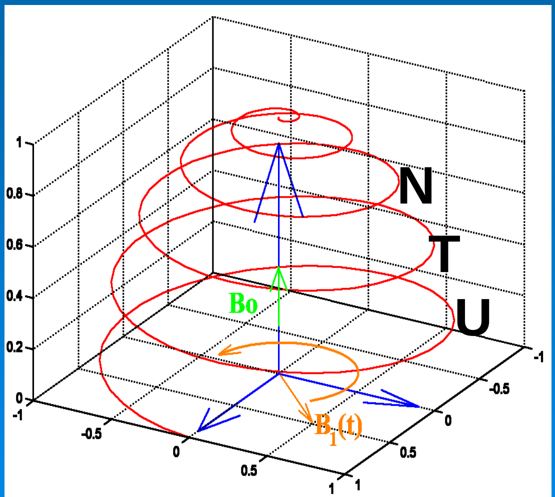
Quantitative monitoring of liquid ingress in heterogeneous layered materials using a Mobile Universal Surface Explorer (MOUSE®)

Hans Adriaensen[1],
Martin Bencsik[1], Stuart Brewer[2], Glen McHale[1]

[1]: Nottingham Trent University (NTU), UK
[2]: Defence Science and Technology Laboratory (DSTL), UK



Presentation for the 9th International Conference on Magnetic Resonance Microscopy

4th September 2007

Work Content

1. A brief overview of the porous medium

- Engineering repellent fabric
- Porosity of the fabric
- Application of the work
- Coating effect

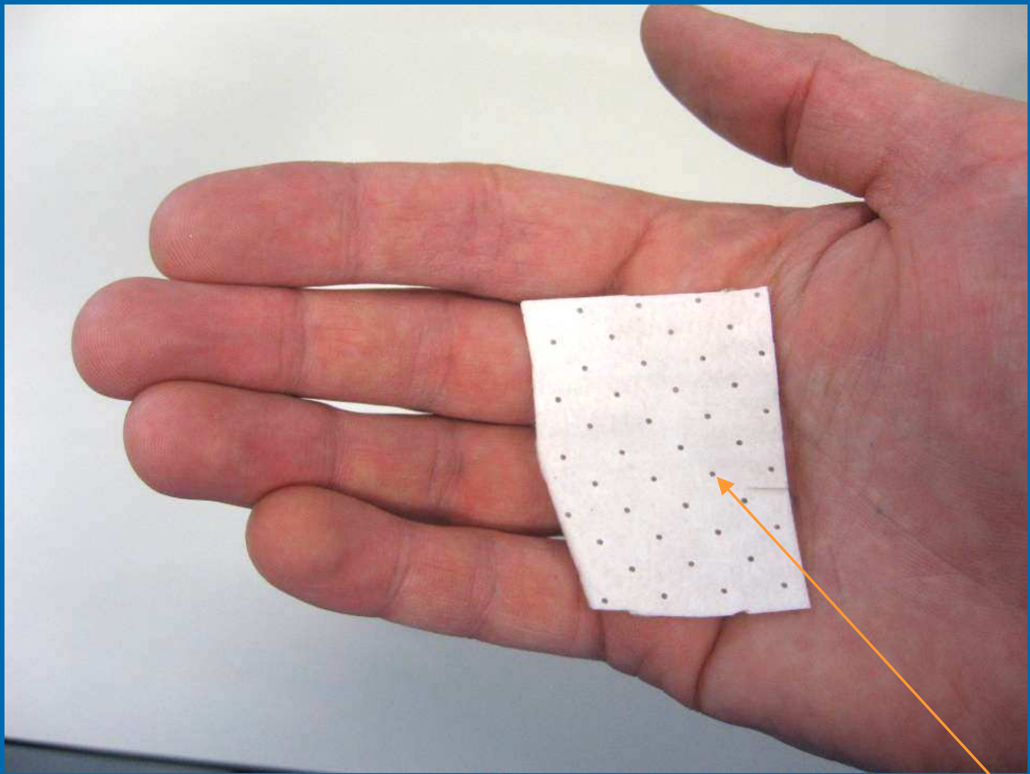
2. NMR Study on layered fabric

- Suitability of the *profile* NMR Mouse® for this study
- Experimental protocol: liquid ingress into the fabric under vertical pressure
- CPMG sequence
- Field gradient

3. Exploration of the porous medium

- Fate of oil after applied pressure
- Conservation of the liquid in the system
- Repeatability
- Conclusion and further work

Engineering repellent fabric



A 400 µm thick fabric, welded with dots to hold 5 different layers together.

A fabric is made repellent by the Defence Science and Technology Laboratory (DSTL), UK with a deposition of a thin fluoro polymer coating.

To render a textile liquid repellent, a silicone or fluorocarbon coating is applied [1,2,3,4,5].

One 80 µm thick fabric layer is made of entangled individual polymer fibres.

The welding dots are placed in squares (edge: 5 mm).

[1]: E. Kissa, In *Handbook of Fibre Science and Technology*; edited by M. Lewin, S. B. Sello (Marcel and Dekker Inc. New York, 1984).
[2]: S.R. Coulson, I.S. Woodward, J.P.S. Badyal, S.A. Brewer,C. Willis, J. Phys Chem B **104** (37): 8836-8840 21 (2000).
[3]: S.R. Coulson, I.S. Woodward, J.P.S. Badyal, S.A. Brewer,C. Willis, *Langmuir* **16** (15): 6287-6293 25 (2000).
[4]: S.R. Coulson, I.S. Woodward, J.P.S. Badyal, S.A. Brewer,C. Willis, Chem Materials **12** (7): 2031-2038 (2000).
[5]: *Adv. Mater.* **2006**, *18*, 3063–3078 *Xinjian Feng* and *Lei Jiang*.

Application of the work

Design of fabrics that are strongly protective against specific liquids for individuals and for materials.

For the design of the textile, it is crucial to know how does liquid interact with it.

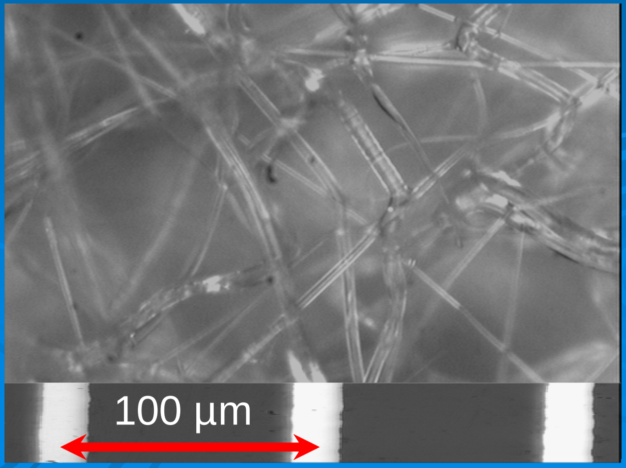
The best combination of layered fabrics for strong repellency needs to be worked out.

Some fabrics are structural, some are not.

We combine three 80 µm thick layers of non-repellent and repellent fabric of non-woven material (17 gm^{-2}).

Each layer of fabric is composed of meltblown polypropylene fibres.

The porosity of a non repellent fabric is around 80 %, but the porosity for a repellent layer remains difficult to measure as it is not wettable.



Entangled fibres from a repellent layer seen through an optical microscope.

Effect of coating

Deposition of 7 µL of oil on a non-repellent layer

Deposition of 7 µL of oil on a repellent layer

Suitability of the NMR Mouse® for this study.

***Profile* MR Mouse® (on the RHS)[2].**
The polarising magnets assembly and the rf coil has the shape of a box (13 x 11 x 10 cm³).

By stacking three fabrics one on top of the other, a 240 µm thick sample is generated.

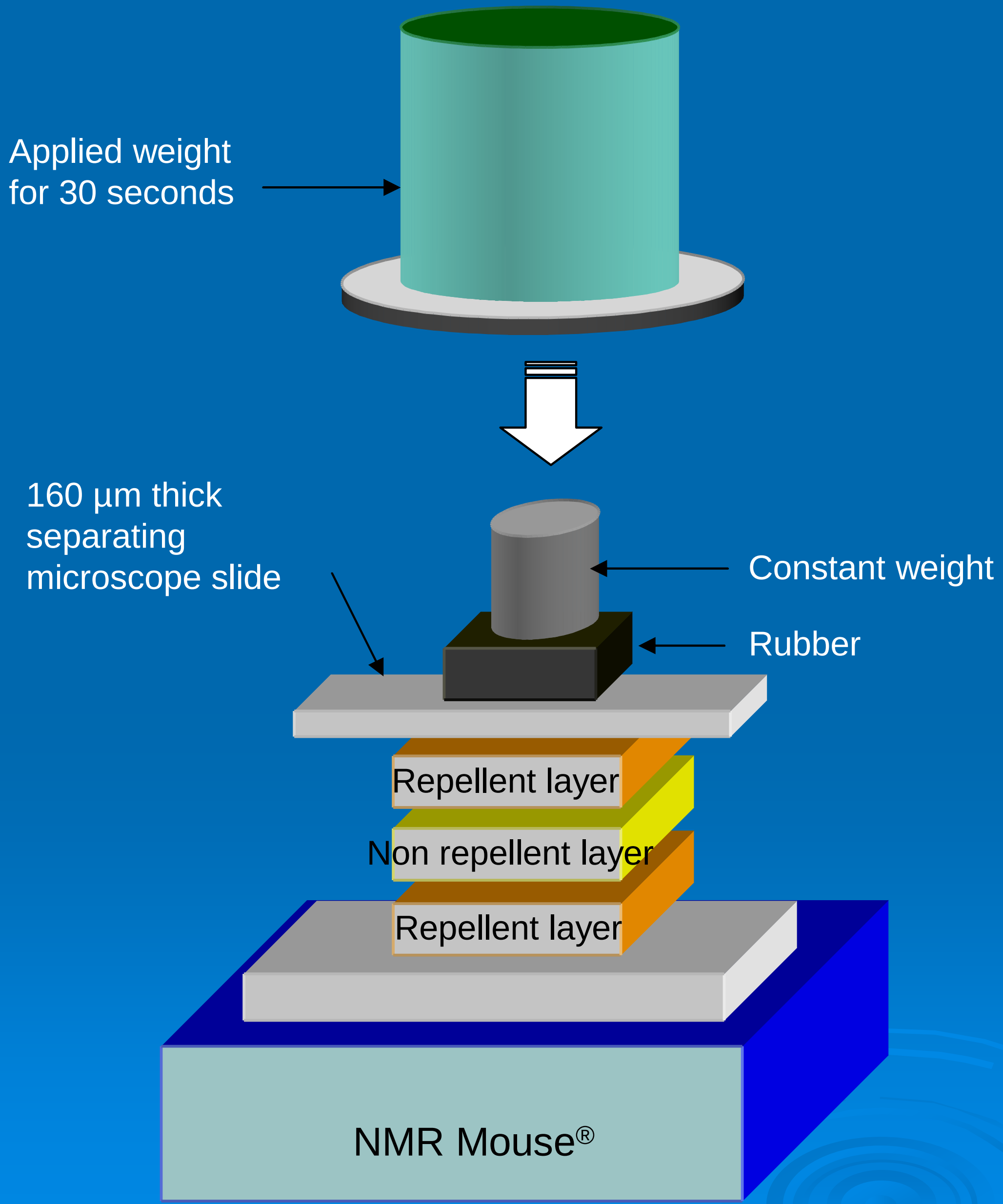
The *profile* MR Mouse®[1] allows to monitor the ingress of liquid through the layered fabric with a non-invasive and non-destructive technique.

The MR Mouse generates a rectangle parallelepiped volume of sensitivity of (500 µm x 20 mm x 20 mm) at 5 mm above the instrument.

For spatial resolution, the spin echoes can be fourier transformed rather than physically moving the instrument relative to the sample.

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

Experimental set-up

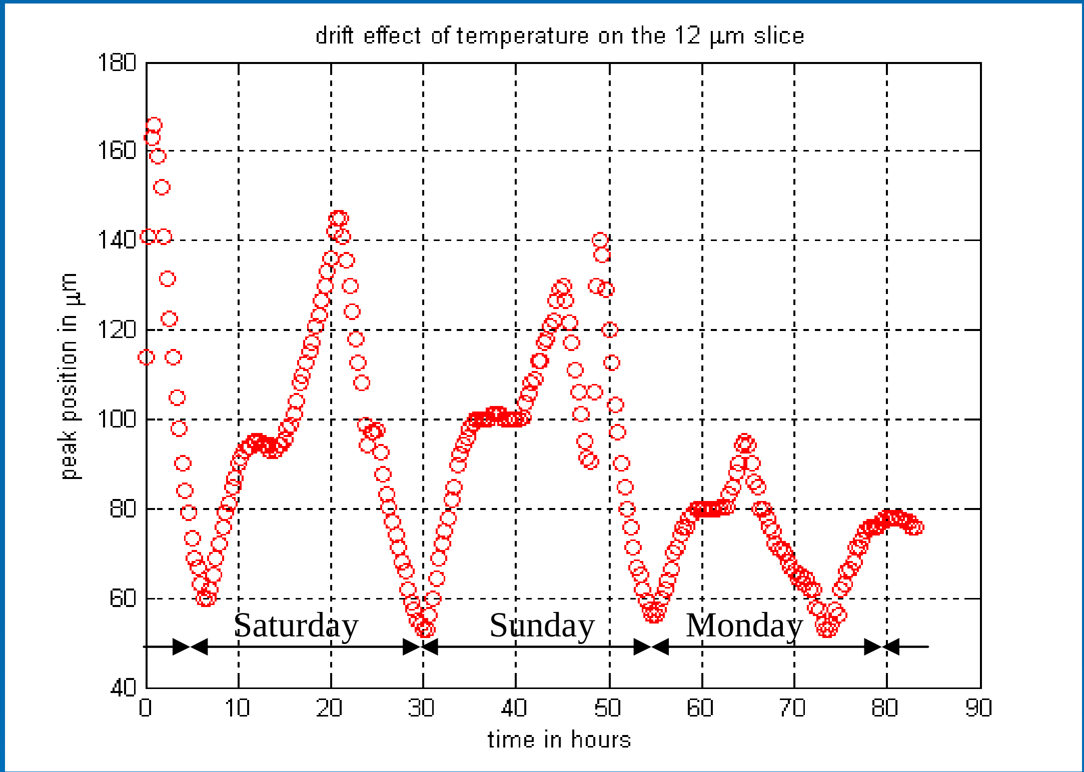


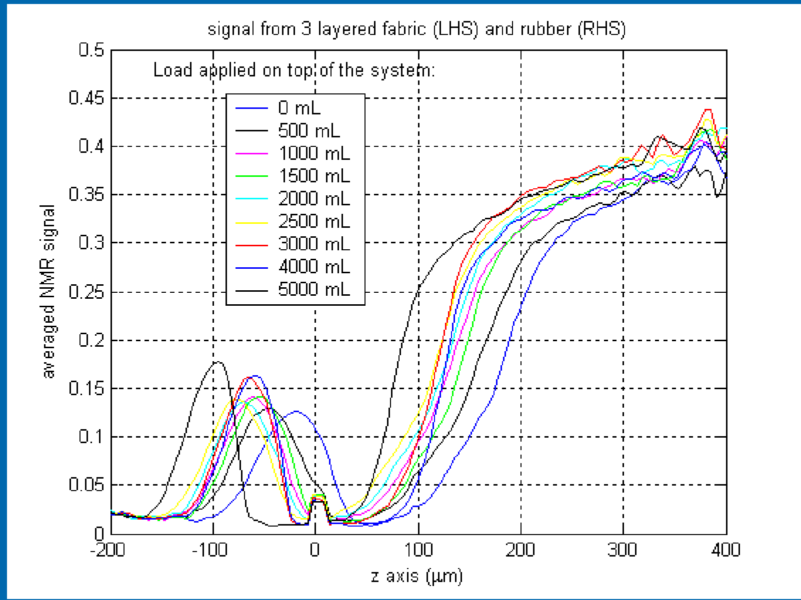
Protocol:

- deposit 7µL of liquid on top of the system.
- apply a known pressure for a constant time of 30 sec.
- acquire NMR signal over 4 minutes.
- increase the load applied on top of the system by 60 g.
- acquire a new NMR profile.

A 4 mm thick sample of rubber is placed above the separating cover slip to provide a static reference for the z-position.

3 reasons to have the rubber reference

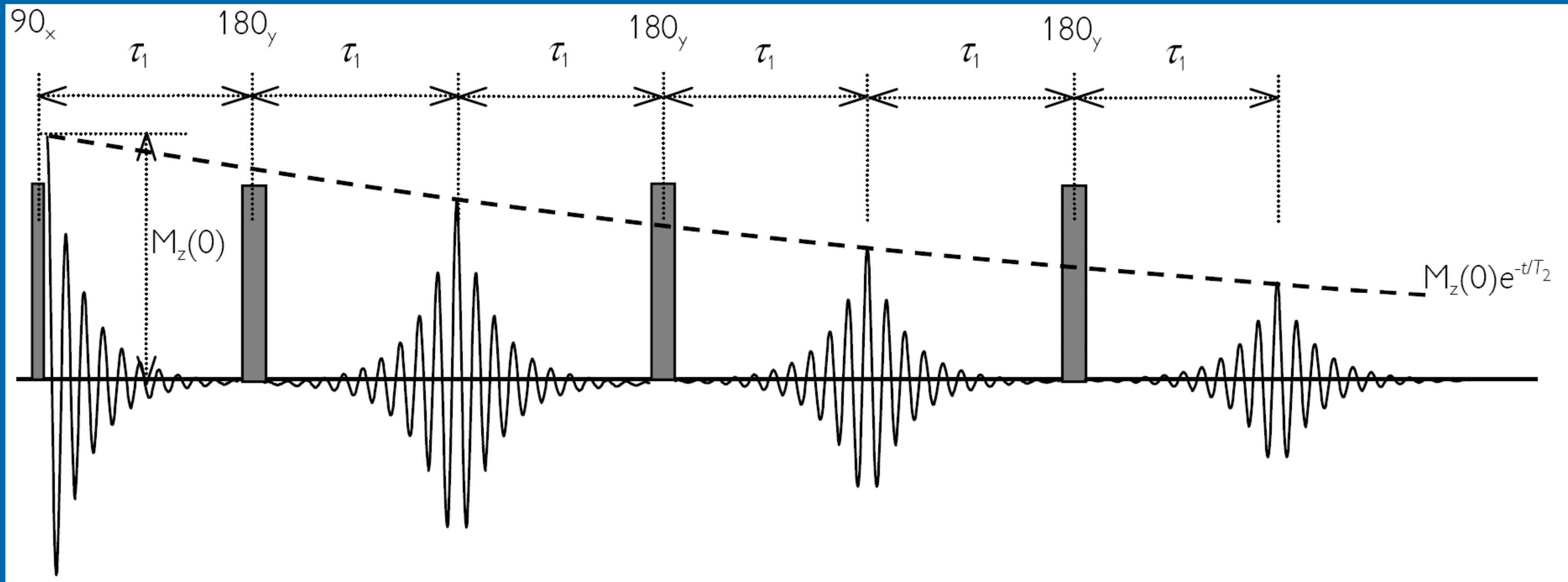




Rubber fronts are digitally aligned with the first profile

Sensitivity of the height of the polarising field under minor variations of temperature.

CPMG Sequence

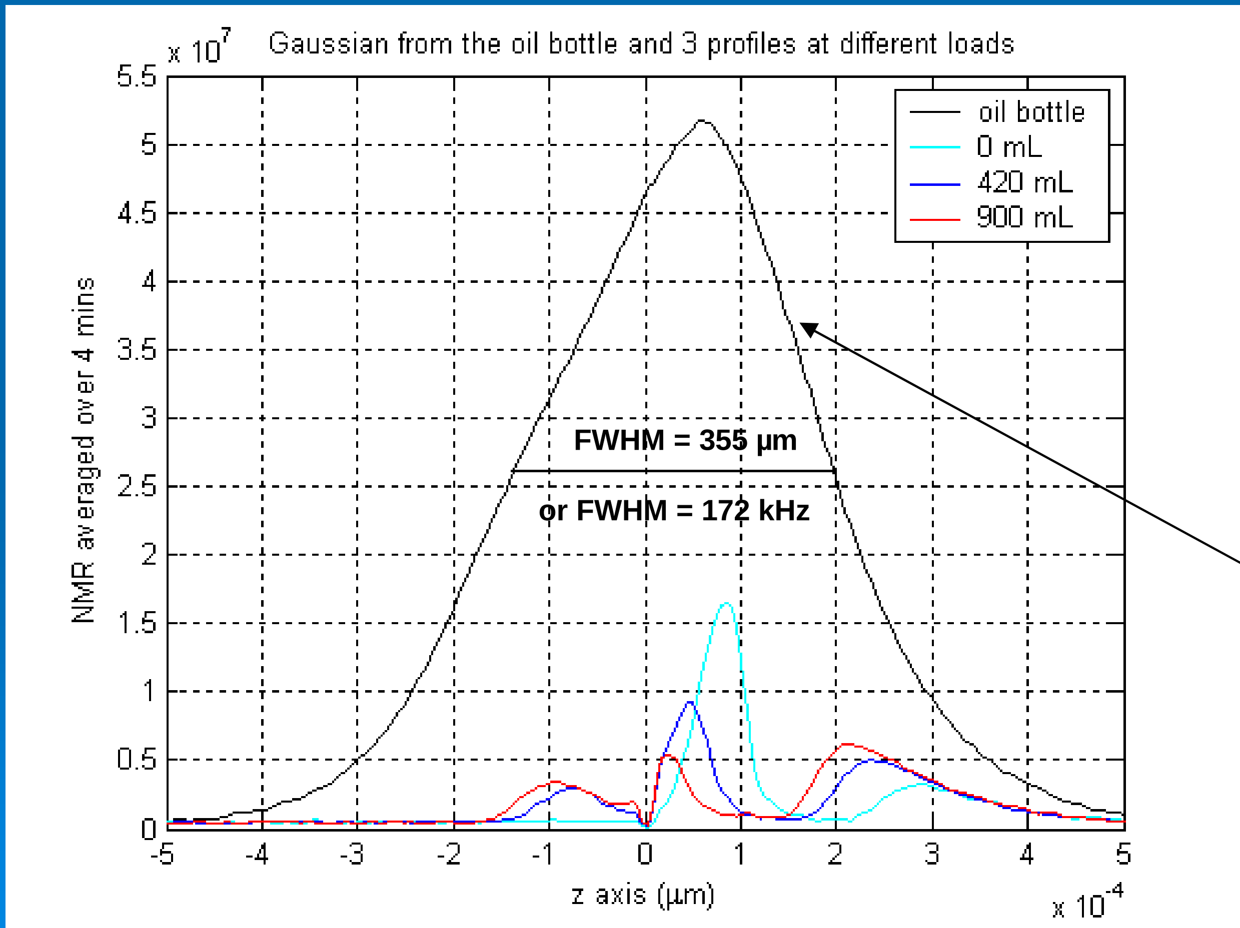


The CPMG (Carr-Purcell-Meiboom-Gill) sequence is used to run a T_2^{eff} experiment with the following settings:

- rf pulse time duration: 2.7 µs
- 40 echoes
- Echo time, TE = 510 µs
- Acquisition time: 400 µs
- Repetition time, T_R = 170 ms
- number of accumulated scans: 1500, representing approx. 4 min of averaging

Fourier transformation

From the CPMG sequence, NMR data is collected and we take the FT of each spin echoes.
To obtain a profile, we fit an exponential decay for each pixel to get the local T_2^{eff} and the local NMR signal amplitude.

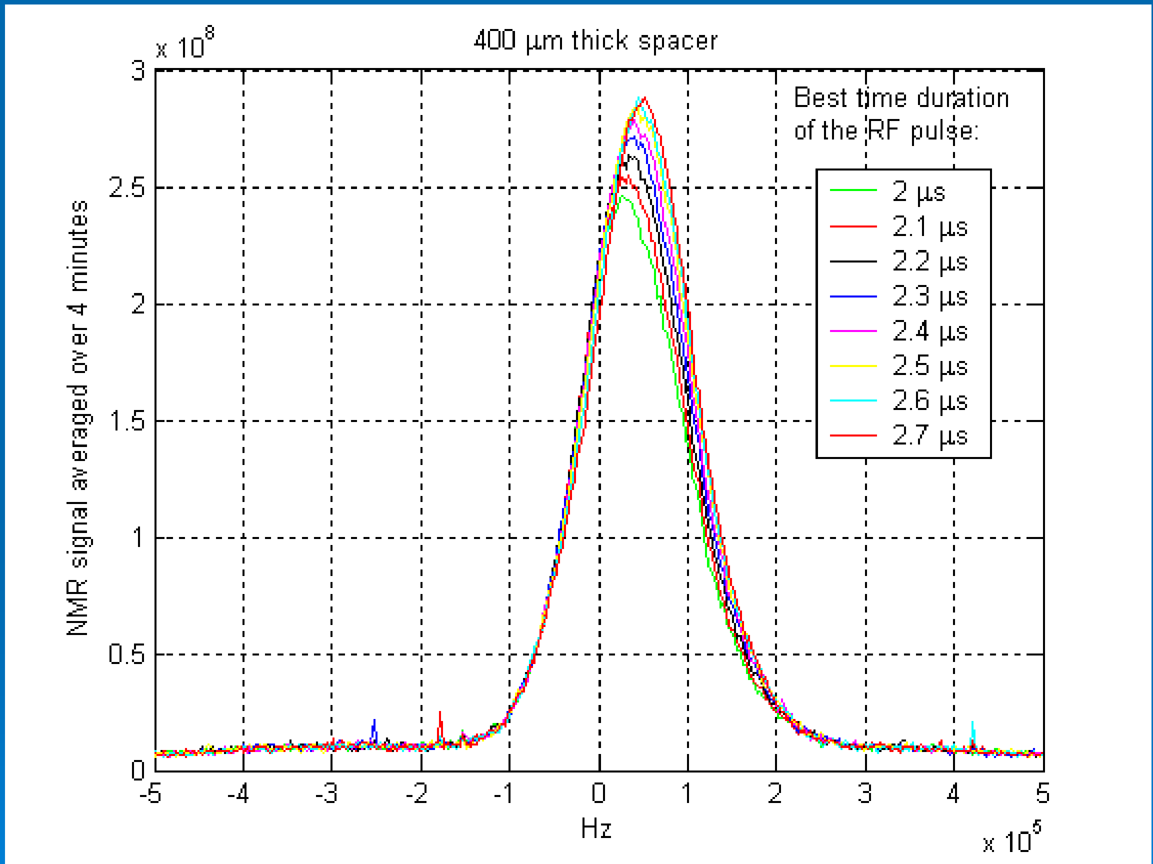


The variation of the NMR signal detected across the slice thickness depends on the rf pulse shape and was measured with a with a homogeneous sample filled with oil.

A small vial (i.d.:19.8 mm, o.d.:23 mm) was used for calibration purposes.

Optimum RF pulse

The height of the of the selected slice relative to the instrument can be changed from 10 mm to a smaller value



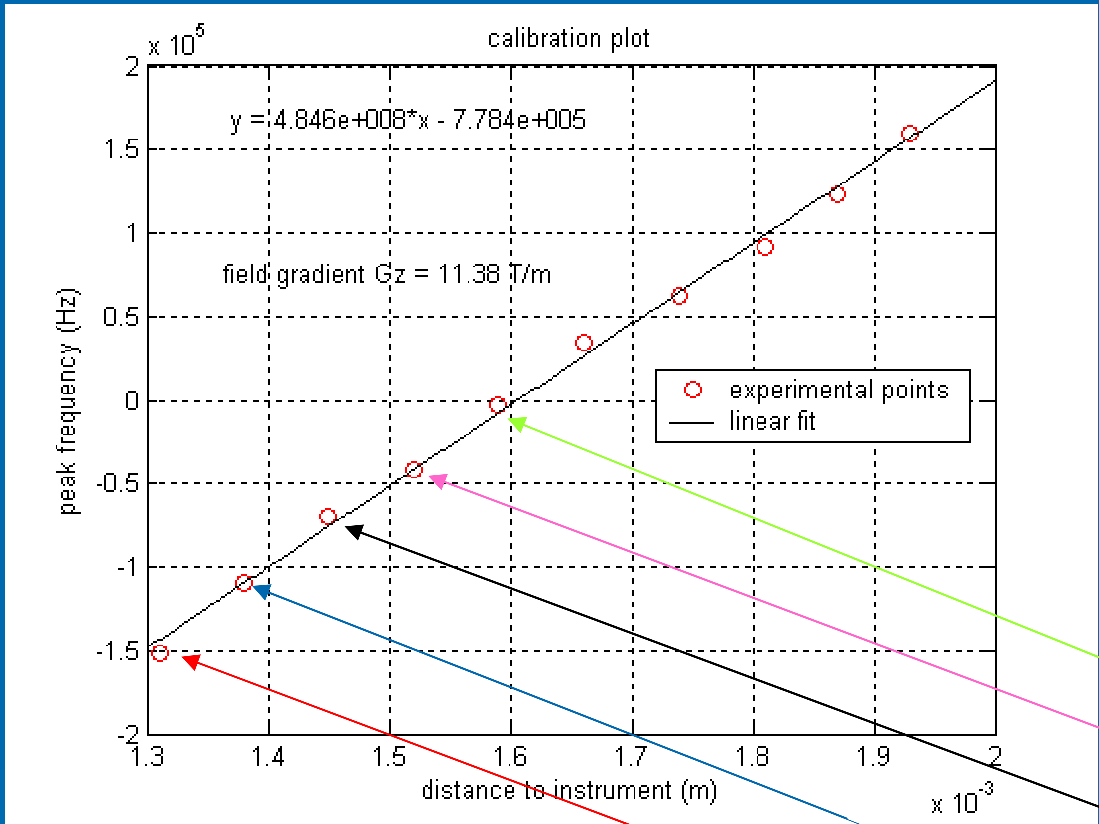
The selected slice was brought 2 mm above the rf coil with a 400 µm thick spacer.

The new 90° rf pulse time duration of 2.7 µs was found.

A broader range of frequencies can thus be selected, and a thicker sensitive volume is obtained.

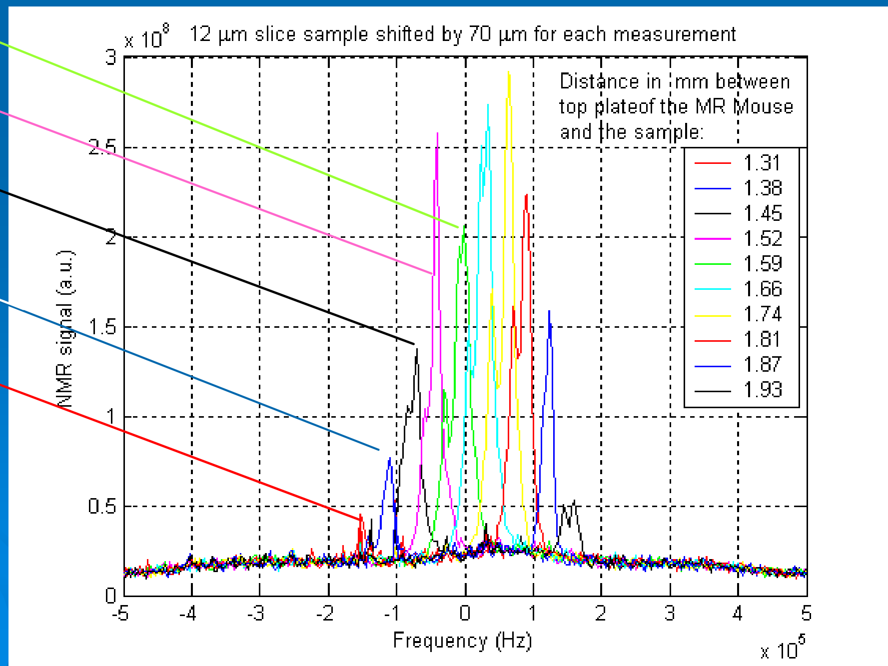
The closer the selected slice is from the instrument, the thicker it is because the rf pulses need to be made shorter resulting in a broader bandwidth.

Strength of the field gradient



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:

$\omega = \gamma B_0$

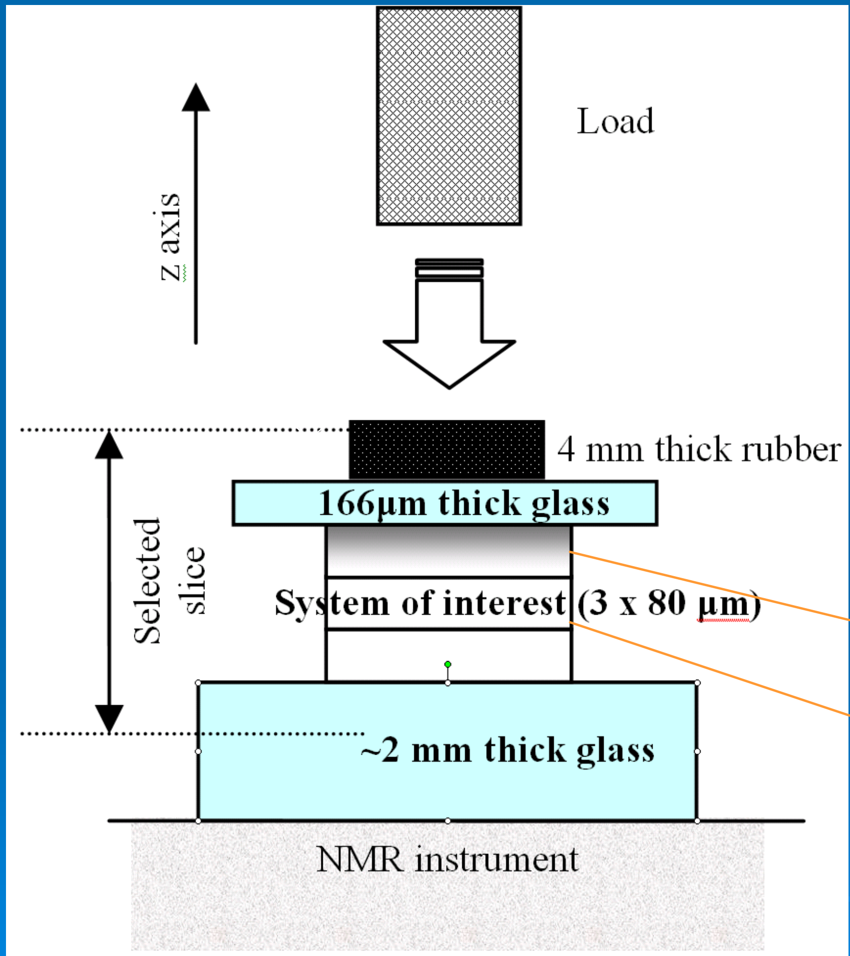
$2\pi\delta f = \gamma G_z \delta z$

$G_z = (2\pi/2.67522 \text{ x } 10^8)(4.846 \text{ x } 10^8)$

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

we are grateful to G.G. Wells,NTU, to help us make this sample

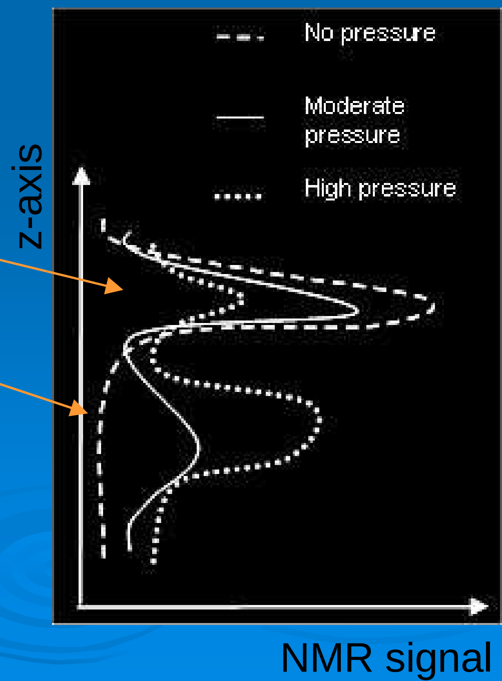
Applying the method to a combined 3 layered fabric



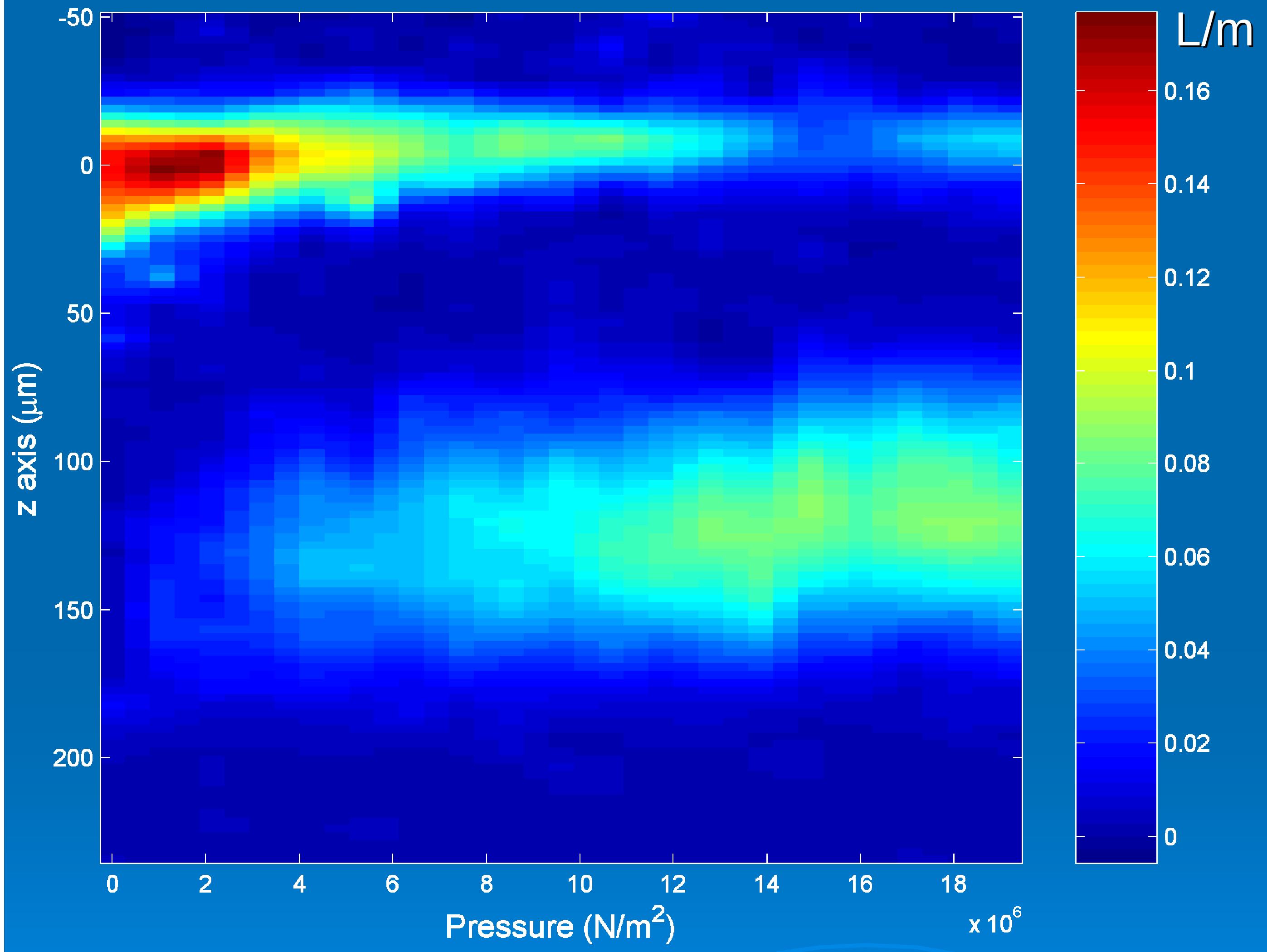
The layers are square in shape: (24 x 24mm^2)

They yield no NMR when they are scanned dry

7 µL were deposited on top of the fabrics



Fate of oil after applied pressure



Product of the local porous media saturation by the transverse (x, y) saturated surface.

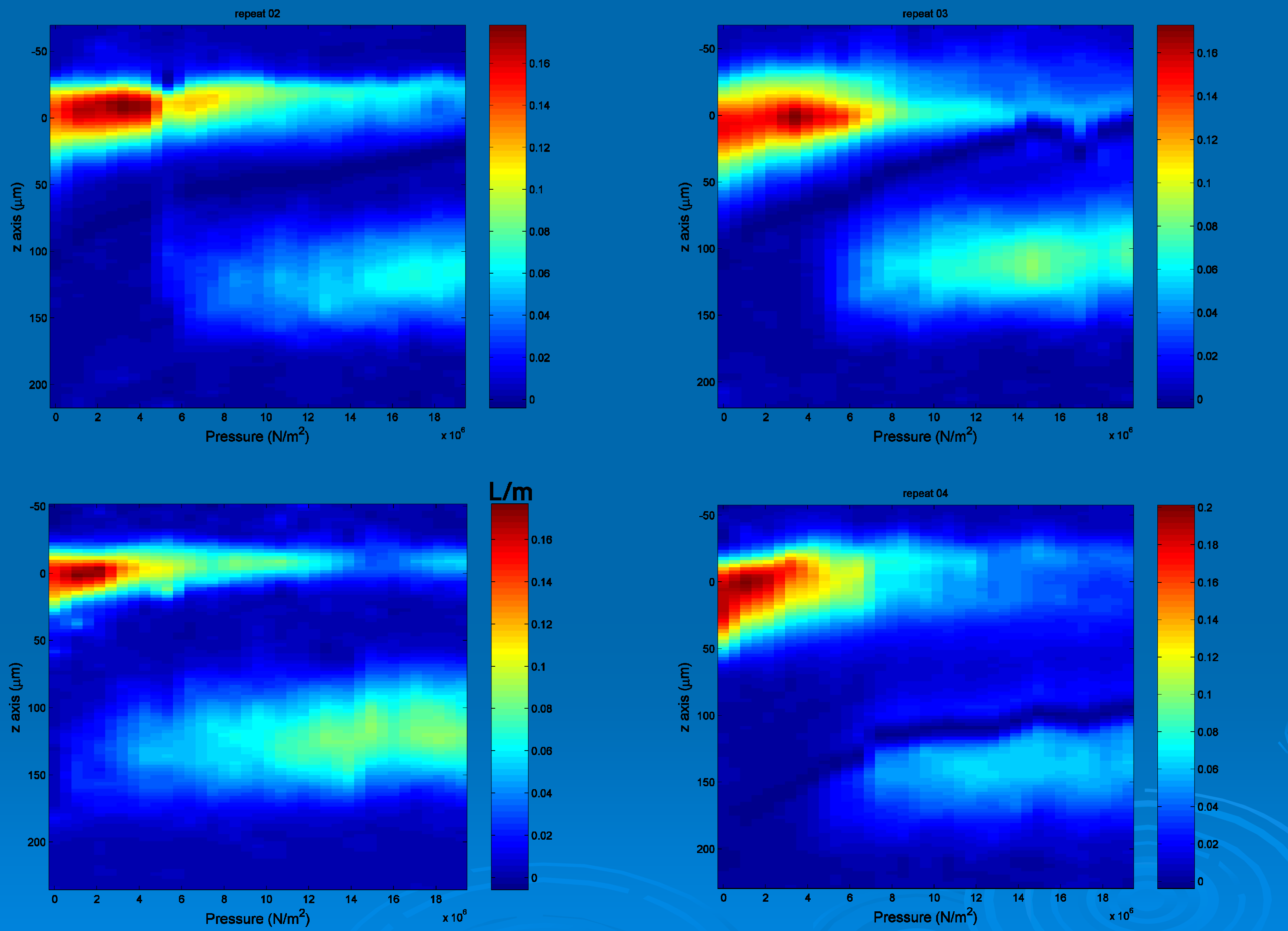
The processed NMR profiles were stacked from left to right, interpolated and color coded.

The ingress of oil is clearly displayed.

The oil must penetrate by a few microchannels, as is suggested by the lack of signal around z = 50 µm.

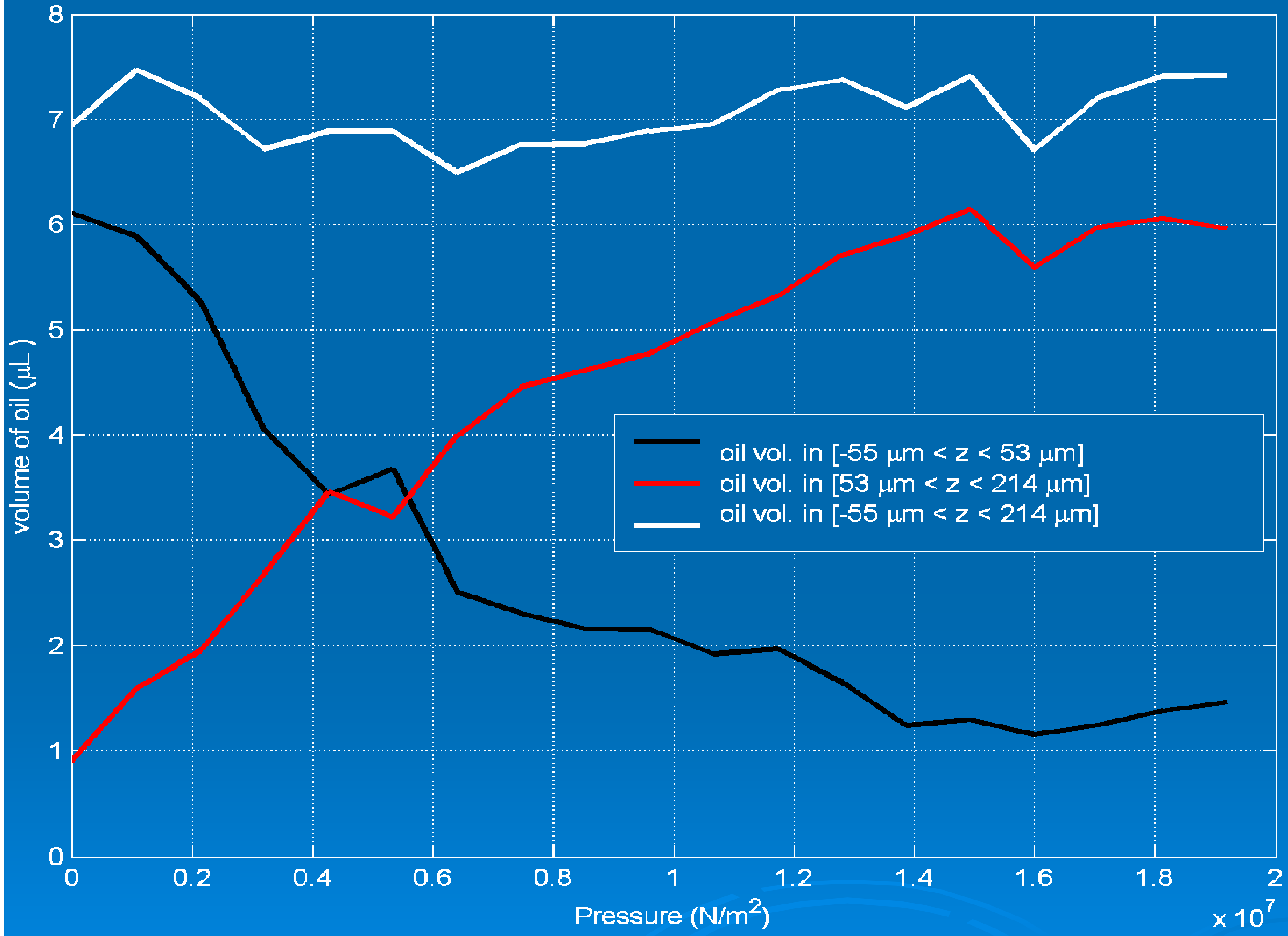
The second layer, non-repellent, pulls the oil from the first repellent layer.

Repeatability



The same experiment was repeated 4 times. Good repeatability can be seen.

Conservation of the liquid in the system



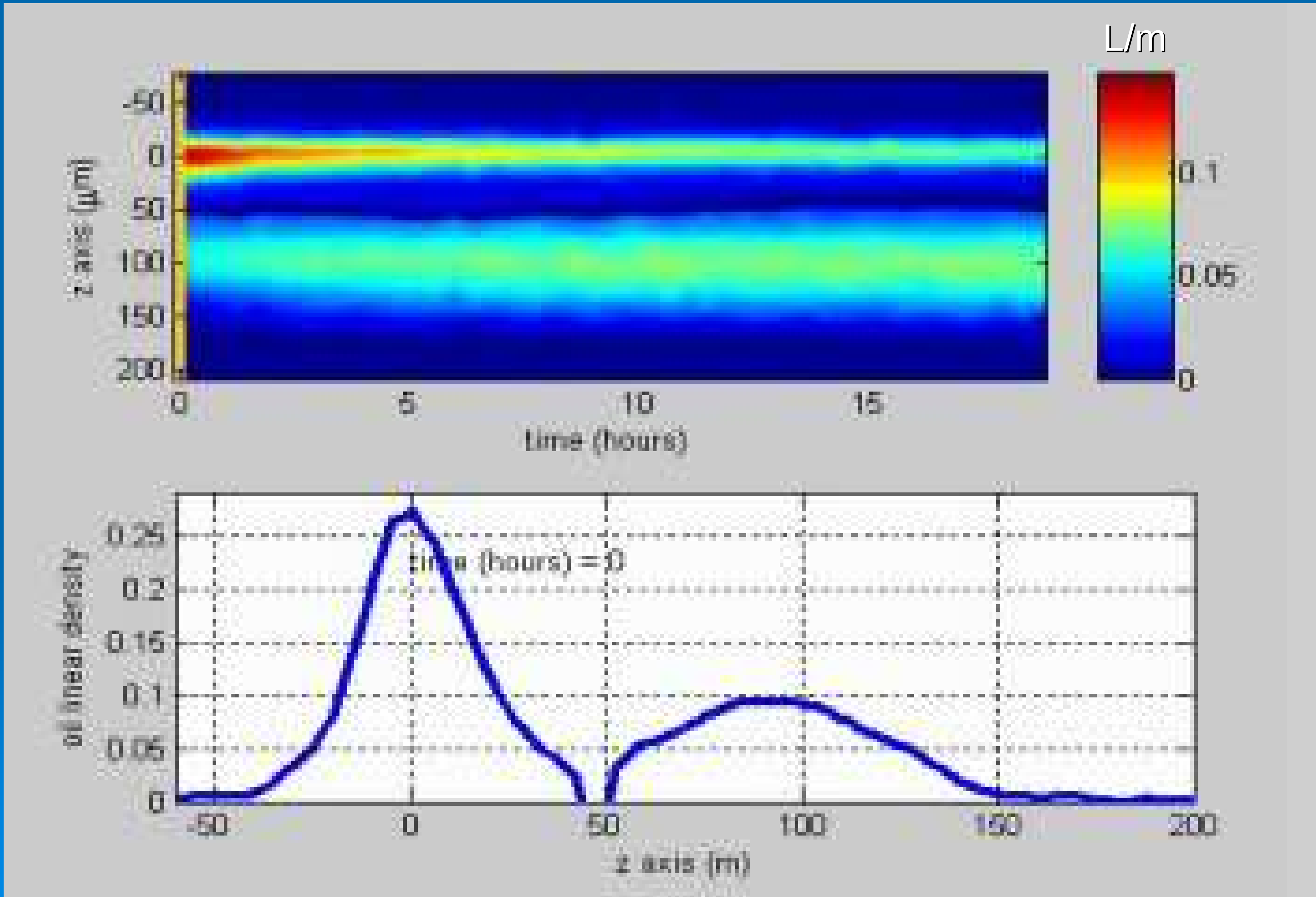
The falling curve was integrated for z between -55 µm and 53 µm

The increasing curve was integrated for z between -53 µm and 214 µm

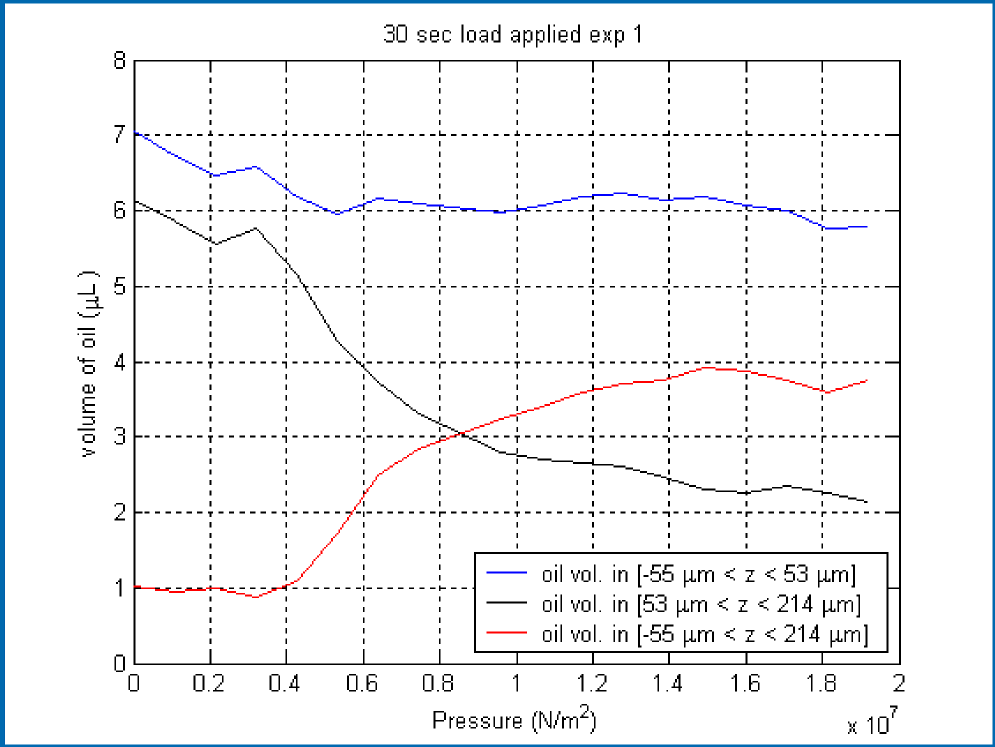
The upper curve was integrated for z between -55 µm and 214 µm

Dynamics of volume of oil found in each layer.
The data is quantitated.

Overnight experiment: time course of the ‘spontaneous’ ingress



Hurdles to overcome



Two main problems:

• Signal loss due to oil laterally moving outside the sensitive volume of the instrument.

• Quantitating the pressure that the liquid actually experiences.

Pressure is applied with air and controlled by a pressure sensor.

Conclusion

The penetration of a known volume of liquid deposited on top of a layered fabric has been successfully measured with a non invasive and non destructive method. With increasing pressure, the local liquid can be quantitated when it ingresses from one layer into another.

The geometry of the system under investigation matches elegantly the features of our device. This results in a method which is rapid and accurate, in spite of using a relatively cheap, low field and simple NMR instrument.

Further work

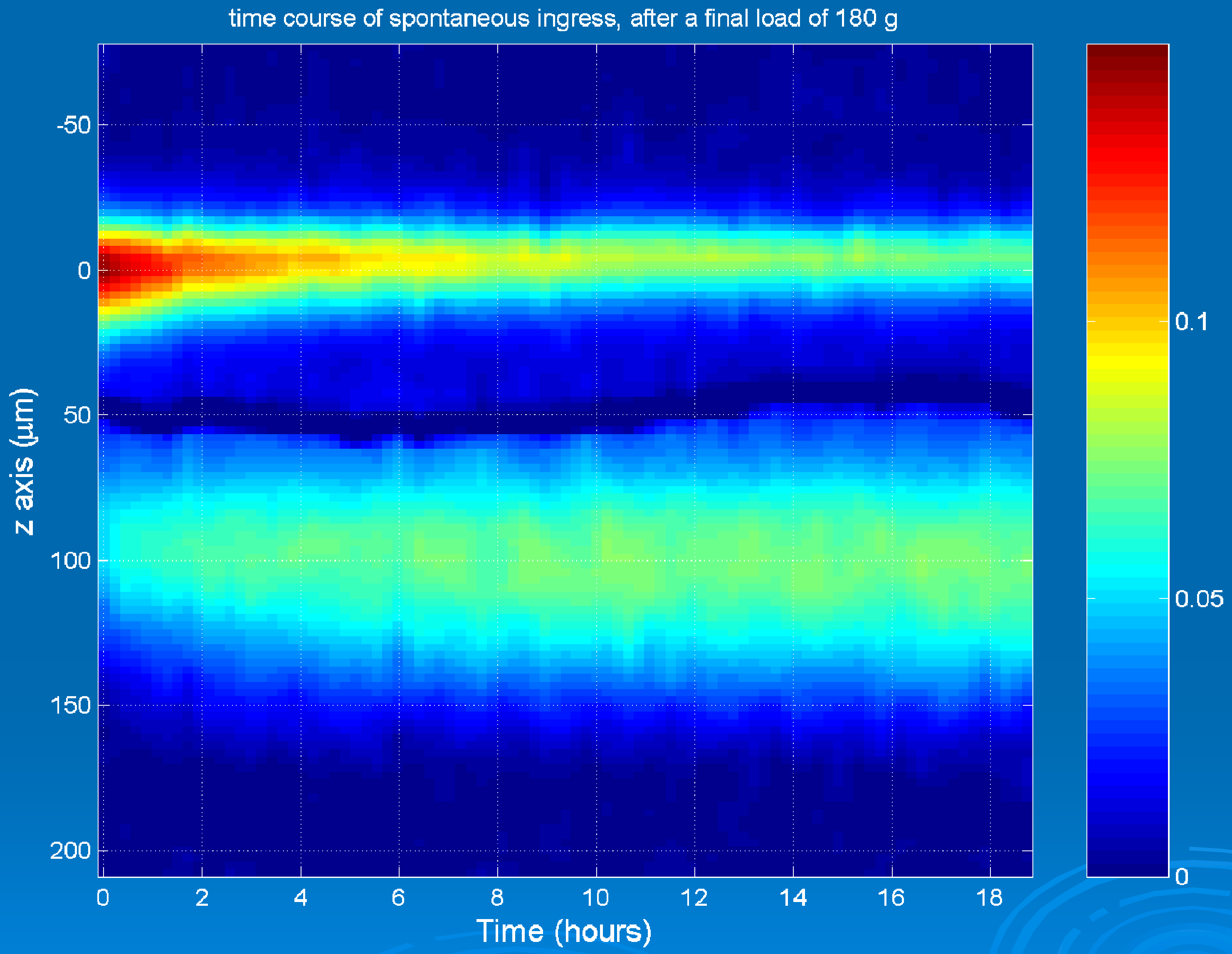
Modeling of the penetration of liquid into our sample with the knowledge of:

- the viscosity of the liquid
- its contact angle
- the combination of the fabrics

Do experiments to explore the potential lateral spread of the liquid using the gradients coil[1].

[1] ACT, Rheinisch-Westfalicish Technische Hochschule (RWTH), university in Aachen, Germany

Thanks for listening



Any questions?