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Determination of Gravimetric Water Content of Porous Media Based on Time Domain Reflectometry

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ABSTRACT

Time domain reflectometry (TDR) has become the most commonly utilized method for calculating the water content of porous media due to its advantages of rapidity, safety, and non-destructiveness. Nonetheless, the dielectric constant of porous media with the same gravimetric water content varies considerably due to their different porosity, so the TDR method cannot be directly used to measure the gravimetric water content. Although the traditional thermo-gravimetric method has high measurement accuracy, sampling is cumbersome and measurement is time-consuming. Therefore, it is essential to develop an accurate and effective method to calculate the gravimetric water content of porous media. Based on the dielectric measurement results, a third-order polynomial fitting equation describing the relationship between volume water content and apparent dielectric constant of quartz sand is obtained by employing the least square method. Then a TDR method, which can eliminate the influence of gap through secondary compression, is proposed to calculate the gravimetric water content of porous media. The comparative experiment with the thermo-gravimetric method demonstrates that this method has high measurement accuracy, and provides an approach for predicting the gravimetric water content, bulk density, and porosity of porous media, which can be used in field and laboratory applications.

Keywords: TDR; apparent dielectric constant; quartz sand; bulk density

1. INTRODUCTION

In agricultural and industrial applications, it is often necessary to measure the gravimetric water content of porous media. For example, in the production of glass products, the water content of the raw material quartz sand is very important. If the water content is too high, it will lead to blockage; While the water content is too low, it will cause dust and environmental pollution. The common method for measuring the gravimetric water content of porous media is the thermo-gravimetric method, which has the advantage of high measurement accuracy, but its main disadvantage is that it cannot be measured in situ, the sampling is cumbersome and the measurement is time-consuming. For example, to accurately obtain the water content in the soil, it usually needs to be dried at 105°C for 24 hours [1]. Consequently, it is imperative to establish an accurate and efficient method to measure the gravimetric water content of porous media.

The porous media represented by quartz sand and soil are three-phase mixtures consisting of a solid phase, liquid phase (i.e., water), and gas phase (i.e., air), with dielectric constants of ~5, 80, and 1, respectively [2]. The theoretical basis for the dielectric method to measure the water content of porous media is the huge difference in dielectric constants between the three constituent phases, that is, the apparent dielectric constant K_a of porous media is very sensitive to the volumetric water content θ_v of the media [3-4]. Time Domain Reflectometry (TDR) is the most widely used method for determining water content of soil and other porous media due to its rapidity, safety, non-destructiveness, and automatic timing of measurements and multiplexing of TDR equipment [5]. Many researchers have researched this and proposed a series of models to describe the relationship between K_a and θ_v, including empirical models, semi-empirical models, and theoretical models. However, the dielectric constants of porous media with the same gravimetric water content vary

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considerably due to their different porosity, so the TDR method cannot be directly used to measure the gravimetric water content of porous media. In this paper, firstly, using the dielectric measurement results of quartz sands with different water contents, based on the least squares method, a third-order polynomial fitting equation describing the K_a-θ_v relationship of quartz sands is obtained. On this basis, using the conversion relationship between volumetric water content, gravimetric water content, and bulk density, a method for measuring the gravimetric water content of porous media based on TDR is proposed, and the measurement accuracy and repeatability of the method are experimentally verified.

2. MATERIALS AND METHODS

2.1 Materials

When studying the dielectric properties of porous media, many researchers use quartz sand as the material to be tested [6-9]. The two types of quartz sand selected in this experiment are the raw materials of glass products, which are made of high-purity quartz ore after crushing, screening and washing. Among them, refined quartz sand is mainly used to produce glass cups and other products that have high glossy requirements, while ordinary quartz sand is mainly used to produce glass jars and other products that have no or low glossy requirements. The densities of these two types of quartz sands were measured by the pycnometer method and it was found that the density values ρ_s of both were 2.65 g/cm^3. The particle size analysis of quartz sand was carried out by the sieving method, and the results are shown in Table 1.

Table 1. Particle size distribution of materials

Sand type	**Particle size distribution of quartz sand, %**			
	>600 μm	*200-600* μm	*75-200* μm	*<75* μm
Fine sand	0	31.5	67.5	1
Ordinary sand	0	49.7	50.2	0.1

2.2 Measurement of the apparent dielectric constant

The apparent dielectric constant of quartz sand under different water content was measured by MiniTrase TDR (6050X3K5B) cable tester. The time domain reflectometer transmits the generated excitation electromagnetic wave to the waveguide probe through a standard BNC connector, coaxial cable, and waveguide connector. When the electromagnetic wave propagates back and forth along the waveguide probe, the propagation speed is affected by the dielectric constant of the porous medium around the probe. Android devices equipped with the TraseTerm APP are connected to TDR through Bluetooth, and 1200 reflected signal data points are collected at a sampling frequency of 10 ps to complete a single measurement of TDR. Analysis, including tangent fitting, is then auto-made on this TDR waveform, to determine the start and end time of reflection. The acquired transit time information in turn provides the key to calculating the volumetric water content θ_v of porous media. This test uses a custom-made two-rod stainless steel waveguide probe with a length of 100 mm, a diameter of 6 mm, and a center-to-center spacing of 50 mm.

2.3 Preparation of quartz sand samples with different water content

4000g refined quartz sand is dried in an oven (electric constant temperature blast drying oven, 105 ° C, 24h) and cooled to room temperature, then put into a graduated PVC cylindrical container with a diameter of 195 mm and a height of 265 mm, add 160 g tap water and stir evenly to obtain a quartz sand sample with a weight water content of 4%; using the same method, the quartz sand samples with the gravimetric water content of 8%, 12%, 16%, 20%, 24%, and 28% were prepared. These seven samples are used as group one to calibrate the relationship between K_a and θ_v. Using the above method, the second group of quartz sand samples with a gravimetric water content of 5%, 6%, 7%, 8%, 9%, and 10% were also prepared. Combined with the quartz sand water content testing requirements of glass products factories, the TDR-based gravimetric water content determination method proposed in this paper is verified. In order to test the accuracy and the repeatability of the proposed measurement method, six samples of refined quartz sand and six samples of ordinary quartz sand with unknown water content were randomly selected from the quartz sand raw material warehouse at different periods.

2.4 Experimental procedures

The surface of a sand sample is flattened, then its volume is recorded and its dielectric constant is measured. Apply a force of 400 N (i.e., compressive stress σ=0.013 MPa) to the sand sample and maintain the pressure for 3 minutes, record its volume and measure its dielectric constant. To ensure the accuracy of the measurement results, the dielectric constant was measured 5 times for different positions of the same sample, and the average value was taken. Apply a force of 800 N to the sand sample and maintain the pressure for 5 minutes, record its volume and measure its dielectric constant. Repeat the above steps until all samples are tested. For the 12 random samples of quartz sand with unknown water content, the method proposed in this paper and the traditional thermo-gravimetric method were used to measure the gravimetric water content, and the experimental results were compared.

3. RESULTS & DISCUSSION

3.1 Dielectric constant and volumetric water calibration

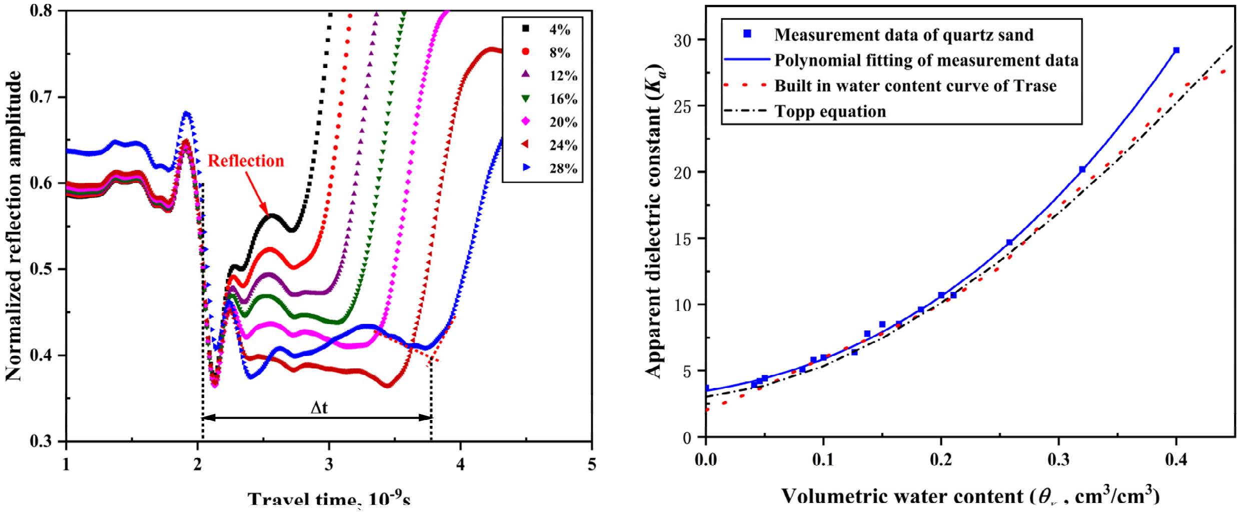
Fig. 1(a) shows the measured TDR waveforms of the first group of seven kinds of refined quartz sand samples with different gravimetric water content in the compressed state 2. It can be seen from the figure that with the increase of water content, the propagation time of electromagnetic wave along the waveguide probe increases monotonously. The TraseTerm APP automatically analyzes the TDR waveform by using the double tangent analysis method, gives the apparent dielectric constant value K_a of a single measurement as well as the corresponding volume water content according to the built-in water content table of the MiniTrase. Using the measured volume value of each sample, the gravimetric water content was converted into volumetric water content, and compared with the TDR measurement results, it was found that the measurement error of TDR in the initial state was large (the error analysis will be further compared and analyzed later.), so only the measurements in the two compression states are used in the following specific calibrations. The classical general empirical formula describing the relationship between the volumetric water content θ_v of porous media and the measured apparent permittivity K_a is the third-order polynomial calibration curve given by Topp [3]:

$$K_a = 3.03 + 9.3\theta_v + 146.0\theta_v^2 - 76.7\theta_v^3 \tag{1}$$

Topp equation is based on 18 kinds of experimental results of four mineral soils and porous media such as vermiculite, organic soil, and glass beads based on factors such as bulk density, texture, salinity, and temperature. Based on the dielectric constant measurement results of seven kinds of quartz sands in group 1 under two compressed states, referring to the fitting form of the Topp empirical formula, the third-order polynomial curve is shown in the blue solid line in Fig. 1(b). The fitting equation between the apparent dielectric constant K_a and the volumetric water content θ_v of the refined quartz sand obtained in this experiment is:

$$K_a = 3.48 + 14.12\theta_v + 90.17\theta_v^2 + 88.38\theta_v^3 \tag{2}$$

Two other curves are also drawn in Fig. 1(b), one is the built-in water content curve in Trase drawn with a red dotted line, and the other is the Topp equation curve drawn with a black chain dotted line based on (1). Comparing these three curves, it can be seen that the three curves are very close when the volume water content is 5%-25%, which also verifies the higher accuracy of the dielectric method for measuring the volume water content of porous media to some extent. However, when the volume water content is lower than 5%, the three curves, especially the Trase water curve, are quite different from the other two curves. Further analysis found that, corresponding to the apparent dielectric constant value of dry quartz sand (i.e., the volume water content is 0%), the built-in water content table of the Trase is 2.0, the Topp curve is 3.03, while the curve obtained by this fitting is 3.48, the measured value is 3.7 and in [7] is 3.8. Therefore, among the three curves, the curve obtained by this fitting has the highest prediction accuracy under the condition of low water content. When the volumetric water content is higher than 25%, the difference between the three curves gradually becomes larger as the water content increases. One possible reason for this difference is that the Topp curve and the Trase built-in water content table are mainly used to describe the relationship between K_a and θ_v of the soil. Compared with quartz sand, the agglomeration phenomenon of soil decreased its apparent dielectric constant with increasing water content. Another possible reason is that compared with the sand sample volume (2800 cm^3-3900 cm^3) in this experiment, the sample size of the Topp experiment (615 cm^3 or 1844 cm^3) is smaller and the TDR equipment, as well as probe types used, are also different [3]. Further analysis of the fitting curve based on the Least Squares Method shows that its coefficient of determination R^2=0.9981, which is very close to 1, and the residual scatter plot shows disorder. In conclusion, the fitting curve can describe the relationship between K_a and θ_v of quartz sand very well.



(a) TDR waveforms under different gravimetric water content

(b) Comparison between fitting curves of K_a and θ_v

Figure 1. TDR waveforms and fitting curves of quartz sand

3.2 Measuring the gravimetric water content of quartz sand by a dielectric method

Although the dielectric method has high accuracy in measuring the volumetric water content of porous media, and after specific calibration, the measurement error is less than 1%, but for porous media with the same gravimetric water content, the change in gas phase composition caused by the change in porosity, its dielectric constant varies greatly, so the TDR method cannot be directly used to measure the gravimetric water content of porous media. To our knowledge, few studies are using the dielectric method to measure gravimetric water content. References [8, 10] proposed a method to jointly solve the soil gravimetric water content θ_m and bulk density ρ_b by using the soil permittivity K_a and electrical conductivity EC_b. However, in the actual use of this method, the permittivity and conductivity need to be calibrated respectively, and conductivity is comprehensively affected by salinity, temperature, water content and TDR probe length, so the measurement accuracy of this method needs to be further studied [5, 11].

According to the definition of volumetric water content, gravimetric water content and porosity f, it can be known that:

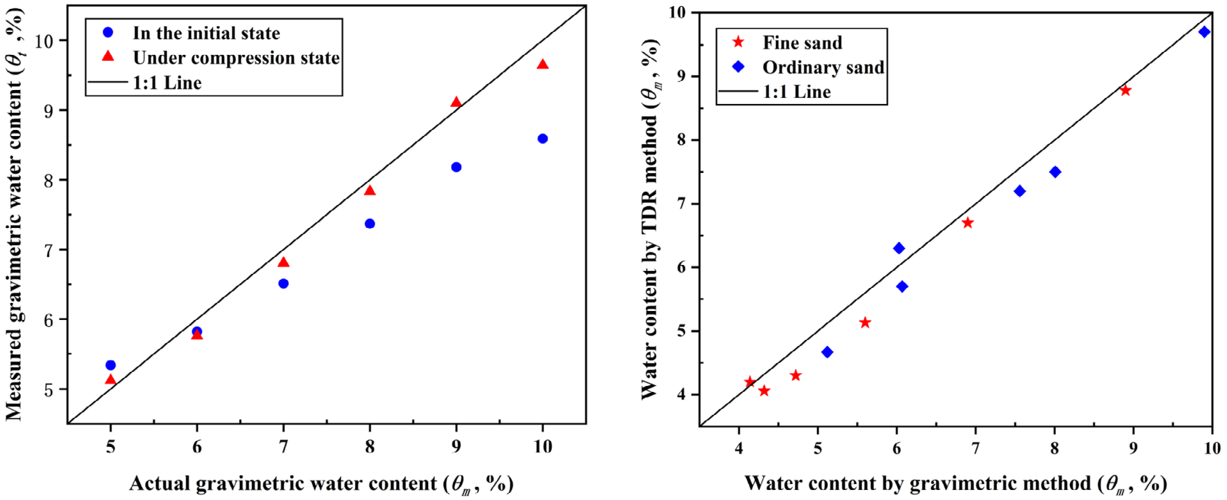
$$\theta_m = \theta_v \frac{\rho_w}{\rho_b} \tag{3}$$

$$f = 1 - \frac{\rho_b}{\rho_s} \tag{4}$$

Where f is the porosity of porous medium, ρ_s is the density of the porous medium (as mentioned in Part II, the density of quartz sand ρ_s=2.65 g/cm^3), and other symbols have the same meanings as above. In agricultural production, it is often necessary to obtain information such as the gravimetric water content and porosity of the undisturbed soil. Due to factors such as the uneven distribution of undisturbed soil pores, the volumetric water content measured by TDR has a non-negligible error. The measurement after compressing the soil sample improves the measurement accuracy but also destroys the initial state of the soil, which seems to enter a dead cycle. To solve this problem, this study adopts the following methods: record the volume value of the sample in the initial state; measure the volume and dielectric constant of the sample in two compression states, and use (2) to obtain the volumetric water content and convert it into the corresponding gravimetric water content; based on (3) and (4), the bulk density and porosity in the initial state are obtained. The measurement results of the second group of samples obtained by the above method are shown in Fig. 2(a). The blue dot in the figure represents the measured value in the initial state, and the red solid triangle represents the average value of the measured value in the two compression states. It can be seen from the figure that the measurement accuracy in the compressed state is significantly higher than that in the initial state, and the absolute value of the measurement error in the initial state tends to increase with the increase of water content. The possible reason for this phenomenon is that under the condition of medium and low water content, with the increase of water content, the fluffy of quartz sand increases, and increasing pore heterogeneity, which leads to the increase of measurement error. The maximum absolute error of measurement in the initial state is -1.41%, and the average error is - 0.53%, while the corresponding values in the compressed state are -0.36% and -0.12%, respectively.

3.3 Validation of the accuracy and repeatability of the proposed method

In order to further verify the accuracy and repeatability of the proposed method, six refined quartz sand samples and six ordinary quartz sand samples randomly selected from the quartz sand raw material warehouse in different periods were tested, and the measurement results were compared with the traditional thermo-gravimetric method, as shown in Fig. 2(b). Among them, the maximum measurement absolute error of refined quartz sand is -0.47%, and the average error is -0.24%; the corresponding values of ordinary quartz sand are -0.51% and -0.27%, respectively. Although there are certain differences in particle size (see Table 1) and composition between the two kinds of quartz sands, the method proposed in this paper is based on the fitting data of refined quartz sand for gravimetric water content prediction, both obtained higher prediction accuracy. In order to verify the repeatability of the experimental results, the dielectric test is carried out on five parallel samples with uniform mixing, and the measured dielectric constant is almost unchanged. Therefore, it can be concluded that the repeatability of the experiment is good, and the main source of experimental error is not caused by the measurement error of the dielectric constant.



(a) Comparison of water content measured in the initial state and compression state with actual gravimetric water content

(b) Comparison of water content measured by TDR with water content measured by thermo-gravimetric method

Figure 2. Accuracy comparison of measuring gravimetric water content by TDR method and by thermo-gravimetric method

3.4 Discussion on the experimental phenomenon and its influence on the accuracy

It is necessary to further explore the three phenomena in the experiment. The first phenomenon is found in the analysis of the TDR waveform, that is, many TDR waveforms measured have undulating bands indicated by the red arrow in Fig. 1(a), which indicates that there is a reflection in the middle stage during the propagation of electromagnetic wave along the waveguide probe. In the process of this experiment, the sample volume is much larger than the sampling volume measured by TDR, and the uniformity of the sample is high after repeated stirring. The measurement avoids the influence of the container wall and the bottom of the container, so there should be no such reflection points. Through repeated experiments many times, it is found that the reason for this phenomenon is the influence of the gap after the last measurement on the next measurement. As shown in Fig. 3 (a), the parallel spacing between the two measurements is 25mm. Compared with the measurement results, it is found that although this kind of gap causes the change in TDR waveform, it actually has little impact on the measurement results in the compressed state, but has an impact on the measurement results in the initial state. The second phenomenon is that the distance between the upper end of the waveguide probe and the upper surface of the sample during measurement, as illustrated in Fig. 3 (b) and (c), has quite an impact on the measurement results. The measurement accuracy in the state shown in Fig. 3 (b) is higher than that in Fig. 3 (c). Hence, we should pay attention to the indentation depth of the waveguide probe when measuring. The third phenomenon is that when the quartz sand is nearly saturated (28% in Fig. 1(a)), the waveform segment marked Δt is generally low on the left and high on the right, indicating that the water is unevenly distributed along the vertical plane. Therefore, the characteristics of the TDR waveform can be used to measure the wetting front of soil.

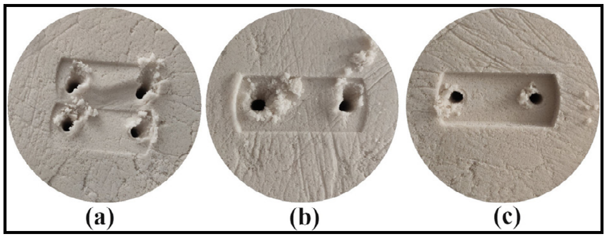


Figure 3. The surface of quartz sand sample after TDR measurement

4. CONCLUSIONS

By measuring the dielectric constants of refined quartz sands with different volumetric water contents, the least squares method is used to obtain a third-order polynomial fitting equation describing the relationship between the apparent dielectric constant K_a and the volumetric water content θ_v of quartz sands. This equation is compared with the Topp equation and the built-in water content curve of the Trase. On this basis, a method for measuring the gravimetric water content and porosity of porous media by TDR is proposed. This method eliminates the influence of uneven gap distribution of porous media under the condition of high porosity through secondary compression differential. Using the gravimetric water content measured under the compression state and the volume value under the initial state, the gravimetric water content, porosity, and bulk density of porous media under the initial state can be obtained. Taking the thermo-gravimetric method as the standard value, the measurement results of two kinds of quartz sands with different particle sizes and different components show that the maximum absolute error of measuring gravimetric water content by this method is less than 0.51%, and the average error is less than 0.27%. Therefore, it can be concluded that this method provides a means to predict the gravimetric water content, bulk density, and porosity of porous media with high accuracy for field and laboratory applications. Higher measurement accuracy can be obtained by preparing calibration samples with smaller water content intervals and further optimization of the K_a-θ_v fitting form.

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