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Time of soil water thermodynamic equilibrium during retention curve establishment using gamma-ray beam attenuation

Luiz Fernando Pires, Osny Oliveira Santos Bacchi, Klaus Reichardt

Abstract The soil water retention curve (SWRC) represents a fundamental part of the characterization of the soil hydraulic properties. The establishment of SWRC is usually time-consuming and presents several problems such as the difficulty of a correct judgment of the time of equilibrium. This work presents a new methodology that involves gamma-ray beam attenuation technique associated with the traditional pressure chambers, having as objective the more precise judgment of the time to attain equilibrium. The gamma-ray source used has an activity of 3.7 GBq consisting of ^{241}Am, with peak energy of 59.54 keV. For the determination of retention curves using the gamma-ray attenuation technique, an acrylic pressure chamber was projected and constructed to be positioned between the source and detector with the gamma-ray beam crossing the center of the chamber and the soil sample during water extraction. The proposed technique allowed, through a specifically elaborated software for data acquisition, to evaluate with precision the exact instant of the equilibrium and, consequently, to obtain the right moment to change the chamber pressure to a new desired level, leading to a reduction of the time required for the whole retention curve determination. The results obtained show that the gamma-ray attenuation technique can be very useful as an auxiliary tool to evaluate SWRC, utilizing the Richards chamber.

Key words gamma-ray attenuation • time of equilibrium • pressure chamber • soil water retention curve

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Introduction

The soil water retention curve (SWRC) is one of the main soil hydraulic characteristics that relates soil water content to water potential and involves the soil pore space system [18]. The establishment of soil water retention curves by the Richards method [17] is time-consuming and presents several difficulties. One particular problem is related to the correct judgement of the instant of the equilibrium condition, once the method involves a series of equilibrium points between the water in the soil sample and the water at chosen potentials imposed to a pressure cell [7].

Each equilibrium condition involves the period of time necessary to remove the excess of water retained in the soil at a higher energy step, in relation to the next step. When these conditions are reached the soil water content (θ) is measured and paired with each chosen soil water matric potential (ψ_m), equal to the air pressure (P) applied on the soil. According to Klute [7], for core samples of about 2 to 3 cm in height, a time of 2 to 3 days has been found to be sufficient to attain equilibrium. However, this equilibrium time depends on several boundary and system conditions, e.g. soil sample diameter and height, soil texture or particle distribution, temperature control in the pressure chamber during SWRC determination, conductance of the porous plate, and contact between sample and plate [4, 11, 16]. The main difficulty is, therefore, to know the time required to maintain the pressure in the chamber for each energy step before opening to measure the correspondent soil water content.

Taking these facts into account a new procedure for soil water retention curve evaluation, using gamma-ray beam attenuation was suggested in a theoretical approach by Bacchi *et al*. [3] and implemented by Pires *et al*. [15]. This new technique includes an adaptation of the conventional pressure chamber to a gamma-ray beam attenuation system. The chamber was built to permit the gamma-ray beam to pass through the soil sample allowing continuous soil water monitoring during each energy step of the SWRC determination [15].

In the present study, we used the gamma-ray beam attenuation with a more precise judgment of the time to attain equilibrium and with a better comprehension of the effects of potential gradients on the extraction of soil water during SWRC evaluations.

Material and methods

Gamma-ray beam attenuation system

The soil water content was monitored using a radioactive gamma-ray source of ^{241}Am having an activity of 3.7 GBq emitting monoenergetic photons of 59.54 keV. The detector was a 7.62 × 7.62 cm NaI(Tl) scintillation crystal coupled to a photomultiplier tube. Rectangular slit (1.0 cm high × 0.1 cm wide) lead collimators were adjusted and aligned between source and detector in order to produce a vertical slit beam. The counter is interfaced with a PC and the acquired data stored using the software MICROVIS (2000) [10], developed by Embrapa Agricultural Instrumentation (CNPDIA – São Carlos, Brazil). The radioactive source and detector were mounted 18.0 cm apart and the chamber containing the soil sample was centred and aligned between them (Fig. 1). During the water extraction period, θ evaluations by gamma attenuation were obtained at intervals of 99 s, continuously.

SWRC determination

Four core samples (3.0 cm high, 4.8 cm diameter, 55 cm^3 volume) were collected from profiles of a soil characterized as Geric Ferralsol (Table 1) at an experimental field in Piracicaba, Brazil (22°4'S; 47°38'W; 580 m above sea level) with aluminum cylinders (rings) at the soil surface layer (3–8 cm depth).

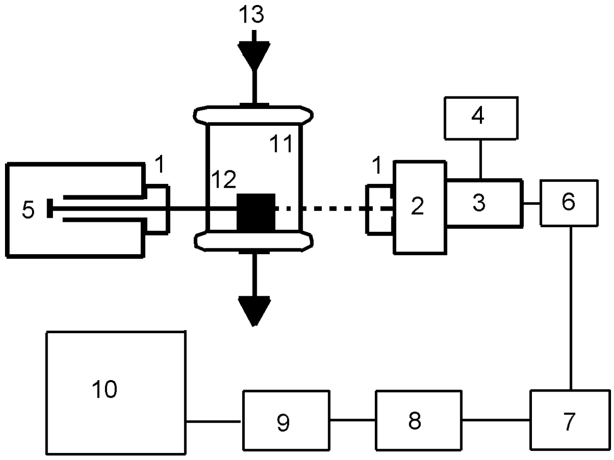

Fig. 1. Schematic diagram of the gamma-ray beam attenuation system associated to an adapted pressure chamber: 1 – lead collimators; 2 – NaI(Tl) detector; 3 – photomultiplier; 4 – high-voltage unit; 5 – ^{241}Am source; 6 – amplifier; 7 – monocanal analyzer; 8 – counter; 9 – timer; 10 – microcomputer; 11 – acrylic pressure chamber; 12 – soil sample; 13 – pressure input; 14 – water outflow.

In the laboratory, the excessive soil was carefully trimmed off each cylinder so that the volume of the ring was completely filled with the soil. The soil saturation process was the capillary rise, soaking soil cores in water at a level just below the top of the core [7]. This procedure was made over a period of 1 to 2 days to obtain best saturation of the samples, minimizing entrapped air in soil pores. After saturation cores were placed on the saturated ceramic plate inside the special acrylic pressure chamber (one sample for each SWRC determination) and respective air pressures were applied to the system allowing the samples to lose water and to come to the next step of equilibrium. Soil samples with a rigid structure and low clay contents were chosen in order to avoid possible changes in the soil structure induced by swelling and shrinkage.

Data processing

Assuming the soil structure remaining rigid and, consequently, the soil density constant during the SWRC determination by the gamma-ray attenuation technique, it is possible to obtain θ by solving the Beer-Lambert equation [19]:

$$\theta = \frac{-1}{x \cdot \mu_w \cdot \rho_w}\left[\ln\left(\frac{I}{I_0}\right) + x \cdot \mu_s \cdot \rho_s\right] \quad (1)$$

Table 1. Physical soil characteristics of 0–10 cm layer

Characteristics	Sand [%]	Silt [%]	Clay [%]	ρ_p* [g·cm^{-3}]	ρ_s [g·cm^{-3}]	ϕ** [%]
Geric Ferralsol	77	5	18	2.47	1.62	34.4

* ρ_p is the soil particle density.
** ϕ is the soil porosity obtained by $\phi = 1 - (\rho_s / \rho_p)$.

where: I and I_0 are, respectively, the rates of the emerging and the incident photon beams; μ_s and μ_w, $cm^2 \cdot g^{-1}$, the mass attenuation coefficients of soil and water; ρ_s and ρ_w, $g \cdot cm^{-3}$, the soil and water densities; and x, cm, the soil thickness (4.73 cm) crossed by the beam. The emerging radiation (I) was obtained after the photon beam passed through the acrylic chamber, aluminum cylinder, soil and air, and the incident radiation (I_0) after crossing all these materials without soil. I and I_0 were measured in sequence since one cylinder without soil was placed under the one containing soil. It was possible to measure I_0 moving the pressure chamber in the vertical direction immediately after determining I.

The linear attenuation coefficients for the soil ($\bar{\mu}_s$) and water ($\bar{\mu}_w$) were measured using the counting sequence of Conner *et al.* [5] and the mass attenuation coefficients[1] were then obtained from the measurements of $\bar{\mu}_s$ using knowledge of ρ_s and of $\bar{\mu}_w$ considering $\rho_w = 1\ g \cdot cm^{-3}$.

For the evaluation of $\bar{\mu}_s$, air-dried soil was passed through a 2.0 mm sieve and packed into a thin wall acrylic box ($10 \times 10 \times 10\ cm^3$). The intensities of monoenergetic photons having energies of 59.54 keV were measured in different positions of the soil into the box. The soil mass attenuation coefficient determined represents an arithmetic mean value of twenty repetitions. The very same experimental setup was used for the measurement of $\bar{\mu}_w$.

The determination of θ by the nuclear method has associated experimental errors. Through the error propagation of Eq. (1), it was possible to estimate the error associated to θ measurement:

$$\sigma^2\theta = \left(\frac{\partial\theta}{\partial I_0}\right)^2 \sigma^2(I_0) + \left(\frac{\partial\theta}{\partial I}\right)^2 \sigma^2(I) + \left(\frac{\partial\theta}{\partial X}\right)^2 \sigma^2(X) + \left(\frac{\partial\theta}{\partial \rho_s}\right)^2 \sigma^2(\rho_s) + \left(\frac{\partial\theta}{\partial \mu_w}\right)^2 \sigma^2(\mu_w) + \left(\frac{\partial\theta}{\partial \mu_s}\right)^2 \sigma^2(\mu_s) \quad (2)$$

where: σ(θ) is the total standard deviation; $\sigma(I_0)$ and $\sigma(I)$ are the standard deviation of I_0 and I, respectively. In laboratory conditions, the parameters x, ρ_s, μ_w and μ_s can be minimized turning very small the third, fourth, fifth, and sixth terms in Eq. (2). In this case, the spatial rates of change of θ may be considered directly related to the uncertainty of the radioactive decay process ($\sqrt{I}$). Solving the first and second partial derivatives in Eq. (2) the uncertainty in θ can be obtained using:

$$\sigma\theta = \frac{1}{x \cdot \mu_w \cdot \rho_w} \cdot \sqrt{\frac{\sigma^2(I_0)}{I_0^2} + \frac{\sigma^2(I)}{I^2}} \quad (3)$$

[1] The experimental mass attenuation coefficient μ was obtained by the Beer-Lambert law: $\frac{\bar{\mu}}{\rho} = \frac{1}{x \cdot \rho} \ln\left(\frac{I_0}{I}\right)$.

Time of equilibrium

To define the exact equilibrium time (t_e), θ was continuously monitored by gamma-ray beam attenuation for long periods of time (two or more days). The acquired data stored on a PC was used to obtain counts *vs.* time of count data. More than two thousand θ measurements were obtained for each soil core. The θ monitoring procedure was initiated 124 minutes before pressure application until a complete period of about 2000 min. After pressure application, the total counting time was divided at intervals of 124 min and average $\bar{\theta}$ values were obtained for each interval. The time of equilibrium was defined when $\bar{\theta}$ changes were within the error (σθ) associated with the uncertainty of the radioactive decay process and $\bar{\theta}$ values were statistically equal at the 95% probability level.

Results and discussion

The mass attenuation coefficients for soil and water were 0.25672 ± 0.00349 and 0.19895 ± 0.00245 $cm^2 \cdot g^{-1}$, respectively, for the 59.54 keV photons. For the analysis of the time of equilibrium, the pressure step (ΔP), between 0 and 0.10 MPa, was chosen since this interval is the most important during SWRC determinations due to the fact that it is related to the largest pores of the soil, which have great importance in agricultural research and practical agriculture.

In order to define t_e, I *vs.* t and θ *vs.* t plots were constructed (Fig. 2) for the Geric Ferralsol.

The greatest quantity of water removed from the soil sample occurs at the initial moments of pressure application, because of macropore emptying. Macropores are responsible for most water movement during the soil drainage processes [8]. On the other hand, due to the fact that the photon emission by a radioactive source is a random process, the determination of the equilibrium time using only photon counts is not a good criterion to be used. This happens because there is an interval of counts ($I + \Delta I$), related to the photon emission

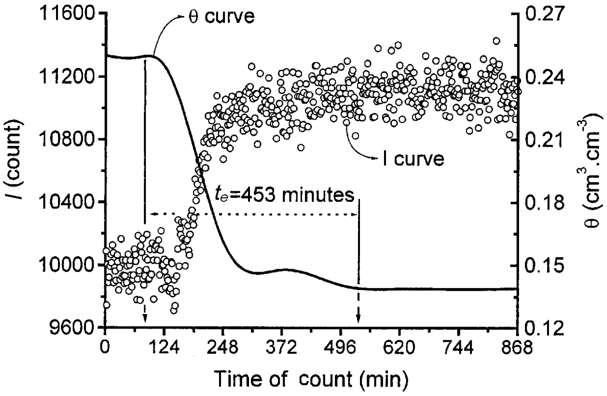

Fig. 2. Equilibrium curve of the data representing the variation of photon counts *vs.* time (empty circles) during pressure application and soil water variation (solid line) before (0–124 min) and after (124–868 min) pressure application *vs.* time to determine the equilibrium time (t_e) during soil water retention curve evaluations.

Table 2. Variation of the soil water content along the time during the determination of the equilibrium time

Interval of time [min]	$\bar{\theta}$ [$cm^3 \cdot cm^{-3}$]	$\bar{\theta} + \sigma\bar{\theta}$* [$cm^3 \cdot cm^{-3}$]	$\bar{\theta} - \sigma\bar{\theta}$* [$cm^3 \cdot cm^{-3}$]
A (0 to 124)	0.2524 (0.4)[a]	0.2588	0.2460
B (124 to 248)	0.2452 (5.7)[a]	0.2516	0.2388
C (248 to 372)	0.1613 (8.7)[b]	0.1677	0.1549
D (372 to 496)	0.1478 (1.7)[b,c]	0.1542	0.1414
E (496 to 620)	0.1406 (0.3)[c]	0.1470	0.1342
F (620 to 744)	0.1388 (2.0)[c]	0.1452	0.1324
G (744 to 868)	0.1386 (0.9)[c]	0.1450	0.1322
H (868 to 992)	0.1391 (2.0)[c]	0.1455	0.1327

The numbers in front of the parentheses are mean values; the numbers in the parentheses are coefficients of variation (%). The coefficient of variation represents the scatter of the 75 θ values for each specific interval of time. Superscripts represented by the same letter show subsets of the mean values that are statistically identical according to the Tukey test at $P = 95\%$.

* $\theta \pm \sigma\theta$ is related to the maximum and minimum θ values considering the error associated to the uncertainty of the radioactive decay process.

uncertainty, that does not permit to identify, with assurance, if the samples are retaining water at energies greater or lower than the chosen pressure step ΔP.

In order to obtain a more precise judgement of the equilibrium condition, the variations of θ were followed at distinct intervals of time (Table 2 and Fig. 2).

The interval of time between 450 and 536 min was required for an effective equilibrium for the four core samples. Each interval represents the average of seventy-five θ values, respectively. Although, $\bar{\theta}$ are statistically equal at the 95% probability level from the intervals *D* to *H* (Table 2), $\bar{\theta} + \sigma\bar{\theta}$ evaluated for the interval *E* is smaller than $\bar{\theta}$ for the interval *D* and the precise time of equilibrium was defined 40 min after this last interval, when the next $\bar{\theta}$ values were within the error ($\sigma\theta$) associated with the uncertainty of the radioactive decay process. An equilibrium time of 453 min was found for the soil sample presented in Fig. 2. The error ($\sigma\bar{\theta}$) associated with the uncertainty of the radioactive decay process was 0.0064 $cm^3 \cdot cm^{-3}$. The values of incident and emerging photons after pressure (*P*) application and respective equilibrium used for calculating $\sigma\bar{\theta}$ were 11161 ± 60 and 70397 ± 186 counts. The differences in the equilibrium time among samples should be related to soil sample heterogeneity or possible problems during sampling operations [14]. Although, samples have been collected next to each other, the procedure of sampling may have induced small undesirable modifications in the soil structure [2]. Alterations in the soil structure such as the deformation of large macropores during sampling, having as consequence changes in the representativeness of SWRCs, has been intensely discussed in the literature [1, 6, 9, 13]. Moraes *et al.* [12], working with an Eutric Nitosol, investigated the heterogeneity of the SWRC experimental points, identifying problematic points due to differences in the equilibrium time.

However, the results obtained for all samples allow concluding that about 500 min could be defined as the equilibrium time necessary for the Geric Ferralsol for low pressures. Reginato and van Bavel [16] reported, for sandy soils, periods of about 600 min for the equilibrium condition to be reached for low pressures. It is important to recognize that for each pressure step there is a specific time of equilibrium, which increases increasing the imposed applied *P*. This occurs due to the fact that for extremely small soil water potentials (higher *P*), it becomes hard to drive out the soil water retained in the pores.

Concluding remarks

This study confirms the ability of the nuclear method to guarantee a higher accuracy in the determination of the equilibrium time, which reduces the time required for the whole retention curve determination. The greatest advantage of this methodology based on nuclear techniques is to avoid sample weighing after each equilibrium, it is also not practical to remove the core from the pressure chamber because of the difficulty of re-establishing a good hydraulic contact between the sample and the porous plate [16] and the risks of soil structure damage due to frequent sample manipulation. In conclusion, a better definition of the equilibrium time is very important to minimize possible methodological problems during SWRC evaluations as reported by Moraes *et al.* [11].

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References

1. Albrecht BA, Benson CH (2001) Effect of desiccation on compacted natural clays. J Geotech Geoenviron 127:67–75
2. Assouline S, Tavares-Filho J, Tessier D (1997) Effect of compaction on soil physical and hydraulic properties: experimental results and modeling. Soil Sci Soc Am J 61:390–398
3. Bacchi OOS, Reichardt K, Oliveira JCM, Nielsen DR (1998) Gamma-ray beam attenuation as an auxiliary

technique for the evaluation of soil water retention curve. Scientia Agricola 55:499–502
4. Chahal RS, Yong RN (1965) Validity of the soil water characteristics determined with the pressurized apparatus. Soil Sci 99:98–103
5. Conner AL, Atwater HF, Plassmann EH, MacCrary JH (1970) Gamma-ray attenuation coefficients measurements. Phys Rev A 1:539–544
6. Croney D, Coleman JD (1954) Soil structure in relation to soil suction. J Soil Sci 5:75–84
7. Klute A (1986) Water retention: laboratory methods. In: Black CA (ed) Methods of soil analysis: I. Physical and mineralogical methods. ASA-SSSA, Madison, pp 635–662
8. Kutílek M, Nielsen DR (eds) (1994) Soil hydrolog. Catena Verlag, Germany
9. Lipiec J, Hatano R (2003) Quantification of compaction effects on soil physical properties and crop growth. Geoderma 116:107–136
10. Microvis (2000) Programa de Reconstrução e Visualização de Imagens Tomográficas, Guia do Usuário. EMBRAPA/CNPDIA, São Carlos, Brasil
11. Moraes SO, Libardi PL, Dourado Neto D (1993a) Problemas metodológicos na obtenção da curva de retenção da água pelo solo. Scientia Agricola 50:383–392
12. Moraes SO, Libardi PL, Reichardt K, Bacchi OOS (1993b) Heterogeneidade dos pontos experimentais de curvas de retenção da água no solo. Scientia Agricola 50:393–403
13. Ng CWW, Pang YW (2000) Experimental investigations of the soil-water characteristics of a volcanic soil. Can Geotech J 37:1252–1264
14. Pires LF, Bacchi OOS, Reichardt K (2004) Damage to soil physical caused by soil sampler devices as assessed by gamma ray computed tomography. Aust J Soil Res 42:857–863
15. Pires LF, Bacchi OOS, Reichardt K (2005) Soil water retention curve determined by gamma-ray beam attenuation. Soil Till Res 83:89–96
16. Reginato RJ, van Bavel CHM (1962) Pressure cell for soil cores. Soil Sci Soc Am J 26:1–3
17. Richards LA (1941) A pressure membrane extraction apparatus for soil solution. Soil Sci 51:377–386
18. Richards LA, Fireman M (1943) Pressure plate apparatus for measuring moisture sorption and transmission by soils. Soil Sci 56:395–404
19. Wang CH, Willis DL, Loveland WD (1975) Characteristics of ionizing radiation. In: Wang CH, Willis DL, Loveland WD (eds) Radiotracer methodology in the biological environmental, and physical sciences. Prentice Hall, England, pp 39–74