CONSIGLIO NAZIONALE DELLE RICERCHE PROGRAMMA

SHORT TERM MOBILITY 2016

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Istituto di afferenza: **Istituto di Cristallografia (IC) sede di Bari**

Istituzione ospitante:

ISIS Neutron and Muon Source Science and Technology Facilities

Council. Rutherford Appleton Laboratory

Beamline NIMROD

Harwell Oxford

Didcot, Oxfordshire

Regno Unito

Titolo del programma: **Combinazione della diffusione a basso angolo dei raggi X e neutroni (SAXS/SANS) per lo studio dei nanomateriali coniugati con biomolecole**

The work performed during the Short Term Mobility 2016 at NIMROD beamline (ISIS) was:

1) The study of the system RGD peptide-Conjugated Silica Coated PbS (PbS@SiO2 NP) by neutron scattering, integrated those results with the information obtained from SAXS (Small Angle X-ray Scattering).
   This material with tunable emission in the near infrared region will be used for Molecular Targeted Imaging

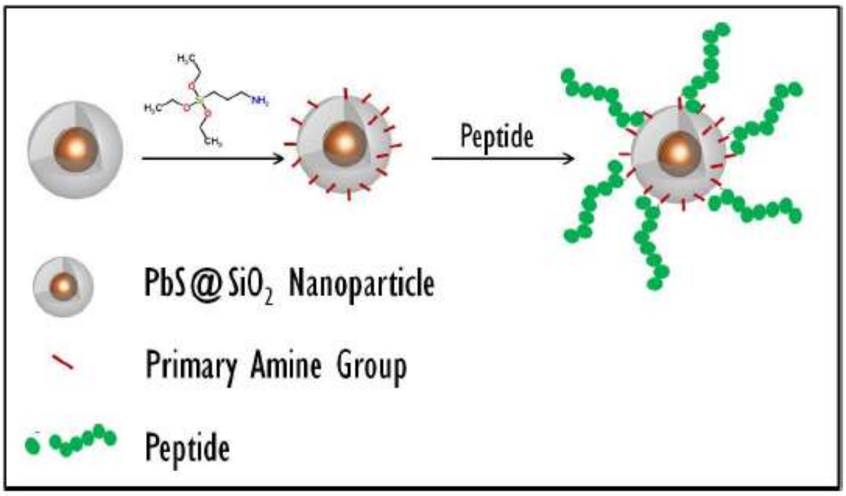


Figure 1. Schematic presentation of PbS@SiO2 NPs conjugated with peptide

The samples were prepared from IPCF –CNR (Bari) in collaboration with IBB-CNR (Napoli). The samples contained:

- PbS@SiO2 NPs (2.5 *10-10 mol) in ethanol (V=0.5 mL)
- PbS@SiO2 NP(5 *10-10 mol)/RGD-Nano-FITC-Peptide (7.4 * 10-9 mol) Conjugates in dimethyl sulfoxide, Borate Buffer (50 mM, pH 8) (V=1 mL). Peptide/NP molar ratio 15

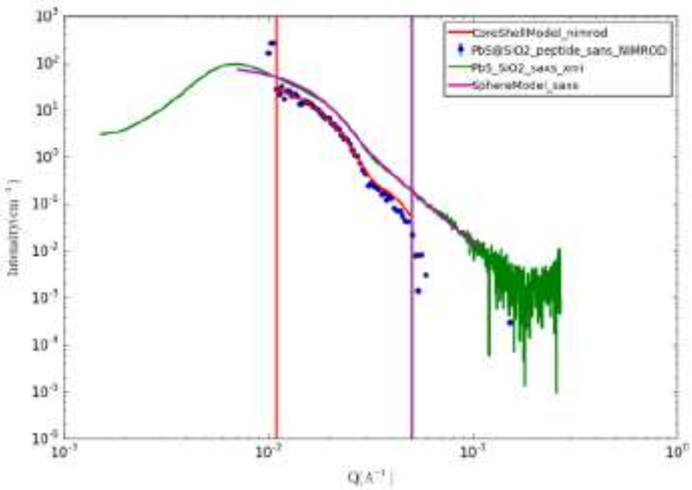
We performed the neutron experiments, using and express proposal (number 1690286), at NIMROD beamline, which is a near and intermediate range order diffractometer designed to provide continuous access to length scales ranging from the interatomic (<1 Å) through to the mesoscopic (>300 Å). Nimrod uses a coupled cold hydrogen and water pre moderator. The detectors consist of ZnS scintillators with 1854 detector eleme.nts (increased to 2337 in October 2012). A temperature controlled (-10°C to 100°C) 15 position automatic sample changer is available, along with a pulse-tube CCR and a high-dynamic range circulating oil bath (operating range -40 to 250°C). The instrument also supports the standard range of cryostats, furnaces and gas handling/pressure systems that are available through the ISIS sample environment support groups [D.T.Bowron, A.K.Soper, K.Jones, S.Ansell, S.Birch, J.Norris, L.Perrott, D.Riedel, N.J.Rhodes, A.Botti, M.-A.Ricci, F.Grazzi and M.Zoppi. Review of Scientific Instruments, 81, 033905 (2010)]. The experiment were done at 13/05/2016 by the help of Dr. Tom Headen.

The x-ray experiments (SAXS) were performed near to our xmi@lab at IC-CNR. Both experiment can give some complementary information. From x-ray scattering it should be possible to estimate the core-shell size of PbS@Sio2 and from neutron scattering the size of the peptide size, which is not possible to be obtain from x-ray scattering due to high scattering from PbS@SiO2.

The experimental result are shown at the Fig. 1 (green and blue curves). Then the data area analyzed by SasView software (http://www.sasview.org/).

For SAXS-xmi data we used a spherical model. The interval for modeling in Q range is: min = 0.011 Å, max = 0.05 Å. The calculated curve for the refined parameters is shown in cyan at the Figure 2.

Figure 2 Experimental and calculated curves for x-ray and neutron data



Refined parameters

Neutron scattering (NIMROD)

Background = -0.024525 [1/cm]
Scale = 1.8757e-09
core_sld = 2.82e-06 [1/A^(2)]
Radius = 120.02 +- 7.5845 [A]
Shell_sld = 0.002686 +- (fixed) [1/A^(2)]
Solvent_sld = 0.0048717 +- (fixed) [1/A^(2)]
Thickness = 15.276 +- 4.4412 [A]
Radius.width = 0.21159 +- 0.059842 [A]
Thickness.width = 0 [A]

X-ray scattering (SAXS-XMI@lab)

Background = 0.0053703 +- 0.00030196 [1/cm]
Scale = 0.00033171 +- 2.7619e-07
Radius = 100.31 +- 0.23581 [A]
sldSolv = 9.47e-06 +- (fixed) [1/A^(2)]
sldSph = 2.4501e-05 +- (fixed)

radius.width = 0.39

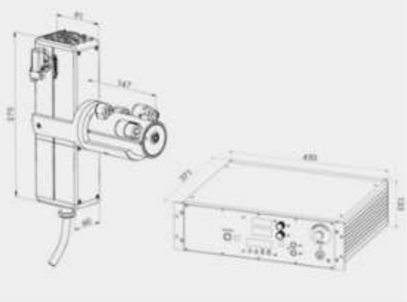
Combining both data, the conclusion is:

The size of coreshell PbS@SiO2 NPs are 20nm but with a dispersity of 0.39 means that the distribution of size is between 12-28 nm. Therefore, the system seems not to be very homogenous. For the system, PbS@SiO2 conjugated with peptides, the core PbS@Sio2 seems to have a distribution between 14-31 nm and the thickness of shell peptide 1.5nm.

Our collaborators from IPCF-CNR/IBB-CNR are valuating these date and combining with the data from TEM , previously performed .

2) The study of feasibility of created a joint x-ray and neutron system at NIMROD. This consist on mount an x-ray tube (with its generator) and a detection system (x-ray detector) inside the neutron camera at NIMROD. In collaboration with the my host Dr. Tristan Youngs and their engineers

Incoatec micro-source & generator



Technical details IuS

IuS	
Length	238 mm
Height	275 mm
Width	60 mm
Weight	6.6 kg

Technical details generator

Generator 19 inch	
High voltage	≤ 50 kV
Current	≤ 650 µA
Power	≤ 30 W
Weight	8.5 kg

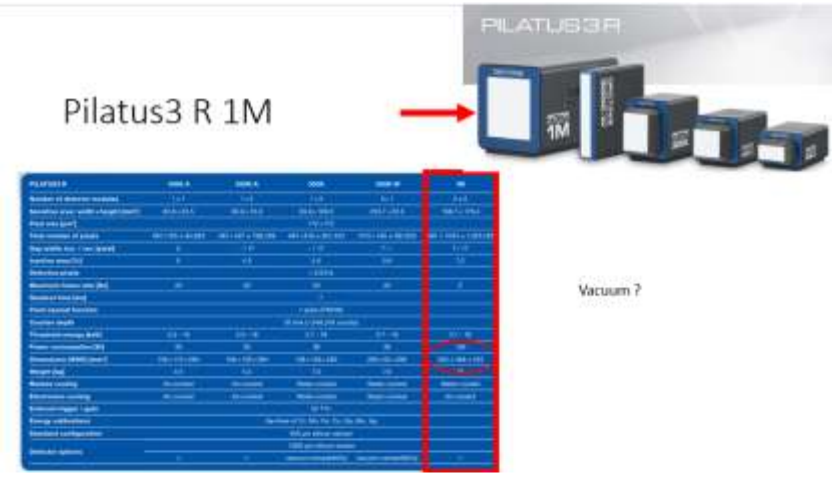


Figure 3. X-ray tube and the x-ray detector with their respectively dimensions

The first step is to find a solution where to place and fix them. The issues are the following:

- Distance x-ray tube/sample holder at least 50cm.
- Distance Sample holder/Detector at least 1-1.5 m for SAXS geometry
- Check if both can stand in the vacuum or not

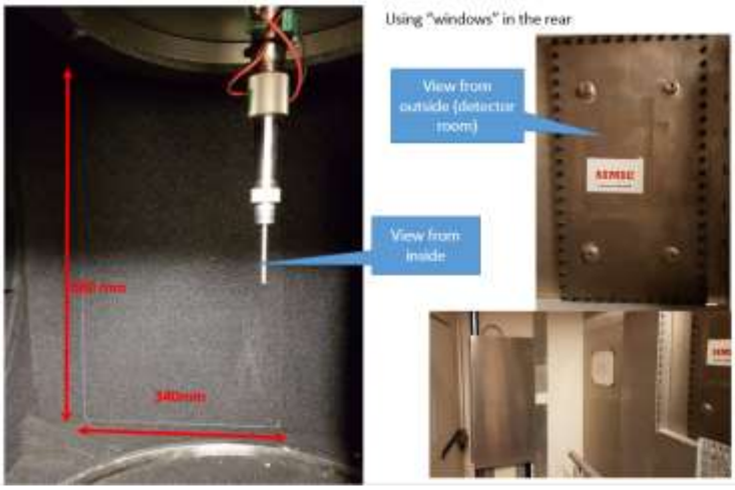
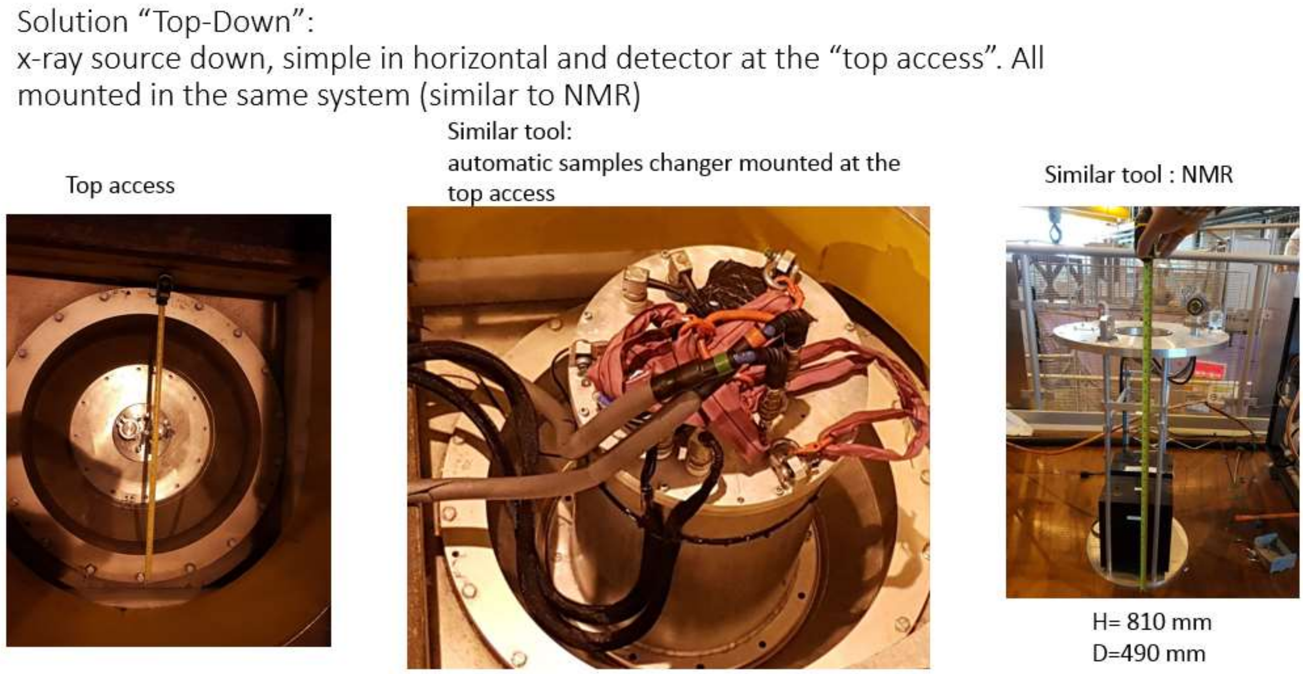


Figure 4. Inside /rear view of “camera” at NIMRO



a) b) c)

Figure 5. The vertical solution.

The best solution we found it is a system similar that in the figure Fig.5c. We have to mount the x-ray tube (generator) at the bottom and detector in the top. But in that case we have to modify the top door at the figure 5a.

Another problem is the vacuum. The measurements should be done in the vacuum but the x-ray tube has a air-cooling system, means we have to create a “air room” inside vacuum camera. Another problem is the cuvette for the sample. That used for the neutron scattering are metallic and cannot be used for x-ray scattering (strong scattering from them). After the discussion may be the best solution is to design and produce double face cuvette, in one direction metallic (for neutron) and in perpendicular in quartz (for x-ray).

Those solution will be presented, in joint with my host Dr. Tristan Youngs and my colleague Dr. Cinzia Giannini, at the PANAREA meeting and next to ISIS Neutron and Muon Source Science and Technology Facilities.

Bari, 13 /06/2016

Il Fruitore

Dritan Siliqi