

50 years of D11

A history of SANS
at the ILL



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Multiscale micro-architecture of pore space in rocks: size, shape, deformation and accessibility (remote)

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The interface between rock matrix and pore space in sedimentary rocks is rough over seven orders of magnitude of the linear scale, from sub-nanometers (nearly molecular) to centimeters. Since the early 1980's, pore-matrix roughness has often been described using the framework of Mandelbrot's fractal geometry and the corresponding mathematical formalism for correlation functions; this formalism has been applied to successfully model the power-law SANS and SAXS results for many types of sedimentary rocks (e.g. sandstones, shale, carbonates and coal). P.W. Schmidt first established the connection between pore-size distribution (as an alternative expression of roughness) and the power-law dependence of small-angle scattering intensity, and within a decade, the vast world of fractals in natural porous media was revealed.

Small-angle scattering is uniquely suited to microstructural geological applications, given its capacity to provide volume-average nano- and microstructural information, and its sensitivity to the chemical composition of pore content. In the early days, the crucial issue was the extent of the Q-range (hence the pore sizes). The capacity to study pore sizes was limited by the small-Q limit of the longest-base SANS instrument D11; this range was then greatly improved in 1997, following the construction of a Bonse-Hart type USANS instrument by M. Agamalian. Since that time, pore sizes ranging from sub-nanometers to approximately 20 micrometers can be investigated using SANS-USANS. The capability of long base SANS instruments using wavelengths of ca. 5 Å to provide overlap with USANS data in the region around $Q \approx 10^{-3} \text{ Å}^{-1}$ has been crucial to the elimination of multiple scattering, a problem specific to strong scatterers (like most rocks), and inescapable for the modern lens-geometry and TOF SANS instruments. Technical developments resulting in low background noise of modern SANS detectors have enabled precise insight into the nano- and sub-nanometer scale regions, which led to the discovery of the ubiquitous phenomenon of gas condensation in the nano-pores of shales and carbonates. Similar results have been obtained for coal and aerogels.

A seminal step occurred with the gradual development of SANS and USANS contrast matching capability, delivered by high pressure gas environmental cells, which offered significant progress compared to the use of deuterated liquids in earlier contrast matching experiments. This made it possible to independently characterise the total porosity, specific surface area and pore size distribution for porous spaces that are accessible and inaccessible to penetrating fluids (greenhouse gases in particular). Significantly, for a great majority of measured rocks, the roughness of the matrix - pore interface of the accessible pores turned out to be less accentuated than that of the inaccessible pores. This provided an interesting insight into the long-standing fundamental question of the origin and temporal persistence of porosity in rocks; in addition to the antisintering mechanism proposed by M. Cohen, the reactive transport of brine through the rock matrix likely plays a significant role. The recently added uniaxial stress capability enabled SANS and USANS measurements under simulated pressure conditions encountered in unconventional shales subjected to hydraulic fracturing. The results showed that the porosity response to simulated well-management is complex: it is both pore-size-dependent and thermal-maturity dependent. Armed with these capabilities, small angle neutron scattering has become a mainstream tool in petrology and geology on the nano- and microscale, applied in tandem with electron microscopy, gas adsorption measurements, mercury intrusion porosimetry and SAXS-USAXS.

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