ADVANCES IN CLOG STATE MONITORING FOR USE IN AUTOMATED REED BED INSTALLATIONS

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Abstract

Constructed wetlands are a popular form of waste-water treatment that have proliferated across Europe and the rest of the world in recent years as an environmentally conscious form of waste water treatment. The ability to monitor the conditions in the bed and control input factors such as heating and aeration may extend the lifetime of the reed bed substantially beyond the ten year lifetime normally reached. The Autonomous Reed Bed Installation (ARBI) project is an EU FP7 initiative to develop a reed bed with automated control over input parameters based on readings taken from embedded sensors. Automated remedial action may improve bed treatment efficiency, and prolong the life of the bed and avoiding the need to refurbish the bed, which is both time consuming and costly. One critical parameter to observe is the clog state of the reed bed, as this can severely impact on the efficiency of water treatment to the point of the bed becoming non-operable. Magnetic resonance (MR) sensors can be a powerful tool in determining clogging levels, and has previously been explored in the literature. This work is based on a conference paper (2nd International Conference "Water resources and wetlands", 2014) and details magnetic sensors suitable for long-term embedding into a constructed wetland. Unlike previous studies this work examines a probe embedded into a wetland.

Keywords: constructed wetlands, waste water, magnetic resonance, clogging, sensor, monitoring

1. INTRODUCTION

Constructed wetlands are an environmentally considerate means of water treatment. After the initial invasiveness of the reed beds installation,

the bed will over time integrate with its surrounding ecosystem. As such these systems have gained popularity and have proliferated across the globe. The basic design is uncomplicated, where a gravel matrix is used to create a porous structure for reeds to grow in. The reed root and rhizome network provides a substrate where microorganisms can grow as biofilm on the bed media. Under optimal conditions these microorganisms remove approximately 90 % of pollutants from waste water, with the remaining pollutants being dealt directly by the plants.

Effluent is then trickled through the reed bed and will come out the other side with a number of undesirable components removed, such as ammonia and phosphorus (Fig. 1). Effluent is not exclusively domestic sewage with reed beds being employed to filter and purify water for wastewater in mines as well as landfill leachate and air strip run-off (Adeola *et al.* 2009; Cooper, 2007).
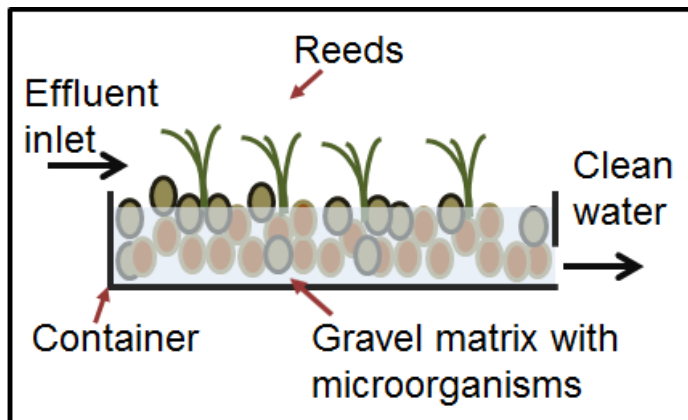

Fig. 1. Basic schematic of a sub-surface reed bed. A gravel matrix has reeds planted in it and effluent is flown through. A combination of the reeds and microorganisms remove unfavourable material from the effluent, resulting in clean water.

Once operational a reed bed ideally should require little maintenance. After effluent flow has begun, the simplest design can allow for a wetland to operate with no external interference for many years: original predictions estimated a lifetime of a bed to be in excess of 50 years (Conley *et al.* 1991) however practical experience has seen lifetimes as short as 10 years (Griffin *et al.* 2008).

Work presented here was partially reported as a conference paper at 2nd International Conference "Water resources and wetlands", 2014 (Hughes-Riley *et al.* 2014a). This research was conducted as part of the

Automated Reed Bed Installation (ARBI) project, an EU FP 7 funded initiative to design an automated constructed wetland module. The ability to control the environment in the reed bed may extend lifetime, and possibly increase efficiency. For example, reed bed efficiency decreases in the winter months as it is a less favourable environment for the microorganisms and reeds (Kadlec&Wallace, 2009).

The main limiting factor to a beds lifetime is that over time the pores in the gravel matrix become clogged with microorganisms and particulate matter, severely reducing the reed beds efficiency to treat water, and ultimately rendering it inoperable (Rousseau et al., 2005). The process of reconditioning the bed after such a time involves removing the bed material (gravel) and either replacing or washing it (Nivala et al., 2012). This is both time consuming and costly. Indirect measures for clogging assessment include the analysis of accumulated solids in filter media (Caselles-Osorio et al., 2007), system hydrodynamics by means of tracer tests (Bowmer, 1987) and hydraulic conductivity measurements (Knowles et al., 2010; Pedescoll et al., 2009). However, acting upon a prohibitively clogged bed can only be done in a timely and efficient manner if the internally clogging conditions of the reed bed can be monitored and understood. Work presented here will look at monitoring clogging levels in reed beds. Sensors will be fitted to each module to monitor the clogging level, oxygenation, and temperature of each bed. This information will be fed into a control system which will determine where and when action is needed.

In a laboratory environment it has been shown that the magnetic resonance (MR) relaxation parameters T_1 and T_2^{eff} are sensitive to the level of clogging on extracted wetland samples (Morris *et al.* 2011; Shamim *et al.* 2013). MR utilises nuclear spin which is an intrinsic quantum property. Spins align in a magnetic field and are excited with a radio frequency coil, this both moves the spins into a plane perpendicular to the magnetic field, and results in the spins precessing. Precession of the spins within the coil induces a current; this is the signal that is collected (Rabi *et al.* 1938; Bloch, 1946). Diverse manipulation of spins allows for the collection of different information about a substance.

Relaxation deals with excited spins returning to a thermal equilibrium state; T_1 (spin-lattice) relaxation deals with spins returning to the same orientation as the magnetic field, and T_2 (spin-spin) relaxation regards precessing spins falling out of phase with one-another. T_2^{eff}, discussed in this work, regards spin dephasing where various factors are responsible, not just spin-spin interactions.

As well as determining clog state MR can be used to analyse components of clogging and therefore better understand the problems being

experienced in the reed bed (explored in model systems by Bencsik *et al.* 2013). MR sensors used in these studies are designed to detect protons (^{1}H) and therefore most MR measurements taken on wetland sludge will be on the water component. However the MR relaxation behaviour of water will be different depending on other factors, such as the water containing particulate matter. Different relaxation behaviour can be extracted from T_1 and T_2^{eff} data using a polyexponential fitting and may be utilized to determine important factors for the reed beds health. The scope for using this to separating out components, such as biofilm has been explored to some extent elsewhere (Hughes-Riley *et al.* 2014b).

Various sensor designs have been investigated for their use with the ARBI module. A pilot study was also conducted using Earth's field nuclear magnetic resonance (EFNMR; Hill-Casey *et al.* 2014 *in press*). T_1 and T_2^{eff} measured using EFNMR were sensitive to clog state, and the probe could be successfully embedded into a gravel matrix filled with water. However when attempts were made to embed the probe into a wetland module the surrounding ferrous cage introduced inhomogeneity to the localised Earth's field, making measurements impossible. Building the outer case of a wetland module from a non-metal material is likely impractical at the size needed for sufficient treatment, making the use of EFNMR for the desired application non-ideal.

Unilateral magnet arrangements have been explored for this application in the laboratory, including using the stray field of an arrangement of four magnets and bar magnet (Hughes-Riley *et al.* 2014b; Hughes-Riley *et al.* 2014c *under review*; Hughes-Riley *et al.* 2014d). Unilateral designs are preferred over an internal volume design as they are less disruptive to the flow-path of effluent and cannot become physically clogged by gravel. The four-magnet sensor showed promise in a lab environment, however further development (unpublished) to deploy the sensor into a reed bed has been problematic. Signal intensity is not sufficient to take T_2^{eff} measurements in a timely manner. Another issue is that in its current configuration only about 2 mm above the probe can be explored. This small volume (~1000 mm^3) is not ideal as it may not be representative of the health of the reed bed overall.

Subsequently this work re-examines the use of a Helmholtz magnet assembly which probes a far larger volume. The sensor used in this work was small (sensitive region of ~10 cm^3), however up-scaling should not prove to be a significant challenge and is only limited by the size of available magnets. Ongoing work is investigating the usefulness and practicalities of a larger sensor. Previous reports have either used mock systems constructed in a laboratory or samples extracted from an actual

wetland and taken to a laboratory for investigation, here for first time MR measurements taken *in situ* from a reed bed are presented. This work also presents a brief study comparing clog state sensitivity of T_1 and T_2^{eff} using a Helmholtz permanent magnet arrangement of this design.

2. METHODS
2.1 Prototype constructed wetland for MR testing

A static wetland module was constructed at Nottingham Trent University with the express purpose of testing MR sensors (previously described elsewhere; Hill-Casey *et al.* 2014 *in press*). An intermediately bulk container (1.02 m x 0.92 m x 0.90 m; DV Containers Ltd, Wrexham, UK) was used as the basis of the module. The container was filled with gravel (average length = 9.6 mm; Travis Perkins Trading, Bulwell, UK), water, and had common reeds (*Phragmites australis*) planted in it, making the module a close representation of a real operational wetland (Fig. 2). The wetland did have an outlet pipe to allow for liquid to flow through; however this was not utilized during these experiments. Effluent was also not present in this system, as it would be in an operational wetland.

Fig. 2. An overhead photo of the prototype wetland at the Nottingham Trent University. The basis of the module was an IBC container.

2.2 MR sensor design

A Helmholtz MR sensor, first presented elsewhere (Hughes-Riley *et al.* 2014d) and very similar to a design by Morris *et al.* 2011, was used in this study. The magnet arrangement comprised a pair of neodymium

magnets (Fig. 3; height = 20 mm, radius = 17 mm; Magnet Monster, Flensburg, Germany). The magnets were separated by 20 mm (slightly over the magnet radius) and arranged with anti-parallel polarization. This created a region of homogeneous magnetic field where an eight-turn solenoid coil was installed.

     The solenoid was attached to a tuning board with three capacitors, a 12 – 100 pF variable capacitor for matching (Johanson Manufacturing, New Jersey, USA) and a 12 – 100 pF variable capacitor and a single 55 pF fixed capacitor for tuning the resonant circuit to 13.87 MHz, the required frequency for the field generated. A slight modification was made to the circuit before the experiments comprising Fig. 7 as part of the ongoing improvement of the sensor. As the change was to the circuit only this will not have affected T_1 or T_2^{eff} taken by the sensor.

     The sensor had to be water-tightened so that it could be embedded into a wetland. Therefore the sensor was coated with a silicon elastomer potting compound (RS Components Ltd., Northhants, UK). Laboratory tests confirmed that this sufficiently water-tightens the sensor to allow for operation. This method is likely unsuitable for long term embedding due to impairment that may occur to the silicon coating over time or physical damage from motion during transport.
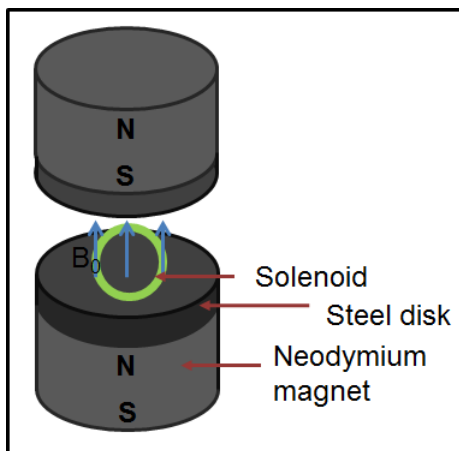

Fig. 3. **Helmholtz sensor design.** Magnets are separated by 20 mm, in the gap there is a homogeneous magnetic field in which the solenoid is installed. Steel disks are place on each magnet to reduce the field gradient.

2.3 Magnetic resonance protocol

     For this study most experiments utilized the Prospa v3.12 software that was used to drive a Kea 2 spectrometer (Magritek, Wellington, New

Zealand) to collect magnetic resonance measurements. All measurements were taken using CPMG sequences (Meiboom & Gill, 1958). For T_2^{eff} measurements a CPMG was taken with a single parameter set, and echo integrals were re-fit using Igor Pro v6.3 (WaveMatrics, Oregon, USA). For T_1 measurements CPMG sequences were run with different repetition times. A CPMG was used so that the echo train could be summed, reducing the number of repeat experiments needed. Summed echo integrals were normalised and then fitted to a mono-exponential curve. All errors specified for T_1 and T_2^{eff} values are given as the error on the fitting provided by Igor Pro.

The experiments shown in Fig. 7 used TNMR software and an Apollo spectrometer (Tecmag Inc., Texas, USA). Fittings were conducted using Matlab (The MathWorks, Inc., Massachusetts, USA) to provide the final T_2^{eff} values.

2.4 Sample preparation

Samples for additional laboratory validation experiments (ARM, Rugeley, UK) were stored in either a 50 mm section of acrylic tube (i.d. = 10 mm, o.d. = 12 mm) or a modified 2 ml syringe (BD Plastipak, Oxford, UK; i.d. = 9 mm, o.d. = 10 mm) filling most of the coil region of the sensor. To calculate the mass of the dry solids in each sample, samples were weighed, then dried in a convection oven (Binder, Tuttlingen, Germany) and finally re-weighed using an electronic balance (Type 120-4M, Kern & Sohn GmbH, Balingen, Germany).

3. RESULTS

Fig. 4 shows a T_2^{eff} measurement taken in the Nottingham Trent University prototype reed bed. Four sets of experiments with of 512 averages each were taken in sequence so that a standard deviation could be taken, giving an estimate for the error on each point. Overall these measurements took approximately 35 minutes. Due to the slow moving nature of the sludge in the reed bed, measurements taking several hours are possible. Therefore additional signal averages are possible, which would reduce the errors for each averaged echo integral and therefore the accuracy of the exponential fitting. A longer experimental time would also allow for a slower repetition between experiments (a repetition time of 1s was used,

later experiments showed that a time exceeding 5s would yield far superior results).
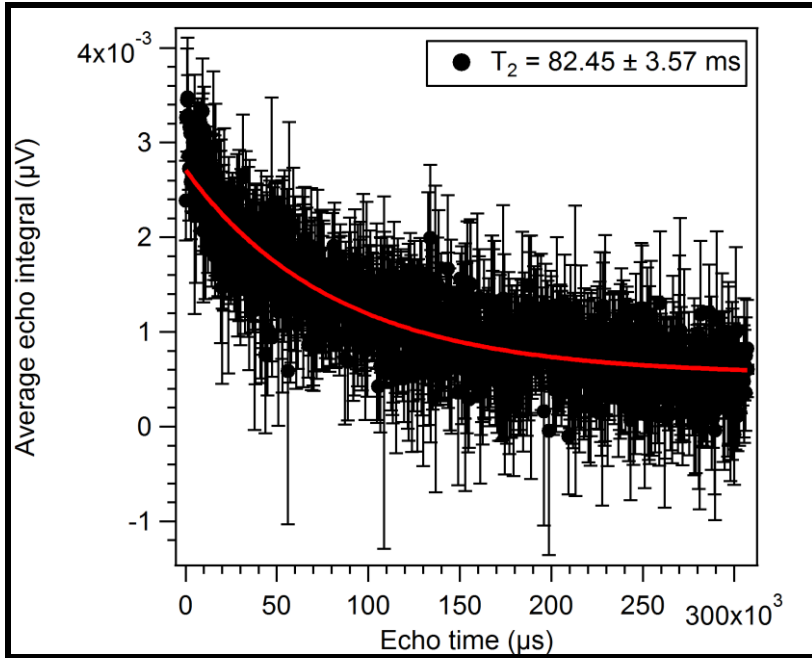

Fig. 4. T_2^{eff} measurement taken using the embedded sensor. Echo time = 300 µs, repetition time = 1000 ms, 2048 averages.

With the successful collection of a T_2^{eff} measurement validating the use of the sensor in a reed bed, a T_1 measurement was recorded, as shown in Fig. 5 (black markers). Also displayed in Fig. 5 are measurements of two other samples recorded in the laboratory environment on the same sensor. Analysis of the reed bed material showed that 0.15 % of its mass was dry solids. This would be expected as the effluent has not been run through this bed. The percentages of dry solids for other samples are shown on the graph.
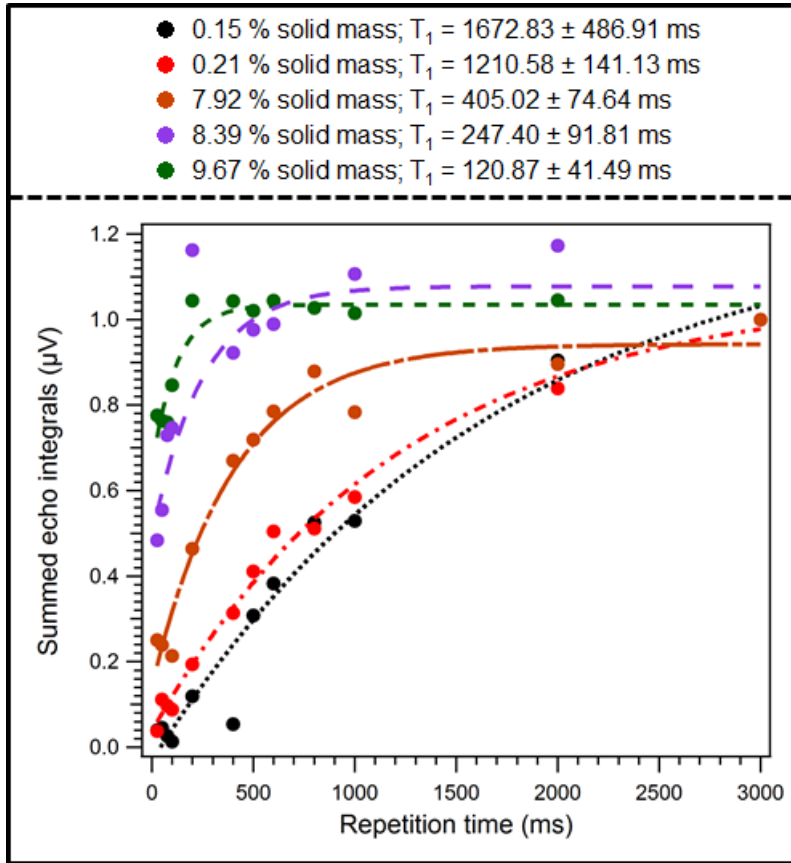
Fig. 5. T_1 measurements taken using the embedded sensor, with additional laboratory measurements. Echo time = 300 µs, 512 averages. Errors in the normalised integral sum are small, and therefore have not been included in the graph. A systematic error occurred for the final two points of the 8.39 % solid mass curve at 200 ms (shown) and 800 ms (not shown). These points have not been included in the fitting. The 75 ms point is omitted from 7.92 % solid point due to an equipment failure.

As expected, additional solid material (i.e. clogging) results in a shorter T_1. The relationship between the T_1 measurement and percentage of solid material in the sludge has been plotted (Fig. 6). A linear relationship is observed. This is expected based on the work of Morris *et al.* 2011, where hydraulic conductivity was shown to have a linear relationship with T_1 relaxation. Hydraulic conductivity should be proportional to the percentage of solid material, and this work appears to confirm that.
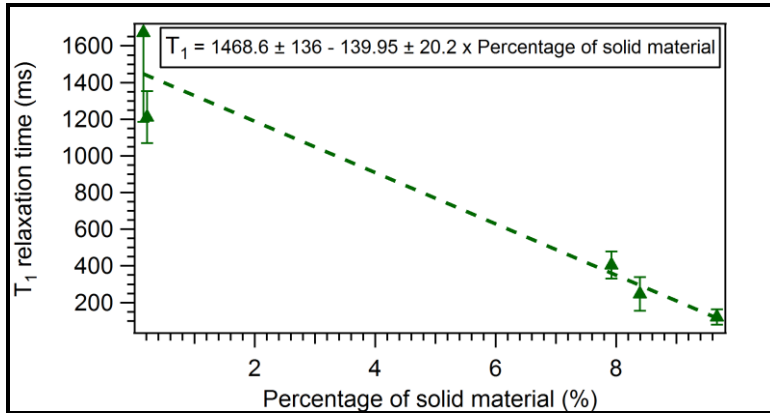
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Fig. 6. T_1 plotted against the percentage of solid material in the sludge sample.
A linear relationship is observed. Errors in the percentage of the solid material due to weight are minimal, and therefore error bars are too small to plot.

As the T_2^{eff} time is short when compared to T_1 time, it is unlikely that T_2^{eff} will be a useful indicator of clog state in wetlands, and certainly not the preferable gauge when using a sensor such as the one described in this work: T_2^{eff} is short due to poor field homogeneity of a permanent magnet system. T_2^{eff} measurements take less time to collect than T_1 as only one, and not multiple CPMG measurements need to be made. As a result it might prove useful to be able to use a T_2^{eff} measurement as a rough gauge of clog state when sensors are initially embedded into a system. Regardless, for completeness, a short laboratory study comparing T_2^{eff} to the percentage of solid material in a sample was conducted and is presented in Fig. 7.
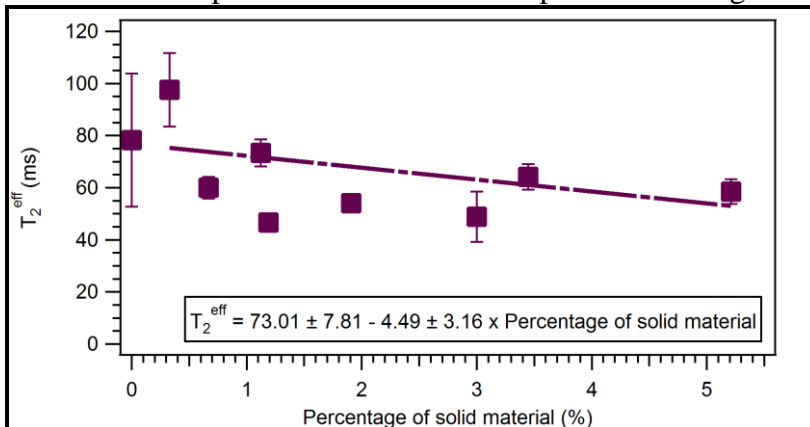

Fig. 7. T_2^{eff} plotted against the percentage of solid material in the sludge sample. For each data-point: Echo time = 300 µs, repetition time = 1000 ms, 1024 averages. A linear relationship is observed. Errors in the percentage of the solid material due to weight are minimal, and therefore error bars are too small to plot.

As expected T_2^{eff} is shorter for samples with a greater level of clogging (Bencsik *et al.* 2013). While this relationship does exist the difference in T_2^{eff} for the clogged and unclogged states are small, varifying that it is an unfavourable meassure of clog state on the presented sensor.

The error on the gradient of the fitting in Fig. 7 is substantial. Error on T_2^{eff} values are given as being the error on the exponential fitting and is generally minimal for clogged samples. Potenitial sources of error not accounted for here may be introduced by temperture effects however any temperture change would have been minimal (less than 5 °C). It is possible that errors in calculating the percentage of solid material may also contribute however this seems unlikely due to the superior linear fitting apparent in Fig. 6 (compared to that in Fig. 7). A better linear fitting could be obtained by repeating meassurements on different samples at other percentages of solid material.

4. CONCLUSION

An automated reed bed module will be able to use incorporated sensors to monitor the internal conditions of the bed and allow for real-time optimisation of environmental parameters by controlling factors such as aeration and heating. This should lead to an increased bed lifetime, and superior water treatment. The most important sensor is the MR probe as this is required to monitor clog state. An important development towards using these probes is to prove that sensors can work *in situ*, which has been shown for the first time for an MR probe in this work.

A short study exploring a comparison between T_1 relaxation and quantity of solids in samples has been conducted. As seen from previous studies increasing particulate matter results in shortening T_1 values (Morris *et al.* 2011).

T_2^{eff} has also been investigated. A small change in T_2^{eff} as a function of solid material in a sample is evidenced. The change is far smaller than the relationship observed for T_1 (about a factor of thirty more sensitive than T_2^{eff}) when using a sensor of this type, confirming that T_1 is the desirable measurement to record when monitoring reed bed clog level.

Further development of MR sensors is desirable. A large Helmholtz sensor or a unilateral magnet assembly would be less likely to become physically clogged and therefore inoperable, or unrepresentative, than the small internal volume design presented here.

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REFERENCES

Adeola, S., Revitt, M., Shutes, B., Garelick, H., John, H. & Jones, C., 2009, Constructed wetland control of bod levels in airport runoff, *International Journal of Phytoremediation*, **11**(1), 1-10, DOI: 10.1080/15226510802363220.

Bencsik, M., Shamim, M.F., Morris, R.H. & Newton, M.I., 2013, Monitoring accelerated clogging of a model horizontal sub-surface flow constructed wetland using magnetic resonance transverse relaxation times, *International Journal of Environmental Science and Technology*, **178**, 48-52, DOI: 10.1007/s13762-013-0336-7.

Bowmer, K.H., 1987. Nutrient removal from effluents by artificial wetland: influence of rhizosphere aeration and preferential flow studied using bromide and dye tracers. *Water Reserach* **21** (5), 591–599, DOI: 10.1016/0043-1354(87)90068-6.

Bloch, F., 1946, Nuclear Induction, *Physical Review*, **70**, 460-473, DOI: http://dx.doi.org/10.1103/PhysRev.70.460.

Caselles-Osorio, A., Puigagut, J., Segú, E., Vaello, N., Granés, F., García, D., García, J., 2007. Solids accumulation in six full-scale subsurface flow constructed wetlands. *Water Reserach* **41** (6), 1388–1398, DOI: 10.1016/j.watres.2006.12.019.

Conley, L.M., Dick, R.I. & Lion, L.W., 1991, An assessment of the root zone method of wastewater treatment, *Research Journal of the Water Pollution Control Federation*, **63**(3), 239 – 247, available online: http://www.jstor.org/stable/25043987 (accessed on 19/08/2014).

Cooper, P. 2007, The constructed wetland association UK database of constructed wetland systems, *Water Science and Technology*, **56**(3), 1-6, DOI: 10.2166/wst.2007.490.

Carr, H. Y., & Purcell, E. M. 1954, Effects of diffusion on free precession in nuclear magnetic resonance experiments, *Physical Review*, **94**(3), 630-638, DOI: 10.1103/PhysRev.94.630.

Hill-Casey, F., Hughes-Riley, T., Bradley, C.R., Newton, M.I., & Morris, R.H. 2014, Magnetic resonance relaxation measurements using earth's field magnetic resonance to assess the clog state of constructed wetlands, *Diffusion Fundamentals*; ***in press***.

Hughes-Riley, T., Newton, M.I., Webber, J.B.W., Puigagut, J., Uggetti, E., Garcia, J. & Morris, R.H. 2014a, Advances in Automated Reed Bed Installations, *2nd International Conference "Water resources and wetlands", 11 – 13 September 2014, Tulcea, Romania*.

Hughes-Riley, T., Xu, Z., Hill-Casey, F., Newton, M.I. & Morris, R.H. 2014b, Using Unilateral Sensors to Assess the Clog State of Constructed Wetlands, *12th International Bologna Conference on Magnetic Resonance in Porous Media (MRPM12), 9 – 13 February 2014, Wellington, New Zealand*.

Hughes-Riley, T., Webber, J.B., Newton, M.I. & Morris, R.H. 2014c, Magnetic resonance relaxation measurements using open-geometry sensors to assess the clog state of constructed wetlands, *Diffusion Fundamentals*; ***under review***.

Hughes-Riley, T., Newton, M.I. & Morris, R.H. 2014d, Temperature dependence of magnetic resonance sensors for embedding into constructed wetlands, *1st International Electronic Conference on Sensors and Applications, 1-16 June 2014*, DOI: 10.3390/ecsa-1-c006.

Griffin, P., Wilson, L. & Cooper, F., 2008, *Proceedings of the 11th Int Conf. on Wetland Systems for Water Pollution Control, 1-7 November 2008, Indore, India.*

Kadlec, R.H. & Wallace, S.D., 2009, *Treatment Wetlands*, CRC Press, FL, USA, 101p.

Knowles, P.R., Griffin, P., Davies, P.A., 2010. Complementary methods to investigate the development of clogging within a horizontal sub-surface flow tertiary treatment wetland. *Water Reserach* **44** (1), 320–330, DOI: 10.1016/j.watres.2009.09.028.

Meiboom, S., & Gill, D. 1958, Modified spin-echo method for measuring nuclear relaxation times, *Review of scientific instruments*, **29**(8), 688-691, DOI: 10.1063/1.1716296.

Morris, R.H., Newton, M.I., Knowles, P.R., Bencsik, M., Davies, P.A., Griffin, P. & McHale, G. 2011, Analysis of clogging in constructed wetlands using magnetic resonance, *Analyst*, **136**, 2283-2286, DOI: 10.1039/c0an00986e.

Nivala, J., Knowles, P., Dotro, G., García, J., Wallace, S. (2012) Clogging in subsurface-flow treatment wetlands: Measurement, modeling and management. *Water Research* **46** (6), 1625–1640.

Pedescoll, A., Uggetti, E., Llorens, E., Granés, F., Garcia, D., García, J., 2009. Practical method based on saturated hydraulic conductivity used to asses clogging in subsurface flow constructed wetlands. *Ecological Engineering* **35** (8), 1216–1224, DOI: 10.1016/j.ecoleng.2009.03.016.

Rabi, I.I., Zacharias, J.R., Millman, S. & Kusch, P. 1938, A New Method of Measuring Nuclear Magnetic Moments, *Physical Review*, **53**(4), 318-327, DOI: 10.1103/PhysRev.53.318.

Rousseau, D.P.L., Horton, D., Vanrolleghem, P.A., De Pauw, N., 2005. Impact of operational maintenance on the asset life of storm reed beds. *Water Science Technology* **51**(9), 243–250

Shamim, M.F., Bencsik, M., Morris, R.H. & Newton, M.I. 2013, MRI measurements of dynamic clogging in porous systems using sterilised sludge, *Microporous and Mesoporous Materials*, **178**, 48-52, DOI: 10.1016/j.micromeso.2013.04.025.