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Determination of Moisture Content of Hygroscopic Building Materials Using Time Domain Reflectometry

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Abstract: The application of Time Domain Reflectometry (TDR) technique for measuring moisture content in highly hygroscopic building materials is analyzed in the study. Flue Gas Desulphurization (FGD) gypsum is used as a typical representative of this type of materials. Experimental results show that common methods for calculation of moisture content from measured relative permittivity used in soil science are not applicable for building materials in general. The verification of commonly used conversion functions and mixing formulas by a reference method is necessary case by case.

Key words: Time-domain reflectometry, hygroscopic building materials, calibration

INTRODUCTION

The Time Domain Reflectometry (TDR) method was developed originally for cable testing. It utilized the well known principle in the electromagnetic theory that if a propagating electromagnetic wave meets an interface between two media with different impedances, it is partially or fully reflected. In a cable appearance of such an interface means a fault. The identification of the spatial location of the fault which was crucial for making the necessary repair was done by launching the waves into the cable, detecting the reflection and calculating the distance the electromagnetic wave passed between the launch and the reflection.

The application of TDR in cable testing assumed the known propagation velocity of electromagnetic waves. However, an opposite way of TDR data assessment was feasible as well. If the dimension of a coaxial probe containing a dielectric, i.e., the position of the reflection point was known, one could use the measured time between reflections on both ends for determination of the propagation velocity of the waves, thus to measure the relative permittivity of the material in the probe.

For some time, the application of TDR for determination of dielectric properties of materials was confined to liquids. However, already in 1980s it expanded to other types of materials. A fast development of the TDR technology was initiated in soil science where the method found an increasing use in soil moisture measurement (Topp *et al*., 1980; Dasberg and Dalton, 1985; Dalton and van Genuchten, 1986). TDR also became a recognized technique in the determination of electrical properties of liquid crystals and biomaterials (El Kadiri *et al*., 1985; Bose *et al*., 1986). Later, in 1990s, first experimental setups suitable for compact solid materials began to appear. Wullschleger *et al*. (1996) used TDR for the measurements of water content of wood, Al-Qadi *et al*. (1997) for the characterization of Portland cement concrete.

During the last couple of years the number of ISI references of TDR measurements was stabilized at approximately 120-130 per year and its increase was not as dramatic as during 1990s. Besides the well-established measurements on traditionally analyzed materials mentioned before, some additional applications appeared. For instance, TDR was used for the dielectric characterization of coals (Fornies-Marquina *et al*., 2003), compact building materials (Pavlík *et al*., 2002, 2007; Wansom *et al*., 2006), compact rock materials (Cerepi, 2004; Sass, 2005), foods and agricultural materials (Miura *et al*., 2003, Nelson, 2005) or human skin (Hayashi *et al*., 2005). Following the traditional cable testing procedures, some advanced applications of TDR also appeared in extensometry (Lin and Tang, 2005) and in crack propagation monitoring (Abu Obaid *et al*., 2006).

This study deals with application of TDR measuring technique for monitoring moisture content in hygroscopic building materials. The main attention is paid to the calibration accuracy. The practical example of the measuring technology is given on measurements of moisture profiles in Flue Gas Desulphurization (FGD) gypsum.

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MATERIALS AND METHODS

In the experimental work in this study, the cable tester LOM/RS/6/mps produced by Easy Test was used which is based on the TDR technology with $\sin^2$-like needle pulse having rise-time of about 200 ps (Malicki and Skierucha, 1989). It is computer aided instrument originally designed for measurements of soil moisture. The built-in computer serves for controlling TDR needle-pulse circuitry action, recording TDR voltage-versus time traces and calculating the pulse propagation time along particular TDR probe rods and the relative permittivity of measured material.

A two-rod miniprobe LP/ms (Easy Test) was used for the determination of moisture profiles that was designed by Malicki *et al.* (1992). This probe is designed for monitoring changes in water and salt distribution in material samples. The sensor is made of two 53 mm long parallel stainless steel rods, having 0.8 mm in diameter and separated by 5 mm. The probe cable length from the sensor to the multiplexer is 1 m and cable feeder length from the multiplexer to LOM is 3 m. The sphere of influence was determined with the help of a simple experiment. The probe was fixed in the beaker and during the measurement, there was added water step by step. From the measured data (relative permittivity in dependence on water level) there was found out that the sphere of influence creates the cylinder having diameter about 7 mm and height about 60 mm, circumference around the rods of sensor.

The experiment was arranged in the form of vertical suction of water into naturally dried samples of Flue Gas Desulphurization (FGD) gypsum in air-conditioned laboratory at 23±1°C and 30±2% relative humidity. The sample was prepared by casting. The material, which was used for sample preparation, was β-form of calcined gypsum with purity higher than 98% of FGD gypsum, produced at the electric power station Poèerady, CZ. The water/gypsum ratio was 0.627. The size of the specimens was 70×50×330 mm. Sixteen TDR probes were installed into each sample during the process of casting and connected through multiplexers with the TDR device. After 28 days of hardening and natural drying, the specimens were water- and water-vapor-proof insulated with epoxy resin on four lateral sides to ensure one-dimensional moisture transport. The moisture profiles were then continuously monitored and the experiment was stopped before the water front reached the end of the measured sample. Experimental setup is shown in Fig. 1a and b.

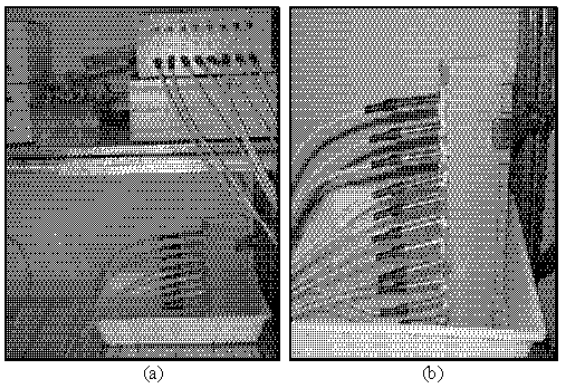

Fig. 1a, b: Experimental setup for measuring moisture profiles using the TDR method

CALCULATION OF MOISTURE CONTENT USING THE MEASURED APPARENT RELATIVE PERMITTIVITY

There are three basic approaches to the determination of moisture content from measured relative permittivity. The first possibility is utilization of empirical conversion functions generalized for a certain class of materials, which, however, are always limited for specific groups of materials only, for which they were proposed. The second possibility is application of dielectric mixing models, which assumes knowledge of the relative permittivities of the material matrix, water, air and other parameters, that can not be measured directly but have to be determined by empirical calibration of the model. The third method for evaluation of moisture content from measured relative permittivity consists in empirical calibration for the particular material using a reference method, such as the gravimetric method. This method is the most reliable until now but the most time consuming one.

In this study, the empirical calibration was done using the final moisture profiles of all analyzed specimens. After finishing each experiment, the sample was cut to 1 cm wide pieces and the relative permittivity data obtained by the TDR device were assigned to the moisture content of the particular 1 cm segments determined by the gravimetric method. The empirical calibration curve was then used for the assessment of two other empirical conversion functions frequently used is soil science and of several calibration functions based on dielectric mixing models.

Among the empirical calibration curves, the conversion functions proposed by Topp *et al.* (1980) and Malicki *et al.* (1996) were tested. The Topp's third order polynomial relation, which is expressed by equation

$$\theta = -5.3*10^{-2} + 2.92*10^{-2}*\varepsilon_a - 5.5*10^{-4}*\varepsilon_a^2 + 4.3*10^{-6}*\varepsilon_a^3 \quad (1)$$

where ε_a is the apparent relative permittivity and θ the moisture content in the porous body [m^3/m^3], had for the originally studied materials standard error of estimate 0.0468 and was proposed for materials having the bulk density close to 1500 kg m^{-3}. The normalized conversion function proposed by Malicki *et al.* (1996) is considered presently by some researchers as universal for different types of materials. The Malicki's function

$$\theta = \frac{\sqrt{\varepsilon_a} - 0.819 + 0.168 * \rho - 0.159 * \rho^2}{7.17 + 1.18 * \rho} \quad (2)$$

where, ρ is the bulk density of dry material, had for the originally studied materials standard error of estimate 0.0269.

The first dielectric mixing model analyzed in this study was the 4-phase α-model proposed by Dobson *et al.* (1985) expressed in formula

$$\theta = \frac{\varepsilon_{eff}^{\alpha} - \theta_{bw}(\varepsilon_{bw}^{\alpha} - \varepsilon_{fw}^{\alpha}) - (1-\psi)\varepsilon_s^{\alpha} - \psi\varepsilon_a^{\alpha}}{\varepsilon_{fw}^{\alpha} - \varepsilon_a^{\alpha}} \quad (3)$$

where, ε_{eff} is the measured value of relative permittivity of the porous medium, θ the moisture content in the porous body [m^3/m^3], θ_{bw} the amount of water bonded on porous walls, ε_{bw} the relative permittivity of bonded water (3.1), ε_{fw} the relative permittivity of free water (79 at 20°C), ε_a the relative permittivity of air (1.0), ψ the total open porosity (60%) determined using vacuum water saturation test and α is an empirical parameter.

The Maxwell-De Loor (1968) mixing model was the second formula which was tested for the application with the studied gypsum material. The Maxwell-De Loor model is expressed by equation

$$\theta = \frac{3(\varepsilon_s - \varepsilon_{eff}) + 2\theta_{bw}(\varepsilon_{bw} - \varepsilon_{fw}) + 2\psi(\varepsilon_a - \varepsilon_s) + \varepsilon_{eff}\theta_{bw}\left(\frac{\varepsilon_s}{\varepsilon_{fw}} - \frac{\varepsilon_s}{\varepsilon_{bw}}\right) - \varepsilon_{eff}\psi\left(\frac{\varepsilon_s}{\varepsilon_a} - 1\right)}{\varepsilon_{eff}\left(\frac{\varepsilon_s}{\varepsilon_{fw}} - \frac{\varepsilon_s}{\varepsilon_a}\right) + 2(\varepsilon_a - \varepsilon_{fw})} \quad (4)$$

RESULTS AND DISCUSSION

Figure 2 shows the results of empirical calibration of the moisture content vs. apparent relative permittivity relation for FGD gypsum. We can see that the shape of the ε (w) function is rather unusual, with practically no dependence of ε on w in a wide range of moisture content from 0.17 to 0.42 m^3/m^3. This may be related to the high hygroscopicity of the material. The relative permittivity of water bonded in a monomolecular layer is

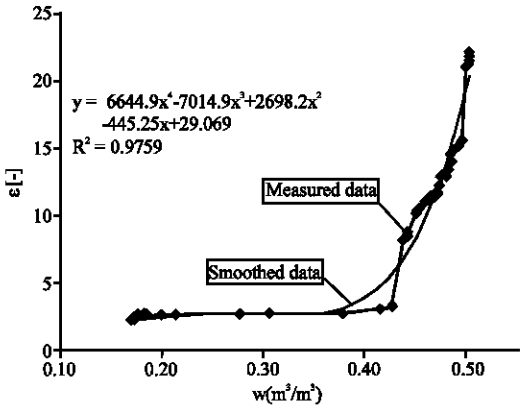

Fig. 2: Empirical calibration curve of FGD gypsum

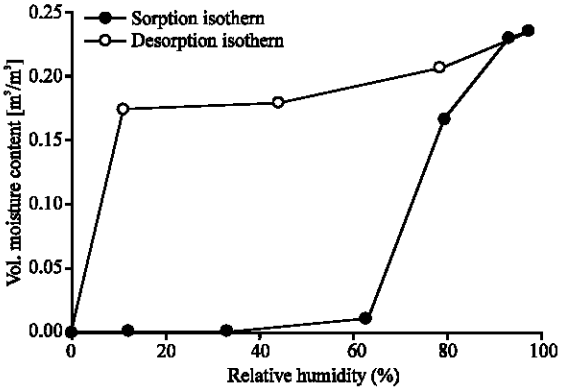

Fig. 3: Sorption and desorption isotherms of FGD gypsum

approximately 2.5, but for further layers it increases relatively fast and for free water it achieves the value of about 80. Therefore, moisture in the hygroscopic range which is bonded mainly by sorption forces to the material matrix can not be detected by the TDR method. This phenomenon is generally more remarkable for low moisture content measurements. However, for highly hygroscopic materials it can be significant in wider moisture ranges.

In order to verify the effect of hygroscopicity on the shape of the ε (w) function of FGD gypsum, adsorption and desorption isotherms were measured. Figure 3 shows that the lowest measured value of moisture content of 0.17 m^3/m^3 corresponds to the relative humidity of about 40% on the desorption curve, the maximum hygroscopic moisture content corresponding to the value of 97% relative humidity is 0.235 m^3/m^3. This means that a very substantial part of the almost constant relative permittivity values on the ε (w) function corresponds to the overhygroscopic moisture.

The most probable reason for this unusual behaviour of the FGD-gypsum ε (w) function is that in relatively

wide range of moisture content, including a part of the overhygroscopic range, water is bound in the porous space very firmly as it is also indicated by the shape of the desorption isotherm. This may be related to the presence of chemically bound water in hardened gypsum, which possibly can establish some bonds with a part of water vapour and even liquid water molecules penetrating into the porous system during the wetting process. However, at the current state of research this is merely a hypothesis which should be verified by further experiments.

Figure 4 presents the moisture profiles in Boltzmann form, calculated using the empirical calibration function in Fig. 2. Clearly, the particular profiles fall into a single Boltzmann profile very well. This indicates that the moisture transport process meets well the conditions of validity of Boltzmann transformation.

Figure 5 shows that both Topp *et al.* (1980) and Malicki *et al.* (1996) empirical functions that proved to be very useful for many soils clearly failed for FGD gypsum. This is, however, not very surprising result. The empirical and semi-empirical formulas for evaluation of moisture content from measured permittivity designed for application in soil science cannot be universal. As most soils exhibit a very low hygroscopicity, the application of soil-science formulas for highly hygroscopic building materials is not straightforward and should always be done with care.

On the other hand, the formulas derived on the basis of dielectric mixing models should be more universal. Their derivation is not confined specifically to soils and they also contain free parameters which can be fitted to match the experimental data. However, the results of extensive parametric studies performed for the Dobson 4-phase α-model and the Maxwell-De Loor model were not very successful as well. This is documented in Fig. 6 and 7.

Figure 6 shows characteristic results obtained by the application of the Dobson 4-phase α-model for different values of the empirical parameter α and different values of the amount of bound water. The first curve with θ_{bw} = 0.2706 and α = 0.25 corresponds to the optimized values using a least square procedure. The other two curves present limiting cases concerning the amount of bound water, θ_{bw} = 0.2374 corresponds to the maximum hygroscopic moisture content, θ_{bw} = 0.4267 to the sudden change on the experimental ε (w) function. The agreement of the calculated results with the calibration curve of FGD gypsum determined by gravimetric method was in all cases clearly very poor.

The first curve in Fig. 7 representing the Maxwell-De Loor mixing model with θ_{bw} = 0.2042 is the least-square

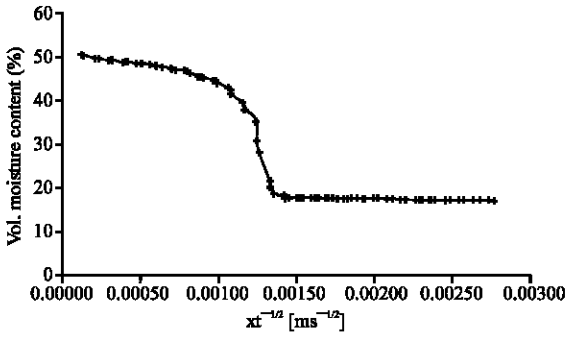

Fig. 4: Moisture profiles in FGD gypsum in Boltzmann form

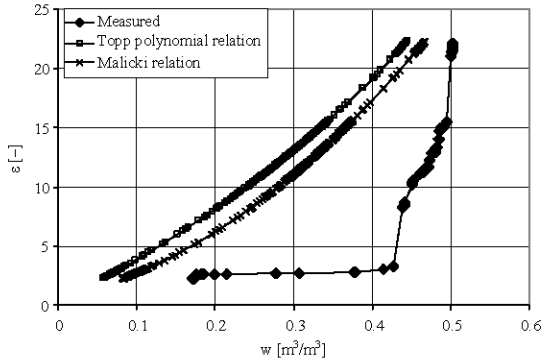

Fig. 5: Moisture dependent relative permittivity of FGD gypsum calculated by two empirical models used in soil science

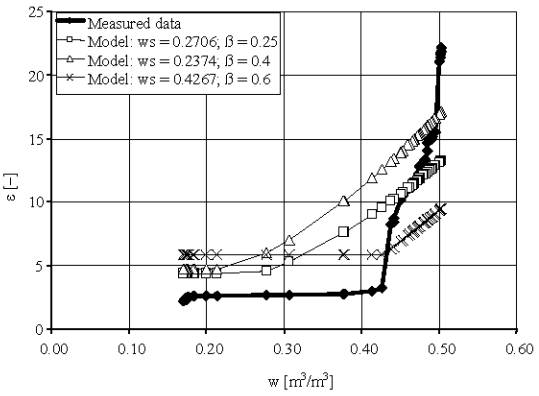

Fig. 6: Moisture dependent relative permittivity of FGD gypsum calculated by 4-phase α-model

optimized case. The other two curves are characteristic examples illustrating the influence of changes of the amount of bound water on the shape of the calculated curve. The agreement with experimental data was very poor as well in this case.

As both the empirical conversion functions used in soil science as well as the Dobson 4-phase α-model and

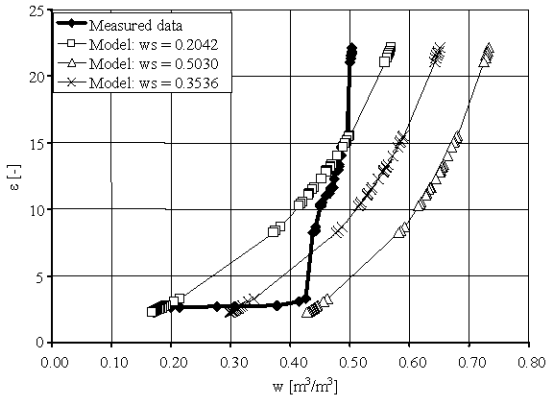

Fig. 7: Moisture dependent relative permittivity of FGD gypsum calculated by the Maxwell-De Loor model

the Maxwell-De Loor model clearly failed in the application to the calibration curve of FGD gypsum, an empirical 4-th order polynomial formula for the calculation of moisture dependent relative permittivity of gypsum was proposed in the following form:

$$\varepsilon_a = 6644.9\theta^4 - 7014.9\theta^3 + 2698.2\theta^2 - 445.26\theta + 29.069 \quad (5)$$

where, ε_a is the apparent relative permittivity and θ the moisture content in the porous body [m^3/m^3].

Figure 2 shows that the empirical formula (5) correlates reasonably well with experimental data, much better than all other analyzed models. The formula can be considered valid for FGD gypsum and similar types of hygroscopic building materials; for other material types its validity should be verified.

CONCLUSIONS

The experiments and calculations performed in this study can be considered as a first step towards the application of TDR technique for monitoring moisture content in hygroscopic building materials. It was found that methods for calculation of moisture content from measured relative permittivity used in soil science are not applicable for building materials in general. A new empirical formula valid for FGD gypsum and similar types of hygroscopic building materials was developed instead. However, the validity of the formula for other types of building materials should be verified. Therefore, in future work we will focus on testing the new formula for other types of materials, on the development of more general empirical conversion functions and on the application of more sophisticated dielectric mixing formulas for specific groups of building materials.

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