DETERMINATION OF THE HUMIDITY AND DIELECTRIC CONSTANT OF BENTONITE USING AN FDR BASED PROBE

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Abstract: This work presents the determination of the humidity and the dielectric constant of bentonite using a probe based on Frequency Domain Reflectometry (FDR). The basic principle of the probe used in this study is the measurement of the refractive index of the soil $\sqrt{\varepsilon}$ at 100 MHz to allow the determination of the humidity through a known relation between these quantities. In this work, a Profile Probe type PR2 (Delta-T Devices, Cambridge, England) and a Speedy Moisture Tester (Thomas Ashworth & Co. Ltd., England) were used. The value of 0.99 for the correlation coefficient obtained in this study demonstrated an accurate relationship between the humidity and the dielectric constant of bentonite. The results of the dielectric constant of bentonite are compared with the values encountered in the literature. Also in this study, the humidity of the bentonite using the FDR method was compared to the values obtained using the calcium carbide method.

Keywords: Bentonite; humidity; permittivity; refractive index; Profile Probe

1. INTRODUCTION

Bentonite is defined as any material composed primarily of the montmorillonite group of minerals and whose physical properties are dictated by the montmorillonite minerals [1]. The characteristics of montmorillonite minerals include large cation exchange capacity, large specific surface area, high swelling potential, and low hydraulic conductivity to water. The properties of bentonite are greatly affected by the interactions between its particles and the surrounding pore fluid. The net electrical charge on bentonite particles is negative [1], which causes dissolved cations in the pore water to be attracted to the surfaces of the bentonite. The layer of water and absorbed ions that surrounds a bentonite particle is referred to as the diffuse double layer (DDL). The size of the DDL is dominated by ionic strength, dielectric constant, and temperature. The overlapping of DDLs of bentonite makes it difficult for water and electrolytes to flow through [1].The determination of water content of soil is important for water management, hydrological studies, and for the development of soil-water balance models. The methods generally used in the laboratory for the determination of soil-water content are based on gravimetric methods and the calcium carbide method [2].

The main disadvantages of the gravimetric method include:

The destructive nature of the test.
The inability of replication of the test on a single sample.
The requirement of a 24-hour period to conclude the test.
The necessity of removing the soil samples from the site.
The existence of potential inaccuracies in data acquisition.

Another method widely used in soil analyses to determine water content is the calcium carbide method, commonly known as Speedy Moisture Test. This test is based on the chemical reaction that occurs between the water contained in the soil and calcium carbide. This procedure involves a potentially dangerous chemical reaction. Basically, when calcium carbide reacts with water, acetylene gas is produced, causing a gas pressure inside a chamber containing the soil sample. Breathing acetylene gas and running the measurements in environments where sparks and fire can occur must be avoided.

The main disadvantages of the calcium carbide method are:

The usage of chemical compounds.
The exposure to high dangerous gases.
The destructive nature of the test.
The inability of replication of the test on a singlesample.
The existence of potential inaccuracies during the data acquisition.

Despite some problems of inaccuracy and risks related to these methods, they are commonly used worldwide to determine water-soil content.

2. PURPOSE

The frequency-domain reflectometry (FDR) method proposed in this work employs a radio frequency signal at 100 MHz for the determination of the electrical capacitance

of the rod of a probe having a bentonite sample as dielectric. Thus, the soil (bentonite) water content can be easily determined and therefore, its dielectric constant obtained.
The main advantages of using the FDR method to determine the humidity and dielectric constant of the bentonite in comparison to the previously reported methods are:

The non-usage of chemical compounds.
The non-destructive nature of the test.
The possibility of replication of the test on a single sample.
The existence of reliable equipment.
The possibility of real-time measurements.
Good accuracy.

The results obtained with the FDR method presented in this work exhibited high accuracy and can be useful in studies of soils and clay minerals.

3. MATERIAL

3.1. Sample
The samples of bentonite used in the experiments were provided by Bentonisa Co., Soledade-PB, Brazil. A quantity of approximately 6.5kg of bentonite was used in the measurements.

3.2. Bentonite
Bentonite is a clay mineral derived from in-situ chemical alteration of volcanic ash. It is composed mainly of montmorillonite and is widely used in a variety of industrial products such as paints, ceramics, pesticides, pharmaceuticals, and cosmetics. Due to its high swelling and adsorption capability, bentonite is also widely used as a soil admixture for the construction of seepage barriers in waste containment facilities [3].

4. EQUIPMENT

A Profile Probe type PR2 from Delta-T Devices in Cambridge, England, an HH2 moisture meter, and a Speedy Moisture Tester from Thomas Ashworth & Co. in England were used in this work.

4.1. The Profile Probe type PR2

The Profile Probe PR2 is essentially a FDR sensor that detects the ratio between the voltage of an oscillator operating at 100 MHz and the voltage of the reflected signal by a sealed polycarbonate rod (25 mm diameter) installed in the soil [2]. The ratio of the two voltages is dependent on the apparent dielectric constant of the soil (ε), which depends on the soil-water content. A sixth-order polynomial for the sensor analogue output voltage V (in volts) is used to estimate the square root of the dielectric constant (refractive index) of the vermiculite samples ($\sqrt{\varepsilon}$) [5].

$$\sqrt{\varepsilon} = -0.057 - 0.66V + 8.00V^2 - 27.91V^3 + 49.23V^4 42.46V^5 + 14.47V^6 \quad (1)$$

The volumetric soil-water content θ_v is calculated from the dielectric constant by using the soil calibration constants a_0 and a_1 in the following equation:

$$\theta_v = (\sqrt{\varepsilon} - a_0)/a_1 \quad (2)$$

where,
a_0 is the square root of the dielectric constant of the dry soil ($\sqrt{\varepsilon_0}$) calculated using the corresponding measured voltage of the probe in equation (2). a_1 is the difference between the square root of the dielectric constant of saturated soil ($\sqrt{\varepsilon_w}$) and that for dry soil divided by soil-water content at saturation (θ_{vs}), given by

$$a = \left(\sqrt{\varepsilon_w} - \sqrt{\varepsilon_0}\right)/\theta_v \quad (3)$$

P.A Farré and G.C. Topp [4] have shown that there is a simple linear relationship between the complex refractive index and the volumetric water content given by the following equation:

$$\sqrt{\varepsilon} = a_0 + a_1\theta_v \quad (4)$$

These coefficients determined by Topp have taken into account the output voltage of a Time Domain Reflectometry (TDR) equipment and a curve relating the output voltage and the volumetric water content. In this work, these coefficients were not determined because the manufacturer of the Profile Probe PR2 provides the relationship between the refractive index $\sqrt{\varepsilon}$ and the probe output voltage. The provided values for a_0 and a_1 are 1.6 and 8.4 for mineral soil and 1.3 and 7.3 for organic soils, respectively [5]. In this work, a generic calibration was used for mineral soils.
The use of the generic calibration for mineral soils presented good accuracy, as seen in the experimental results. The relationship between the refractive index and the water content using the generic calibration is given by the following equation:

$$\sqrt{\varepsilon} = 1.6 + 8.4\theta_v \quad (5)$$

where the values 1.6 and 8.4 correspond to a_0 and a_1 of equation (4) for mineral soils, respectively, and θ_v is the volumetric water content read by the HH2 meter.
Although a generic calibration curve for mineral soil was used in this work, it is possible to make a specific calibration curve procedure for the soil studied. To do this, it is necessary to use another probe ML2x (Delta-T Devices, Cambridge, England) type. With this procedure, it is possible to determine the a_o and a_1 coefficients that are adequate for the soil being studied. In our work, we did not make a specific calibration curve procedure because an M2Lx probe was not available,, and because the general calibration curve gives good accuracy, this did not interfere with the results of the study. When using the general calibration curve for mineral soil or organic soil, variations

in the soil will not cause variations in the coefficients a_o and a_1 because their values are pre-defined in the equipment. The particle size of the soil also has no influence in the coefficients, but it can cause errors in the readings due to the presence of air between the particles. This can cause the equipment to give a value of the permittivity of a soil that was affected by the presence of air. For coarser particles there is more air between the particles, which, can increase the error. To avoid this, it is necessary to use a specific calibration and to carefully prepare the samples when doing tests in a laboratory.

4.2. The HH2 moisture meter

The HH2 moisture meter is a compact hand-held unit designed specifically for in-situ measurements. The readings are displayed on an LCD and can be stored for later download. The device can store up to 1,500 readings. Data can be transferred to a PC via an RS232 cable. Up to five user-defined soil calibration tables can also be adjusted [5].

4.3. The Speedy Moisture Test

The Speedy Moisture Test is widely used worldwide to determine the humidity of soils, fine aggregates, sand, and clay. It uses a tester whose operation is based on the principle of a calcium carbide reagent that is introduced in the free moisture of the soil sample. The resulting chemical reaction creates a gas that is contained inside a sealed chamber. The pressure of the gas produced during the chemical reaction can be measured with the built-in gas pressure gauge. The speedy moisture set includes the speedy moisture tester, a weigh, half-weight reagent, measure scoop, brushes, cleaning clothes, and three 12.5 mm steel balls.

5. EXPERIMENTAL PROCEDURE

A bentonite sample from Bentonisa Co. in Soledade, PB Brazil, was selected, and then the sample was put in a plastic bag to avoid exposure to the room humidity. The Profile Probe PR2 gives volumetric water content readings at six levels of depth: 100 mm, 200 mm, 300 mm, 400 mm, 600 mm, and 1,000 mm. In field tests, it is possible to vary the depth of the measurement by simply putting the probe or group of probes placed in the different depths, using holes distributed at different depths in a way to cover the variations of the humidity that can occur in the soil due to the variation in its composition and by water presence that can be influenced by the depth. A sample of bentonite was placed inside the PVC tube, and then the probe was inserted into the access tube; during this procedure, it is important to be certain that the access tube is exactly perpendicular to avoid a bad propagation of a signal through the soil. After this procedure, the HH2 meter was connected to it. The HH2 meter readings were transferred to a notebook for data analysis. Five sets of measurements were performed, resulting in 30 readings. Then another sample was selected and applied to the measurements made using the gravimetric and the calcium carbide method. The gravimetric method is

based on the weighting of the sample of bentonite in a wet condition and after being heated to 110 C for 24 hours. The gravimetric water content is then calculated by dividing the mass of water by the mass of the soil. This relationship is given by the following equation:

$$W = m_w / m_s \qquad (6)$$

where,

m_w is the mass of the water;
m_s is the mass of solids.

From the gravimetric water content, the volumetric water content (θ_v), which represents a volume of water to a volume of soil relationship, is calculated using the following equation:

$$W = m_w / m_s \qquad (7)$$

where,

W is the gravimetric water content;
ρ is the soil density;
ρ_w is the water density.

All data obtained in the three measurements were submitted to statistical analysis to determine the correlation coefficients, the standard deviation, and the confidence interval for 95% reliability. All measurements were made at the Soil Laboratory of the Center of Technology of Federal University of Paraiba at João Pessoa, PB, Brazil, at a room temperature of 26.8 C and a relative humidity of 74%.
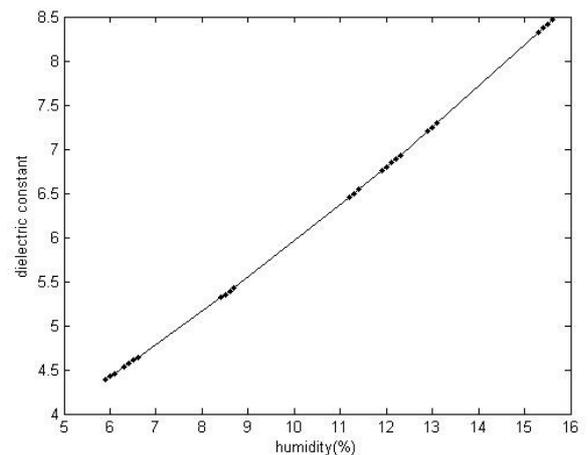
6. MEASUREMENT DATA

Fig1. Humidity (%) vs. Dielectric Constant of bentonite.

Figure 1 shows the relationship between the dielectric constant and the humidity. The direct influence of the humidity on the dielectric constant is observed for a correlation coefficient of 0.997. An increase of the humidity of the sample after a depth of 200 mm was expected because bentonite is very sensitive to variations in the environment humidity. This was confirmed during the realization of the

measurements when higher values for the humidity were read by the probe at depths of 300 mm, 400 mm , 600 mm, and 1,000 mm when therewas an increase in the humidity inside the tube and a greater compaction of the material. In fact, this increase in humidity is due to the environmental conditions, which, in turn, increases the specific surface of the samples, and therefore of their volumetric water content. The end effect is an increase of the dielectric constant of the material (bentonite) and its humidity value. An average value of 6.47 was found for the dielectric constant of bentonite with a standard error deviation of 0.04. The average humidity obtained was 11.1%, with a standard error of 0.163.The obtained confidence interval for the dielectric constant for 95% reliability is CI=[6.15;6.80]. Comparing the value for the dielectric constant obtained in this work with data found in the literature (5.4-7.0) at 20°C , it was noticed that the FDR method is very accurate and gives good results when applied in studies of soils and clay minerals. The average value of 11.1% for the humidity measured by the FDR-based probe is close to the average value of 12.8% obtained in the calcium carbide method that is commonly used to determine the humidity of soils.

7.CONCLUSION

The measurement of humidity and dielectric constant of bentonite using an FDR-based probe proposed in this work is a reliable and accurate method for studies of soil and clay minerals. Compared to the gravimetric and the calcium carbide methods, the FDR method presents good accuracy in almost real-time because it takes only two seconds to obtain a reading, while the gravimetric method needs 24 hours to complete a reading and the calcium carbide method needs two to four minutes. The FDR method also makes possible the simulations for different depths in the laboratory, which is important when simulating in-situ situations. The dielectric constant is an important quantity in soils studies because it influences the size of the DDL. The overlapping of DDLs due to variations in the dielectric constant of bentonite makes it difficult for water and electrolytes to flow through it. Comparing the FDR with the commonly used calcium carbide method, the FDR method shows good accuracy and gives an almost real-time reading . Another advantage of the proposed FDR method   is that it is possible to make measurements using large quantities of soil simulating different depths, which is important when simulating *in situ* conditions in the laboratory.

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