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28 years of nano-pore analysis by NMR Cryoporometry : background, capabilities, comparison with other techniques, instrumentation & protocols, and recent developments

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Abstract :

NMR Cryoporometry (NMRC) is a powerful technique for the measurement of pore-size distributions and total porosities on a pore length scale from sub 1nm to over 1micron. This technique is suitable for measuring poresizes in a wide range of polymers and porous materials, including porous glass, rock, clays and porous carbons including biochar. It offers various advantages over other techniques, including the ability to study wet samples. By swelling rubbers and polymers with added organic liquids cross-link density and nano- to micro-porous properties of the polymer may be obtained. In biochar, progressive changes to the quantity and mobility of hydrocarbons, as well as changes in pore-blocking, as a function of preparation temperature, have been demonstrated. The capabilities of NMRC have in recent years been extended in a number of directions, to greater sensitivity, to sub-nanometric pore sizes (lower temperatures) and to above micron sized pore-sizes (tiny melting point depressions). NMRC has been used to probe the effect of using different probe liquids on measured pore volume. in the nearly 30 years that Lab-Tools have been developing NMRC, the protocols have evolved. The data formats have been formalised, and the programs to access and display the NMRC control variables and results data have been optimised. Some of these protocols are now encoded into Graphical User Interface (GUIs) and their associated process and graph windows. As part of the evolution of NMRC, Lab-Tools have developed a highly compact precision NMR time-domain relaxation spectrometer, based on a Field Programmable Gate array (FPGA) module, with associated Peltier thermo-electrically cooled variable temperature probe, which together make a high-performance NMR Cryoporometry instrument. A high proportion of the R.F. circuitry in a digital form, implemented as firmware in the FPGA. The FPGA module is credit-card sized, and the NMR receiver and NMR transmitter are each even smaller. The advantage of using the Peltier cooling is that one obtains the precision temperature control and smoothness needed by NMR Cryoporometry, particularly near the probe liquid bulk melting point. This enables the NMRC measurement of pore diameters in excess of 1 micron. The measurement protocols that have been developed will be outlined. Complete with a Graphical User Interface (GUI) for control and on-line analysis, this precision instrument is particularly suitable for material science studies both in the field and in university, research institute, company and even school laboratories. A range of international companies, universities and research institutes are now using NMRC as part of their arsenal of research tools to study their samples

Biography:

Beau Webber gained his PhD at the University of Kent, UK (thesis : "Characterising Porous Media"). He has published 50 papers in refereed journals, and is a called upon referee. He makes use of a wide range of measurement techniques for studying porous materials and liquids contained in them and has made extensive use of Central Facilities neutron scattering instrumentation at Grenoble, Paris and Abingdon, for these studies. He is director of Lab-Tools (nano-science) Ltd. a small UK spin-off research laboratory that performs academic and commercial contract nano- to meso- materials-science research, studying the structure, dynamics and phases of liquids and their solids (and also gas hydrates) in confined geometry and at and near surfaces.

Note: - This work is partly presented at Webinar on Nano-Engineering and Its Applications on July 22, 2020.