An Approach to Make More Technical Thermal Properties Measure in Porous Media

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Abstract
There are three variables measured for the characterization of the thermal properties in porous media and soft rocks. These are the specific variables: heat capacity, thermal conductivity and their relationship with the thermal diffusivity. Recently, Decagon Devices has developed the KD2-Pro meter. This instrument is a useful device, which permits storing more than 4000 thermal data. This company has also improved the design of sensors for their specific use. KD2-Pro uses the infinite line heat pulse technique based on current standard ASTM D 5334. Nevertheless, it is not clear how to obtain the required reliability and accuracy in the measurements since neither the standard nor manufacturer’s user manual include any method or recommendation to develop a measuring set up. Therefore, a strong methodology is required to achieve the maximum efficiency when KD2-Pro is used. This work presents ThermalHyd as the first “stadium” towards the development of a laboratory and field methodology to obtain reliability, accuracy and rapidity in the analytical dataset of thermal properties in porous media and soft rocks. To carry out the objective several soils from Llobregat river delta plain were used to obtain very acceptable results such as the wetting up samples, being the static method more accurate than dynamic one. Other results were based on the mineralogy of the samples in terms to transferring the heat pulse. Eventually, the microwave oven as a technique to obtain reliable water content dataset was a robust method, especially for its accuracy and faster performance.

Keywords: soil, rock, thermal conductivity, thermal diffusivity, volumetric heat capacity, water content, microwave

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1. Introduction
In 1822 Fourier published the Théorie Analytique de la Chaleur. Many authors have presented several scientific works related to the thermal properties of soil. Carslaw and Jaeger [1] and DeVries [2] for instance.

Fourier’s law presents the relationship between heat flux and temperature gradient. This sort of dependence between these variables can only be understood as a heat transfer by conduction mechanism. Typically, three parameters are measured to characterize the thermal properties of any porous media: volumetric 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 scales.

Recently, Decagon Devices has developed the KD2-Pro meter logger, and two specific sensors; the SH-1 and TR-1. The SH-1 thermal sensor (1.3 mm diameter, 30 mm long and 6 mm spacing) measures the three thermal properties by employing the dual needle heat pulse method (DNHP), whereas the TR-1 thermal sensor (2.4 mm diameter and 100 mm long.) is a single needle employing an infinite line heat pulse method (ILHP). The methodology of these devices is based on ASTM D5334. American’s standard D5334 is applicable for both undisturbed and remoulded soil specimens as well as soft rocks specimens, yet this test method is suitable only for isotropic materials [3]. In order to obtain a reliable thermal dataset, a simple field and laboratory methodology needs to be adapted and depicted according to existing standards. The fact is because of neither the manufacturer nor the standard gives us a clear methodology to be used. There are also soil scientists, engineers and other current users that are demanding these kinds of data for their 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 while taking into account the current accepted standard [3] as a work method that KD2-Pro is based on. The name ThermalHyd corresponds to the relationship between thermal properties and moisture content as one of the most important variables to concern the heat pulse transfers in porous media. It is a friendly laboratory method nick-name.

2. Methodology
The first group of soil samples were obtained from the top soil horizon (0-30 cm depth) of a plot placed at Can
Solé Road (Figure 1), located in the Llobregat delta plain (Northeast of Spain). A second group of soil samples were collected 250 m north from the Can Solé Road plot. This sampling point was in the alluvial floodplain of the Llobregat river (Figure 1).

![Map of the Can Solé Road and alluvial floodplain at Llobregat delta plain, showing location of the two sampling plots](image)

**Figure 1.** Map of the Can Solé Road and alluvial floodplain at Llobregat delta plain, showing location of the two sampling plots

### 2.1. Characterization of the Soil Samples

To characterize the soil of Can Solé Road, the physical variables, particle size distribution, bulk density, total organic carbon content, and 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 [4], and a device by dispersion laser beams (Malvern Mastersizer/E) for particles smaller than 500 µm [5,6]. Bulk density and total porosity were determined from undisturbed sample volumes [7]. Total carbon content was analyzed by loss on ignition at 900°C, and inorganic carbon content by loss on ignition at 200°C [8], both using a Shimadzu SSM-5000A and solid sample module. These results allowed calculations of both total organic carbon content and calcium carbonate content. Also, a second group of soil samples was collected 250 m north from Can Solé Road in the alluvial Llobregat river floodplain. To characterize the alluvial soil samples group, the same variables and analytical treatment was performed as the Can Solé Road soil samples. An exception was for the variable calcium carbonate content, which determinations were carried out according to Skinner et al. [9]. Residual water content was determined by loss in weight after drying the samples at 105°C for 24h [1,10,11,12].

### 2.2. Measuring Soil Thermal Properties and Water Content

De Vries [13] showed that temperature as a function of time at a radial distance from the heat pulse source is given by the following equation:

\[
T(r,t) = \left[ \frac{q}{4\pi \alpha C_v} \left( E_i \left( \frac{-r^2}{4\alpha (t-t_0)} \right) - E_i \left( \frac{-r^2}{4\alpha t} \right) \right) \right] t > t_0 \tag{1}
\]

Where \( t \) is time (s), \( t_0 \) is the duration of the heat pulse (s), \( r \) is the radial distance (m), \( \alpha \) is the soil thermal diffusivity \((m^2 \cdot s^{-1})\), \( q \) is the amount of heat applied \((W \cdot m^{-1})\), \( C_v \) is the volumetric specific heat capacity \((J \cdot m^{-3} \cdot C^{-1})\) and \(-E_i(-x)\) is the exponential integral. The exponential integral can be evaluated using the formula 5.1.53 of Abramowitz and Stegun [14] for \( 0 \leq x \leq 1 \) and formula 5.1.56 for \( 1 \leq x \leq \infty \).

Measurements of thermal-hydrodynamic properties were made on soil columns (10 cm height and 13 cm diameter) constructed specifically for this experiment. Several sensors were placed inside of the device allowing control of moisture content and thermal properties, as well as temperature of the sample during the experiment. Finally, to minimize the effects of the temperature drift, the samples were placed into an isothermal chamber.

To determine the thermal properties, two thermal sensors, one small dual-needle sensor (SH-1) and one single needle sensor (TR-1) were employed. These sensors use the heat pulse methodology to yield reliable soil thermal data. Using a SH-1 sensor, a data set with thermal diffusivity (\( \alpha \)), thermal conductivity (\( \lambda \)) and volumetric specific heat capacity (\( C_v \)) estimations were obtained. On the other hand, the TR-1 sensor only measured the thermal resistivity and its inverse thermal conductivity.

The thermal data were collected using a KD2-Pro reader-logger. The KD2-Pro resolves 0.001 °C in temperature. It uses special algorithms to analyze measurements made during a heating and a cooling interval. It also uses special algorithms to separate the effects of the heat pulse from ambient temperature changes. The algorithms are based on the transient line heat source analysis given in Carslaw and Jaeger [1] and Kluitenberg et al. [15]. Thus, from both thermal properties the volumetric specific heat capacity was calculated. To determine the volumetric water content (\( \theta \)), the soil column was monitored with ECH2O EC-5 frequency domain probe. A Decagon Device, Em-5b data-logger, was required to collect the water content data and the temperature inside of an isothermal chamber.

### 2.3. Field Sampling Design

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

Some considerations must be taken during this stage:
- Calibration of the thermal sensor
- 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

### 2.4. Analytical Laboratory Procedure

This method is applicable for both unaltered and repacked soil specimens, which are suitable only for isotropic materials. Hetero-metric materials must be taken into account for repacking 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 a large quantity of coarse elements, these must be taken into account when the sample is repacked. In this case, an important decision should be taken in order to decide what the most significant diameter of the coarse elements is. Once the soil sample was examined, with respect to the stony particles, 12 mm sieve seems to be a significant value for these coarse particles. The diameter value achieves a good contact between soil matrix and stony elements. It also minimizes the contact resistance between sensor and sample when the material was repacked.

Once the soil sample column was ready the next step was to place the thermal sensors inside the device. Usually, we recommend inserting more than one TR-1 or SH-1 thermal sensor for each column device. The experience indicated that few measurements were required to obtain reliable results and assessing the uncertainty of the measurements. A comparison between both sensors was not capable owing to the different volume fraction measured for each type of sensor.

On the other hand, KS-1 thermal sensor is strongly not recommended for these types of samples, since KS-1 is only useful and recommended for measuring thermal conductivity in liquids.

To wet the sample, we used two different techniques; (i) dynamic technique, where thermal properties and water content measurements were taken as water rises by capillarity from the bottom of the column; and (ii) static technique, where measurements were taken after water was added to the soil, mixed thoroughly and repacked. Afterwards, thermal sensors were placed in two different positions, vertically and horizontally, and left up to reach the steady state conditions for each scenario or water content. One of the most important steps during the preparation of the set up was to avoid the evaporation fluxes between the surface of column device and the environment. The time required to reach equilibrium inside the column device depends on the temperature of both the sample and the water (Rubio et al., 2011). To avoid convective fluxes the device was placed inside an isothermal chamber which maintained the experiment temperature around 17ºC.

Other aspects taken into account were the quantity of observations, and the number of measurements for each observation. An observation was defined as one thermal sensor placed in the sample where several measurements were collected per sensor. At this point, it ensured sufficient data to determine the significance of the values (Table 1). Thermal sensor TR-1 measured every 15 minutes, and it used 2 minutes to perform every determination. Thermal sensor SH-1 measured every 30 minutes, and used 2 minutes to estimate the measure as well. Each TR-1 thermal sensor collected 7 measures per water content, i.e. a total of 21 measures per scenario.

3. Results and Discussion

3.1. Soil Properties

The studied soil from Can Solé Road was classified as silt loam textural class [16], 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%. The group of soil samples collected from alluvial Llobregat river floodplain was split up into three different textural classes according to Soil Survey Staff [16], they are the following; sandy, sandy loam, and silt loam as well. The average values of the variables characterized for alluvial soil samples are shown in Table 1.

| Textural Class | Sand (%) | Silt (%) | Clay (%) | O.M. (%) | BD (g·cm⁻³) | CaCO₃ (%) |
|----------------|----------|----------|----------|----------|-------------|----------|
| Sandy          | 88       | 8        | 3        | .25      | 1.76        | 20       |
| Sandy loam     | 65       | 29       | 7        | .95      | 1.71        | 29       |
| Silt loam      | 17       | 62       | 21       | .95      | 1.66        | 33       |

On the other hand, the SH-1 thermal sensor recorded 630 data using a KD2-Pro continuous mode. As far as the water content is concerned, several scenarios were determined using a FDR probe. The measures were collected at the same instant as that of the thermal data. Thus, thermal data and water content could be related. The average temperature during the experiment was 17.3 °C.

3.2 Thermal Properties and Hydrodynamics of the Soil

![Figure 2](https://example.com/figure2.png)

**Figure 2.** Comparing different wetting processes to determine the thermal resistivity (R) as a function of volumetric water content (VWC) for a silt loam soil. H: parallel to surface; V: perpendicular to surface.
Figure 2 shows the comparison for different positions of the thermal sensor and different wetting processes, to determine the thermal resistivity. In this case, we chose the inverse thermal conductivity variable as it is the most frequently thermal property used in many applications such as civil engineering and basic thermal science, among others. The thermal sensor used in the experimental design was the TR-1 single needle.

The influence of water content in the thermal resistivity 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 thermal resistivity on the whole of the dry out curve 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.

Table 2. Paired samples T-test calculated for each moisture content scenario. To compare the thermal resistivity values between vertical and horizontal sensor placement. Each scenario means one group class of soil moisture defined by FDR probe

| Moisture | Correlation | Std. Desv. |
|----------|-------------|------------|
| Scenario 1 | .845 | 1.362 |
| Scenario 2 | .959 | 1.531 |
| Scenario 3 | .826 | 0.996 |
| Scenario 4 | .689 | 0.288 |
| Scenario 5 | .170 | 0.158 |

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 as water content increased [17]. The soil presented 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. This fact did suggest a discrete wetting front occurred when the dynamic method was used. Therefore, higher decreasing in thermal resistivity measurements occurred during the wetting process range, assuming a constant slope [18,19]. Similar results were showed by Al Nakshabandi and Kohnke [18] 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, m3·m-3) [20,21]. An experiment using the SH-1 thermal sensor was carried out. This type of thermal sensor estimates the three thermal properties; i.e. thermal conductivity, thermal diffusivity and volumetric specific heat capacity [19] as a function of λ, α and Φ. SH-1 uses a dual needle heat pulse. Dual needle allows estimate thermal conductivity and thermal diffusivity to calculate the volumetric specific heat capacity.

Figure 3 shows the relationship between thermal conductivity (λ), thermal diffusivity (α) and volumetric specific heat capacity (Cv) versus volume fraction of air (Φ). Volume fraction of air was calculated once water content and particle density were known, since the sum of the volume fraction is 1. The thermal conductivity data in

Figure 3 shows the variation in λ can be explained by the variation in Φ values. On the whole, the increase of fraction of air in the soil sample showed an inverse relationship with the thermal properties, except for α values. The thermal diffusivity values did not present a linear dependence on the volume fraction of air. The relationship between λ, Cv and Φ was stronger (r = 0.98, Figure 3) than the relationship between α and Φ (r= 0.95, Figure 3). Therefore, volume fraction of air exerts a limiting effect on thermal conductivity [20] and volumetric heat capacity in these measured conditions for silt loam soil. However, in the case of specific heat capacity the relationship with fraction of air is approximately the mirror image of the relationship with water content [22,23].

On the other hand, the variations in the volume of the air fraction explained much of the variation in the thermal diffusivity data compared to other variables. The α versus Φ relationship is similar to, but weaker than, the other thermal properties versus fraction of air. Also, of note, is the fairly strong relationship between α versus Φ [20]. The data shows that thermal diffusivity increases steadily except in the driest samples of the silt loam soil. At this point, the relationship between α versus Φ could be much weaker than the more commonly studied relationship between thermal diffusivity and water content.

Soil thermal conductivities (λ) for sandy, sandy loam and silt loam soil textural class measured using the single probe are shown in Figure 4 as a function of soil gravimetric water content (ω). Here, we also see the typically low thermal conductivity for dry soil, a rapid increase in thermal conductivity as the soil water increases on the whole of the textural classes [26]. There is better connectivity between soil particles and then a further slow increase in thermal conductivity as water content continues to increase towards saturation, especially for sandy loam and silt loam texture class. Similar results were found by Bristow [19] analyzing the same sort of soil. Regarding the sandy soil textural class, we assumed
that the soil mineralogy would be dominated by quartz. This fact resulted in a fast reaction of the heat pulse transfer among the soil particles for this textural class more than others, which increased gradually [27].

**Figure 4.** Thermal conductivity ($\lambda$) as a function of water content (\(w\)) for alluvial floodplain soil, from Llobregat river delta plain.

**Figure 5.** Comparing soil moisture content determined according to Reynolds (1970) with ones using a microwave oven.

The possible use of microwave oven for soil moisture determination seemed ideal to eliminate the 24 hours drying period required with air forced or non-forced drying ovens. The soil samples were wetted up following the procedure mentioned above. Miller et al. [24], and Routledge and Sabey [25] showed that the drying time in a microwave oven increased when increasing water content and increasing sample size. Sixty two subsamples containing around 70g of soil of the different textural classes studied were dried in the microwave oven. Also, to evaluate and validate the effectiveness of the method, it was made determining the soil moisture of the same subsamples in a non-forced air oven at 105ºC during 24 hours, such as it is recommended by Reynolds [10,11,12]. The results in Figure 5, show a very acceptable adjustment between two variables. A paired samples T-test indicated a standard deviation around 0.006gH2O·gsoil-1, and a correlation coefficient about 0.998 for a confidence interval 99%.

4. Conclusion

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

Indeed, one of the most relevant contributions in this work was about the dynamic method. It is not useful especially when measuring interval time is shorter, thus it is not recommended. On the other hand, mineralogy of the sample plays a crucial role in terms of transferring the heat pulse, as well as the particle size distribution especially when thermal properties are related with water content. Also, the use of the microwave oven to calculate the gravimetric moisture content was found entirely satisfactory as much as its accuracy as its reliability.

In spite of these facts it would be beneficial to continue the investigations of the soil thermal behavior and its variables, such as bulk density, mineralogy and stony porous medium. Eventually, a new approach in thermal properties for anisotropic and hetero-metric media should be performed.

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**Competing Interests**

Authors have declared that no competing interests exist.

**Authors’ Contributions**

The Author has designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript.

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