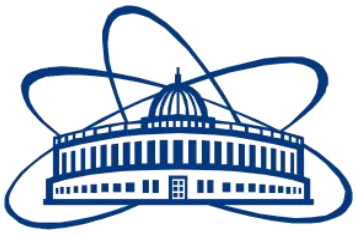

JOINT INSTITUTE FOR NUCLEAR RESEARCH
Frank Laboratory of Neutron Physics

FINAL REPORT ON THE
INTEREST PROGRAMME

Introduction to Neutron Scattering Experiments at Large Scale Facilities

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Abstract

In this report the next neutron techniques are presented: Neutron Scattering, Neutron Spectroscopy and Small Angle Neutron Scattering (SANS). The practical part introduces the software package SasView which is an open source, collaboratively developed software for analysis and modeling of small angle scattering (SAS) data. Also, I am reporting some theoretical experiments experimental data and data fitting procedures for structure factor determination.

Table of Content

Introduction

Neutron

The neutron is the particle in the atomic nucleus with a mass = 1 and charge = 0. Neutrons are found together with protons in the atomic nucleus. The number of neutrons in an atom determines its isotope.

Although a neutron has a net neutral electrical charge, it does consist of charged components which cancel each other out with respect to charge.

Neutrons are the largest of the particles that make up the atom. The neutron is a baryon, which means it is a massive particle that is made up of 3 quarks (1 up quark and 2 down quarks). Quarks are what build all matter. You may have heard that atoms are the building blocks of all matter and this is true, but now scientists have figured out that quarks build all the parts that make up atoms.
Neutrons are found in the nucleus of the atom. They are tightly packed with the protons. The protons and neutrons are held together in the nucleus with a force called the strong nuclear force. The nucleus is a very dense area that is found in the center of the atom.

Neutrons have a neutral charge or actually no charge at all. Its partner in the nucleus, the proton, does have a positive charge. The proton's positive charge is matched with the electron's negative charge to make a neutral atom. Even though the neutron does not impact the charge of the atom, it still has many properties that influence the atom, including the atom's level of radioactivity, but we will get to that later

The neutron is 0.2% larger than the proton. Together, the neutron and proton make up 99.99% of all the mass of the atom. The neutron actually has the same mass as an electron and proton put together. The atomic mass of an atom is found by adding the number of protons and neutrons. Since the atomic number refers to the number of protons of an atom, we can use both the atomic mass and the atomic number to figure out the number of neutrons. When using the simple equation below, we are able to find the number of neutrons in an atom.

Atomic Mass - Atomic Number = Number of Neutrons

For instance, Carbon has an atomic mass of 12 and an atomic number of 6. So if we place these numbers in our equation, it will look like this…

12 (Atomic Mass) - 6 (Atomic Number) = 6 Neutrons.

Neutrons are produced at large research infrastructures. Researchers use them to look inside materials. With neutrons one can e.g. look inside a car engine, investigate drug delivery, see how plants uptake water, get insights into the development of superconductors.

For research purposes, neutrons are released from their role as a component part of matter, and are used as probes with which researchers can look inside a very wide variety of materials. In a typical neutron experiment, a neutron beam passes through the object under investigation. The experimenters then observe how the characteristics of the beam have been changed by its interaction with the sample. By doing this, they can obtain information about the internal structure or composition of the sample.

When beams of neutrons are used to probe small samples of materials they have the power to reveal what cannot be seen using other types of radiation. Neutrons appear to behave either as particles or

as waves or as microscopic magnetic dipoles. It is these specific properties which enable them to yield information which is often impossible to obtain using other techniques.

Beams of neutrons can be scattered by materials. If the neutron energy is chosen correctly, then their wavelength is similar to the distances between atoms or molecules, and the pattern of scattered neutrons can be used to produce an image of the atomic structure.

Neutron Fusion

Nuclear fusion is a reaction in which two or more atomic nuclei are combined to form one or more different atomic nuclei and subatomic particles (neutrons or protons). The difference in mass between the reactants and products is manifested as either the release or the absorption of energy. This difference in mass arises due to the difference in nuclear binding energy between the atomic nuclei before and after the reaction. Nuclear fusion is the process that powers active or main sequence stars and other high-magnitude stars, where large amounts of energy are released.

When two light nuclei fuse to form a larger nucleus, energy is released, since the larger nucleus is more tightly bound, as seen from the binding energy curve. Some examples of such energy liberating nuclear fusion reactions are

$$_1^1\mathrm{H} + {}_1^1\mathrm{H} \rightarrow {}_1^2\mathrm{H} + e^+ + \nu + 0.42\ \mathrm{MeV}$$

$$_1^2\mathrm{H} + {}_1^2\mathrm{H} \rightarrow {}_2^3\mathrm{He} + n + 3.27\ \mathrm{MeV}$$

$$_1^2\mathrm{H} + {}_1^2\mathrm{H} \rightarrow {}_1^3\mathrm{H} + {}_1^1\mathrm{H} + 4.03\ \mathrm{MeV}$$

In the first reaction, two protons combine to form a deuteron and a positron with a release of 0.42 MeV energy. In 2nd reaction two deuterons combine to form the light isotope of helium. In 3rd reaction, two deuterons combine to form a triton and a proton. For fusion to take place, the two nuclei must come close enough so that attractive short-range nuclear force is able to affect them. However, since they are both positively charged particles, they experience coulomb repulsion. They, therefore, must have enough energy to overcome this coulomb barrier. The height of the barrier depends on the charges and radii of the two interacting nuclei. It can be shown

X-RAY

X-ray, electromagnetic radiation of extremely short wavelength and high frequency, with wavelengths ranging from about 10^{-8} to 10^{-12} metre and corresponding frequencies from about 10^{16} to 10^{20} hertz (Hz).

X-rays are commonly produced by accelerating (or decelerating) charged particles; examples include a beam of electrons striking a metal plate in an X-ray tube and a circulating beam of electrons in a synchrotron particle accelerator or storage ring. In addition, highly excited atoms can emit X-rays with discrete wavelengths characteristic of the energy level spacings in the atoms. The X-ray region of the electromagnetic spectrum falls far outside the range of visible wavelengths. However, the passage of X-rays through materials, including biological tissue, can be recorded with photographic films and other detectors. The analysis of X-ray images of the body is an extremely valuable medical diagnostic tool.

X-rays are a form of ionizing radiation—when interacting with matter, they are energetic enough to cause neutral atoms to eject electrons. Through this ionization process the energy of the X-rays is

deposited in the matter. When passing through living tissue, X-rays can cause harmful biochemical changes in genes, chromosomes, and other cell components. The biological effects of ionizing radiation, which are complex and highly dependent on the length and intensity of exposure, are still under active study (*see* radiation injury). X-ray radiation therapies take advantage of these effects to combat the growth of malignant tumours.

X-rays were discovered in 1895 by German physicist Wilhelm Konrad Röntgen while investigating the effects of electron beams (then called cathode rays) in electrical discharges through low-pressure gases. Röntgen uncovered a startling effect—namely, that a screen coated with a fluorescent material placed outside a discharge tube would glow even when it was shielded from the direct visible and ultraviolet light of the gaseous discharge. He deduced that an invisible radiation from the tube passed through the air and caused the screen to fluoresce. Röntgen was able to show that the radiation responsible for the fluorescence originated from the point where the electron beam struck the glass wall of the discharge tube. Opaque objects placed between the tube and the screen proved to be transparent to the new form of radiation; Röntgen dramatically demonstrated this by producing a photographic image of the bones of the human hand. His discovery of so-called Röntgen rays was met with worldwide scientific and popular excitement, and, along with the discoveries of radioactivity (1896) and the electron (1897), it ushered in the study of the atomic world and the era of modern physics.

In the early 1920s, experimental studies of the scattering of X-rays from solids played a key role in establishing the particle nature of electromagnetic radiation. In 1905 German physicist Albert Einstein had proposed that electromagnetic radiation is granular, consisting of quanta (later called photons) each with an energy hf, where h is Planck's constant (about 6.6×10^{-34} joule·second) and f is the frequency of the radiation. Einstein's hypothesis was strongly supported in subsequent studies of the photoelectric effect and by the successes of Danish physicist Niels Bohr's model of the hydrogen atom and its characteristic emission and absorption spectra (*see* Bohr atomic model). Further verification came in 1922 when American physicist Arthur Compton successfully treated the scattering of X-rays from the atoms in a solid as a set of collisions between X-ray photons and the loosely bound outer electrons of the atoms.

The defining characteristics of X-rays—their ability to penetrate optically opaque materials, their wavelengths of atomic dimension, the high energy of individual X-ray photons—lead to a wide range of industrial, medical, and scientific applications. Specialized X-ray sources, detectors, and analysis techniques have been developed to address a range of questions from the study of the interactions of the simplest molecules to the structure of the human brain.

X-ray images of the body are an indispensable diagnostic tool in modern medicine. Medical imaging allows for the nonintrusive detection of dental cavities, bone fractures, foreign objects, and diseased conditions such as cancer.

Neutron Sources

A neutron source is any device that emits neutrons. Neutron sources have many applications, they can be used in research, engineering, medicine, petroleum exploration, biology, chemistry and nuclear power. A neutron source is characterized by a number of factors:

- Significance of the source
- Intensity. The rate of neutrons emitted by the source.
- Energy distribution of emitted neutrons.
- Angular distribution of emitted neutrons.
- Mode of emission. Continuous or pulsed operation.

Some of Neutron Sources are-

Nuclear Reactors. There are nuclei that can undergo fission on their own spontaneously, but only certain nuclei, like uranium-235, uranium-233 and plutonium-239, can sustain a fission chain reaction. This is because these nuclei release neutrons when they break apart, and these neutrons can induce fission of other nuclei. Uranium-235 which exists as 0.7% of naturally occurring uranium undergoes nuclear fission with thermal neutrons with the production of, on average, 2.4 fast neutrons and the release of ~ 180 MeV of energy per fission. Free neutrons released by each fission play very important role as a trigger of the reaction, but they can be also used fo another purpose. For example: One neutron is required to trigger a further fission. Part of free neutrons (let say 0.5 neutrons/fission) is absorbed in other material, but an excess of neutrons (0.9 neutrons/fission) is able to leave the surface of the reactor core and can be used as a neutron source.

- Fusion Systems. Nuclear fusion is a nuclear reaction in which two or more atomic nuclei (e.g. D+T) collide at a very high energy and fuse together. Thy byproduct of DT fusion is a free neutron (see picture), therefore also nuclear fusion reaction has the potential to produces large quantities of neutrons.
- Spallation Sources. A spallation source is a high-flux neutron source in which protons that have been accelerated to high energies hit a heavy target material, causing the emission of neutrons. The reaction occurs above a certain energy threshold for the incident particle, which is typically 5 – 15 MeV.

Neutron Spallation Sources-

Neutron scattering allows scientists to count scattered neutrons, measure their energies and the angles at which they scatter, and map their final positions. This information can reveal the molecular and magnetic structure and behavior of materials, such as high-temperature superconductors, polymers, metals, and biological samples. In addition to studies focused on fundamental physics, neutron scattering research has applications in structural biology and biotechnology, magnetism and superconductivity, chemical and engineering materials, nanotechnology, complex fluids, and others.The spallation process at SNS begins with negatively charged hydrogen ions that are produced by an ion source. Each ion consists of a proton orbited by two electrons. The ions are injected into a linear particle accelerator, or linac, which accelerates them to an energy of about one GeV (or to about 90% the speed of light) The ions pass through a foil, which strips off each ion's two electrons, converting it to a proton. The protons pass into a ring-shaped structure, a proton accumulator ring, where they spin around at very high speeds and accumulate in "bunches." Each bunch of protons is released from the ring as a pulse, at a rate of 60 times per second (60 hertz). The high-energy proton pulses strike a target of liquid mercury, where spallation occurs. The spalled neutrons are then slowed in a moderator and guided through beam lines to areas containing special instruments where they are used in a wide variety of experiments

Neutron Techniques

Neutron techniques are based on the detection of the nuclear properties of possible threats rather than their density or chemical properties. One may summarize the advantages of these techniques as follows:

1. Determine elemental composition not just density: This is the major advantage of nuclear techniques as compared with conventional X-ray scanners. Conventional X-ray techniques generally measure the apparent density and shape of objects. Using dual-energy X-rays, one also gets an approximation to the average atomic number (Z) of the material, and can it and spatial extent as a clue to the presence of an explosive material. Neutron techniques offer the possibility of determining the elemental composition of a suspicious object.

2. Great penetration and potential for cargo inspection: The effective range of neutrons even in metals enables penetration of much greater thicknesses of materials than the usual low-energy (140 kV) X-ray scanner, although it should be noted that high-energy X-ray sources, such as from multi-MeV electron linacs, offer similar penetration. Of course, each technique has its limitations; neutrons are shielded by low-Z materials, particularly organics, and high-energy X-rays by high-Z metals.

3. Difficult to shield contraband against probing radiation, usually neutrons or gammas: This is a corollary of the previous statement; however, in the interest of completeness, one should recognize that the presence of a large absorber in an object under inspection is in itself an indication of a potential threat and will also trigger a “shield alarm” in many X-ray imaging systems.

4. Detection of nuclear materials: This is a different aspect of the use of neutrons in that nuclear materials may be detected by their response to probing neutrons. In some cases, depending on the neutron energy and the material under inspection, this can be a very clear identifier of such materials.

Neutron Scattering

Neutron scattering, the irregular dispersal of free neutrons by matter, can refer to either the naturally occurring physical process itself or to the man-made experimental techniques that use the natural process for investigating materials. The natural/physical phenomenon is of elemental importance in nuclear engineering and the nuclear sciences. Regarding the experimental technique, understanding and manipulating neutron scattering is fundamental to the applications used in crystallography, physics, physical chemistry, biophysics, and materials research.

Neutron scattering is practiced at research reactors and spallation neutron sources that provide neutron radiation of varying intensities. Neutron diffraction (elastic scattering) techniques are used for analyzing structures; where inelastic neutron scattering is used in studying atomic vibrations and other excitations.

Because neutrons are electrically neutral, they penetrate more deeply into matter than electrically charged particles of comparable kinetic energy, and thus are valuable as probes of bulk properties.

Neutrons interact with atomic nuclei and with magnetic fields from unpaired electrons, causing pronounced interference and energy transfer effects in neutron scattering experiments. Unlike an x-ray photon with a similar wavelength, which interacts with the electron cloud surrounding the nucleus, neutrons interact primarily with the nucleus itself, as described by Fermi's pseudopotential. Neutron scattering and absorption cross sections vary widely from isotope to isotope.

Neutron scattering can be incoherent or coherent, also depending on isotope. Among all isotopes, hydrogen has the highest scattering cross section. Important elements like carbon and oxygen are quite visible in neutron scattering—this is in marked contrast to X-ray scattering where cross sections systematically increase with atomic number. Thus neutrons can be used to analyze materials with low atomic numbers, including proteins and surfactants. This can be done at synchrotron sources but very high intensities are needed, which may cause the structures to change. The nucleus provides a very short range, as isotropic potential varies randomly from isotope to isotope, which makes it possible to tune the (scattering) contrast to suit the experiment.

Scattering almost always presents both elastic and inelastic components. The fraction of elastic scattering is determined by the Debye-Waller factor or the Mössbauer-Lamb factor. Depending on the research question, most measurements concentrate on either elastic or inelastic scattering.

Achieving a precise velocity, i.e. a precise energy and de Broglie wavelength, of a neutron beam is important. Such single-energy beams are termed 'monochromatic', and monochromaticity is achieved either with a crystal monochromator or with a time of flight (TOF) spectrometer. In the time-of-flight technique, neutrons are sent through a sequence of two rotating slits such that only neutrons of a particular velocity are selected. Spallation sources have been developed that can create a rapid pulse of neutrons. The pulse contains neutrons of many different velocities or de Broglie wavelengths, but separate velocities of the scattered neutrons can be determined *afterwards* by measuring the time of flight of the neutrons between the sample and neutron detector.

Neutron Spectroscopy

Inelastic neutron scattering measures the change in the energy of the neutron as it scatters from a sample. This can be used to probe a wide variety of different physical phenomenon: diffusional or hopping motions of atoms, the rotational modes of molecules, sound modes and molecular vibrations, recoil in quantum fluids, magnetic and quantum excitations or even electronic transitions.

Often knowing the atomic structure of a material will be sufficient to understand its nature. But to gain a deeper insight into the underlying physics of say a phase change, it is necessary to also understand the atomic dynamics. The vibrational motion of atoms is, entirely or in part, responsible for a large number of the characteristic properties of a material, such as the specific heat, thermal conductivity, optical and dielectric properties and electrical resistance, but it is also a direct way of understanding the nature of atomic bonding. And with the current interest in smart or functional materials whose properties are often determined by a complex balance or strong coupling between competing phenomena, understanding the atomic and magnetic dynamics is essential.

The most commonly used spectroscopies are the light scattering techniques: Raman and infrared. Compared to neutron scattering they are cheap, readily available and highly sensitive. But they do have certain limitations: They can only measure near the Brillouin zone centre and are only sensitive to certain vibrational modes. The calculation of the scattered intensity is also difficult and prone to error and so information is usually only taken from the positions of the observed modes. However, the simplicity and sensitivity of the techniques means that they are often used to identify or ‘finger print’ compounds something that is rarely done with neutrons. Inelastic neutron scattering is usually used to understand the physics of a system. It is a highly quantitative probe whose results are directly comparable to numerical and analytical calculation. It can be used to understand the nature of a phase transitions or linked directly to thermodynamics quantities, like specific heat or thermal conductivity, or structural properties such as force tensors or bulk and shear moduli. It is still one of the few methods available to measure phonon and magnon dispersion curves. And, due to the unique

nature in way that hydrogen scatters neutrons, it is a natural technique for measuring the vibration or diffusion of hydrogen in a material.

Small Angle Neutron Scattering (SANS)

Small-angle neutron scattering (SANS) provides a unique method to probe soft matter in the 10–100 nm length scale in solutions. In order to determine the shape and size of biological macromolecular structures correctly with SANS, a background-subtracted, undistorted scattering curve must be measured, and the required accuracy and precision is especially needed at the short-length-scale limit. A true scattering curve is also needed to discern whether intermolecular interactions are present, which also are probed in the SANS experiment. This article shows how to detect intermolecular interactions so that subsequent structure modeling can be performed using only data that do not contain such contributions. It is also shown how control of many factors can lead to an accurate baseline, or background, correction for scattering from proteins, especially to account for proton incoherent scattering. Failure to make this background correction properly from proteins, polymers, nucleic acids and lipids can result in incorrect values for the calculated shapes and sizes of the molecules as well as the derived magnitudes of the inter-molecular interactions.
During a SANS experiment a beam of neutrons is directed at a sample, which can be an aqueous solution, a solid, a powder, or a crystal. The neutrons are elastically scattered by nuclear interaction with the nuclei or interaction with magnetic momentum of unpaired electrons. In X-ray scattering, photons interact with the electronic cloud so the bigger the element, the bigger the effect is. In neutron scattering, neutrons interact with nuclei and the interaction depends on the isotope; some light elements like deuterium show similar scattering cross section as heavy elements like Pb.

SASVIEW

Introduction

SasView is an open source, collaboratively developed software for analysis and modeling of small angle scattering (SAS) data. The core functionality of SasView includes fitting of model functions, pair-distance distribution function inversion, model-independent analysis and correlation function calculations. SasView provides a large collection of form and structure factors and allows for easy incorporation of user-defined models. SasView also supports calculations on Graphical Processing Units (GPUs), which allows for faster evaluation of complex model functions.
SasView was originally developed by the University of Tennessee as part of the Distributed Data Analysis of Neutron Scattering Experiments (DANSE) in 2006 project funded by the US National Science Foundation (NSF) but is currently being developed as an Open Source project hosted on GitHub in 2013 and managed by a consortium of scattering facilities. Participating facilities include (in alphabetical order): the Australian National Science & Technology Centre for Neutron Scattering, the Diamond Light Source, the European Spallation Source, the Federal Institute for Materials Research and Testing, the Institute.. Laue Langevin, the ISIS Pulsed Neutron & Muon Source,

Interface of SasView

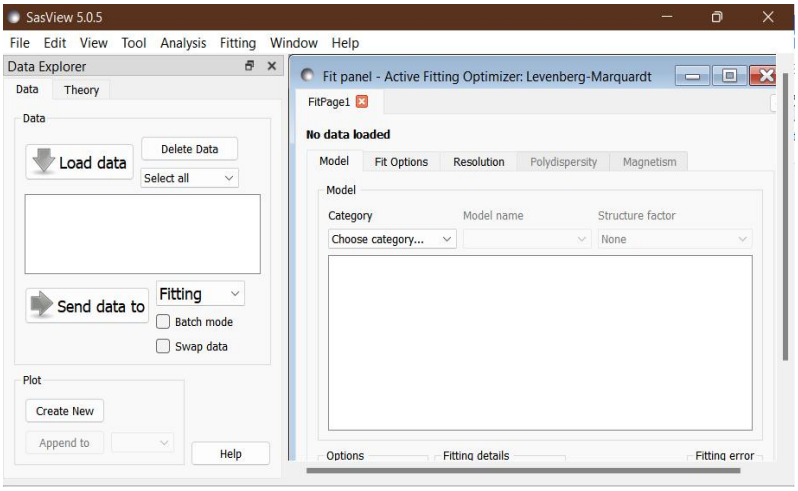


Fig- Typical Interface of SasView

Typical SAS data analysis workflow

1. Acquire data
2. Define a model with some free parameters
3. Calculate scattering pattern for that model for a given set of parameters
4. Fit the parameter to simulated data based on model matches experimental data.

Experiments

Objective - To demonstrates a fit requiring a structure factor and introduces the use of multiple FitPages

From the \test\1d_data folder in the SasView installation directory, load the hSDS_D2O_2p0_percent.xml dataset and send the data for fitting.

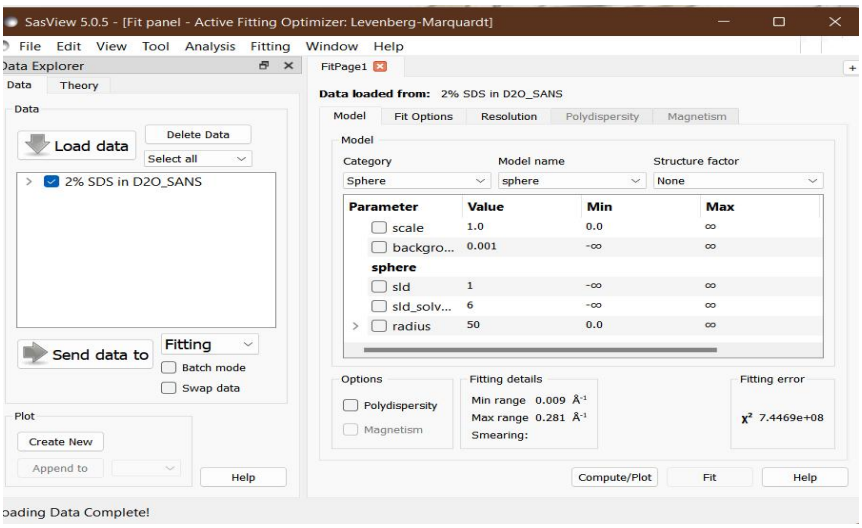


Fig: Loading data in SasView

This dataset is the SANS from a 2 %w /w solution of the widely-studied surfactant h25- sodium dodecyl sulphate (SDS) in heavy water (D2O).

The critical micelle concentration for SDS is about 0.2 %w /w, so the scattering must represent a concentrated micellar solution, SDS is also an anionic surfactant, so the micelles will be charged. Interactions between the charged micelles can be expected to manifest themselves in the scattering as a structure factor. And in the absence of electrolyte SDS micelles are reported to be approximately spherical. To start with, select the sphere model.

This model has 5 parameters. Two of them, sld (for the SDS) and sld_solvent (for the D2O), can be calculated explicitly.

• SDS: Na1C12H25S1O4 ; density 1.01 g/cm3
• D2O: D2O1 ; density 1.11 g/cm3

The values of sld = 0.337x10-6 Å -2 and sld_solvent = 6.39x10-6 Å -2

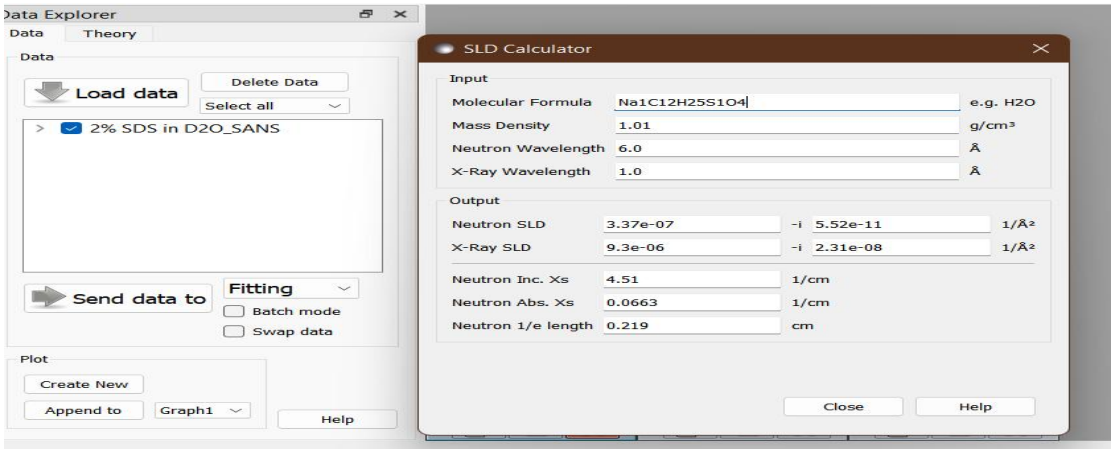


Fig- Calculating sld and sld_solvent for Na1C12H25S1O4

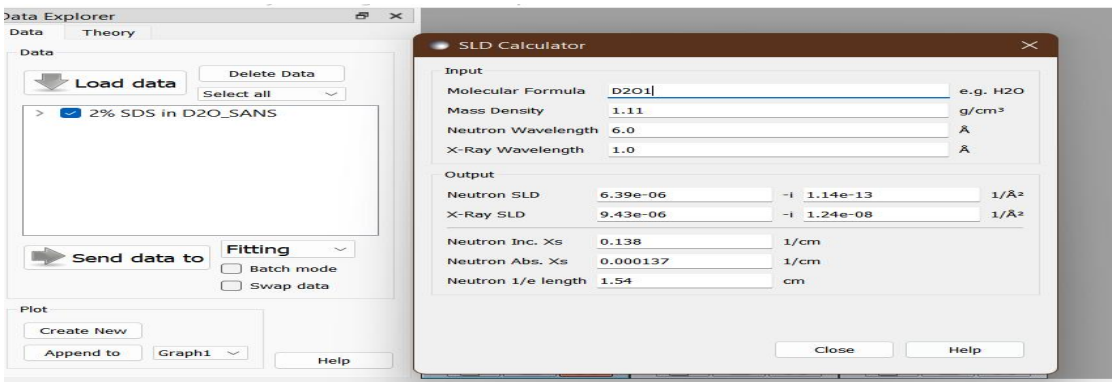


Fig: Calculating sld and sld_solvent for D2O

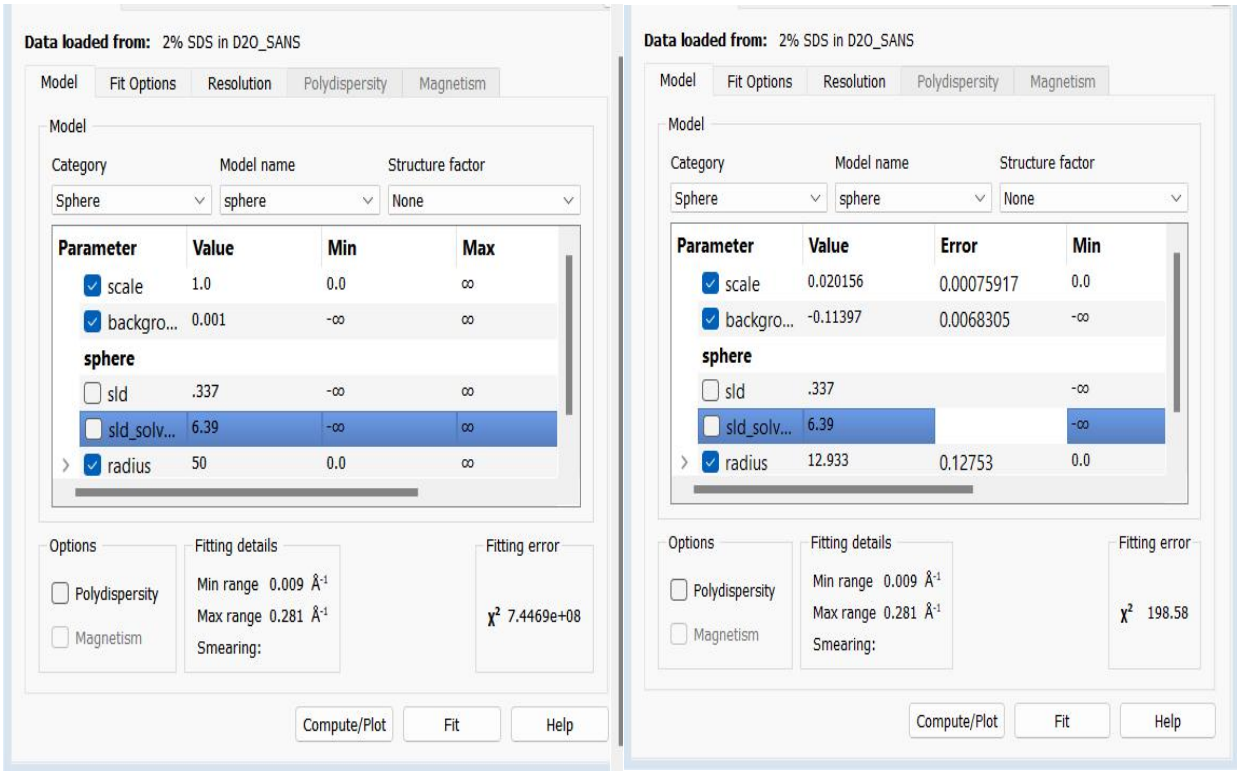
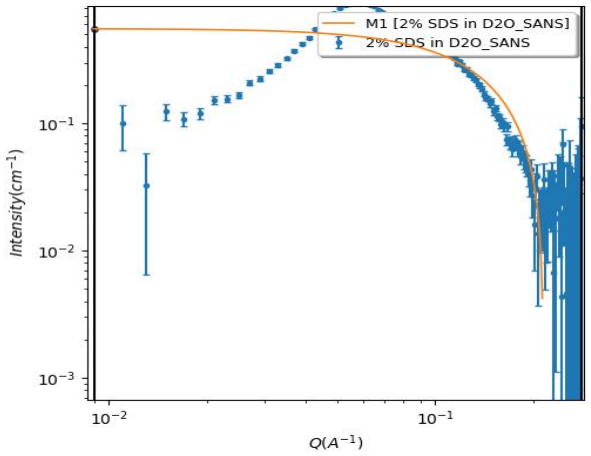
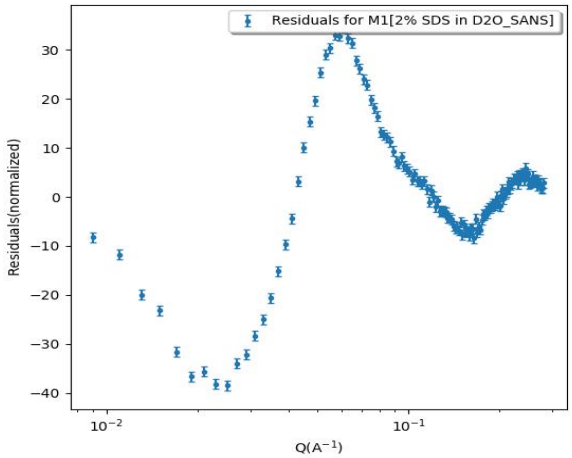


Fig- Assigning Parameter into the SasView and After fitting results.



(a) Experimental data and its fitting



(b) Residual after fitting

The orange theory curve now represents the scattering from non-interacting, monodisperse, spherical micelles ~13 Å in radius. And the scale parameter in this case represents the volume fraction of micelles.

Applying hayster_msa parameter, In hayster_msa, filling paramters whose values is known,

- volfraction: 2 %w /w is approximately (2/1.01) %v /v = 0.019
- charge:
- temperature: 298 K
- concentration_salt: 0 M
- dielectconst: 78.06 (Vidulich et al, J Phys Chem, 1967)

However, the volfraction parameter now duplicates the scale parameter, set scale=1.0 and uncheck it, and check the volfraction parameter instead. Also check the charge parameter. Run the fit again

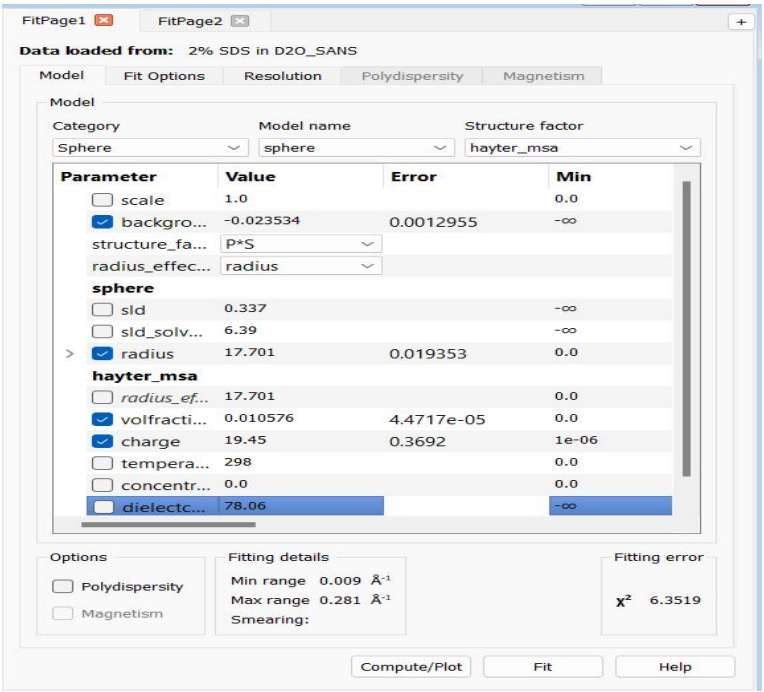
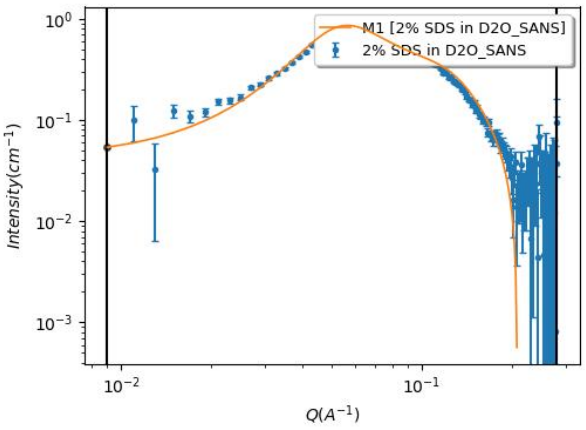
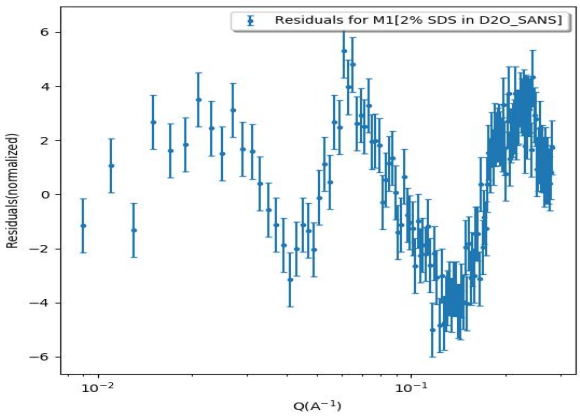


Fig- Choosing hayter_msa as structure factor for better fitting



(a) Experimental data and its fitting



(b) Residual after fitting

This improve the fit further. The Reduced Chi2 has dropped to 0.855, the residuals are within +/- 2, and the volfraction parameter has increased to almost 1.7 %.

To improve the fit further, we are switching to Elipsoid,

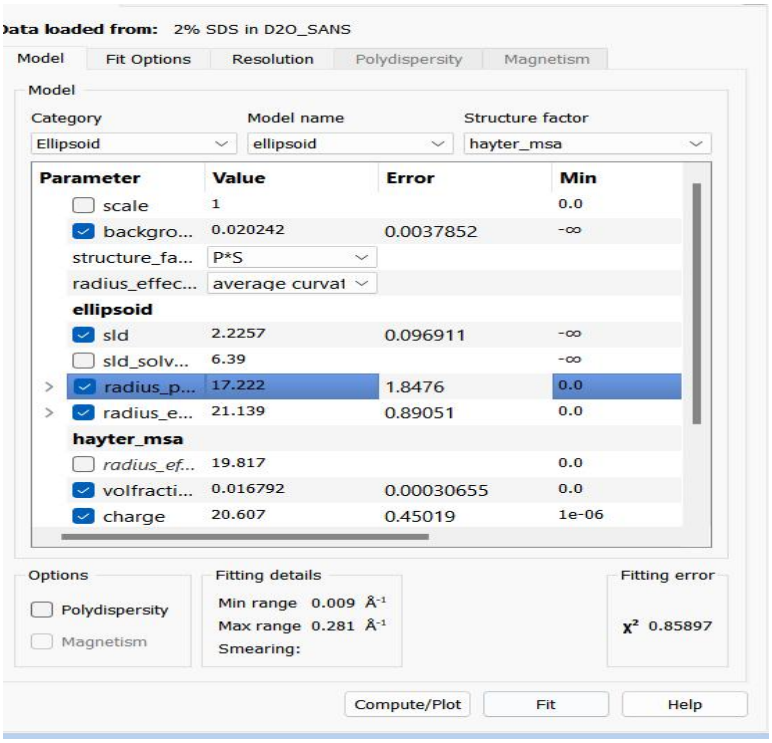
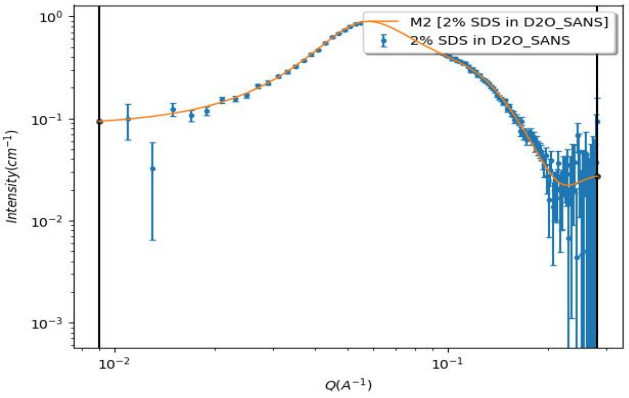
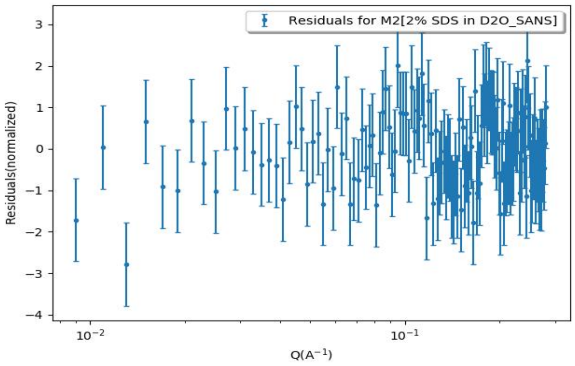


Figure- SDS Absorption in D_2O Using SANS Technique Parameters



(a) Experimental data and its fitting



(b) Residual after fitting

A fit at least as good as the one to the sphere model is obtained, suggesting that the micelles are ellipsoids with an axial ratio of 1:1.2. So the SasView fitting is certainly consistent with the literature reports. But unambiguously distinguishing between the two cases would require more precise measurements and higher quality data.

Conclusion

After the successful completion of this project on Introduction to neutron scattering experiments at large scale facilities, I have implemented an experiment To demonstrates a fit requiring a structure factor and introduces the use of multiple FitPages, where I have implemented different approach for the model to fit the data well such as sphere and elipsoid while introducing structure hayster_msa and providing the required parameters and the resulting curve came out to be fitting the data well.

References

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