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Simple models for the release kinetics of dissolved organic carbon from woody filtration media

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ABSTRACT

The mechanisms and kinetics of dissolved organic carbon (DOC) release from woody materials (pine, hardwood and compost) under non-equilibrium leaching conditions was examined through batch and column tests. Mechanistically based kinetic models (first and second order) had a low predictive power for DOC release compared to those based solely on regression (Elovitch, power law). The DOC release data showed a bi-phasic response, with an early period of rapid release (<24 h) controlled by film diffusion followed by a slower rate controlled by intra-particle diffusion. After flow interruption, DOC release was primarily controlled by intra-particle diffusion; however, the specific rate parameters generally varied with each flow cycle and between different wood types.

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1. Introduction

There is widespread interest in finding beneficial reuse opportunities for woody organics in order to promote diversion of these materials from landfill. Environmental applications include using woody organics in subsurface barriers to treat acid mine drainage, stormwater runoff and groundwater contamination. This water treatment is achieved through the well recognised ability of woody organics to sorb dissolved metals (Seelsaen et al., 2006, 2007; Sud et al., 2008) as well as petroleum hydrocarbons (Hong et al., 2006) from percolating water. Woody organics also has the ability to release dissolved organic carbon (DOC) which can act as an electron donor and therefore, drive the microbial processes which underpin nitrogen removal (Gibert et al., 2005) and the degradation of chlorinated and aromatic hydrocarbons (Shen and Wilson, 2007). DOC release may involve both the short-term release of pre-existing DOC from the substrate as well as substrate degradation of insoluble organic carbon which then generates further DOC. DOC release kinetics is therefore, critical knowledge for a number of subsurface barrier applications involving woody filtration media. Further research is needed to study controls on DOC release from plant litter (Hagedorn and Machwitz, 2007) and mulch under conditions relevant to subsurface barriers (Ahmad et al., 2007; McLaughlan and Al-Mashaqbeh, 2009). While there has been significant research into DOC as a component in soils (Münch et al., 2002), there is considerably less published work about DOC leaching under the sustained hydraulic leaching conditions relevant to the engineering applications of mulch-based filtration media. Simple approaches are needed describe DOC release kinetics from woody materials.

Whilst there has been little work directly on woody filtration media, previous studies in soils have shown that DOC release is generally a multi-phasic process where there is an initial rapid release followed by a rapid decline (Cao et al., 1999; Reemtsma et al., 1999; Schaumann et al., 2000). This has described in terms of the mobilisation of fractions or pools of dissolved organic carbon and their associated rate constants. In common with many soil chemical phenomena these processes have been described in terms of kinetic equations as well as diffusion models (Aharoni et al., 1991). Whether these DOC pools and the models used to describe DOC release from soils are applicable to woody filtration materials have yet to be studied.

The most appropriate models or equation for fitting DOC release data from plant matter to soil has yet to be agreed. Adequate fits of first-order models to DOC release for leaf litter, bark and twigs have been found by some authors (O'Connell et al., 2000) while others have data which show an adequate fit for some leaf litter types but not for others (Wallace et al., 2008). For a continuously leached soil Schaumann et al. (2000), found that while a power law function did not fit early time data (<15 min) it did fit later time data (15–200 min). They reported the power law function fitted the data DOC concentration data better than a one or two exponential decay function. Kaiser and Zech (1998) found while time-dependency of the release was described adequately by the Elovich and power model equations, in a few cases the DOC release from soil was represented better by the intra-particle diffusion equation.

The goal of this paper is to investigate the nature and rates of release of dissolved organic carbon from woody filtration materials. This was achieved by measuring the quantity of dissolved or-

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ganic carbon released using techniques over a short period (24 h). Continuous-flow experiments were also undertaken over a longer timescale (<6 days) to determine the DOC release behaviour under the high flow non-equilibrium conditions relevant to stormwater treatment device design. Several commonly applied predictive kinetic equations and a kinetic-diffusion model was evaluated to determine if simple models could be used to describe the DOC release process under these conditions.

2. Experiment methodology

2.1. Materials

Pine, hardwood and compost (yard waste) were selected for testing due to their common use as woody filtration materials. The received pine (Superior Pine Chip, Soilco Pty Ltd.) and hardwood (Hardwood chips, Soilco Pty Ltd.) material were furthered sorted to remove bark fragments. The compost (Composted Leaf mulch, Australian Native Landscapes) was compliant with the Australian Standard for composts, soil conditioners and mulches (AS4454, 2003) and consisted of a screened composted mulch (nominally 10–35 mm) sourced from garden organics. Garden organics include putrescible garden organics (grass clippings); non-woody garden organics; woody garden organics; trees and limbs; stumps and root balls. All woody filtration materials were then dried at 45 °C for 48 h and then ground using a hammer mill (Model No. ADEB80N2, John Morris Pty Ltd.) until the required particle size range was obtained. This study used material which passed through a 2.36 mm sieve but was retained over a 1.18 mm mesh sieve (referred to as 1.18 mm material). All these materials were subsequently dried at 45 °C for 48 h prior to use. All weights reported in the paper are as dry matter (see Table 1).

2.2. Analytical methods

Water samples were measured for electrical conductivity (EC) and pH (TPS WP-81). Dissolved organic carbon (DOC) for the high DOC samples (first four samples) was determined in duplicate with a Phoenix 8000 TOC analyser, while the rest of samples were calculated from UV absorbance (Shimadzu UV-1700). The UV derived DOC values (referred to in this paper as DOC) was calculated for each material using a regression between UV_{280} and DOC based on values obtained from the sequential batch leaching experiment (McLaughlan and Al-Mashaqbeh, 2009). The correlation co-efficient for hardwood and compost was 0.97 and for pine was 0.99. Total carbon and nitrogen for all solid materials were determined in duplicate using a Truspec Carbon Nitrogen determinator. Ash content was measured using the method for total volatile solids (APHA/AWWA/WEF, 1998) at 550 °C. All water used in the study was distilled unless otherwise specified. The distilled water had a pH of 4.8 which is representative of the lower end of Australian field data for rooftop runoff (Engineers Australia, 2006). The cumulative release data is reported as the sum of all DOC released until that particular time. Since microbial uptake was not explicitly inhibited in this study, the reported DOC reflects abiotic release minus an undetermined amount of microbial uptake. All plasticware and glassware in contact with samples was acid washed for 24 h with 0.1 M HNO_3 and rinsed with Milli-Q water, unless otherwise noted

Table 1
Properties of woody material.

Woody material	Ash (%)	C/N	TOC (mg/g)
	Ratio		
Pine	0.3	428	480
Hardwood	0.2	374	487
Compost	10.9	32	382

2.3. Batch leaching tests

Numerous batch extraction methods have been reported in the literature and used to obtain water-soluble organic carbon values. Salt extractable organic matter fractions have commonly been used as measures of dissolved organic carbon. Salt extracts have been used for ease of extraction since they cause flocculation of soil particles (Haynes, 2005). Extractants used include NaCl, $CaCl_2$, K_2SO_4 and distilled water. To evaluate their performance against other types of extraction techniques, the following extractants were used in this study; water, K_2SO_4 (0.5 M), NaCl (1 M) and $CaCl_2$ (0.01 M). The 24 h batch leaching test was performed in duplicate at a liquid to solid sample ratio of 10:1 L/kg using a 150 g oven dried (100 °C) sample, shaken on an orbital shaker at 200 rpm for 24 h using the extractants described above. A sequential batch leaching procedure was used to measure DOC leached over longer time periods. The method used in this study consisted of a sequencing the 24 h batch test described above. It is described in detail in McLaughlan and Al-Mashaqbeh (2009).

2.4. Column leaching test

The column leaching test involved pumping water from a 45 L polyethylene container using a peristaltic pump (Masterflex Model 7553-85) in up flow mode into the column at an average flow rate was 12 cm^3/min. The column residence time was approximately 30 min. The columns were constructed in PVC class 18 with an inner diameter of 5.3 cm, a column length of 56 cm and cross-sectional area of 22 cm^2. They were constructed by using Silastic to seal a PVC end cap onto a PVC tube. A layer of plastic mesh and then a 2–3 cm depth of 3 mm glass beads were placed upon the end cap at the influent end of the column. A plastic mesh was then placed over the glass beads to support the filtration media. All columns had a mix of 68 g of 1.18 mm woody material and 1.663 g of 0.6 mm glass beads ($\approx$20–25% v/v compost) which gave a bulk density of approximately 1.5 g/cm^3.The mix was wetted and then added in increments using continuous column vibration without any compaction over the filtration media surface (Oliviers et al., 1996). Since the ratio of column diameter to average particle diameter is high, the effects of channelling were expected to have a negligible effect (Reynolds and Richards, 1996). The column test had periods of flow interruption. The first flow interruption period for the compost and hardwood was 266 h and for the pine was 530 h, while the second interruption period was 450 h for the compost and hardwood and 448 h for the pine. The column test data is presented in terms of flow time corresponding with the actual time when flow was occurring through the column as well as elapsed time when the flow interruption periods are included. The total elapsed time for the column test was approximately 1100 h. Column studies were terminated when the DOC of the effluent was in the range below 2 mg/L (or leaching had reached a steady state of DOC release).

2.5. DOC release equations

A number of predictive equations have been used in the literature to quantify the leachable fraction of an element and its associated release rate constants. In this study five equations were evaluated for their ability to model DOC release kinetics from woody materials. These equations are the first order, second order, simple Elovitch, power law function and the intra-particle diffusion model.

The cumulative release for first-order reaction kinetics is expressed as following (Gérard-Marchant et al., 2005):

$$D(t) = M_0[1 - \exp(-t/\tau_1)] \quad (1)$$

where $D(t)$ is the cumulative solute released per unit mass of material (mg g^{-1}) at any given time (t), M_0 is the initial soluble DOC, τ_1 is a characteristic time and t is leaching time in hours.For second-order kinetics model (Gérard-Marchant et al., 2005):

$$D(t) = M_0[t/(t + \tau_2)] \quad (2)$$

where τ_2 is a characteristic time and the other parameters have been defined above.

The parameters, τ_1 and τ_2 are rate constants and indicate how fast the cumulative concentration reaches the maximum value. For large characteristic times the solute is released more slowly than for short characteristic times.

The simple Elovich equation can be expressed as (Gérard-Marchant et al., 2005):

$$D(t) = (1/\beta)\text{In}(1 + \alpha\beta t) \quad (3)$$

where τ (mg g^{-1}) and β (mg g^{-1} h^{-1}) are fitted parameters.

The power law function can be expressed:

$$D(t) = At^B \quad (4)$$

where A (mg g^{-1} h^{-1}) and B (dimensionless) are fitted parameters.The intra-particle diffusion model can be expressed as;

$$D(t) = kt^{0.5} \quad (5)$$

where k (mg g^{-1} h$^{-0.5}$) is the intra-particle rate constant which is a fitted parameter.

This form of the intra-particle equation is based on a simplification of Crank's solution using spherical particles (Gerente et al., 2007). This equation is also known as the parabolic diffusion equation.

In order to apply these equations a number of simplifications must be made to the leaching process (Gérard-Marchant et al., 2005). Firstly, only the water-soluble organic carbon is available for leaching and this value can be determined experimentally or analytically. Secondly, it is assumed that chemical and microbial transformation of DOC to non-extractable forms and vice-versa are negligible. Thirdly, during the leaching event, the DOC release can be modelled as a net desorption process.

The fitted parameters for these equations were calculated using least squares regression (Microsoft Solver). This approach was used rather than linearization of the equations since linearization may result in improperly weighted data points during the analysis (Bilo, 2007). The mathematical models were tested for best fit using regression co-efficients (R^2), the standard errors of the estimate (SE) as well as visually plotting the data. Aharoni et al. (1991) have previously noted a solution may not be unique and that in some cases a number of these equations seem to equally well describe time-dependent data if a simple correlation co-efficient and standard errors of the estimate are used to evaluate the data.

3. Results and discussion

3.1. Leachate pH and EC

Soluble constituents leached from the woody materials during all extraction tests were found to alter the EC of the effluent. Within the column test under sustained flow, the effluent pH for the hardwood (4.5–5 U) was similar to the influent pH 4.8, while the pine (~5–5.5 U), and compost (7–5.5 U) effluent maintained a higher pH throughout the test and which increased during periods of flow interruption. These results are consistent with those reported in McLaughlan and Al-Mashaqbeh (2009).

The EC of the column leachate showed a general decline throughout the three leaching cycles with increased values after flow interruption. These increased values during periods of flow interruption are typical of non-equilibrium flow conditions. The compost showed the greatest amount of leached ions (EC increase) followed by hardwood and pine. There was over an order of magnitude difference between the compost and the pine.

3.2. Mass of DOC leached

Cumulative DOC leached from the three woody materials during the batch test, sequential batch test and column tests are presented in Table 2. The 24 h batch test shows that the DOC leached using MQ water was greater than that using the various salt extractants (K_2SO_4, NaCl and $CaCl_2$).

In contrast to this study, Na-based salts extractants have been found to release similar levels of DOC to that of water by destabilising organic matter held in Ca complexes (Reemtsma et al., 1999). Differences between the amounts of DOC extracted using a soil matrix and a wood matrix may be attributed to the interaction between the salt extractants and DOC sorption surfaces. Within soils a number of DOC sorption studies have indicated that extractable Fe and Al, organic C content, and mineralogy are important controls on their ability to sorb DOC (Kalbitz et al., 2000). These sites are likely to have greater reactivity with salt extractants. In woody materials the nature of the sorption sites and where DOC is located will differ. It has also been suggested that the nature of the DOC extracted by distilled water and salt extractants may differ (Rennert et al., 2007).

The magnitude of the DOC values leached from the hardwood to compost are consistent with data reported by Godley et al. (2005) for twigs (~5% TOC) and green grass (~9% TOC). The DOC/TOC ratio for pine is around an order of magnitude less than for hardwood or compost. The total mass of DOC leached for hardwood are broadly consistent with values of 5–20 mg C/g reported for twigs and are less than the values (20–200 mg C/g) for leaves and gumnuts (O'Connell et al., 2000; Wallace et al., 2008; Francis and Sheldon, 2002) using sequential extraction techniques.

In this study the DOC leached after 24 h for the three different leaching methods (batch, sequential batch and column) only comprises only 47–58% (compost), 49–72% (hardwood) and 36–64% (pine) of the DOC that was extracted at the end of the third leach cycle of the column test (Table 2). The lower DOC values reported

Table 2
Cumulative DOC leached for all extraction methods.

Method	Compost	Hardwood	Pine
	DOC leached (mg C/g)	DOC leached (mg C/g)	DOC leached (mg C/g)
24 h Batch test			
K_2SO_4	16.5	13.4	2.2
NaCl	14.0	13.2	2.2
$CaCl_2$	11.6	15.0	2.3
MQ water	17.8	16.6	3.1
Sequential batch test			
24 h	15.3	14.7	2.3
160–480 h	24.6	23.8	3.9
Column tests			
24 h	18.8	21.4	4.1
End of first leach cycle	26.3	27.7	4.7
End of third leach cycle	32.6	29.9	6.4
Total organic carbon (mg C/g)	382	487	480

from the 24 h batch tests when compared to the 24 h data from the column test reflects the influence of frequent flushing (48 cycles/day) in the column which creates a strong diffusion gradient compared with the two batch tests that were only flushed daily.

3.3. Rate of DOC release

The column leaching test for each media comprised three cycles of flow each separated by a flow interruption (no flow) period. This multi-cycle test was designed to provide an insight into DOC leaching under continuous and intermittent flow. The column test shows initially high values DOC values which rapidly decline with time except where the two flow interruption periods occur (Fig. 1). When flow was restarted after flow interruption there was elevated DOC which subsequently decreased to a baseline pseudo-steady state level. A period of rapid release followed by a lower sustained release is commonly observed in leaching studies. Within the first 2 h over 50% of the DOC that was released in the first leaching cycle had been released. This is consistent across all materials. It can also be seen the amount of DOC released from the pine over the three cycles is significantly less than the other woody materials.

3.4. Kinetic modelling of the first leaching cycle

The DOC release data is presented as cumulative release data for modelling purposes. The various kinetic models were fitted to the first cycle of the DOC release data for each of the three woody materials (Table 3).

The modelling process for the first and second order equations involved using the measured cumulative DOC release value (M_0) at the end of the first cycle of the column leaching test and then a fitting process to derive the values for the characteristic times (τ_1, τ_2). The results (Table 3) show that first and second-order models generally have a poor goodness of fit for all the data. This was evidenced by the relatively low R^2 (<0.88) and SE (>1.6) for compost and hardwood. These poor fits are evident when modelled data over predicts leaching at early times and then under predicts at later times (Fig. 2a–c). Even when both the characteristic time and initial soluble DOC (M_0) were treated as fitting parameters, the fit only improved marginally. The two other models (power law function, simple Elovich model) are regression based equations which therefore, do not have any independently measured values and consequentially all parameters are fitted. Both of these models show an excellent fit as evidenced by a high R^2 and low SE (<0.4). The goodness of fit is also evident in Fig. 2a–c where the Elovitch and power law function correctly match the release data for both early and late stage data. However, it should be recognised that these regression models result in an individual best fit pseudo-rate constant for the system studied and that a different rate constant will apply when system variables are changed.

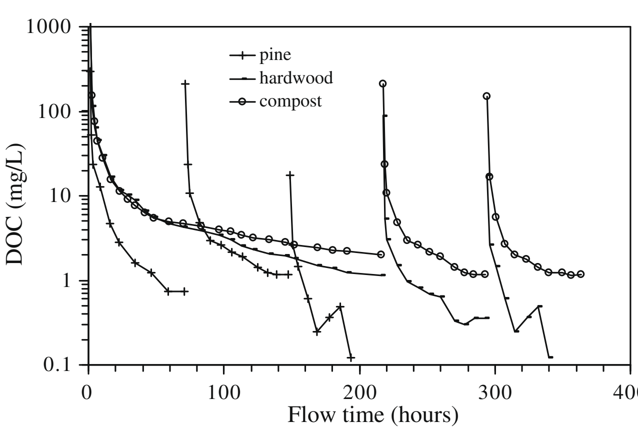

Fig. 1. DOC concentrations during the three leaching cycles.

Table 3
Fitted and measured parameters for the various kinetic models.

Parameters	Woody material		
	Pine	Hardwood	Compost
First-order model			
M_0*	4.25	24.72	22.55
$\sim\tau_1$	2.28	3.08	3.92
R^2	0.874	0.683	0.695
SE	0.372	2.496	2.591
Second-order model			
M_0[a]	4.54	25.84	23.85
$\sim\tau_2$	1.63	2.07	2.88
R^2	0.963	0.839	0.838
SE	0.189	1.647	1.786
Power law function model			
A (mg g^{-1} h^{-1})	2.26	14.44	11.68
B, dimensionless	0.18	0.12	0.15
R^2	0.953	0.998	0.995
SE	0.199	0.175	0.280
Elovitch equation model			
a (mg g^{-1})	19.9	370.0	99.5
β (mg g^{-1} h^{-1})	1.6	0.37	0.35
R^2	0.992	0.998	0.989
SE	0.080	0.175	0.435

[a] Measured parameter.

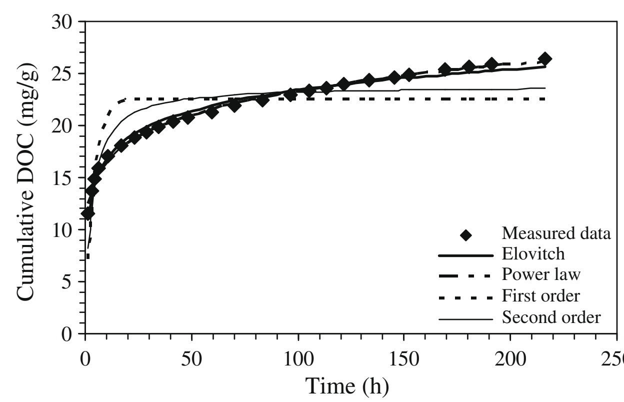

Fig. 2a. Fitted cumulative DOC leached data from measured data and predicted values for compost.

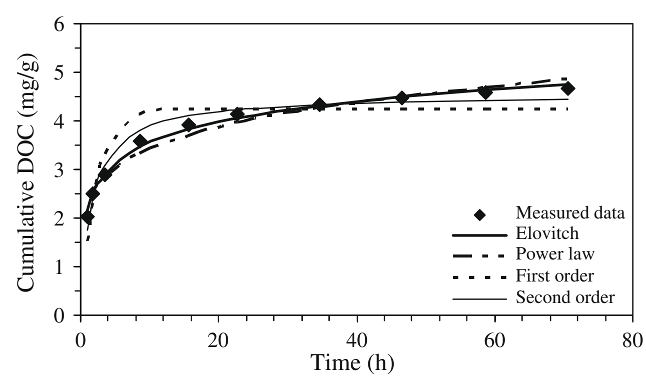

Fig. 2b. Fitted cumulative DOC leached data from measured data and predicted values for pine.

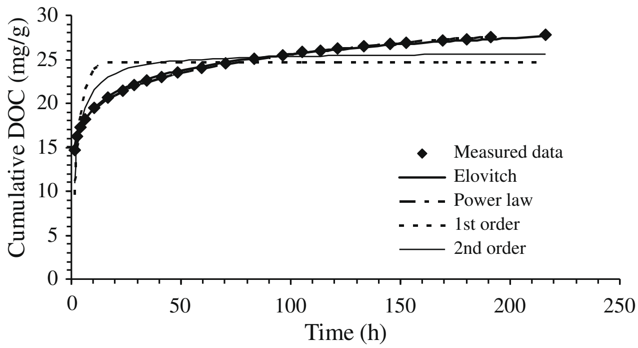

Fig. 2c. Fitted cumulative DOC leached data from measured data and predicted values for hardwood.

This study shows that a kinetic based approach to predicting DOC release from these woody materials using process-based parameters and first and second order equations has a substantially lower predictive power than a regression based approach using a power law function or Elovitch equation. Whilst the power law and Elovitch have been demonstrated to have good predictive power for continuous leach conditions, they give little insight into the mechanisms controlling the rate of DOC release within the woody materials.

3.5. DOC leaching mechanisms

One approach to gaining an insight into the DOC release process for woody materials is to examine rate-limiting release mechanisms;

- Film diffusion: Diffusion/mass transport across the liquid film surrounding the particle.
- Intra-particle diffusion: Internal diffusion/mass transport within the particle boundary.
- Desorption from the site.

While both diffusion processes and desorption can affect the rate at which solutes are released from porous media, they are conceptually different but are mathematically analogous (Haws et al., 2006). Within this study we have focussed on using diffusion concepts to determine the rate controlling mechanisms. At any particular time, release kinetics will be controlled by the slower of either the film or the intra-particle diffusion process. An appreciation of which diffusion processes controls the leaching process can be obtained by applying the intra-particle diffusion model to the experimental data. Numerous studies on sorption show that initial curved portions of the intra-particle diffusion plot suggest film diffusion processes while linear portions suggest intra-particle diffusion (Gerente et al., 2007). A third stage may occur where intra-particle diffusion decreases due to a low solute concentration and the release rate plateaus. In reality these various stages may actually be a gradual transition. At long enough time frames then substrate degradation may generate further DOC.

In this study with the pine and compost, intra-particle diffusion dominates the leaching process after approximately 22 h in the first cycle. This is evident in the good data fit for a linear trendline (Table 4) when cumulative DOC (mg/g) is plotted against the square root of contact time.

The trend for hardwood is more curvilinear for early time periods and a strong correlation could be established after 100 h but a slightly poorer correlation was apparent after 23 h. Hardwood clearly has a broader transition period between film and intra-particle diffusion dominance compared with pine and compost. By extrapolating the trendline for intra-particle diffusion back to zero and subtracting this from the final DOC leached at the end of the first cycle, the amount of DOC leached which is controlled by intra-particle diffusion and that controlled by film diffusion can be estimated. The data shows that of the DOC leached, intra-particle diffusion only controls 28% (pine), 42% (compost) or 32% (hardwood). Whilst intra-particle diffusion does not control the dominant portion of the DOC leached during the first cycle, it has great significance as a rate controlling mechanism for subsurface barrier design where it is the longer term data which creates the sustained levels of DOC release. During the second and third cycle a near linear release trends indicate that intra-particle diffusion is the dominant rate controlling process when each of these events is treated independently (data not shown). A different intra-particle rate constant for each flow cycle and type of wood material was also evident. At very long time periods the rate of release drops to a very low level and DOC desorption/generation may control the release.

Table 4
Intra-particle model parameters.

Material	Time period (h)	Regression equation of trendline	R^2
Pine	22–71	$y = 0.148x + 3.4$	0.990
Compost	16–216	$y = 0.768x + 15.3$	0.997
Hardwood	100–216	$y = 0.450x + 21.15$	0.991
Hardwood	23–216	$y = 0.631x + 18.9$	0.978

This data shows that under continuous flow (e.g. first cycle) these woody materials have a transition from film diffusion controlled release to intra-particle controlled release of approximately 24 h. The more rapid leaching rates that occur in this time period can therefore, be approximated by measured values from short-term batch tests. This could represent the 'readily soluble DOC pool' described in many studies. The sustained rates of DOC release after this time period are dependent on intra-particle diffusion. Under the relatively short time periods used in the first stage (80–200 h) then a single intra-particle rate constant would be sufficient to predict DOC release. However, for longer time periods then multiple rate constants would be needed to reflect the changing limiting conditions for diffusion controlled flow. However, factors such as substrate degradation may also need to be incorporated into a long term model. It should be realised that the relative significance of each diffusion processes and therefore, which rate is limiting at a particular time is system dependent. As yet there few if any reported studies which analyse leaching processes in terms of film and intra-particle diffusion using these types of porous media. It is therefore, not understood how these rates may change between different materials and how the rate controlling process changes with system parameters such as flow and particle size.

4. Conclusion

The analysis of leaching from woody materials in terms of rate controlling diffusion mechanisms provides useful insights for woody material. The strong control of intra-particle diffusion over DOC release rates after an initial period provides an opportunity for improved predictive modelling using some independent measurement of material properties. Further work is needed to establish relationships between DOC release and rate controlling diffusion processes under a range of system conditions relevant to engineering applications of woody filtration media. The increased DOC release using low rather than high ionic strength water contrasts with many soil studies and suggests that DOC is bound within soils in different ways compared with woody materials.

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