ISSN 1064-2293, Eurasian Soil Science, 2012, Vol. 45, No. 4, pp. 416–422. © Pleiades Publishing, Ltd., 2012.
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SOIL PHYSICS

Column-Centrifugation Method for Determining Water Retention Curves of Soils and Disperse Sediments

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Received July 27, 2010; in final form, July 26, 2011

Abstract—A new instrumental method was proposed for the rapid estimation of the water-retention capacity of soils and sediments. The method is based on the use of a centrifugal field to remove water from distributed soil columns. In distinction from the classical method of high columns, the use of a centrifugal force field stronger than the gravity field allowed reducing the height of the soil samples from several meters to 10–20 cm (the typical size of centrifuge bags). In distinction from equilibrium centrifugation, the proposed method obtained an almost continuous water retention curve during the rotation of the soil column only at one–two centrifuge speeds. The procedure was simple in use, had high accuracy, and obtained reliable relationships between the capillary-sorption water potential and the soil water content in a wide range from the total water capacity to the wilting point.

DOI: 10.1134/S1064229312040126

INTRODUCTION

The soil water retention curve (SWRC) is a universal basic state parameter of a physical soil system, which is widely used in the mathematical simulation of water, heat, and solute transfer in soils and landscapes [3, 4, 16]. Its experimental determination is performed under equilibrium thermodynamic conditions with the use of special equipment and methods for the separation of the soil water from the solid-phase matrix. The most common methods are based on the withdrawal of water under vacuum (tensiometry, capillarimetry, tensiostats) or its extrusion (with a membrane press) at increasing pressure on the other side of a porous membrane separating the water from the stationary solid phase of the soil sample [3, 4, 6, 12, 14, 16]. These methods are quite labor- and time-consuming if the conditions of the thermodynamic equilibrium are adequately maintained [13]. Moreover, each of them covers only a segment of the SWRC rather than the entire function of the matrix (capillary-sorption) potential in the natural variation range of the water content. The specific conditions (a highly decreased or increased pressure of the soil moisture) used in these methods rarely have analogues in natural environments (with a probable exception for the quasi-equilibrium root uptake of water or vacuum drainage).

Tensiometric methods act only in a relatively narrow range of absolute soil water pressure up to 0.7–0.8 atm, while its value can exceed 5–10 atm under actual conditions of soil drying; therefore, the simulation of the liquid-phase mass transfer in hydrophysics frequently faces the problem of insufficient data on the SWRC. The conventional solution of this problem by the combination of the tensiometric segment of the SWRC with its sorption segment, which is obtained by the hygroscopic method of liquid–vapor phase equilibrium, cannot be considered methodologically correct, because it joints the lines of the matrix and total potentials of the soil water. Even if it is granted that such joining is formally possible with consideration for the effect of the nondissolving volume (a decrease in the osmotic pressure with the drying of the soil), a significant part of the SWRC in the region of the absolute matrix potential from 1 to 20 atm remains undetermined.

The use of the thus obtained SWRCs in hydrophysical models most frequently cannot describe the actual moisture conditions without preliminary adjustment, which essentially involves the empirical fitting of the models. Some authors relate this to the large-scale (the variability of the hydrophysical properties) or kinetic (nonequilibrium) effects and attempt to unify the procedure of the data acquisition, e.g., using pedotransfer functions, and complicate the hydrophysical models themselves [2, 4, 12, 13, 16–19]. Along with these studies, the experimental methods of the SWRC's assessing can be improved, and the presented work deals with this field of study. It is based on the classical approaches to the assessment of the water-retention capacity of soils.

In the early 20th century, Lebedev, the founder of Russian hydrophysics, proposed two independent methods (the high-column method and centrifugation) for the determination of the so-called maximum molecular water capacity of soils [5]. Later on, Dolgov and Rode with researchers from the Dokuchaev Soil Science Institute used these methods to study the water-retention capacity and the rupture of the capillary bonds in soils [7]. In elaboration of Lebedev's ideas and some subsequent studies performed in Russia (Moscow State University and the Dokuchaev Soil Science Institute) and abroad (Rothamsted), we developed and introduced a version of the equilibrium centrifugation method for obtaining SWRCs [11, 14]. This work used the theoretical basis of the above study, as well as an original idea of combining the classical method of gravity columns with centrifugation to separate water from the solid-phase soil matrix. This allowed obtaining the first almost continuous SWRC in wide ranges of soil water contents and matrix pressures (potential) of the soil moisture during one–two centrifugation cycles with a constant rotation speed.

OBJECTS AND METHODS

In the centrifugation method, which was the prototype of this project, water was removed from the sample under the effect of centrifugal force. The developed pressure on the liquid phase (P) can be determined from the equation [11]

$$P = \frac{\rho_\ell \omega^2}{2}\left(R_2^2 - R_1^2\right), \quad (1)$$

where ω is the rotation speed, ρ_ℓ is the liquid's density, R_1 is the distance from the rotation axis to any point of the sample beginning from its upper boundary, and R_2 is the distance from the rotation axis to the free surface of the removed liquid or the lower point of the sample.

The construction of the centrifuge frequently involves an inclined position of the sample with respect to the rotation axis; therefore, a correction factor $\cos\alpha$, where α is the angle between the horizontal and the central symmetry axis of the sample, should be included in Eq. (1). For the horizontal rotor spider, $\alpha = 0°$ and $\cos\alpha = 1$. For low speeds of the centrifuge, the gravity component of the pressure ($P_g = -gh\sin\alpha$, where h is the sample's height, and g is the acceleration of gravity) should be taken into consideration. The rotation speed (ω) is calculated from the number of centrifuge revolutions per minute (N) using the equation $\omega = \pi N/30$, where $[\omega] = s^{-1}$. The final equation for determining the absolute values of the soil water pressure (the matrix potential ψ_m) depending on the centrifugation parameters for a distributed sample (soil column) has the following form [10, 11, 14]:

$$P[\text{kPa}] = \psi_m[\text{J/kg}] = 0.0055N^2(R_2^2 - R_1^2)\cos\alpha + gh\sin\alpha, \quad (2)$$

where $[N]$ = rpm, $[R]$ = m, and $[h]$ = m.

An analogue of the high-column method (according to Lebedev) implemented in the centrifugal field, which is stronger than the gravity field, can be the most promising procedure for the determination of the SWRC with a centrifuge. This can reduce the size of the columns proportionally to the separation factor $F = \omega^2R/g$), which shows by how many times the centrifugal field is stronger than the gravity field. For common laboratory centrifuges with a rotation radius of 0.1 m and a rotation speed of 500–5000 rpm, the field can be stronger by 30 to 3000 times. In this case, common centrifuge bags (about 0.1 m high) with soil moistened up to the total water capacity (TWC) can be used in place of long soil columns several meters in length [7]. Thus, an almost continuous SWRC can be obtained at a fixed rotation speed of the centrifuge by analyzing the water content of the soil in the centrifugal tube depending on the distance from its bottom during a single experiment (rather than a series of experiments). To maintain the zero capillary-sorption pressure at the lower boundary of the column, the perforated bottom of the tube should be installed on a water-soaked hygroscopic material (cotton or foam). After a characteristic time interval (2–4 h [11]), the plastic tube can be cut into fragments to determine the distribution of the water by weighing, or soil portions can be carefully removed from the top to bottom. In the longer term, sectional tubes of heat-resistant material should be used, which can be fragmented after the end of the experiment to weigh the fragments (sections) and determine the soil water content by drying.

The calculation of the soil water pressure in the distributed soil sample depending on the variable radius R_1 (which is apparently related to the sample's height h as $R_1 = R_2 - h\cos\alpha$) using Eq. (2) shows that a pressure of 1500 kPa will be created at the top of a 10-cm soil column at the maximum rotation speed of a TsLS-3 laboratory centrifuge (Russia) equal to 6000 rpm; i.e., a pressure corresponding to the wilting point can be reached (Fig. 1). At the same time, a pressure of 40–50 kPa will be observed at a distance (h) of only 2 mm from the free water surface (a 2-mm layer at the tube's bottom). A single experiment with this rotation speed can obviously be used only for studying the SWRCs of heavy soils and sediments with high water retention capacity. In sands, the distribution of water throughout the column will be almost homogeneous, because almost all the capillary water will be extruded already at 40 kPa. At the same time, a decrease in the rotation speed to 1500 rpm can create a gradient of the centrifugal field from ~3 kPa at the bottom to ~100 kPa at the top of a 10-cm soil column, which is suitable for sand SWRCs. Thus, a single centrifugal experiment with this rotation speed can determine an almost continuous SWRC in the range of 3–100 kPa, which significantly reduces the time and cost of the analysis. To obtain SWRCs in the range of higher absolute pressures between 200 and 1500 kPa, an additional experiment should obviously be performed with a sample of the same soil in a 10-cm column at the maximum rotation speed of the centrifuge (6000 rpm).

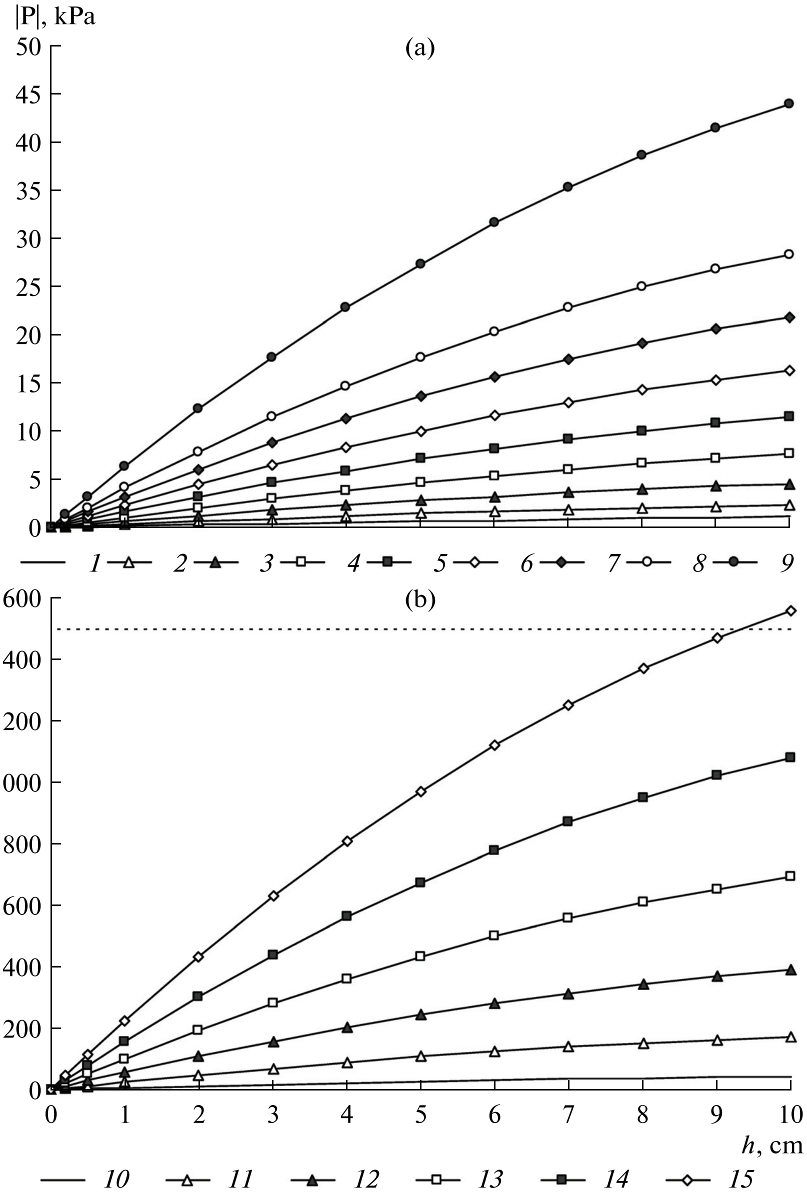

Fig. 1. Calculated curves of the soil water pressure as a function of the soil column height for a TsLS-3 laboratory centrifuge: (A) low centrifugation speeds (*1*)–(*9*): 100, 200, 300, 400, 500, 600, 700, 800, and 900 rpm, respectively; (B) medium and high centrifugation speeds (*10*)–(*16*): 1000, 2000, 3000, 4000, 5000, and 6000 rpm, respectively; (the dotted line) the pressure corresponding to the wilting point (1500 kPa).

Along with the drying SWRCs conventionally used in hydrophysics, this work theoretically allows obtaining the wetting branch of the SWRC (an analogue of the capillary fringe) if the soil is placed in centrifugal cells with water on the bottom and a hard spring ensuring the submersion of the lower surface of the sample into the free water at the given rotation speed [11]. The experimental study of the SWRC hysteresis thus becomes possible in a wide range of soil water pressures. In our opinion, the proposed version of the equilibrium centrifugation method will be especially promising for the information support and adjustment of water transfer models such as HYDRUS, where the SWRCs are preset in the form of the vertical water distribution in a homogeneous soil layer (column) of fixed thickness. These models describe such an equilibrium distribution identical to the SWRC's shape under the free outflow of gravity water and zero flow at the upper boundary of the profile [10, 18].

Experimental procedure. We describe the procedure for the simplest version of the method to obtain the drying SWRC with the use of a TsLS-3 centrifuge and an electronic analytical balance with an accuracy of 0.001 g. Filled or monolith soil samples were placed in preweighed centrifugal tubes 10 cm high and 1 cm in diameter with perforated bottoms. A sample was simultaneously taken to determine the water content in the initial (air-dry or natural) state. The weighed tubes with the soil samples were installed in laboratory bakers with water and capillary fed with water from the bottom; then, water was added to the bakers to the level of the sample's surface for the complete saturation of the soil with water. After weighing, the tubes with the soil samples were installed in the laboratory centrifuge holders (four tubes per holder) with a water-wetted cotton or foam piece on the bottom. Then, the centrifuge's rotation speed was selected from plot A (Fig. 1) to obtain the SWRC in the initial pressure range corresponding to the tensiometric region (no more than 50 kPa). Some samples were centrifuged at this speed for 2 (light soils) or 4 (heavy soils) hours; then, the tubes were cut (or unscrewed) into fragments of 3–10 mm, weighed, and dried to constant weight (m_s) at 105°C. Another procedure of soil extraction is also possible: the transparent plastic tube is marked out in segments, and soil portions are carefully extracted from each depth and transferred into glass cups. From the known equation $W = 100\ (m_{\text{в}} - m_s)/(m_s - m_f)$, where m_f is the weight of the empty tube fragment or the cup, the corresponding values of the water content in the profile of the soil column were found [1]. Another series of soil samples was treated analogously, where the SWRC should be determined in the higher range of absolute soil water pressure up to 1500 kPa with the corresponding rotation speed being selected from plot B (Fig. 1). The plots obviously served for the approximate selection of the experimental conditions, and the exact values of the soil water pressure in the studied layers of the distributed samples (columns) were found from Eq. (2).

From the weight of the empty tube (m_0), the initial weight of the soil in the tube (m_w) at the initial water content (W_0), and the weight of the water-saturated soil in the tube (m_s), the TWC value was estimated:

$$\text{TWS}, \% = 100\left(\frac{m_s - 100m_w/(100 + W_0)}{100m_w/(100 + W_0) - m_0}\right). \quad (3)$$

Taking into consideration that $R_1 = R_2 - h\cos\alpha$, the water pressure was calculated for the given centrifuge rotation speeds depending on the distance from the tube's bottom. These data formed an SWRC coordinate. The values on the other axis of the function were found as gravimetric the water contents (W) of the soil fragments corresponding to their height from the tube's bottom (h) (see above). Given the diameter and height of the tube's segment and the weight of the soil fragment after drying, the soil density can be easily determined, and the gravimetric water content can be converted to the volumetric water content if necessary.

The proposed procedure was tested using three samples with different degrees of dispersion and physical states: quartz glass sand, soil from the A (20- to 30-cm) horizon of a loamy ordinary chernozem (Krasnodar region), and soil from the T (10- to 20-cm) horizon of a low-moor peat soil (Moscow oblast). The selection of the samples was performed with consideration for the sufficiently wide distribution of the corresponding soils and sediments and the possibility of estimating the water retention capacity in a wide range of water content with their use. The chernozem was sampled as a monolith; bulk samples were used in the other cases. The air-dry peat material was preliminarily wetted with a hand sprayer to remove the effect of hydrophobicity. After saturation, 16 to 32 columns for each sample were centrifuged on a TsLS-3 laboratory centrifuge with the following stages of the rotation speeds: 200, 400, 600, 2000, 3000, 4000, and 6000 rpm. Such a wide and dense range of speeds was used for methodological purposes. At each stage, two to four columns of each sample were taken and analyzed for their water content, which provided two to four replicates, respectively. SWRCs obtained by equilibrium centrifugation were available for the samples used in this work [11], as well as an SWRC obtained by the high-column method for quartz sand [4, 7]; therefore, we managed to compare the new method with those used in soil hydrophysics.

The statistical processing of the results and the preparation of the supporting data were performed using MS Excel-2003. To approximate the experimental data of the SWRC by the nonlinear van Genuhten function, RETC software (available at http://www.hydrus2d.com/) was used in the HYDRUS-1D environment for the computer simulation of the energy and mass transfer in the soils.

RESULTS AND DISCUSSION

Selected experimental results sufficient to illustrate the potential of the new method are shown in Fig. 2. The data were grouped according to the following stages of the centrifuge rotation: 200–400, 1000–2000, and 6000 rpm. For the measurements performed in four replicates, the data were averaged by pairs. It can be seen (Fig. 2) that the method gave typical SWRCs as a continuous series of points in a wide range of absolute soil water pressures from 0 to 1500 kPa for all of the studied samples. Thus, for the first time in soil science, the theoretically and practically important problem of assessing the SWRC by a single rapid method within the natural variation of the water content from the TWC to the WP was solved.

The comparative analysis revealed specific features of the samples studied. The quartz sand expectedly had the lowest water-retention capacity (from the gravimetric water content), the peat soil had the highest water-retention capacity, and the chernozem occupied an intermediate position. The comparative estimation of the differential water capacities was performed by the Voronin secant method [2], although it 3
could be considered only as conventionally suitable for coarsely dispersed and organic substrates. Nonethe-

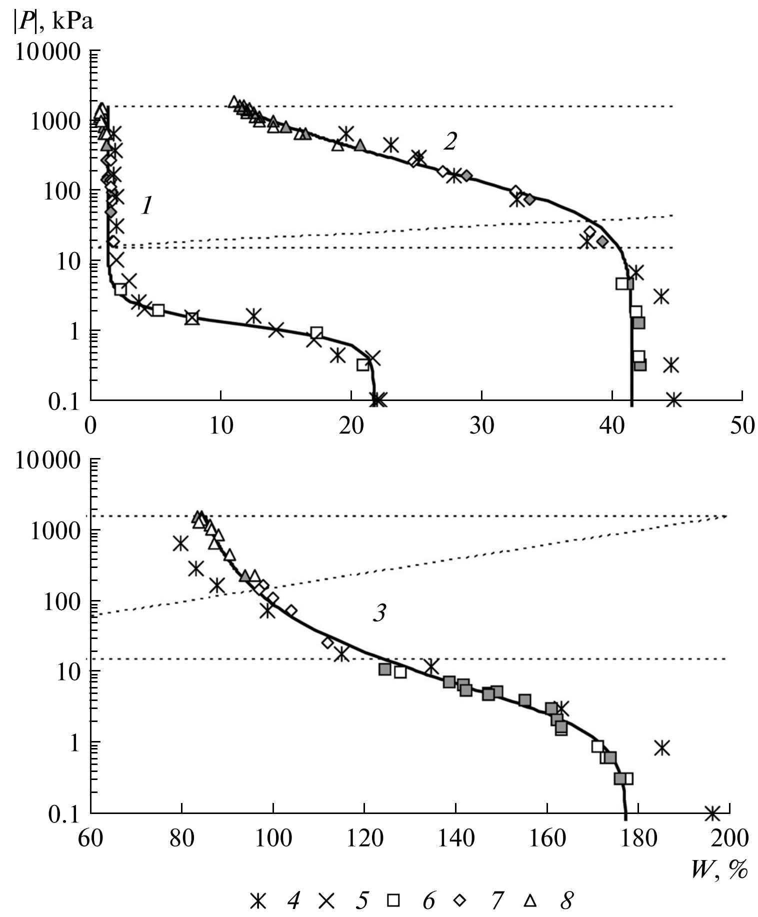

Fig. 2. SWRCs of some studied samples (the combined column–centrifugation method): (*1*) monomineral quartz sample; (*2*) loamy ordinary chernozem; (*3*) low-moor peat soil; (curves) data approximation by the van Genuhten model; (upper dotted
3 line) WP (1500 kPA); (lower and medium dotted lines) the CW and FC, respectively (Voronin's secant method [2]); (*4*) equilibrium centrifugation [11]; (*5*) high-column method [4, 7]; centrifuge rotation stages, rpm: (*6*) 200–400, (*7*) 1000–2000, (*8*) 6000; (closed symbols) first replicate; (open symbols) second replicate.

less, the method can compare the numerical values of the so-called soil-hydrological (energy) constants marking the changes of the water-retention mechanisms in soils as manifestations of some physical (gravity, capillary, or surface) forces [2, 16]. The TWC values were close to 180% for the peat, exceeded 40% for the chernozem, and exceeded 20% for the sand. The values of the capillary water (CW) and field capacity (FC) were almost equal in the mineral samples: 1.5–2% in the sand and 38–40% in the chernozem. For the peat, the FC (95%) was significantly lower than the CW (125%). The WP values regularly increased with the increasing degree of dispersion and the content of organic matter in the samples from ≤1% in the sand to 11–12% in the chernozem and 85% in the peat soil. The obtained range of the values well agreed with the available quantitative estimates of these parameters for the studied classes of physical soil objects [7, 15, 16].

After the recalculation of the gravimetric water content to the volumetric water content, the SWRC data were approximated by the van Genuhten function most frequently used in the current foreign models of energy and mass transfer [16]. The approximation revealed good agreement between the actual SWRC data obtained by the new method and the above model with the parameters θ_r (the residual water content), θ_s (the saturated water content), α (the reciprocal of the bubbling pressure), and n (the function exponent) (Table). It can be seen that the maximum possible estimates of the approximation significance $R^2 = 0.99$ were reached for all of the studied samples at low standard errors of the approximation $s = 0.008$–0.015 and statistically significant model parameters. The latter varied in the ranges permissible for each of the studied sample classes according to the known international geophysical databases HYPRES and UNSODA [14]. For the loamy chernozem, only the α value controlling the SWRC slope in the near-saturation region was untypically low. In our opinion, this could be related to the use of a monolith sample obtained from a dried ped after the abundant irrigation of the soil under the field conditions. As a result, the swelled sample lost its

Parameters and approximation statistics of the SWRC data by the van Genuhten function

Object	θ_r	θ_s	α	n	s	R^2
Quartz sand	0.018 ± 0.002	0.282 ± 0.006	0.093 ± 0.003	3.720 ± 0.200	0.008	0.99
Ordinary chernozem	–	0.456 ± 0.003	0.001 ± 0.000	1.441 ± 0.014	0.008	0.99
Low-moor peat soil	0.397 ± 0.012	0.887 ± 0.020	0.036 ± 0.008	1.449 ± 0.052	0.015	0.99

granular structure and loose consistence typical of rainfed ordinary chernozems. Therefore, the pressure of the air bubbling in the sample was untypically high, and its reciprocal α in the van Genuhten equation was low. This could be seen from the shape of the SWRC, which was almost parallel to the ordinate axis up to pressure values of 10–20 kPa.

After the reverse conversion of the volumetric water content into the gravimetric water content, the continuous SWRCs calculated from the van Genuhten function were plotted in Fig. 2 with the experimental data. The visual analysis confirmed the complete agreement of the calculated and experimental data, which indicated the adequacy of the van Genuhten approximation function, on the one hand, and the potential suitability of the new method for the informational and experimental support of the current models for the transport of water and solutes in soils and porous environments, on the other hand.

The comparison of the results obtained by the new method with the physically closest data from the equilibrium centrifugation [11] and high column [4, 7] methods (denoted with cross symbols in Fig. 2) also revealed satisfactory agreement. The best agreement of the data was noted for the coarsely dispersed sand sample, and the worst agreement was observed for the organic material of the peat-bog soil. By all appearances, this was related to the probable partial compaction (shrinkage) of the swelled finely dispersed samples in the centrifugal field. In equilibrium centrifugation, 9 or 10 points on the SWRC corresponded to the same number of sample exposure stages in the centrifugal field of successively increasing intensity; therefore, the probability of compaction in this case was higher than in the new combined method with the lower number of centrifugation stages. For an organic sample, the compaction in the equilibrium centrifugation method resulted in decreasing porosity and, hence, the equilibrium water content at the same pressure as in the new method. For mineral samples with a rigid structure (sand) or initially dense monolith packing of the particles (chernozem), the additional load had no strong effect on the sample's volume (water content), but it increased the absolute matrix potential of the soil water, probably because of its localization in microcapillaries and at the surface of soil particles. Therefore, the data of the equilibrium centrifugation method in the region of the capillary and sorption moisture were located above the SWRCs obtained in the new combined method. An analogous excess in the region of saturated moisture for swelling samples *2* and *3* was apparently related to the initially different water contents. A higher excess was found in the equilibrium centrifugation method, probably because of the smaller samples (4 cm in size) and their more complete free swelling compared to the samples in the new method (Fig. 2).

The following approach was used for the visual demonstration and statistical processing of the data compared. From the van Genuhten equation with the previously determined coefficients (table), the equilibrium water content (W_1) corresponding to the matrix pressure at which the water content (W_2) was experimentally found using the known methods of the SWRC's determination was calculated for each of the studied samples. This was necessary, because the pressure values obtained in the new method usually did not coincide with those in the equilibrium centrifugation and the water distribution in the high columns. As a result, two series of equilibrium water contents corresponding to the same values of the matrix pressure were obtained; one series characterized the new method, and the other series characterized the previously known methods of the SWRC determination. The mutual correlation of these values was then analyzed with the approximation of the data by a linear equation in the form $W_2 = \alpha W_1$ (Fig. 3). At the complete coincidence of the data from the different methods, the slope of this line should be equal to one ($\alpha = 1$), as well as the coefficient of the determination ($R^2 = 1$). In our case, $\alpha = 0.97$–0.99 and $R^2 = 0.93$–0.97, which indicated a close correlation between the data compared. The standard errors of the approximation varied from 1.6–2.3% in the mineral samples to 9.2% in the organic sample, which was lower than the errors and the confidence intervals that we found earlier for the equilibrium centrifugation method [9, 11]. Thus, it can be concluded in a first approximation that the results of the new method are quite comparable with those of the known hydrophysical methods within the limits corresponding to the natural variation of the

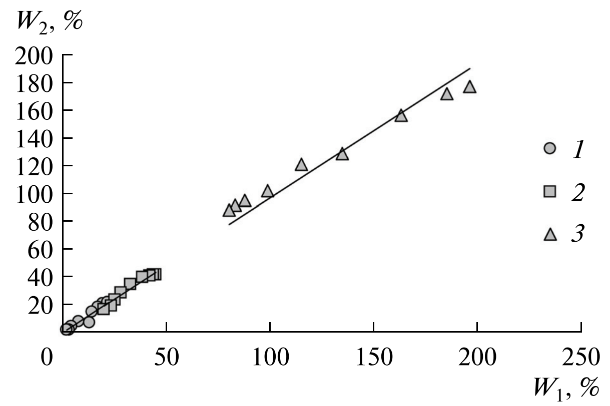

Fig. 3. Comparison of the data for the equilibrium water content from the column–centrifugation method and other methods of the SWRC's determination: (*1*) monomineral quartz sample, (*2*) loamy ordinary chernozem, (*3*) low-moor peat soil; (W_1) equilibrium water content determined by the known methods of centrifugation [11] and high columns [4, 7]; (W_2) the same for the new column–centrifugation method; the linear regression equation, coefficients of determination, and standard errors of the approximation: (*1*) $W_2 = 0.986W_1$, $R^2 = 0.97$, $s = 1.6$; (*2*) $W_2 = 0.965W_1$, $R^2 = 0.95$, $s = 2.3$; (*3*) $W_2 = 0.970W_1$, $R^2 = 0.93$, $s = 9.2$.

measured equilibrium water contents in soils and sediments.

CONCLUSIONS

(1) A fundamentally new method for the determination of SWRCs was developed that estimated the equilibrium profile distribution of the water in centrifuged soil columns of short length.

(2) The analysis is not laborious; it can be implemented on common laboratory centrifuges (TsLS-3) and allows obtaining SWRC data for 32 soil samples of no more than 10–15 g each within 4–8 h.

(3) The method can give an almost continuous SWRC in the range of the absolute capillary-sorption water content from 0 to 1500 kPa corresponding to the water content range from the FC to the WP.

ACKNOWLEDGMENTS

This work was supported by the program of basic research Biological Resources of Russia of the Division of Biological Sciences of the Russian Academy of Sciences, the program of the Presidium of the Russian Academy of Sciences Biological Diversity, the Russian Foundation for Basic Research (project no. 509-04-00929a), and the Moscow Government (state contract no. 8/3-66p-10/11).

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