Assessment of pore clogging in gravel filters using magnetic resonance.

Robert H. Morris[1], Michael I. Newton[1], Paul R. Knowles[2], Martin Bencsik[1], Philip A. Davies[2], Paul Griffin[3] and Glen McHale[1].

[1]School of Science and Technology, Nottingham Trent University, Clifton Lane, Nottingham NG11 8NS, UK

[2]Sustainable Environment Research Group, Aston University, Birmingham B4 7ET, UK

[3]Severn Trent Water Ltd, Avon House, St. Martins Road, Coventry CV3 6PR, UK

PACS:

47.56.+r	Flows through porous media
87.64.kj	NMR
92.40.Yy	Wetlands
89.20.-a	Interdisciplinary applications of physics
92.40	Hydrology

Keywords: Magnetic Resonance, Porous Media, Clogging, Filtration

Abstract

In this work we demonstrate the potential of permanent magnet based magnetic resonance sensors to monitor and assess the extent of pore clogging in filtration systems. The performance of the sensor was tested on artificially clogged gravel substrates and on gravel bed samples from constructed wetlands used to treat wastewater. Data indicates that the spin lattice relaxation time is linearly related to hydraulic conductivity in such systems. In addition, within biologically active filters we demonstrate the ability to determine the relative ratio of biomass to abiotic solids, a measurement which is not possible using alternative techniques.

Main Text

Porous media based filtration systems are a common feature of many industrial processes. They are often characterised using the median diameter of the media particles and their operation is limited by the bulk hydraulic conductivity of the filter. Filtration of process water leads to pore occlusion (clogging), which can cause substandard treatment, hydraulic malfunction and ultimately limits the filter lifetime [1]. Whilst the impact of clogging can be determined by hydraulic conductivity measurements, there are few methods which can easily elucidate the corresponding form and nature of clogging (e.g. assessing the local association of interstitial water with different clogging components) and therefore reveal the exact cause of the clogging.

A particularly important filter system is the sub-surface flow constructed wetland, typically consisting of a gravel substrate in which *Phragmites Australis* (the common reed) are planted[2]. These are used as an environmentally friendly method of sanitising wastewater before it is discharged into the watercourse. Wastewater flows through the gravel, always below the media surface, where the correct conditions for purification are encountered: retention of solids is aided by the gravel substrate and the root network of the reeds, which provide surface area to trap particulates and support biofilms. Removal of organic matter, pathogens and nutrients is mainly achieved by biofilms; whilst chemical compounds may be absorbed or precipitated according to the physicochemical conditions of the wastewater and filter environment[3]. Over time these processes result in the pore volume between the gravel becoming clogged, which leads to undesirable hydraulic short-circuiting and flow surfacing.

Measurement techniques exist to allow the hydraulic conditions within the filter to be determined *in situ* [4,5], whereas techniques to measure the composition and quantity of clog material usually require sample extraction for subsequent laboratory analysis. Resultantly poor correlation between these measures has led to the conclusion[6] that it is the form and not the quantity of the clogging that is of primary importance, and *in situ* techniques that simultaneously measure the nature of clog matter and influence on hydraulics are necessitated.

Magnetic resonance imaging has been previously demonstrated as a useful technique to assess the clogging of pores in model packed bead systems[7] but requires the extraction of part of the filter matrix. In this work, we present a magnetic resonance device which is sufficiently small that it can be embedded into a non-consolidated porous medium based filter to determine both the extent of clogging and the relative quantities of biomass and particulates. Three types of experiments are presented here which demonstrate the suitability of magnetic resonance for the measurement of clogging in gravel substrates. The first type uses a commercially available instrument, the profile NMR MOUSE®[8], with samples collected from a functional wetland which contain both biomass and particulate material. The second uses a custom built permanent magnet probe in a low permeability system to correlate the magnetic resonance results with the macroscopically measured hydraulic conductivity. The third uses smaller embeddable probes in a gravel bed filter at different stages of clogging.

In the first experiments, samples of gravel filter beds were collected from a constructed wetland near the inlet of the system; this type of sample displays high levels of clogging from both biofilms and particulates. The NMR MOUSE® was used to perform saturation recovery experiments[9] to determine the value of the spin lattice relaxation time (T_1). This parameter is strongly influenced by the environment in which the protons in the sample are found and as such can be used to determine the properties of the clog matter. The saturation recovery experiment is based on the loss of signal due to an insufficiently long recovery time. The relationship between the recovery time and the signal intensity is an exponential with time constant T_1. Figure 1 shows the effect of systematically varying the recovery time between 30ms and 1s on the averaged amplitude of the corresponding train of spin echoes. Bi-exponential fitting is performed on the collected data which suggests that there are two very distinct relaxation times corresponding to the two water environments. Measurements from other samples taken nearer the outlet showed a negligible weighting on the short $T_{1,}$ suggesting that it represents water predominantly associated with biomass.

In order to confirm that it was also possible to assess the clog state using an embeddable system, an MR sensor was produced using two 40 mm diameter neodymium iron boron magnets in a Helmholtz arrangement. A 20 mm diameter radio frequency coil within the centre of the Helmholtz pair defined the sensitive volume. This sensor was used to assess the correlation of the MR signal with standard hydraulic conductivity measurements. A 17 mm outer diameter pipe filled with coral sand as a filter medium was inserted into the radio frequency coil. At either side of the sensor, manometer take off tubes (see schematic in Figure 2) were used to estimate the head loss in the system and hence to determine the hydraulic conductivity. Diluted surface sludge collected from the same wetland as in the NMR MOUSE® experiments was sterilised by oxygenation and agitation. This removed the possibility of biological clogging resulting in particulate material clogging only. The wastewater mixture was pumped through the tube at a constant flow rate and as the filter material clogged, the variation in T_1 and hydraulic conductivity were recorded. The fairly high flow rate through the comparatively narrow bore of the tube during conductivity measurements needed to be stopped for measurements of T_1 to be made accurately with no flow artifact. This is because the sample must remain within the sensitive volume for at least the time it takes for one train of echoes to be acquired (~10ms). This situation is not an issue in a constructed wetland in which the flow rates are typically slower (trickle flow). The results of the experiment are shown in Figure 3, including a linear regression line which emphasises the good correlation between hydraulic conductivity and T_1.

The sensor in the previous set of experiments is too large to be fully representative if used in a typical porous filtration system. The final set of laboratory experiments demonstrate the application of a smaller sensor for measuring two distinctly different states of clogging in filter media: a) no clogging and b) 50% particulate clogging by volume. Gravel beds were prepared from 10 mm gravel extracted from the same wetland as the samples in the NMR MOUSE® experiments. Using the sterile sludge, a wastewater mixture was created with a concentration of 50% solids by volume. This suspension was then mixed with one gravel sample to simulate a clogged system, whilst a second gravel sample was filled with only water. Saturation recovery experiments were performed with the smaller embedded probe in these samples (Figure 4). To demonstrate the large change between these two systems, the raw magnetic resonance signal at two recovery times (500ms and 50ms) is also shown as insets for both samples in Figure 4. Such a data collection strategy could also allow for ultra-fast simple observations of clogging, by monitoring the signal amplitude at a pre-determined recovery time relating to the clog state of interest. It is also clear from the signal collected using the embeddable sensor that there is a significant difference in amplitude between the samples, not only due to changes in T_1 but also due to the volume of particulates in the clogged sample which do not produce any NMR signal. This in combination with bi-exponential fitting should allow access to the relative proportions of the three components which comprise the clog matter (biofilms, abiotic solids and water).

We have shown that magnetic resonance is a powerful tool for assessing the clogging state of gravel filters and, in particular, sub-surface flow constructed wetlands. It allows the relative proportions of biological and abiotic solid materials to be directly related to the hydraulic conductivity; an ability which cannot be easily or accurately achieved using conventional methods. The sensors have been used to fully characterise the state of clogging in typical wetland gravels, thus proving the usefulness of this technology for constructed wetlands. Although this article has focused on the application in constructed wetlands, the technique is equally applicable to many large scale industrial filters, such as trickling filters or recirculating sand filters, which experience a similar clogging process.

References

[1] S. Redner, and S. Datta, Phys. Rev. Lett. 84, 6018 (2000).

[2] UN-HABITAT, Constructed Wetlands Manual (2008). ISBN: 978-92-1-131963-7

[3] H. Brix, Wat. Sci. Tech. 29, 71 (1994).

[4] P.R. Knowles, P. Griffin, and P.A. Davies, Water Res. 44, 320 (2009)

[5] A. Lin, J. Debroux, J.Cunningham, and M. Reinhard, Ecol. Eng. 20, 75 (2003).

[6] A. Caselles-Osorio, J. Puigagut, E. Segú, N. Vaello, F. Granés, D. García, and J. García, Water Res. 41, 1388 (2007).

[7] O. Lamrous, D. Houi, C. Zarcone, and J. Pradere, Rev. Phys. Appl. 24 607 (1989)

[8] G. Eidmann, R. Savelsberg, P. Blümler, and B. Blümich, J. Magn. Reson. Ser. A, 122, 104 (1996).

[9] McDonald, G.G., Leigh, J.S., J. Magn. Reson. 9, 358 (1973).

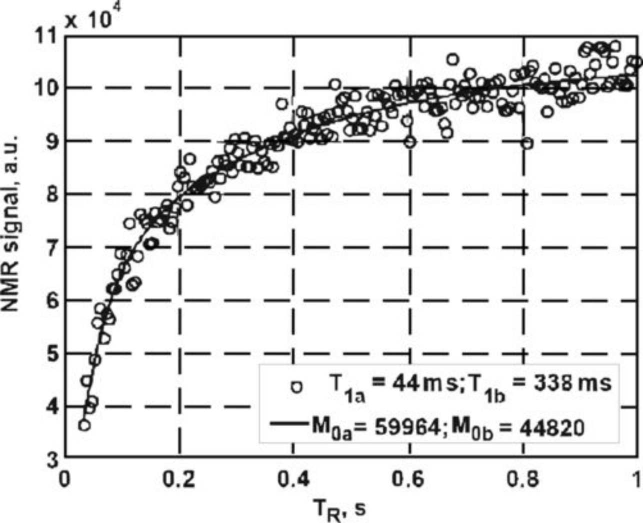
Figure Captions

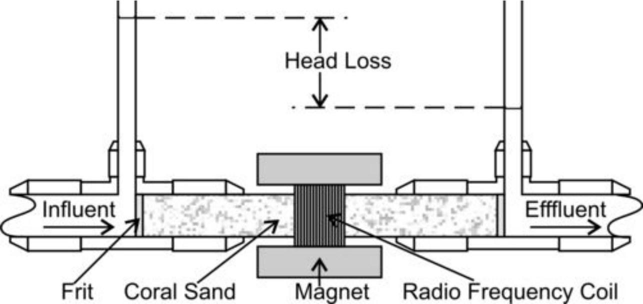
Fig. 1. Saturation recovery curve for inlet sample showing bi-exponential behaviour as expected from a sample with two water environments. The weight of each exponential is given by M_{0a} and M_{0b}.

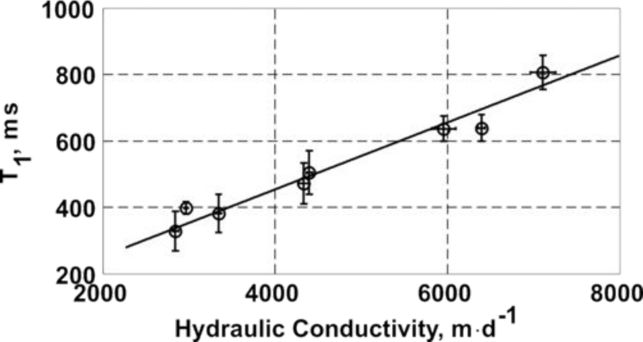
Fig. 2. Schematic representation of the system used to correlate the spin lattice relaxation time (T_1) and the hydraulic conductivity.

Fig. 3. Correlation between hydraulic conductivity and spin lattice relaxation time for clogging in coral sand.

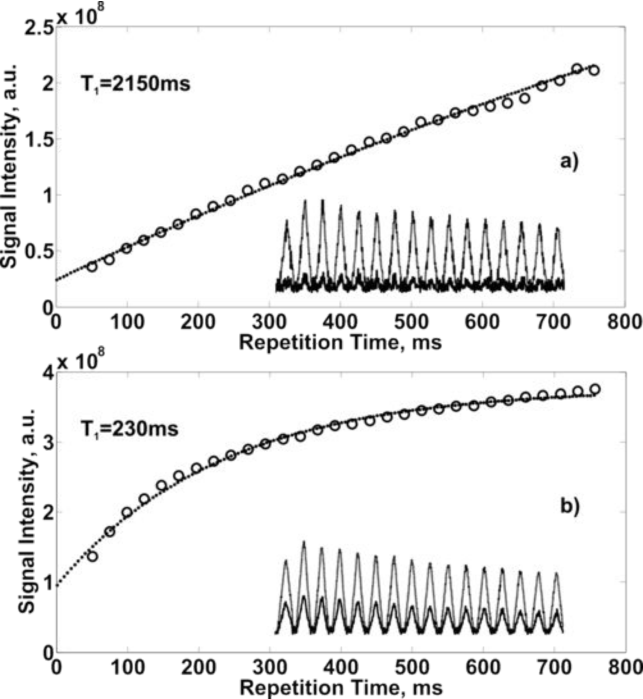
Fig 4. Saturation Recovery from embedded probe in unclogged sample a) and 50% clogging by pore volume b) with monoexponential fits for T_1. Raw spin echoes are shown as insets in both graphs for two recovery times: 50ms (continuous line) and 500ms (dashed line). Results shown are from 256 averages, representing a total experimental time duration of 50 minutes.


x 10^4
NMR signal, a.u.
T_R, s
0
0.2
0.4
0.6
0.8
1
3
4
5
6
7
8
9
10
11
T_1a = 44 ms; T_1b = 338 ms
M_0a = 59964; M_0b = 44820


Head Loss
Influent
Effflluent
Frit
Coral Sand
Magnet
Radio Frequency Coil



1000
800
600
400
200
2000
4000
6000
8000
T1, ms
Hydraulic Conductivity, m·d-1



x 10^8
2.5
2
1.5
1
0.5
0
T1=2150ms
a)
Signal Intensity, a.u.
0 100 200 300 400 500 600 700 800
Repetition Time, ms
x 10^8
4
3
2
1
0
T1=230ms
b)
Signal Intensity, a.u.
0 100 200 300 400 500 600 700 800
Repetition Time, ms