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Retention and Solute Transport Properties in Disturbed and Undisturbed Soil Samples

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ABSTRACT: Solute transport parameters can be determined in miscible displacement experiments, usually performed in columns with disturbed (sieved) soil samples. Experiments with undisturbed samples are uncommon, due to the difficulty of taking undisturbed samples at the size required for these experiments. Structural alteration of the disturbed material implies modifications in the pore geometry that determines hydraulic properties, including hydraulic conductivity and retention and properties related to miscible displacement. An existing model for prediction of breakthrough curves based on retention properties was tested using material from a medium-textured Ferralsol, and alterations caused by sample disturbance were investigated. Soil water retention curves and miscible displacement parameters were determined in breakthrough experiments with nitrate salts in columns filled with undisturbed and disturbed soil samples. Data obtained from the undisturbed samples showed a higher dispersion, suggesting homogenization of pore geometry and a reduction in the representative elementary volume by the disaggregation and sieving of the soil material. The transport parameters for nitrate determined in disturbed and undisturbed samples were significantly different and the model was able to simulate the observed breakthrough curves after fitting the pore connectivity parameter.

Keywords: sampling, retention, miscible displacement, solute transport, leaching.

INTRODUCTION

Quantifying the components of the solute transport process is important for understanding management practices to control ion movement in soil (Bresler, 1981). Studies on soil solute dynamics not only provide a means to determine solute flow in soils but can also give a physical explanation of phenomena that occur during the movement of mineral fertilizers and other salts, such as percolation, exchange, and adsorption of ions (Nielsen and Biggar, 1961).

The miscible displacement process affecting solutes in porous media can be physically described by the solute transport equation, relating displacement to the movement of the interface between transported and transporting fluids. The solution of this equation includes three components describing solute transport: the dispersion-diffusion coefficient (D), the retardation factor (R), and the Péclet number (P) (Ruiz et al., 2010). These transport parameters can be determined in the laboratory by performing a breakthrough experiment, to be analyzed by analytical or numerical physical-mathematical models (Anami et al., 2008; Fonseca et al., 2009; Rose et al., 2009; Doltra and Muñoz, 2010; Silva et al., 2012). In contrast with analytical solutions, the numerical models do not require boundary conditions that usually are not satisfied during experiments.

Due to the difficulty of collecting undisturbed samples at the required size, in the order of 1000 cm^3, breakthrough experiments are normally performed in columns filled with disturbed and sieved soil material. The water retention curve, however, is normally performed in smaller samples (in the order of 100 cm^3), allowing the use of samples with undisturbed structure.

Any deformation implies modification in the pore space geometry that determines hydraulic properties, including retention and hydraulic conductivity, as well as properties related to miscible displacement. Therefore, the use of samples with a natural structure is preferable for evaluation of the behavior of naturally occurring soils.

Water retention and ion displacement is governed, among other factors, by the geometry of pore space, and it is our hypothesis that a functional relation between both processes is to be expected. Therefore, a model that predicts the breakthrough curve based on water retention properties would be sensitive to modifications in soil structure. Such a model was proposed by Wang et al. (2002), who employed a water retention parameter related to pore size distribution, and another parameter related to pore size connectivity to be estimated independently.

The objective of this paper was to analyze the modifications in breakthrough properties caused by disturbance of soil structure by sieving and repacking, and the correlation of these modifications to changes in the corresponding water retention curves using the model of Wang et al. (2002). To do so, modifications in the water retention parameters were determined from experimental retention desorption curves, whereas changes in miscible displacement parameters were measured by breakthrough experiments using nitrate salts in soil material samples from a medium-textured Ferralsol from Brazil.

MATERIALS AND METHODS

Soil

The soil material used in this study was sampled at depths from 0.20 to 0.50 m in a *Latossolo Vermelho-Amarelo* (Santos et al., 2013), a Ferralsol (IUSS Working Group WRB, 2015) from the municipality of Piracicaba, São Paulo, Brazil (22° 44' S and 47° 38' W, altitude 500 m) under conventional tillage management.

Particle size analysis was performed using the densimeter method (Donagema et al., 2011) and yielded 0.198 kg kg^{-1} clay, 0.032 kg kg^{-1} silt, and 0.770 kg kg^{-1} sand, consisting of 0.320 kg kg^{-1} coarse sand (2.00 – 0.210 mm) and 0.450 kg kg^{-1} fine sand (0.210 – 0.053 mm). Bulk density, determined in a soil sample ring, was 1,230 kg m^{-3}, and particle density, determined with a pycnometer, was 2,600 kg m^{-3}, resulting in a total porosity of 0.527 m^3 m^{-3}. Soil chemical properties were determined by standard methods according to Donagema et al. (2011), which showed pH(CaCl$_2$) of 6.2, S of 14 g m^{-3}, organic matter of 13 kg m^{-3}, P of 3 g m^{-3}, K$^+$ of 2.0 mol$_c$ m^{-3}, Ca^{2+} of 22 mol$_c$ m^{-3}, Mg^{2+} 10 of mol$_c$ m^{-3}, and H of 15 mol$_c$ m^{-3}, while Al exhibited a total base sum of 34 mol$_c$ m^{-3}, CEC of 49 mol$_c$ m^{-3}, and base saturation of 69 %.

Breakthrough experiments

Six breakthrough experiments were performed in PVC tubes with an external diameter of 0.05 m, consisting of three tubes filled with undisturbed and three with disturbed soil samples. To allow maintenance of a constant hydraulic head, an outlet was placed at the top of the tube, 0.1 m above the soil sample surface, to drain excess water.

Undisturbed soil samples were taken with a specially designed auger (Figure 1). Excess soil on the top and bottom of the samples was removed, resulting in a final sample height of 0.23 m. Figure 1 shows some details of the undisturbed sampling process.

For experiments with disturbed samples, the soil material was air dried and sieved through a 0.002 m mesh. Columns were filled with the material in order to obtain uniformity and homogeneity, subdividing the total mass in portions. The portions were subsequently added to the tube and compacted, applying light pressure with a wooden disc.

The breakthrough experiments were performed at the Soil Physics Lab of the Biosystems Engineering Department of the University of São Paulo in Piracicaba, São Paulo, Brazil. Breakthrough curves (BTC) were determined by adding a solution with 50 g m^{-3} of nitrate to the soil surface contained in the columns. For each of the three columns with disturbed and undisturbed samples, a distinct inorganic nitrate salt was used: Ca(NO$_3$)$_2$, NH$_4$NO$_3$, and KNO$_3$. Nitrate was used due to its high solubility and for its agronomic and environmental importance. In addition, from an electrochemical standpoint, there is a difference between nitrate and other oxyanions regarding adsorption by soil particles. Nitrate is adsorbed exclusively by electrostaticity, thus depending solely on soil particle charges (Alcântara and Camargo, 2005).

Saturation and leaching were performed according to Rossi et al. (2007) and Silva et al. (2012). After verification of a permanent flow regime, a solution containing the respective

(a)   (b)   (c)   (d)   (e)
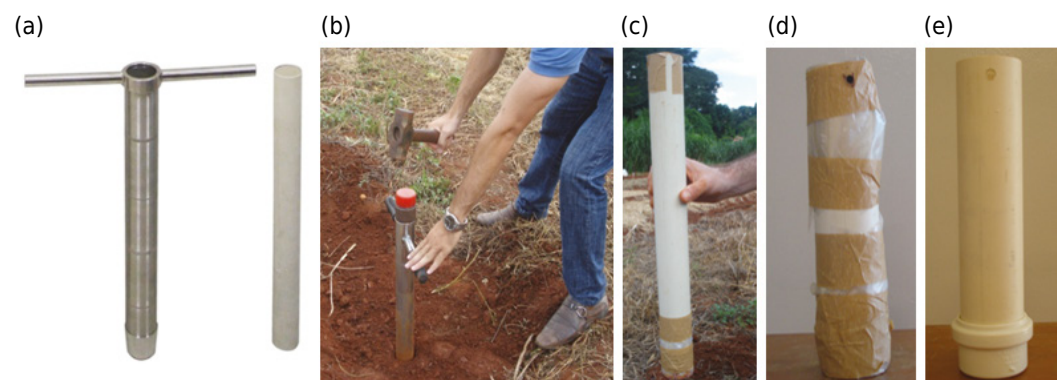
Figure 1. Details of the undisturbed sampling process. (a) Auger used to take the undisturbed samples; (b) Sampling; (c) PVC tube with undisturbed sample; (d) Sample after removing excess and drilling the outlet; (e) PVC tube with drain and cap at the bottom.

50 g m^{-3} nitrate source was applied to the columns, one component per column. Mariotte flasks were used to ensure a constant nitrate application rate to the column.

The column outflow was sampled in 15 mL acrylic flasks. The accumulated outflow was expressed relative to the column pore volume V_P (m^3), calculated as the product of column volume V (m^3) and soil porosity α (m^3 m^{-3}). The NO_3^- concentration in each sample was determined by spectrophotometry (Yang et al., 1998).

Values of nitrate concentration (C) as a function of applied volume (V_a) for each soil column were transformed into their relative equivalents. The concentration was divided by the concentration of the applied solution (C_0), and the applied volume was expressed relative to the column pore volume (V_P). Using the obtained relative solute concentration (C/C_0) and relative volume (V_a/V_P), transport parameters (retardation factor – R, Péclet number – P, slope – S, dispersion-diffusion coefficient – D, and dispersivity – λ) were calculated by inverse modeling using the CFITIM module from Stanmod 2.0 (STudio of ANalytical MODels) (Šimůnek et al., 2008). The water flux density in the soil was determined by dividing the outflow volume by the column cross-section and by the time.

Water retention experiments

Water retention (desorption) curves were determined for the disturbed and undisturbed soil samples in metal rings of 0.03 m height and 0.05 m diameter, with three replicates. The sample water content was determined after equilibrium at nine tensions: 0.10, 0.20, 0.40, and 1.0 m on a tension table, and 3.0, 5.0, 10, 50, and 1.5 10^2 m in a pressure chamber. The Brooks and Corey (1964) equation was fitted to the data using the RETC software (van Genuchten et al., 1991):

$$S = \frac{\theta - \theta_r}{\theta_s - \theta_r} = \left(\frac{h_b}{h}\right)^n \qquad \text{Eq. 1}$$

in which S is the effective saturation, θ is the water content (m^3 m^{-3}), $θ_s$ is the saturated water content (m^3 m^{-3}), $θ_r$ is the residual water content (m^3 m^{-3}), h_b is the air entry matric potential (m), and n is a fitting parameter related to pore size distribution.

Relation between retention and breakthrough properties

To correlate retention and breakthrough properties, the Wang et al. (2002) model was applied. According to this model, relative solute concentration $c(t)/c_0$ is described by:

$$\frac{c(t)}{c_0} = 1 - \left[\frac{nR}{(n(m + 1) + 2)V_a/V_P}\right]^{\frac{2+(m + 1)n}{2 + mn}} \qquad \text{Eq. 2}$$

in which n is the retention curve fitting parameter (Equation 1), m is a parameter related to pore connectivity, normally considered equal to -2, and R is the retardation factor given by:

$$R = 1 + \frac{\rho k}{\theta} \qquad \text{Eq. 3}$$

in which k (m^3 kg^{-1}) is the distribution coefficient, $ρ$ (kg m^{-3}) is the soil bulk density, and θ (m^3 m^{-3}) is the soil water content.

The applied volume of solution in relation to pore volume is calculated as

$$\frac{V_a}{V_P} = \frac{tv_0}{L} \qquad \text{Eq. 4}$$

where t (d) is time, L (m) is the length of the soil column, and v_0 (m d^{-1}) is the average velocity of the solution, given by

$$v_0 = \frac{K_s}{\theta_s - \theta_r}$$

Eq. 5

where K_s (m d^{-1}) is the saturated hydraulic conductivity, estimated in each experiment as a function of the observed solute flux.

RESULTS AND DISCUSSION

Breakthrough experiment

The disturbed soil material in the columns had a bulk density of 1230 kg m^{-3}, corresponding to a total porosity of 0.527 m^3 m^{-3}. The bulk density of the soil in the undisturbed columns was higher, 1355 kg m^{-3} (corresponding to a total porosity of 0.482 m^3 m^{-3}), with a standard deviation of 11 kg m^{-3} (coefficient of variation of 0.8 %). The difference in density and porosity observed between the disturbed and undisturbed columns results in different hydraulic properties and solute flux properties.

Saturated hydraulic conductivity in the disturbed soil columns was 0.535 m d^{-1}, on average, and 0.134 m d^{-1} in the column with undisturbed structure. The difference between both values is in agreement with the retention curves, which show an increase in macroporosity for the disturbed soil.

A summary of the analysis of variance for the solutes $Ca(NO_3)_2$, KNO_3, and NH_4NO_3 is shown in table 1, showing no significant difference between the transport parameters evaluated, either for the disturbed or the undisturbed samples. From these results, the experiments performed with any of the cations can be considered replicates.

The multiple means comparison test (Tukey at 5 % probability) for the main nitrate transport parameters obtained from disturbed and undisturbed soil showed differences for all parameters (Table 1). It is clear that these differences are related to structural modifications in the disturbed samples caused by the process of disaggregation and sieving. A similar

Table 1. Summary of the analysis of variance (squared means) for solute transport parameters obtained from the solutes $Ca(NO_3)_2$, KNO_3, and NH_4NO_3 in experiments with disturbed and undisturbed soil samples

| Source of variation	Degree of freedom	v	P	R	S	λ	D
Disturbed soil							
Treatment	2	0.00268^{ns}	21.99^{ns}	0.00014^{ns}	0.025^{ns}	0.038^{ns}	0.031^{ns}
Residue	6	0.01103	29.39	0.00083	0.032	0.079	0.042
Overall mean	-	9.81[1] A	22.75 A	0.94 A	1.42 A	0.0095[2] B	0.092[3] A
CV (%)	-	15.73	23.83	3.07	12.56	28.44	32.07
Undisturbed soil							
Treatment	2	0.00541^{ns}	3.51^{ns}	0.042^{ns}	0.099^{ns}	0.075^{ns}	0.045^{ns}
Residue	6	0.01334	7.08	0.019	0.032	0.381	0.071
Overall mean	-	2.0[1] B	8.69 B	0.82 B	1.03 B	0.0241[2] A	0.048[3] B
CV (%)	-	81.86	30.62	16.61	17.46	25.65	80.94

v: average velocity of solution; P: Péclet number; R: retardation factor; S: slope; λ: dispersivity; D: dispersion-diffusion coefficient; and CV: coefficient of variation. Overall means followed by different letters in the columns are different at a 5 % probability level by the Tukey test. ^{ns}: non-significant (p≥0.05) by the F-test. [1] In m d^{-1}. [2] In m. [3] In m^2 d^{-1}.

analysis was performed by Celestino Ladu and Zhang (2011), who found lower values of dispersivity in disturbed and repacked soil, due to a homogenization of structure.

During a flow process through a heterogeneous porous medium, a major part of the solution is transported through some wider channels, making solute molecules move rapidly, a phenomenon called preferential flow (Rosqvist and Destouni, 2000; Gerke, 2006). More preferential flow can be expected in soils with natural structure (Dousset et al., 2007), corroborating the differences observed in transport parameters, especially in reference to dispersivity, as larger values of dispersivity are associated with a predominance of preferential flow (Aggelopoulos and Tsakiroglou, 2008).

Retention experiment

The results from the retention experiments were in agreement with expected behavior: for matric potentials less negative than 1 m, the disturbed soil showed higher water contents than the undisturbed soil (Figure 2), exhibiting the formation of macropores as a result of the disturbance. Under drier conditions (at more negative potentials), the water contents for disturbed and undisturbed soils were very similar. Fitting parameters of the Brooks and Corey (1964) equation show a higher n parameter for the disturbed soil (Table 2). A higher n indicates a more concentrated pore size distribution and directly
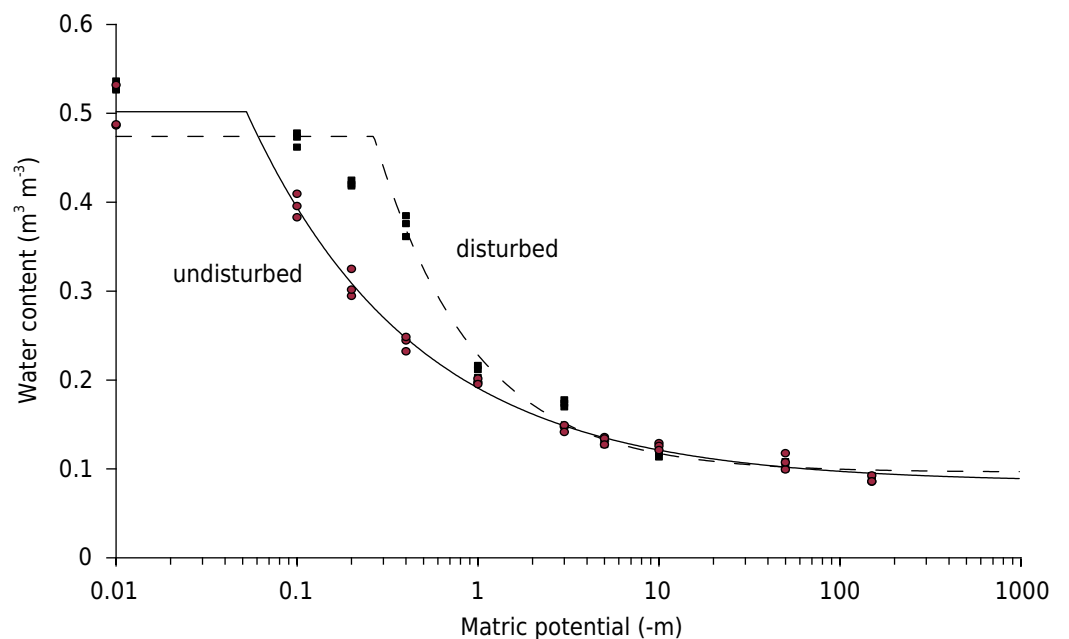
Figure 2. Water content versus matric potential ("water retention curve") for disturbed and undisturbed soil samples. Data points correspond to observed values; lines represent the fitted equation 1 (parameters in table 2).

Table 2. Fitting parameters for equation 1 (Brooks and Corey, 1964) in disturbed and undisturbed samples

Parameter	Disturbed		Undisturbed	
	Value	Standard Error	Value	Standard Error
θ_s	0.474	0.0095	0.502	0.0065
θ_r	0.096	0.0128	0.085	0.0064
n	0.795	0.1424	0.464	0.0303
h_b^{-1} (m^{-1})	3.8	0.45	19.1	1.41
R^2	0.972		0.994	

affects the predictions of the Wang et al. (2002) model. The h_b parameter increased and q_s decreased in the disturbed soil; however, these values do not affect the predictions of the Wang et al. (2002) model.

The three replicates showed similar behavior, with differences in the order of 0.01 to 0.03 m^3 m^{-3}. Equation 1 fitted the data points well, with a higher deviation between saturation and matric potential 0.2 m in the disturbed soil, resulting in a slightly lower R^2 in this treatment.

Relation between retention and breakthrough properties

The retardation factor (Table 1) was smaller than 1, showing that only part of the liquid phase takes part in the transport process. This is the case when the chemical substance (solute) undergoes anionic exclusion or when immobile water regions are present that do not participate in convective movement (Wierenga and van Genuchten, 1989). Consequently, equation 2 from Wang et al. (2002) for reactive solutes was employed.

The diffusion-dispersion equation contains two independent parameters (P and R) fitted to results from a breakthrough experiment by software like Stanmod/CFITIM. The Wang et al. (2002) model, in contrast, is based on information contained in the water retention curve (parameter n), adding an independent parameter, m. Therefore, the statistical performance of CFITIM is expected to be better than that of the Wang et al. (2002) model, as confirmed by the Nash-Sutcliffe efficiency values (Table 3). Using the standard value $m = -2$, the performance of the Wang et al. (2002) model was slightly inferior to CFITIM for disturbed soil, and the shape of the predicted curve simulates the experimentally observed behavior well (Figure 3). For the undisturbed soil, a fit with $m = -2$ resulted in large deviations and a shape that differs from the tendency observed.

The Wang et al. (2002) model predictions were fitted to the experimentally obtained values, resulting in $m = -2.06$ (disturbed soil) and $m = -3.55$ (undisturbed soil). These values confirm that $m = -2$ is an adequate value for the disturbed soil material, but not for the undisturbed soil material, where m assumes a more negative value. In this respect, it is interesting to note that m is considered to be related to pore connectivity, with more negative values corresponding to lower connectivity and higher retardation.

Figure 3 also shows that the Wang et al. (2002) model, because of its mathematical structure, does not describe well the beginning of the breakthrough curve, predicting an abrupt increase in relative concentration, which is not observed experimentally.

Observing the experimental data in figure 3, relative maximum concentration ($C/C_0 = 1$) was reached between 1.5 and 2 V_a/V_p. Dispersion between observed values was higher in the experiments with undisturbed soil than in the disturbed soil. The reason for this can be found in the destruction and sieving of soil aggregates, which homogenizes larger structural elements. The small dispersion observed in the experiments with disturbed soil leads to the conclusion that the size of these samples exceeded the representative elementary volume (REV), as described in Bear (1972), Bachmat and Bear (1986), and Kutilek and Nielsen (1994), whereas the REV for the undisturbed soil is larger than the soil column used in our experiments.

Table 3. Nash-Sutcliffe efficiency coefficient for CFITIM and for the Wang et al. (2002) model predictions for m = -2 and for a fitted m
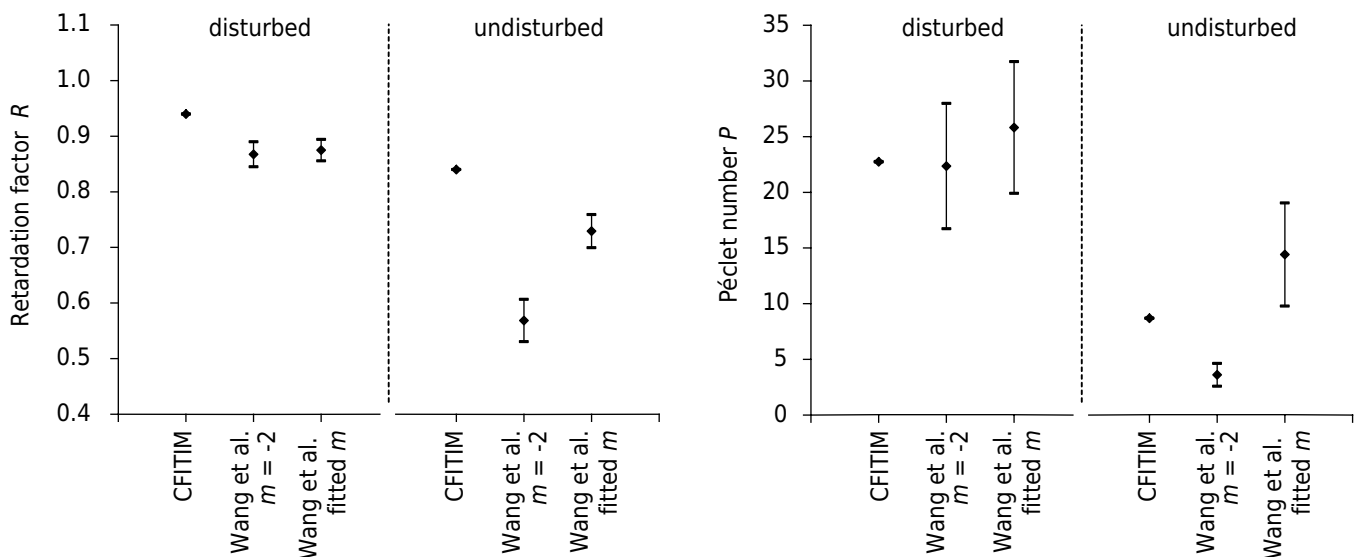
	CFITIM	Wang et al. (2002), $m = -2$	Wang et al. (2002), fitted m
Disturbed	0.998	0.978	0.982 ($m = -2.06$)
Undisturbed	0.994	0.886	0.957 ($m = -3.55$)

The higher dispersion of values observed in the undisturbed soil leads to a lower Nash-Sutcliffe coefficient and a higher standard deviation (Table 3). Similar observations are reported by several authors. For example, Rossi et al. (2007), in an experiment with two Ferralsols of different texture (a loamy versus a clayey soil), showed that all parameters except dispersivity increased for the disturbed soil. Sampaio et al. (2010) found differences in nitrate transport parameters between disturbed and undisturbed samples of a Ferralsol and made reference to possible compaction and modification of preferential pathways for water flow in their discussion.

An alternative to verify the predictive capacity of the Wang et al. (2002) model is by using the C/C_0 values predicted by the model as a function of V_a/V_p, fitting the diffusion-dispersion equation to them by Stanmod/CFITIM, thus verifying if the Wang et al. (2002) model is capable of reproducing the experimentally obtained values. Figure 4 shows the results from this exercise. The Wang et al. (2002) model and the diffusion-dispersion equation predict different curve shapes; therefore, the dispersion of fitted values of the Péclet number P and retardation factor R by Stanmod/CFITIM is higher in the case of the original experimental breakthrough data. Good fits are observed both for P and for R in the
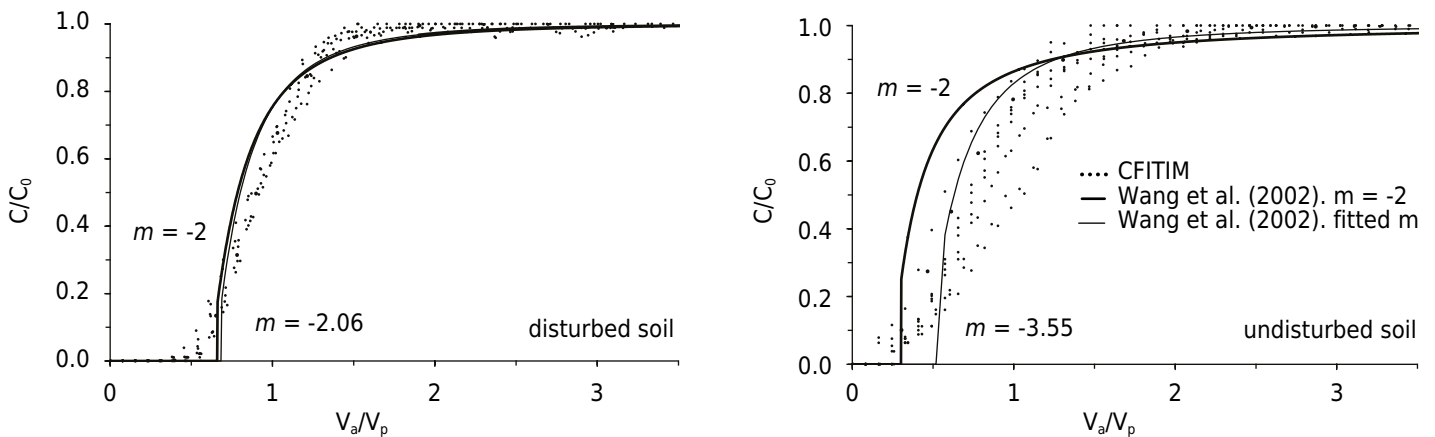
Figure 3. Relative solute concentration C/C_0 in the effluent of disturbed and undisturbed soil, as a function of the relative applied volume V_a/V_p: values observed in three experiments (data points), fitted line by Stanmod/CFITIM, and values predicted by the Wang et al. (2002) model for default parameter value m = -2, and for fitted m.

Figure 4. Péclet number P and retardation factor R obtained from fitting Stanmod/CFITIM to experimental breakthrough data, and to values predicted by the Wang et al. (2002) model for m = -2 and for fitted m, for disturbed and undisturbed soil material. Error bars represent the confidence interval of 95 %.

disturbed soil. For the undisturbed soil, fitting the *m* parameter is an essential step for obtaining better predictions.

CONCLUSIONS

The Wang et al. (2002) model allows good predictions of breakthrough experiments for the disturbed soil using the standard pore connectivity parameter value of *m* = -2. For undisturbed soil samples, a more negative value was fitted to the soil used in this study (*m* ≈ -3.5). More research in different soils is needed in order to apply the model.

Experimental breakthrough data dispersion was higher in columns filled with undisturbed material than in those filled with disturbed soil, suggesting that the homogenization of pore structure leads to a reduction in the size of the representative elementary volume (REV) for these properties.

Nitrate transport properties determined in disturbed and undisturbed samples showed significant differences, especially the Péclet number (about 3 times higher in the disturbed soil), mean solution velocity (about 5 times higher in the disturbed soil), dispersivity (about 3 times higher in the undisturbed soil), and the dispersion-diffusion coefficient (about 2 times higher in the disturbed soil).

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