Unilateral NMR of Activated Carbon

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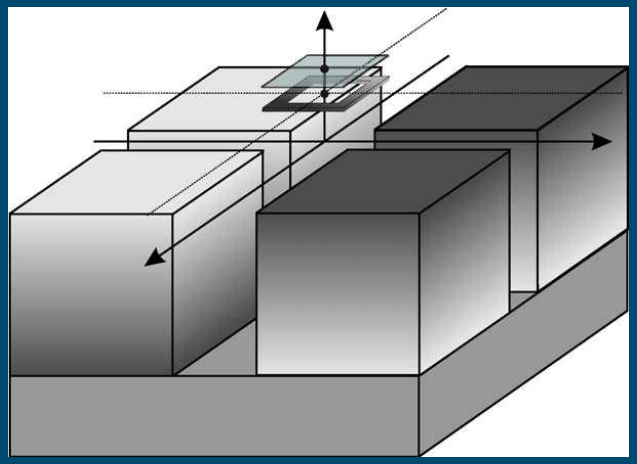
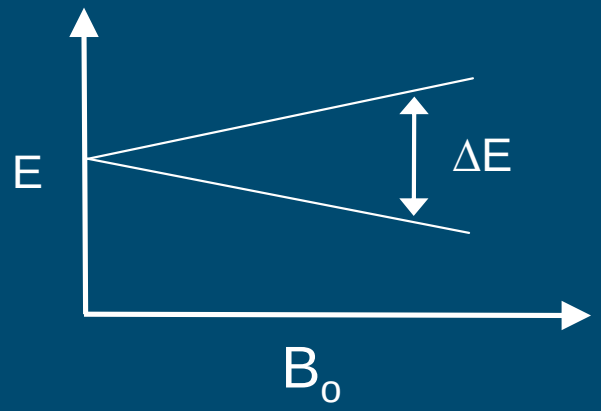
Background- Unilateral NMR

- The measurements are done on an object placed on top of the NMR Mouse®.
- The NMR Mouse® is designed to project a sensitive area outside the instrument that can be probed.
- Therefore with this design, experiments are performed outside rather than within the instrument.
- The polarising magnets assembly and the RF coil has the shape of a box (13 x 11 x 10 cm^3).

[1]: D.G. Rata, F. Casanova, J. Perlo, D.E. Demco, B. Blümich *, JOURNAL OF MAGNETIC RESONANCE, 180 (2006) 229–235

Suitability of the NMR Mouse®

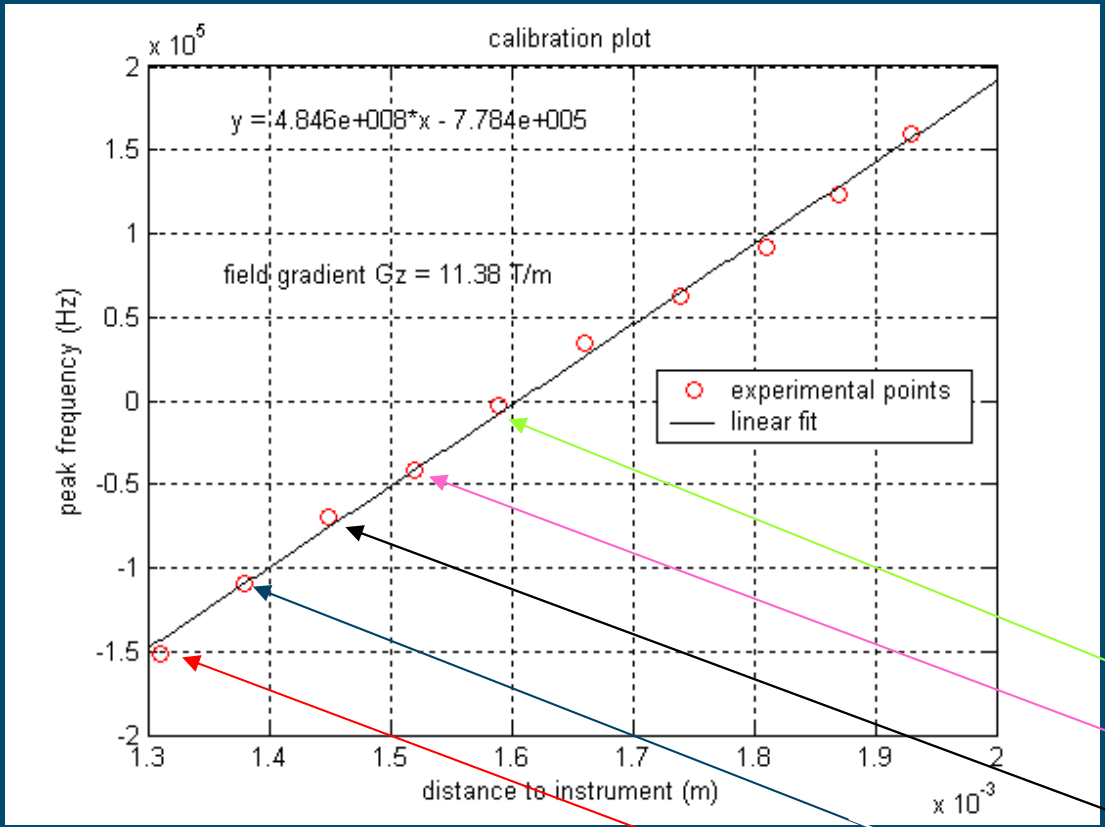




- The magnet geometry generates a flat sensitive volume of an adjustable size of approximately 1.5 cm^2 and 0.6 mm thick at 2-10mm away from the instrument.
- A radio frequency field coil excites and detects the proton nuclear spins.
  - The measurement essentially yields; the proton density
  - the relaxation rate of the nuclear spins.

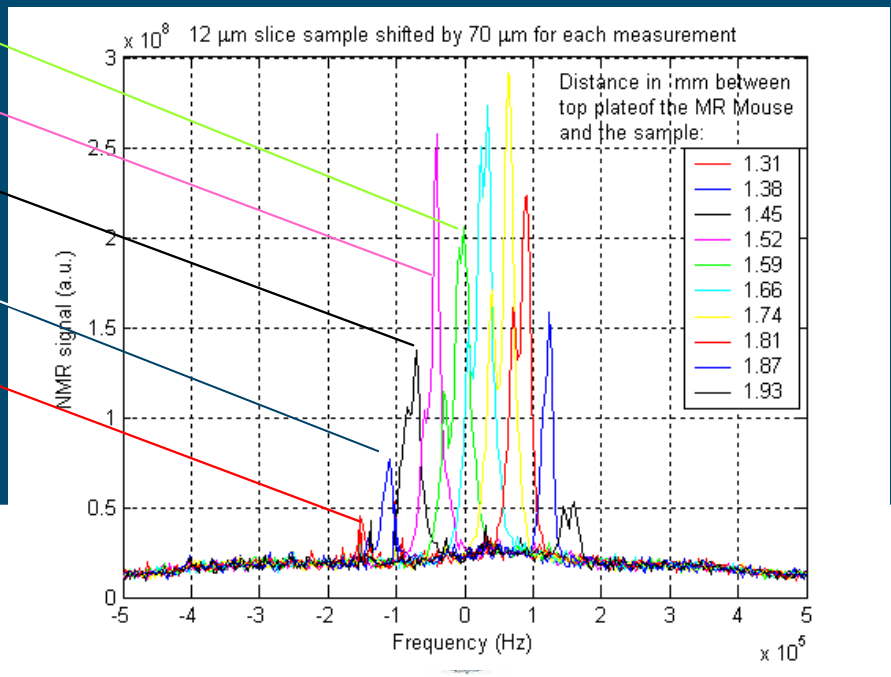
[1] **J.Perlo,** JOURNAL OF MAGNETIC RESONANCE 176 (1): 64-70 SEP (2005)
[2] ACT, Aachen, Germany.

Field Gradient Effect



The z-coordinate of a 12 µm thin film of sandwiched oil between two microscopes slides was incrementally changed by interleaving a gradually increasing stack of 70 µm thick tracing paper sheets.

Excellent linearity is found resulting in an approx Gaussian profile.



Larmor frequency:

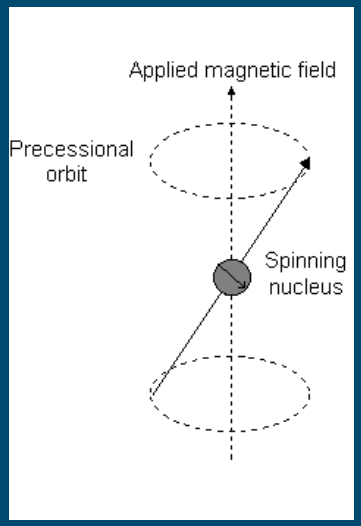
$\omega = \gamma B_0$

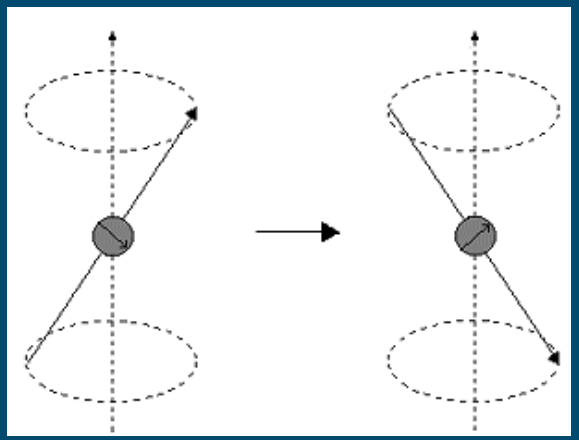
$2\pi\delta f = \gamma G_z \delta$

$G_z = (2\pi/2.67522 \times 10^8)(4.846 \times 10^8)$

$G_z = 11.38\ T.m^{-1}$

Information from Relaxation Rates

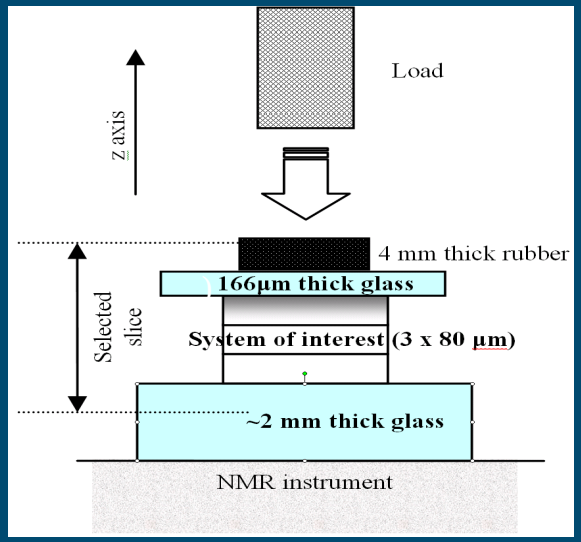


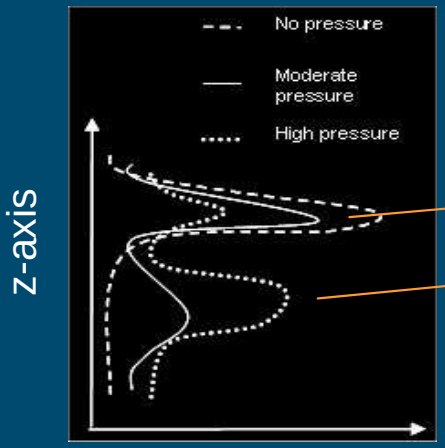


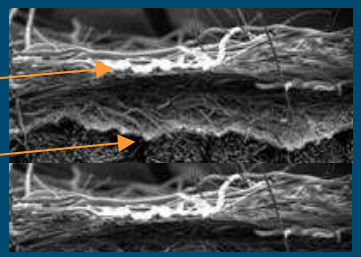
- Nuclei in the higher energy state return to the lower state is by the relaxation process
  - Spin - lattice (longitudinal) relaxation
  - Spin - spin (transverse) relaxation
- **Spin - lattice relaxation** T_1
- **Spin - spin relaxation** T_2
- CPMG sequence used to collect NMR data. A profile is obtained by fitting an exponential decay for each pixel to get the local T_{2eff} and the local NMR signal amplitude.

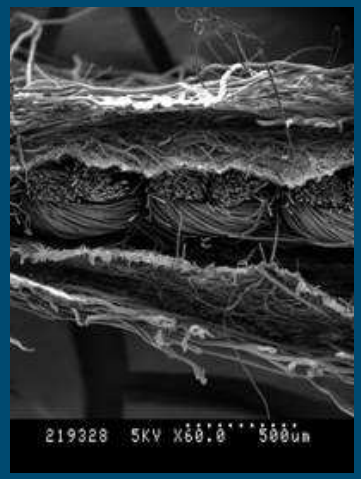
http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/nmr1.htm

Assessment of Textile Substrates



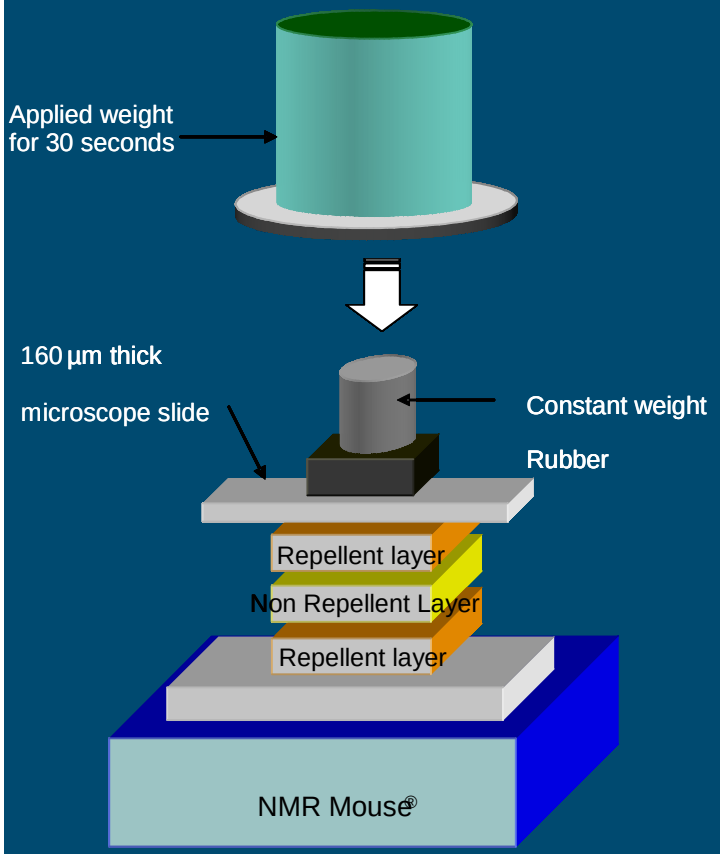


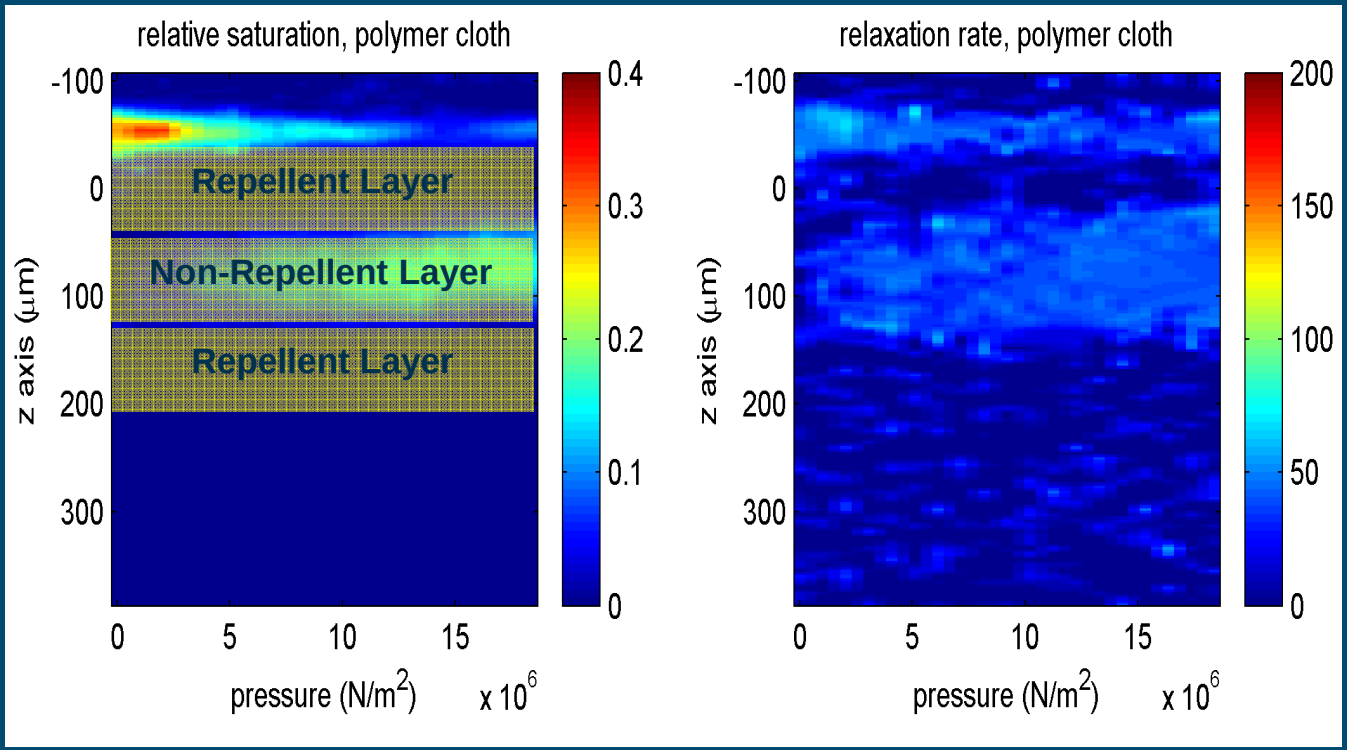




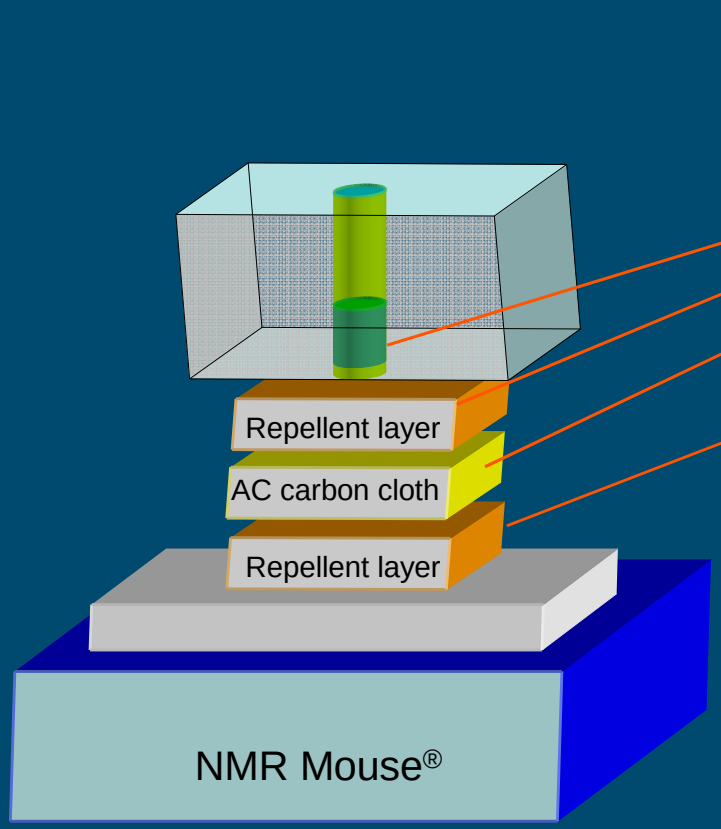
- Textiles that provide protection against toxic chemicals need to prevent the ingress of aerosols, vapours and liquids
  - Aerosols: particle capture
  - Liquid: repellent and wicking layers
  - Vapour: activated carbon
- Test methods that image a textile's performance are desirable

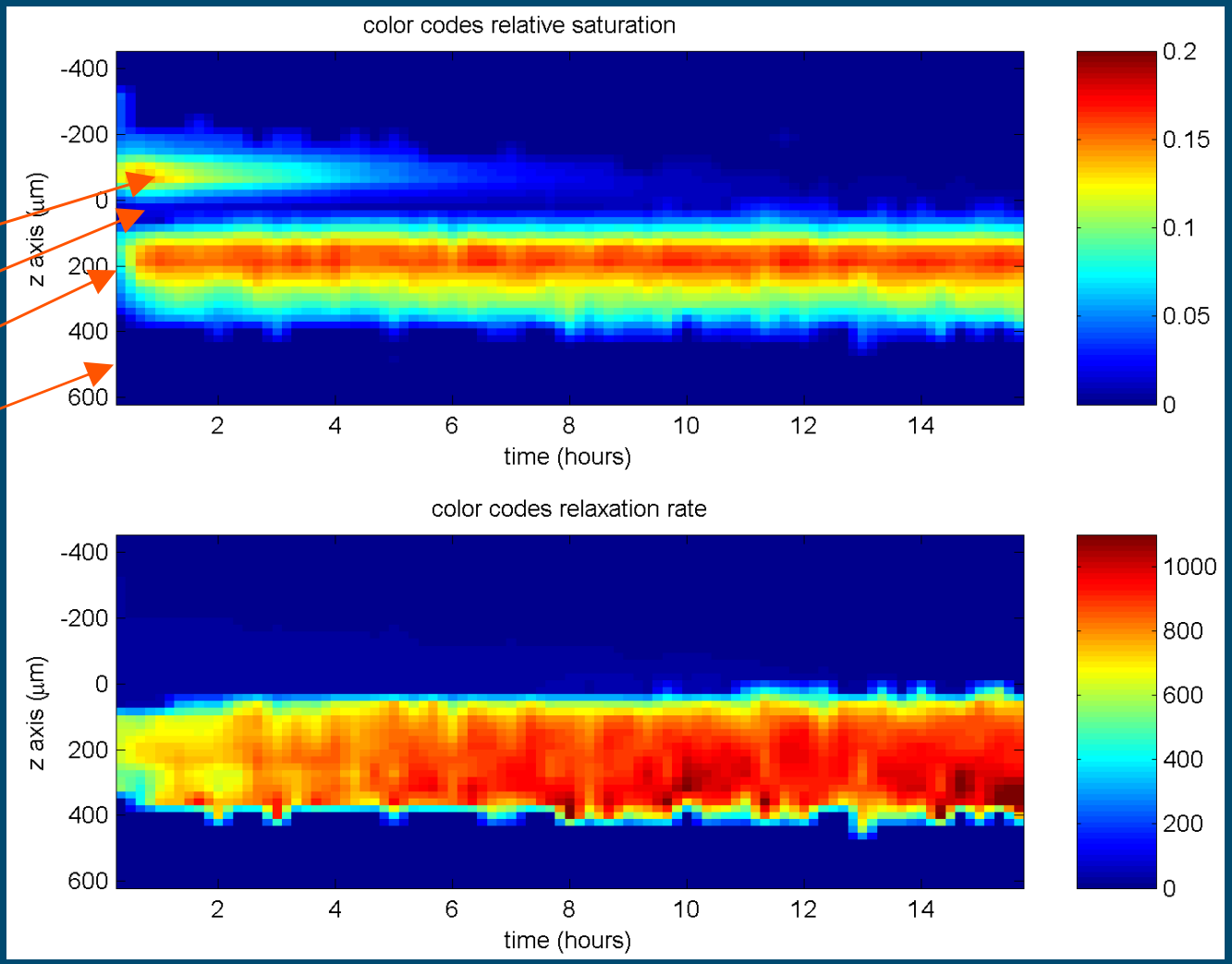
Spatially resolved measurement





Spatially resolved measurement of Vapour uptake through a repellent layer





Different types of Activated Carbon (AC) for this study

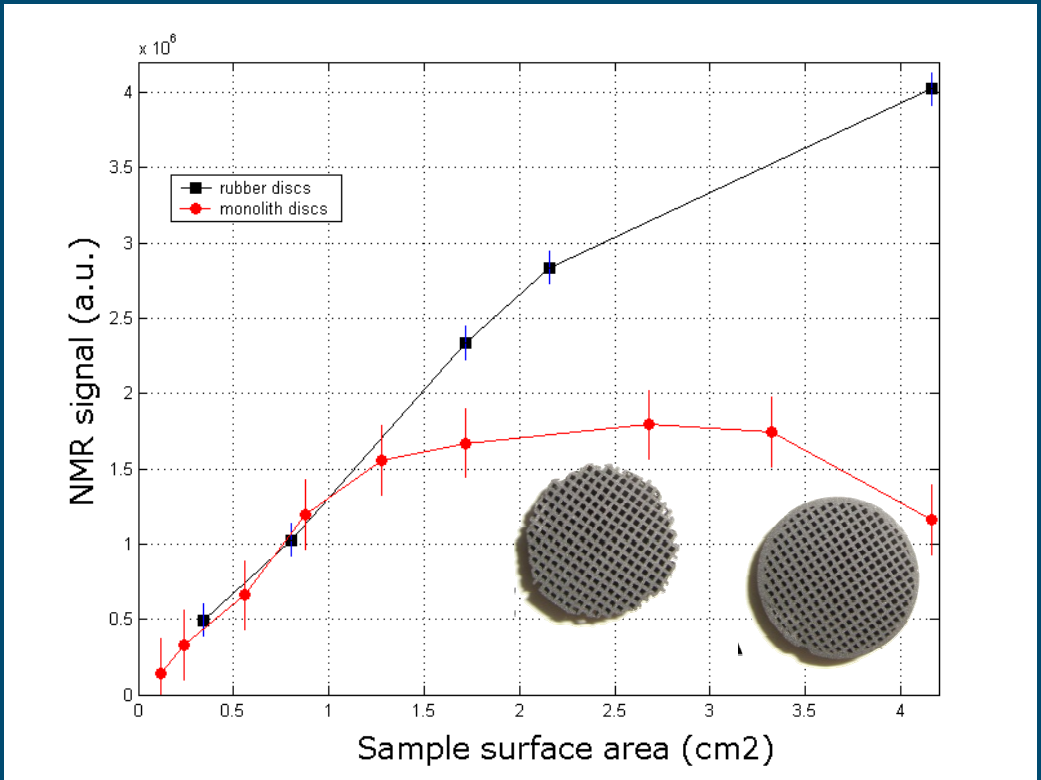
AC cloth

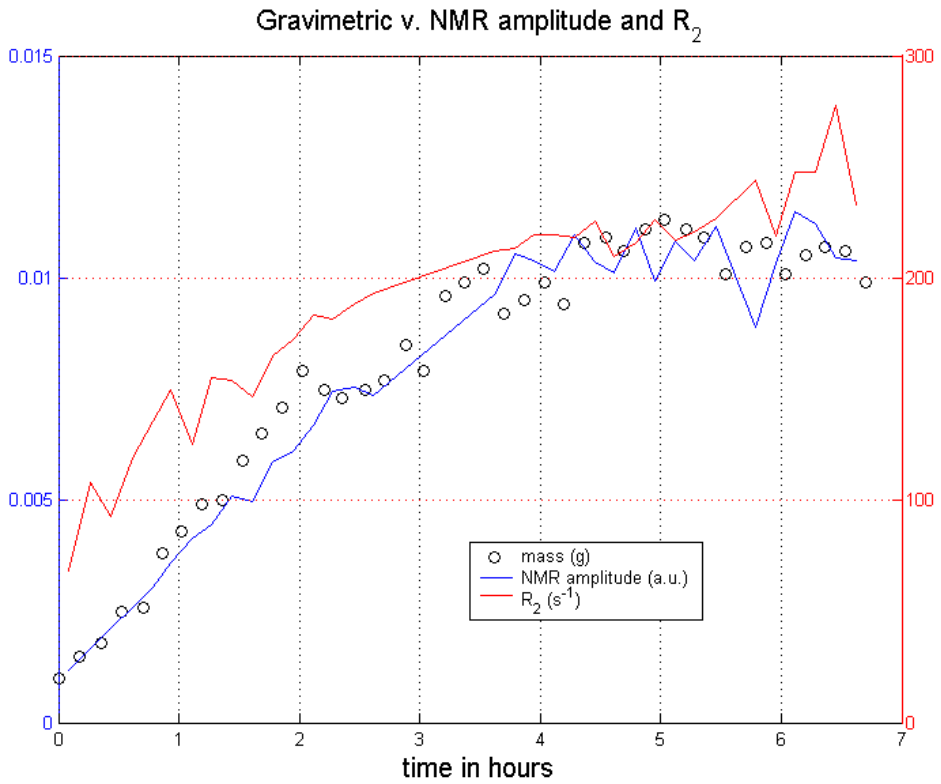
AC monolith:
Is made from pure carbon extruded.

AC composite

Conductivity and its Affect On The NMR Amplitude

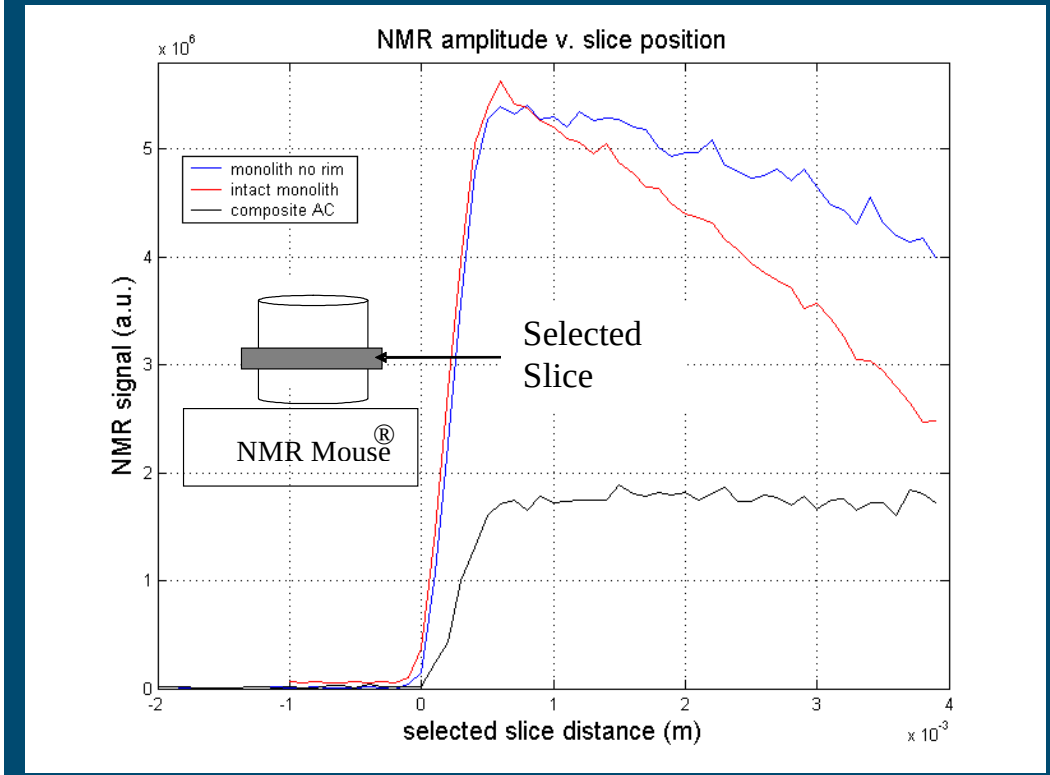
- Graph showing signal from rubber discs v. monolith discs as a function of disc area
- Graph of gravimetric v. NMR amplitude for the thin sample

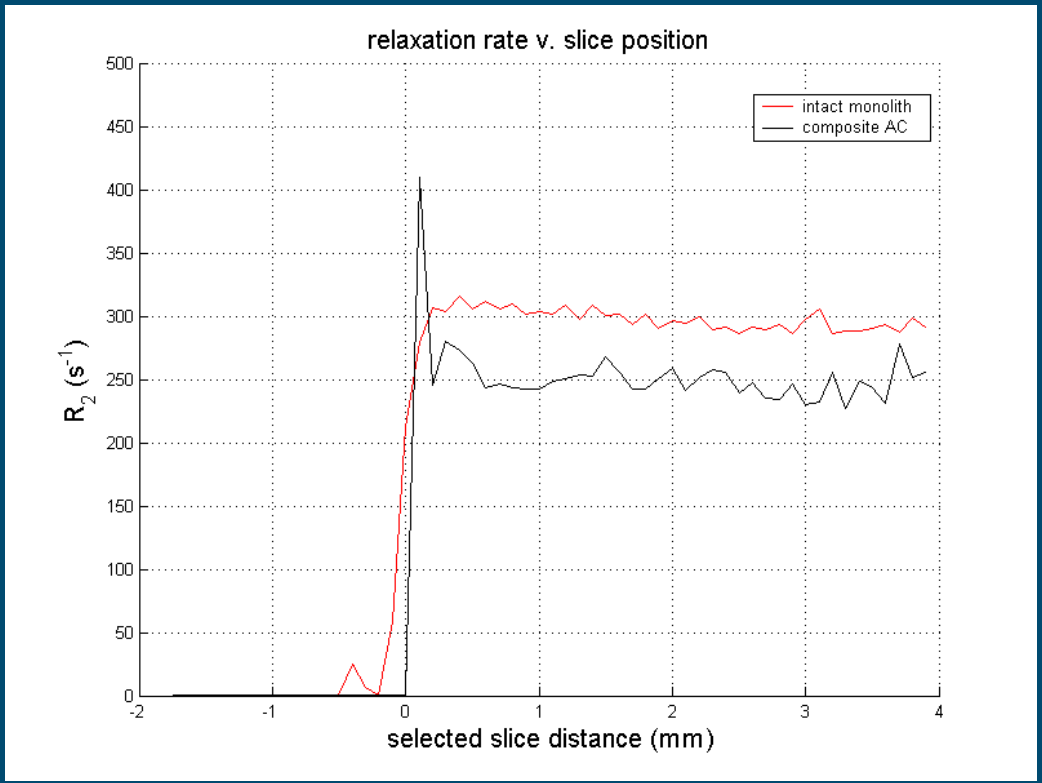




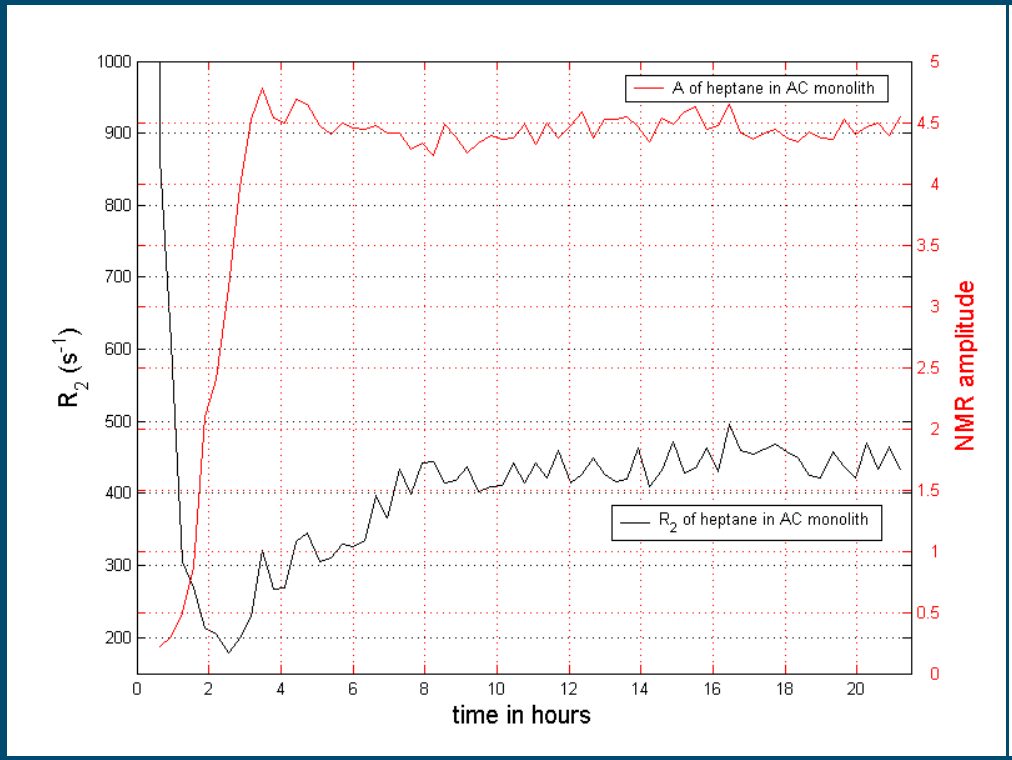
Quantification of the NMR Signal Intensity as a Function of Sample Diameter

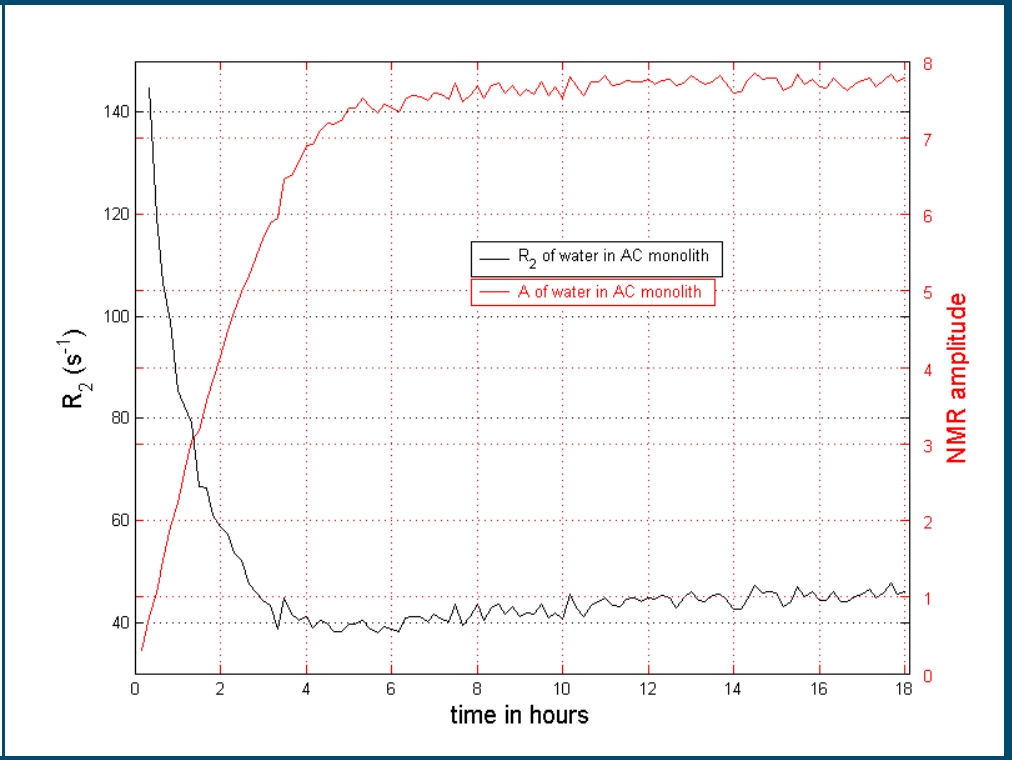
- Graph of the NMR amplitude for heptane-saturated AC, as a function of depth
- Variation of T2 with z at equilibrium





Relaxation rate variation with Time For Water and Heptane Adsorption (pore filling?)





Conclusions

- Unilateral NMR is capable of imaging both liquid ingress and vapour adsorption in a multi-layered fabric.
- The instrument provides information on the quantity of liquid or vapour present in a x-y plane.
- Depth profiling (z- direction) is also achieved without the need to move the sample physically.
- In spite of the high conductivity of carbon substrates, the unilateral NMR instrument is able to monitor vapour uptake in dense activated carbons.
- The time course of the relaxation rate clearly exhibits two different regimes, a rapid and early decrease corresponding to pore filling, followed by a slower rise that we do not understand yet.
- Unilateral NMR is a simple and relatively inexpensive instrument that provides very useful insight in the uptake of molecules in a porous material under standard test regimes.

Acknowledgements