

České vysoké učení technické v Praze

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**Methods for Determination of Moisture Content of Porous
Building Materials**

Metody měření vlhkosti porézních stavebních materiálů

Summary

Moisture in building materials represents an important issue in building science. There is evident that the rising of moisture content in buildings leads to the serious negative events, like degradation of materials (disintegration of inorganic plasters, porous stones, ceramic bricks, binder decomposition, surface erosion, etc.). It has also negative effects on biological devaluation of constructions, and on the hygienic conditions of interior climate. Significant is also the effect of moisture rising on mechanical properties of bearing-structures materials, and on the thermal performance of materials. On that account, there is clear that is necessary to prevent the presence of higher moisture content in building structures during their service life. In the case of moisture induced damage of buildings, there is necessary to access and monitor moisture content and to classify its amount in respect to optimization of restoration process. The knowledge of moisture distribution is also important for the material research especially for determination of moisture transport and storage properties of materials.

This lecture provides an overview of the currently applied methods for moisture measurement in porous building materials. At first, the harmful action of liquid water on performance of materials is introduced. Then, the moisture sources, and moisture forms are briefly discussed. In the following text there are described particular methods of moisture measurement including their main physical principles, accuracy and reliability. The specific attention is paid to the TDR method that is introduced as highly advanced tool for moisture measurement in laboratory as well as in-situ conditions. Finally, the studied theme is summarised and the practical recommendations for moisture measurement in porous materials are given.

Souhrn

Vlhkost stavebních materiálů představuje důležité téma stavebního výzkumu. Je zcela evidentní, že nárůst vlhkosti stavebních konstrukcí vede k závažným negativním důsledkům, jaký představuje například degradace materiálů (rozpad pevné struktury anorganických malt, poréznych kamenů, keramických cihel, rozklad pojiv apod.). Nadměrná vlhkost způsobuje také biologické znehodnocení konstrukcí a negativně působí také na hygienické podmínky vnitřního prostředí budov. Významný je vliv nárůstu vlhkosti na pokles mechanické pevnosti materiálů a na zhoršení jejich tepelně izolační funkce. Z výše uvedených skutečností vyplývá nutnost zamezit pronikání vlhkosti do stavebních konstrukcí a to po celou dobu jejich životnosti. V případě, že dojde k proniknutí vlhkosti do konstrukce a k následnému poškození její funkčnosti, je pro optimální návrh sanačních a rekonstrukčních postupů nezbytné stanovit a monitorovat obsah a distribuci vlhkosti. Znalost distribuce vlhkosti je nezbytná také v materiálovém výzkumu, zejména v případě stanovení vlhkostních transportních a akumulčních parametrů materiálů.

Tato přednáška se zabývá v současnosti aplikovanými metodami pro měření obsahu vlhkosti poréznych stavebních materiálů. Nejprve jsou stručně popsány negativní vlivy zvýšené vlhkosti materiálů na jejich chování. Dále jsou uvedeny zdroje vlhkosti, její formy a vazba případného transportu vlhkosti k porézni struktuře materiálů. Následuje popis jednotlivých metod měření vlhkosti, včetně jejich přesnosti a spolehlivosti. Zvláštní pozornost je pak věnována metodě TDR, která je prezentována jako univerzální metoda nejen pro měření v laboratoři, ale i přímo na stavbě. V této části jsou prezentovány výsledky vlastních měření a výpočtů demonstrující použitelnost metody TDR pro měření vlhkosti stavebních materiálů. V závěru je proveden souhrn jednotlivých metod měření a uvedeny praktické připomínky a doporučení pro měření vlhkosti ve stavebních materiálech.

Klíčová slova

porézní stavební materiály, obsah vlhkosti, vliv vlhkosti na chování materiálů, zdroje vlhkosti, formy vlhkosti, definice a klasifikace vlhkosti, metody měření obsahu vlhkosti, metody přímé a nepřímé, kalibrace metod měření vlhkosti, stanovení obsahu vody z naměřených fyzikálních veličin

Keywords

porous building materials, moisture content, effect of moisture on materials performance, moisture sources, moisture forms, definition and classification of moisture, methods of moisture measurement, direct and indirect methods, calibration of moisture measurement methods, evaluation of water content from measured physical quantities

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1. Introduction

Present building industry and its modern trends are characteristics by enhanced requirements on quality and comfort of buildings interior climate and prolonged durability and service life of structures. These requirements are induced not only by the building users and occupiers, but also by investors of newly built or reconstructed buildings that pay attention on quality as well as on price of construction works and materials. The continuous rising of above given requirements can be meet only with the use of newly developed advanced building materials that should maintain their properties even in the case of unfavourable environmental conditions.

Among the environment action on building materials, especially the moisture changes represent crucial negative factor that can seriously worsen their utility properties and performance. Therefore, the properties of porous building materials should be accessed as functions of moisture content to ensure their optimised performance in real climatic conditions. From this fact comes the need of moisture content measurement in inner structure of materials, especially in laboratory conditions.

On the other hand, there is also need to measure moisture content in real conditions of buildings. Typical examples represent monitoring and investigation of current state of existing buildings that have serious harmful problems because of negative water action. In this case, not only the moisture content is measured, but also its distribution and classification must be measured and accessed.

The above given facts clearly document the actuality of problem of moisture measurement in porous media and on that account the study on moisture measurement methods is presented in this lecture.

1. Moisture action in building materials and structures

Deterioration and the functionality decrement of porous building materials are strongly related to the presence of moisture. Most chemical degradation processes of buildings and inbuilt materials require moisture [Roels, 2000].

From the point of view of mechanical properties, the presence of higher moisture content significantly decreases the compressive and bending

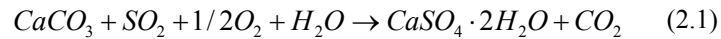
strength of bearing-structures materials and whole structures. The seriousness of this effect clearly documents fact that the amount of contained moisture is taken into account in calculation of mechanical properties according to Czech and international standards. For example in Czech standard ČSN 73 0038, that was replaced in August 2005 by ČSN ISO 13822 (Bases for design of structures - Assessment of existing structures) is the computational compressive strength of masonry reduced by the coefficient of reliability that is dependent on the masonry moisture content and the quality of the brick bound. Probably the worse compressive strength problems can be observed for materials, where the binders forming the solid matrix react with water. Typical examples represent adobes (unfired clay bricks) that exhibit in saturation state the compressive strength nearly zero, whereas the compressive strength in the dry conditions is in the range 5 – 8 MPa in dependence on production method and composition of rare materials [Morel et al., 2005]. The higher moisture content is also serious problem for gypsum based materials. For example Tesárek and his colleagues [Tesárek et al., 2007] have analysed in details the effect of moisture on compressive and bending strength of gypsum blocks based on the flue gas desulphurization gypsum. They have found, for moisture content higher than approximately 0.20 kg/kg both the compressive and bending strength decreased very significantly, to less than one half, whereas the main decrease was observed between 0.20 kg/kg and 0.40 kg/kg.

Due to hindered hygric expansion of materials inbuilt in structures, the micro-cracks can originate as well.

Furthermore, in damp conditions, porous building materials are susceptible to frost damage, because of the volume changes accompanying the phase conversion from the liquid into the solid phase. The volume changes are typically about 9%. This volume rising can lead, in dependence on the shape and dimension of porous system, to the damage of the material and in some cases to its total destruction, whereas the decrease of frost resistance will be more remarkable for materials having lower strength and elastic modulus.

Water can deteriorate building materials and structure surfaces by acid decomposition reactions. Typical example is sulphur dioxide that dissolves in water and partly forms sulphurous acid and sulphur trioxide forms acid as well. Both acids decompose lime and lime-mixed binders in coatings

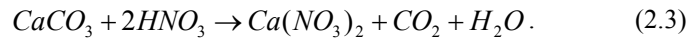
[Rovnaníková, 2007]. The final result is formation of gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The reaction can be expressed in a simplified manner as



and



Another problem for lime based building materials represents water that reacts with nitrogen dioxide in atmosphere and oxygen and forms nitric acid. This acid decomposes calcium carbonate according to the reaction expressed in equation (1.3)



Calcium nitrate is highly soluble substance, and can be leached from the surface layer of structure by rainwater action.

Moist or damp buildings have been in several studies related to the negative health effects [Bornehag et al., 2001]. Most building materials contain excess water when are moisten during their service life and this water must be removed to prevent deterioration of the indoor quality due to the biological growth [Scherer et al., 2001], ranging from bacteria, algae and fungi to moss [Fog Nielsen, 2002]. Micro-organisms cause damage by producing acidic secretions such as oxalic acid, whereas plants can create mechanical stresses by sending roots into crevices.

Another serious problem for the functionality of building materials and buildings represents the decrease of thermal insulation function of materials in dependence on the moisture content rising. This feature is evident from the following figure where is plotted the dependence of thermal conductivity of five different types of lime-based composites on moisture content rising measured at our laboratory.

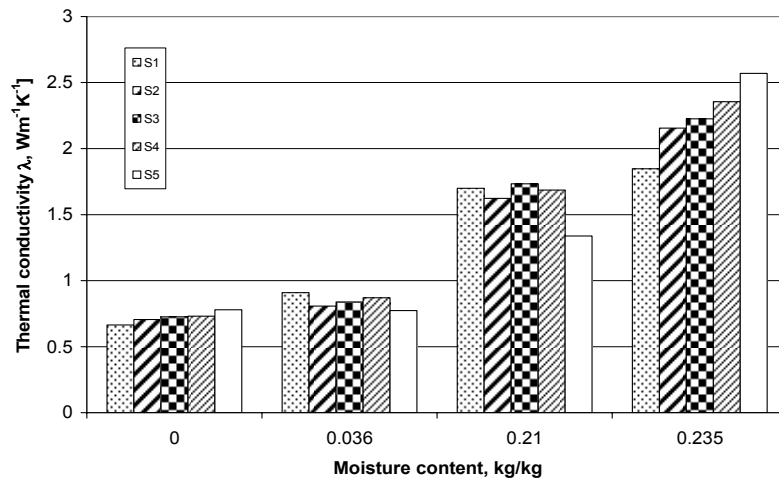


Figure 1: Thermal conductivity of lime-based composites.

The thermal insulation function is based on fact that the thermal conductivity of the air is only 0.026 W/mK [Lide, 1998], whereas the thermal conductivity of the porous matrix is much higher. Therefore, the total porosity of material defines its thermal resistance. Even in usual service conditions, porous building materials contain certain (in some cases significant) amount of water. Since the thermal conductivity of water is approximately 0.60 W/mK at 20°C [Lide, 1998], which is more than 20 times higher than of the air, the thermal conductivity of damp material rapidly increases. This effect was already studied by many authors and is often taken into account in computational modelling of hygrothermal performance of buildings (e.g. [Grunewald, 2000], [Jiříčková et al., 2006]).

2. Moisture sources, effect of porosity, moisture forms

Basically, the moisture sources can be according to the mode and place of moisture leakage divided into the following basic groups:

- atmospheric water – moisture from the environment in all its phases, it is formed by air humidity, atmospheric precipitation and air movement,

- production water (technological, initial) – affected by the wet processes within the material production and building build-up,
- underground water – water contained in the ground and buildings subsoil, this water is transported into the building structures on the capillary uptake principle, and is very harmful especially for historical buildings, where the horizontal hygric insulation is often missing,
- condensed water – this water can be observed as on the material surface as well as in the interior porous structure,
- sorption water – moisture adsorbed by building materials from the ambient humid air,
- supply water – moisture that acts on building structures due to the technological processes.

Particular moisture sources are in the real conditions blending together. More precise information on water penetration in to the building structures can be derived on the basis of physical principals of moisture transport and bonding.

It should be pointed out that the main presumption of moisture transport in buildings is the presence of pores in building materials, whereas only the open pores take part in the transport process. Regarding to the moisture and water solutions transport, the pore size and its distribution are the most important parameters that can be used for estimation of dominant mechanism of moisture transport. According to Hochmann and Setzer [Hochmann and Setzer, 2001] the pores in building materials can be classified into the following groups:

- submicroscopic (ultra-capillary) pores – pore radius $< 10^{-9}$ m, the pore size is comparable to the dimensions of molecules, the water movement is not realized,
- capillary pores – pore radius lies in the range 10^{-9} - 10^{-3} m, water and gases act as in the system of capillary tubes, the water movement is induced by surface tension (capillary forces), the capillary pores can be subdivided into capillary micro-pores (pore radius $2 \cdot 10^{-9}$ – $2 \cdot 10^{-6}$ m), capillary transition pores (pore radius $2 \cdot 10^{-6}$ – $60 \cdot 10^{-6}$ m), and capillary macro-pores ($60 \cdot 10^{-6}$ – $2 \cdot 10^{-3}$ m),

- macro-pores and aerial pores – pore radius $> 10^{-3}$ m, the capillary forces do not participate on moisture transport since the pore size is too large, and the effect of gravity is dominant.

Dealing with the moisture related durability and functionality problems of buildings we should look also at the forms of water present in materials. With reference to the water bonding and pore sizes, five basic phases of water in material can be recognized, nominally free water (fills the large pores and cavities), physically bonded water (bonded by van der Waal's forces), capillary water (filling of small pores and capillaries), adsorbed water (fills the smallest pores and covers the walls of porous space), chemically bonded water (part of crystal lattice of materials).

Roels [Roels, 2000] describes in his work the moisture adsorption and identifies two basic phenomena: molecular surface adsorption in lower relative humidity range, and the capillary condensation in the relative humidity range above approximately 40%. He subdivides molecular absorption in two stages: in a first stage a mono-molecular layer develops on all pore walls. The layer becomes filled at a relative humidity of approximately 25%. The process is then followed by multilayer adsorption. If the porous are large enough, the adsorbed layer reaches a thickness of five molecules at 99% relative humidity. Capillary condensation occurs in pores of size 2-50 nm and was first described by Thomson-Lord Kelvin. It is based on the thermodynamic equilibrium between the relative humidity, the liquid water and the capillary pressure in the pores (see for example [Gregg and Sing, 1982]).

Information on the form of water in material is highly important, because the particular form of water exhibits different physical and chemical properties [Hilhorst et al., 2001], [Kaatze, 2005], [Sihvola, 2000], [Wagner et al., 2007].

3. Definition and classification of moisture

Dealing with the problem of moisture measurement, there is necessary to express and define the term “moisture” (resp. moisture content). Moisture can be defined as a partial density of water, which is the water volume mass of material

$$\phi_w = \frac{m_w}{V}, [\text{kg}/\text{m}^3] \quad (4.1)$$

where m_w [kg] is the mass of water in the measured sample and V [m^3] is sample volume .

The next definition of moisture as volumetric water content can be expressed as

$$\phi_w = \frac{V_w}{V} * \rho_w = u * \rho_w = u * \frac{m_w}{V_w}, [\text{kg}/\text{m}^3] \quad (4.2)$$

where ρ_w [kg/m^3] is the water density, V_w [m^3] is its volume, and u is volumetric moisture content

$$u = \frac{V_w}{V} = \frac{m_s - m_d}{V * \rho_w}, [\text{m}^3/\text{m}^3] \quad (4.3)$$

where m_s [kg] is water saturated mass of sample, and m_d [kg] is the dry mass sample [kg]. The value of the volume moisture content does not exceed 100 % as noticeable from the definition.

The moisture content by mass u_m is defined as a ration between water mass in the defined material volume m_w [kg], and the mass of the solid material in this volume m_d [kg]

$$u_m = \frac{m_s - m_d}{m_d} = \frac{m_w}{m_d}. [\text{kg}/\text{kg}] \quad (4.4)$$

In this case, the value of mass moisture content can exceed 100 %.

The moisture can be defined also with help of saturation degree [%] or relative moisture content [-] defined as

$$\Psi = \frac{u_m}{u_s}, [\%], [-] \quad (4.5)$$

where u_s is mass moisture content of fully saturated sample.

The relationship between volumetric and mass moisture content is as follows

$$u = u_m * \frac{\rho_s}{\rho_w}, \quad (4.6)$$

where ρ_s is the dry material bulk density [kg/m³].

As the measured or calculated moisture content is defined, it shall be classified. In Table 1, there is given classification of moisture in masonry according to Czech standard ČSN P 73 0610, where u_m is the moisture content by mass in [%].

Table 4.1: Classification of moisture in masonry.

Moisture content u_m [%]	Classification
$u_m < 3.0$	very low moisture content
$3.0 \leq u_m < 5.0$	low moisture content
$5.0 \leq u_m < 7.5$	increased moisture content
$7.5 \leq u_m < 10.0$	high moisture content
$10.0 < u_m$	very high moisture content

The given classification pertains to structures of residential premises, whereas it is assumed that the masonry is built from ceramic bricks and lime, lime-cement or cement mortar, from lime-sand bricks, and from traditional stones like sandstone, and calcareous marly limestone, having water absorptivity higher than 10% per mass. The gravimetric moisture content is related mainly to the samples taken from the studied structure in the depth of 100 – 150 mm from the face side of the plaster. Table 4.1 gives only basic information on the hygric conditions of the investigated building.

However, in this way evaluated measured moisture concentrations can put wise about the seriousness of moisture related problems.

4. Moisture measurement methods and principles

Water in all its phases possesses many anomalous properties, which also affect the properties of a porous material. Therefore, there exist various methods of determination of moisture content in porous materials, and various moisture meters [Černý and Rovnaníková, 2002].

The moisture measuring methods can be classified in two basic groups:

- absolute methods (direct methods), which determine the real water content in the material after its removal from the specimen (by drying, extraction),
- relative methods (indirect methods), which determine the amount of water in the specimen on the basis of measuring another physical quantity (permittivity, electrical conductivity, absorption of radiation energy, etc.) having a clear relation to the amount of water in the material.

5.1 Gravimetric method

This method is the most frequently used in the practice. Water is removed from the specimen by drying in exactly defined conditions, and its amount is determined from the loss of weight after drying or by titration. Water contained in a material can also be removed by firing in dry air flow and trapping in a tube filled by drying agent. This method is fundamental to many standard treatments for determination of moisture content in materials and its application requires basic knowledge on the bond energy of water to porous matrix. The knowledge of the bond energy enables determination of total amount of residual water content in material within the gravimetric experiment. In practice, no correction for residual water in porous material after drying is done. Instead of this, such drying conditions are chosen that the amount of residual water in the studied material sample is negligible compared to the measured water loss within the drying process. Since the gravimetric method is usually used for calibration of relative methods of moisture measurements, the weighing of the specimens must be done very precisely because of the possible substantial errors within the weighing process especially for the drying of hot specimens.

5.2 Extraction method

Water in a porous material can be extracted using an organic solvent. As such solvents, glycerol, acetone or ethanol can be applied. The amount of water in the specimen is determined on the basis of the density change of the solvent, permittivity, etc. It should be noted, that not all water contained in the material, but only the amount of water, bonded up to a certain limit of bond energy given by the applied solvent, is determined by the method.

5.3 Resistance methods

Electric resistance of porous materials depends on the amount of water, which can be utilized in determining moisture content. A typical value of the resistivity of a dry porous material is within the range of $10^8 - 10^{13} \Omega\text{m}$. The presence of water in this material can decrease its resistivity to $10^{-4} \Omega\text{m}$. The dependence of the electric resistance R on moisture content u at the constant temperature has usually the form

$$R = a \cdot u^{-b}, \quad (5.4.1)$$

where a , b are empirical constants. However, this relation is valid in a limited range of moistures only, typically for hygroscopic moisture content corresponding to the relative humidity of 30 – 90%. For higher moistures, the resistance decreases more slowly than according to the given exponential relation, because the importance of the volume phase of water in bigger pores is increasing. With increasing temperature the electrical resistance of a moist material decreases, which is due to the ionic conductivity.

Resistance moisture meters have to be always calibrated for the given material and the given moisture range. Determination of moisture content by these moisture meters is very simple. They can be applied for a wide range of materials. Therefore, their application in the practice is very frequent especially for in-situ measurement and test house measurement. For example [Nore et al., 2006], and [Shi and Burnett, 2006] used the resistance (pin) method for monitoring of moisture distribution in semi-scale and full scale testing.

5.4 Dielectric methods

Dielectric methods of moisture content determination consist in an analysis of the behaviour of dielectrics in a time varying electric field. They are based on measuring real (and in some applications also imaginary) part of the complex permittivity as a function of the amount of water. The determination of moisture content using the permittivity measurements is based on the fact that the static relative permittivity of pure water is equal to approximately 80 at 20°C, while for the most of dry building materials it ranges from 2 to 6. The relative permittivity is a complex quantity with real part (ϵ_r') that characterizes the moisture and imaginary part (ϵ_r'') which is a measure of energy loss and electrical conductivity. Both parts depend mainly on frequency, so that the measuring frequency of an electromagnetic technique is a decisive criterion. Based on the frequency of the applied electric field, the dielectric methods can be divided in two groups: capacitance methods and microwave methods (see Figure 5.5.1 for the schematic illustration of the dependence of dielectric methods on measuring frequency).

Capacitance methods are employed in the range of lower frequencies typically from 100 KHz to 100 MHz. The permittivity is determined using a capacitor with the measured material as its dielectric. The measuring capacitor has usually either a simple plate form (for measuring solid materials) or a form of two coaxial cylinders (for measuring loose materials). For the capacity of a plate capacitor, we have the known relation (see, e.g. [Smythe, 1968])

$$C = \epsilon_0 \epsilon_r \frac{S}{d}, \quad (5.5.1)$$

where $\epsilon_0 = 8.854 \cdot 10^{-12}$ F/m is the permittivity of vacuum, ϵ_r is the relative permittivity, S is the area of the capacitor plates, and d the distance between the plates. The measuring capacitor with the capacity C_0 is filled by the measured material, and the changes of its capacity are determined by a proper method.

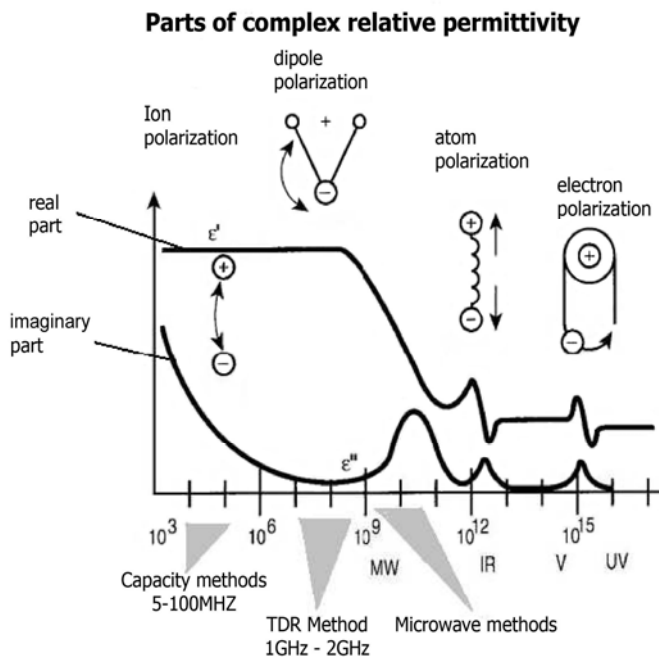


Figure 5.5.1: Dependence of relative permittivity on frequency changes.

The capacitance aquameters found application for example in monitoring the moisture content in early hydration stages of cement mortar [Tydlitát et al., 2000], in measurement of moisture profiles for the calculation of moisture dependent moisture diffusivity [Drchalová et al., 2002], in in-situ measurement of soil moisture [Kuráž et al., 1999], [Johnson, 2002]. Kuráž and his colleagues have used capacitance sensor for measurement of moisture in concrete samples [Kuráž et al., 2000], and Matoušek reported about the universal applicability of cylindrical electrode capacity sensor for moisture measurement in porous building materials [Matoušek and Kuráž, 2007].

The microwave methods for measurement of relative permittivity related to moisture content are quite different from methods working on lower

frequencies. The most often used techniques of relative permittivity determination employ waveguides, cavity resonators, or the measurements are performed directly in a free space.

The measurements in a waveguide are based on the fact, that if a part of the waveguide is filled by a dielectric, the velocity of propagation of electromagnetic waves is changed and the wave is reflected by all air-dielectric and dielectric-metal interfaces.

The measurements in resonators are based on the determination of the change of the resonance frequency induced by putting the dielectric in. In the practical experimental setups, there are almost exclusively used cylindrical cavity resonators with the dimensions corresponding to the formation of transverse electric or transverse magnetic waves.

The measurements in free space are based on the determination of either microwave attenuation after the transmission through a material sample or microwave reflection from its surface.

A specific methodology among the microwave impulse techniques is the time-domain reflectometry (TDR) method which will be closely described in section 6.

The high frequency microwave methods found use especially for measurement of moisture content and its distribution in laboratory conditions because of the complexity of the designed experimental setups and microwave moisture meters. Their accuracy is quite high comparing with other electrical methods of moisture measurement.

5.5 Radiometric methods

Radiometric methods are based on the absorption of radioactive radiation in a material. The most commonly used are the absorption of fast neutrons or γ radiation.

The neutron method utilizes the deceleration (thermalization) of fast neutrons due to their interaction with nuclei of atoms with small atomic

mass. The loss of energy of a neutron due to a collision with an atomic nucleus depends on the mass of the nucleus. In common inorganic materials, the most important substance containing hydrogen, which is the most effective moderator among the elements, is water. Therefore, the amount of water in a material substantially affects the absorption of neutrons, which can be utilized in moisture measurements. For classical experimental setup of moisture measurement by neutron method see e.g. [Van Bavel, 1965].

The γ -ray attenuation method employs for the determination of moisture content the absorption of γ radiation in materials. The intensity of radiation after passing through a material of thickness d is expressed as

$$I = I_0 \cdot e^{-\mu\rho d} + I_s, \quad (5.6.1)$$

where I_0 is the intensity of the incident radiation of the source, μ the mass absorption coefficient (in m^2/kg), ρ the density of material, I_s the intensity of the scattered radiation. The most frequently used sources of γ radiation are ^{60}Co , ^{137}Cs and ^{241}Am . Measuring the moisture content by the γ -ray attenuation method is based on applying the superposition principle to the effects of dry material and moisture on the γ radiation absorption. The effective absorption coefficient of the mixture (water and dry material) is then accessed using the following formula

$$\mu_m = \mu_w c + \mu_d (1 - c), \quad (5.6.2)$$

where μ_m , μ_d represent absorption coefficients of water and dry studied material and c the mass concentration of water

$$c = \frac{m_w}{m_w + m_d}. \quad (5.6.3)$$

For more details on γ -ray method see [Rovnaníková and Černý, 2002]. The main advantages of the γ -ray method are its objective physical character and

a minimal effect of salts on the measured values of moisture. The main disadvantages are a substantial dependence of results on density, and the necessity to measure the absorption on both dry and moist material, what can be a substantial source of errors particularly for inhomogeneous materials, where the density is generally function of the position in the specimen.

5.6 The nuclear magnetic resonance method (NMR method)

The absorption of high-frequency energy in a material exposed to the magnetic field is measured as function of water content. The method can distinguish between the free and bound water. For detailed description and explanation of NMR method see [Černý and Rovnaníková, 2002]. The application of NMR methods for measuring moisture content in building materials does not have a long history, the first experimental setups appeared within the last decade (see e.g. [Pel et al., 1996]). Nevertheless, the method is very perspective, and it can be anticipated that its application frequency will increase in future.

5.7 Ultrasonic methods

The velocity of ultrasound propagation or its attenuation in a material depends on the composition of the material and temperature. At constant temperature, the velocity is affected by the amount of the solid phase, which can be employed for moisture measurements. The dependence of the sound velocity on moisture is for the most of materials nonlinear, and its dependence on temperature is so important, that either the measurements have to be performed in a conditioning chamber, or temperature compensations have to be done. For these disadvantages, this method is not very often used in the practice.

5.8 Infrared spectroscopy method

In measuring the moisture content of porous materials by the infrared spectroscopy method, the reflection of infrared radiation from the surface is employed. The magnitude of the reflected energy depends on the moisture

content in the material. The effect is most pronounced for the wavelengths of 1.4 μm and 1.9 μm . In order to utilize the change of reflectivity to the measurements of absolute moisture content in materials, all other factors affecting the reflection of infrared radiation, such as the roughness, have to be excluded. The main application of the infrared spectroscopy method is in the range of small moistures up to 10% of the maximum moisture content, where the errors are smallest. Experimental devices on the basis of this method require an empirical calibration for every material. The disadvantage of the method is that it determines the surface moisture content only, and not the average moisture content in the specimen.

5.9 Chemical methods

Any chemical method for determination of moisture content is based on the chemical reaction of water in the porous material with a chemical agent. The agent must have the following properties: its reaction with water has to be fast, and it has to make possible and exact quantification of water content from the reaction products, the reaction has to be specific just for water, and its finishing has to be easily detectable. One of the most known chemical methods is based on generation of gases due to the reaction of water with added agent. The amount of gas or its pressure in a closed space is then a measure of the amount of water in the material. The most often used reagents are CaC_2 , CaH_2 and LiAlH_4 .

6. Strong and weak points of moisture measurement methods

The accuracy of moisture content measurement using the electrical methods (dielectric methods, resistance methods) is highly dependent on the temperature. In case of dielectric methods, the changes of permittivity due to the change of moisture are of the same order as the changes due to the change of temperature. In practical application, either the temperature must be maintained constant or temperature compensation has to be included. In TDR moisture measurement is the temperature compensation included in calibration procedure, where calibration media as water, benzene, acetone,

etc. are used. For example, the temperature compensation for water permittivity (ε) is implemented using following equation

$$\varepsilon = 0.2492 \cdot 10^3 - 0.79069 \cdot T + 0.72997 \cdot 10^{-3} \cdot T^2. \quad (6.1)$$

Most of the methods of moisture measurement are characteristic by lower accuracy in case of low moisture content. This feature is most remarkable especially for capacitance methods and resistance methods, where is the problem with measurement of big resistances.

The accuracy and reproducibility of several moisture methods is also strictly dependent on the amount of dissolved salts in water and present in porous system of materials. This feature is remarkable especially for relative methods, where the measured physical quantity is highly dependent on salt concentration. Typical examples are resistance moisture meters although they are often used in building practice even in materials containing salts. For materials with a high amount of salts, the resistance moisture meters are practically inapplicable, because the errors rapidly increase with the increasing moisture content.

The salts dissolved in water are present in form of dissociated ions. On the conduction of electrical current take part free salt ions in solution. In dependence on ion concentration, the electrical conductivity of material is increasing. For lower concentrations of salt water solutions, there is valid direct proportionality between salt concentration and electrical conductivity.

Since the presence of dissolved salts affects the conductivity of water in a significant way (see Figure 6.1), the low frequency operating dielectric methods are not applicable for moisture measurement in salt laden materials. On the other hand, with increasing frequency of microwaves, the importance of salt content for the measured values decreases. The imaginary part of water permittivity increases with frequency (it has a maximum at so called critical frequency, $f_{cr} = 23.4$ GHz), and therefore the relative importance of conductivity decreases. On this account, the application of high frequency microwave method looks very promising for moisture measurement in materials containing salts.

In paper [Pavlik and Mihulka, 2008] we reported about the applicability of TDR method for moisture measurement in building materials containing salts. We have found out, the accuracy of this method is not limited by the

presence of salt ions. Looking on the results presented in Figure 6.1, it is quite clear that TDR method looks like logical solution for moisture measurement especially in the cases, where the popular electrical resistance and capacitance methods fail.

From other methods of moisture measurement, also γ -ray attenuation method and nuclear magnetic resonance (NMR) method exhibit minimal effect of salts on the measured values of moisture.

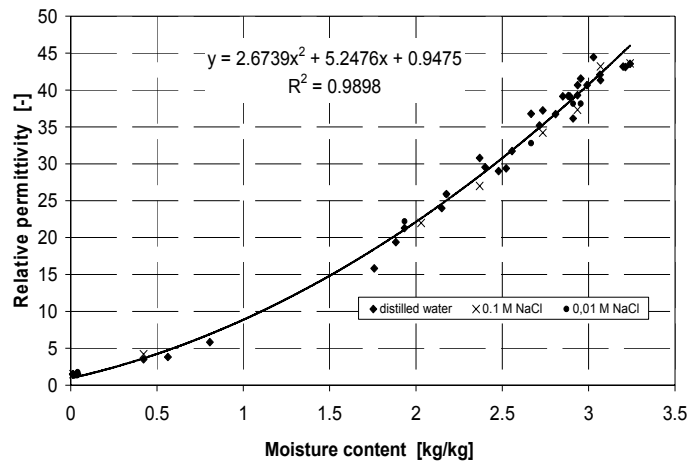


Figure 6.1: Calibration curve of TDR method for calcium silicate material in dependence on NaCl concentration.

7. TDR (Time-domain reflectometry) method

TDR method represents a specific methodology among the microwave impulse techniques. The principle of TDR device consists in launching of electromagnetic waves and the amplitude measurement of the reflections of waves together with the time intervals between launching the waves and detecting the reflections. The fundamental element in any TDR equipment used for the determination of moisture content in porous materials is a device to observe the electromagnetic pulse echo in time domain. The method application originates from the application of electric cable tester. The measuring device usually consists of four main components: a step or

needle pulse generator, a coaxial cable wave guide, a sampler and an oscilloscope to register or visualize the trace of echo.

Time/velocity of pulse propagation depends on the apparent relative permittivity of the porous material, which can be expressed using following formula

$$\varepsilon = \left(\frac{ct_p}{2L} \right)^2, \quad (7.1)$$

where ε is the relative permittivity of the porous medium, c the velocity of light ($3 \cdot 10^8$ m/s), t_p the time of pulse propagation along the probe rods measured by TDR meter and L the length of the rod inserted into a measured porous medium. The determination of moisture content using the permittivity measurements is then based on the fact that the static relative permittivity of pure water is equal to approximately 80 at 20 °C, while for most dry building materials it ranges from 2 to 6.

7.1 Evaluation of moisture content from measure apparent permittivity values

For evaluation of moisture content from measured relative permittivity values, three basic approaches can be used. The first possibility is utilization of empirical conversion functions generalized for a certain class of materials. On the basis of analysis performed it can be stated that empirical conversion functions used in current research for TDR data conversion are anything but universal. They are always limited to specific groups of materials. The second is application of dielectric mixing models, which assumes knowledge of the relative permittivities of the material matrix, water, air and other parameters, that cannot be measured directly but have to be determined by empirical calibration of the model. Dielectric mixing models were tested in many practical applications and their perspectives for further use seem to be better than those of the empirical conversion functions. 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.

7.2 Practical example of calibration of TDR method

For the TDR measurements in the presented experiment, the cable tester TDR/MUX/mts produced by Easy Test which is based on the TDR technology with \sin^2 -like needle pulse having rise-time of about 250 ps, was used. The working frequency of this device is 1.8 GHz for the relative permittivity measurement. Two-rod miniprobes LP/ms (Easy Test) were used. The sensors are made of two 53 mm long parallel stainless steel rods, having 0.8 mm in diameter and separated by 5 mm. The sphere of sensor's influence creates the cylinder having diameter about 7 mm and height about 60 mm, circumference around the rods of sensor. The accuracy of relative permittivity and electrical conductivity reading, and the measuring range of applied sensors are given in Table 7.2.1.

Table 7.2.1: Accuracy and measuring range of applied TDR sensors.

Measured quantity	Measuring range	Accuracy
Relative permittivity ϵ	2 ÷ 90	Absolute error: ± 2 for $\epsilon \geq 6$ ± 1 for $2 \leq \epsilon \leq 6$
Electrical conductivity σ	0 ÷ 1 S/m	Relative error: $\pm 5\%$

The applied TDR device including the sensor is shown in Figure 7.2.1.



Figure 7.2.1: Device TDR/MUX/mts (model 2007).

The presented measurements were done on Ytong cellular concrete samples. At first, two parallel holes having the same dimensions as the sensor rods were bored into each sample. Then, the sensors were placed into the samples and sealed by silicon gel. The samples were partially saturated by water and insulated to prevent water evaporation. The relative permittivity of wet samples was then continuously monitored until the measured values reached the constant value. Then, the experiment was interrupted, sensors removed from the samples and moisture content in the samples was determined using gravimetric method. Finally, the measured values of permittivity were assigned to the gravimetric moisture content. In Figure 7.2.2 there is presented empirical calibration curve of the TDR technique for the moisture measurement in investigated cellular concrete.

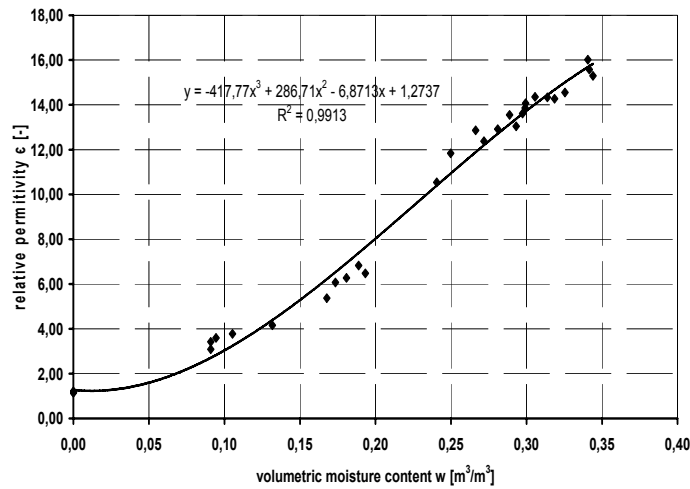


Figure 7.2.2: Empirical calibration curve for studied air aerated concrete.

Also the applicability of dielectric mixing models for calibration of TDR method was studied. The best agreement between the calculated and measured results was obtained for four-phase model which contain the amount of bound water as free parameter. The used four-phase model was proposed by Dobson [Dobson et al., 1985] in the following equation

$$\theta = \frac{\varepsilon_{eff}^\alpha - \theta_{bw}(\varepsilon_{bw}^\alpha - \varepsilon_{fw}^\alpha) - (1-\psi)\varepsilon_s^\alpha - \psi\varepsilon_a^\alpha}{\varepsilon_{fw}^\alpha - \varepsilon_a^\alpha}, \quad (7.2.1)$$

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_s the relative permittivity of solid phase, ε_a the relative permittivity of air (1.0), ψ the total open porosity (0.77) determined using vacuum water saturation test, and α is an empirical parameter. Data obtained by Dobson's model are presented in Figure 7.2.4. In this figure, also the Wiener bounds are presented that represent upper and lower limit of permittivity function.

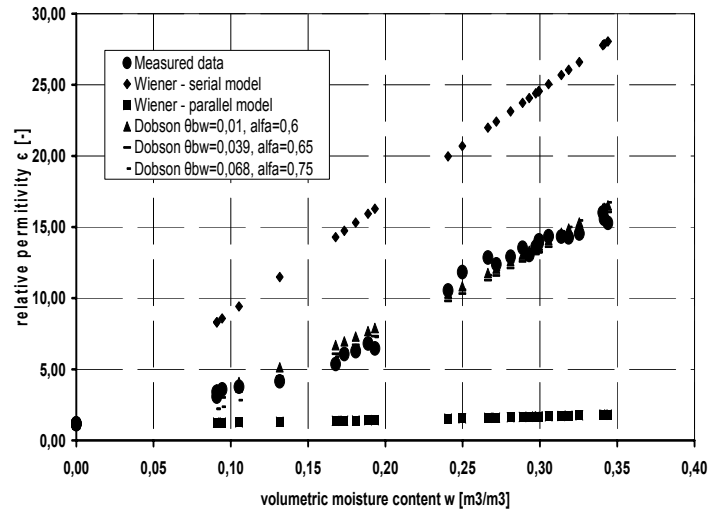


Figure 7.2.4: Moisture dependent relative permittivity calculated by Dobson 4-phase α model

The presented data clearly validate the applicability of TDR method for moisture measurement in porous building materials. Also the dielectric mixing models look highly promising for the shortening of time necessary for calibration.

8. Conclusions and the trends for future research

The presented lecture gives complex summary of the problem of moisture content measurement in porous building materials. The lecture points out to the fact that the currently used moisture measurement methods are not universal for all the types of materials.

Probably the biggest problem can be assumed for materials containing not only pure water, but water containing several types of dissolved substances, like salt ions. In this case, the properties of such water solution are quite different from the properties of the pure water, and as a consequence, also the measured physical quantities based on moisture concentration are different. In this lecture, the TDR method was presented as an advance tool that can be used for moisture measurement even in the case of salt ions presence.

Another problem, especially within the application of electrical moisture meters, represents electrically conducting parts of building structures or materials that highly affect the accuracy and reproducibility of moisture measurement. As a typical example can be introduced concrete containing steel reinforcement. This problem reveals the necessity to use very small moisture sensors which action will be not affected by the conducting particles.

Also the layered materials and structures represent serious problems from the point of view of moisture measurement, because the calibration of relative methods must be usually done for all the material inbuilt in the material structure. In this case, optimised homogenization procedure must be applied. The same feature can be observed for inhomogeneous materials.

Looking at the studied problem from the point of view of environmental conditions of moisture measurement experiments, also the effects of relative humidity, temperature, air pressure etc., must be considered and implemented within the calibration procedure of particular methods of moisture measurement.

Current research in the field of moisture measurement methods is not focused on the development of new methods from the point of view of their basic physical principles, but it is oriented especially on improvement of existing methods and on generalization of calibration procedures that are still very time consuming and expensive.

The researchers intend to design more complex dielectric models that should find application within the calibration of TDR method for different groups of building materials. In these models, spectral density functions will be introduced. In this way, the inner structure of materials will be represented using measurement of pore size distribution curves and the models will be more related to the real structure of materials.

Also the TDR measurement of moisture content for higher salt concentrations and mixtures of salt solutions represent difficult tasks for future research. In literature, there were already published first attempts for simultaneous measurement of moisture and salt ions concentration measurement within the simple suction experiment. In the near future, such experiments performed using TDR technique will be more often and more advanced. From these experiments, the moisture and salt concentration profiles can be accessed and using the inverse analysis procedure, the moisture and salt transport material parameters can be accessed.

Since there is a need to have reliable and precise moisture meter for building materials and structures, the results presented in this lecture significantly contributed to the solution of this problem. The TDR method, originally designed for application in soil science, looks like logical solution for building researches and civil engineers. However, more complex research outlined above must be still done.

In building practise, not only the total moisture content is measured, but also the knowledge of surface moisture is required. On that account, the intensive research on the development of TDR surface probe has already started. The first results on the sensitivity analysis of designed sensors are promising and make good prerequisites for future research.

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Acknowledgment

This research has been supported by the Czech Ministry of Education, Youth and Sports, under project No MSM 6840770031.

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