Estudios en la Zona no Saturada del Suelo. Vol IX, O. Silva et al.
Barcelona, 18 a 20 de Noviembre, 2009

A NEW ANALYTICAL LABORATORY PROCEDURE FOR DETERMINING THE THERMAL PROPERTIES IN POROUS MEDIA, BASED ON THE AMERICAN STANDARD D 5334-05

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Palabras clave: thermal conductivity, thermal diffusivity, volumetric heat capacity, water content, volume of air

RESUMEN. *Actualmente, tres son las variables medidas para la caracterización de las propiedades térmicas en medios porosos y rocas blandas: la capacidad de calor específico, la conductividad térmica y/o su opuesto la resistividad, y la difusividad térmica. Recientemente, Decagon Devices Inc. ha desarrollado el lector KD2-Pro que permite registrar más de 4000 datos térmicos y, a su a vez, ha mejorado el diseño de sensores para este uso específico, como por ejemplo el sensor de aguja simple y/o el sensor de doble aguja, que utilizan el método del pulso térmico en las determinaciones de éstas propiedades. Sin embargo, para obtener la fiabilidad deseada en los datos medidos, el método debe ser normalizado según las indicaciones de los estándares actuales (ASTM D 5334-05). Este trabajo presenta el primer estadio hacia el desarrollo de una metodología de laboratorio y campo que permita obtener fiabilidad, precisión y rapidez en los datos analíticos de propiedades térmicas en diferentes medios porosos.*

ABSTRACT. *Nowadays, three are the variables measured for the characterization of the thermal properties in porous media and soft rocks: the volumetric specific heat, the thermal conductivity and the thermal diffusivity. Recently, Decagon Devices Inc. has developed the meter KD2-Pro. This instrument is a reader-logger which permits storing more than 4000 thermal data. Also, Decagon Devices has improved the design of sensors for this specific use, as is the sensor of simple needle, and the sensor of dual needle, which use the method of the thermal pulse for the determinations of these properties. Nevertheless, to obtain the required reliability in the measurements, this method must be normalized according to the indications of the current standards (ASTM D 5334-08). This work presents the first "stadium" towards the development of a laboratory and field methodology for obtaining reliability, accuracy and rapidity in the analytical data of thermal properties in different porous media.*

1. INTRODUCTION

Typically, three parameters are measured to characterize the thermal properties of any porous media: specific heat capacity, thermal conductivity and thermal diffusivity. Thermal properties are strongly influenced by physical properties such as bulk density, water content, particle-size distribution, and structural arrangement. Therefore, these factors have to be taken into account when performing measurements at laboratory and field scale. Recently, Decagon Devices Inc. has developed the KD2-Pro meter logger, and two specific sensors: the SH-1 thermal sensor, to measure the thermal properties employing the dual needle heat pulse method (DNHP), and KS-1 thermal sensor that is a single needle employing an infinite line heat source method (ILHS). In order to obtain reliable data, field and laboratory procedures to determine thermal properties with the KD2-Pro need to be normalized, according to existing standards and manufacturer's indications, since soil scientists, engineers and other users are demanding these kind of data for different applications. The present work describes the first step towards the development of a laboratory procedure to obtain reliable, accurate and rapid thermal properties dataset in soils, taking into account the current accepted standard (ASTM D-5334-08).

2. MATERIALS AND METHODS

Samples were obtained from the top soil horizon (0-30 cm) of a plot located at Can Solé Road, located in the Llobregat delta plain (Northeast of Spain).

2.1. Soil properties

To characterize the soil of Can Solé Road, the physical variables, particle size distribution, bulk density, total organic carbon content, calcium carbonate content were measured. In addition, the residual water content (hygroscopic water) was determined. Particle-size distribution was determined using the wetting sieve method for 2000 to 500 μm, and a device by dispersion laser beams (Malvern Mastersizer/E) for particles smaller than 500 μm. Bulk density and total porosity were determined from undisturbed sample volumes. Total carbon content was analyzed by loss on ignition at 900ºC, and inorganic carbon content by loss on ignition at 200ºC, both using a Shimadzu SSM-5000A and solid sample module. These results allowed to calculate both, total organic carbon content and calcium carbonate content. The residual water content was determined by loss in weight after drying the samples at 105ºC during 24h.

Measurements of thermal-hydrodynamic properties were made on soil columns, constructed specifically for this experiment. Several sensors were placed inside of the device, allowing control of moisture content and thermal properties.

To determine the thermal properties, two thermal sensors, one small dual-needle sensor (SH-1) and one single needle sensor (KS-1) (Decagon Devices Inc.) were employed. These kind of sensors use the heat pulse methodology and yield reliable soil thermal diffusivity ($\boldsymbol{\alpha}$) thermal resistivity (**R**) and the inverse thermal conductivity ($\boldsymbol{\lambda}$) and volumetric specific heat capacity ($\mathbf{C_v}$) estimations, obtained by a non-linear least squares procedure during both processes.

The thermal data were collected using a KD2-Pro reader-logger. To determine the volumetric water content ($\boldsymbol{\theta}$), the soil column was monitored with ECH$_2$O EC-5 frequency domain probe (Decagon Devices Inc.). A Decagon Devices Em-5b data-logger was required to collect the water content and temperature chamber data.

2.2. Field sampling design

The first step to develop a protocol to measure the thermal properties, begins with the field sampling design, i.e. to choose a representative unit for sampling. Field observations and preliminary prospection must be performed. In this work, disturbed samples from a silty loam soil were taken.

Some considerations must be taken during this stage:

- To verify and prepare the thermal sensor (calibration)
- Definition of the thermal sensor placement
- Position of the needle with respect to surface
- Extraction of the sample
- Determination of bulk density *in situ*

- Determination of water content and thermal properties *in situ*

2.3. Analytical laboratory procedure

This method is applicable for both, unaltered and repacked soil specimens, which are suitable only for isotropic materials. Heterometric materials must be taken into account on repacked soil samples.

After the sample is air dried, it is sieved to 2000 µm and repacked inside the column device to a target bulk density. In this case, the bulk density should be similar to the value measured in the field. If the sample presents large quantity of coarse elements, these must be taken into account as the sample is repacked.

Once the soil sample column is ready the next step will be to place the thermal sensors inside the device. Usually, we recommend inserting more than one KS-1 or SH-1 thermal sensor for each device. The experience indicated that few measurements are required to obtain reliable results, and to analyze and to evaluate the uncertainty of the measurements.

To wet the sample, we use two different techniques; (i) dynamic technique: Thermal properties and water content measurements are taken as water rises by capillarity from the bottom of the column; and (ii) static technique: measurements are taken after water has been added to the soil, mixed thoroughly and repacked.

3. RESULTS AND DISCUSSION

3.1. Soil properties

The studied soil from Can Solé Road was classified as silt loam textural class (USDA, 1975), with a particle size distribution for silt content, always, higher than 60%, mean sand content about 34%, and mean clay content about 4%. Mean bulk density is 1.47 g·cm^{-3} and total porosity 45%. Mean total organic carbon content was about 3.1%, mean calcium carbonate content was 40.3%.

3.2. Thermal properties and hydrodynamics of the soil

Fig. 1, shows the comparison for different positions of the thermal sensor and different wetting processes, as well, to determine the thermal resistivity. For this example, we have chosen the inverse thermal conductivity variable just that is a most frequently thermal property used in many experiences, as are civil engineering, basic thermal science, among other. In this experiment the thermal sensor was the KS-1 single needle.

The influence of water content in the thermal resistivity is observed when using either one of the two methods (static and dynamic) and two different sensor placements. The thermal resistivity obtained with the dynamic wetting technique always presented higher R values than obtained by static technique. The effect of the position of the sensor inside the soil sample (perpendicular or parallel to sample surface) did not present significant differences between both positions respect to the thermal resistivity values. In spite of this, minimal differences could be observed among the measurements. The data obtained with the perpendicular sensor showed large thermal resistivity values when the sample was air dried. However, the thermal resistivity values were lower than the data obtained with the parallel sensor when the water content was close to saturation. Therefore, for a silt loam soil, the thermal resistivity (*R*) showed a gradual decrease insofar water content increased (Singh and Devid, 2000), presenting a strong reaction when soil moisture was higher than 10% vol·vol^{-1} for the static technique, and close to 20% vol·vol^{-1} for the dynamic technique. Even thought, we suspect a discrete wetting front occurred with the dynamic method. Therefore, greatest decrease in *R* became during the wetting processes range, assuming a constant slope (Nakshabandi and Kohnke, 1964; Bristow, 1998). Similar results were showed by Al Nakshabandi and Kohnke (1964) with the same type of soil textural class.

Often, a common approach to present soil thermal properties has been to plot these properties as a function of water content. But less commonly, thermal properties have been plotted as a function of volume fraction of air (Φ, m^3·m^{-3}) (Ochsner et al., 2001). Fig. 2, shows the relationship between λ, α and *Cv vs* Φ. Volume fraction of air was calculated once water content and particle density was known, since the sum of the volume fraction is 1. The thermal conductivity data in Fig. 2 shows that the variation in λ can be explained by the variation in Φ

between the measurements. On the whole, the increase of Φ was related linearly to the decrease of the thermal properties, except for α values, which did not present a linear dependence with the volume fraction of air values. The relationship between λ, Cv and Φ was stronger (r = 0.98, Fig. 2) than the relationship between α and Φ (r= 0.95, Fig. 2). Therefore, volume fraction of air exerts a limiting effect on thermal conductivity (Ochsner et al., 2001) and volumetric heat capacity in these measure conditions for silt loam soil.

Also, the variations in the volume of the air fraction explained much of the variation in thermal diffusivity data rather than other variables, just that in driest measurements for this soil the relationship was not typically linear.

4. CONCLUSIONS

Sampling is a crucial stage in the evaluation of soil thermal properties, and a correct decision must be taken following a validated procedure. This fact is especially important to obtain reliable results and a lower uncertainty of the registered data.

Preliminary results showed that the new procedure was suitable for all cases, though the soil properties measured *in situ* were not relevant for the studied case. Only, the different characteristics of each porous media must be taken into account. The method also can be used in cases where the structure of the porous media is relevant to evaluate the hydrodynamic properties.

On the other hand, the special design of the column device was highly effective. The experiment showed several interesting features. Thermal properties showed an acceptable relationship with water content. Also, these measurements could be described as a decreasing linear function of the air-filled porosity.

The dynamic method is able to use whether the measuring interval time is shorter, which it is strongly not recommended, just that it is not possible to reach a system equilibrium.

In spite of this, it would be convenient to continue the investigations of the soil thermal behavior, studying more variables, which can be especially sensitive.

Acknowledgements. This work was funded by the project HYDRUS-LAB (PTQ05-02-03451) from Ministerio de Ciencia e Innovación while carrying out this research. This research was funded by the Lab-Ferrer Soils and Environmental Consulting, Department of Research and Development. We thank the Ministerio de Ciencia e Innovación for the Torres Quevedo award to Dr. C. M. Rubio. Also we thank to Dr. G. S. Campbell and Dr. C. S. Campbell, both from Decagon Devices, Inc. for their help and for fruitful discussions.

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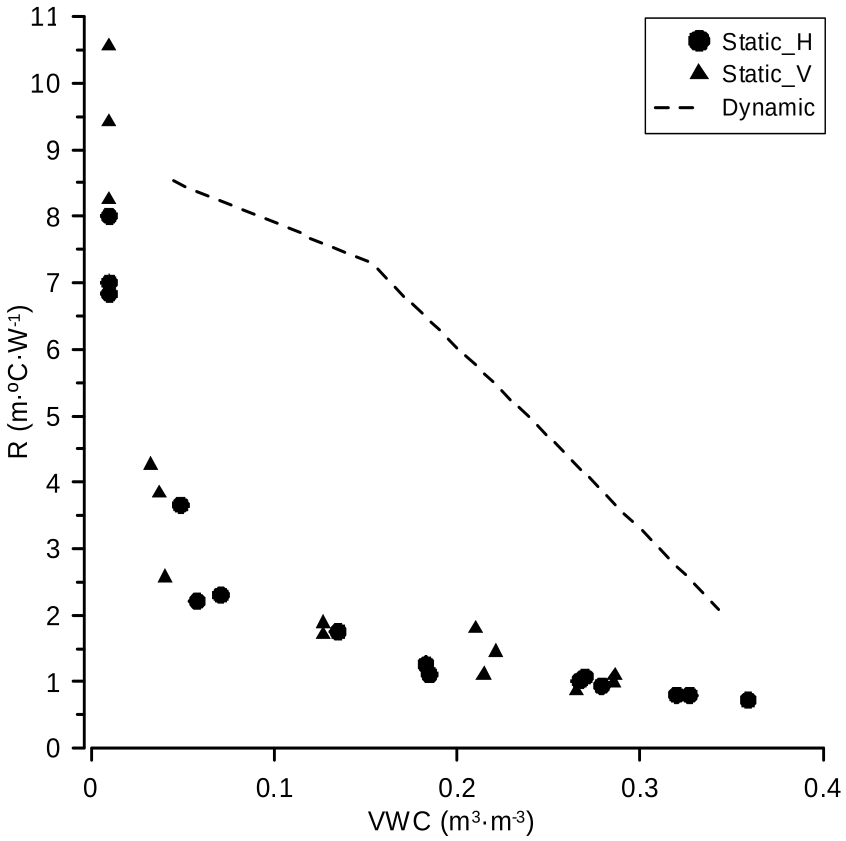

Figure 1. Comparing different wetting processes to determine the thermal resisitivity (R) as a function of volumetric water content (VWC) for a silt loam soil. H: parallel to surface; V: perpendicular to surface

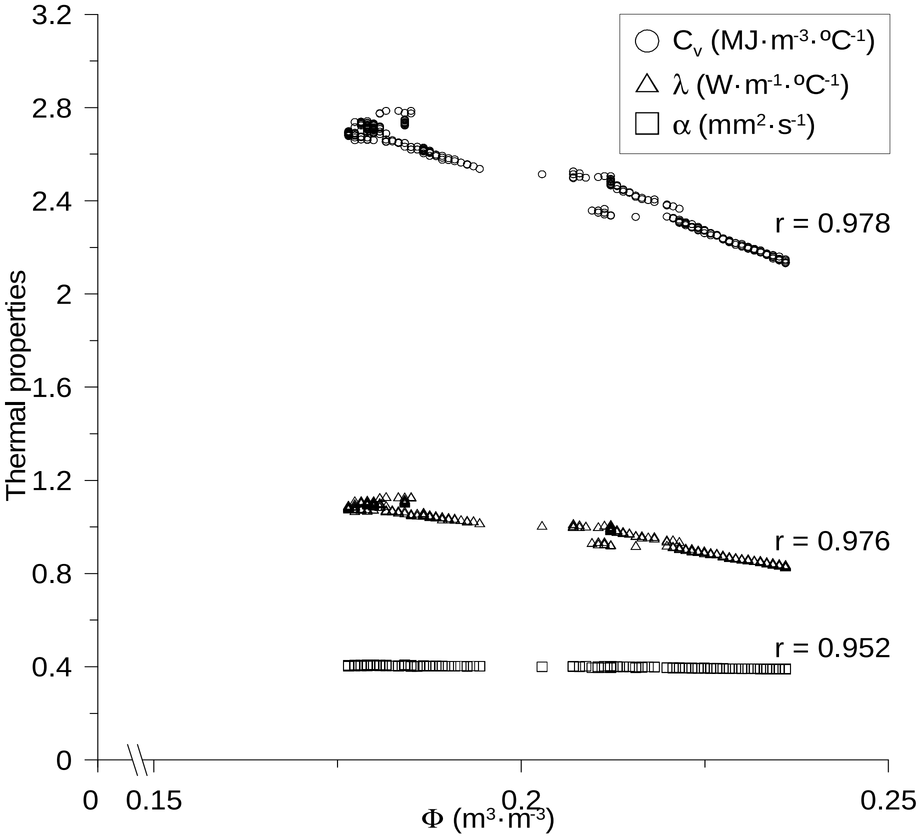

Figure 2. Thermal conductivity (λ), volumetric heat capacity (C_v), and thermal diffusivity (α) versus volume fraction of air (Φ) for the studied soil. R indicates the coefficient of correlation between Φ and thermal properties data-set.