

Division - Soil Processes and Properties | Commission - Soil Physics

# Rock size fragments reduction allow including their effect on water retention properties determined with a dew point potentiometer

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**ABSTRACT:** The relation between water content and water potential is a key soil hydraulic property. The presence of rock fragments in soils can affect this property in bulk soil. In this study, we focused on the relation property determined with a WP4 dew point potentiometer. The objective was to evaluate the hypothesis that breaking large gravel-size particles into smaller fragments is a suitable strategy for accurate WP4 measurements in coarse materials without affecting the retention properties. Ten initial samples of basalt rock fragments with different weathering degrees were collected from the A horizon of an Entisol (*Neossolo*). Rock fragments initially sized between 38 to 4 mm were successively fractured into smaller fragments (size classes of 4-2, 1-0.25, and <0.25 mm). For each size class, the retention properties (water content versus water potential) were determined with WP4 equipment. No significant effect of fragment size was detected on the determined retention properties. Using supplementary water retention data from a previous study, we found an overestimation of water retention at -1.5 MPa ranging from 0.08 to 0.12 g g<sup>-1</sup> when rock fragments were removed, which is of the order of magnitude to the available water capacity of several soils. This study showed that fragments larger than the diameter of the WP4 sample cup could be broken to sizes smaller between 4 and 0.25 mm without affecting the retention properties. This is a suitable strategy to allow accurate WP4 measurements in coarse materials while considering the effect of such coarse material on retention properties.

**Keywords:** soil hydraulic properties, stony soil, water potential.

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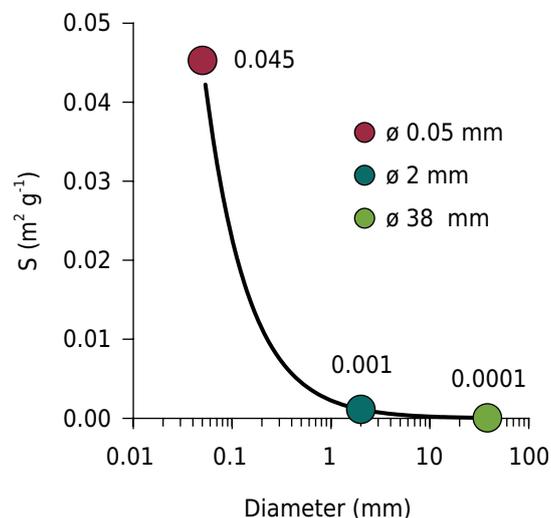


## INTRODUCTION

Several water flow models require parameters of the water retention curve. In some models, soil water content at the permanent wilting point (PWP) is a key hydraulic parameter related to water uptake by plants. For example, PWP is needed in the bucket soil-water balance module of several models of DSSAT - Decision Support System for Agrotechnology Transfer (Jones et al., 2003). The physically-based model Hydrus-1D (Šimůnek and van Genuchten, 2008) also needs PWP or a close value to parametrize the transpiration reduction function (Feddes, 1978) in the root water uptake submodel. Furthermore, PWP is a parameter in water availability indicators to plants, such as the least limiting water range and integral water capacity (Kazemi et al., 2020).

Water potential measurements in the water vapor phase in equilibrium with the liquid phase of a soil sample inside a closed chamber, where the dew point depression is measured, is a fast, accurate technique for determining water potential, especially in dry soils (Andraski and Scanlon, 2002). The WP4 dew point potentiometer (Decagon Devices, Inc., Pullman, WA, USA) uses a chilled mirror dew point technique to measure water potential from 0 to -10 MPa with an accuracy of 0.1 MPa and from -10 to -300 MPa with an accuracy of 1 % (Decagon Devices, 2000). In terms of water potential, the value of -1.5 MPa is frequently considered a close estimate of PWP (Soil Science of Society America, 1997). As WP4 measurements in soils are sufficiently accurate for water potential values below -1 MPa (Klein et al., 2010; Gubiani et al., 2012), the determination of PWP with WP4 has become common in recent years.

Measurements of water potential in WP4 are frequently done with the soil fraction passed through a 2 mm mesh sieve. However, if the soil solid phase is composed of particles coarser than 2 mm, the water content and water potential relationship in the fine fraction would not correspond to the water content and potential relationship of the bulk soil (Reinhart, 1961; Khaleel and Relyea, 1997). The amount of water a soil retains at PWP depends considerably on the soil specific area (Petersen et al., 1996; Ghanbarian-Alavijeh and Millán, 2009). The specific surface area ( $S$ ) of a spherical particle is a function of its diameter ( $\phi$ ) and density ( $\rho$ ),  $S = 6/(\rho\phi)$  (Hillel, 2003). Although spherical particles are rare in soils, the relationship of  $S$  with  $\phi$  over the range of  $0.05 \leq \phi \leq 38$  mm (Figure 1) is useful to illustrate the two issues addressed in our study. By removing particles coarser than 2 mm from the sample, an overestimation of bulk soil  $S$  and water retention is expected. It is not possible to include particles larger than the diameter of the WP4 sample cup (approximately 38 mm). However, the relationship of  $S$  and  $\phi$  (Figure 1) also suggests that 38 mm-sized particles can be fractured into sand-sized particles without causing



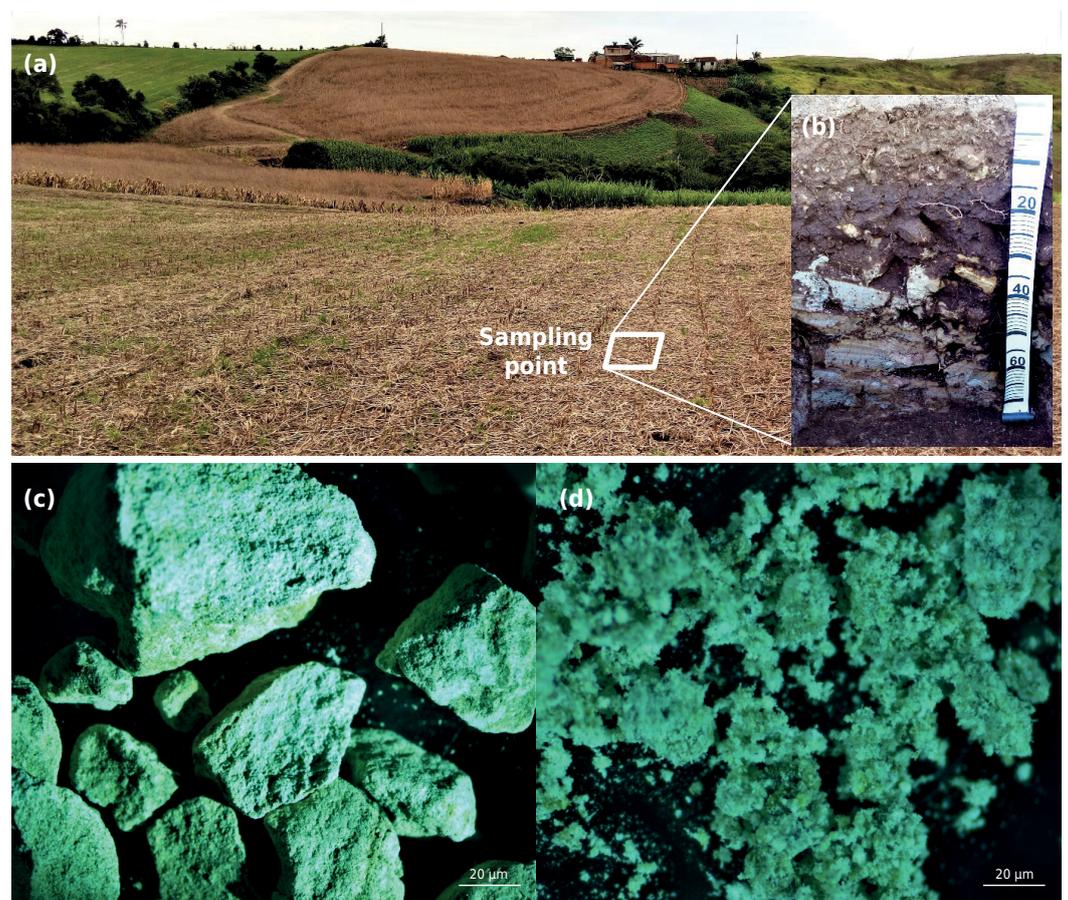
**Figure 1.** Specific surface area ( $S$ ) of a spherical particle as a function of its diameter ( $\phi$ ).

a significant increase in *S*. If the reduction of coarser material does not affect water retention, this procedure would allow us to mix fragmented coarser material with the fine particle fraction to compose a WP4 sample with an *S* similar to that of bulk soils.

A systematic evaluation of these issues is still scarce and necessary. Several factors are forcing agriculture to expand to marginal areas around the world (Bruinsma, 2003; Laurance et al., 2014). To effectively manage water resources and plant species and cultivars in such areas or any soil containing rock fragments, accurate hydraulic parameters need to be determined for gravel soils. Thus, in this study, we evaluated the hypothesis that turning large gravel particles into smaller fragments is a suitable strategy to allow accurate WP4 measurements in coarse materials. Based on supplementary data, we also evaluated the overestimation of water retention caused by removing coarser fragments from the WP4 sample.

## MATERIALS AND METHODS

Saprolite fragments were collected from the A horizon of a Entisol (Soil Survey Staff, 2014), which correspond to a *Neossolo*, according to the Brazilian Soil Classification System (Santos et al., 2013), of a farm located in the Ivorá municipality, Southern Brazil (Figure 2a). Most of the soils in this region are formed from volcanic rocks as basalt (Pedron et al., 2011) and cultivated by small farmers. A representative location was sampled to collect remaining basalt rock fragments with different weathering degrees in bulk soil (Figure 2b). The coordinates of the sampling point are 29° 30' 20.1" S and 53° 37' 34.6" W. The fragments were referred to as fragments of saprolite (FR<sub>sapr</sub>). In the



**Figure 2.** Typical relief of areas in the upper part of the transition from the central plain to the plateau of the state of Rio Grande do Sul, Brazil (a). Soil profile in the sampled location (b), stereo microscope images of saprolite fragments before (c), and after breakdown (d).

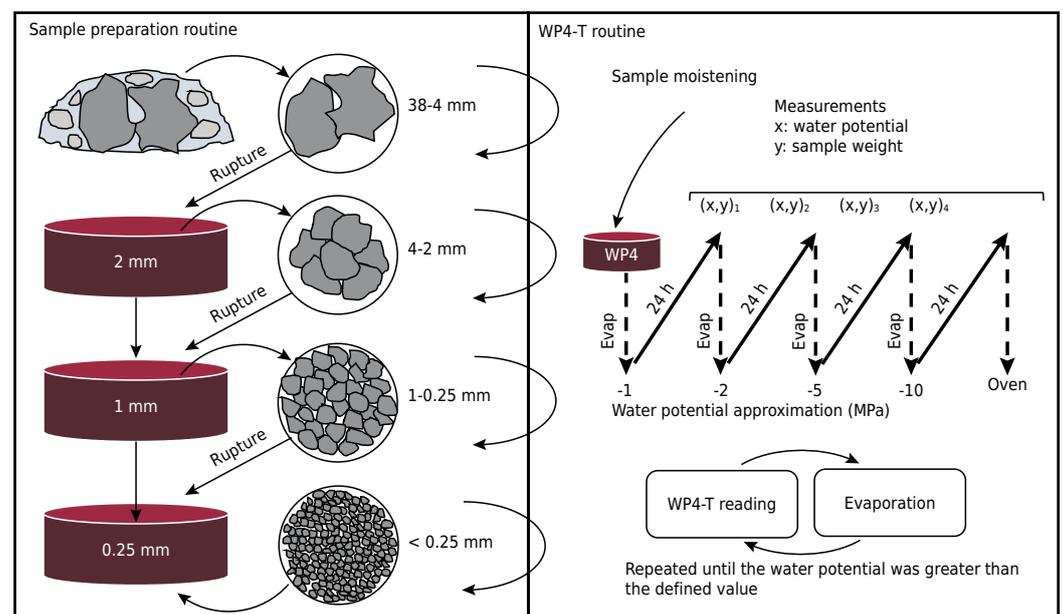
laboratory, the air-dried bulk material collected from the A horizon was sieved with a 2-mm mesh. The  $FR_{sapr}$  larger than 2 mm were immersed in NaOH (1 %) solution to disperse smaller mineral and organic particles attached to them. Afterward, these  $FR_{sapr}$  were rinsed in water and dried in an oven at 105 °C for 24 h.

A general visual characterization of some  $FR_{sapr}$  was performed with a Zeiss Stemi 508 stereo microscope (Carl Zeiss Microscopy GmbH, Jena, Germany) with 50 × magnification. The images were collected using an integrated camera connected to the Zeiss Zen software. Figure 2c shows porous units that can be easily broken (Figure 2d). Evaluation of water retention in nine sampled points over the area performed in another study (data not yet published) showed that the amount of water content in saturated  $FR_{sapr}$  ranged from 0.2 to 0.4 g g<sup>-1</sup>. Thus,  $FR_{sapr}$  can be characterized as a porous material that contributes significantly to soil water retention capacity.

Some  $FR_{sapr}$  like that of figure 2c were used in the water retention analysis. In general, the procedure sequence consisted of: (i) preparing  $FR_{sapr}$  samples within predefined particle size classes; (ii) measuring water potential in four different water contents, and (iii) drying samples in an oven at 105 °C for 24 h, followed by determining sample weight (Figure 3). To start a new sequence, the  $FR_{sapr}$  of the previous sequence were fractured into a new smaller-size class, and steps (ii) and (iii) were repeated. The details in each of the four sequences (Figure 3) are described in the following section.

### Saprolite fragmentation

Saprolite fragments were transferred into ten WP4 sample cups. As the diameter of the WP4 sample cup is approximately 38 mm, the size of  $FR_{sapr}$  varied from 38 to 4 mm (Figure 3). After measuring water potential in these samples (described later), the  $FR_{sapr}$  were fractured into a new smaller-size class (Figure 3). Inside a porcelain mortar, saprolite was fractured with a pestle until all particles passed through a 4 mm mesh sieve. The material was then passed through a 2 mm mesh sieve, and  $FR_{sapr}$  larger than 2 mm were transferred to the WP4 sample cup. The size of  $FR_{sapr}$  of these new samples varied from 4 to 2 mm. The  $FR_{sapr}$  smaller than 2 mm were stored to be added to the next smaller-size class. The  $FR_{sapr}$  in the range of 1-0.25 mm (Figure 3) were prepared by fracturing the fragments of the 4-2 mm class plus the stored  $FR_{sapr}$  smaller than 2 mm until all particles passed through a 1 mm mesh sieve. The material was sieved with a 0.25 mm mesh, and



**Figure 3.** Schematic view of how the samples were prepared and water potential was measured.

the  $FR_{sapr}$  larger than 0.25 mm were transferred to the WP4 sample cup. The  $FR_{sapr}$  smaller than 0.25 mm were stored. Finally, the  $FR_{sapr}$  smaller than 0.25 mm were prepared by fracturing the  $FR_{sapr}$  of the 1-0.25 mm class until all passed through a 0.25 mm mesh sieve. This material was joined to the previously stored  $FR_{sapr}$  lower than 0.25 mm, and the total material was transferred to the WP4 cup.

### Water potential measurements

Initially, the samples were moistened by spraying water on them, followed by homogenization. Immediately, the water potential ( $\Psi$ , MPa) was read in the WP4 (Decagon Devices, Inc.; METER Group, Inc). Previously the WP4 calibration was checked using a KCl 0.5 mol L<sup>-1</sup>. In general,  $\Psi$  was higher than -1 MPa. As the WP4 has low accuracy for  $\Psi$  above -1 MPa (Klein et al., 2010; Gubiani et al., 2012), the samples were removed from the chamber, left unsealed for water evaporation, and  $\Psi$  was measured again. This procedure was repeated until water potential remained higher than -1 MPa. As water potential became equal or lower than -1 MPa, the WP4 sample cup was sealed for 24 h to allow water redistribution in the sample (Campbell et al., 2007). The following day, water potential was measured and this value was then recorded. Immediately, the weight of sample was measured and recorded. The sequence composed of  $\Psi$  approximation, water redistribution for 24 h, and  $\Psi$  and water content (WC, g g<sup>-1</sup>) measurement aforementioned was repeated in the same sample to take  $\Psi$  and its corresponding WC close to other pre-defined  $\Psi$  values of -2, -5, and -10 MPa (Figure 3). At the end of the fourth step, the samples were transferred into a metal cup, dried in an oven at 105 °C for 24 h, and weighed to calculate WC corresponding to the measured  $\Psi$  values.

### Supplemental data

To highlight the effect of excluding coarser particles on the relationship between WC and  $\Psi$  determined with WP4, we evaluated data presented at the 10th South-Brazilian meeting of soil science (Pigatto et al., 2016), which is not published elsewhere. Four water retention curves were determined by combining water retention data from undisturbed samples (120 cm<sup>3</sup>) at saturation and at -6, -10, -33, and -100 kPa (sand column and pressure-plate extractor) with data from disturbed samples analyzed with WP4, both collected from a gravelly Ultisol (*Neossolo*). These samples contained 35 to 58 % of quartz gravel. As the size of these coarse particles ranged between 2-10 mm, it was not needed to break them to evaluate water retention in the WP4. Two types of samples were used in the WP4. One containing all particle sizes and another composed of only fine fraction of particles smaller than 2 mm. In samples of approximately 5 g,  $\Psi$  was measured using the normal mode (not continuous) of the WP4, following the procedure described in Gubiani et al. (2012). The van Genuchten (1980) equation was fitted on the dataset combining WC and  $\Psi$  from undisturbed and those from disturbed samples containing all particle sizes. The continuous curve of the fitted function was depicted, and all measured data was shown in a graph for visual evaluation. Special attention was given to comparing WP4 data of samples containing all particle sizes and samples composed of only particles smaller than 2 mm.

### Data analysis

The forty relationships between WC and  $\Psi$  (10 samples × 4 subsamples = 40 curves) were shown in graphs used to visually evaluate the effect of the size of the saprolite fragments ( $SZ_{frag}$ ) on the relationship of WC and  $\Psi$ . In each curve, the power function  $WC = a|\Psi|^b$  (Campbell and Shiozawa, 1992) was fitted using the Marquardt nonlinear procedure (SAS Institute, 1999). The coefficient of determination ( $R^2$ ) and the sum of squared residuals (SSR) were used to evaluate the goodness of fit. Using the estimated parameters  $a$  and  $b$  of the power function, the WC for  $\Psi = -1.5$  MPa was calculated and considered the WC at permanent wilting point ( $WC_{PWP}$ ). Finally, the effect of  $SZ_{frag}$  on  $WC_{PWP}$  was evaluated by ANOVA, and the Tukey test was used to evaluate the differences in means.

## RESULTS

The monotonically decreasing trend of curves of the relationship between WC and  $\Psi$  (Figure 4) indicate that WP4 measurements in  $FR_{sapr}$  are consistent. A visual analysis of these curves revealed no systematic effect of the  $SZ_{frag}$  on the relationship between WC and  $\Psi$ , because the relative position between curves is random.

The minimum weight of  $FR_{sapr}$  samples was 0.96 g, which allows accurate measurements with WP4. Even better, in 81 % of the samples, weights ranged between 2 and 5.4 g (Figure 5a). All fitting of model  $WC = a|\Psi|^b$  on curves of figure 4 were significant at  $p = 0.05$ . The frequency of  $R^2$  in classes  $0.6 \leq R^2 < 0.7$ ,  $0.7 \leq R^2 < 0.8$ ,  $0.8 \leq R^2 < 0.9$ ,  $R^2 \geq 0.9$  was 2, 5, 6, and 34, respectively. The sum of squared residuals of WC was lower than  $0.003 \text{ g g}^{-1}$ , which proves that the model  $WC = a|\Psi|^b$  fitted very well to the measured data.

The F test of ANOVA did not detect significant effect of  $SZ_{frag}$  on  $WC_{pWP}$  (Figure 5b). The average  $WC_{pWP}$  in  $SZ_{frag}$  classes varied within a narrow range of 0.06 to 0.09  $\text{g g}^{-1}$ , and a consistent trend in increasing  $WC_{pWP}$  with  $SZ_{frag}$  decreasing was not observed.

A significant difference in water WC determined with WP4 between samples containing (i) all particle sizes of a gravel soil (35 to 58 % of quartz gravel) and (ii) the same samples that had these large particles removed (sample with particles smaller than 2 mm) was observed in favor of the samples containing only particles smaller than 2 mm (Figure 6). The difference in WC ranged from 0.08 to 0.12  $\text{g g}^{-1}$  at -1500 kPa (-1.5 MPa).

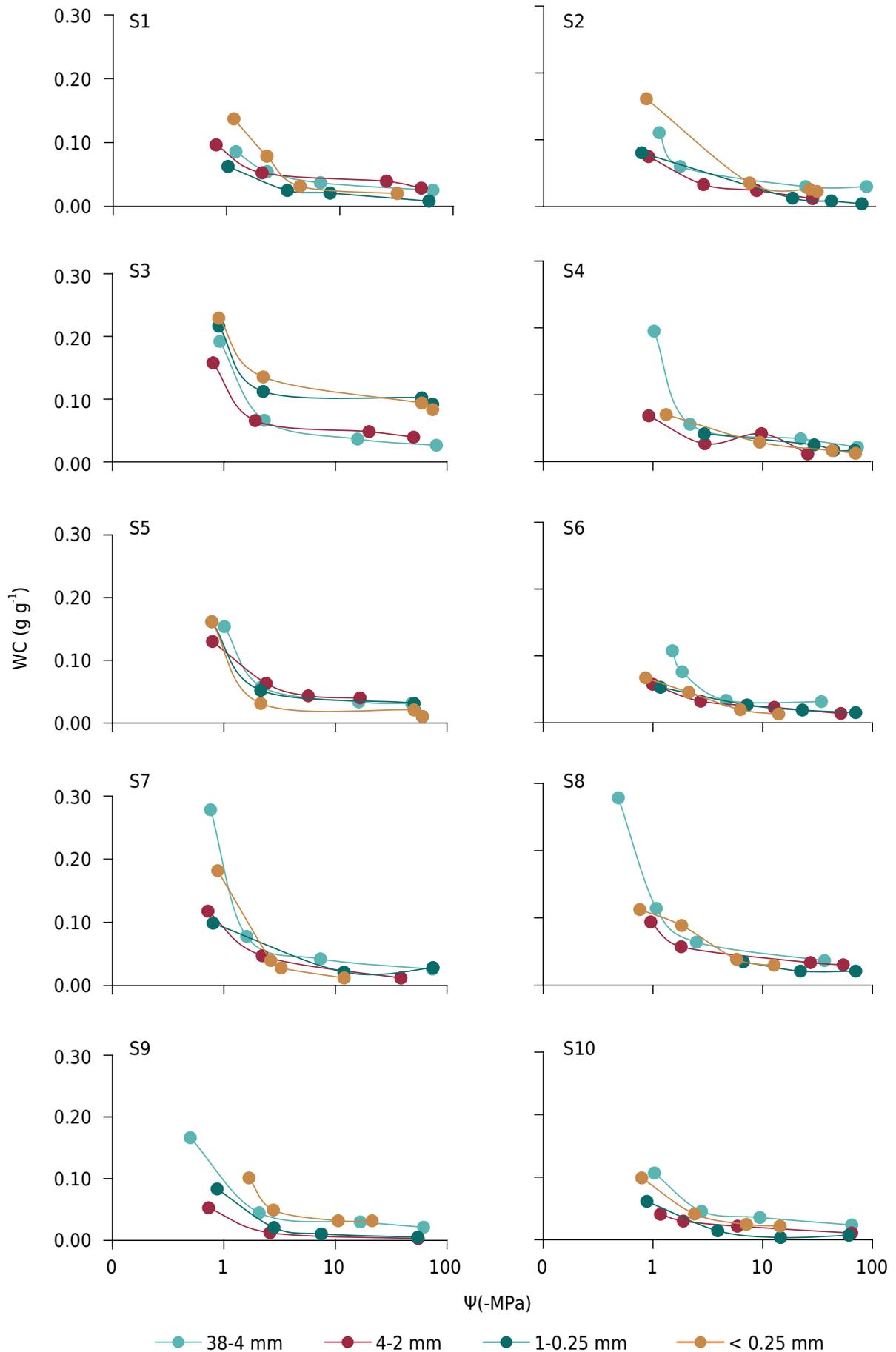
## DISCUSSION

If the reduction of  $SZ_{frag}$  implied an increase in water retention, the curves of smaller size classes should be shifted upwards. Curves of the smallest  $SZ_{frag}$  classes (orange and green lines) are above the others only in sample S3 (Figure 4). In the other samples, all curves are close to one another, and their relative positions seem to be random.

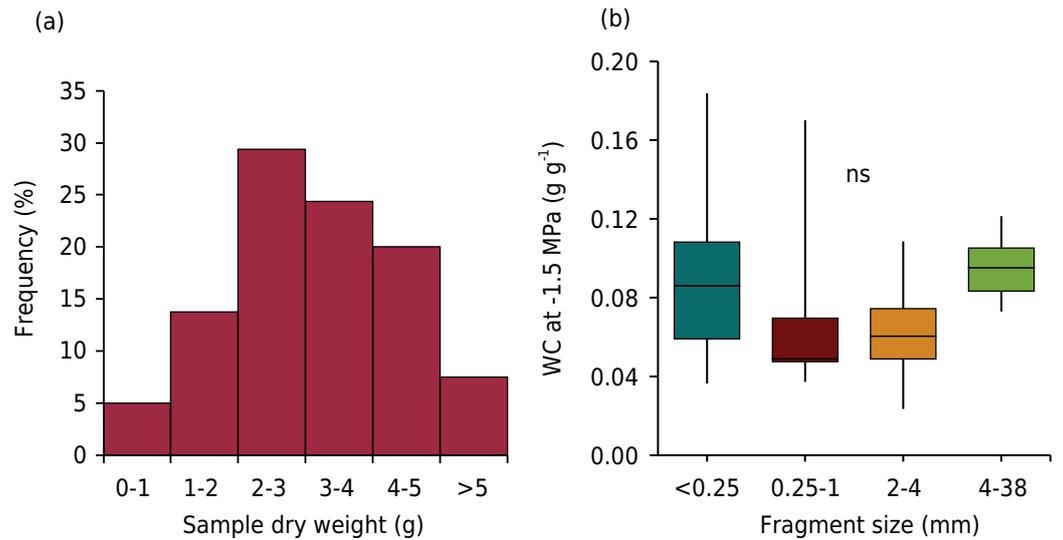
The shape of the curves (Figure 4) indicates they were accurately determined with samples weighing from 0.96 to 5.4 g (Figure 5a). Although WP4 can accurately measure a sample that does not (or cannot) cover the bottom of the cup (Decagon Devices, 2000), samples with weights of around 4 g are preferred to minimize the unavoidable errors in determining weight due to scale resolution limit (a scale with 0.01 g was used). Furthermore, larger samples speed up reading by shortening the time needed to reach liquid-vapor equilibrium and increase instrument efficiency by providing more stable infrared sample temperatures (Decagon Devices, 2000).

As previous reported in several studies (Campbell and Shiozawa, 1992; Gubiani et al., 2012), the power function accurately described all the relationships of WC and  $\Psi$  of this study (Figure 4). Their  $R^2$  values above mentioned indicate that the  $WC_{pWP}$  estimated with the fitted function at  $\Psi = -1.5 \text{ MPa}$  (Figure 5b) is statistically reliable.

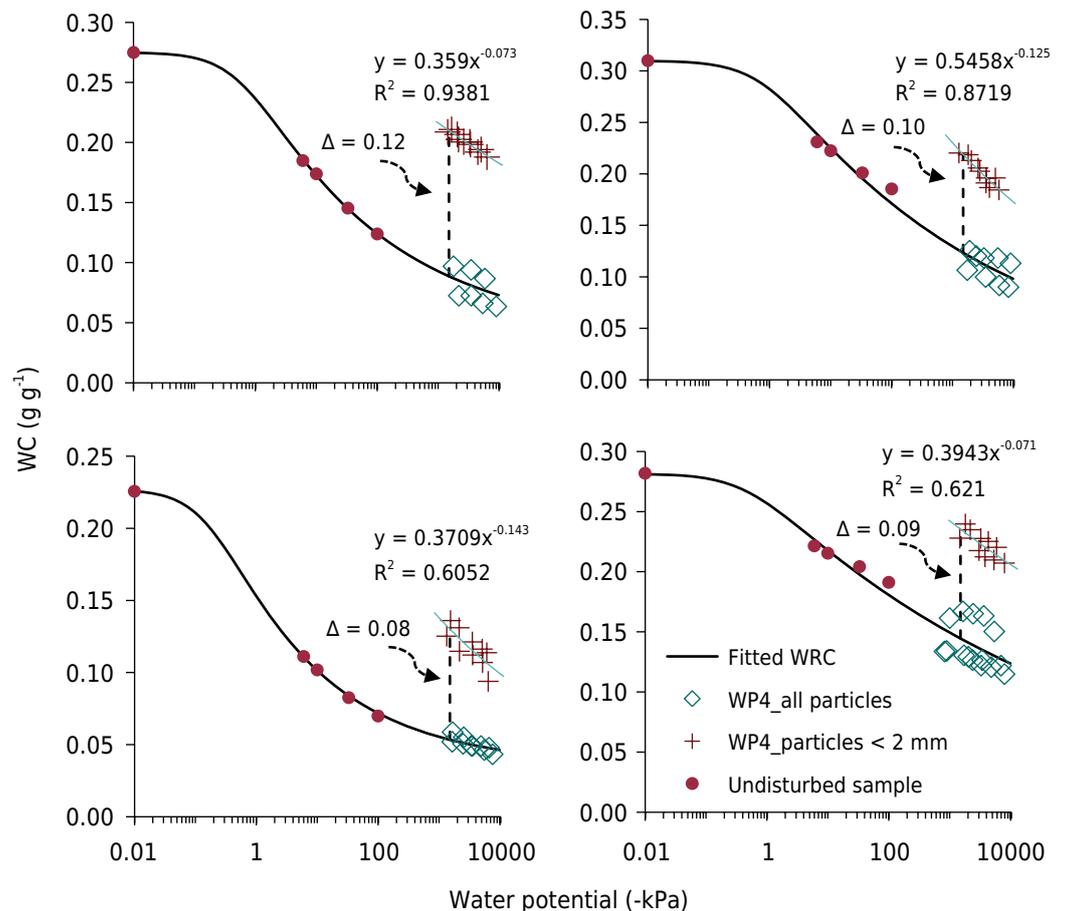
The no systematic effect of the  $SZ_{frag}$  on the relationship between WC and  $\Psi$  (Figure 4) indicates that fracturing saprolite samples from sizes of 38-4 mm to sizes smaller than 0.25 mm did not sufficiently increase specific surface area ( $\text{m}^2 \text{ g}^{-1}$ ) to provoke a detectable change in  $WC_{pWP}$ . Using the relationship shown in figure 1, a mere increase of  $0.01 \text{ m}^2 \text{ g}^{-1}$  in specific surface area would be caused in non-porous, spherical particles by reducing their diameter from 38 to 0.25 mm. The increase in surface area should have been much lower in the  $FR_{sapr}$ . As the  $FR_{sapr}$  were porous material (Figure 2d), which retained from 0.2 to 0.4  $\text{g g}^{-1}$  of water at saturation (data not shown), they have internal surface area. Thus, fracturing porous  $FR_{sapr}$  creates a lower surface area compared to the fracturing of a non-porous material. Furthermore, if the purpose of reducing the size of  $FR_{sapr}$  is only to enable samples



**Figure 4.** Water content (WC) and potential ( $\Psi$ ) relationship for ten samples of saprolite (S1 to S10) fractionated into different sizes (legend of the upper left graph).



**Figure 5.** Frequency of saprolite sample weight classes (a) and distribution of water content (WC) estimated at -1.5 MPa with the fitted power function  $WC = a|\Psi|^b$  in saprolite samples with different sizes (b).  $n = 10$  in each size class; ns = not significant by the F test of ANOVA at  $p = 0.05$ .



**Figure 6.** Water retention curve combining data from undisturbed and disturbed samples containing all particle sizes. Water retention of disturbed samples composed of only particles smaller than 2 mm were disregarded in the fitting procedure. Data from Pigatto et al. (2016).

to fit in the WP4 sample cup, fragment sizes do not need to be reduced to as much as 0.25 mm, because the operability of WP4 is not restricted to using sizes such as 2-4 mm. By reducing the diameter of either non-porous or porous particles from 38 to 2 mm, the increase in the specific surface is too little ( $0.001 \text{ m}^2 \text{ g}^{-1}$ ), and its effect

on  $WC_{pWP}$  may be negligible. Thus, reducing saprolite sizes into this range makes WP4 measurement feasible yet accurate.

All evidences of this study indicate that the WC and  $\Psi$  relationship in soils containing rock fragments larger than the diameter (38 mm) of the WP4 sample cup can be accurately assessed with WP4 after fracturing the larger fragments into workable sizes. This allows us to take in account coarser particles in WP4 measurements and avoid overestimations in the soil water retention curve if only fine earth is used. These overestimations were well evidenced by Pigatto et al. (2016) when they removed large soil particles and analyzed WC in WP4 using a sample containing only particles smaller than 2 mm (Figure 6). The magnitude of overestimation ( $0.08$  to  $0.12 \text{ g g}^{-1}$  at  $-1500 \text{ kPa}$ ) errors is closer to the amount of available water capacity in most soils (Reichert et al., 2020). Furthermore, when all particles were used in WP4 measurements, the fitted water retention curve matched very well the wet range of WP4 measurements performed in undisturbed samples. Using only WP4 measurements from particles smaller than 2 mm would result in an unreliable water retention curve.

Our results showed that the  $WC_{pWP}$  needs to be measured on the same bulk sample when testing soils with rock fragments (potentially crushed to fit in the WP4 sample cup), not on a subsample of particles that pass through a 2 mm sieve. The WP4 measurements are generally performed using only particles smaller than 2 mm, because the fraction of particles larger than 2 mm could be negligible for heavily weathered soils. However, agriculture is expanding to marginal areas around the world (Bruinsma, 2003; Laurance et al., 2014). The coarser material of these soils must be taken into account in WP4 measurements to avoid overestimating water retention from using only the fine particle sizes. Our results suggest that soil fragments larger than the diameter of the WP4 sample cup can be crushed down to a size of 0.25 mm without affecting the relationship of water content and water potential. However, we suggest breaking coarse fragments apart from fine earth (to avoid sand breakdown) into sizes between 4-2 mm. Coarse particles of 4-2 mm can be easily remixed in the fine earth, and the resulting composed material can be easily poured into the WP4 sample cup.

## CONCLUSION

Disregarding coarser soil particles in WP4 measurements may result in a significant overestimation of water retention for bulk soil. For the soils evaluated in this study, the overestimation of water retention at  $-1.5 \text{ MPa}$  ranged from  $0.08$  to  $0.12 \text{ g g}^{-1}$ , which is equivalent to the available water capacity of several soils.

Particles larger than the diameter of the WP4 sample cup can be broken into several sizes from 38 down to 0.25 mm without affecting the water content and water potential relationship. By reducing coarse particles into this range, the WP4 measurements are feasible and accurate. Thus, we can avoid overestimating water retention in bulk soil when determined only with fine particles.

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**Writing - review and editing:**  Caroline Andrade Pereira (equal) and  Paulo Ivonir Gubiani (equal).

**Supervision:**  Caroline Andrade Pereira (equal) and  Paulo Ivonir Gubiani (equal).

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