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Analysis of clogging in constructed wetlands using magnetic resonance

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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 water 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.

1. Introduction

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 (reed bed), 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]. In contrast, 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 required for reliable measurements.

Magnetic resonance (MR) is a technique which is best known for its use in imaging in medical diagnostics [7]. It has however been previously demonstrated as a useful technique to assess the clogging of pores in model packed bead systems [8]. In this work, we employ similar pulse sequences but without the imaging component, so that the average signal from the whole sample volume is interrogated. It is intrinsically sensitive to the presence of water molecules in the sample under interrogation and can manipulate them to determine the properties of their environment. A typical measurement using an MR Imaging scanner relies on extraction of a sample sufficiently small to fit within the bore of the magnet (typically 70 mm to 1 m diameter) which often leads to changes in the sample structure. 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.

2. Theory

Magnetic resonance is a phenomenon which enables the intrinsic physical properties of water molecules to be probed non-invasively using an appropriate combination of static magnetic field, radio frequency coil and suitable radio frequency pulse sequence [9]. Although the principles of magnetic resonance are most commonly applied to chemical shift spectroscopy and imaging, the technique used here is relaxometry. In this technique, a system of protons is excited using a radio frequency field. This energy is then gradually transferred to the molecular lattice. The decay constant of this energy transfer is measured and provides information regarding the system. The basic experiment for magnetic resonance sensors is a CPMG sequence [10]. This allows signal to be acquired many times before the system has returned to equilibrium [11]. This small signal is then averaged after a delay known as the repetition time. If this delay is shorter than the time it takes for the system to return to equilibrium, there will be a loss in signal. This phenomenon is used to measure the T_1 'spin lattice relaxation time'. By varying the repetition time and recording the averaged signal intensity an exponential relationship is found with time constant T_1 [12]. This parameter is strongly affected by its environment and hence is used in this study to determine the local association of water.

The basic setup for a magnetic resonance sensor is a strong magnetic field (typically over 0.1 T) inside which a copper coil is placed that is used both to generate the changing magnetic field and also to collect the resulting current [13]. For in situ measurements it is possible to collect signal using strong permanent magnets [14] such as those used in the devices presented here. Electronic consoles are used to generate the required radio frequency pulses at around 1 kW power and also to collect the resulting signal which is many orders of magnitude smaller. These often employ the use of a duplexer to allow the transmission and reception to take place over a single cable and coil.

The use of permanent magnet sensors for in situ measurements was first conceived for bore hole measurements to determine suitable blasting sites for oil recovery in the late 1970s [15]. This led to the development of the profile NMR MOUSE® [16] and NMR MOLE [17] which are both intermediate sized (under 0.03 m^3) unilateral magnetic resonance devices; these have found several niche applications particularly in materials testing of large samples. Very recently the possibility of using one or more small permanent magnets (under 10 cm) to provide the static magnetic field has been demonstrated as disposable long term embeddable sensors for civil engineering [18].

3. Experimental Protocol

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®, with samples collected from a functional wetland which contain both biomass and particulate material. Use of this commercial instrument provides confidence in the validity of the MR approach. The second uses an in-house custom built permanent magnet probe in a low permeability system to correlate the magnetic resonance results with the macroscopically measured hydraulic conductivity. This enables the work to be moved towards embeddable sensors. The third uses physically smaller (again in-house built) embeddable probes in a gravel bed filter at different stages of clogging and so demonstrates the potential for field deployment.

In the first set of 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 (with a commercial console to drive the system and process the signal) to perform saturation recovery experiments to determine the value of the spin lattice relaxation time (T_1). This sequence uses a train of RF pulses in the saturation period, applied at non-regular intervals to ensure saturation [19]. T_1 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.

In the second set of experiments, 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 (Figure 1) 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. MATLAB (Mathworks, MA) was used to control off-the-shelf hardware [20] to generate the pulse sequence and process the signal. Visual measurements were also made of the take off tubes for the hydraulic conductivity. 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 (~10 ms). This situation is not an issue in a constructed wetland in which the flow rates are typically slower (trickle flow) with water taking typically a week to pass along the length of the bed.

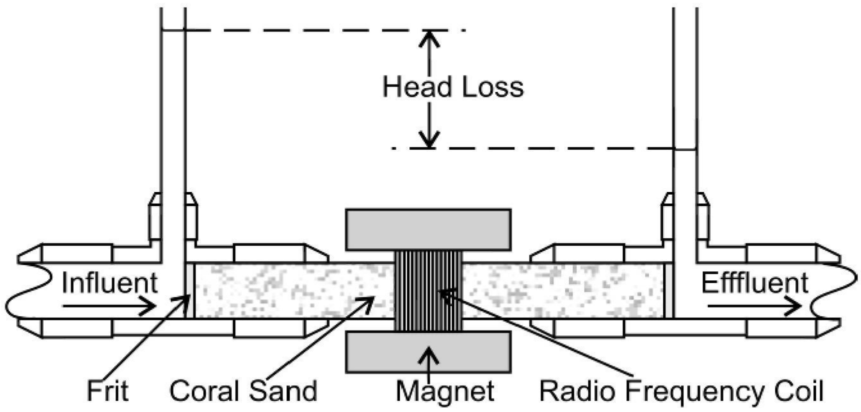

Fig. 1 Schematic representation of the system used to correlate the spin lattice relaxation time (T_1) and the hydraulic conductivity in the second set of experiments.

The sensor in the previous set of experiments is too large to be fully representative if used in a typical porous filtration system. The third and final set of laboratory experiments demonstrate the potential for a smaller field deployable embeddable sensor for measuring two distinctly different states of clogging in filter media: no clogging and 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. Again 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 shown in Figure 2 in these suspensions.

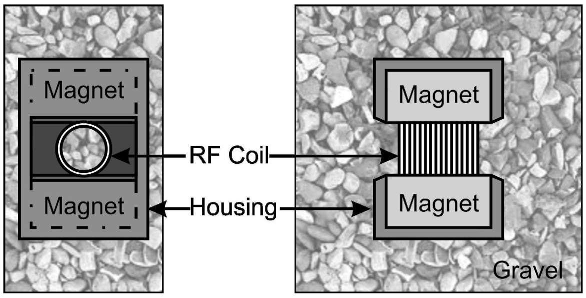

Fig. 2 Schematic of smaller embedded probe used to conduct the third set of experiments.

4. Results and Discussion

The data in Figure 3 shows the effect of systematically varying the recovery time used for the NMR MOUSE® between 30ms and 1s on the averaged amplitude of the corresponding train of spin echoes; a bi-exponential fit is also shown along with the corresponding fit parameters. The results from the first set of experiments demonstrate the ability to separate the proportions of several different water environments in a sample as bi-exponential fitting is required to obtain a good fit to the collected data. This is possible as 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 particulates. For a given system it should be possible to determine the relative ratios of different water environments at different bed locations and hence determine the state of clogging across the entire bed.

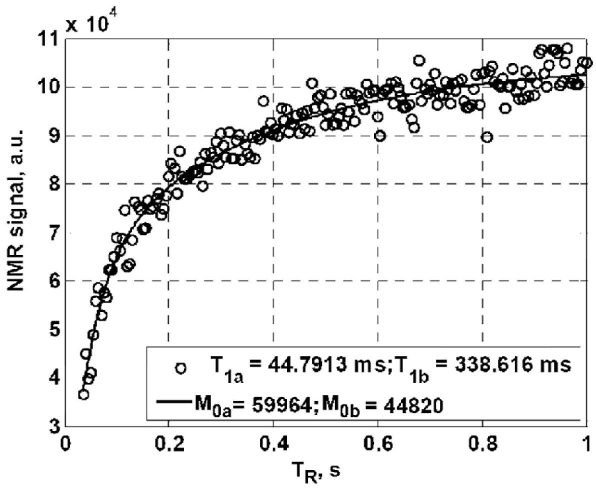

Fig. 3 NMR MOUSE® determined saturation recovery curve for inlet sample showing bi-exponential behaviour as expected from a sample with two water environments. The weight of each exponential is given by M_{0a} and M_{0b}.

The measured T_1 values of the second experiment are shown in Figure 4 for the corresponding hydraulic conductivity, including a linear regression line which emphasises the good correlation between them. In the second set of experiments the strong correlation between the hydraulic conductivity and the T_1 parameter indicate that in a simple system, a single relaxation measurement is sufficient to determine the clog state due to pore occlusion by solids. Such a calibration could be used in a semi-automated system which reports back the clog state of the bed as opposed to the hydraulic conductivity or T_1 which require interpretation. The error on the measurements is determined by the quality of the fit to the saturation recovery curves and is better than 10% for all measurements made. Improvements to this can be made at the expense of experimental time. By using a single point measurement, the overall time spent could be better optimized to deliver more accurate results.

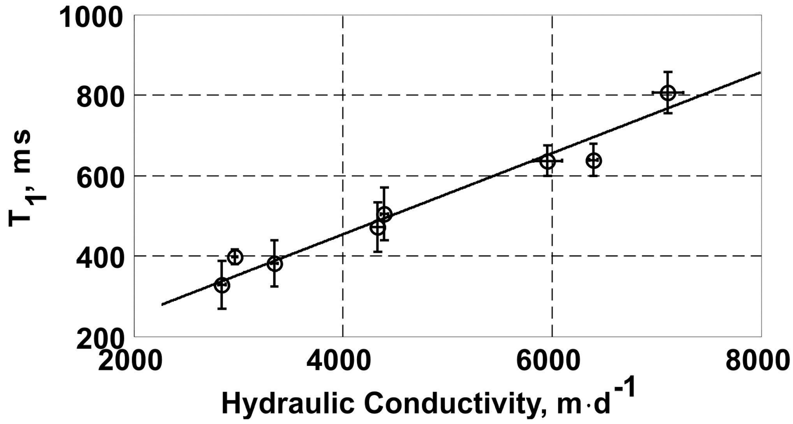

Fig. 4 Correlation between hydraulic conductivity and the spin lattice relaxation time from the monoexponential decays collected during clogging in coral sand using the system shown in fig. 1 in the second set of experiments. Data was not normalised to account for pore occlusion.

Saturation recovery curves from the clogged and unclogged samples investigated with the embedded probes in the third set of experiments are shown in Figure 5. The T_1 of the unclogged sample is an order of magnitude longer than that of the clogged sample. Both fits are within 5% which is more than sufficient for a qualitative assessment of the clog state. The large difference between the clogged and unclogged systems agree well with the results found in the second set of experiments despite the use of a different MR sensor. The large difference between the two allows for the value of T_1 to be approximated from a single data point collected at an appropriate repetition time (for example 200 ms in this case). 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).

The results presented here were collected from model systems constructed in the laboratory. The next stage will be to produce sensors which are sufficiently robust that they can be embedded in the ground without detriment to the collected data. This requires investigation of several factors.

The first is the temperature dependence of the static magnetic field which following preliminary experiments can be compensated with automatic tuning (for a typical sensor the drift appears to be around 0.26%K^{-1}). Due to the presence of a strong magnetic field the sensors will encourage build up of magnetic material over time. In the experiments conducted in this study we found a very small proportion (<0.1%) of the gravel contained magnetic chips although these were not sufficiently large to cause field distortion. In the field it will be important to ensure that the substrate is not largely magnetic and that the influent does not have a large paramagnetic or ferromagnetic compound content. Further investigation into influent containing ochre is necessary as this is a very common compound in mine water treatment [21].

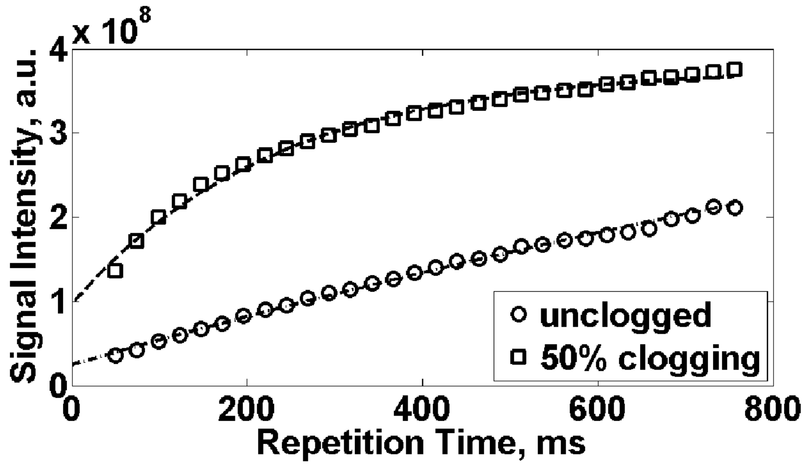

Fig. 5 Saturation Recovery from an embedded probe in unclogged sample (o) and 50% clogging by pore volume (□) with monoexponential fits for T_1. Results shown are from 256 averages, representing a total experimental time duration of 50 minutes and are obtained using the embeddable probe system shown in fig. 2.

5. Conclusion

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.

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