Determining the clog state of constructed wetlands using an embeddable Earth's Field Nuclear Magnetic Resonance probe

F. Hill-Casey, T. Hughes-Riley, C.R. Bradley, M.I. Newton, R.H. Morris

School of Science and Technology. Nottingham Trent University, United Kingdom

Corresponding author: Rob Morris, School of Science and Technology, Nottingham Trent University, NG11 8NS Nottingham, E-Mail: rob.morris@ntu.ac.uk

(Received xxx, Accepted xxx)

Abstract

The recent rise in interest of green technologies has led to significant adoption of the constructed wetland as a waste water treatment technique. This increased popularity has only been mired by the decline in operational lifetime of wetland units, leading to the need for more regular, time consuming, and expensive rejuvenation techniques to be performed than initially anticipated.

To extend operational lifetimes and increase efficiency of wetland units, it is crucial to have an accurate method to determine the internal state of the wetland system. The most important parameter to measure within the reed bed is the clog state of the system, which is representative of the overall system health.

In previous work, magnetic resonance (MR) measurements, parameters of T_1 and T_2^{eff} , have been demonstrated as extremely powerful tools to determine the internal clog state of a wetland [1, 2]. Measurements have been performed in a laboratory, setting using low field permanent magnet arrangements. This work presents an Earth's Field Nuclear Magnetic Resonance (EFNMR) probe suitable for *in situ* measurements within constructed wetlands.

We show T_2^{eff} and T_1 measurements using the EFNMR probe. T_1 values are shown to be sensitive to the change in the clog state with 1498 ms for the thickly clogged sample and 2728 ms for the thinly clogged sample. T_2^{eff} values are shown to be marginally more sensitive to clog state with 630 ms for a thickly clogged sample and 1212 ms for the thinly clogged sample. This gives distinguishable variation within both parameters suggesting that this probe is suitable for embedding into an operational constructed wetland.

This work was conducted as part of an EU FP7 project to construct an Automated Reed Bed Installation, ARBI.

Keywords

Constructed wetlands, Earth's Field Nuclear Magnetic Resonance, clog state, spin lattice relaxation, spin-spin relaxation, embedded sensor.

1. Introduction

Constructed wetlands have been used all over the world as a green, environmentally friendly waste water treatment method. Initial predictions of operational lifetimes were shown to be in excess of 50 years [3]. With practical implementation it is clear that 50 years was an over estimation, with most wetlands reaching a fully clog state within ten years. From these reduced lifetimes it is found that the need for more regular, time consuming, and expensive rejuvenation techniques to be performed is far more than initially anticipated.

Typical wetlands are comprised of a gravel matrix in which plants such as *Phragmites australis* (the common reed) are planted. Through this gravel matrix biochemically treated effluent is flown. This effluent then undergoes a number of treatment regimens such as chemical separation, biological degradation and physical filtration to culminate in clean, safe water, which is able to be released back into the water course [4].

It has been demonstrated that it is possible to perform measurements upon wetland material in a laboratory setting using magnetic resonance (MR) to record the values T_1 or T_2^{eff} , which have been shown to be correlated to clog state [1, 2]. Previously presented sensors have been two cylindrical permanent magnets in a Helmholtz like arrangement with a solenoid for both the transmission of the radio frequency pulse and the collection of the resultant signal, with a central aperture of 10 mm. Despite the proficiency of these sensors at determining the clogging state of a system issues are identified to the prospect of long term embedding as the bore hole configuration can become clogged over time in a manner which is misrepresented of the rest of the system. Further work has been performed into the construction and deployment of unilateral sensors however, all designs rely upon expensive permanent magnets and only a small volume of the wetland can be explored. Ideally as large a volume as possible would be probed using MR to ensure that readings were representative of the wetland as a whole.

One of the benefits of an Earth's Field Nuclear Magnetic Resonance Probe is that it does not require the use of permanent magnetic arrays, instead using the Earth's magnetic field. The Earth's field, while weak, is extremely useful for performing nuclear magnetic resonance, due to its extremely high homogeneity, world wide availability and by its nature, free of cost. A well-known and often raised concern of EFNMR is that the device needs to be in an extremely low noise, and highly homogeneous environment, this concern is partly laid to rest when considering the locations and environments that constructed wetlands are often deployed in being away from highly developed centres. Additionally, because it is not limited by available magnet sizes, EFNMR probes can be built to any size, allowing for them to investigate large volumes of material.

2. Materials and Methods

2.1 The Earth's Field Probe

The EFNMR probe was comprised of two solenoids. The inner solenoid acted as a transmit receive coil, tuned to the necessary resonant frequency at a given location (at Nottingham Trent University this is around 2 kHz) using the variable capacitor inside the commercial spectrometer (described below). The second coil applied a polarising field to the sample. This was used to better align the nuclear magnetisation of the sample with the Earth's field. This is described in detail in the literature by Rabi *et al.* [5] and others [6, 7].

The transmit-receive coil Fig. 1(b) was hand wound and consists of 3200 turns over eight layers of 0.315 mm enamelled copper wire (Scientific Wire Company Essex UK), wound on a section of extruded acrylic pipe (outer diameter. = 8.3 cm, inner diameter. = 7.3 cm, length = 24.5 cm; The Plastic Shop, Coventry, UK). The transmit-receive coil was then waterproofed using an additional acrylic tube that fit closely around the outside of the coil. The tubes were then sealed using two acrylic face plates.

The polarising coil (Fig. 1(a)) consisted of 400 turns, in four layers of 1.80 mm enamelled copper wire (Scientific Wire Company Essex UK). This was hand wound on a section of extruded acrylic pipe (outer diameter. = 15.5 cm, inner diameter. = 14.4 cm, length = 24.0 cm; The Plastic Shop, Coventry UK). Thick enamelling on the wire for this coil made additional waterproofing measures unnecessary.



Fig. 1: The Earth's field nuclear magnetic resonance probe.(a) Polarising coil.(b) Transmit-receive coil.

2.2 Magnetic resonance protocol

Signal generation, collection and processing used a Magritek Terranova MRI spectrometer running on Prospa version 3.12 software (Magritek, Wellington, New Zealand). To collect the T_1 and T_2^{eff} measurements two pulse sequences are utilised.

 T_1 was collected using a built in pulse sequence which involved performing multiple single pulse experiment where the length of the polarising field pulse was varied (' T_1 in the polarising field', described in detail here[8]). The net nuclear magnetisation was allowed to develop along the coils axis within the polarising field for a given time and then rotated adiabatically back to the earth's field, thus increasing the ratio of polarised nuclei. The amplitude of the signal was greater with longer polarising times. As the polarising time increased a saturation was reached. The polarising time was varied in 32 steps from 100 ms to 3200 ms. A T_1 value was determined by plotting the signal attenuation against the polarising time, and fitting it to a mono exponential curve using IGOR Pro version 6.3 (WaveMetrics, Oregon, USA)

 T_2^{eff} measurements were collected using a Carr Purcell Meiboom Gill (CPMG) sequence [9]. The mono exponential fitting of the echo integrals plotted against echo time was performed with IGOR Pro.

2.3 Test wetland set-up

The ability to accurately assess the suitability of an EFNMR probe for *in situ* measurements required a system in which to perform these measurements. Two on-site wetlands were constructed using a non-magnetic gravel matrix (9.6 mm gravel, Travis Perkins Trading, Bulwell, UK) and filled with water, however they differed in scale and functionality.

The first of the units was based on a repurposed intermediately bulk container (102 cm x 92 cm x 90 cm; DV Containers Ltd, Wrexham, UK) with an outlet valve to allow for flow through the system, and a metal base and struts for support as the primary structure for the system.

A further small scale system was constructed using an 80 litre polythene container (60 cm x 40 cm x 40 cm; Wilkinson Plc, Worksop, UK) based upon the same principals as the large scale unit. However, this unit was temporary in nature and thus did not receive either an outlet flow valve or plantations of reeds. This system was filled with the same non-magnetic gravel to 400 mm, and with the water level at 390 mm from the base of the container.

To perform measurements, the probe was embedded within the test wetland so that the top of the coil was level with the surface of the gravel and the bore of the system was completely submerged. At the time of embedding the wetland units only consisted of a simple gravel and water matrix and were receiving no effluent flow.

2.4 Sample preparation

Two samples were used in these experiments. The first sample was thickly clogged sludge and taken from the outlet of an operational horizontal, sub-surface flow wetland (ARM, Rugeley, UK). The sample contained both particulate and biomass material and was considered to be representative of a heavily clogged wetland, nearing the time which it required rejuvenation.

The second sample was thinly clogged sludge and represents the initial state of a wetland. This was taken from the prototype wetland built at Nottingham Trent University as described above.

Samples were stored in 500ml polypropylene bottles with a wall thickness of approximately 1 mm.

3. Results and Discussion

3.1 Measurements at the prototype wetland

Initially measurements were performed in the large prototype wetland described in section 2.3 with the sensor resting upon the surface of the gravel. A series of T_2^{eff} experiments were attempted, however where unsuccessful. It was believed that magnetic field inhomogeneity's introduced by the ferrous cage and base of the IBC container were significant enough (at a distance of 15 cm) to not allow for EFNMR to be conducted.

This highlighted a limitations of the EFNMR probe, as the final ARBI unit will likely have to be constructed from metal to allow for easy transportation. This restriction should not be an issue for low-field permanent magnet systems, as the magnetic field would be substantially higher (~0.5 T [11, 12]).

All further experiments were conducted with the probe embedded into the smaller test bed. This allowed for EFNMR to be conducted as the small test bed did not include ferrous materials in its construction.

3.2 T₁ measurements

Fig. 2 shows the T_1 measurements of both the thick sludge and thin sludge samples. T_1 for the thick sample was 1498 ms whereas for the thin sample it was 2728 ms. This is approximately a factor of 1.8 for the two clog states, showing a clear difference.



Fig. 2: T_1 measurements of the thick sludge and thin sludge samples taken using a T_1 in the polarising field pulse sequence (discussed earlier) with 32 averages. The errors in the normalised integral of the thin sample data and the polarising time for both data sets are insignificant and therefore those error bars are not visible on this graph.

3.3 T₂^{eff} Measurements

 T_2^{eff} measurements were taken using a CPMG sequence on both samples, with the results displayed in Fig. 3. The difference between T_2^{eff} values is a factor of 1.9, making it marginally more pronounced than for the T_1 values. Thick sludge gave a T_2^{eff} value of 630 ms and the thinly clogged sample gave a of 1212 ms.



Fig. 3: T_2^{eff} measurements taken using the EFNMR probe with a CPMG sequence, 32 echoes, 32 scans. The integral is normalised to the first data point. The errors in the normalised integral and the echo time are insignificant and thus error bars are not visible on this graph.

4. Conclusions

The EFNMR probe presented here has been shown to be suitable for determination of the internal clog state of a constructed wetland, with measurements being performed while embedded within an onsite test bed.

Clear variation has been observed between the two clog state samples, through both the T_1 and T_2^{eff} measurements, though it has been shown that there is a minor difference in sensitivity to clogging between the two parameters. The T_2^{eff} results were shown to provide a factor of 1.9 distinction between the samples compared to a factor of 1.8 when looking at T_1 results.

An issue identified with the previously published designs was that they could become clogged to the point where it was no longer representative of the wetland as a whole. However, due to the bore of this system being many times larger than that of the gravel used in the wetland borehole clogging should be a rare occurrence.

A further limitation of the EFNMR design comes from the fact that wetlands have an active flow: As the Earth's field probe needed to be aligned in respect to the Earth's magnetic field lines, in a non-static wetland the bore of the system may not be aligned along the flow regime of the wetland. This might lead to distorted clogging measurements.

The major restriction identified when using the EFNMR is its dependence upon the homogeneity of the local field. As shown when experiments were performed at the IBC wetland, the significant presence of ferrous materials can prevent EFNMR from working. This would limit the materials that could be used in wetland module construction.

Further work should be performed including embedding a probe for a long duration into an operational wetland.

Acknowledgment

The research leading to these results was funded by the European Union's Seventh Framework Programme managed by the REA – Research Executive Agency <u>http://ec.europa.eu/research/rea(FP7/2007_2013)</u> under project reference 606326.

References

[1] R.H. Morris, M.I. Newton, P.R. Knowles, M. Bencsik, P.A. Davies, P. Griffin, G. McHale, Analyst 136(11) (2011) 2283-2286.

[2] M. F. Shamim, M. Bencsik, R.H. Morris, M.I. Newton, Microporous and Mesoporous Materials 178 (2013) 48-52.

[3] P. Griffin, L. Wilson, F. Cooper, Proceedings of the 11th Int Conf. on Wetland Systems for Water Pollution Control, India, (2008) 419–426.

[4] R. H. Kadlec, S. Wallace, Treatment Wetlands (2nd edition), CRC Press, Boca Raton, 2008.

[5] I.I. Rabi, J.R. Zacharias, S. Milkman, P. Kusch, Physical Review 53 (1938) 318.

[6] C.A. Michal, Meas. Sci. Technol., 21 (2010), 105902.

[7] A. Mohorič, J. Stepišnik, Progress in Nuclear Magnetic Resonance Spectroscopy 54 (2009) 166-182.

[8] H. Dong, Y.L. Wang, S.L. Zhang, Y. Sun, X.M. Xie, Superconductor Science and Technology 21 (2008) 115009.

[9] S. Meiboom, D. Gill, Review of Scientific Instruments 29 (1958) 688-692.

[10] R.H. Morris. M.I. Newton, M. Bencsik, P.R. Knowles, P.A. Davies, P. Griffin, Sensors IEEE, New Zealand (2009) 1733-1737.

[11] T. Hughes-Riley, Z. Xu, F. Hill-Casey, M.I. Newton, R.H. Morris, The 12th International Bologna Conference on Magnetic Resonance in Porous Media, New Zealand, (2014).

[12] T. Hughes-Riley, M.I. Newton, J.B. Webber, J. Puigagut, E. Uggetti, J. Garcia, R.H. Morris, The 12th International Bologna Conference on Magnetic Resonance in Porous Media, New Zealand, (2014).