Latest developments in laboratory SAXS/WAXS instruments

S. Rodrigues¹*, P. Panine¹, S. Desvergne¹, P. Høghøj¹, R. Mahé¹ and F. Bossan¹

¹Xenocs, 19 rue François Blumet, F-38360 Sassenage, France

Keywords: laboratory SAXS/WAXS, instrumentation, in-situ studies, macromolecules, block copolymers

*e-mail: sergio.rodrigues@xenocs.com

In parallel to the advent of dedicated synchrotron radiation sources and beamlines several breakthroughs have been accomplished for laboratory analytical X-ray instrumentation and in particular for Small Angle X-ray Scattering (SAXS) instrumentation. Breakthroughs include X-ray micro-focus sources with aspheric multilayer coated optics, scatterless collimation [1], sample environment, software and hybrid pixel photon counting detectors as well as instrument design with for example multiple source energy capability [2].

Today, these technologies combine to provide inlaboratory SAXS instruments, with a performance comparable to that previously achieved only at synchrotrons. Flexible instrument designs provide simultaneous measurement of Wide Angle X-ray Scattering (WAXS) signal in various sample forms (including thin film) and experimental conditions. The state-of-the-art performance opens the way for a wide range of applications, including scattering from soft matter. Performance and possibilities will be illustrated through a few application examples such as characterization of highly diluted macromolecules or in-situ dynamic studies of complex soft materials.

Study of 3D protein enveloppes with laboratory SAXS is now widely used as a complementary technique to electron microscopy and NMR. Different high brilliance sources technologies can be used to study highly diluted proteins including large macromolecular complexes. Data modelling of various proteins will be presented and compared including with synchrotron data.. Performance is now at a level where traditional sample holders such as capillaries, could be a limiting factor, overcome by innovative flow cells.

Physical behavior of soft materials is often complex and couples to environmental conditions such as stress and temperature. Studying the structure variations upon external fields over 2 or 3 decades in real space, i.e. from atomic scale up to the mesoscale is thus particularly useful in understanding complex materials. If fast transitions require intense flux of third generation synchrotrons, slow phenomena can be studied fruitfully in laboratory systems, offering in one hand less instantaneous flux but with unrivalled availability of beam time. Among others, this will be illustrated by the SAXS/WAXS kinetic study of washing powder exposed to moisture. Moreover fast transition studies are also possible with laboratory SAXS/WAXS systems with relative exposure times which match the standard measurement conditions used in Differential Scanning Calorimetry (DSC) of few degrees per minute for instance. Capability to measure simultaneously nanoscale structure and crystalline features during in-situ studies such as temperature controlled measurements on copolymer samples will be illustrated.

[2] S. Koppoju, et al., J. Appl. Cryst. 48 (2015) 2040.

^[1] Y.Li et al., J. Appl. Cryst. 41 (2008) 1134.