



A methodological framework to determine optimum durations for the construction of soil water characteristic curves using centrifugation

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Abstract

During laboratory assessment of the soil water characteristic curve (SWCC), determining equilibrium at various pressures is challenging. This study establishes a methodological framework to identify appropriate experimental duration at each pressure step for the construction of SWCCs via centrifugation. Three common temporal approaches to equilibrium – 24-, 48- and 72-h – are examined, for a grassland and arable soil. The framework highlights the differences in equilibrium duration between the two soils. For both soils, the 24-h treatment significantly overestimated saturation. For the arable site, no significant difference was observed between the 48- and 72-h treatments. Hence, a 48-h treatment was sufficient to determine 'effective equilibrium'. For the grassland site, the 48- and 72-h treatments differed significantly. This highlights that a more prolonged duration is necessary for some soils to conclusively determine that effective equilibrium has been reached. This framework can be applied to other soils to determine the optimum centrifuge durations for SWCC construction.

Keywords

centrifuge • equilibrium • soil water characteristic curve

Introduction

The soil water characteristic curve (SWCC) describes the relationship between volumetric water content (θ) of a soil and the matric potential (Ψ) (Fredlund and Xing, 1994). It has been termed 'the most important curve in soil physics' (Vogel, 2014), due to its many applications in agriculture and environmental research (Hillel, 1998). The curve has been used to describe plant-soil water relations (Gardner and Ehlig, 1963); to assess shear strength (Vanapalli *et al.*, 1996), physical quality (Dexter, 2004a, 2004b, 2004c), water and solute transport (Šimůnek *et al.*, 2013; Vero *et al.*, 2014) and pore size distribution (Aschonitis *et al.*, 2012), among other applications. Hydraulic parameters derived from the SWCC (e.g. van Genuchten, 1980; Brooks and Corey, 1966) are critical inputs for numerical models used to ascertain solute and nutrient travel times through the unsaturated zone (Vero *et al.*, 2014; Fenton *et al.*, 2015; Vero *et al.*, In press).

Traditionally, the drying phase of the SWCC has been assessed using laboratory devices, including pressure plates (Richards, 1948, 1965), Tempe cells (Reginato and Bavel, 1962; ASTM D6836-02, 2008) and hanging columns (ASTM D6836-02, 2008), which apply pressure or suction, thus forcing water from the samples. However, these methods can be slow and arduous (Gee *et al.*, 2002; Bittelli and Flury, 2009; Dexter *et al.*, 2012; Gubiani *et al.*, 2012) and susceptible to

errors, particularly at low water potentials (Peck and Rabbidge, 1969; Campbell, 1988; Cresswell *et al.*, 2008; Bittelli and Flury, 2009). Cresswell *et al.* (2008) noted that shrinkage and dispersion of colloids may impair the ability of some soil types to equilibrate at specified Ψ , using pressure plates. Furthermore, these methods typically pertain to small and disturbed soil samples, which may not reflect the influence of soil structure on hydraulic properties (Young *et al.*, 2001; Lin, 2011). Wetting curves, representing the period during which fluid is imbibed by a soil sample on being subjected to various pressures, also may be measured (Fredlund and Xing, 1994; Yang *et al.*, 2004), or they may be inferred in cases where more easily measured drying curve data are available (Šimůnek *et al.*, 1999). This study focusses only on the drying curve of intact soil samples. Current commercial options offer more rapid assessment of the SWCC, e.g., HYPROP (Schindler *et al.*, 2010) or the vapour sorption analyser (both Decagon Devices, Pullman, WA, USA). A non-commercial alternative is to adopt the centrifuge method (ASTM D6572, 2008; ASTM D6836-02, 2008), which can be adapted for specific analytical purposes, to accommodate standardised bulk density (ρ_s) rings, according to the model of centrifuge available (Nimmo, 1990; Šimůnek and Nimmo, 2005; Reis *et al.*, 2011). Recent research on the SWCC has addressed knowledge gaps pertaining (but not limited) to hysteresis (Caron *et al.*,

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2014), thermal effects (Campbell and Wacker, 2014), the number of applied pressure steps (Cobos, 2014), modelling approaches (Diamantopoulos and Durner, 2014; Durner, 2014) and the influence of inherent soil properties (such as ρ_s , organic matter content and texture) on SWCC shape (Jensen *et al.*, 2014). Another acute knowledge gap pertains to the identification of hydraulic equilibrium. This is the point at which no pressure gradient exists within the soil sample and the water held in the pores is at an equal potential to the applied pressure or suction. In each of the aforementioned methods, Ψ is applied until dewatering ceases, indicating that hydraulic equilibrium has been attained, at which point the Ψ is increased incrementally (Briggs and McLane, 1907). However, determining total equilibrium (E) presents a major practical challenge, as dewatering of a sample in response to applied Ψ is non-linear and may continue at low levels over a prolonged period (Cropper *et al.*, 2011; Dexter *et al.*, 2012). Nuth and Laloui (2008) and Malaya and Sreedeeep (2012) note that 'characteristic' implies that a unique and distinctive SWCC exists for a specific soil. This suggests that measurement effects may lead to the construction of curves that are neither reflective nor characteristic of the soil in question, with implications for practical applications based on such data and on the durations required to attain complete dewatering/ E (Fredlund and Xing, 1994). It is possible that a level of dewatering may be attained for each individual soil, which approximates equilibrium for all practical purposes, beyond which, further dewatering may not significantly influence the shape of the SWCC.

Centrifugation was initially proposed as a method of SWCC assessment in the early 20th century (Briggs and McLane, 1907; Russell and Richards, 1938). This method applies Ψ by artificially inducing a high gravitational force via increases in revolutions per minute (rpm) (Khanzode *et al.*, 2002; Rao and Singh, 2012). This force causes the downward movement of pore water through, and then from, the soil sample. However, high-speed centrifuges were not widely available prior to the distribution of commercial centrifuges in the 1940s and 1950s (Thackery and Myers, 2000). The limitations of these early models (including temperature fluctuations and restricted rotor speeds) meant that the slower methods, such as pressure plates and hanging columns, became established as *de rigueur* (Reatto *et al.*, 2008). Modern high-speed, refrigerated centrifuges allow such technological limitations to be overcome, which has led to renewed interest in the method. This method is not without its disadvantages; soil structure may be altered both by centrifugation, particularly at high speeds (Nimmo and Akstin, 1988), and by the exertion of a range of Ψ along the soil sample (Khanzode *et al.*, 2002; Smagin, 2012). However, due to the large degree of control over Ψ in the centrifuge method, decreased experimental duration and the capacity for high-resolution measurements

of dewatering over a wide range of Ψ , the centrifuge method has gained popularity for the construction of detailed SWCCs and the measurement of hydraulic properties (Nimmo *et al.*, 1987; Smagin *et al.*, 1998; ASTM D6527, 2000; Khanzode *et al.*, 2002; Caputo and Nimmo, 2005; ASTM D6836, 2008; Reatto *et al.*, 2008; McCartney and Zornberg, 2010; Cropper *et al.*, 2011; Smagin, 2012). Satisfactory agreement has generally been found in the literature between SWCC measurements obtained via centrifugation and other methods (Khanzode *et al.*, 2002; Caputo and Nimmo, 2005). Smagin (2012) noted that no method has yet been established as a benchmark against which SWCCs may be assessed, which is particularly true considering the diversity of sample formats and sizes observed throughout the literature (Table 1).

It is accepted that the optimum method of SWCC analysis maintains each pressure step until dewatering has entirely ceased (ascertained by monitoring the weight of the sample until no further changes are observed when subsequent pressure is applied) (Nimmo, 1990; Khanzode *et al.*, 2002; Šimůnek and Nimmo, 2005). However, this may take weeks, particularly when large or undisturbed samples are used, and successive changes in weight become increasingly minor as equilibrium is approached (Vomocil, 1965). This has resulted in the imposition of rules regarding the duration of the pressure step, which assume that hydraulic equilibrium is attained after a predetermined duration. A wide range of experimental durations are proposed in the literature (Table 1), but these typically correspond only to very small or disturbed soil samples and, hence, may not wholly reflect the hydraulic properties of in situ soils (Schjønning *et al.*, 1999; Costa *et al.*, 2008; Lin, 2011). Furthermore, a conflicting array of sample dimensions, formats and methods of preparation has been documented (Table 1), making the identification of the appropriate duration extremely difficult. Even when these rules are well suited to the conditions described therein, the cores used in such experiments tend to be non-standardised and challenging to replicate; in addition, a range of sample sizes, conditions and experimental durations are observed throughout the literature (Table 1). Consequently, determination of the true or total equilibrium state in many instances has relied upon arbitrary decisions (Vomocil, 1965), reflecting methodological limitations. While the importance of achieving equilibrium prior to adjustment of applied pressure is universally acknowledged in the literature (Russell and Richards, 1938; Nimmo, 1990; Khanzode *et al.*, 2002; Hunt and Skinner, 2005; Šimůnek and Nimmo, 2005), there is no practical method of assessing its status in standard laboratory centrifuges without complex in-flight monitoring equipment (Reis *et al.*, 2011), which may be unavailable in many laboratories. It is hypothesised that an 'effective equilibrium' may be reached (at each pressure step) beyond which prolonged centrifugation will not yield further significant dewatering or changes in the SWCC. As

assessment of true/total equilibrium may in many cases be uncertain (Vomocil, 1965; Vogel, 2014), methodologies in which effective equilibrium may be discerned would allow soil physics practitioners to determine testing durations that best reflect true equilibrium.

The objective of this study was to examine the differences in the SWCC resulting from the application of temporal rules commonly seen in the literature (Table 1) and to establish a methodological framework to identify the appropriate experimental duration to ascertain effective equilibrium (at each pressure step) within the bounds of an imposed experimental design. This framework facilitates reliable raw data collection for the construction of SWCCs.

Materials and methods

Centrifuge method setup

The SWCC was measured in accordance with the centrifuge method described by Nimmo *et al.* (1987), Reis *et al.* (2011) and Šimůnek and Nimmo (2005). The apparatus used was a Sigma 6-16KS refrigerated centrifuge (Sigma Laborzentrifugen GmbH, Ostrode, Germany), with an 11150 model four-bucket rotor and bespoke adaptors designed to fit within the centrifuge buckets. The bespoke adaptors (Figure 1) were designed to facilitate the use of 5 × 5 cm (Peerlkamp and Boekel, 1960; Reatto *et al.*, 2008; Moncada *et al.*, 2015) ρ_s rings commonly used in the field (Creamer, 2015). An Acculab Atilon ATL2202 balance (Sartorius, Goettingen, Germany), with a precision of

0.01 g was used to weigh the soil cores at specified intervals. During the structured experiment, the following pressure steps were applied: -50, -100, -150, -200, -1,000 and -1,500 kPa (grassland site) and -33, -100, -150, -200, -1,000 and -1,500 kPa (arable site). The gravitational force g required to achieve the desired pressures was as follows: 73 (48 at -33 kPa), 147, 220, 293, 1,467 and 2,201 g , respectively. This equated to 670 (544 at -33 kPa), 948, 1,161, 1,341, 2,998 and 3,672 rpm, respectively. Centrifuge speeds were determined according to Gardner's equation (Gardner, 1937) (Equation 1):

$$\psi = \frac{\rho\omega^2}{2} (r_2^2 - r_1^2) \quad (\text{Eqn. 1})$$

where r is the density of the pore fluid (grams per cubic centimetre), ω is the angular velocity (radians per second), r_1 is the radial distance to the midpoint of the soil sample (centimetres), and r_2 is the radial distance to the free water surface (centimetres). After each specified time step (e.g., 24-, 48- or 72-h, pressure was incrementally raised by adjusting the speed of the centrifuge. Further details of the apparatus and methods used are available in the reports of Hassler and Brunner (1945), Cronney *et al.* (1952), ASTM D6836 (2008) and Dane and Topp (2002). At the end of each complete cycle, the cores were dried at 105 °C for 48-h to determine the water content (θ , in percentage). SWCCs were then constructed for each of the three temporal treatments (24, 48 and 72 h) in the structured experiment by plotting θ against ψ in kilopascals.

Table 1. Summary of methodologies used in the literature to determine the SWCC

References	Methods	Sample description			Pressure step duration (h)
		Height (cm)	Diameter (cm)	Format	
Russell and Richards (1938)	Centrifuge	0.5	3.5	Disturbed	<4
Nimmo (1990)	Centrifuge	3.8	2.5	Intact	3–72
Smagin <i>et al.</i> (1998)	Centrifuge	1–3	Unspecified	Disturbed	0.25–48
Vanapalli <i>et al.</i> (1996)	Pressure plates	0.63	10	Disturbed	Unspecified
Khazode <i>et al.</i> (2002)	Centrifuge	1.2	7.5	Disturbed	2–48
	Tempe cells		Unspecified	Disturbed	336–2,688
Bittelli and Flury (2009)	Pressure plates	3	5.35	Intact	48
		1	5.35	Disturbed	48
McCartney and Zornberg (2010)	Centrifuge	12.6	Unspecified	Compacted	Average: 10
Cropper <i>et al.</i> (2011)	Centrifuge	4.78	3.89	Disturbed	2–18
Reis <i>et al.</i> (2011)	Centrifuge	5	2	Intact	Unspecified
Smagin (2012)	Centrifuge	10	1	Intact	4–8
				Disturbed	

Physical characterisation of the soil (Table 2) was conducted at Teagasc, Environment Research Centre, County Wexford, Ireland. Particle size analysis was performed using laser diffraction (Konert and Vandenberghe, 1997), and particle density was determined using the pycnometer method, in accordance with ASTM D854-14 (2014).

Initial testing

Intact soil cores ($n = 4$) from a permanent grassland site were excavated from the top 10 cm of soil, within a 1-m² area, on a local research farm. Textural analysis is given in Table 2. Centrifugation was initially conducted at -20 kPa (29 g/424 rpm). During the centrifuge run, samples were weighed every 60 min up to 76-h. This was a simple, preliminary test to observe the rate and shape of dewatering at a single pressure. Due to the arduous nature of this frequent data collection, this measurement resolution was not maintained for the complete SWCC. The mean high-resolution dewatering curve at -20 kPa is presented in Figure 2. Intervals similar to those observed in the literature (Table 1; Nimmo, 1990; Vanapalli *et al.*, 1996; Smagin *et al.*,

1998; Bittelli and Flury, 2009) (24-, 48- and 72-h) are indicated on the figure. The 48- and 72-h measurements exhibited lower θ (37.35% and 36.22%, respectively), compared to the 24-h measurement (38.99%) equivalent.

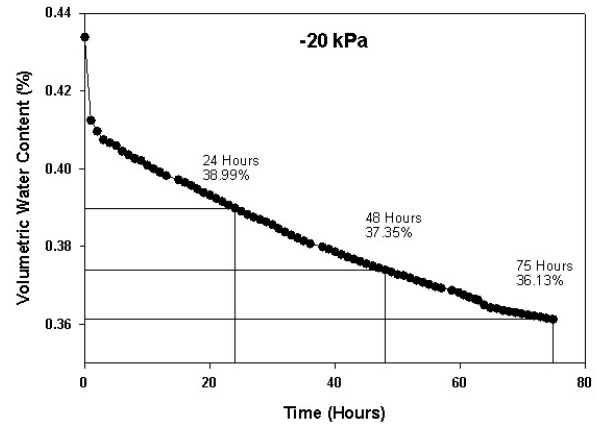


Figure 2. Dewatering at -20 kPa, assessed on an hourly basis.

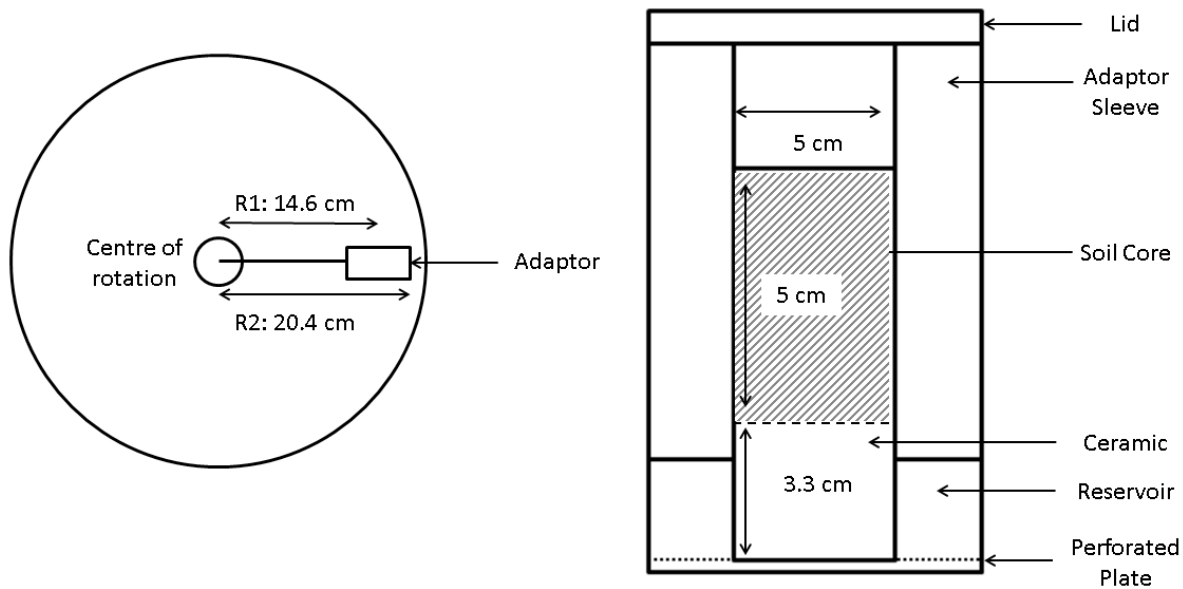


Figure 1. Diagram of the centrifuge (left) and bucket adaptor (right).

Table 2. Summary of site and soil information

	Site	Land use	Texture	Sand (%)	Silt (%)	Clay (%)	ρ_b (g/cm ³)	Particle density (g/cm ³)
Initial testing	Local soil	Permanent grassland	Loam	40	36	24	1.41	2.31
	Grassland	Permanent grassland	Clay loam	33	31	36	0.86	2.41
Experimental design	Arable	Malt barley	Clay loam	43	24	33	0.93	2.49

It was therefore decided that three durations commonly observed in the literature (Table 1) should be applied as treatments in a structured experiment, in which incremental changes in Ψ would be implemented after centrifugation for predetermined durations (24-, 48- or 72-h), and the resulting SWCCs should be statistically compared to develop a framework for other practitioners.

Experimental design and data analysis

The experimental treatment was centrifuge run time, which was applied for 24-, 48- or 72-h at seven pressure steps to the intact soil cores. After each time iteration, the sample was weighed. A completely randomised experimental design with four replicates was used to investigate the treatment effect for two separate sets of soil cores. The first set of soil cores was taken from a well-drained, permanent grassland site used for beef production (referred to as 'grassland') and the second set from a well-drained arable site under malt barley production (referred to as 'arable') in the south of Ireland. In both sites, the samples were taken from a 1-m² area. In terms of water quality, both watersheds are classified as vulnerable to nutrient transport through the vadose zone. A summary of the site and soils used in the study is presented in Table 2.

Unlike homogenised soil samples, the nature of intact soil cores is such that there is variability in the r_s and the initial saturated water content (θ_s). This is in agreement with the study by Šimůnek and Nimmo (2005), who reported significant scatter of θ_s , particularly close to saturation. In order to compare the effects of treatment without the confounding effect of differing initial saturation, θ_s (0 kPa) was taken as 100%, and the subsequent water contents were expressed on a relative basis (Figure 3) (Fredlund, 2002). The relative saturation approach enables direct comparison across the

treatments, irrespective of initial water content. Henceforth, these adjusted water contents will be referred to as 'effective saturation'.

The PROC MIXED procedure of SAS 9.3 (© 2002–2010, SAS Institute Inc., Cary, NC, USA) was used to conduct a repeated-measures analysis of variance (ANOVA) to test the effect of treatment on the effective saturation over incremental pressure steps for the two sites individually. The parameters included in the model for each site were time (24-, 48- and 72 h) and pressure (seven steps: 0, -33 or -50, -100, -150, -200, -1,000 and -1,500 kPa), as well as their interaction terms. Pressure was the repeated measure. Using the Akaike information criterion (AIC) to test the fit model, the compound symmetry covariance matrix structure was chosen. The AIC approach is commonly used as a means of model selection in soil physics (Verbist *et al.*, 2009; Savva *et al.*, 2010) and indicates the relative performance of two or more models by comparing the goodness of fit that they achieve. Least-squares means (LSmeans) are presented, and mean comparisons are conducted by *F*-protected least significant difference test. For all statistical analyses, *P*-values <0.05 were considered significant.

Results and discussion

The soils from the grassland and arable sites were similar in textural class but differed structurally owing to their different management and cropping history. While the r_s values of the grassland samples have been found to be low (Table 2), a 0.80–1.00 cm⁻³ range has been frequently reported for the surface horizon of Irish grassland sites (Lalor, 2004; Kiely *et al.*, 2009, Herbin *et al.*, 2011; Vero *et al.*, 2013). The values

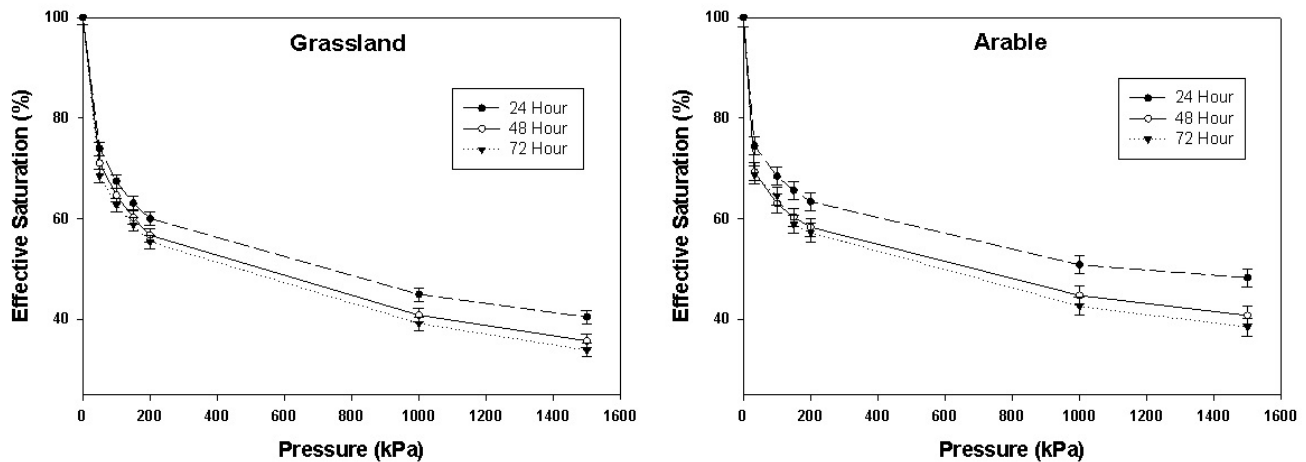


Figure 3. Effective saturation relative to initial water contents for the grassland and arable sites, according to treatment. Error bars indicate pooled standard error of the mean: 1.33 for the grassland site; and 1.79 for the arable site.

herein reflect minimal trafficking at the site and its high vulnerability to nutrient transport through the vertical pathway (Fenton *et al.*, 2011; Vero *et al.*, 2014).

Figure 3 displays the mean SWCCs constructed according to each treatment for both soils. There was a significant effect of pressure for both sites ($P < 0.0001$), which is expected, because dewatering increases as pressure is raised incrementally. Critically, there was a significant effect of treatment ($P < 0.0001$) for both the grassland and arable sites. Neither site exhibited a pressure \times treatment interaction ($P = 0.5539$ and $P = 0.0872$, for the grassland and arable sites, respectively). The 24-h treatments exhibited significantly greater effective saturation compared with the 48- and 72-h treatments, for both sites ($P < 0.001$). This suggests that a 24-h duration is likely to be insufficient to allow dewatering of intact samples. For the grassland site, the 48- and 72-h treatments were also significantly different from one another ($P = 0.0312$), indicating that effective equilibrium was not reached after 48-h and that a greater duration would be required to attain equilibrium for this soil. For the arable site, there was no significant difference between the 48- and 72-h treatments, indicating that 48-h is adequate to characterise the effective saturation of samples from this site. As such, 48-h can be recommended as the minimum centrifuge run time for soils from this specific arable site, less than which dewatering was insufficient to approximate effective equilibrium. For the grassland site, the minimum threshold cannot be established from the treatments described herein. As the P -value is approaching the significance threshold, the methodological framework suggests that the optimum duration for this soil may be close to the 72-h treatment already applied. These results demonstrate the difference between two sites that have similar textures but different structural properties. Based on the outcomes of the current study, additional replicates of soil samples should be collected to facilitate longer durations if needed. The methodological framework defined herein identifies the optimum experimental duration for a particular soil sample and therefore allows for the construction of a reliable SWCC. The construction of SWCCs within this methodological framework gives a practitioner greater confidence with respect to the application of the associated soil hydraulic data, provided that temporal durations are determined for their specific soils beyond which further dewatering is no longer significant.

Although not the case for the current soils, the duration required to attain effective equilibrium at specific Ψ may differ for other soils, which probably reflects the extent and tortuosity of the corresponding pores and, further, may probably be influenced by the composition (texture and structural characteristics) and size of the soil samples. Herein, the statistical analyses indicated that there was no significant interaction of Ψ and treatment for either site.

Hence, a single duration may be applied across all Ψ (e.g., 48-h for the arable site), in cases where it is not significantly different from a greater duration. The interaction on the arable soil was almost significant, indicating that this should be considered on a soil-by-soil basis. The important point here is that the current framework will identify the situations where this is the case, e.g., it is possible that for some soils, a more prolonged treatment would be required at high Ψ , in accordance with the report of Šimůnek and Nimmo (2005). The implication of determining the appropriate duration using the current experimental structure is that practitioners will be prevented from either using unnecessarily prolonged experimental durations, which limit the number of samples that may be processed, or applying too brief a duration, which results in SWCCs that are statistically different from the true measurement.

These results suggest that where the SWCC is measured, a level of dewatering exists approaching E_e , beyond which further centrifugation will not yield further significant information. Vogel (2014) alluded to the impracticalities of reaching true or total equilibrium, asking whether “we really need more accurate measurements for a curve that in principle is not really measureable?”, with the implication that such sufficient threshold levels of dewatering probably exist for specific applications. It is proposed that this threshold be termed ‘effective equilibrium’ (E_e), to distinguish it from true hydraulic equilibrium (E). It is clear from the results that this threshold is likely to be different for various soils, and testing at multiple temporal treatments (which may exceed the 72-h limit examined in the current study) is required in order to identify suitable experimental durations. Sufficient additional soil sampling may be required to facilitate this assessment, and eventual development of pedotransfer functions may allow inference from more easily assessed properties such as texture and density.

Conclusions

High-resolution dewatering measurements demonstrated the misleading effect of arbitrary temporal rules in the centrifuge method; however, for practical purposes, it remains necessary to identify suitable experimental durations, which will be influenced by soil-specific characteristics. The methodological framework presented herein highlighted the differences between the two soils tested in terms of equilibrium duration thresholds. For the arable soil, a 48-h time step was deemed most appropriate, whereas for the grassland soil, there was no time step within the experimental design (24-, 48- or 72-h) at which equilibrium was established. Hence, further prolonged treatment duration for this soil would be required, highlighting the need to take many replicates during field

work. This methodological framework can be applied by other practitioners to determine optimum durations for the construction of SWCCs using their specific setup. In future, this methodological framework can be applied to a greater range of sample sizes, soil textures and structural classifications.

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