SMALL ANGLE X-RAY SCATTERING

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INTRODUCTION

Small Angle X-ray Scattering (SAXS) is a well-established technique for investigating the structure of soft matter such as polymers, proteins, and nano-materials. By probing structural inhomogenities, SAXS provides insights into the internal organization of these materials.

There are two primary experimental configuration in SAXS. The first one is Transmission SAXS(T-SAXS), where X-ray pass through the sample and the scattered radiation is analyzed to extract bulk structural information. The second one is Grazing Incidence SAXS (GISAXS), in which X-ray are directed at the sample surface at a very shallow angle. In this report, I describe a training experiment conducted at the 4C beamline of PLS-2, focusing on the principles and practical applications of T-SAXS. Through this experience, we aim to understand how SAXS techniques can be applied to analyze the nanostructure and measured scattering patterns translate into meaningful structural interpretations.

BASIC KNOWLEDGE OF SAXS

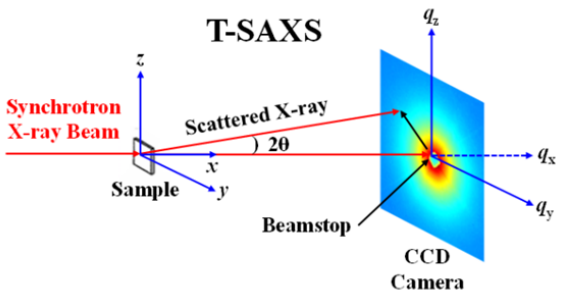

Figure 1: Overview of SAXS

For T-SAXS, the sample is typically in solution or in powdered form. A monochromatic X-ray is collimated and directed toward the sample. As X-rays interact with the sample, some photons are elastically scattered at small angles due to electron density. The scattered X-ray are collected by a 2D detector placed several meters downstream from the sample. The detector records the intensity distribution as a function of a scattered angle, typically represented as a function of the scattering vector q:

$$q = \frac{4\pi}{\lambda} \sin \frac{\theta}{2} \tag{1}$$

One of the most important thing of SAXS is setting the sample-to-detector distance(SDD) This distance critically determines the angular range and resolution of the scattering data. When the sample stage and the detector are positioned close to each other, the system is optimized for observing wide-angle scattering features. In contrast, a longer SDD allows for better resolution at small angles, which is essential for analyzing larger-scale structures.

Adjusting the SDD often requires moving the sample stage. However, this process can be cumbersome, as it typically involves venting the vacuum chamber to access and reposition the stage. To avoid this inconvenience, some experimental setups are designed to move the detector instead of the sample stage, enabling flexible SDD adjustment without breaking the vacuum.

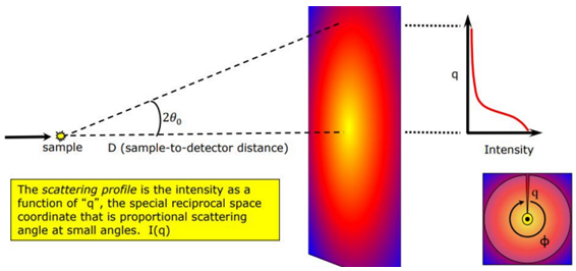

Figure 2: Radial averaged 1D SAXS curve data

2D pattern represents the projection of the 3D scattering intensity onto a flat plane. By averaging the intensity over concentric rings the data are further reduced to a 1D scattering profile(intensity as a function of q). This 1D curve contains rich information about the form factor(size, shape), and structure facotr(interaction of the scattering objects). The intensity I can be expressed in terms of several parameters:

$$I(q) \propto Mc(\rho_1 - \rho_2)^2 |F(q)|^2 S(q) \tag{2}$$

M : molecular weight

c : concentration

ρ : scattering density (electrons per unit volume)

$F(q)$: Form factor

$S(q)$: Structure factor

Many well-established form factor models and structure factor models are available in SAXS analysis software to fit the experimental data accordingly. By fitting the 1D scattering profile with appropriate models, the following structural parameters can be extracted radius of gyration, molecular weight and pair-distance distribution function. Through these analyses, SAXS provides a powerful, non-destructive approach for characterizing nanoscale structural features in biological, polymeric, and nanomaterial systems.

EXPERIMENT AT 4C BEAMLINE

A distinctive feature of the 4C beamline is its ability to flow the sample during X-ray exposure, which helps to mitigate radiation damage. Rather than keeping the sample stationary, the setup enables the sample to flow through the sample stage. This approach ensures that new material is continuously introduced into the beam path, thereby limiting prolonged exposure of any single region and preserving the structural integrity of radiation-sensitive samples.

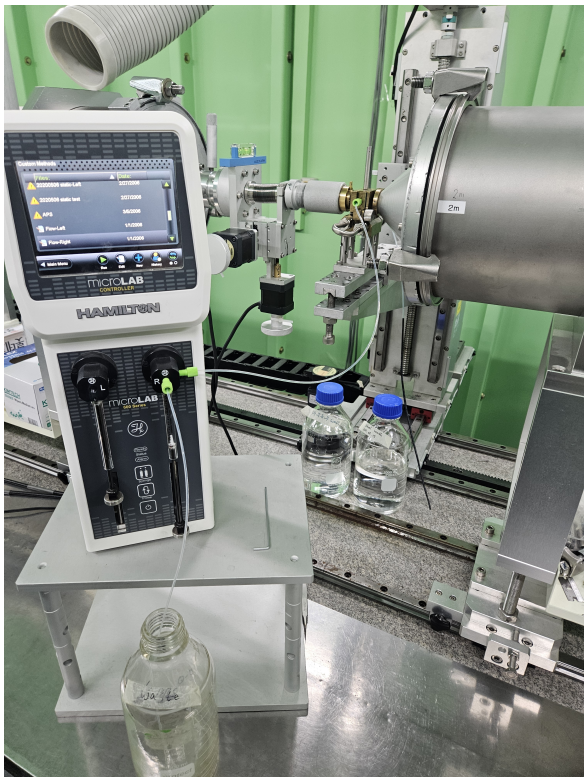

Figure 3: Sample stage of SAXS

Before measuring the sample, it is necessary to calibrate the absorption of air as a reference depending on the X-ray exposure time. For example, air exposure for 60 seconds was set to an intensity of 1 (100% transmission). After placing the glass capillary, the amount of absorption due to the capillary is subtracted. When the solvent was added, the additional absorption is also accounted for by subtracting it from the reference.

The actual measurements were performed under an exposure time of 5 seconds per image, and a total of 8 images were collected for each condition. In the static condition, background scattering from pure water was first measured and later subtracted from the scattering data obtained with the actual solvent. In the flowing condition, the same subtraction process was performed.

To minimize the impact of noise, radiation damage, and other experimental errors, the sets of data showing the most consistent trends were grouped and averaged.

From this analysis, it was evident that the sample experienced measurable X-ray-induced damage, especially under static conditions where fresh material was not continuously supplied.

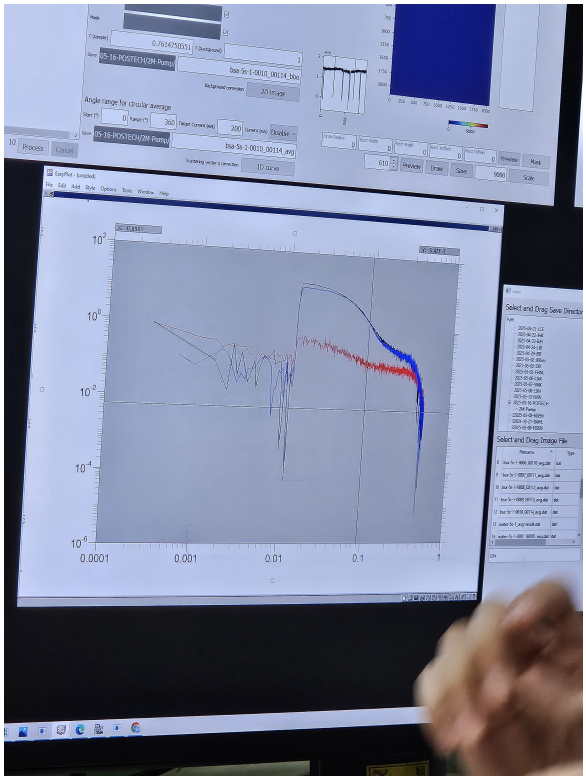

Figure 4: Results of SAXS

CONCLUSION

In this study, we explored the principles and practical implementation of Small Angle X-ray Scattering (SAXS) using the 4C beamline at PLS-II. Through a series of controlled measurements under both static and flow conditions, we considered how experimental parameters—such as sample-to-detector distance, exposure time, and sample movement—critically influence the quality and reliability of scattering data.

One of the key findings was the clear evidence of X-ray-induced damage under static conditions, where the same region of the sample was exposed continuously. In contrast, under flow conditions, where fresh sample continuously passed through the beam path, radiation damage was significantly reduced. This highlights the importance of dynamic sample handling, especially when dealing with radiation-sensitive materials such as biological or polymeric samples.

REFERENCES

[1] Kyung-sik Jin , *Lecture Notes on NUCE719P*, Division of Advanced Nuclear Engineering, POSTECH, PowerPoint presentation, 2025.