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New opportunities provided by modernized small-angle neutron scattering two-detector system instrument (YuMO)

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Abstract. Main features of the modernized small-angle neutron scattering spectrometer (YuMO) at IBR-2M pulsed reactor are described. New installations for sample environment of the spectrometer are highlighted. The modernized SANS instrument (YuMO) is equipped with a new type of position sensitive detector as well as two detector system which provide a unique dynamic range (Q_{\max}/Q_{\min} ratio is about 90). Sample environment is extended with a magnetic system (magnetic field about 2.5 Tesla), automated high pressure setup which allows simultaneous SANS and volumetric high pressure studies and light illumination system. In particular, these developments led to considerable improvements of resolution of the instrument (about 1%) and opened the possibility to study anisotropic materials and perform efficient high pressure studies.

1. Introduction

In 2011 the IBR-2M pulsed reactor (JINR, Dubna, Moscow region) will face new challenges. One of the most efficient instruments at the IBR-2M is small angle neutron scattering spectrometer YuMO which allows one to obtain $I(q)$ curves in absolute scale. The spectrometer is characterized by relatively short data acquisition time which depends on the type of the studied material and varies from minutes to hours. The YuMO spectrometer is used for wide range of scientific and technical applications.

Main features of the YuMO spectrometer are as follows: high flux, absolute scale, two-detector system, high (relative to typical SANS instruments at the steady neutron sources) resolution and circle geometry. Due to direct view of the surface of the moderator of the reactor and time-of-flight method for wavelength determination the instrument has higher flux [1, 2].

Most of the studied materials via small-angle neutron scattering (SANS) require data collection in a wide range of momentum transfer (Q -range). Larger Q -range means more complete and reliable determination of structural parameters of the investigated materials. The dynamic range (a ratio of

Q_{\max} to Q_{\min} measured simultaneously) of SANS instruments is normally determined by the sizes of the detector, which are limited mainly by technical reasons, and by the wavelength range of available thermal neutrons in the neutron beam. It was shown [3,4] that the time-of-flight method provides necessary conditions for a considerable increase of the dynamic range of a SANS instrument by using two or more detectors of scattered neutrons with central holes placed at different distances from the sample. Excellent resolution of YuMO spectrometer was recently further improved by using new type of position sensitive detector PSD and is about 1% of module scattering vector [5-6].

In addition, the sample environment of the SANS instrument was also improved providing new opportunities for the researches working in different fields of science. We describe here recently installed at the YuMO magnet (2.8 T), high pressure setup (4 kbar) and a system for light illumination of the samples.

2. High-pressure setup

High penetration of neutrons into the materials and the possibility of contrast variation by using different sample preparation techniques make neutron scattering a valuable tool for high pressure studies. First high pressure setup at the small angle neutron scattering facility was realised using the Ti/Zr “null matrix” alloy [7]. The experiments with the surfactants C14MDAO and TDMAO provided new important information [7, 8].

Recently we used developed high pressure setup (HPS) to perform the studies of main (chain melting) phase transition in lipid membranes. Membrane fluidity predetermines many of the membrane properties. Pressure influences membrane fluidity, structure and dynamics of membranes. Some bacteria (for example algae) live under extreme conditions. Therefore, high pressure studies of the membranes are of significance for better understanding of their biological and biophysical properties [9].

However high pressure setup (HPS) with Ti/Zr alloy chamber provides satisfactory results only in q -range up to 0.03 \AA^{-1} . Second step of the development of HPS was based on a new approach, namely: separation of the liquid used to create a pressure from the studied samples. One of the parts of the device is the SITEC hand pump. The HPS was automated by homemade mechanical and electronic devices which allowed us to considerably increase the efficiency of the corresponding studies. New scientific results with surfactant systems have been obtained with the mentioned above high pressure setup [10, 11].

Next step of modernization of hydrostatic high-pressure setup was adaptation of existing device to the volumetric measurements. For this purpose the rotation angle sensor was mounted at the hand pressure pump. It provides control of the change of the volume with accuracy up to $\pm 2 \cdot 10^{-6} \text{ ml}$. In addition, pressure pump was equipped with a motor which gives the possibility to change volume of the system with a rate about $2 \cdot 10^{-3} \text{ ml/s}$. Electronic pressure sensor allows one to control pressure with accuracy of about $\pm 0,1 \text{ bar}$. The temperature in the pressure cell is controlled by the LAUDA thermostat with an error $\pm 0,05^\circ\text{C}$. First P-V-T experiments with the water-dimyristoylphosphatidylcholine (DMPC) system have been done in [8]. Pressure dependence of isothermal compressibility of lipid membranes was studied. Dependence of the pressure corresponding to phase transition from gel to liquid crystal phase in the studied system was obtained in the temperature range $(24 \div 35)^\circ\text{C}$ and a P-V-T phase diagram was determined in the pressure range $(1 \div 800) \text{ bar}$.

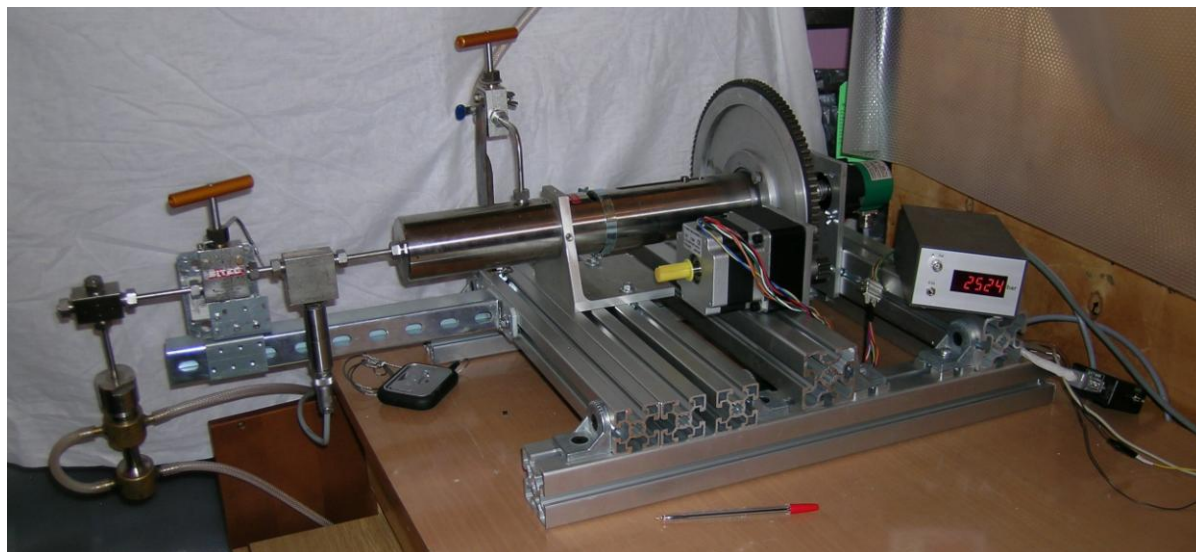


Figure 1. Volumetric high-pressure setup for the YuMO spectrometer, adapted for SANS measurements.

The typical experimental curve of compressibility via pressure near main phase transition point is shown in figure 2.

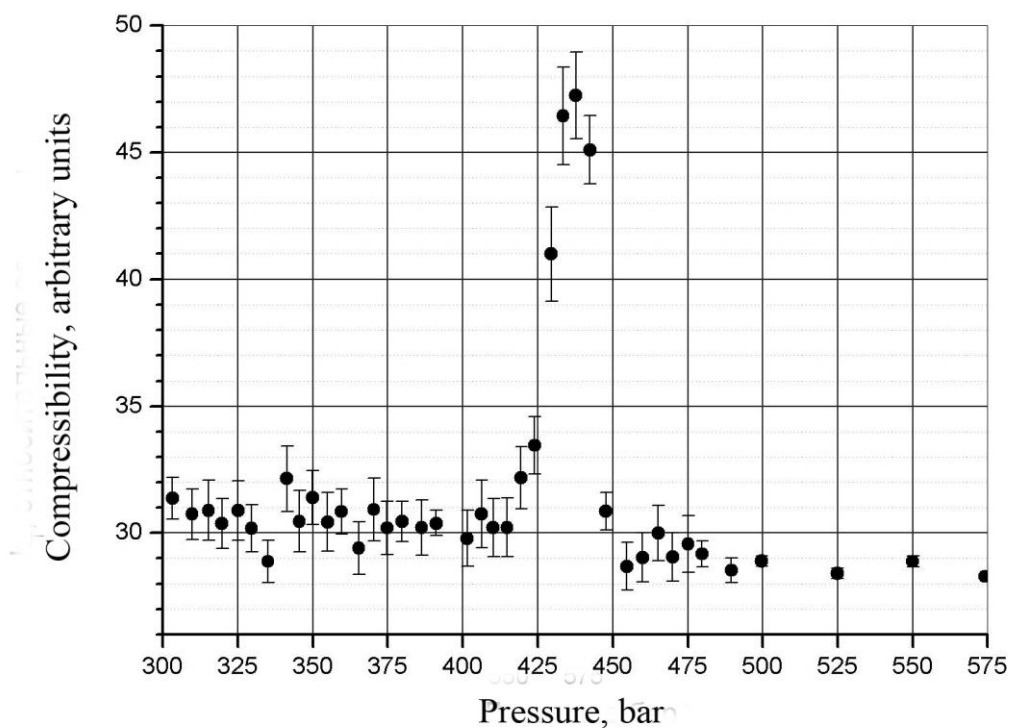


Figure 2. Compressibility of DMPC at 33.2°C as a function of pressure.

3. Magnetic field system

The magnetic field, like temperature or pressure, is one of the important parameters influencing the state of many materials. The applied magnetic field is tangible not only for magnetic materials but also in nonmagnetic ones such as paramagnetic and diamagnetic materials.

It is very important to control the microstructure of magnetic materials because textured or anisotropic magnetic materials usually show better performance in practical applications. Magnetic field processing is a well-proven technique to impose the desired texture and magnetic properties of magnetic materials. Macroscopic materials comprising a large number of nanostructures in crystalline alignment with one another would constitute highly anisotropic materials with wide potential applications. It has been also found that magnetic field can make inclusions migrate in a melt and align grains in fabrication processes for the magnetic and non-magnetic materials.

The “control of materials” for materials science by means of a magnetic field, during the materials synthesis, chemical reactions or crystal growth in high magnetic fields is useful method for creating and investigating new types of materials [13].

A new dimension in advanced materials (such as magnetic materials, oxide superconductors and organics) processing, and in the microstructure research of nanometer-scaled materials, spintronics, polymer, bio-chemistry and so on, through the possibility of SANS investigation in situ of the phenomena induced by applied magnetic fields has been discovered.

Other aspect of using magnetic field in small angle neutron scattering experiment refers to the possibility of determination of the nuclear and magnetic elastic scattering contributions to the scattering. When investigating magnetic materials in a SANS with nonpolarized neutrons experiment, measuring the scattering intensity without magnetic field and with a saturation magnetic field oriented perpendicular to the scattering momentum transfer, the nuclear and magnetic contributions can be differentiated [14-16]. Previously, this differentiation was possible to be obtained at SANS instrument only in the case of magnetic liquids by means of the contrast variation method [17-19].

For common goals the magnitude of magnetic field inducing changes in samples, (namely: anisotropy and orientation of magnetic domains in magnetic materials, as well as in the liquid crystal polymers [20]) is about few Tesla.

A new magnetic system for small angle neutron scattering at YUMO instrument was put into operation. The system includes about 3T (factory setup) electromagnet fixed on a two-axes goniometric table, power supply, cooling system and PC based control equipment. Main features of magnetic system are: big changeable gap for the samples (up to 130 mm size), computer controlled horizontal and vertical rotation and sufficiently big space for the sample holders [21, 22].

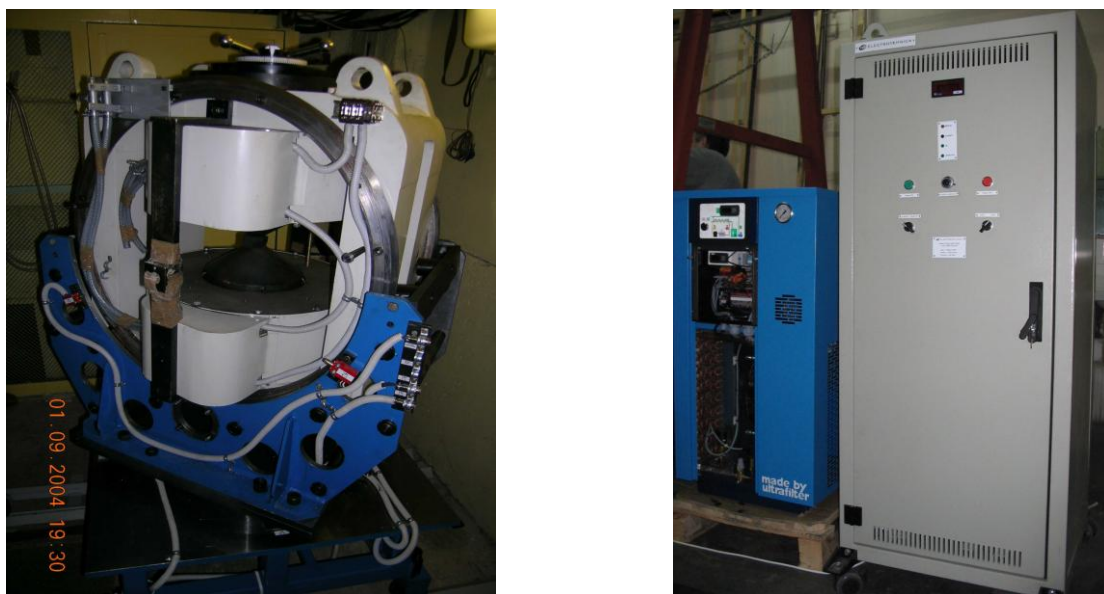


Figure 3. Electromagnet setup on a two-axes goniometric table (left); power supply and chiller system (right).

First experimental results of SANS in ferrofluids, magnetic elastomers, ferromagnetic powder have been obtained at YUMO spectrometer equipped with the new magnetic system.

4. Illumination of the samples

A sample in a SANS experiment is located in the air gap between neutron guide and detector tube. Thus there is a possibility to illuminate a sample with a laser beam or a special light beam. The YuMO system comprises the cold source of continuous high-intensity visible light (Schott KL 1500 electronic (SCHOTT Glas)). Light is guided to the object using flexible movable light guides. Taking an advantage of this light source system, functional structural changes of a membrane protein - bacteriorhodopsin (BR) have been investigated [23].

BR absorbing a photon uses its energy to transport protons H^+ through the membrane in which BR is located. It results in electro-chemical gradient on the membrane. This first step of accumulation of energy (creation of electro-chemical gradient by proton pumping) is an universal step in energy transformation in bacteria, plants and animals. BR is widely used for investigation of mechanism of proton pumping. A major question to be answered is whether protein conformational changes are involved in proton pumping and what is their amplitude. The SANS experiments revealed a substantial difference between the thickness of purple membrane (a part of bacterial membrane containing solely BR) dark-adapted ($T=50.3 \text{ \AA}$) and exposed to continuous illumination ($T=44.7 \text{ \AA}$). The reduction of the membrane thickness triggered by light illumination most probably is connected to structural changes of BR molecule. Difference of the scattering intensities of light illuminated and nonilluminated purple membranes BR is presented in figure 4.

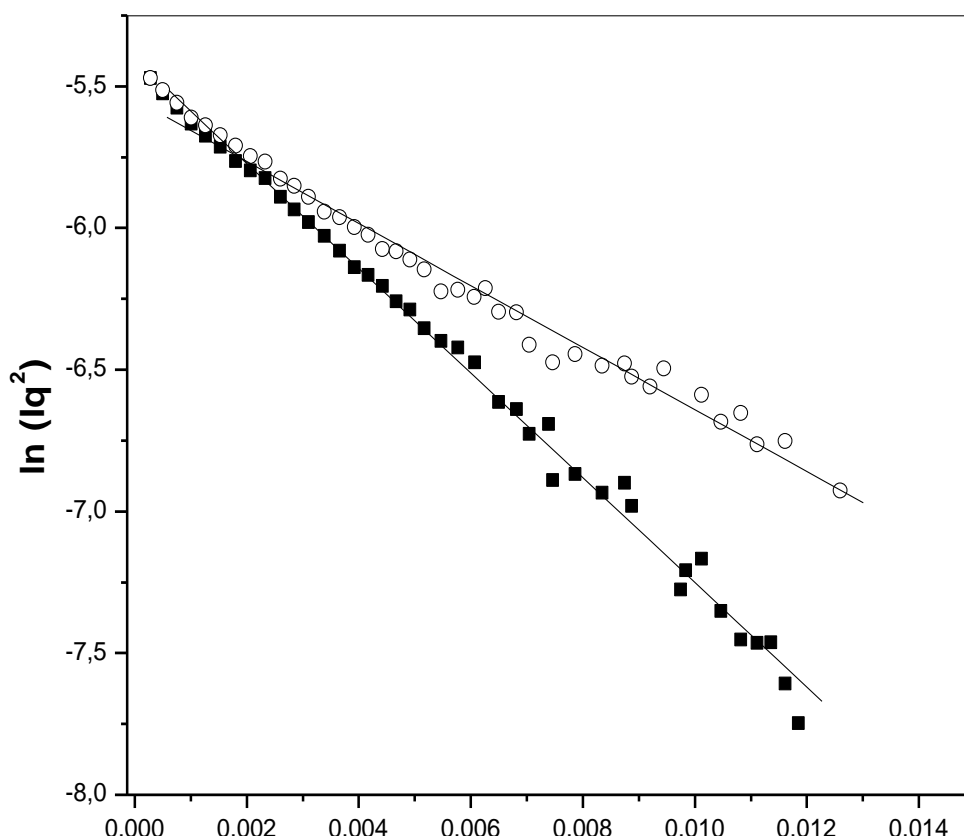


Figure 4. Dependence of the scattering intensity of PM in D₂O at T=16.8 °C on the scattering vector in $\ln(I(q)q^2)$ vs. q^2 plot: (■) dark-adapted state; (○) upon illumination.

5. Conclusions

The modernized SANS instrument (YuMO) was equipped with a new type of position sensitive detector as well as two detector system which provide a unique dynamic range (Q_{\max}/Q_{\min} ratio is about 90).

Sample environment was extended with a magnetic system (magnetic field about 3 Tesla), automated high pressure setup which allows simultaneous SANS and volumetric high pressure studies and light illumination system.

In particular, these developments led to considerable improvements of resolution of the instrument (about 1%), opened possibility to study anisotropic materials and perform efficient high pressure studies.

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