Baltic Sea Region
Programme 2007–2013

SCIENCE LINK

Project name: **Utilization of X-rays and neutrons for future formulations of white paint**

Beamtime Report

dd.mm.yyyy - dd.mm.yyyy *(Date of the report to be added)*

General information

Name of the rapporteur	**Name of user organization**
Andreas Lassesson	Dyrup - PPG Industries
Type of research (nanotechnology/health care/chemistry etc.)	**Name of the research facility**
Chemicals	MAX-lab
Date of the measurement, duration	**Location of the event**
2013-02-08; 2013-06-19	Lund, Sweden
Facility personnel participating in the measurement	
Tomas Plivelic (BL I911-SAXS)	

Description of the project

Research description (short summary as written in the application)

White paint consists of titaniumdioxide (TiO2) nanoparticles embedded in a polymer matrix. We wish to apply SAXS and XRR in the study of such a particle/polymer system.

Traditional R&D work in the coating industry is based on indirect characterization of properties well known. Complex formulations and empirical rules govern the methods utilized in the laboratory instead of scientifically based strategies. Legislations (based on environmental issues) and increased commodity prices (reduced margins) stresses the need for a more innovative and scientific approach in the development phase.

Tools based on X-rays and neutrons offer such an approach, as it becomes possible to characterize the coating itself, and also to establish the individual material inside the coatings. As a first step to achieve this knowledge, SAXS will be applied to capillaries containing naked TiO2-particles, as well as particles embedded in different polymer matrices (different stages of wet and dry paint). XRR will subsequently be used to characterize the same particle/polymer system on a surface. The extension and detail of the study will be adapted to fit the beamtime awarded. The ultimate study would reveal size and position of particles in the matrix and establish if particle migration in the matrix occurs.

Summary of activities (experiments performed, beamtime used, preliminary overview of results, next steps and other relevant information)

Experiments at MAX-lab were performed at the I911-SAXS beamline.

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1) SAXS analysis of "HO" and "LO" samples in different states: solid or liquid.
Main target of the experiment: differences between both samples

2) Analysis of the SAXS data during the drying process of the paint. Experiment explored during the beamtime.

Experimental conditions:
Data: 08-02-2013
Beamline: I911-4
Detector: Pilatus 1M
Sample-detector distance: 1360.332mm
wavelength:0.91 Å
q range: 0.01- 0.65 1/Å
Software for data analysis: bli911-4
Sample-holder: Multiple positions sample and holder (5 positions)
Total amount of hs used at the beamline: 4 hs
Total amount of hs used for the analysis: 5:30 hs

Experiments:

1.a) Comparison of the data. Dry samples (films)

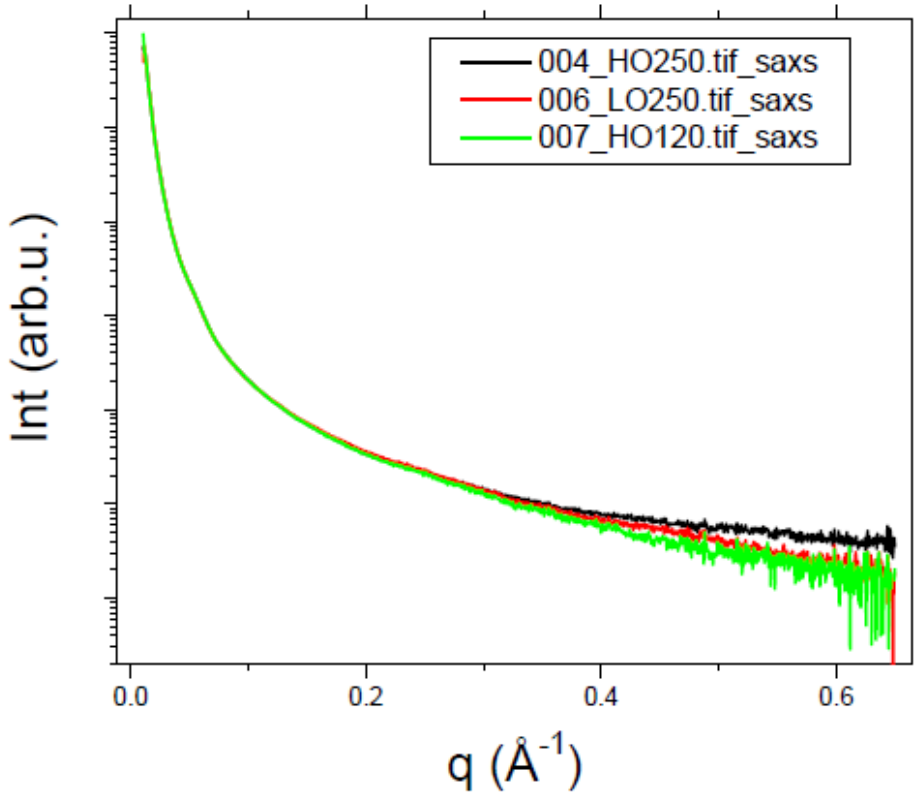

Fig. 1: SAXS signal for HO and LO dry samples.
The SAXS signal for HO and LO 250 samples seems very similar. It only differs at high q-values which represent the signal for the smaller size objects of the system.

The SAXS signal seems to be produced for a highly polydisperse system of scattering objects (in this case and based on discussions at the beamline, the scattering objects can be the empty spaces between particles). This result should be considered as qualitative information.

However the difference between the SAXS signal from different samples do not seem to be consistent. The scattering from the same sample HO with different thickness, 250 and 120, doesn't produce the same scattering signal at high-q values after normalization. Even more the scattering is lower for HO sample than for LO sample.

In the BL scientist's view, Fig. 1 just reflects differences due to small inhomogeneites on the samples.

The conclusion for these results is that the SAXS signal of LO and HO dry samples is equivalent considering the experimental error of the measurement.

1.b) Comparison of the data. Wet samples

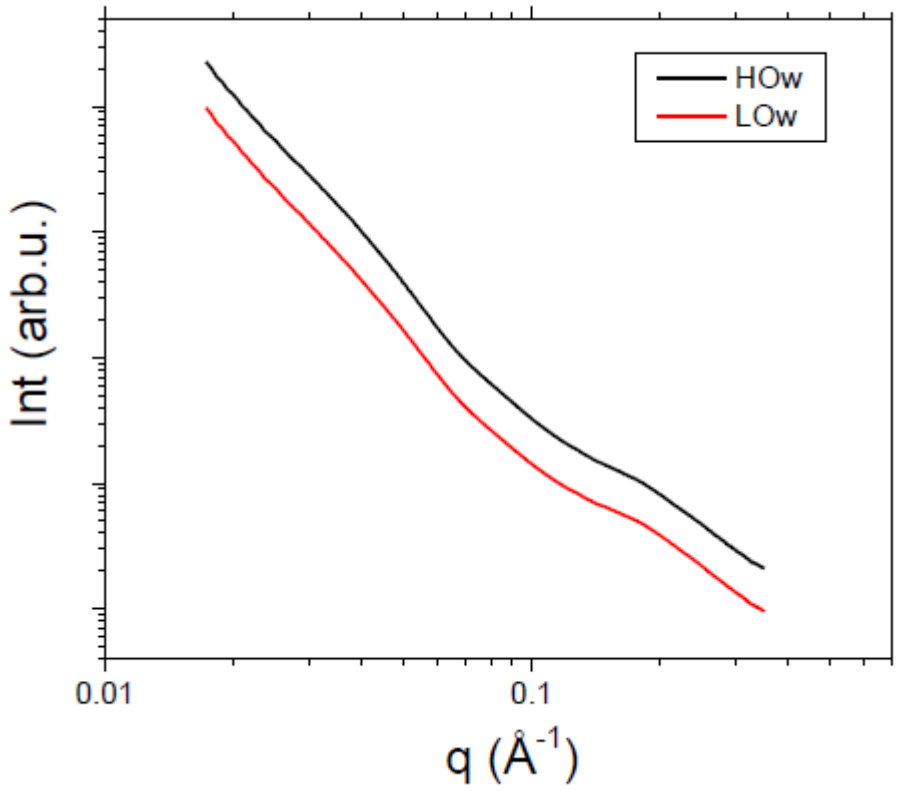

Fig. : Log-log plot for the wet samples HO and LO. Data are shifted vertically for a clear comparison.

The SAXS signal for HO and LO samples are the same. Data are vertically shifted just for comparison. This result is coherent with the previous conclusion: the SAXS signal of both samples, LO and HO, is equivalent, also in the wet state.

However, the SAXS signal from wet vs. dry samples is different. This result is emphasized in Fig 3 for the LO case.

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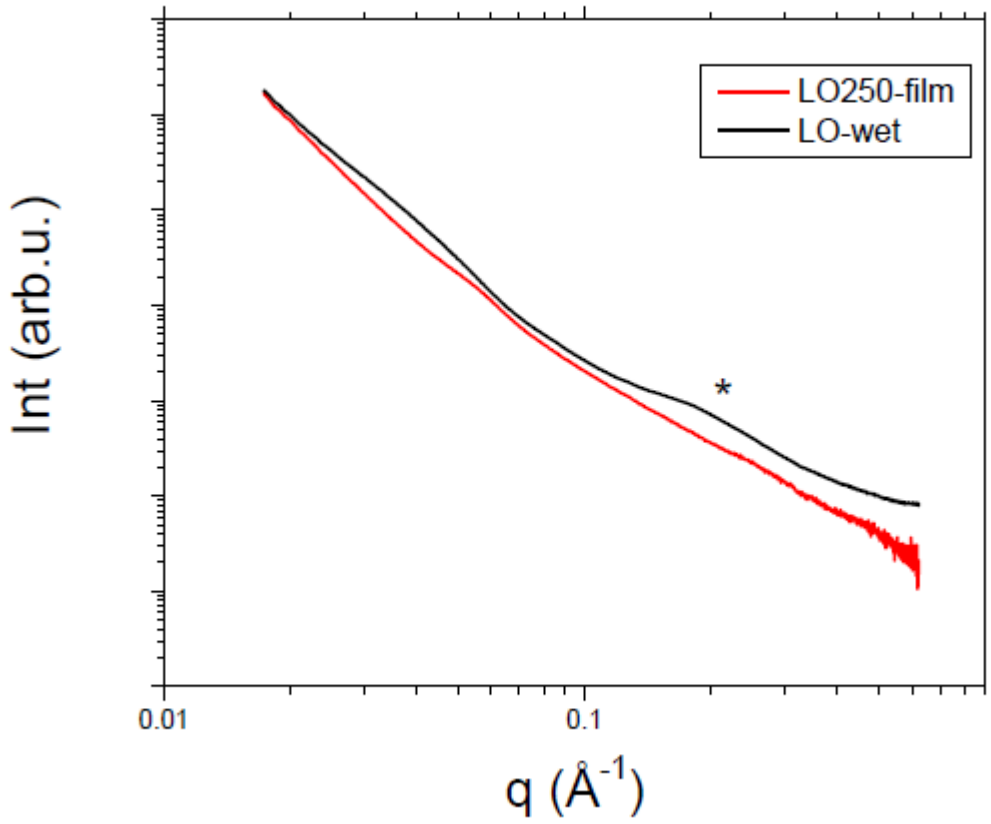

Fig.3: Log-log plot for LO sample in the dry (film) and wet state. * mark the most pronounced "bump" on the wet sample.

In general, broad peaks (or bumps) in SAXS can be correlated with characteristic distances or sizes on the system. The peak mark as (*) as a d-value of 34Å (d= 2π/qm and qm the maximum of the peak).

The first bump, at low q, is poorly defined but it has a characteristic distance of around 160Å. The results of Fig. 3 seem to indicate that there are structural features which can change during the drying process and eventually can be detected by SAXS experiments.

This was the main motivation of the results presented below.

2) SAXS signal during the drying process. Exploratory experiments with HO experiments

The sample used for the experiments was HO wet and it was spread on top of a mica sheet. The total time of the experiments was 30 min, time expected to have completely dry sample.

Fig. 4 shows the data collected during the first 5 minutes of the experiment. Each frame corresponds to 30sec of exposure. During the first 3 min of the drying process, the scattering curves do not change. After that, the two broad peaks with d-values of 180 and 34 Å decrease in intensity. The first peak reaches the minimum values after 5 min of drying. The second peak has a slower evolution reaching the minimum values after

15 min (see Fig. 5-6). The scattering curves doesn´t change anymore between 15-30 min of starting the drying process.

When the sample was removed from the sample holder, it was not completely dry.

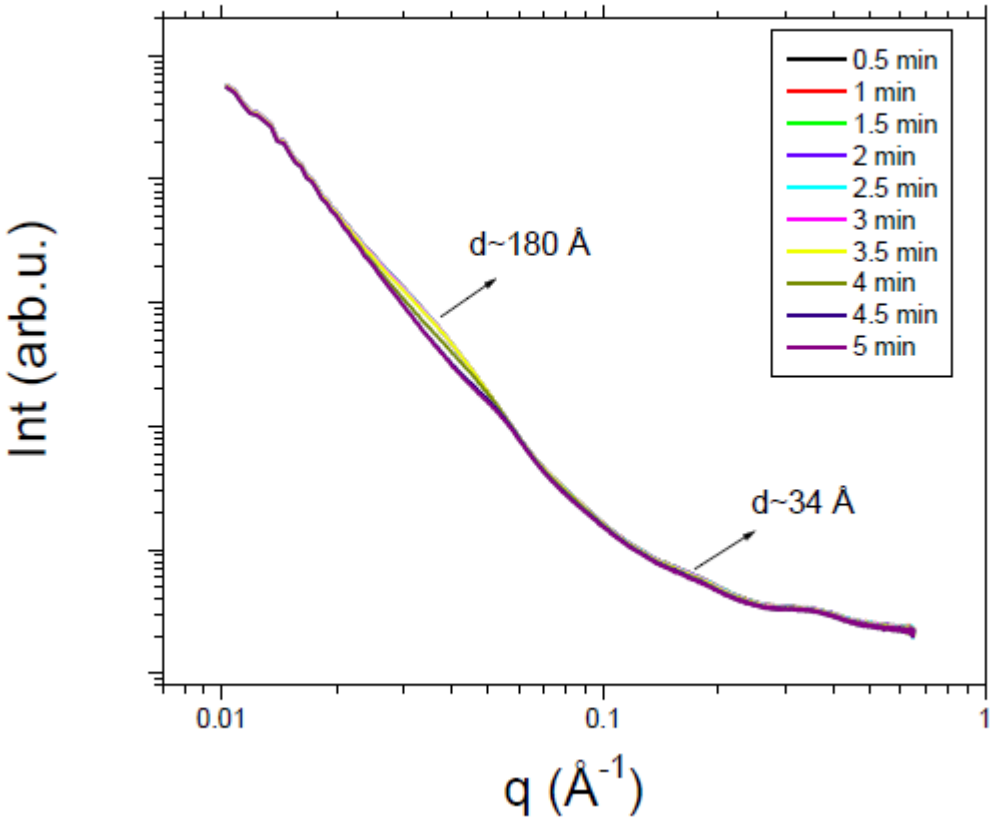

Fig. 4: Drying kinetics during the first 5 min of the process. Two bumps with characteristic distances of 180 and 34Å are observed. Both decrease in intensity after 3 min of incubation time.

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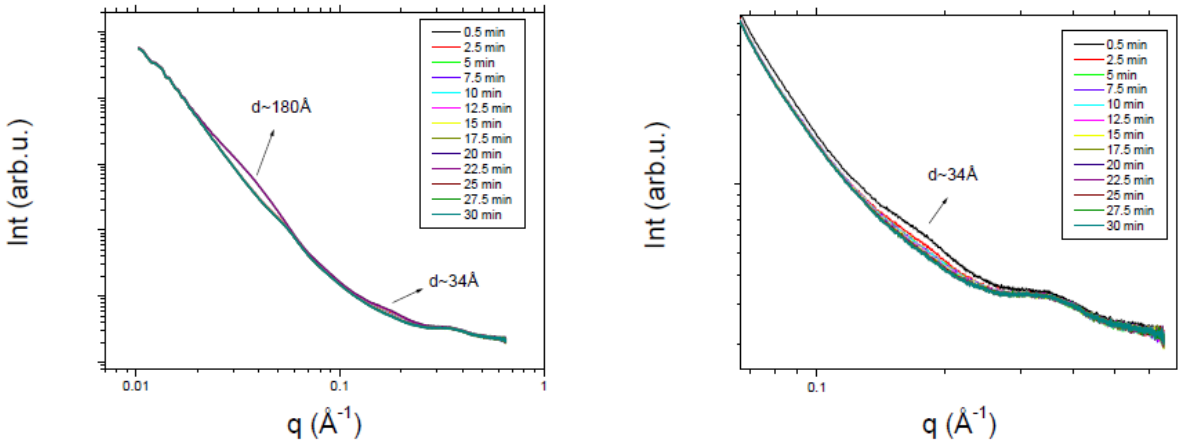

Fig. 5 : Left, Drying process observed for 30 min. The time between frames is 2.5min.
Fig. 6: Right, Amplification of the SAXS curve at high-q values.

Conclusion: changes of the SAXS signals are observed during the drying process. After an incubation time of 3 min, the 2 main peaks decrease in intensity. The first peak, with characteristic distance 180Å, reaches the minimum value after 5 min of drying. The second peak, with characteristic distance 34Å, has his minimum value after 15min.

How would you describe cooperation and assistance from industrial liaison officers and national contact points while preparing and carrying out the research at large scale facilities?

NA

Other personal remarks

NA

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Annexes

Annexes (list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)

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