TDR Moisture Measurements in Sand-Like Materials: a Comparison between Different Calibration Procedures

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Abstract- Time Domain Reflectometry (TDR) is a well-established technique for the development of microwave sensors devoted to the dielectric characterization of materials. In particular the TDR has become the standard method for measuring soil water content. Although the response of the TDR signal is strictly related to the medium moisture content, it is also affected by several others factors related to the physical and chemical composition of samples and to the characteristics of soil solution. This way, for each test-case, the investigation of specific calibration procedures, plays a crucial role. This paper is focused on the feasibility of the adoption of three calibration procedures, and on the investigation of the most representative empirical and partly deterministic TDR calibration methods for real-time moisture monitoring of sand-like materials.

I. Introduction

Time domain reflectometry (TDR), is a popular electromagnetic method for measuring soil water content. The advantages of the TDR method over other methods for soil water content measurement are 1) uncertainty level in the range of 1-2%; 2) minimal calibration requirements; 3) excellent spatial and temporal resolution (in the millimetric scale); 4) easiness of use; 5) small dependence on temperature and hysteresis effects, 6) capability of providing continuous water measurements and 7) possibility of several signal controls through multiplexing. Another advantage of TDR is the possibility of simultaneously measuring water content and bulk electrical conductivity with a single probe [1]. Therefore, the TDR method is a suitable method for field measurements as well as for laboratory use. For TDR measurement the relationship between volumetric water content (θ) and the effective permittivity of soil (ε) is needed. Empirical calibration curves are often used for this purpose [2-7]. Besides the empirical calibration curves, several dielectric mixing models have been proposed for describing the $\theta-\varepsilon$ relationships. The dielectric mixing models [8-11], for example, can estimate ε from the permittivities of the medium's components. Therefore, the dielectric mixing model is an adequate method allowing the evaluation of the $\theta-\varepsilon$ relationship for non-homogeneous granular material, for example, with low dry bulk density, large amount of bound water, or relatively large permittivity of the solid phase. Moreover, the dielectric mixing models can be used for understanding the dependency of the effective permittivity on water content and medium's physical properties. This paper examine the robustness of TDR technique for estimating the water content of sand-like materials, through a comparative approach among different methods. Particularly, the following methods are comprehensively compared: (1) the polynomial equation of Topp [2] with its original parameters, (2) a 3rd degree polynomial with four adjusted parameters obtained by calibrations procedures on the materials under test, (3) the partly deterministic simplified models based on the theory of mixtures of three or four components and (4) the probabilistic model of Ansoult [12] based on the random propagation of the electromagnetic pulse in the soil that is schematized as an assembly of capacitors. The paper is structured as follows. Section II describes the proposed approach for the measurement method and the adopted calibration procedures. Section III proposes and discusses results. Finally conclusions are drawn.

II. Proposed approach

In the considered TDR method, a fast rise time (approximately 200 ps) step voltage pulse impinges on a metallic three-rod-probe, placed in the material under test. A portion of the incident electromagnetic wave is reflected back at the beginning of the probe, due to the impedance difference between the sample under test and the characteristic impedance of the probe-system. The propagation velocity of the pulse is strictly related to the dielectric properties of the sample under test [13]. For a TDR probe in a porous medium, the dielectric material between the probe rods is a complex mixture of air, water, and solid particles that exhibits a variable apparent permittivity, ε_a. Since the water dielectric constant

(approximately 80) is much higher than those of the other mixture components (the permittivity of air is 1; while soil minerals and organic matter typically present low permittivity values, around 3-5), a small variation of the medium moisture implies a large variation of the mixture apparent permittivity (ε_a). After a specific calibration, an estimation of the moisture level (θ) of the materials under test can be obtained through the TDR measurement of the dielectric constant. For a porous medium, several factors, besides θ, affect the value of ε_a such as: the physical and chemical nature of the medium, the solute characteristics, the temperature, the bulk density ρ, the material granulometry, the measurement frequency, the water status (free or bound), etc. In order to investigate the specific calibration requirements a first set of testing measurements have been performed on siliceous sand. Three different calibration methods have been considered, thus evaluating the sensitivity of the calibration procedure:

i) Method 1. The medium was watered continually from the surface, with continuous dripping, using a procedure similar to that described in [14].

ii) Method 2. This calibration method, proposed in [15], is based on the progressive insertion of the probe in a medium with homogeneous water content.

iii) Method 3. The medium was watered by a discontinuous process: material samples were placed on a vessel and the fixed reference water content is obtained using a water sprayer, then mixed and packed in graduated glass beakers.

For all the experiments, the samples were dried at 105°C for 16 hours. For the *Method 1* and *Method 2* the samples were placed in a 30cm-length- three-rod-probe immersed in a 7.7cm-diameter cylinder. For the *Method 3*, instead, graduated glass beakers were used (9.4x9.4x13 cm; volume 800 ml). The sample holders were weighed with a high precision electronic balance (Sartorius BL1500) in order to determinate the samples gravimetric water content from the difference between dry and moist sample weight. In order to analyse the appropriateness of TDR technique for estimating the water content of the materials under test, the calibration curves obtained for all the materials are compared to those commonly used in the state of the art.

Firstly, the relationship proposed by Topp [2] has been considered:

$$\vartheta = -5.3*10^{-2} + 2.92*10^{-2}\varepsilon_a - 5.5*10^{-4}\varepsilon_a^2 + 4.3*10^{-6}\varepsilon_a^3 \tag{1}$$

This model provides an adequate description for a water content range <0.5 and for a low mineral organic content.

Secondly, two typical dielectric mixing models have been implemented; one is a theoretical model based on the Maxwell equation proposed by De Loor [16] (*Maxwell-De Loor model*), and the other one is based on the semi-empirical model proposed by Birchak et al. [17] (*α model*). In these models, the water phase of the material under test is considered as a unique phase. However, since the water close to the medium surface is restricted to its molecular movement and it has a permittivity value lower than that of free water, Dobson et al. [9] extended these two models, thus deriving a four-component dependent model that accounts for free water, bound water, solid phase, and air. The Maxwell-De Loor model (*MD model*) is given by:

$$\theta = \frac{3(\varepsilon_m - \varepsilon_a) + 2\theta_{bw}(\varepsilon_{bw} - \varepsilon_{fw}) + 2\phi(\varepsilon_{air} - \varepsilon_m) + \varepsilon_a\theta_{bw}\left(\dfrac{\varepsilon_m}{\varepsilon_{fw}} - \dfrac{\varepsilon_m}{\varepsilon_{bw}}\right) - \varepsilon_a\phi\left(\dfrac{\varepsilon_m}{\varepsilon_{air}} - 1\right)}{\varepsilon_a\left(\dfrac{\varepsilon_m}{\varepsilon_{fw}} - \dfrac{\varepsilon_m}{\varepsilon_{air}}\right) + 2(\varepsilon_{air} - \varepsilon_{fw})} \tag{2}$$

while the *α model* is given by:

$$\theta = \frac{\varepsilon_a - \varepsilon_m^\alpha(1-\phi) + \theta_{bw}\left(\varepsilon_{fw}^\alpha - \varepsilon_{bw}^\alpha\right) - \phi\varepsilon_{air}^\alpha}{\left(\varepsilon_{fw}^\alpha - \varepsilon_{air}^\alpha\right)} \tag{3}$$

where θ is the volumetric water content, θ_{bw} is the volumetric water content of bound water, $\varepsilon_m, \varepsilon_{fw}, \varepsilon_{bw}$, and ε_{air} are dielectric constants for medium particles, free water, bound water and air respectively, while ϕ represents the porosity. The coefficient α accounts for the effects of the geometrical arrangement of the medium components. This coefficient ranges from -1 to 1 (α =-1 and α =1 correspond to the medium stratification perpendicular and parallel to the electromagnetic field direction, respectively). We have considered the standard values of the dielectric constant of the different constituents [18]: ε_{fw} =81, ε_{air} =1, ε_{bw} =3.2 (permittivity of ice, according to [19]), ε_m =value calculated on the dry medium sample. For the α parameter, a value of 0.5 was proposed by [17] and confirmed by [20]. Finally, the theoretical model of Ansoult et al. [12] has been considered. It is a probabilistic model based on the random propagation of the pulse in the medium that is schematized through a system of different capacitors. In this approach, in fact, the granular medium is considered as

a set of three kinds of capacitors associated to the dielectric constants ε_{ij} that correspond to the three medium components (water, air and mineral particles). The computational algorithm derives the total amount of the equivalent dielectric constant encountered by an electrical pulse randomly transmitted through the equivalent electric circuit associated to the medium modelling.

III. Experimental results

In this Section the feasibility of the adoption of the TDR measurements for real-time moisture monitoring of granular materials is assessed. This way, starting from measurements on siliceous sand an in depth analysis, useful for industrial quality control applications, is also developed on some agro-food materials. The data ranges of the most significant parameters are reported in Table I. The table also reports the different moisture levels to which the samples were wetted, according to the three calibration methods described in Section II. The dielectric constant was measured with a 30 cm-length three-rod metallic probe (*CS610, Campbell Scientific*) connected to the reflectometer (*TDR 100, Campbell Scientific*). The probe was vertically inserted from the top of the sample. The sand porosity was derived through a mercury porosimeter. For the agro-food materials the porosity was determined taking into account the air percentage contained among the grains.

Table I. Typical physical properties of materials under test.

	Dry bulk density (g/cm^3)	Porosity	Hygroscopic water content (cm^3/cm^3)	Moisture levels (cm^3/cm^3)
Siliceous Sand	1.30-1.47	0.45-0.52	0.005-0.006	0-0.48
Corn Flour	0.659	0.600	0.071	0-0.25
Soya Bean	0.609	0.475	0.018	0-0.16

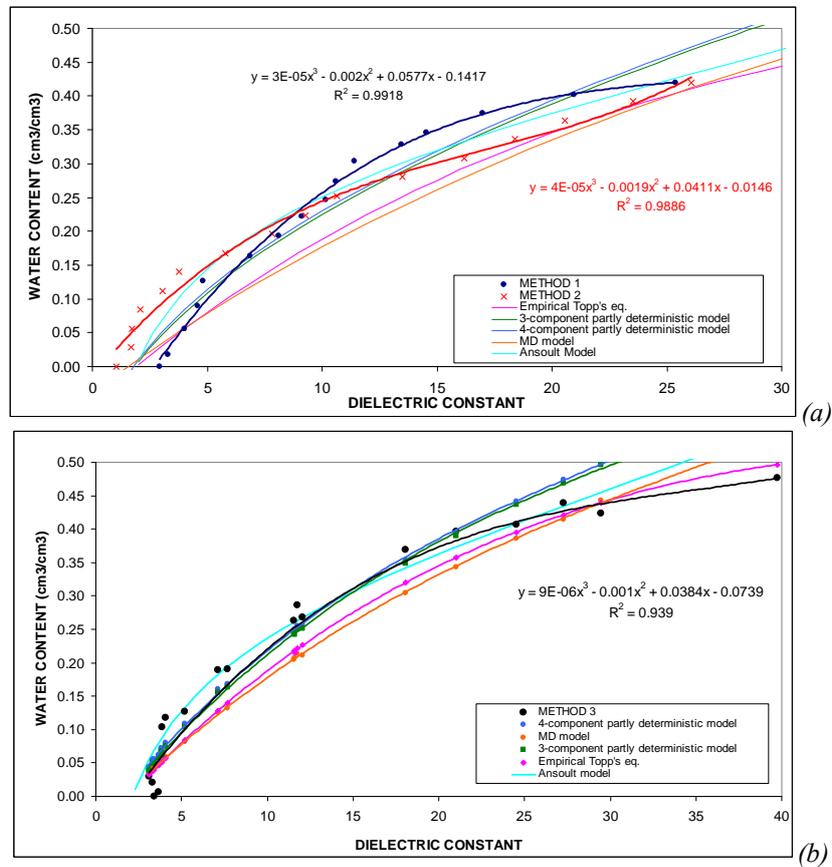#### A. Comparison between calibration methods

Siliceous sand is a very homogeneous material. The use of the sand favoured a quick infiltration of the water, allowing a sufficiently uniform moisture advancing front. However, according to *Method 1*, a vertical water content gradient in the watered sample can not be avoided. This effect can introduce some biases in the TDR signal, due to the possible heterogeneous distribution of sample portions around the probe rods [21]. These water content variations are partly taken into account by the measurement itself. According to [22], in fact, TDR measurements give a weighted average value of the layered water content when the calibration function shows a linear relation between θ and $\sqrt{\varepsilon_r}$, according to the Topp's equation. The experimental data are shown in Figure 1. Small differences are observed among the three calibration procedures, mainly attributable to: *Method 1*), the irregularities of the advance of the wetting front; *Method 2*), the introduction of air between the probe rods and the sand sample, and *Method 3*), the difficulty of uniformly distributing the water portion, as well as the intrinsic irregularities deriving from the manipulation and packaging of humid sand samples. Additionally, the deviations among the three methods can be attributed also to the porosity of the samples varying in the range reported in Table I.
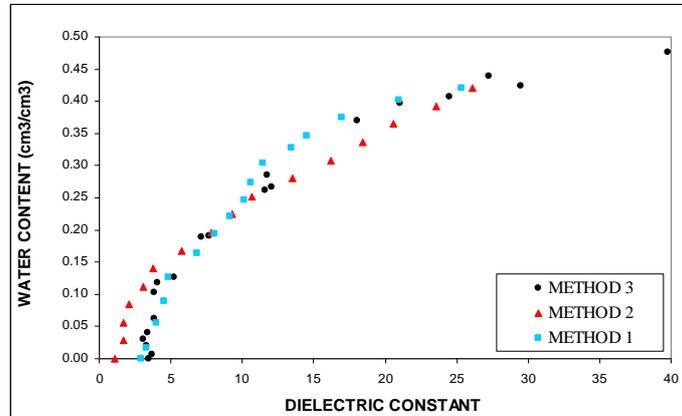
Figure 1. Experimental comparison among the three considered calibration procedures for siliceous sand: *Method 1*, moistening by continuous dripping , M*ethod 2*, progressive introduction of the probe in a constant water content medium, *Method 3*, moistening with spraying water and packing.

B. Comparison between calibration curves

Siliceous sand was considered to individuate the functional relationship between the dielectric properties and the moisture levels and to compare among them the calibration curves derived from Topp's equation, α model, MD model and Ansoult's model. Figures 2.a and 2.b show the dielectric constant measured for the sand at room temperature with all the three calibration methods as a function of the sample moisture. The experimental data are superimposed to the curves obtained through Topp's calibration function, α models (to 3 and 4 components), MD model and Ansoult's model. A good fitting quality is observed between the calibration curves of all the models under test. The partly deterministic models, in particular the Ansoult's and α models, show the best agreement with the experimental data. For the α models this can be attributed to the fact that the sand porosity was explicitly taken into account, thus extending the validity range of these models. In order to assess the quality of the adjustment we calculated the root mean square error (RMSE) between the measured and the simulated values of water content. Results for siliceous sand samples are reported in Table II. Considering all the calibration methods, the RMSE of Topp's equation ranged between 0.039-0.054, while the RMSE of mixing models ranged between 0.030-0.062. The Ansoult's model seems to be the most adequate method, since it presents the best data-fitting.

Figure 2. Experimental measurements on sand samples and comparison between empirical and partly deterministic models.
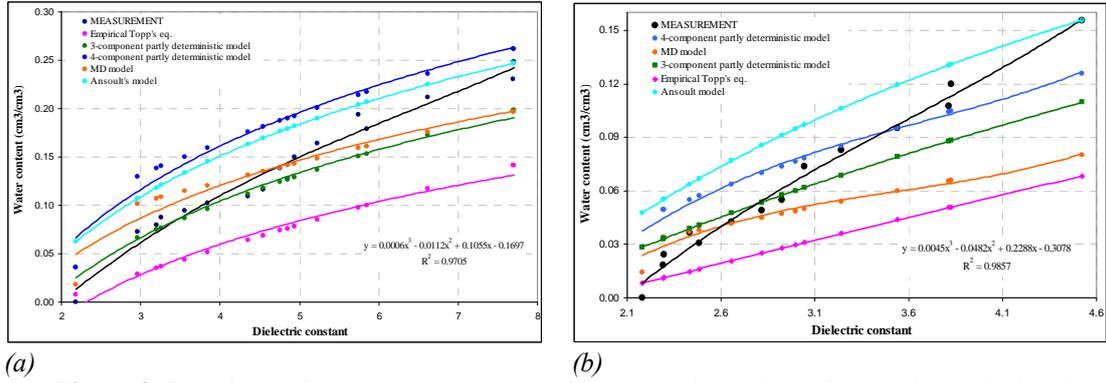
Table II. RMSE between measured and modelled water content data on siliceous sand samples for the different calibration methods.

Model	Calibration Method 1	Calibration Method 2	Calibration Method 3
Topp's Equation	0.054	0.050	0.039
α model (3 component)	0.031	0.043	0.041
α model (4 component)	0.030	0.042	0.045
MD model	0.062	0.052	0.047
Ansoult's model	0.035	0.030	0.035

C. A key-note on agro-food materials

The permittivity of two different agro-food materials, corn flour and soya bean, have been measured with the moisture level ranging among the values reported in Table I. Measurements on these materials have been reported only for the calibration *Method 3*, since experimental results confirmed the best suitability of this method in terms of uncertainty minimization. Figure 3 shows the results. Similarly to the sand case, the curves representing the dielectric constant behaviour versus the moisture level, derived from Topp's equation, α models, MD model and Ansoult's model, are superimposed to the experimental data. The RMSE between the measured and the simulated values of water content are reported in Table III. Results confirm that the empirical Topp's equation seems to be the less suitable for the agro-food materials. Also in this case, as for the siliceous sand, the partly deterministic models show the best agreement with the experimental data.

Considering both the agro-foods materials under test, the RMSE of Topp's equation ranges between 0.042-0.063, while for the mixing models it ranges between 0.019-0.048.

(a)   *(b)*

Figure 3. Experimental measurements on corn flour (a) and soya bean (b) samples and related comparison between empirical and partly deterministic models.

Table III. RMSE between measured and modelled water content data for the different agro-food materials under test.

	Topp's Equation	α model (3 component)	α model (4 component)	MD model	Ansoult's model
Corn flour	0.063	0.022	0.048	0.022	0.038
Soya Bean	0.042	0.019	0.020	0.031	0.030

IV. Conclusion

In this paper the feasibility of the adoption of the TDR technique for moisture monitoring of granular materials, such as siliceous sand and agro-foods, has been investigated. We have demonstrated that TDR moisture measurement on porous media is a valid alternative to gravimetric and to other non-destructive methods, such as gamma radiation probe, neutron probe etc [23]. When dealing with the electromagnetic wave interaction with a dielectrically complex system, like granular materials, the simple adoption of a common $\theta-\varepsilon$ relation is affected by substantial limitations in evaluating the effect of water content. On such bases, specific calibration procedures must be individuated for each specific test case. Starting from measurements on siliceous sand an in depth analysis, useful for industrial quality control applications, has been also developed on some agro-food materials. Particularly, three different calibration procedures have been tested. A specific 3rd degree polynomial relationship between ε and θ has been compared with the empirical and partly deterministic calibration curves, that are commonly used in the state of the art, be them: Topp's equation, α models, MD model and Ansoult's model. Results indicate a good fitting quality between the calibration curves and between all the other models under test. In particular, the partly deterministic Ansoult's model and α models show the best performance in terms of experimental data fitting.

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