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# METHODOLOGY OF MOISTURE MEASUREMENT IN POROUS MATERIALS USING TIME DOMAIN REFLECTOMETRY

## METODYKA PROWADZENIA BADAŃ WILGOTNOŚCI W OŚRODKACH POROWATYCH ZA POMOCĄ REFLEKTOMETRII W DOMENIE CZASU

**Abstract:** The article presents the description of measurement methodology of moisture transport in unsaturated porous materials using Time Domain Reflectometry (TDR) technique on the example of measurement of capillary uptake phenomenon in the sample of autoclaved aerated concrete (AAC). In the paper there are presented basic principles of the TDR method as a technique applied in metrology, its potential for measurement of moisture in porous materials like soils and porous building materials. Second part of the article presents the experiment of capillary rise process in the sample of AAC. Within the experiment moisture content was monitored in the sample exposed on water influence. Monitoring was conducted using TDR FP/mts probes. Preparation of the measuring setup was presented in detail. The TDR readouts post-processing, graphical presentations of the obtained results, short discussion and comparison of TDR readouts to gravimetric measurement were also presented.

**Keywords:** Time Domain Reflectometry, TDR, porous materials, building materials, moisture

Time Domain Reflectometry (TDR) is considered to be one of the most effective methods for moisture estimation in porous media. Contrary to other popular electric techniques of quantitative moisture determination like resistance or Frequency Domain (FD) methods, it gives the possibility of moisture estimation with satisfying uncertainty and without dependence on other factors influencing moisture readouts like temperature or salinity [1-3].

The first papers about the application of TDR method for estimation of soil moisture were noted at the early eighties of 20th century [4] and since then, a constant technical development was noted in the field of electronics [5], sensor constructions [6-13] calibration techniques [4, 14, 15] and also possible applications [16-18].

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According to its name, the TDR technique is based on determining of propagation time of electromagnetic pulse along the sensor buried into the measured material. Basic parameter determined using TDR is apparent permittivity $\varepsilon$ (dimensionless) [19], being the measure of molecules behaviour under the alternate electromagnetic field and energy dissipation of the material after electromagnetic field is released [4, 20]. Apparent density of porous media varies depending on their structure, shares of particular phases but mainly water. This is caused by the dipole character of water molecule. Contrary to other phases of porous media, electric loads distribution of the molecule is asymmetric and the relative dielectric permittivity value reaches 80. Compared to other shares, it is several time greater in value [19]. According to above mentioned sources $\varepsilon$ equals 1[3] for the air, 4-9 for granite, 2-3 for sandstone, 2-6 in case of clay and 4-5 for sand. The difference between the apparent permittivity values of the air, solid phases and water is crucial for moisture determination using TDR technique.

To compare the TDR to other electric techniques of moisture detection it must be mentioned that the dielectric permittivity of the materials is a complex number, divided into the real ($\varepsilon'$) and imaginary ($\varepsilon''$) part. $\varepsilon'$ represents the amount of released energy in alternating field, being the base value for moisture estimation using TDR. $\varepsilon''$ represents energy loses caused by ionic conductivity, which in practice depends on medium salinity. Below formula described complex dielectric permittivity of saline medium [2, 20]:

$$\varepsilon_\omega = \varepsilon'_\omega - i\left(\varepsilon''_\omega + \frac{\sigma_0}{\varepsilon_0 \omega}\right) \tag{1}$$

where: $\varepsilon'_\omega$ - real part of dielectric permittivity of medium at ω frequency, $\varepsilon''_\omega$ - imaginary part of dielectric permittivity of medium at ω frequency, $i$ - imaginary unit, $\sigma_0$ - electrical conductivity, $\varepsilon_0$ - dielectric permittivity of vacuum ($\varepsilon_0 = 8.85\cdot 10^{-12}$ F/m), $\omega$ - angular frequency of the external electric field.

According to the above formula one can conclude, that the imaginary part of the formula influences measurements in low frequencies of electromagnetic field, applied in FD method for example. TDR operating frequency is about 1 GHz [2], which is enough to minimize the influence of imaginary part on the value of complex dielectric permittivity of a saline medium. It can be assumed that ionic conductivity does not influence TDR readouts, which is the greatest advantage of this method comparing to resistance and capacitance (FD). Also, according to literature sources, it is also possible to measure medium salinity basing on TDR trace interpretation [21].

With the above mentioned assumptions about TDR method it is possible to calculate the relative apparent permittivity of the material basing on the following formula [7]:

$$\varepsilon = \left(\frac{c \cdot t_p}{2L}\right)^2 \tag{2}$$

where: $c$ - light velocity in vacuum [m·s$^{-1}$], $t_M$ - travel time along the TDR sensor [ps], $L$ - length of measuring elements of the TDR sensors [m].

Construction of the TDR sensors enables to determine electromagnetic pulse travel time along the sensor basing on the reflections on particular discontinuities of the sensor waveguide, which are intentionally applied in the probe construction. In many cases those

[3] It is dimensionless

discontinuities are the entrance to the probe and its termination. Figure 1 presents an example of TDR probe construction with black arrows showing the discontinuities of waveguide for the electromagnetic pulse.

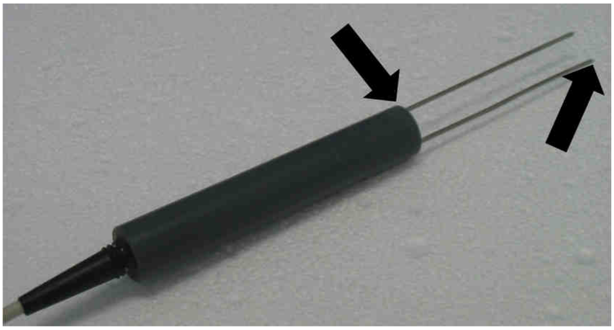

Fig. 1. FP/mts probe (EasyTest, Lubiln, Poland) for moisture determination using TDR method

Figure 2 presents the exemplary traces obtained by measurement of dry and wet material using TDR FP/mts probe. Upper trace is representative for the dry materials (or air) with low value of relative apparent permittivity. Bottom trace represents wet material. The difference in traces is visible in peaks shown in circles. Position of these peaks in the TDR trace depends on the apparent permittivity of material. In case of the dry materials, representative peak is closer to the left part of the trace. In case of wet materials the representative peak is shifted towards right direction, which means that time of the signal propagation along the sensor buried in wet material is longer.

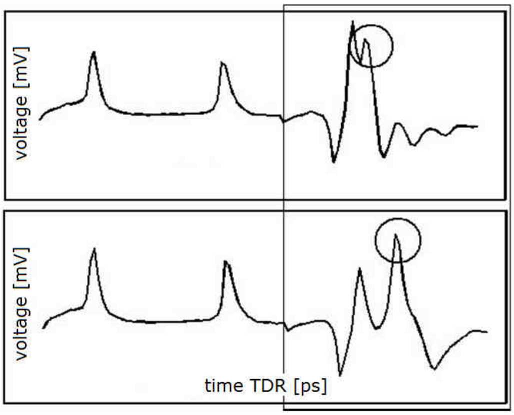


Fig. 2. Example of TDR traces for dry (top) and moist (bottom) material obtained for FP/mts TDR probe (EasyTest, Lublin, Poland)

With measured dielectric permittivity, moisture can be calculated using theoretical and physical models [22-24] or the empirical calibration formulas obtained by the experimental examinations [4, 14, 15, 25]. Practical attempt to the automatic moisture measurement using the TDR technology is presented in the further part of this article.

## Materials and methods

The aim of the research was to evaluate the unsaturated water transport in porous building material using the technique of indirect moisture detection - Time Domain Reflectometry (TDR). For that aim it was conducted an experiment of capillary uptake process in the sample of the aerated concrete.

For the experiment the following materials and equipment were applied:

- sample of the aerated concrete, apparent density in dry 400 kg/m$^3$,
- bitumen isolation,
- laboratory dryer,
- laboratory scales - WPT 6C/1, RADWAG, Poland,
- water container with necessary equipment to sustain the constant water level,
- TDR equipment consisting of laboratory multimeter LOM (EasyTest, Lublin, Poland), TDR probes (FP/mts, EasyTest, Lublin, Poland), PC computer as a control station.

Before the samples of aerated concrete were prepared the maximum water content of measured aerated concrete applied for further experiment was determined. For that aim three samples were prepared. Dimension of each sample was 50 mm×50 mm×45 mm. Next the samples were dried in 105°C to constant mass. Dry samples were weighed and then saturated with water being regularly weighed. Gravimetric and volumetric saturation water content were determined using the following formulas [26]:

$$w_{sat} = \frac{m_n - m_s}{m_s} \tag{3}$$

$$\theta_{sat} = \frac{m_n - m_s}{V} \tag{4}$$

where: $w_{sat}$ - gravimetric water content [kg/kg], $\theta_{sat}$ - volumetric water content [cm$^3$/cm$^3$], $m_n$ - mass of wet sample [kg], $m_s$ - mass of dry sample [kg], $V$ - sample volume [m$^3$].

Apparent density, gravimetric and volumetric saturation water content of the measured material is presented in Table 1.

Table 1

Apparent density, gravimetric and volumetric saturation water content of measured AAC

| **No** | **Mass of dry sample** | **Mass of saturated sample** | **Apparent density** | $w_{sat}$ | $\theta_{sat}$ |
|---|---|---|---|---|---|
| | **[g]** | **[g]** | **[kg/m$^3$]** | **[kg/kg]** | **[cm$^3$/cm$^3$]** |
| A1 | 43.1 | 89.5 | 383.1 | 1.08 | 0.41 |
| A2 | 41.7 | 87.2 | 370.7 | 1.09 | 0.40 |
| A3 | 40.2 | 89.2 | 357.3 | 1.22 | 0.44 |
| A4 | 41.1 | 88.4 | 365.3 | 1.15 | 0.42 |
| A5 | 42.5 | 87.3 | 377.8 | 1.05 | 0.40 |
| A6 | 41.3 | 87.9 | 367.1 | 1.13 | 0.41 |

Basing on the obtained results it was established, that the apparent density of the measured sample was equal 370.2 kg/m$^3$. Average gravimetric saturated water content was 1.12 kg/kg and volumetric saturated water content 0.41 cm$^3$/cm$^3$.

Contrary to the classical observations of capillary uptake process TDR method enabled constant monitoring of the phenomenon and its dynamics quantitative description. For that aim a sample of aerated concrete was prepared. Dimensions of the sample were the following: 24×16×6 cm. The sample was dried in 105°C to constant mass and weighed. Mass of dry sample was equal to 851.8 g. Then the sample was insulated with bitumen mass to prevent any influence of ambient air on moisture parameters of the measured sample. The bottom side of the sample was left uncovered to absorb water from its source.

After the bitumen isolation was matured, the sample was equipped with the necessary TDR probes. For the experiment there were used FP/mts TDR probes produced by EasyTest, Lublin, Poland. Four probes were distributed according to Figures 3 and 4 at the following levels above the water: 7 cm (probe 0), 12 cm (probe 1), 17 cm (probe 2), 22 cm (probe 3). The sample was placed into a water container on small supports (Fig. 4). The container was filled with tap water to the level of 2 cm above the bottom edge of the sample, which meant that particular TDR probes are placed 5, 10, 15 and 20 respectively above water level. Constant water level was kept by the specially prepared system for such type of experiments consisting of long pipette filled with water.

Before water was poured into the container, the TDR equipment was initiated to measure moisture content at particular levels of the sample. Time step of the experiment was set for 15 minutes. Duration of the experiment was about 11 days until no significant water content increase was observed and near-saturation state was read by the measuring setup. The research was conducted in semi-isothermal conditions about 20°C (±0.5°C), air relative humidity was about 50% which was not important for the experiment due to bitumen insulation of the sample.

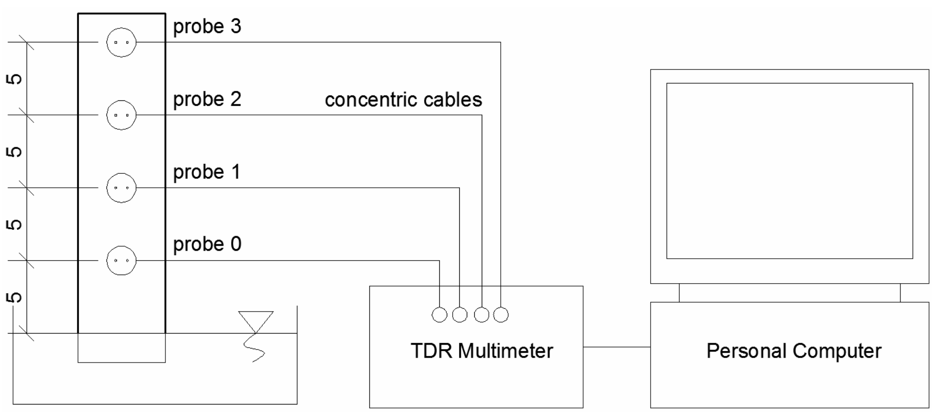


Fig. 3. Measuring setup for capillary uptake experiment - scheme

After 11 days the experiment was terminated and the obtained data was post-processed according to the procedure described in the next sub-chapter.

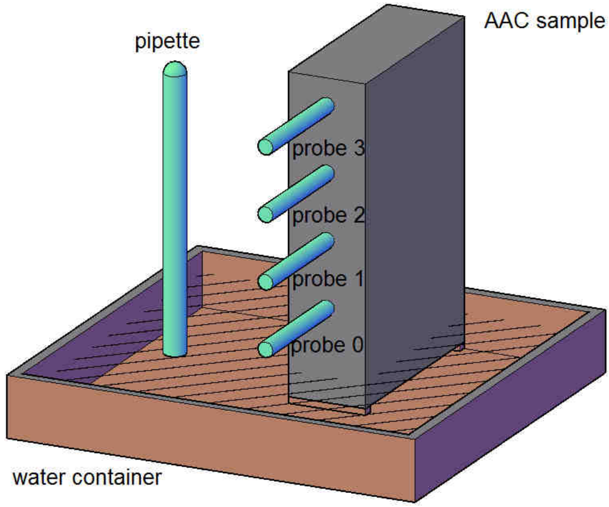


Fig. 4. AAC sample with TDR probes ready for experiment - visualisation

Moisture determination using TDR method required several attempts before final readouts as volumetric water content were obtained. Among them there can be mentioned: calibration of the TDR setup, calculation of signal travel time along the TDR sensor, calculation of apparent permittivity and finally - moisture estimation.

Before the experiment was initiated, the TDR equipment was calibrated. First stage of calibration was devoted to establish the parameters of the device and particular TDR probes. These parameters are reference time $t_{ref}$ and characteristic probe length $l_p$ which depend on particular experimental setup and are connected to probe geometry [27]. According to literature sources [28-30] this stage of calibration ought to be conducted using two media, that significantly differ in the dielectric permittivity value. According to [28] water ($\varepsilon$ = 80) and air ($\varepsilon$ = 1) could be used. However, according to [29] it is strongly recommended to use water ($\varepsilon$ = 80) and benzene ($\varepsilon$ = 2.3) for this stage of calibration. Application of the air as the reference medium is controversial due to low time-shift of between peaks of reflectogram which may often run to falsified results. For the described experiment the TDR equipment was calibrated in water and benzene and reference time $t_{ref}$ and characteristic probe length $l_p$ were determined. With the reference time it was possible to establish travel time of signal along the sensor according to [27]:

$$t_M = t_{probe} - t_{ref} \tag{5}$$

where: $t_M$ - travel time along the TDR sensor [ps], $t_{probe}$ - particular travel time reading on the trace, measured by TDR multimeter [ps], $t_{ref}$ - reference time of sensor [ps].

Data acquired by the TDR setup were saved into the files separately for each sensor. In each file the following information was stored - time of measuring step and time of pulse propagation ($t_{probe}$). Time of pulse propagation $t_{probe}$ was determined by the TDR multimeter automatically by analyzing of the traces, discussed in the previous sub-chapter of the article, presented on Figure 2.

With $t_{probe}$ value determined by the TDR multimeter it was possible to calculate travel time along the TDR sensor $t_M$ which was used for further calculations. With known geometry of the sensor and the $t_M$ value it was possible to determine apparent permittivity for each time step of measurement and each sensor using (2) equation.

As it was discussed above apparent permittivity $\varepsilon$ is substantial information for moisture determination using the TDR method, and it was used for determination of volumetric water content. For that aim two approaches are possible - the physical one and the empirical one. Both approaches are widely discussed by Černý [27].

In many cases of moisture determination, there are applied two basic formulas elaborated experimentally by Topp et al [4] and Malicki et al [3]. It was proved by literature information that the most significant formula applied in the TDR technology [4] is only valid for materials with bulk density about 1500 kg/m$^3$ [27] and the standard measurement uncertainty varies between 0.05 and 0.15, depending on material [31]. Also, as it was presented in the table 1, the investigated material density is about 400 kg/m$^3$. For that aim it is suggested to apply semi-empirical Malicki's formula [3] which, together with apparent density considers bulk density. According to [27] standard uncertainty of this approach is better for TDR data post-processing and equals 0.0269 cm$^3$/cm$^3$.

Literature review prove that even universal models for moisture determination are prone to significant uncertainties [11, 32, 33], especially in case of porous media like building materials [27]. That's why in case of many porous materials, individual calibration is required. In the following papers the TDR measurement is proceeded with individual calibration for the examined material [34-36].

The procedure of individual calibration of TDR readouts relies on comparison of TDR readouts and gravimetric measurements of material water content. Basing on the obtained results the calibration curve is generated and regression formula obtained to be used as calibration empirical formula. Such an attempt was described in above mentioned [34-36] papers. For the experiment described in the following paper it was applied formula which was proposed for aerated concrete with bulk density 400 kg/m$^3$ in [15]

$$\theta = -0.001\varepsilon^2 + 0.0426\varepsilon - 0.0337 \qquad (6)$$

where: $\theta$ - volumetric water content [cm$^3$/cm$^3$], $\varepsilon$ - apparent permittivity determined with TDR [-]. According to estimations of the authors of this paper, standard uncertainty of this formula equals about 0.01 cm$^3$/cm$^3$, which is a better value when comparing to popular models by Topp and Malicki.

## Results

With all above presented calculations a set of data containing moisture was obtained for each probe and time step. This enabled to determine the dynamics of the examined phenomenon. It is presented in the form of diagram on Figure 5 where readouts of each probe are presented.

The diagram presented on Figure 5 presents four curves describing the phenomenon of capillary rise. It is clearly visible that the lowest probe (probe 0) showed moisture increase after 6 hours after the experiment was started. The pace of the moisture growth was very dynamic and within one day TDR readouts showed moisture values exceeding 0.3 cm$^3$/cm$^3$. Probe 1 installed 10 cm above water level showed moisture increase after the first day of process and within next two days moisture readouts exceeded 0.03 cm$^3$/cm$^3$. Readouts of probes 2 and 3 were shifted in time for the period of 1-2 days. What was important and worth of discussion is that after seventh days of experiment duration all probes showed moisture value about 0.3 $cm^3/cm^3$ and since that period the moisture started to grow to reach values close to 0.4 $cm^3/cm^3$ at the end of measurement, which was a value close to saturation determined for the samples before the experiment of capillary uptake had started.

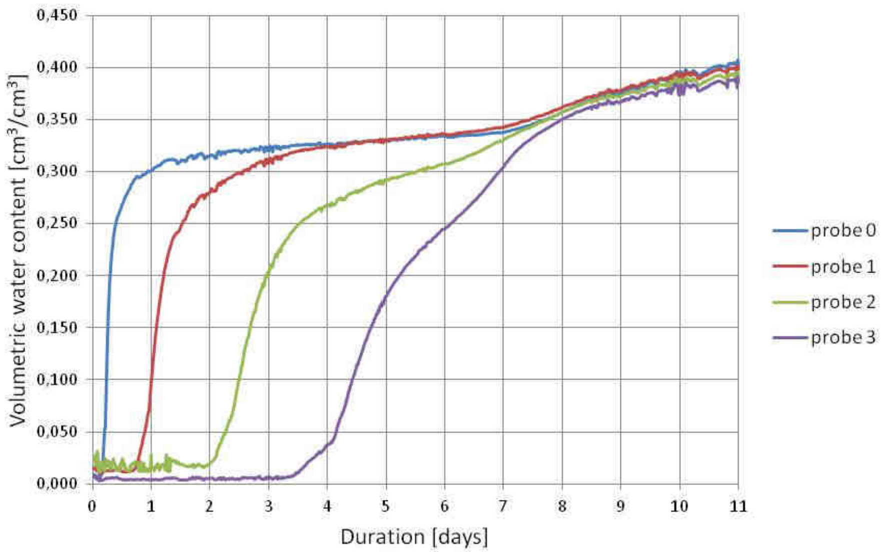


Fig. 5. Volumetric water content read by TDR probes during the capillary uptake process

Two stages of moisture increase on each probe ought to be explained with complicated porous structure of examined material and uneven distribution of pores dimensions. It can be supposed that at the first stage of experiment the smallest pores are filled with water due to capillary suction and great capillary forces. After the smallest pores are saturated the next phase of process occurs and the greater pores, with lower capillary forces, are filled with water. That's why this stage of capillary suction process is slower.

After the experiment was finished the sample was weighed to compare the TDR readouts with direct gravimetric data. Mass of saturated sample was 1752.4 g, which means that the average volumetric water content was about 0.39 $cm^3/cm^3$, and was similar volumetric water content read by all TDR probes at the end of the process, being 0.03 $cm^3/cm^3$ smaller than material saturation determined in the initial determinations presented in Table 1.

## Conclusions

The following conclusions can be drawn from the analysis of the literature and results:

- The conducted research confirmed hydrophilic properties of aerated concrete as a porous medium.
- Use of Time Domain Reflectometry enables constant monitoring of water change in porous materials.

- Readouts of TDR equipment in near-saturation state can be compared to gravimetric estimation of the sample before and after capillary suction experiment. Both estimations show the same moisture values equal 0.39 $cm^3/cm^3$.
- Gravimetric and reflectometric readouts at the end of the capillary suction experiment (about 0.39 $cm^3/cm^3$) were smaller than saturated water content determined before the experiment (0.42 $cm^3/cm^3$). The difference can be explained by both methods measurement uncertainty, inhomogeneous material structure or impossibility to reach saturation by capillary suction in the upper parts of the sample.

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## METODYKA PROWADZENIA BADAŃ WILGOTNOŚCI W OŚRODKACH POROWATYCH ZA POMOCĄ REFLEKTOMETRII W DOMENIE CZASU

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**Abstrakt:** W artykule przedstawiono opis metodyki pomiaru transportu wilgoci w nienasyconych porowatych materiałach przy wykorzystaniu techniki Time Domain Reflectometry (TDR) na przykładzie pomiarów zjawiska podciągania kapilarnego przez próbkę autoklawizowanego betonu komórkowego. Zaprezentowano podstawowe informacje na temat metody TDR jako techniki stosowanej w metrologii. Omówiono jej potencjał do pomiarów wilgoci w takich ośrodkach porowatych, jak gleby i porowate materiały budowlane. Druga część artykułu przedstawia eksperyment podciągania kapilarnego przez próbkę autoklawizowanego betonu komórkowego. W trakcie trwania eksperymentu monitorowano zmiany wilgotności w próbce wystawionej na oddziaływanie wody. Monitoring realizowano za pomocą sond TDR FP/mts. Rozdział „Materials and Methods" przedstawia szczegółowo przygotowanie stanowiska pomiarowego. W rozdziale „Results" podano odczyty miernika TDR przeliczone na wilgotność oraz zaprezentowano uzyskane wyniki w postaci graficznej. Zawarto w nim również krótką dyskusję wyników i porównanie odczytów TDR z pomiarami grawimetrycznymi.

**Słowa kluczowe:** Reflektometria w Domenie Czasu, TDR, ośrodki porowate, materiały budowlane, wilgotność