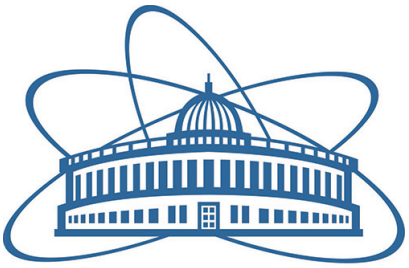

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Introduction to Neutron Scattering Experiments at Large Scale Facilities

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Abstract

X-ray and neutron small-angle scattering experiments are two of the most important techniques to visualize the internal structure of samples which are soft or hard. In this project, examples from SasView are used to clarify how SAXS and SANS are facilitated to study the structural properties of different samples such as Sodium Dodecyl Sulfate (SDS) and silica010. In the end, a proposal is written on studying the AOT sample using the small-angle neutron scattering technique, by depending on the PAXY detector, in addition, the anticipated results and fitting parameters are shown through SasView.

Acknowledgment

It was a pleasure to participate in the INTEREST Programme for the fourth time. This helps me to get more specific and boost my knowledge in different fields of physics. I would like to thank Dr. Raul for his great support and patience throughout the whole period of the project, and also for his lectures, which were too informative and contained important information about small-angle scattering techniques and their experimental work.

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1 Introduction

Radiation in its matter form such as neutrons and electrons and X-Rays in its electromagnetic form is used to study the geometrical patterns of matter and to recognize the crystalline structure. Neutron and X-Ray scattering techniques are facilitated in a range of small angles to determine and visualize the internal structure of a sample [1, 2], which may be soft such as species' tissue, liquid, and biological samples, or hard as a magnetic material, metals, and poly or single-crystalline materials. This technique requires a higher value of wavelength in comparison to electrons [1].

1.1 Scattering and Diffraction: A Comparison

Scattering is a term referring to the change in direction of motion of an incident particle or a wave; due to collision with another single or a system of particles. There are different ways of scattering, which depend on the types of the sample to be investigated and the type of incident beam. In contrast to the latter statement, the diffraction cares about the matter in its waveform [3]. Sir Lawrence Bragg and his father studied the phenomena of crystals' effect on the diffraction of the incident x-ray beam and achieved a formula that describes the diffraction of a beam which has a wavelength that is approximately equal to the spacing between atoms of investigated solid [4, 5].

1.2 Small-Angle Scattering (SAS)

Small-angle scattering is a technique that utilizes particles or electromagnetic waves in a range of 1 $\mathring{A}$ limited to 2 degrees; to study the structure of matter. As presented in Figure 1, the beam starting from the very left end is emitted by a source of x-ray or neutrons, then the beam is focused and accumulated through the point collimation, which is followed by the sample of interest. Afterward, the beam extracted interacts with the sample (i.e. the sample analysis starts at this stage). Finally, the scattered and transmitted beams interact with the detector which provides a pictorial representation of the sample's internal structure or the contents beyond the materials' patterns. For the detector -on the left end, as much as the $\vec{q}$ is increasing, the intensity of the scattered beam will be decreased, so -they are inversely proportional to each other.

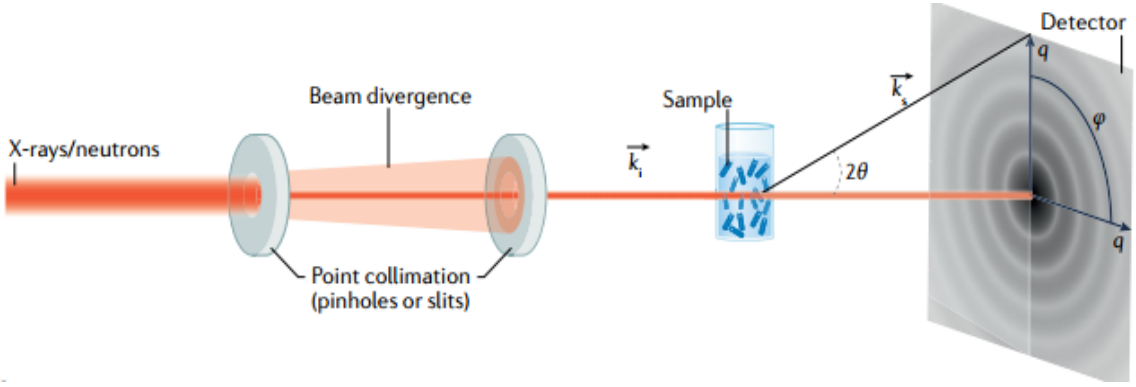


Figure 1: Small Angle Scattering Technique with an x-ray and a Neutron Beam

The intensity of the scattered beam is given as a function of the wave vector (q), it is known that the intensity of the scattered beam will decrease; due to the absorption of a part of the incident beam's energy in the sample itself, hence we can determine the structure depending on the intensity of the beam.

The wave vector is [6] then given mathematically by vector addition rule:

$$\vec{q} = \vec{K_s} - \vec{K_i} \tag{1}$$

Where $\vec{K_s}$ is representing the scattered beam, whereas the $\vec{K_i}$ is for the incident beam. The value of K is the wavenumber and it is given by:

$$K = \frac{2\pi}{\lambda} \tag{2}$$

While λ is the wavelength. The angles θ and ϕ give the scattered beam angles to the horizontal x and y axes.

The magnitude of the wave vector is then calculated as:

$$q = \sqrt{{K_s}^2 + {K_i}^2 - 2K_sK_icos(\theta)} \tag{3}$$

In the elastic scattering mode (i.e. Energy before and after collision is the same), we have [6] $K_s = K_i = K$, hence eq.(3) will be developed to the following form:

$$q = \sqrt{2K^2(1 - cos(\theta))} \tag{4}$$

As $cos(\theta) = 1 - 2sin^2(\frac{\theta}{2})$, so previous equation will become:

$$q = 2Ksin(\frac{\theta}{2}) \tag{5}$$

Finally, from eq.(2), the magnitude of the wave vector will be given as a function of wavelength λ and angle of the scattered beam θ:

$$q = \frac{4\pi}{\lambda}sin(\frac{\theta}{2}) \tag{6}$$

1.3 X-Rays, Neutrons: Their Sources and Scattering Behavior

[1]As a form of beams, x-rays and neutrons are two completely different ones. As, the x-ray is electromagnetic radiation, in contrast, neutrons are particles with a considerable mass. For the energy, neutrons are less energetic with a lower range of wavelength in comparison to x-rays that have more.

1.3.1 X-rays Prodcution

There are many ways to get an x-ray beam, mainly depending on the deexcitation of electrons, that emit this difference in energy as electromagnetic radiation (i.e. x-rays).

1.3.2 Neutrons Prodcution

Neutrons are generated through the following processes:

- Nuclear fission: when a heavy nucleus such as uranium is bombarded with a neutron, this leads to a large number of neutrons and protons to be produced.
- Nuclear fusion: occurs due to the fusion of two nuclei to produce a new nucleus, for example, the fusion of hydrogen nuclei to form deuterium.

[1]Most of the content under this subsection depends on the lectures presented in **SwedNESS Ph.D. school: Small-Angle Neutron Scattering (SANS)**

- Spallation: this phenomenon happens when a proton with high energy hit a heavy metal target (i.e. contains a considerable number of neutrons). This leads to the production of 20-30 neutrons per hit of one proton.
- Bombarding beryllium (^{9}Be) with alpha particles, leads to a nuclear reaction as follows:

$$^4_2He + ^9_2Be \longrightarrow ^{12}_6C + 10 \text{ neutrons}$$

For getting a beam with a high flux of neutrons, spallation and nuclear fission are used.

1.4 Small-Angle Scattering Technique

[2]SAXS (Small-Angle X-ray Scattering), by which x-rays as an electromagnetic wave interacts with the electron cloud and SANS (Small-Angle Neutron Scattering) that boosts scattering of neutrons by the atomic nuclei are two techniques, which are utilized to study the material's internal structure and the arrangement of atoms [6, 7]. It should be noted that this microscopic arrangement is dynamic; due to the motion of the particles such as rotation, in addition, the spacing between particles is not fixed with time [5]. The kind of graphs for data representation in SasView is a 1D graph for the x-ray case, while it is a 2D graph for neutron analysis.

1.4.1 Small Angle Neutron Scattering: SANS

SANS technique provides us with the investigation and measuring of three-dimensional samples with sizes from 1 nm to 100 nm, besides that, it also allows the characterization of the structure of the sample and its parameters such as volume, mass, and shape [8].

1.5 SAXS and SANS: A Comparison

Comparison	SAXS	SANS
Absorption	The absorption of x-rays must be considered in possible analysis.	The absorption of neutrons is not to be considered in the analysis.
Dependency	The form factor of x-rays for an atom depends on the number of electrons.	The neutron scattering length is not constant and varies from atom to atom or even from one isotope to another.
Damage	Damage to the beam must be considered.	There is no concern about the sample's damage.
Flux	The flux of x-rays allows fast measurements.	The flux of neutrons controls the rate of data collection.

Table 1: Comparison between SAXS and SANS

[2]Most of the content under this subsection depends on the lectures presented in **SwedNESS Ph.D. school: Small-Angle Neutron Scattering (SANS)**

2 Experimental Work By SasView

2.1 SasView Software

Sasview is software designed by scientists from the University of Tennessee in the USA. It is used for studying neutron scattering experiments. Figure 2 shows how the SasView window seems, as it is opened before loading and analyzing the experimental data.

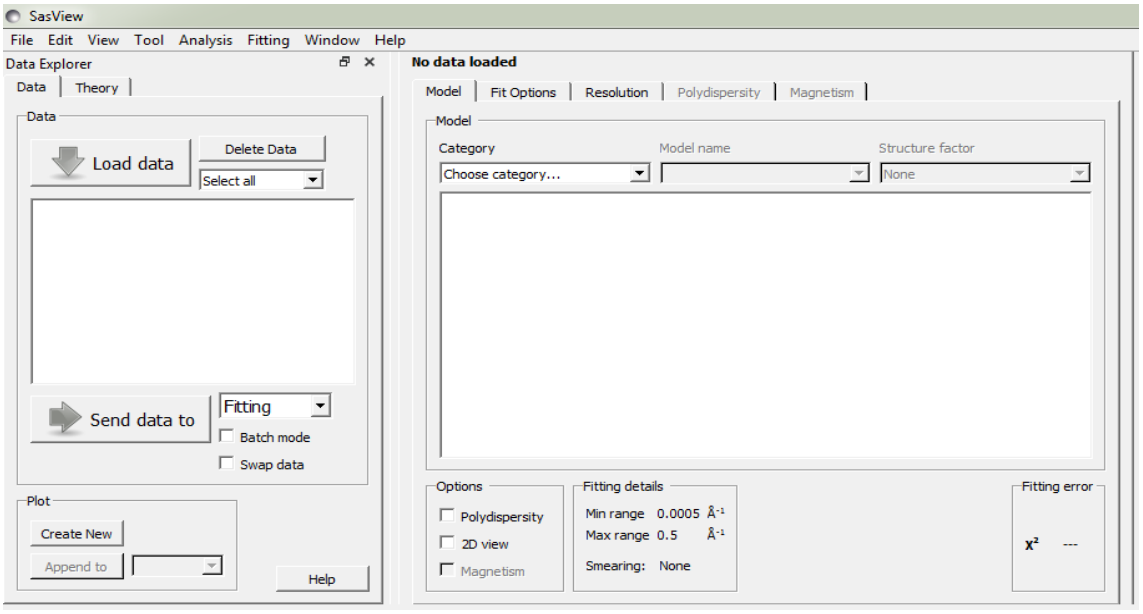


Figure 2: SasView Software Front Screen

2.2 Experimental Data and Analysis

SasView is provided with some examples for analysis made before and provides parameters that should be changed to fit the data and reduce the error factor. Below there is an example of the representation of data and its fitting seems before and after changing parameters such as: and its effect on the error factor.

2.2.1 Procedures

Here are the steps of how data are called and treated:

1. Loading and sending the data file. This happens by pressing the "Load data" button and then sending the data to be fitted through "Send data to" as presented in Figure 2.
2. Selecting the category such as cylinder, sphere, parallelepiped, etc. Each category contains several models, which have been chosen depending on the type of sample and its structure.
3. Fitting the data to get the best agreement between theoretical expectations and experimental findings.

2.2.2 Fitting the Data: Fitting Parameter χ^2

It is a statistical factor which provides us with a comparison between the measured and theoretical data. It is obtained through the following relation:

$$\chi^2 = \sum [\frac{(I_{Exp.} - I_{Theor.})^2}{(I_{err.})^2}] \tag{7}$$

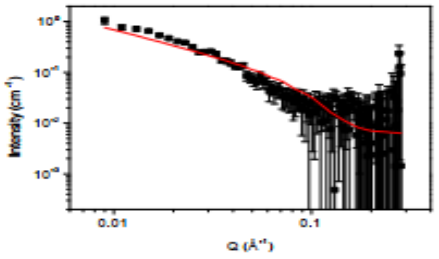
$I_{Exp.}$ is the intensity of the scattered beam from the experiment, $I_{Theor.}$ is the intensity calculated theoretically for the scattered beam, and $I_{err.}$ is the error in measuring.

2.3 Examples

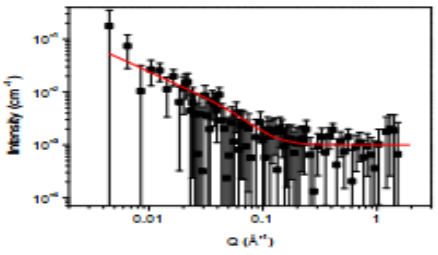
In SasView, from the test folder, we can find various examples that depend on previous studies for small-angle x-ray and neutron scattering techniques. One can choose one of the examples, which are presented in form of data files. After loading and fitting the files, we can fit and show plots after fitting. The plots can give representations such as the intensity of the scattered beam and the residuals as a function Q.

2.3.1 Example-1: [SANS] Sodium Dodecyl Sulfate (SDS)

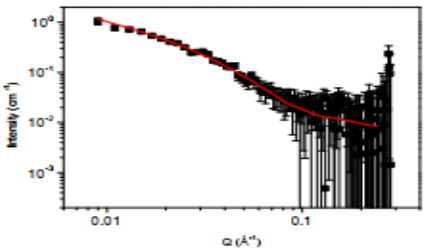
Through the core-shell cylinder model, SDS absorption in carbon nanotubes has been studied by SANS, the samples prepared were investigated in 2 mm of D_2O with the percentage of 1%. The error factors were 243 and 24. Figure 3a and Figure 3b represent data produced from x-ray scattering technique, while in Figure 3c and Figure 3d represent the results from small-angle neutron scattering technique [9].



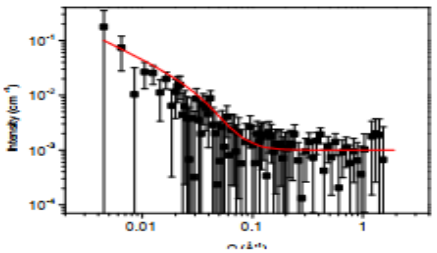
(a) h-SDS in D_2O with SWCNTs



(b) cm-SDS in D_2O with SWCNTs



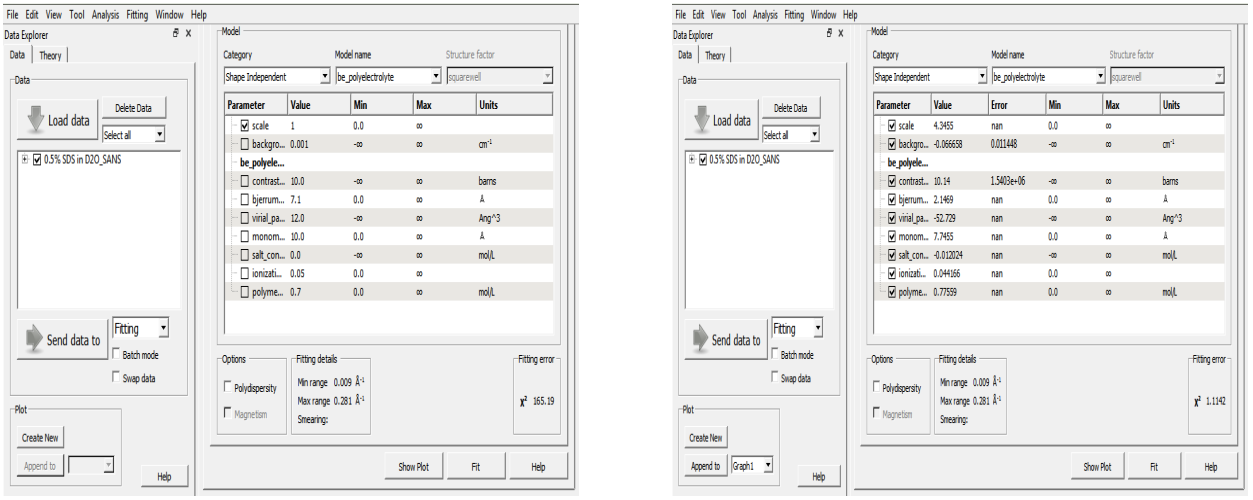
(c) h-SDS in D_2O with SWCNTs



(d) cm-SDS in D_2O with SWCNTs

Figure 3: SAXS and SANS Investigation to SDS in SWCNTs

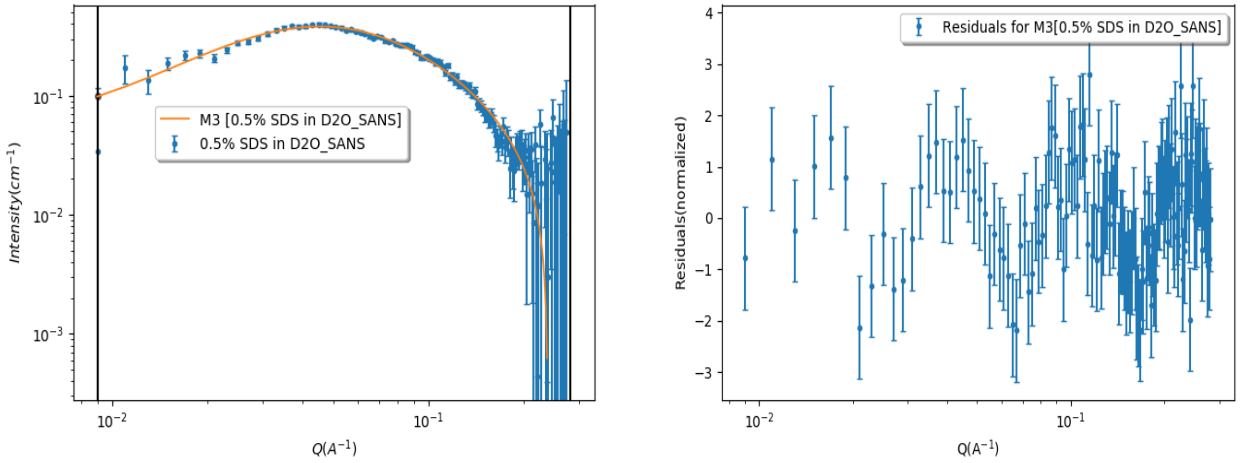
The following figure presents the changes made to reduce the error factor. It is important to enhance it until it reaches 1. In the case of the depicted graphs in Figure 5, the value of χ^2 developed from 165.19 to 1.14. In this example, the SDS absorption in D_2O is investigated through SANS, and data is graphed by SasView.



(a) Before changing parameters

(b) After changing parameters

Figure 4: SDS Absorption in D_2O Using SANS Technique Parameters

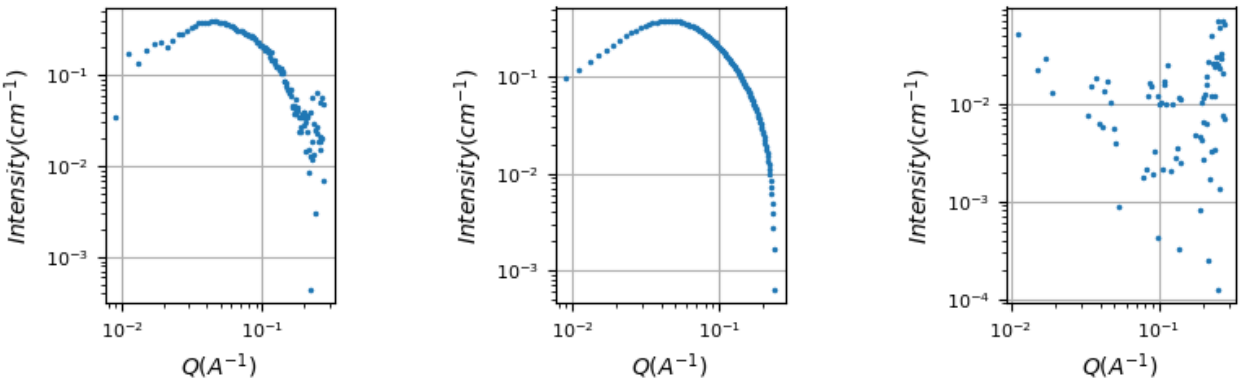


(a) Experimental data and its fitting

(b) Residual after fitting

Figure 5: SDS Absorption in D_2O Fitting and Residual

Another feature of SasView is to perform the subtraction process, which means that firstly, from sasView, we go to "Tool" from the toolbar, secondly, choose data, then we load the data which is needed to work on. Finally, we detect an operation to be performed, for example, "subtraction" which is denoted by "-". **??** shows the subtraction process of fitted data from all data, and the result is in Figure 6c, representing the excluded data.



(a) Data (b) Fitted after Subtraction (c) Remaining

Figure 6: Subtraction Process

2.3.2 Example-2: [SANS] Silica010

The model used for fitting the SILIC010.DAT file is "fuzzy_sphere" with fitting error $\chi^2 = 9.93$. The category to get the fuzzy_sphere model is "sphere", besides structure facto "hayter_msa".

Minimum range	Maximum range	Fitting error
0 $\mathring{A}^{-1}$	0.12083 $\mathring{A}^{-1}$	10.56

Table 2: Fitting Details

In fuzzy_sphere model [**?**] the scattering intensity is calculated by:

$$I(q) = \frac{scale}{V}(\delta\rho)^2 A^2(q) S(q) + background \tag{8}$$

While the amplitude $A^2(q)$ is calculated by:

$$A(q) = \frac{3[sin(qR) - qRcos(qR)]}{(qR)^3} \exp(\frac{-(\sigma_{fuzzy}q)^2}{2}) \tag{9}$$

V is the volume of the sphere, $(\delta\rho)^2$ represents the difference in scattering length density of the sphere and the solvent surrounding it, q is the vector given by $q = \sqrt{{q_x}^2 + {q_y}^2}$, σ_{fuzzy} is the fuzziness parameter of interference and R is the radius of the particle.

Figure 7 depicts the data results from sans analysis for silica010, then we get the shape and the internal structure depending on the model as in Figure 8, hence we have the residual shown in Figure 8.

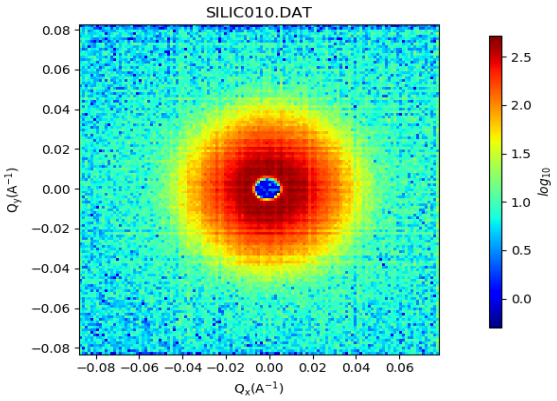


Figure 7: Experimental Data

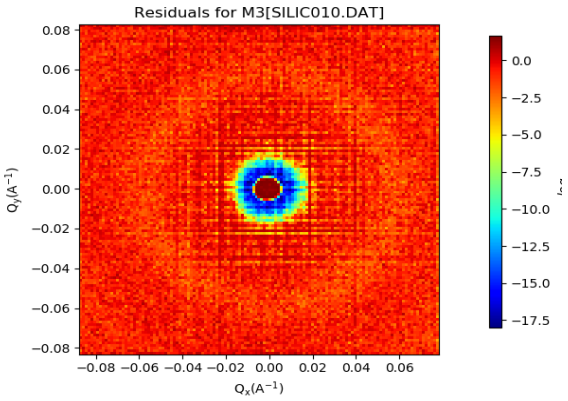


Figure 8: Residual Portion after Treatment

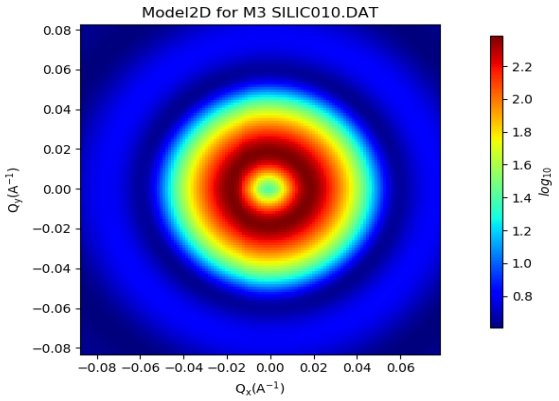


Figure 9: Modelled Result

The following table shows the parameters of sasView used to produce the figures above:

Parameter	Value	Error
scale	1.8648	1.2×10^5
background	3.961	0.036
sld	1.2645	nan
sld_solvent	6.4	2.8×10^5
raduis	75.66	0.0756
fuzziness	0.0049	416.77
radius_effective	20.75	–
volfraction	0.004	0.00046
charge	19	53.85
temperature	318.16	2.38×10^5
concentration_salt	0	0.00099
dielectconst	71.08	63392

Table 3: Fit Panel and Model Details

3 Proposal: SANS Studying for Sodium-2-Diethylhexyl Sulfosuccinate (AOT)

3.1 Aim of the Work and Background

The following work aims to study a sample of Sodium-2-Diethylhexyl Sulfosuccinate (AOT) by SANS. Many studies are performed to investigate AOT as a mixture with other samples. The sodium dodecyl sulfate-poly(propylene oxide) methacrylate mixed micelles is studied through small-angle neutron scattering in [10], the wavelength of the beam used for studying is 10 Å and the scattering range is 0.01 to 0.11 $Å^{-1}$. Another study [13], used SANS with changing the contrast of incident neutron beam for polydispersity and shape variations of droplets in AOT.

3.2 Experimental Setup and Instrumentation

The proposed facility to be used for SANS analysis is PAXY detector, which is shown in Figure 10 [8, 11, 12]. PAXY is used for high resoultion studies, with momentum range of 3×10^{-3} $Å^{-1}$ to 1 $Å^{-1}$. The temperature of the ambient should be 25 °C, while the maxamim and minimum range of q are 0.281 $Å^{-1}$ and 0.009 $Å^{-1}$.

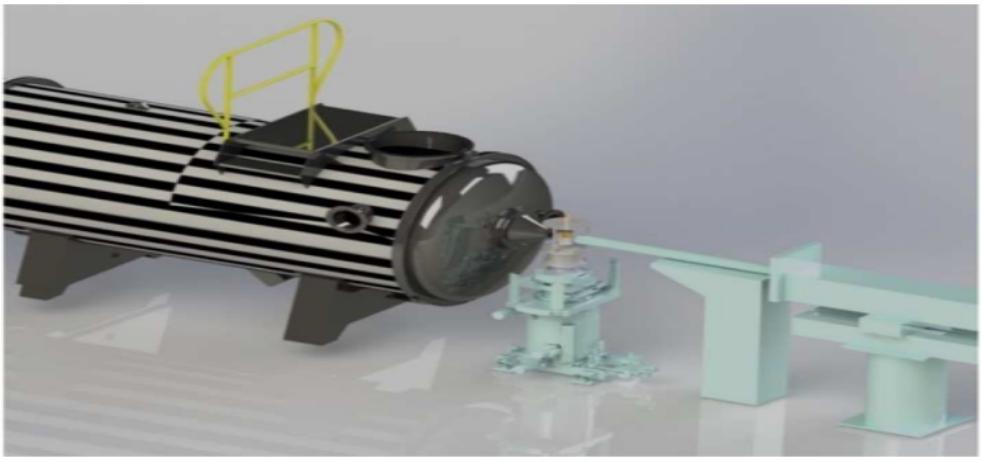


Figure 10: PAXY Facility

3.3 Predicted Results

These analyses are made by SasView, the category is "Sphere" and the treating model is "polymer_micelle". The maximum and minimum ranges of fitting details are 0.009 $Å^{-1}$ and 0.281 $Å^{-1}$, respectively. The fitting error is 6.82.

The following table shows the parameters of sasView used to produce the figures below:

Parameter	Value	Error
scale	262.29	1.16×10^7
background	-1.35	5.51
ndensity	12.328	nan
v_core	73901	nan
v_corona	1.14×10^5	nan
sld_solvent	1.101	2221.2
sld_core	0.237	nan
sld_corona	0.617	nan
radius_core	37.68	32.75
rg	3.13	7.19
d_pentration	-1.56	21.28
n_aggreg	1.706	10.09

Table 4: Fit Panel and Model Details

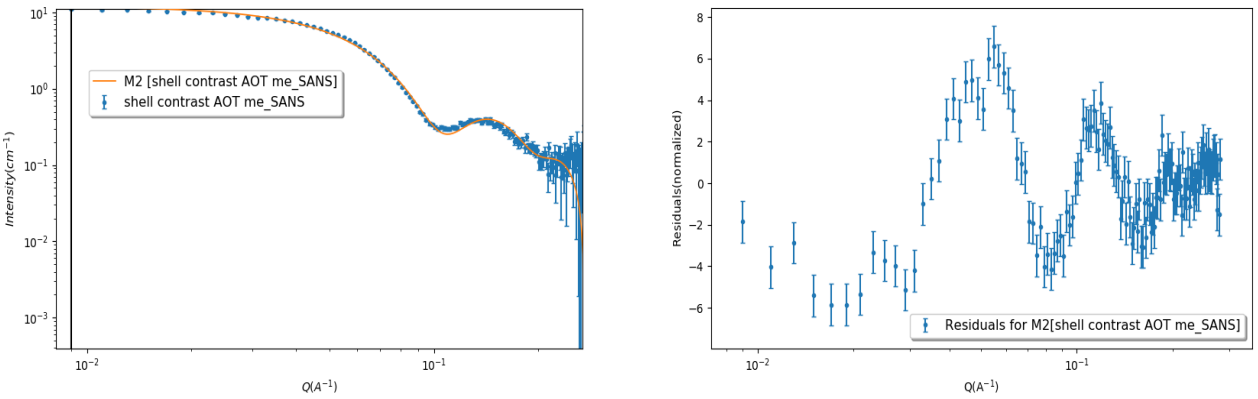


Figure 11: Fitting and Residual for AOT Microemulsions Analysis Through SANS by SasView

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