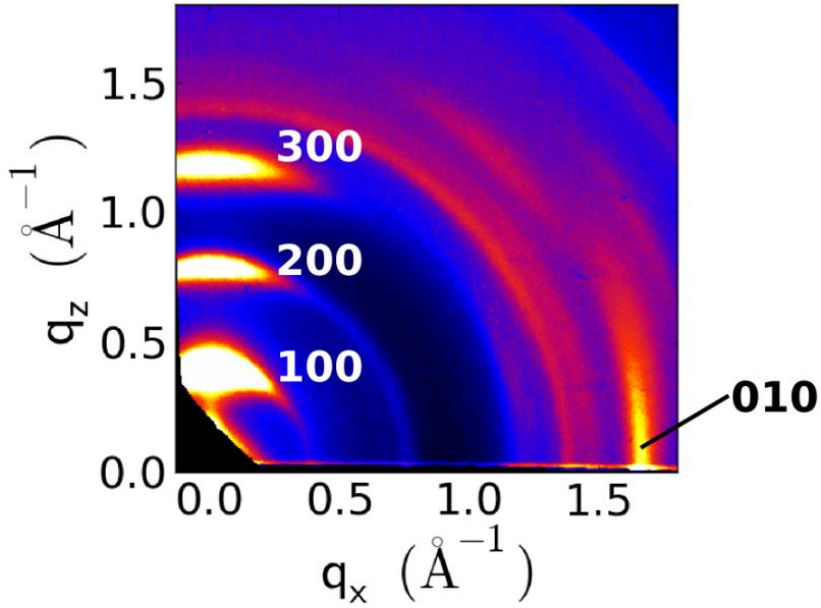


GISAXS e GIWAXS - applications

- Nanoparticles and clusters deposited on solid surfaces - shape, degree of order, morphology, structure
- Surfaces - structure, symmetry, roughness
- Film (single or multilayer) and interphases - structure of surfaces, internal structure, structure at interphase

GISAXS – Film of a semiconducting polymer
Structure and orientation





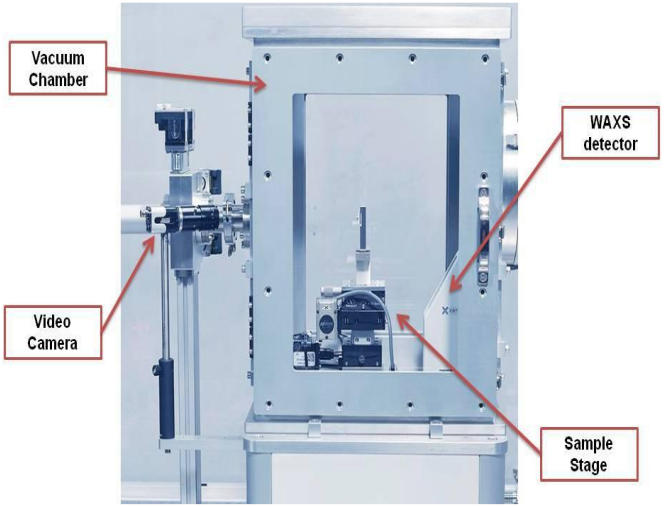
Contact Us

- ***SAXSLab Sapienza –*** A large university equipment
- Dipartimento di Chimica, Ed. Cannizzaro, St. 058
- https://www.chem.uniroma1.it/saxslab/home
- Dott.ssa Alessandra Del Giudice
- alessandra.delgiudice@uniroma1.it
- Nicolae Viorel Pavel
- nicolaeviorel.pavel@uniroma1.it

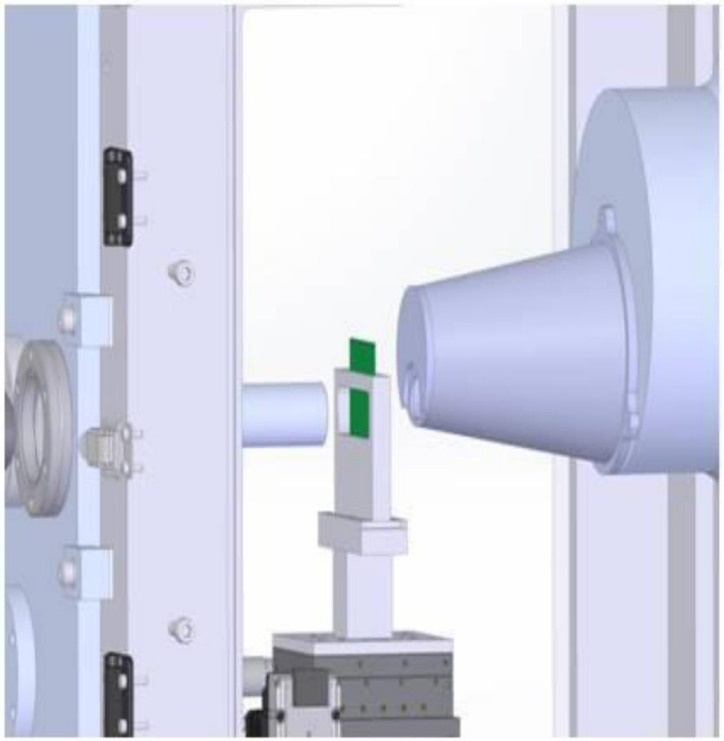
SAXSLab Sapienza

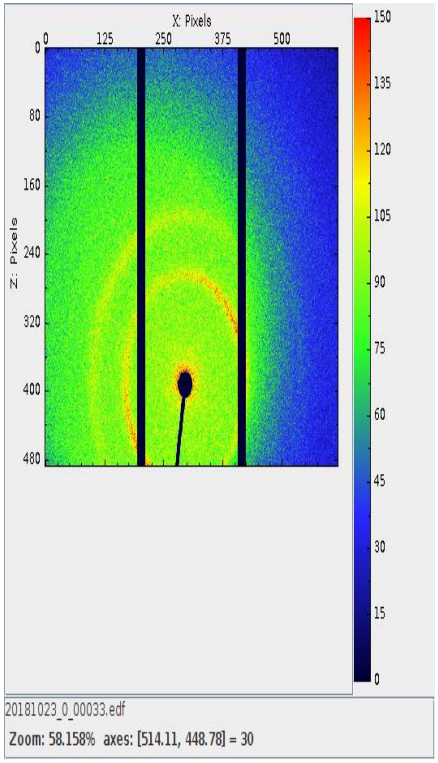
Looking forward for seeing you,
thank you for your attention

Xeuss 2.0 – Qxoom @ SAXSLab Sapienza



IN-AIR setup





1h-image of the empty camera at SD=300 mm,
with IN-AIR configuration

X-ray Source	Xenocs Genix 30W x-ray microsource (Cu $K\alpha$)
Wavelength	1.54 Å
Monochromator/Mirror	Xenocs FOX3D single reflection
Detector «SAXS»	Dectris Pilatus3 R 300K, 83.8 mm x 106.5 mm sensitive area (487 x 619 pixels image)
Detector «WAXS»	Dectris Pilatus3 R 100K
Beam Size at Sample	~ 0.5 mm x 0.5 mm
Flux at Sample position (x-ray power: 50kV x 0.6mA)	1.4×10^{7} photons/s
Source to Sample Distance	1.7 m
Sample to Detector Distance	270-2500 mm, variable
Q range (typical)	0.007 – 0.32 ($Å^{-1}$)
X-ray Optics	2 sets of motorized vacuum slits with “scatterless” blades
Sample Chamber	In vacuum (0.1 mbar <pressure<0.02 mbar) or in air with motorized x-y-z translations, and omega rotation
User Interface	SPEC

Sorgente di raggi X	Xenocs Genix 30W x-ray microsource (Cu Kalpha)
Lunghezza d'onda	1.54 Å
Specchio/Monocromatore	Xenocs FOX3D single reflection
Detector «SAXS»	Dectris Pilatus3 R 300K, 83.8 mm x 106.5 mm area sensibile (immagine 487 x 619 pixels)
Detector «WAXS»	Dectris Pilatus3 R 100K
Dimensione fascio	~ 0.5 mm x 0.5 mm
Flusso (Potenza sorgente: 50kV x 0.6mA)	1.4×10^{7} fotoni/s
Distanza sorgente-campione	1.7 m
Distanza campione-detector (variabile)	270-2500 mm
Range di q (tipico, variabile)	0.007 – 0.32 $Å^{-1}$
Collimazione	2 set di fenditure motorizzate con lame "scatterless"
Camera del campione	In vuoto (0.1 mbar <pressione <0.02 mbar) o in aria. Movimenti motorizzati per le traslazioni x-y-z e la rotazione omega della piattaforma porta-campione
Interfaccia utente	SPEC

MODALITA' DI ACCESSO E DI UTILIZZO

- Gestione tecnica, amministrativa e pagina web: Dipartimento di Chimica
- https://www.chem.uniroma1.it/saxslab/home
- Regolamento (CdD Chimica, 30 Ott. 2018)
- Comitato di gestione
- Assistenza tecnica: Dott.ssa Alessandra Del Giudice
- Tariffario (Sapienza, Enti non profit, Industrie)
- Modulistica: Membership, Application form
- Procedure di uso dello strumento, analisi dati:

incontro preliminare, **application form**, definizione delle procedure sperimentali, prove, misure, analisi dati = chiave di volta del successo

ATTIVITA' SVOLTE E IN CORSO, RISULTATI CONSEGUITI

Bilancio dei primi sei mesi di funzionamento

- **Distribuzione del tempo macchina**

- **Utenza**: Sapienza 12 + 8, CNR 3, altre Università: Cagliari, Firenze, Milano, Lund, ETH-Zurigo, Aalto
- **Dati preliminari**: essenziali per le richieste di tempo macchina su linee di sincrotrone
- **Pubblicazioni**: 1 manoscritto accettato
- **Formazione**:

- Esperimenti per tesi di laurea e di dottorato, corsi della laurea magistrale
- Corso per dottorandi (aa 2019/20)