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Calibration of a New Device to Measure Water Content of Rocks

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1. Introduction

The vadose zone, which extends from the soil–atmospheric interface to the capillary fringe of the water table, is a fundamental part of the hydrologic cycle. It controls how the precipitation splits into infiltration, surface runoff, evapotranspiration, groundwater recharge; it regulates the storage, transfer, filtering, adsorption, retard and attenuation of solutes and contaminants before these reach the groundwater.

Until the last decades, the vadose zone was considered as a black box that merely connect surface water and groundwater. As consequence an incomplete understanding of the complex dynamics of the vadose zone existed.

Nowadays, instead, the monitoring of the hydrological processes that occur through the vadose zone, are receiving increased attention mainly with refer to contaminant transport processes. Fluxes of water and solutes strongly depend on water content thus its monitoring and estimation becomes an important issue.

A wide range of methods, sensors and technologies are available for the measure of soil water content, mainly used in management of precision farming.

The traditional standard method for direct measurement of soil water content is the gravimetric method [1]. This method implies the sampling of the soil to carry out laboratory measurements, modifying the natural condition. As gravimetric method is destructive, labour intensive, not timely and costly, many alternative non-destructive methods for measuring water content have been developed. However, none of all methods measure the water content directly but only some properties, named "surrogate measure", that changes as the soil water content changes. By measuring the value of the surrogate parameter, it is possible to estimated

the value of water content, using a calibration curve that represents the relationship between the surrogate measure and the soil water content values [2].

Neutron Thermalization method [3, 4], Capacitance method [5-8], Time Domain Reflectometry (TDR) method [9-13], Frequency Domain Reflectometry (FDR) method [14, 15], Impedance method [16, 17], Electrical Restistance method [18, 19] and Tensiometer method [20-22] are among the well-known technologies utilized to develop different kind of devices and probes for measuring moisture content and soil water potential, respectively.

More details on these technologies and devices, advantages and limitations of each one, are given in several references [23, 24, 2].

Recently, remote sensing technologies, like passive or active radiometry, have been applied in order to provide wide-area indications of surface soil water content [25, 26]. However, the great influence of vegetation and surface structure on the quality of received signal, restricts the sampling depth to the uppermost part (2–5 cm) of soil [27], strongly limiting the usefulness of this technology in hydrological application.

The use of all these methods and devices in the soil is customary by now but when the vadose zone consists of rocks the monitoring of water content become more difficult for several aspects. The main difficulty regards the installation of the probes that are often very delicate and cannot be hammered or screwed into the rock. Furthermore, after their insertion, a good contact between the rock and the sensor must be ensured in order to minimize the gap effect which causes significant errors in the investigated properties, especially for dielectric ones [28, 29].

At present, very few applications of these techniques to rocks, by creating new devices or by adapting the existing ones, are documented in literature.

The first measurements in sandstone and tuff blocks by means of penetration type probes using TDR are reported in [30]. Topp's equation [9] was used to convert the dielectric constant (K) values into water content (θ) highlighting an overestimation of water content. In fact, especially for rocks with low effective porosity, an individual calibration was needed.

In [31] TDR was applied in granodiorite both in the laboratory and in the field. Employing surface probes, it was showed that K and θ were almost linearly related, revealing that TDR was capable to measure the volumetric water content changes.

Using brass rods, individual K - θ calibration functions were developed in [32] for nine different rock types with porosity values ranging from 1 to 54%. However, the experimentally determined K - θ relationships showed an abrupt jump near fully saturation of samples, suggesting that TDR would be accurate for applications in rocks only when the gap effect is negligible.

By applying TDR in seven different types of rocks [33], the authors demonstrated that conductive silicone fillings, carefully applied, can successfully eliminate gap effect in the case of penetration probes. Moreover, they compared the K-θ relationships obtained with surface probes against that obtained using penetration probes. The results showed

systematic differences between two types of probes, explained on the basis of their geometry.

In this chapter, we intended primarily to give further contribute to the knowledge on the applicability on rocks of methods developed for measuring water content in the soils by focusing on the Electrical Impedance Spectrometry (EIS) method. In particular, this work presents the results obtained to calibrate a new device, called Z-meter 2, based on EIS method. Samples of calcarenite have been used for the calibration procedure carried out in laboratory under controlled condition.

The electrical impedance in complex form and its changes over time have been measured with the aim to:

a. verify the suitability of the device for water content estimation in rocks;

b. characterize the effects on the measured values of the electric frequency applied and of the electrical conductivity (EC) of the solution used to saturate samples;

c. determine specific calibration curves for the investigated lithotype.

2. Materials and methods

2.1. Electrical Impedance Spectrometry (EIS) method

The basic principle of the EIS method [34] is the measurement of the electrical impedance $(\hat{Z})$ which provides a measure of medium's opposition during the passage of a alternating current (AC). The impedance is described not only by the amplitudes of the voltage and the current, but also by their relative phase-displaced.

The impedance of a circuit element can be defined as the frequency domain ratio of the voltage phasor $\hat{U}$ to the current phasor $\hat{I}$ across the element:

$$\hat{Z} = \frac{\hat{U}}{\hat{I}} \tag{1}$$

Impedance is represented as a complex quantity. In the polar form, it is

$$\hat{Z} = |Z| \, e^{j\theta} \tag{2}$$

where $|Z|$ is the magnitude, the argument θ is the phase difference between voltage and current and j is the imaginary unit.

In cartesian form, it is

$$\hat{Z} = R + jX \tag{3}$$

where R is the resistance, that forms the real part of impedance, independent from the frequency, and X is the reactance, that forms the imaginary part of impedance, dependent from the frequency. The values of impedance are expressed, as those of resistance and reactance, in ohms [Ω].

The impedance depends on the characteristics of AC electric circuit which can be made by combination of resistor, inductor and capacitor, either in series or parallel mode.

For a real resistor-capacitor (RC) circuit and resistor-inductor (RL) circuit, the impedance can be expressed by the following relations

$$\hat{Z}_L = R + j \cdot X_L = R_L + j\omega L \tag{4}$$

and

$$\hat{Z}_C = R - j \cdot X_C = R_C - \frac{j}{\omega C} \tag{5}$$

where

R = resistance

$X_L = 2 \cdot \pi \cdot f \cdot L$ = inductive reactance

$X_C = -\dfrac{1}{2 \cdot \pi \cdot f \cdot C}$ = capacitive reactance

f = frequency (Hz)

$\omega = 2\pi f$ = angular frequency

C = capacitance or electrical capacity (F)

L = inductance (H)

The porous medium (e.g. soil and rock) can be described by the impedance of an equivalent electric circuit. It always has the character of a resistor, so that the real component of the total impedance $\hat{Z}$ of the circuit is always present.

In a dry porous medium the resistance is so high that it can be considered as a non-conductor (dielectric). Therefore it is possible to assume that dry porous medium is polarized by the electric field and thus it is charged as a capacitor characterized by its capacitance.

Using this assumption, the capacitance of medium is substantially higher than the inductance so it can be neglected and the dominant component of imaginary part of the impedance $\hat{Z}$ is the capacitive reactance (X_C).

As for mentioned above, the porous medium can be described as an equivalent RC circuit and its impedance, expressed by the equation (5), describes primarily electromagnetic properties of medium.

The solid part (grains) is formed by insulating materials, characterized by their dielectric constants, and hence it represents the imaginary part (X_C) of the measured impedance. If the material is in non-zero moisture condition, it can be considered as a conductor because water always contains a certain quantity of mineral salts (conductive material). In this case the resistance is lower and the degree of saturation of the material strongly influences the real part of the measured impedance.

2.2. Z-meter 2 device

The Z-meter 2 is a new device developed, constructed and realized within the European International Project E!3838 of EUREKA Program [35]. It's based on EIS method and it is able to measure the electrical impedance in complex form. Z-meter 2 was specifically designed to carry out long-term field experiments.

According to the equivalent diagram of the circuit in Figure 1, the resulting measured value of impedance is represented by a parallel combination of resistor and capacitor.
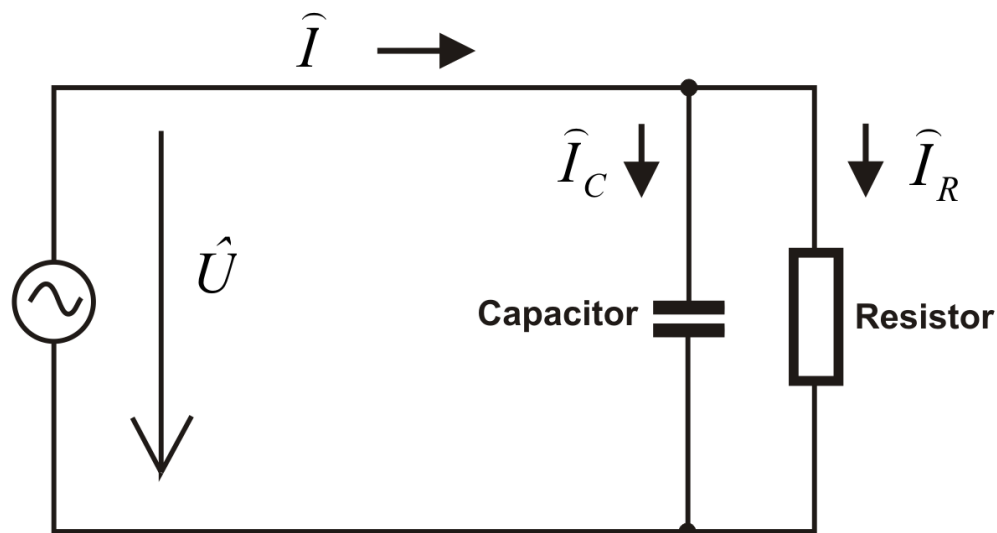
Figure 1. Equivalent diagram of measured impedance.

The electric current is divided into two orthogonal components, real $\hat{I}_R$ flowing through the real component of the equivalent resistor, and imaginary $\hat{I}_C$ flowing through the equivalent capacitor. In this way the Z-meter 2 allows measurement of both parts of the impedance: resistance and reactance. The basic parameters of the device are summarized in Table 1.

Many different kinds of probes can be used with Z-meter 2 for measuring the impedance of soils, either in the field or in laboratory: rod probes, modular probes with variable length constituted by stainless electrical conductive parts alternated with non conductive parts (polyamide), aluminium strip probes with a self-adhesive layer on one side that ensures perfect contact with the medium.

The kind of probe needs to be chosen in relation to the monitored medium, the environmental condition and the aim of the measurements. In this sperimentation, stainless steel road probes, called picket type probe, were used. These probes were chosen because they can be easily installed in the rock without be damaged.

PARAMETERS	Z-METER 2
Range of impedances	$10\ \Omega - 1M\ \Omega$
Frequency range	100 Hz – 20 KHz
Accuracy of modulus of impedance	+/- 2% of the range
Accuracy of phase	+/- 2°
Level of measuring voltage	500mV – 5V with an interval of 500 mV
Communication with a PC bus	USB (COM port)
Integrated electric switcher	128 measuring points, each with 4 electrodes

Table 1. Basic parameters of a Z-meter 2 device.

## 2.3. Rock type and samples preparation

The rock used for the calibration tests was a marine sedimentary rock of Plio-Pleistocene age, known as "Calcarenite di Gravina" Formation. It is a porous rock with granular skeleton consisting of irregularly rounded lithoclasts of limestones and bioclasts, fragments of marine organisms, embedded in a fine, granular, carbonatic matrix, cemented to some degree. The matrix and the sparry cement, made up predominantly of mineral calcite, are entirely subordinate to the granular skeleton. The calcarenite's composition mostly consists of calcium carbonate (>95%) with an insoluble residue characterized mainly by clay minerals. Previous laboratory tests assessed that porosity ranges between 40% and 47% and the hydraulic conductivity (K) is about $6 \div 7 \times 10^{-5}$ m/s for the coarse calcarenite and 3×10^{-5} m/s for the medium calcarenite [36-38]. The calcarenites are diffused in Apulia Region and frequently constitute part of the vadose zone or form significant porous aquifers in the region, playing an important role in drinking supply. Particularly, the samples of calcarenite used for the tests came from an active quarry, located in Canosa di Puglia, a city in the South of Italy (Figure 2).

Three blocks of calcarenite, hereafter called E1, E2 and E3, cut from one large block of rock, were used in order to verify the repeatability of measurements. The rock samples appeared relatively homogeneous at the sample scale. The samples were oven dried in order to obtain the dry weight. The dimensions of samples and their main physical properties are summarized in Table 2.

The lateral surface of the samples was sealed using a bi-component epoxy resin in order to ensure a one-dimensional water flow during the evaporation process.

The picket type probes, 10 mm diameter, 120 mm long with a conical bit 15 mm long, were used as electrodes in the tests (Figure 3). Two guide holes, 9 cm apart, with approximately 10 mm diameter, were drilled into rock samples for the installation of the probes. These ones were then slowly inserted into the sample with twisting motion and, alternatively, with the help of hammer, leaving 2 cm protrusion from the top edge of sample. In this way a good contact between the rock and the sensor was ensured.
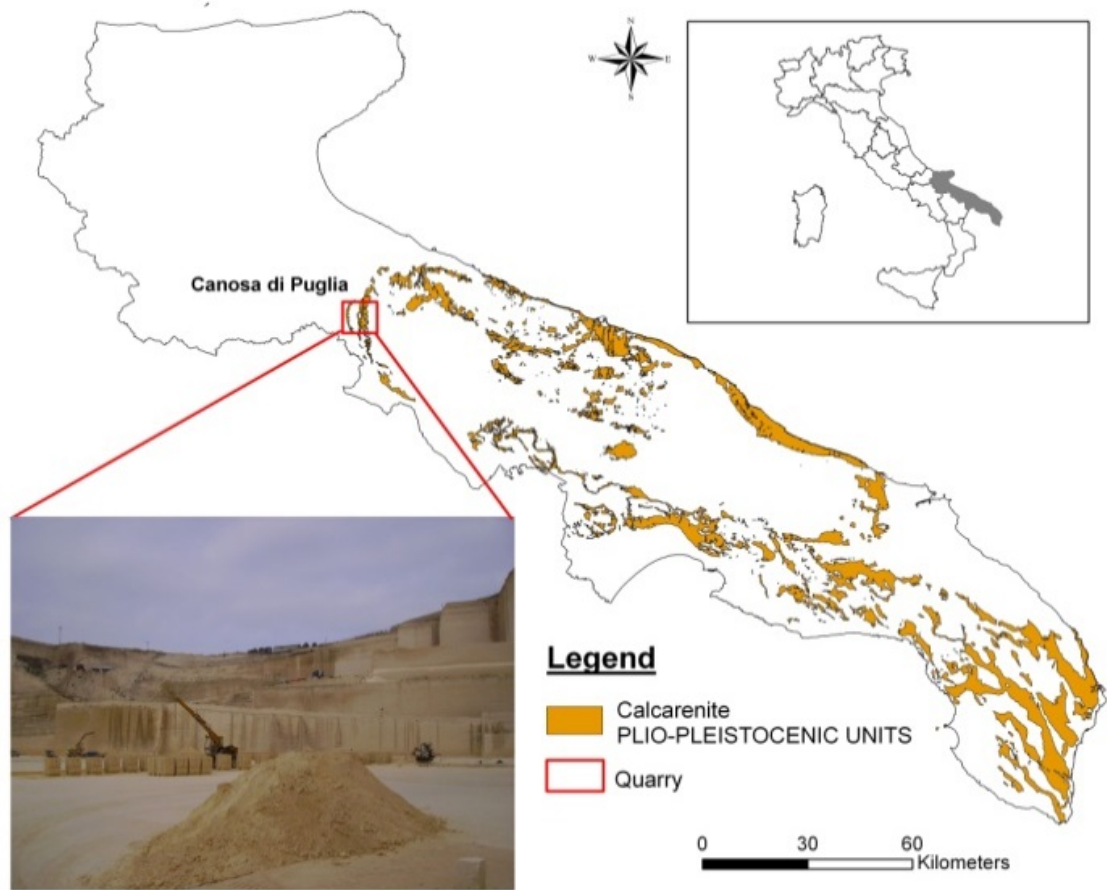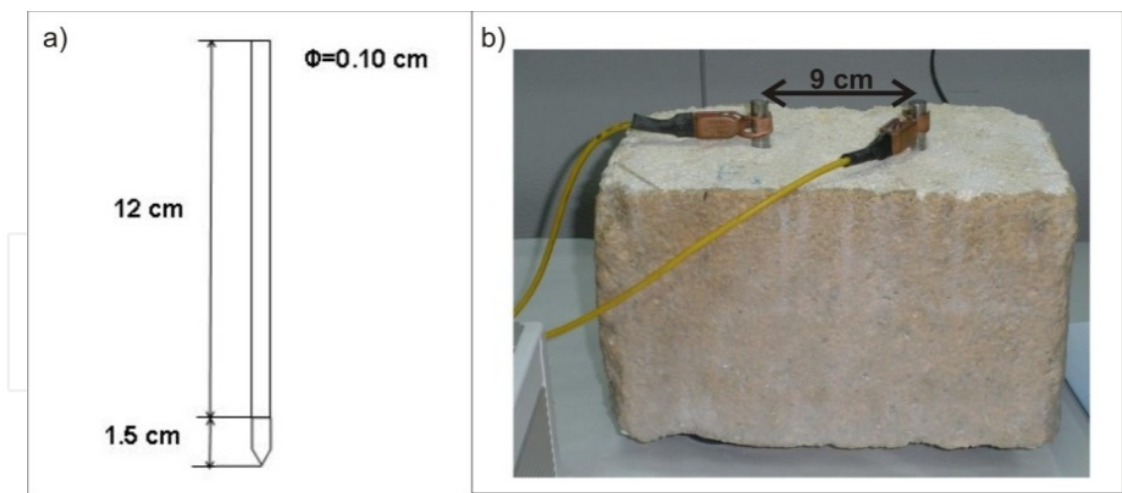

Figure 2. Regional distribution of calcarenites and location of quarry from which the rock samples have been collected.

	Sample E1	Sample E2	Sample E3
Dimensions (cm)	25x16x15	25x15x15	24.9x15x14.7
Bulk density (g/cm³)	1,525	1,515	1,500
Particle density (g/cm³)	2,71	2,71	2,71
Porosity (%)	43,73	44,11	44,66

Table 2. Physical characteristics of the calcarenite samples tested.

Figure 3. a) Scheme of picket type probe; b) probes position.

After the probes installation, the sample was placed in a water-filled vacuum bell jar, under confining pressure of 1 bar for at least 24 hours, in order to achieve almost full saturation. The saturation process took place from bottom to up by avoiding both air entrapment and uneven distribution of water. Three different solutions were used to saturate the samples in order to establish the effect of electrical conductivity (EC) on the output of the device: deionized water, 0,01 M and 0,03 M KCl solutions. The EC value as well as the KCl concentrations and the density of each water solution are reported in the Table 3.

KCl solution molarities	EC values (mS/cm)	Salt concentration g/L	Water solution density g/cm³(at 25°C)
deionized water	$3,4*10^{-3}$	0	0.9971
0.01	1,75	0,745	0.9976
0.03	4,70	2,235	0.9988

Table 3. Physical properties of water solutions used to saturate the samples.

2.4. Measuring procedures

The entire cycle of measurements, comprising the measurements for all chosen frequencies, was repeated over time: 2-3 times per day in the first 15 days, 1 time in the following days, according to the expected rate of the evaporation process.

Contemporarily the weight loss of the rock sample, due to evaporation, was measured by electronic balance. The whole experimental set up is shown in Figure 4.

The tests have been performed in a thermostatic room at temperature of 25 ±1°C provided with a ventilation system. This is because temperature variations cause errors in all indirect moisture measurement methods due to the dependence of the dielectric properties from the

temperature [39,40]. The tests were stopped when the sample's weight reached approximately it's dry weight.

Figure 4. Experimental set up.

3. Results and discussion

3.1. Volumetric Water Content (VWC)

The first stage was to obtain the VWC of samples starting from the weight recorded at different times.

The rock VWC was determined, from the gravimetric one, in a standard way by the following equation (6) [41]:

$$\theta_v = \theta_g \left(\frac{\rho_b}{\rho_w} \right) \qquad (6)$$

where

θ_v (cm^3 cm^{-3}) = VWC volumetric water content,

θ_g (g g^{-1}) = gravimetric water content,

ρ_b (g cm^{-3}) = rock bulk density,

ρ_w (g cm^{-3}) = water solution density.

The dynamics of the weight loss of the sample during the test is shown in Figure 5, in which the data for sample E2, saturated with 0.01 M KCl solution, are reported as an example.

The weight of the sample decreases over time in different way so that the evaporation from a soil or rock may be modelled as a two-stages process. In the first stage, corresponding to high water contents, the evaporation is governed by environmental conditions; in the second, when the water content decreases beyond a critical value, the evaporation is controlled by the ability of the medium to transmit water or, in other words, by its hydraulic properties [42, 43].
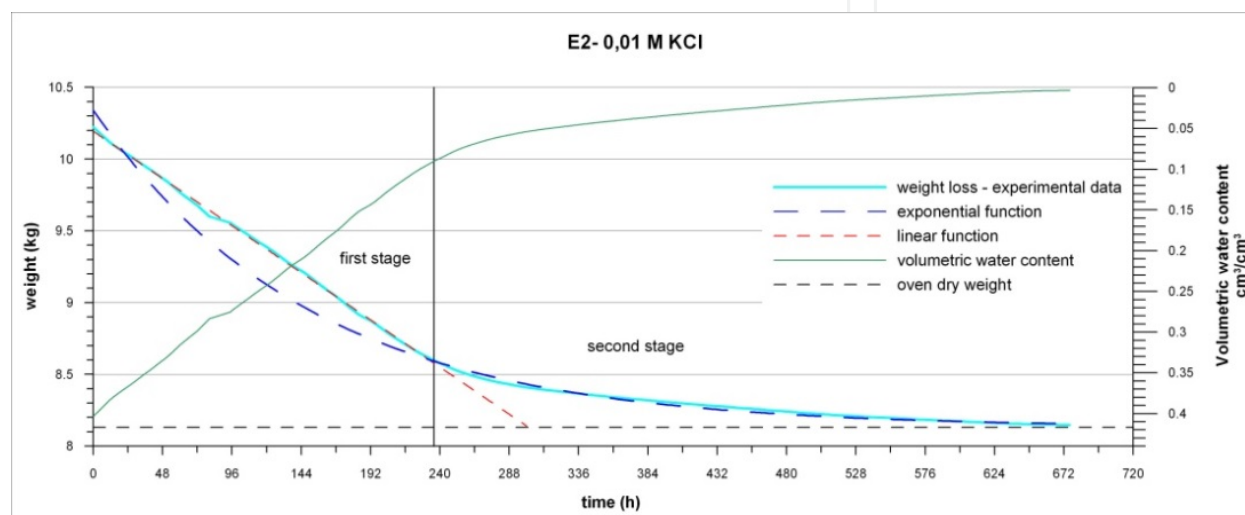
Figure 5. Changes in the weight and volumetric water content vs. time.

This different behavior is reflected in the different equations describing the process: the first part of data, corresponding to high water contents, is described by a linear relation while the second is well described using the following exponential function:

$$y = a + b\exp\left(-\frac{x}{c}\right) \tag{7}$$

where y (kg) is sample weight over time, a (kg) is its dry weight, b (kg) is the weight of water required to saturate the sample, x is the time (h) and c (h) is the only fitting parameter. The transition from the first to the second part occurs at a water content of about 6÷9%.

3.2. Resistance

The use of Z-meter 2 allows to investigate both the real (resistance) and the imaginary component (capacitive reactance) of impedance, being the two components recorded separately. In this study only the resistance was investigated because of its direct correlation with the water content.

The effective independence of the resistance (hereafter called Rx) from the frequency applied was checked and verified. Figure 6 shows the Rx output probe for all the tested measurement frequencies relative to sample E2 saturated with 0.01 M KCl solution.

The Rx values, measured for the same VWC, are very similar to each other for all tested frequencies, except that for VWC close to dry condition (less than 0.02). These means that, for VWC > 0.02, the device has a good accuracy and precision and that changing the frequencies has no effect on the Rx values that are therefore related with the VWC only. This results permits to use the complete dataset, consisting of medium Rx values recorded for each frequencies and the same VWC, in order to obtain the best fit curve, for each solution. For the reason mentioned above, all Rx data corresponding to VWC less than 0.02 are not included in the dataset.
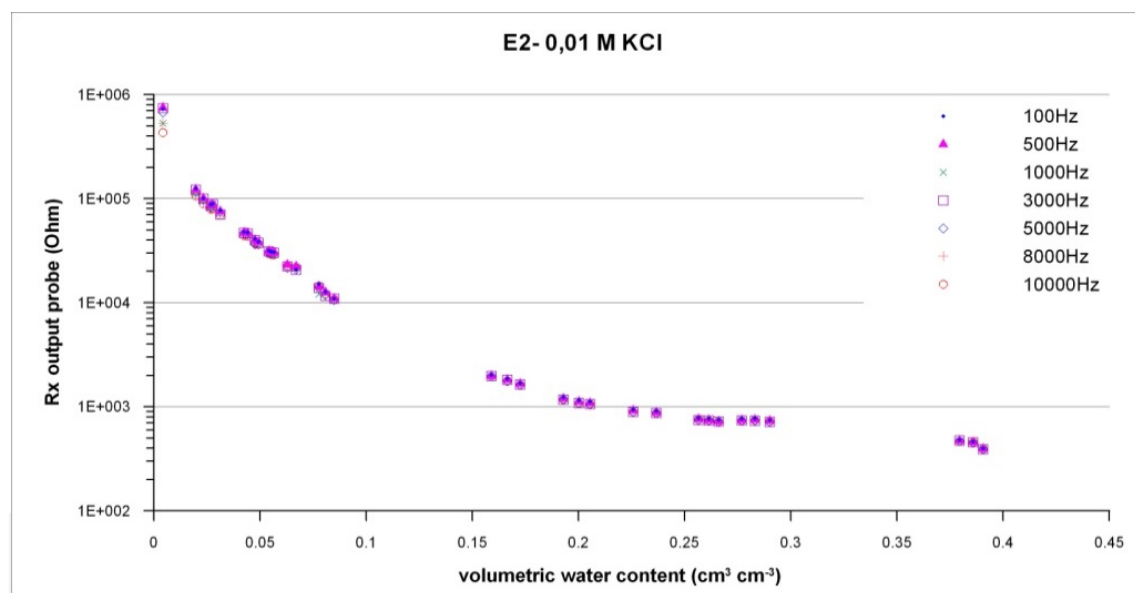
Figure 6. Rx values vs volumetric water content at all tested frequencies for sample E2.

The complete dataset of Rx values, recorded for the sample E2, are shown in Figure 7. Unlike what happens for the frequency, Rx values are strongly affected by changing the molarity, and hence EC value, of the solution used for the sample saturation. Moreover, as expected in a drying experiment, the VWC decreases over the course of the experiment and the corresponding Rx measurements increase significantly.
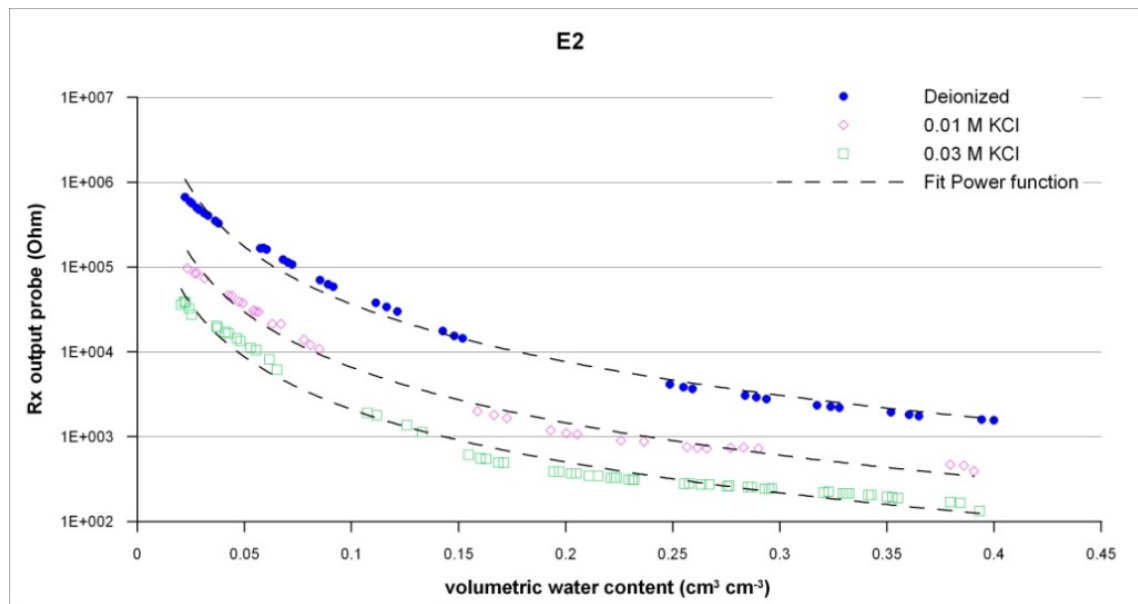
Figure 7. Rx complete dataset for the three solutions used to saturate sample E2.

The best interpolation function of dataset is represented by a power function of the type:

$$y = a \cdot x^b \qquad (8)$$

This model can be linearized by taking logarithms:

$$\ln(y) = \ln(a) + b \cdot \ln(x) \qquad (9)$$

The linearized experimental data, obtained for each solution used to saturate samples E1, E2 and E3, are shown in Figure 8.

Small differences are found in the three replicates obtained for the same solution that can be explained with the natural variability among different samples of the same rock type (see Table 2). The two replicates E1 and E2 have been used, as a unique dataset, to obtain the calibration function that describes the VWC-Rx relationships, for each solution, while the third replicate (E3) has been used for the validation test. In Figure 9, for each solution, the logarithms of calculated VWC are plotted against the logarithms of measured Rx coming from replicates E1 and E2. The regression curves are also reported in the graphs. The changes in Rx due to the changes in VWC are significant and the relationships show a negative slope. The information provided from the linear regression, as indicated by the value of R-squared coefficient, suggests good degree of correlation of the datasets to these functions.

The calibration function is $VWC = a \cdot Rx^b$, by converting the logarithmic function into power function. The values of parameters a and b, for each solution, are reported in Table 4. In order to check the performance of the calibration function obtained by regression of experimental data, a validation test was performed utilizing the data of the sample E3. The actual values of
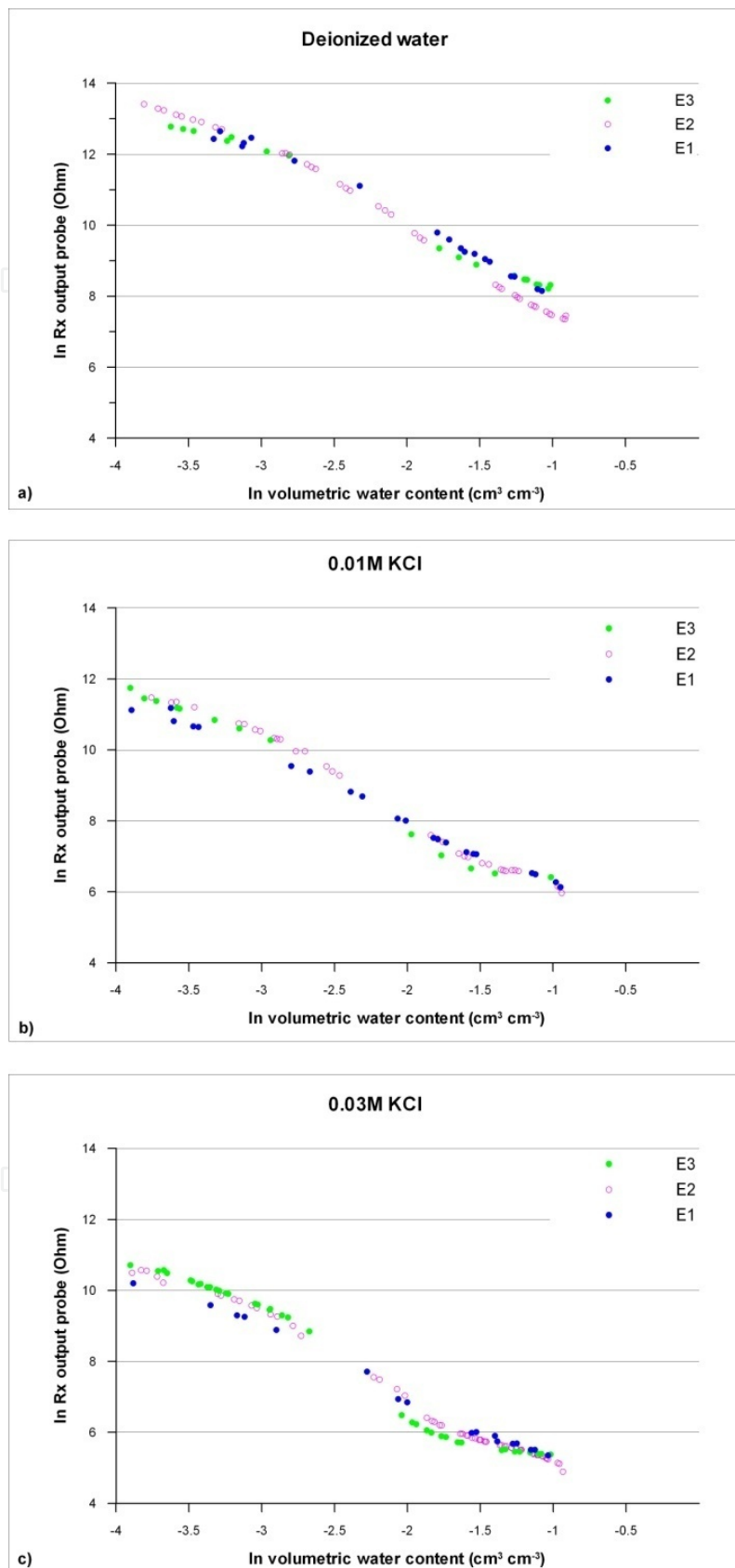
**Figure 8.** Three replicates (E1, E2, E3) for each solution (a, b, c).
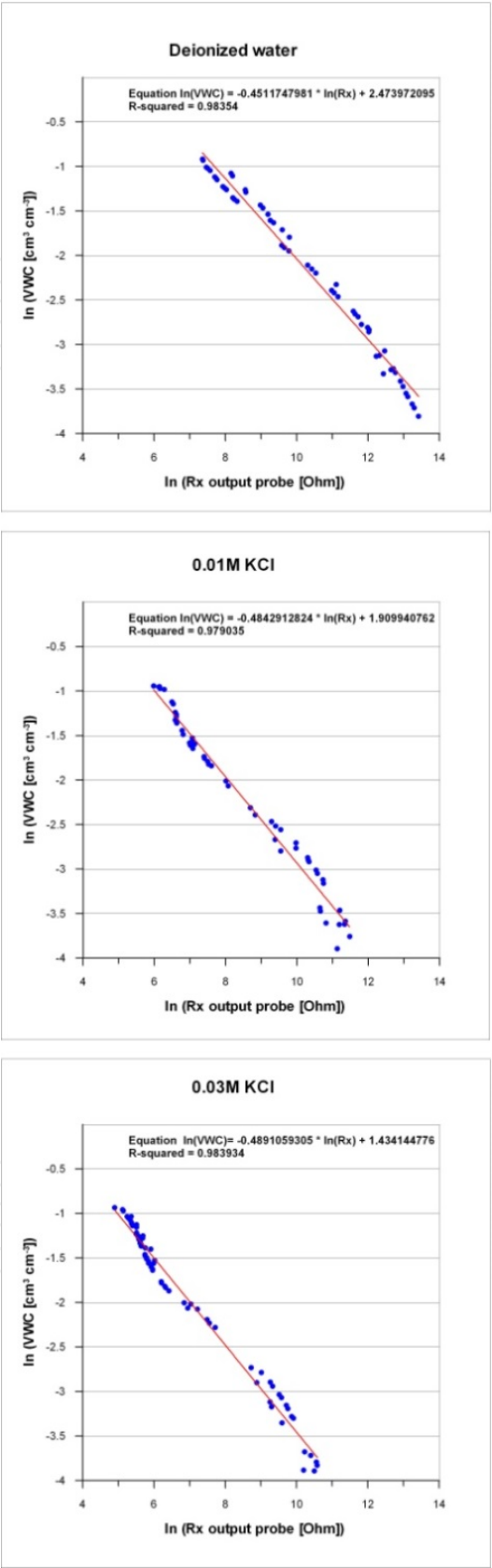
Figure 9. Calibration curves for each solution used to saturate samples.

VWC, measured during the test E3, against the predicted values, calculated using the calibration function, are plotted in the graph of Figure 10. The 1:1 line represents the perfect correspondence between the actual and the predicted value. The points below this line indicate an underestimation of the model while the points above indicate an overestimation. Two absolute error lines of ±0.03 and ±0.06 respectively, are also plotted in the graph. Only the 10% of the data fall outside prescribed limits while the 23% of data fall between the ±0.03 and the ±0.06 error lines. For the remaining 66% of the data, the model provides an estimate of the VWC with an error less than ±0.03. The correlation between the actual and predicted values is much higher for low values of VWC (less than 0.07) while tends to decrease for greater values of VWC.

KCl solution molarities	a	b
deionized water	2.4739	-0.4512
0.01	1.9099	-0.4843
0.03	1.4341	-0.4891

Table 4. Parameters of the calibration curve for each solution used to saturate samples.
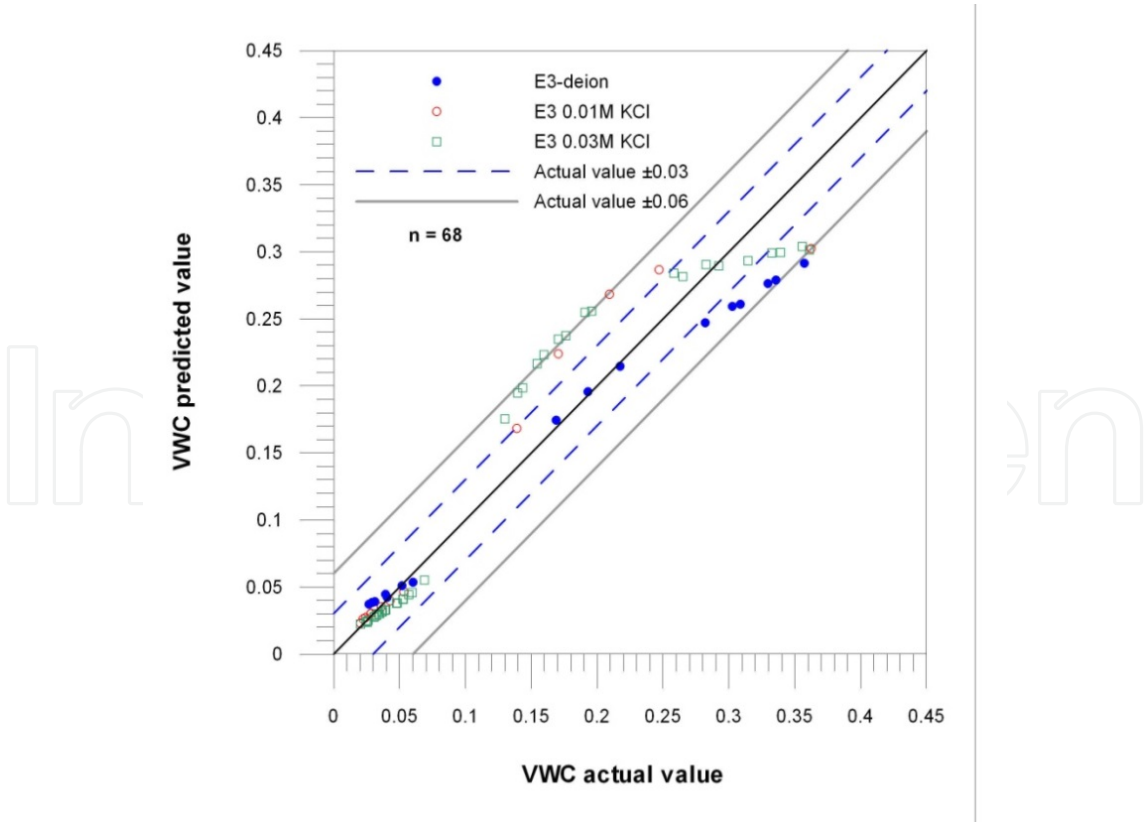
Figure 10. Validation test.

4. Conclusions

In this chapter has been described the experimental procedure used to obtain the calibration curves for a new device Z-meter 2, to measure water content in porous rock. Experimental tests have been carried out in laboratory, using samples of sedimentary rock, known as calcarenite, collected in a quarry located in Canosa, a city in South of Italy.

The device, based on the EIS method, gives as output both the resistance and the reactance values separately; this study considered the resistance because of its high correlation with the water content.

The tests were aimed at understanding the effects of the applied frequency and EC of KCl solution used to saturate the samples, on the device outputs and, consequently, on the calibration functions. For this reason a wide range of frequencies, from 100Hz to 10000Hz, was investigated and three KCl solutions with different EC values were used to saturate the rock samples.

The probes used were short stainless steel road probes, called picket type probes, chosen because they can be easily installed in the rock without be damaged. By analyzing the different data sets, none abrupt change in resistance values was observed during the evaporation process. This means that the probes work well in the calcarenite and that are able to record measurements of the impedance without any gap effects. The evaporation process monitored during the test showed how the weight of the sample decreases over time with different rates leading to model the process as a two-stages process. The first, corresponding to high water contents, was described by a linear relation while the second by an exponential function. The independence of the resistance from the frequency applied was observed. The resistance values obtained for the same water content at all tested frequencies were almost coincident, except close to dry condition (less than 2%). This means that, for water content higher than 2%, the device has a good accuracy and precision and that changing the frequencies has no effect on the resistence values.

Unlike the frequency, the resistance values were strongly affected by the molarity of the solution used for the sample saturation. For this reason the calibration curves have been obtained considering the data sets recorded for each solutions, separately. The calibration functions, that describe the relationships between the water content and resistance values, are of power type with a good degree of correlation as shown by value of R-squared of about 0.98.

Acknowledgements

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