



The application of X-ray micro Computed Tomography imaging for tracing particle movement in soil

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ABSTRACT

Despite significant advances in recent years in the application of sensing and imaging technologies to the study of the hydraulic behaviour of soils, our understanding of how solutes and particulate matter move through soil is still limited and often based on idealised soil structures. The aim of this work was to use an appropriate proxy material, in this case (Decabromodiphenyl ether (DBDE)), to represent 1–2 µm sized chemical particles, frequently used in agricultural practices, in order to trace their spatial and temporal movement through a soil column. X-ray Computed Tomography (CT) was employed to map the 3D pore geometry and facilitate visualization of the concentration distribution of the highly X-ray attenuating proxy material as it was applied and subsequently leached through the soil over a 5 hour period. Simultaneously the soil eluate was collected from the bottom of each column and the concentration of proxy material measured using gas chromatography to compare with the imaged data. The method developed for visualizing the tracer material in 3D at the pore-scale was successful. The results demonstrated that the tracer material initially moved rapidly through the upper portion of the soil but subsequently became immobile, despite repeated elution with water as confirmed by imagery. Similar concentrations to those applied to the soil surface were not recorded at any point in time towards the bottom of the soil column. By visualizing the DBDE distribution in 3D it was possible to correlate the highest concentrations of the tracer material with the location of the smaller size pores (i.e. mesopores). Dynamic X-ray CT imaging of tracer materials has considerable potential as a proxy for the visualization of particulate agricultural chemicals that will assist with understanding how their transport behaviour varies both spatially and temporally.

1. Introduction

The UK National Action Plan for Sustainable Use of Pesticides (Defra, 2013) encourages the sustainable use of pesticides and highlights the importance of the life cycle of chemicals. As intensification of agriculture accelerates to meet increasing food demand, it is becoming increasingly important to understand precisely how agricultural chemicals interact with, and move through, soil (Carvalho, 2006). Such agrochemicals are known to cause contamination in surrounding ecosystems when applied excessively (Gamon et al., 2003), therefore development of sustainable management practices are essential to achieve both increased crop production and decreased pollution. Improved understanding of agrochemical behaviour in soil will contribute not only to the alleviation of environmental impact but also to the possibility for improvement in controlled placement leading to increased

bioefficacy and reduced use rates.

The movement of agrochemicals through soil is strongly influenced by the physical, chemical and biological characteristics of the soil and plants, as well as those of the chemical itself. This intricate combination of influences makes understanding how an agrochemical moves through soil both spatially and temporally extremely difficult. Traditional methods for examining solute flow through soil have focused on conventional bulk ex-situ chemical analysis of soil and eluate (Andreu and Picó, 2004). Continuous soil column tests have been routinely used to produce breakthrough curves to characterise the behaviour of the chemical of interest (Brusseu et al., 1991). Such techniques have also been combined with mathematical models to predict chemical movement e.g. Magga et al. (2012). In combination these approaches have been used to provide detailed information on the fate of chemicals applied to soil, however research in this area has been

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hindered by a lack of real time data, particularly from a three dimensional (3D) perspective. One technique that has shown promise in this regard is X-ray Computed Tomography (CT) which allows the visualization of the internal fabric of a porous media such as soil (Akin and Kovsec, 2003). With CT it is possible to non-destructively visualize the internal pore geometry of the structured media and, with repeating scanning, characterisation of the active processes in situ can be attempted assuming the solute/particulate material can be distinguished from the soil background. X-ray attenuation is linked to the material elemental composition; hence elements with a high atomic number have a higher X-ray attenuation and are potentially good candidates to act as a tracer of solute flow in a soil (Jenssen and Heyerdahl, 1988; Helliwell et al., 2013).

The use of tracers in X-ray CT imaging is routine in medical research to improve the contrast of blood vessels and tumours and a wide range of tracers have been applied such as gold nanoparticles (Nune et al., 2009) and iodinated agents (Lusic and Grinstaff, 2012). There has been limited research into the use of tracers to visualize solute movement in soil (Anderson et al., 1992; Clausnitzer and Hopmans, 2000; Anderson et al., 2003; Luo and Lin, 2008). A key limitation is that X-ray CT is best suited to the visualization of motionless objects (Luo and Lin, 2008) as the scan process generates spatial data of the entire sample over the whole course of the scan, with any effect of change in the material morphology typically corrected at the end of the scan. However with the repeated scanning of the same sample over the shorter timescales that is now achievable (as low as 5 min) (Tracy et al., 2010), it is possible to include time as a variable providing the solute transport is not too rapid. Mooney (2002) used X-ray CT to visualize soil water movement, identifying preferential flow paths, in soil columns by spatial subtraction of images pre- and post-wetting. Anderson et al. (2003) combined X-ray CT imaging with an iodide breakthrough experiment, and was able to compare the results with laboratory derived measurements of saturated hydraulic conductivity. Luo and Lin (2008) passed an iodide tracer through a soil column (30 cm) over a 23 h period and repeatedly scanned the soil column to demonstrate the spatial variation in hydraulic behaviour of the soil.

To date there has been limited research concerned with tracking the movement of particles or suspensions in soil using X-ray CT at the pore (μm – mm) and column (mm – cm) scales. In this work we tested a novel proxy for agrochemical suspension concentrates, Decabromodiphenyl ether (DBDE), chosen due to its X-ray attenuation properties and similar suspension behaviour compared to routinely used compounds such as Chlorothalonil, a broad spectrum fungicide which has an average particle size of $2\ \mu\text{m}$ (Curry et al., 1998). The objectives of this investigation were to determine the efficacy of DBDE as a general proxy tracer for particulate agrochemical movement in soil via CT imagery and to investigate if X-ray attenuation can be used as a surrogate for tracer concentration over time and following elution by water.

2. Materials and methods

2.1. Soil column preparation

Soil was obtained from the '18 acre' field site at Syngenta Jealott's Hill International Research Centre, Bracknell, UK. The soil was a brown earth with a sandy clay loam with 49% sand, 27% silt, and 24% clay. Soil was sieved to $< 2\ \text{mm}$ and wetted to 17% moisture content; centrifuge tube filters were used as soil columns (23 mm diameter, 40 mm length), with holes at the bottom to allow free drainage of soil solution. The outer tube holder was used to hold the columns during scanning and to collect soil eluate. Six tubes, each containing different concentration of the tracer suspensions used for calibration during the image analysis process, were secured to the inside edge of each column, before the soil was packed into the columns at a bulk density of $1\ \text{g cm}^{-3}$. To examine tracer movement through 40 mm of soil, triplicate columns were packed to heights of both 10 and 40 mm and the

bottom 10 mm of each of these columns scanned to represent tracer movement at the top and bottom of a 40 mm soil section, hence six columns in total. Eluate was collected from the bottom of each section after each scan in a small container added to the bottom of the sample for this purpose; the columns were left to saturate overnight the day before scanning.

2.2. Tracer material and calibration

DBDE (from Sigma-Aldrich) was chosen as the tracer material due to its high X-ray attenuation capacity arising from the high electron density of bromine. It was milled using Yttria Stabilized Zirconia Beads (0.3 mm) to generate particles with a size range of 0.1–6 μm in diameter and a volume mean diameter (VMD) of 2–3 μm suspended in DI water with 5% Soprophor® 3D33 (tristyrylphenol polyethoxyester phosphate ester, Solvay) as a dispersing agent. An initial test was undertaken to confirm that DBDE was detectable in soil and then to identify the appropriate concentration for application (cross-sectional X-ray images of the tracer and tracer embedded in soil are shown in Supplementary Fig. 1). From this a DBDE suspension was prepared with a concentration of $200\ \text{mg ml}^{-1}$ and sonicated for 1 h before use. DBDE particle suspensions were then prepared with concentrations of 25, 50, 100 and $200\ \text{mg ml}^{-1}$ and Kelzan S® (CP Kelco), a natural xanthan gum based thickener, was added to reduce settling of particles over time. The suspensions were pumped into rubber tubing (0.89 mm diameter) using a peristaltic pump after which the tubing was cut to 40 mm length and sealed with PVA glue, before being secured to the side of each column with adhesive tape.

2.3. X-ray CT scanning procedures

Each column was scanned using a Phoenix Nanotom® X-ray μCT scanner (GE Measurement & Control Solutions, Wunstorf, Germany). Discrete regions of interest (ROI) only were scanned at settings of 120 kV and 90 μA with a 0.1 mm copper filter. Projections ($n = 1200$) were captured over a 30 minute time period for each column, with a pixel resolution of 12.5 μm , as such we do not resolve individual particulate material below this. Each column was positioned so that only the base of the column (10 mm) was scanned i.e. the region 0–10 mm was scanned for half the treatments and the region 30–40 mm for the other half. This approach allowed us to directly relate the associated physical and chemical measurements of these regions to the obtained X-ray images. Each of the six columns was scanned before, and after, the application of 1 ml of DBDE ($200\ \text{mg ml}^{-1}$) and then scanned a further 10 times continuously over a 5 h period. At the beginning of each post-DBDE scan 1 ml of water was added to the top of the column and the soil eluate collected from the tube holder at the end of each scan. A total of 72 CT scans were undertaken.

2.4. Chemical analysis of DBDE

Gas chromatography was used to determine the DBDE concentration in the eluate collected from the bottom of each soil column after each scan (i.e. 30 min intervals). The aqueous soil eluate samples containing DBDE particles were dried using a Genevac HT-4X centrifugal vacuum evaporator. Toluene was added to the dried deposit to dissolve the DBDE after which the samples were centrifuged to remove soil particles. Samples were diluted further in toluene as required before analysis by gas chromatography using an Agilent 6890 Series GC System, equipped with cool on column injection system, a fused silica DB-5 column (15 m \times 0.25 mm id; 0.25 μm film thickness) (Agilent J&W, Agilent Technologies) and an electron capture detector. The peak area for DBDE was used to calculate the DBDE concentration of the sample, with reference to a calibration standard. By determining the concentration of DBDE collected from the eluate of each section it was possible to construct a chemical breakthrough curve for each section of

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