

Time Domain Reflectometry: Temperature-dependent Measurements of Soil Dielectric Permittivity

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1. Introduction

The purpose of this study is to determine the temperature influence on the soil bulk dielectric permittivity, ϵ_b , calculated from the measurement of the electromagnetic wave velocity of propagation along the parallel waveguide in a TDR probe, i.e. a probe working in Time Domain Reflectometry technique. The experimental evidence shows that the existing models do not completely describe the temperature effect. However, it has been confirmed that the observed temperature effect is the result of two competing phenomena; ϵ_b increases with temperature following the release of bound water from soil solid particles and ϵ_b decreases with temperature increase following the temperature effect of free water molecules. It has been found that there is a soil type characteristic moisture value, θ_{eq} , named the equilibrium water content, having the specific temperature property. The temperature effect for this moisture is not present, which means that for soils with the moisture value equal to θ_{eq} the both competing phenomena mentioned earlier compensate each other. The equilibrium water content, θ_{eq} , decrease is correlated with the soil specific surface area. The temperature correction formula adjusting the soil moisture determined by TDR, θ_{TDR} , at various temperatures to the corresponding value at 25°C, based on knowledge of θ_{eq} , decreases the standard deviation of the absolute measurement error of soil moisture θ_{TDR} by the factor of two as compared to the uncorrected values.

The majority of measurements of physical, chemical and biological properties of porous materials including soil should be accompanied with the measurement of soil water content and temperature. These parameters determine almost all processes in natural environment. It seems obvious to accompany the sensors for the measurement of soil salinity, oxygenation, content of nutrients, soil water potential, and others with the temperature and moisture sensors having the same measurement volume and performing measurements at the same time. Temperature sensors of various accuracy and size are easily available and together with the necessary electronics they can fit into the desired sensor enclosure. More problems are encountered with moisture sensors of porous materials because they do not measure water content directly, but use other parameters of the measured matter that indirectly and selectively determine its moisture. Such a property of porous materials accompanied with moisture is dielectric permittivity.

Measurement of soil moisture using time domain reflectometry (TDR) has become increasingly popular because of simplicity of operation, satisfactory accuracy and fast result available, the process of measurement is non-destructive, portable systems are available and the method gives ability for measurement automation and probes multiplexing (Dirksen & Dasberg, 1993; Malicki et al., 1996; Topp et al., 1980). Beside above advantages there are some drawbacks of this measurement technique including the requirement of excellent contact of the probe to soil and the dependence of dielectric permittivity of soil on temperature affecting the TDR soil moisture readout.

In first applications of the TDR technique for soil moisture determination, the influence of temperature on the TDR determined soil bulk dielectric permittivity, ϵ_b , was neglected. The significant fluctuation of measured data, which was obviously correlated with soil temperature, was noticed with the introduction of soil moisture field monitoring systems based on reflectometric meters. Also, it was found (Pepin et al., 1995; Halbertsma et al., 1995) that ϵ_b decreased with temperature increase for sandy, silt and peat soils and it did not change for the tested clay soil.

The text below uses two terms that need explanation: (i) soil free water – composed from water particles that rotation in the electric field is not hampered, and (ii) soil bound water – composed from water particles so close to solid phase that their rotation in the electric field is hampered by surface charge on the solids. The real part of the complex dielectric permittivity of free water is about 80 at room temperature and is much smaller for bound water (Boyarskii et al., 2002) reaching the minimal value of ice (about 3.2) for the first layer of water particles adsorbed on the clay soil surface.

2. Basics of TDR technique

Time Domain Reflectometry (TDR) technique for the measurement of water content of isotropic and homogenous media becomes popular for the simplicity of operation, accuracy and the non-destructive, as compared to other methods, way of measurement (Malicki, 1999; O'Connor & Dowding, 1999). This measurement technique takes advantage of four physical phenomena characteristic to the porous materials including soil:

- for soils with negligible magnetic properties and electrical conductivity, which is true for the majority of arable soils, and in the frequency range of about 1 GHz, the complex dielectric permittivity of the soil can be approximated by its real value and the electromagnetic wave propagation velocity, v , in the soil can be calculated from:

$$v \approx \frac{c}{[\epsilon(\theta)]^{1/2}} = \frac{c}{n} = \frac{2L}{\Delta t} \quad (1)$$

where: c is a velocity of light in free space, $\epsilon(\theta)$ is a real part of the complex dielectric permittivity dependent on its water content, θ ; $n = [\epsilon(\theta)]^{1/2}$ is the medium refractive index; L is the length of TDR probe rods inserted into the soil; Δt is the time distance between the reflections of TDR pulse from the beginning and the end of the probe rods, inserted into the medium,

- the dielectric permittivity of the medium liquid phase has much higher value than the other medium phases, *i.e.* about 80 against 2-4 for the solid and 1 for the gas phase,

- the relation between the water content of the medium and its dielectric permittivity is highly correlated for the majority of the porous media (Davis et al, 1977; Malicki & Skierucha, 1989; Topp et al., 1980),
- the attenuation of the amplitude of electromagnetic wave traveling along the parallel transmission line inserted into the medium, measured from the amplitudes of the pulse before (U_{in}) and after (U_{out}) attenuation caused by the pulse travel twice a distance of the probe length, L (Fig. 1) depends on its bulk electrical conductivity, EC_b (Dalton et al., 1984):

$$EC_b (\text{Sm}^{-1}) = \frac{(\epsilon')^{1/2}}{120\pi L} \ln \left(\frac{U_{in}}{U_{out}} \right) \quad (2)$$

Therefore, water content of the soil, which is assumed to be an isotropic and homogenous medium, is the main reason determining its bulk dielectric permittivity.

The simplified formula (1) for the determination of the electric pulse propagation velocity in porous medium is derived from more general one:

$$v = \frac{c}{\left(\frac{\epsilon'}{2} (1 + (\text{tg}^2 \delta)^{1/2}) \right)^{1/2}} \quad (3)$$

where: c is the light velocity of propagation in free space, ϵ' is the real value of the complex dielectric permittivity of the medium, $\text{tg} \delta$ is the dielectric loss defined as:

$$\text{tg} \delta = \frac{\epsilon'' + \frac{EC_b}{2\pi f \epsilon_0}}{\epsilon'} \quad (4)$$

The complex dielectric permittivity of the medium, ϵ , is:

$$\epsilon = \epsilon' - j \left(\epsilon'' + \frac{EC_b}{2\pi f \epsilon_0} \right) \quad (5)$$

where: ϵ'' represents dielectric loss connected with the dielectric polarization of soil particles, EC is the medium electrical conductivity, f is the frequency of the electromagnetic field, ϵ_0 is dielectric permittivity of free space, j jest equal to $(-1)^{1/2}$.

The idea of simultaneous measurement soil water content and electrical conductivity as well as the respective hardware setup are presented in Fig. 1.

The TDR probe consists of two waveguides connected together: a coaxial one, called the feeder, and a parallel one, called the sensor, made of two or more parallel metal rods, one of which is connected to the cable hot wire the others to the shield. The sensor should be fully inserted into the measured medium, that by definition should be homogeneous. The initial needle pulse travels from the generator by the feeder towards the sensor. A fast sampling oscilloscope registers this pulse in time. In the junction between the feeder and the sensor, there is a rapid change in geometry of the electromagnetic wave travel path. At this point some energy of the pulse is reflected back to the generator, like in radar, and the remaining travels along the parallel waveguide to be reflected completely from the rods ending. The

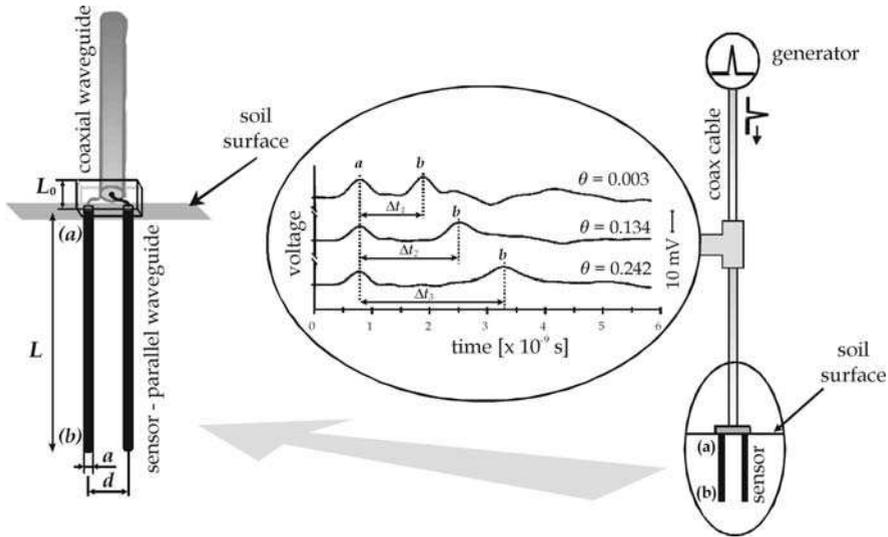


Fig. 1. Hardware setup for simultaneous measurement of soil water content and electrical conductivity using Time Domain Reflectometry technique

successive reflections are recorded for the calculation of the time distance between the two reflections (*a*) and (*b*). Three reflectograms representing pictures from the oscilloscope screen (voltage as a function of time at the chosen point in the feeder) are drawn in Fig. 1. They represent cases when the sensor was placed in dry, wet and water saturated soil. The time Δt_i necessary for the pulse to cover the distance equal to the double length of the metal rods in the soil increases with the soil dielectric permittivity, thus water content. The reason for that is the change of electromagnetic propagation velocity in media of different dielectric permittivity, according to (1). Also, the amplitude of the pulse at the point (*b*) decreases with the increase of soil electrical conductivity, according to (2).

2.1 Temperature effect of soil free water and electrical conductivity

Corrections of the TDR determined moisture data related to the temperature effect of dielectric permittivity of free water was examined by Pepin et al. (1995) and Halbertsma et al. (1995) and the influence of soil texture on the observed temperature effect was reported. Temperature dependence of dielectric permittivity of free water can be described as (Wheat, 1979):

$$n_{fw}(T) = n_{fw} \cdot d(T) = 8.851 \cdot d(T) \tag{6}$$

$$[d(T)]^2 = 1 - 0.4536 \cdot 10^{-2} (T - 25) + 0.9319 \cdot 10^{-7} (T - 25)^2 \tag{7}$$

where $n_{fw} = (\epsilon_{fw})^{1/2} = 8.851$ is free water refractive index at 25°C for the frequency in the range of 10^8 Hz, ϵ_{fw} is dielectric permittivity of free water at 25°C, and *T* is the temperature in °C.

If bulk soil dielectric permittivity had a temperature dependence related solely to free water, measurements of soil ϵ_b would show a negative correlation with temperature that would increase with water content. Results from Pepin et al. (1995) show this negative correlation,

but the overall changes are smaller than predicted from a dielectric mixing model that integrated the temperature change in dielectric permittivity of water. The same was observed by Halbertsma et al. (1995) who made measurements of sand and silt soil samples. Also, he reported the lack of the discussed temperature effect for clay soil. It was obvious that the physical processes involving other soil phases beside the liquid phase should be taken into account when interpreting the temperature effect on the soil ϵ_b .

Literature reports (Nadler et al., 1999; Or & Wraith, 1999) show that soil electrical conductivity does not influence the TDR determined soil bulk dielectric permittivity, ϵ_b , in the soil salinity range where the majority of plants are growing, i.e. up to the corresponding values of soil solution from 1600 to 2600 mSm⁻¹. Following the discussion in Or & Wraith (1999) it is possible to present the frequency dependence of the real and imaginary parts of a salt-water mixture's dielectric permittivity, ϵ' and ϵ'' , as well as the velocity of propagation, v , of electromagnetic wave along the TDR probe rods inserted in this mixture for different temperatures and electrical conductivities, σ_{dc} . The increase of temperature of the salt-water mixture increases the frequency range around the frequency 1 GHz, where the velocity of propagation of an electromagnetic wave along the waveguide, used in TDR technique, does not change. Therefore the temperature effect on the electrical conductivity of soil, resulting mainly from ionic conductivity of soil electrolyte should not influence the bulk temperature effect of soil dielectric permittivity.

2.2 Temperature effect of soil bound water

The discussion of the release of bound water from the solids with the temperature is presented by Or & Wraith (1999), where the authors applied the Debye model for polar liquids and liquid viscosity dependence on temperature, T , for bound water molecules at the distance x from the solid surface to describe their relaxation frequency. They reported that ϵ_b increased with temperature for a silt loam soil for all soil moisture, however another silt loam soil showed the increase of ϵ_b for relatively low water contents, whereas it decreased with temperature at higher water contents. The change of ϵ_b with temperature is not fully explained yet but they discuss its two reasons: ϵ_b decreases with temperature because the real part of soil complex dielectric permittivity decreases following the temperature effect of soil free water and ϵ_b increases with temperature following the release of bound water molecules.

Water molecules that are adsorbed to the solid surface are less mobile in the imposed EM (electromagnetic) field as the not adsorbed ones. For a given temperature, the more distant the water molecules are from the solids, the higher is their relaxation frequency. The increase of temperature increases their kinetic energy that raises the relaxation frequency and they become more mobile in the imposed EM field. Bound water molecules released from the solid surface become now free with higher value of the real part of the complex dielectric permittivity.

The release of water molecules from the solids results in the increase of the real part of the soil dielectric permittivity and decreases its imaginary part describing the loss tangent of dielectric material. This phenomenon leads to a new equilibrium with more free water molecules and less bound water ones.

According to recent studies (Or and Wraith, 1999; Boyarskii et al., 2002), only a few layers of water covering soil particles are subjected to the change of relaxation time in relation to relaxation time of free water. The analysis of nuclear resonance spectra of bound water films

in clay shows the approximated relation between relaxation time of bound water, and thickness of the film covering soil particles (Boyarskii et al., 2002). The resonant frequency of hydrogen in water particles, measured by NMR, is different for various layers of water films on the solids and consequently different frequency response of their dielectric permittivity. The relaxation time of bound water drops rapidly with the number of layers covering the particles and it seems that only one or two layers have significantly longer relaxation times than that of free water.

On the base of Debye model (Debye, 1929; Hasted, 1973) for polar liquids and using relations showing dependence of viscosity of liquid molecules on temperature T and distance x from the solid phase, the relaxation frequency f_{rel} of water molecules can be expressed as (Or & Wraith, 1999):

$$f_{rel}(x, T) = \frac{kT \exp\left(-\frac{1}{T}\left(\frac{a}{x} + d\right)\right)}{8\pi^2 r^3 c} \quad (8)$$

where: k is Boltzman constant ($1.38 \cdot 10^{-23}$ JK⁻¹), T is temperature in Kelvin degrees, $a = 1621 \cdot 10^{-10}$ mK, $d = 2.047 \cdot 10^3$ K and $c = 9.5 \cdot 10^{-7}$ Pas are constants as the consequences of applied simplifications, $r = 1.8 \div 2.5 \times 10^{-10}$ m is the radius of water molecule.

The equation (8) shows that moving closer to solid phase, water particles are increasingly hampered to rotate in the electromagnetic field of high frequency, which is manifested by decreasing relaxation frequency for these molecules. Also, the temperature of water particles will have some effect on the f_{rel} , which is simulated in Fig. 2A.

The dependence of dielectric permittivity (real part) for free and bound water particles on the frequency can be described by Cole-Cole formula (9), where $\omega = 2\pi f$ is the angular frequency of the external electrical field, $\epsilon_{low} = 81$ and $\epsilon_{hi} = 4.23$ are relative dielectric permittivity values for free and bound water particles in the frequency values lower and higher, respectively, from 18 GHz (relaxation frequency of free water particles), $\tau = 1/(2\pi f_{rel})$ is the relaxation time of water particles, $h = 0.013$ is a parameter describing interaction of water dipoles (Hasted, 1973).

$$\epsilon = \epsilon_{hi} + \frac{(\epsilon_{low} - \epsilon_{hi}) \left[1 + (\omega\tau)^{1-h} \sin \frac{h\pi}{2} \right]}{1 + (\omega\tau)^{2(1-h)} + 2(\omega\tau)^{1-h} \sin \frac{h\pi}{2}} \quad (9)$$

Three curves in Fig. 2B represent water particles located at various distances from solid phase and the highest one relates to free water with relaxation frequency of 18 GHz, while the lowest to bound water particles close to solid phase.

The frequency range of an electric field, for which the values of ϵ determined from equation (9) are fixed at about 81, is the largest for free water. Adsorbed water molecules, by virtue of proximity to the solid phase are characterized by lower relaxation frequency and are less mobile in an external electric field. Consequently, according to the formula of Cole-Cole (9), the value of ϵ for frequencies below the relaxation frequencies are lower than for free water. The decreasing number of adsorbed water molecules, due to increasing temperature, increases the amount of free water molecules. Fig. 2A shows that moist soil temperature

increase from 278 K to 338 K increases the relaxation frequency of water molecules located $2 \cdot 10^{-10}$ m away from the solid phase from 0.1 GHz to 0.7 GHz. As a result, this temperature increase also increases the effective value of the dielectric permittivity of adsorbed water. Or and Wraith (1999) recognized the frequency limit of 1 GHz of the TDR method as the cut-off frequency, f^* , that distinguishes water molecules between free and bound. Water layers having dielectric relaxation frequencies lower than the cut-off frequency were considered as bound water having smaller dielectric permittivity than for free water. It was shown (Dirksen & Dasberg, 1993) that the quantity of bound water increases with the volume of clay fraction in the soil due to large specific surface area of clay.

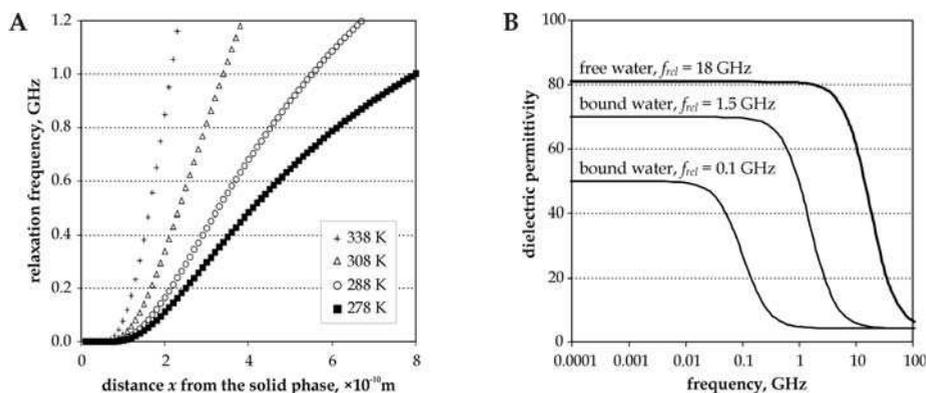


Fig. 2. A - relaxation frequency for water particles bound to solids according to (8) and related to its distance x from the solid phase; B - example relation between dielectric permittivity of water molecules and relaxation frequency for bound and free water particles

3. Materials and methods

TDR measurements were performed in the laboratory on mineral soils with negligible organic content, collected from Lublin region, Poland (Table 1) mainly from the topsoil layer. The soils were spread in layers of about 1 cm thickness on a flat surface in laboratory for drying at room temperature and then they were grinded to destroy big aggregates and passed through 2 mm sieve.

Dry soils were mixed with appropriate amount of distilled water to obtain five soil samples for each analyzed soil, with moisture values from air dry to saturation with regular differences in moisture content, taking care to get homogeneous distribution of water in the soil samples. Eight containers with soil samples covered with plastic foil to minimize evaporation were placed in a specially constructed rack. The volume of the soil containers was 0.5 dm^2 and their shape assured that the TDR probes sphere of influence was in the measured soil samples. The gravimetric moisture content, θ_g , and bulk density, ρ , were determined for each soil sample directly after completion of the TDR measurements. The values of ρ in Table 1 are the mean for all applied moistures for each tested soil. Soil texture was determined by standard Bouyoucos method (Pansu & Gautheyrou, 2006). The values of soil specific surface area, S , were measured by water vapor adsorption method (Oscik, 1983).

Soil No.	Soil type	Specific surface S $\times 10^3 \text{ m}^2\text{kg}^{-1}$	Bulk density ρ $\times 10^3 \text{ kgm}^{-3}$	ISSS soil texture		
				sand 2-0.02 mm	silt 0.02-0.002 mm	clay < 0.002 mm
611	brown soil	9	1.59	94	5	1
566	rendsina	10	1.45	92	7	1
589	soil lessive	10	1.70	88	11	1
605	brown soil	10	1.69	95	4	1
597	soil lessive	11	1.53	94	5	1
604	brown soil	12	1.65	97	2	1
593	brown soil	19	1.35	77	20	3
560	rendsina	20	1.46	87	11	2
569	brown soil	21	1.33	73	23	4
606	muck soil	23	1.42	97	2	1
591	brown soil	25	1.40	70	27	3
601	chernozem	31	1.38	64	31	5
568	brown soil	34	1.35	60	30	10
570	brown soil	35	1.40	63	26	11
623	chernozem	36	1.39	62	25	13
622	chernozem	37	1.40	61	33	6
621	chernozem	42	1.33	60	34	6
562	rendsina	65	1.44	52	35	13
619	chernozem	69	1.16	87	12	1
565	rendsina	83	1.04	77	18	5

Table 1. Selected physical parameters of the tested soils

The applied TDR probes had two parallel metal rods of 10 cm length and they were enhanced with the electronics (microcontroller, digital output temperature sensor, analog-to-digital converter and serial interface) for independent measurement of the probe temperature and soil electrical conductivity. The construction of such a "smart sensor" is presented in Fig. 3.

The electrical conductivity of the soil sample, EC_b , is measured from the voltage drop on the reference resistor R_1 connected in series with the soil equivalent resistor R_2 . Low frequency conductivity, EC_b , of the soil samples were determined from the formula: $EC_b = C/R_2$, where C is a calibration constant determined individually for each TDR probe by the measurement in NaCl solution of known conductivities.

The source voltage for electrical conductivity measurement was a square wave, generated by the microcontroller, of 100 kHz frequency that does not polarize the electrode-soil system, the inductance L is for separation of high frequency TDR signal from much lower frequencies.

Reflectometric measurements for determination of the change of soil samples bulk dielectric permittivity with temperature, moisture and electrical conductivity were performed using the setup that is shortly described below as well as in Fig. 4 and Fig. 5.

It consists of three functional modules controlled by a program running on a PC compatible computer: (i) oscilloscope frame HP54120B with the TDR test set HP54121A, (ii) self-designed and manufactured interface connecting TDR probes (two wire waveguide, 10 cm long and spaced 1.5 cm) to the TDR unit by eight position 0.01 - 2.4 GHz multiplexer and reading selected temperature sensors and controlling the temperature chamber, (iii)

temperature chamber consisting of a freezer, a fan-heater inside it and an additional fan to minimize temperature gradients inside the chamber.

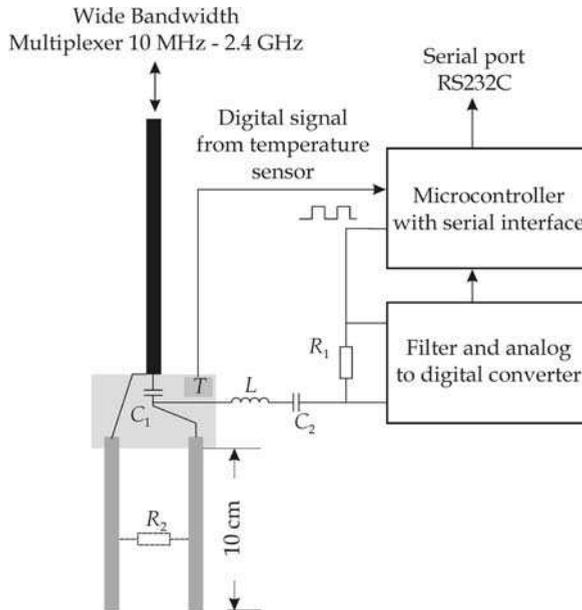


Fig. 3. Block diagram of the TDR probe with the electronics for soil electrical conductivity and temperature measurements

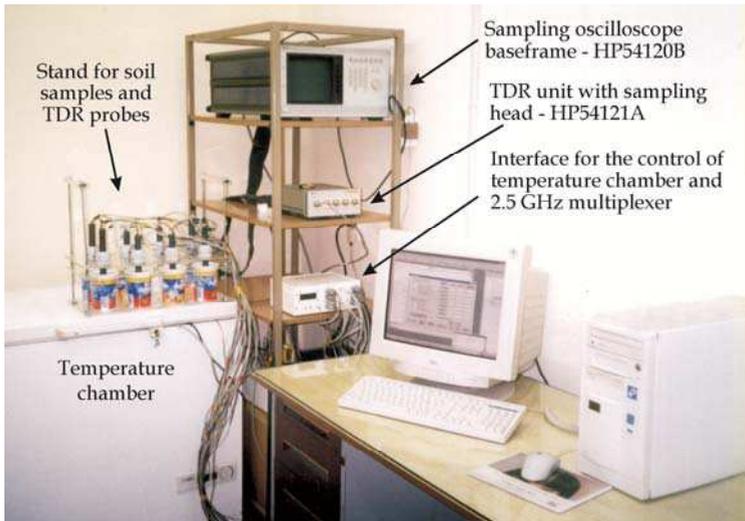


Fig. 4. Laboratory setup for the determination of the temperature variability of the soil dielectric permittivity

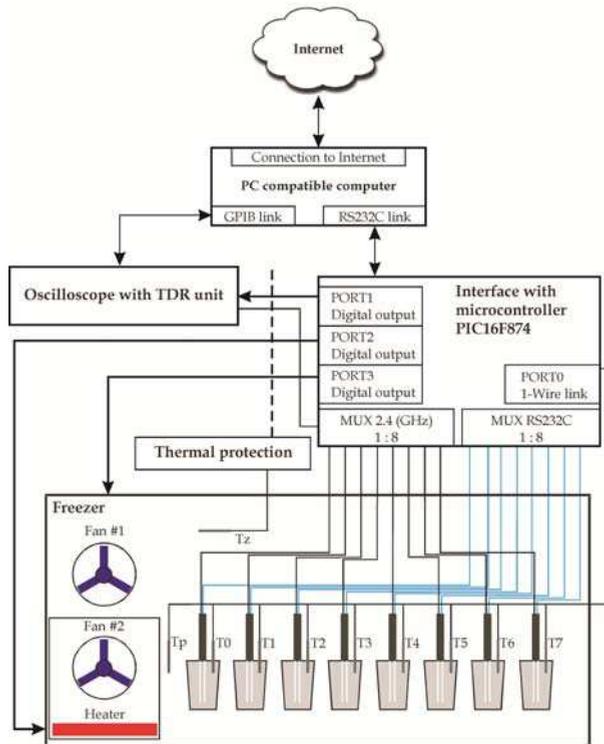


Fig. 5. Block diagram of the laboratory setup for the determination of temperature effect on soil dielectric permittivity

PC compatible computer controlling the dedicated interface by means of the serial RS232C interface and the oscilloscope by universal GPIB link, was connected to Internet to monitor remotely the performance of the experiment. The application software provides a user friendly interface for the operator. Temperature of the soil samples was controlled by switching on and off the fan-heater located at the bottom of the temperature chamber and the freezer, only one working at a time. For security reasons, an independent temperature sensor connected to the main security switch was applied to disconnect all power devices in the system in case of reaching upper limit temperature, $T_z=65^\circ\text{C}$, inside the chamber.

The complete measurement cycle at six temperature values, from 5°C to 55°C in 10°C ($\pm 1^\circ\text{C}$) increments, for the set of eight soil samples took about 12 hours. Starting from the 4th cycle, the measurable decrease of TDR moisture values of soil samples was noticed, which was attributed to the loss of water evaporating from the not perfectly sealed holes of plastic foil where the TDR probe rods entered the soil samples.

The collected data in ASCII format representing reflectograms were processed by proprietary software to calculate travel times along the TDR probe rods, and soil bulk dielectric permittivity, ϵ_{br} , according to the "flat tangent" approach (Fig. 6) described by Wraith & Or (1999).

The bound water volume fraction of the soil θ_{bw} is the product of solid phase specific surface S , temperature dependent thickness of bound water layer $x(T)$ and bulk density ρ :

$$\theta_{bw}(T) = x(T)S\rho \quad (10)$$

and $x(T)$ can be presented after Or & Wraith (1999) from reorganizing (8) as:

$$x(T) = \frac{a}{-d + T \ln\left(\frac{kT}{8\pi^2 r^3 c f^*}\right)} \quad (11)$$

where $f^* = 1$ GHz is the cut-off frequency, that distinguishes water molecules between free and bound. Having determined the dependence of bound water volume fraction of soil, $\theta_{bw}(T)$, on temperature using (10), it was possible to find the overall temperature dependence of bulk soil dielectric permittivity, ε_b , by application of dielectric mixing models. Among many dielectric mixing models describing soil as the mixture of solids, liquid, gas and also bound water phase, there are two applied in the presented study: alpha model (Birchak et al., 1974) and the model of de Loor (1990), given by the (12) and (13), respectively:

$$\varepsilon_b^\alpha = (1-\phi)\varepsilon_s^\alpha + (\phi-\theta)\varepsilon_a^\alpha + (\theta-\theta_{bw})\varepsilon_{fw}^\alpha + \theta_{bw}\varepsilon_{bw}^\alpha \quad (12)$$

$$\varepsilon_b = \frac{3\varepsilon_s + 2\theta_{fw}(\varepsilon_{fw} - \varepsilon_s) + 2\theta_{bw}(\varepsilon_{bw} - \varepsilon_s) + 2\theta_a(\varepsilon_a - \varepsilon_s)}{3 + \theta_{fw}\left(\frac{\varepsilon_s}{\varepsilon_{fw}} - 1\right) + \theta_{bw}\left(\frac{\varepsilon_s}{\varepsilon_{bw}} - 1\right) + \theta_a\left(\frac{\varepsilon_s}{\varepsilon_a} - 1\right)} \quad (13)$$

where: ε_b – bulk dielectric permittivity determined by TDR method; ϕ – soil porosity (m^3m^{-3}); ε_s , ε_a , ε_{fw} and ε_{bw} are dielectric permittivity of dry soil, air, free water and bound water, respectively; θ , θ_{bw} and θ_a (m^3m^{-3}) are volume fractions of water in the soil (free and bound water), only bound water, and air, respectively.

To perform analysis of the models (12) and (13) the following values were assumed:

$\varepsilon_{bw} = 3.2$ (the same as for ice), $\varepsilon_s = 5$ as used by Or & Wraith (1999), $\varepsilon_a = 1$.

4. Results and discussion

The experimental setup described earlier produced reflectograms like in Fig. 6 – upper curves, which after processing by dedicated software were converted to data pairs (ε_b, T) representing bulk dielectric permittivity and temperature of respective soil samples. Lower curve in Fig. 6 is produced by analysing software application software and it represents one differentiated curve, which local maxima localize inclination points in reflectograms for application the “flat tangent” algorithm.

All tested soils were divided into three groups with the soil specific surface areas below $12 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$, between 20 and $35 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$ and above $37 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$. The calibration curves taken for the tested soils are trend lines of 2nd degree polynomials fitted to the data pairs (θ , $\varepsilon_b(T)$) collected experimentally at six applied temperature values. The first group of soils had TDR calibration curves close to the equation given by Topp et al. (1980).

With the increase of the soil specific surface area, S , as well as the decrease of bulk density, ρ , the bulk dielectric permittivity values, ϵ_b , were below the Topp's calibration. This is in agreement with other reports (Dirksen & Dasberg, 1983) showing the influence of S and ρ on the calibration of the TDR method for soil moisture determination. The biggest temperature effect on ϵ_b was observed for the soil 569 at its highest moisture content; with the temperature increase of 50°C its value decreased by 3.49. This temperature change of the TDR determined bulk dielectric permittivity corresponds to a decrease of the calculated soil moisture of 0.037 m³m⁻³, using the calibration from Topp et al. (1980).

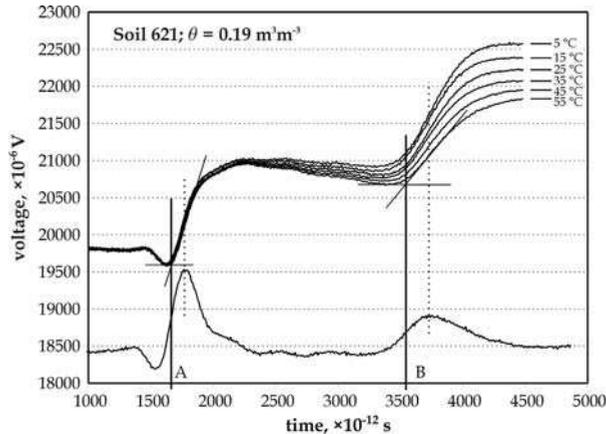


Fig. 6. Set of reflectograms registered by the measurement setup for the same soil sample in different temperatures

The calibration curves for each soil taken for different soil temperatures meet at a characteristic moisture content value θ_{eq} , where the physical phenomena responsible for the temperature effect of soil dielectric permittivity equalize (Fig. 7). This moisture content is named the "equilibrium water content" in this study. For water contents below θ_{eq} , soil bulk dielectric permittivity ϵ_b measured at 5°C is smaller than the one measured at 55°C and for water contents above θ_{eq} the change of ϵ_b with temperature is opposite. The observed temperature effect of soil dielectric permittivity confirms the theory of Or & Wraith (1999), which explains it by the temperature caused exchange of water particles between free and bound phases.

The value of ϵ_b for air-dry soils does not depend on temperature. With the increase of soil moisture from air dry, there is an increase of ϵ_b with temperature. All soils except one (soil 562) have higher values of ϵ_b for 5°C than 55°C at high water contents, and the biggest difference is observed for the soils having medium values of S (Table 1). For the soil 562, there was no equilibrium water content observed in the analysed temperature range, although this soil does not have the highest value of specific surface area from all the tested soils. The bulk dielectric permittivity for this soil is higher at 55°C than for 5°C in the whole range of moisture from air dry state to almost saturation. The soil bulk electrical conductivity in low frequency range, σ_{dc} , for the soil 562 and for example the soil 565 at different temperatures and high moisture was compared.

Bulk electrical conductivity for the soil 562 ranged from 47 mS m⁻¹ at 5°C to 192 mS m⁻¹ at 55°C for $\theta = 0.3$ m³m⁻³, while for the soil 565 the respective values were even bigger, i.e. they

ranged from 50 mSm⁻¹ at 5°C to 221 mSm⁻¹ at 55°C for $\theta = 0.4 \text{ m}^3\text{m}^{-3}$, and still the temperature behaviour of the soil 565 dielectric permittivity was typical. This confirms the literature reports (Nadler et al., 1999; Or & Wraith, 1999) that the increase of soil bulk electric conductivity does not increase the TDR readout of ϵ_b in the presented variability range of σ_{dc} . Further studies are needed to diagnose the temperature effect of the soil 562.

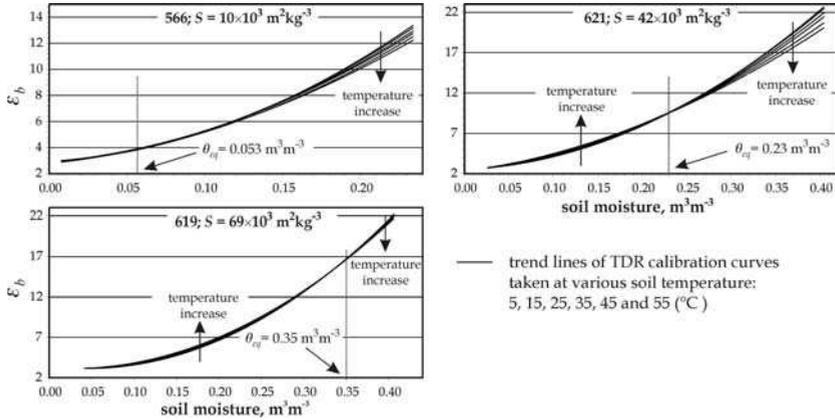


Fig. 7. TDR calibration curves for three tested soils at different temperatures, S is the soil specific surface area, θ_{eq} is the equilibrium moisture where the temperature effect caused by the described two competing physical phenomena is compensated

The value of soil moisture at the equilibrium point, θ_{eq} , depends on the amount of bound water attracted by the soil, which is described by equation (10). The relation between the equilibrium water content, θ_{eq} , and the specific surface for the tested 19 soils, except the soil 562, is presented in Fig. 8.

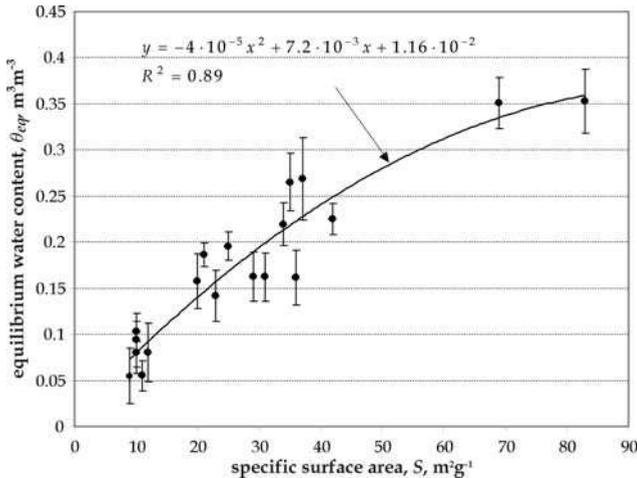


Fig. 8. Empirical relation between the equilibrium water content, θ_{eq} , and soil specific surface, S , for the tested soils

The good correlation between the soil specific surface S and θ_{eq} confirms the assumed physical description of the processes involving the temperature effect of soil dielectric permittivity.

The temperature dependence of the soil bulk dielectric permittivity, ϵ_b , of the selected soils is presented in Fig. 9. The majority of tested soils show similar trends that confirm other experimental data (Pepin et al., 1995; Or & Wraith, 1999). For small and medium moisture values there is a negligible temperature effect and the linear trend lines in Fig. 9 are almost in parallel to the horizontal axis representing no or a small positive temperature change.

This is especially evident for the two trend lines representing the lowest moisture values in $\epsilon_b(T)$ relations for all soils in Fig. 9. For higher moisture values there is a tendency to decrease ϵ_b with temperature proving that the dominant reason for this behaviour is the decrease of free water dielectric permittivity with the temperature increase. However, as expected for the soils with large specific surface area this tendency is much smaller or has the opposite direction, as for the soil no. 562.

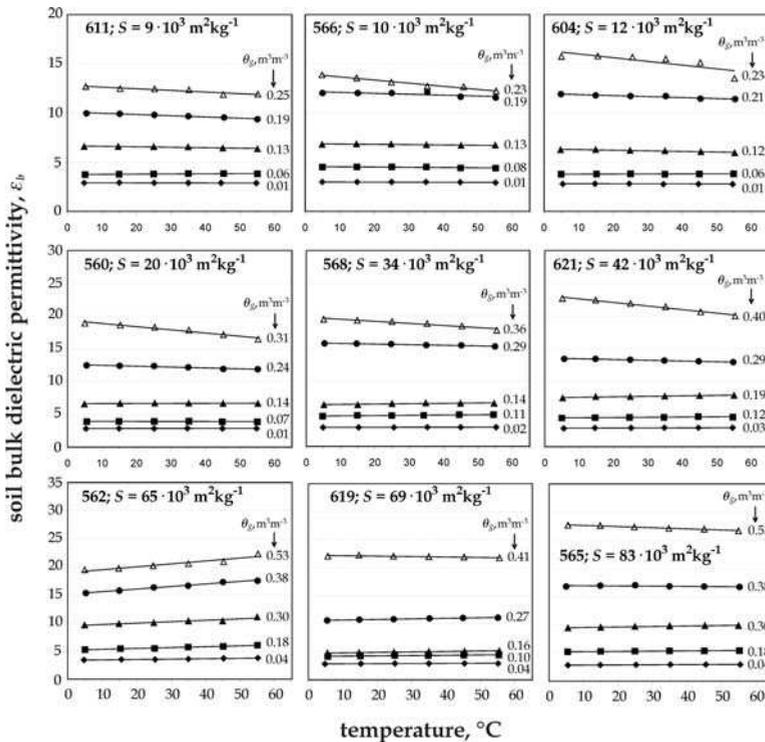


Fig. 9. Bulk dielectric permittivity temperature dependence of selected soils bulk dielectric permittivity for different soil volumetric water contents (θ_g stands for thermo-gravimetrically determined soil water content)

Other soils from the same group as the soil no. 562, i.e. no. 619 and no. 565 having larger values of soil specific surface area, $69 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$ and $83 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$ respectively, show typical temperature effect of soil dielectric permittivity, i.e. for moisture below θ_{eq} , ϵ_b

increases with increasing temperature, and for moisture above θ_{eq} , ϵ_b decreases with increasing temperature. For all tested soils, except the soil no. 562, the slope was small and positive for low and negative for high soil moisture.

The applied models: 4-phase alpha and de Looer models do not follow the measured data $\epsilon_b(T)$ for all soils and for all moisture values. The examples of the performance of these models are presented in Fig. 10, for the soil no. 562 ($S = 65 \cdot 10^3 \text{ m}^2\text{kg}^{-1}$).

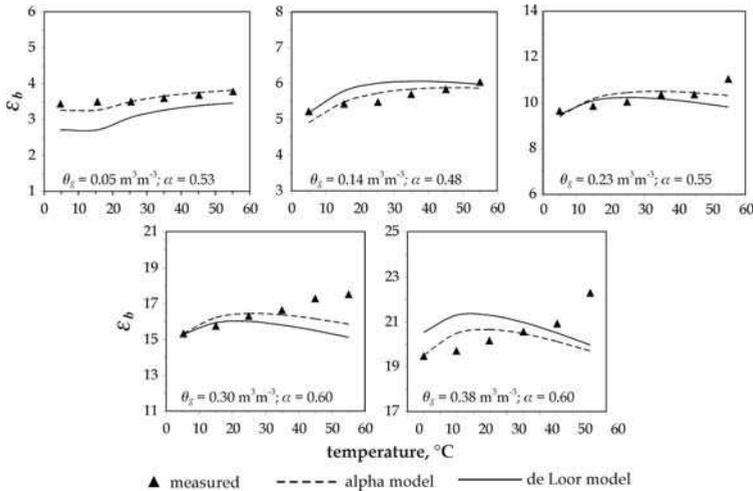


Fig. 10. Performance of applied 4-phase soil dielectric mixing models for the soil no. 562. The assumed values for dielectric permittivity for soil solid phase $\epsilon_s=5$ and bound water $\epsilon_{bw}=3.2$

The alpha model has a fitting parameter representing the geometry of modelled medium (Roth et al., 1990) and enabling to adjust model data to the measured ones, as it was done in Fig. 10. The values of ϵ_b from the model data are generally higher than the TDR determined from the measurement. Therefore the applied correction of the temperature effect on the soil dielectric permittivity is based on empirical data and it accounts for the observed property of θ_{eq} . The applied correction turns the slope of the linear trend lines of the measured values of $\epsilon_b(T)$ to zero. The turning centre is the soil temperature of 25°C (Fig. 9).

For each soil sample the temperature dependent value of soil moisture $\theta_{TDR}(T)$ was determined basing on the individual TDR soil calibration function giving the bulk dielectric permittivity values at different temperatures. Temperature corrected values of volumetric water content θ_{TDR}^{corr} , were determined from the relationship between $\theta_{TDR}(T)$ and θ_{eq} , as follows:

$$\text{For } \theta_{TDR}(T) \leq \theta_{eq} : \theta_{TDR}^{corr} = \theta_{TDR}(T) \cdot \left[1 - A \cdot (\theta_{eq} - \theta_{TDR}(T)) \cdot (T - 25) \right] \tag{14}$$

$$\text{For } \theta_{TDR}(T) > \theta_{eq} : \theta_{TDR}^{corr} = \theta_{eq} + \frac{\theta_{TDR}(T) - \theta_{eq}}{1 + n_{25} \cdot B \cdot [d(T) - 1]} \cdot \frac{\partial \theta_{TDR}}{\partial n} \tag{15}$$

where: $n = (\epsilon_b)^{1/2}$ is the soil refractive index, n_{25} is its value at 25°C, $\partial\theta_{TDR}/\partial n$ is the slope of TDR calibration, for example the one taken from Topp et al. (1980) is 0.127.

Formula (14) refers to TDR determined soil moisture values not exceeding equilibrium water content, θ_{eq} , for which the temperature effect of the soil bulk dielectric permittivity increases its value, which is due to release of water molecules adsorbed by the surface of the soil solid phase. Formula (15) applies TDR determined soil moisture values exceeding equilibrium water content, θ_{eq} , when the temperature effect of the soil bulk dielectric permittivity is mainly from the temperature effect on free water, resulting in declining soil bulk dielectric permittivity with increasing temperature.

Parameters $A = 0.008$ and $B = 0.55$ are empirically adjusted to minimize the slope $\Delta\epsilon_b/\Delta T$ for all examined soils. Because of good correlation between soil specific surface, S , and θ_{eq} (Fig. 8), the former can be applied from the fitted line.

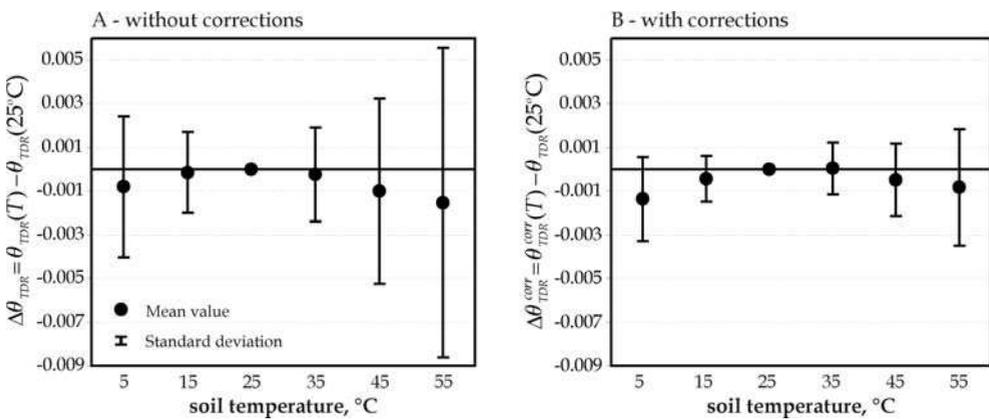


Fig. 11. Errors of TDR determined soil moisture at various temperature values referenced to the respective values at 25°C: A - without temperature corrections, B - with temperature corrections based on (14) and (15)

The comparison of mean values and standard deviations of absolute errors for measured data, $\Delta\theta_{TDR}$, and temperature corrected data, $\Delta\theta_{TDR}^{corr}$, according to the temperature corrections (14) and (15) is presented in Fig. 11. The mean values for the both errors are calculated for 20 tested soils at six values of temperature from 5°C to 55°C with 10°C steps between.

The mean values of TDR determined soil moistures at tested soil temperatures are below the respective values at 25°C. The absolute measurement error of soil moisture measured by TDR, defined as double the standard deviation from the mean value does not exceed 1.5% of measured value. After applying the empirical correction given by (14) and (15) the absolute measurement error decreased almost three times to the value not exceeding 0.54%.

5. Summary

The temperature effect of the examined mineral soils' bulk dielectric permittivity, ϵ_b , determined by TDR method, confirms the theory presented by Or and Wraith (1999), describing it as the result of two competing phenomena; ϵ_b increases with temperature

increase following the release of bound water from soil solid particles and ϵ_b decreases with temperature increase following the temperature effect of free water molecules.

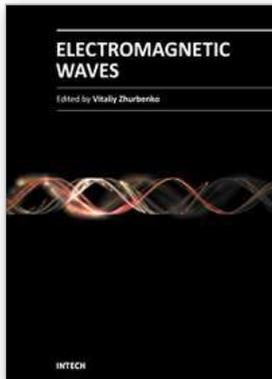
It has been found that there is a soil type characteristic moisture value, θ_{eq} , named the equilibrium water content, having the specific temperature property. The temperature effect for this soil water content value is not present, which means that at θ_{eq} the both competing phenomena mentioned earlier compensate each other. The equilibrium water content, θ_{eq} , is correlated with the soil specific surface area. For soils with water content below θ_{eq} , the temperature effect of soil dielectric permittivity is positive, i.e. it increases with temperature, and for soils with water content above θ_{eq} , the temperature effect of soil dielectric permittivity is negative, i.e. it decreases with temperature.

The temperature correction formula adjusting the soil moisture determined by TDR at various temperature values to the corresponding value at 25°C, based on knowledge of θ_{eq} , decreases the standard deviation almost three times as compared to uncorrected values of θ_{TDR} . The electrical conductivity of the examined soils does not show any influence on the observed temperature effect of soil dielectric permittivity.

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