

AN ACCURACY EVALUATION PROCEDURE FOR TDR QUALITATIVE AND QUANTITATIVE CHARACTERIZATION OF LIQUIDS

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Abstract: In a previous paper the Authors demonstrated that Time Domain Reflectometry (TDR) can be successfully used for an enhanced real-time qualitative and quantitative monitoring of liquids. In this paper, a metrological characterization of the method is formulated. Results are proposed, demonstrating the robustness and liability of the TDR technique.

Keywords- Time Domain Reflectometry (TDR), Dielectric Measurements, Level Measurement.

1. INTRODUCTION

In the present paper an assessment of time domain reflectometry (TDR) measurements for simultaneous detection of dielectric properties and levels of liquids is proposed. In a previous paper [1] the Authors developed a detection method demonstrating that TDR can be successfully used for an enhanced real-time monitoring of liquid inside tanks: in one shot, the determination of levels, dielectric properties and multiple interfaces in layered media are possible, thus opening challenging perspectives for several monitoring applications, especially for industrial and environmental control purposes. It is worth to note that stringent requirements in liquid monitoring applications, frequently limit the use of traditional liquid sensing technologies, such as ultrasonic or capacitive sensors. The above mentioned method can be identified as a substantial improvement with respect to existing approaches [2]. Based on these results, we will present in this work a comprehensive study in order to evaluate the performance level of such technique in terms of measurement accuracy. The main purpose of the present work is to analyse the qualitative and quantitative TDR method proposed by the Authors in [1] from a metrological point of view, leading to an assessment on the measurement uncertainty and to a validation of the method itself.

2. BACKGROUND

The Time Domain Reflectometry (TDR) method, used in this research, measures the reflected voltage along a 50cm-length coaxial probe caused by the travelling of a step pulse with 200 ps-rise time and records the travel time and magnitude of all reflected signals (echo) returning back from

the line [1]. The signal propagating down the line is reflected from a generic line section wherever an impedance mismatching occurs, causing an electromagnetic discontinuity. The TDR signal's velocity propagation v is related to the relative dielectric permittivity ϵ_r of the medium, which is assumed to be lossless (or at least with negligible conductivity), and the relative magnetic permeability μ_r , by the following well-known equation [1, 2]:

$$v = \frac{c}{\sqrt{\epsilon_r \mu_r}} \quad (1)$$

where $c=3*10^8$ m/s is the velocity of light in vacuo, while the relative magnetic permeability is unity in most materials, such as liquids.

When an impedance mismatch occurs, a part of the incident pulse is reflected back toward the step generator: the ratio between the reflected signal amplitude (V) and primary pulse amplitude (V^+) gives the reflection coefficient ρ . Experimental results demonstrated that for the designed 50 Ω -matched coaxial probes filled with the dielectric under test, the dielectric change at the air-dielectric material interface, can be related to the reflection coefficient through the equation:

$$\epsilon_r = \left(\frac{1-\rho}{1+\rho} \right)^2 \quad (2)$$

Furthermore, in the case of a TDR probe traversing from the top to the bottom of a tank or a pipe partially filled with a liquid of level D , we obtain that the dielectric constant of the liquid under test can be evaluated as:

$$\epsilon_r = \left(\frac{D_a}{D} \right)^2 \quad (3)$$

where D is the distance of the signal trip up to the new mismatch point (typically a short or open circuit terminating the probe), and D_a is the corresponding apparent distance in air, measured by the TDR set-up.

On the other hand, the length of the sample under test up to the point where a new mismatch occurs can be measured, as:

$$D = \frac{D_a}{\sqrt{\epsilon_r}} \quad (4)$$

Measuring the reflection coefficient (ρ) and the apparent level of the medium (D_a) we can, simultaneously, evaluate the liquid dielectric properties while performing a quantitative measurement (such as the evaluation of liquid levels in tanks, for instance).

3. PURPOSE AND ADOPTED METHODS

The main purpose of the present work is to analyse the qualitative and quantitative TDR method proposed by the Authors in [1] from a metrological point of view, leading to an assessment on the measurement uncertainty and to a validation of the method itself. There are basically two problems to consider for assessing the accuracy and performance limitation of the TDR system for the previously mentioned applications:

1. the accuracy with which apparent lengths (D_a) can be determined in the liquid sample under investigation when the designed 50Ω -matched coaxial probe is used;
2. the evaluation of the uncertainty (and of the relative level of confidence) in the relative dielectric constant and level estimation, deriving from adoption of Eqs. (2) and (4), respectively.

The uncertainty in the apparent distance estimation is evaluated through a replication experiment. Furthermore, as commonly reported in large part of literature [3-5], TDR liquid level sensing applications require a preliminary knowledge of the dielectric constant of the involved fluid.

The permittivity estimation deriving from the analysis of the reflection coefficient behaviour, according to the procedure discussed in [1], is compared with the expected values calculated using Eq. (3) and is associated to the uncertainty evaluation relationship. In the same way, the level estimation is compared to the liquid level given by the physical length of the coaxial probe central electrode, being the probe fully immersed in the sample.

The test set-up used in our experiments (Figure 1) consists of the TDR miniaturized unit (TDR100, Campbell Scientific), the processing control software, the coaxial cable (Aircell ® 7), and a 49.5 cm-long stainless steel coaxial probe. The sample holder consisted of a 1000 ml-graduated cylinder glass, having a diameter of 6 cm and height of 44 cm. The probe is made up of a central, cylindrical conductor with a coaxial conductive shield, both stainless steel made. The shield is suitably perforated, in order to allow the fluid circulation. The typical coaxial probe configuration is shown in Figure 2.

The probe central conductor is centered on the probe head through a teflon-made ring, while the lower part of the probe can be open-ended or inserted in a steel plug, thus allowing a short circuit at the probe end. The probe head and the coaxial conductive shield are 6 cm and 43.5 cm long, respectively.

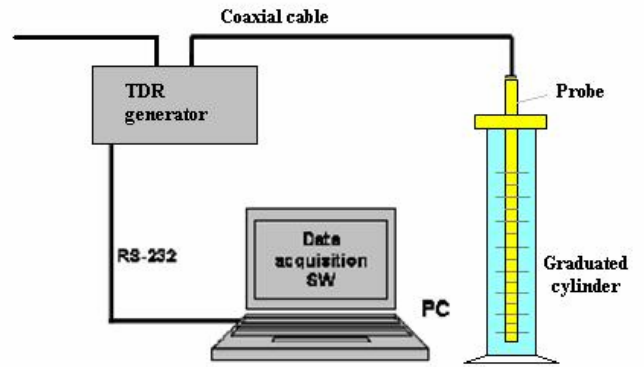


Figure 1. TDR experimental set-up



Figure 2. Configuration of the coaxial stainless steel probe.

The adopted analysis is structured as follows:

1. the experimental uncertainty in the apparent distances estimation is evaluated through the calculation of the standard deviation of different independent measurements on twenty de-ionized water samples (replication experiment);
2. using the same data-set, the experimental uncertainty in relative dielectric constant measurements and in level measurements is evaluated.

Accurate measurements of permittivity and level are made also on fuel, diesel oil and acetone samples, for the amount of four dielectric fluids. The liquids under test are representative of “low” (diesel and fuel), “medium” (acetone) and “high” (de-ionized water) permittivity values, and have also negligible ionic conductivity and relaxation in the considered frequency range (0-1.7 GHz). For each liquid sample ten waveforms were acquired, and the used measuring parameters were: relative propagation velocity equal to 1, number of points equal to 2,048 and measurement average equal to 10.

All the experiments were performed with the cylinder completely full of each liquid, so that the probe was totally immersed in the sample under test (Figure 1). Moreover, the coaxial probe was open-ended, since this configuration seems to be related to a better signal stability. Measurements were performed at different temperature conditions (room temperature), ranging from 23°C to 25°C, allowing to take also into account the effects of this environmental parameter in the uncertainty evaluation procedure.

The experimental results demonstrated that the accurate spatial localization of different interfaces can be performed in faster and easier way than the commonly used tangent method [6], simply considering the reflection coefficient derivative plot versus the distance of the TDR data [1]. Notice that the reflection coefficient value to be considered for applying Eq. (2), as discussed in [1], is related to the

steady-state achievement of the system, clearly detectable in the steady portion of the curve, hence the relative dielectric constant of the involved liquid can be easily evaluated. The steady reflection coefficient also indicates a negligible frequency dependence of the permittivity, thus corroborating the lossless liquid assumption.

This measurement method, based on the reflection coefficient evaluation, showed also a good performance in locating two different layered liquids contained in a tank, such as diesel oil-water layered sample, as reported in [1].

The detection of non-miscible liquids can be particularly useful, in the field of petrochemical, chemical, or agro-food, industrial processing control purposes. For example, during the refinery industrial processing a portion of water is often present in the lower part of the tanks, as a non-miscible fraction, and its monitoring is a very crucial point in terms of quality control detection. Similarly, the above mentioned method could be also implemented for monitoring of sediments in the tank bottom, foams in the top, emulsions etc. In addition, when miscible liquids are unintentionally present in the tank under test, the related deviation of the detected dielectric properties from the expected values, can give a significant feedback on the qualitative control of the industrial process. The detailed metrological analysis of such experimental cases (such as layered or mixed samples) is not considered, since the main scope of the present paper is simply to address to the measurement uncertainty evaluation and to the validation of the method when single liquids are monitored.

4. RESULTS AND DISCUSSION

4.1. Evaluating the uncertainty in apparent distance measurement

In Figure 3, a typical reflection coefficient waveform deriving from de-ionized water measurement, together with the derivative of the data is reported. Figure 3 clearly shows the reflection caused by probe-head-to-liquid interface and probe-end section corresponding to the open circuit. Before proceeding to the experimental estimation of relative dielectric constant, effective liquid level, and measured uncertainties, we performed multiple tests on twenty samples of de-ionized water, in order to evaluate measurement replication performance, precision and reliability, thus taking into account the fluctuation effects associated to the environmental conditions. For each sample we performed ten measurements. Table I summarizes the results: for each apparent distance calculated as mean value of ten measurements, the standard deviation is also reported. To obtain the best value of apparent distance and the respective uncertainty, we calculated the weighted mean on the twenty samples according to:

$$\bar{D}_a = \frac{\sum W_i * D_{a(i)}}{\sum W_i} \quad i = 1, 2, \dots, 20 \quad (5)$$

$$\bar{\sigma}_{D_a} = \frac{1}{\sqrt{\sum W_i}} \quad (6)$$

being:

$$W_i = \frac{1}{\sigma_{D_a(i)}^2} \quad (7)$$

where $\sigma_{D_a(i)}$ is the standard deviation related to i -th sample.

Based on this evaluation the average value is $\bar{D}_a = 375.78$ cm, the standard deviation is $\bar{\sigma}_{D_a} = 0.3$ cm and the extended uncertainty u_{D_a} , with a confidence level of 95%, is:

$$u_{D_a(i)} = 2.093 * \bar{\sigma}_{D_a} = 0.6 \text{ cm} \quad (8)$$

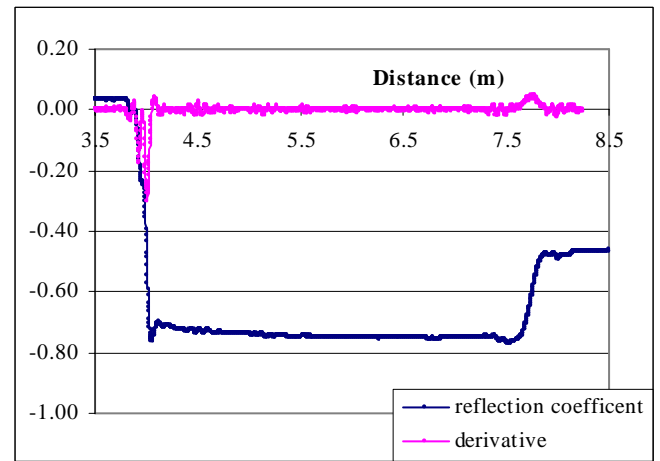


Figure 3: Typical TDR waveform for de-ionized water sample (reflection coefficient and derivative).

Table I: Apparent distance recorded data for 20 de-ionized water sample series.

Number of samples	Apparent liquid length (cm)	Standard deviation (cm)
1	375.43	1.64
2	375.53	1.21
3	375.31	2.06
4	375.01	1.84
5	374.96	1.58
6	374.80	1.38
7	375.13	1.30
8	375.16	1.86
9	376.55	0.89
10	375.62	1.87
11	375.26	1.86
12	374.94	1.67
13	376.70	1.43
14	374.77	1.31
15	376.04	1.93
16	376.09	0.96
17	376.16	1.16
18	374.72	1.12
19	375.01	2.11
20	375.74	1.75

4.2. Evaluating the uncertainty in dielectric constant and level measurement

As explained before, the dielectric constant can be calculated using Eq. (3) so that, the expected value can be derived from this relationship. The physical length of the coaxial probe central electrode, fully filled with water, can be measured with a tolerance of 0.5 mm. Calculating the square of the ratio between the apparent and the effective distance, we can estimate the correspondent expected values of the dielectric constant (ϵ_i^{exp}) and the relative uncertainty levels. Those expected values are therefore compared to the experimental values deriving from the TDR procedure above discussed. The standard uncertainty in ϵ_i^{exp} , due to the uncertainties in D_a and D , is calculated, according to the uncertainty propagation theory, through the equation:

$$u_{\epsilon_i^{exp}} = \sqrt{\left[\left(\frac{\partial \epsilon_i^{exp}}{\partial D_{a(i)}} u_{D_{a(i)}} \right)^2 + \left(\frac{\partial \epsilon_i^{exp}}{\partial D} u_D \right)^2 \right]} \\ = \sqrt{\left[\left(2 \frac{D_{a(i)}}{D^2} u_{D_{a(i)}} \right)^2 + \left(2 \frac{D_{a(i)}}{D^3} u_D \right)^2 \right]} \quad (9)$$

Furthermore, the presented research pointed out that, for accurate measurements of material dielectric properties, the effect of cable losses and additional signal dissipations must be taken into account, so that a calibration procedure can clearly discriminate the attenuations due to the sample and to the measurement set-up. This way, accurate measurements are possible in a wide range of conditions, particularly when long coaxial cables are employed for TDR-probe connection [7, 8]. The loss attenuation constant A was found through a calibration procedure described in [1, 8] hence, the reflection coefficient amplitudes can be corrected, according to:

$$\rho_{corrected} = A \rho_{measured} \quad (10)$$

For the employed test set-up, including the 3 m-length coaxial cable, we have measured $A=1.060 \pm 0.005$. Using this value we can correct the reflection coefficient values (ρ_i^{corr} , $i=1,2,\dots,20$) and the associated standard uncertainty ($u_{\rho_i^{corr}}$, $i=1, 2, \dots, 20$) can be written as:

$$u_{\rho_i^{corr}} = \sqrt{\left(\frac{\partial \rho_i^{corr}}{\partial \rho_i^{meas}} u_{\rho_i^{meas}} \right)^2 + \left(\frac{\partial \rho_i^{corr}}{\partial A} u_A \right)^2} = \\ = \sqrt{\left(A * u_{\rho_i^{meas}} \right)^2 + \left(\rho_i^{meas} * u_A \right)^2} \quad (11)$$

For each sample the dielectric constant value (ϵ_i^{meas} , $i=1,2,\dots,20$) and the effective level estimation (D_i^{meas} , $i=1,2,\dots,20$), can be calculated using Eqs. (2) and (4) respectively [1]. As reported in [1], in fact, if the liquid can be considered as a lossless dielectric the TDR waveform is characterized by steady curve portion, hence the relative dielectric constant of the involved liquid can be suitably evaluated according to Eq. (2). To assess the accuracy in the simultaneous evaluation of the dielectric constant and the level of liquid under test, the uncertainty relationships for ϵ_i^{meas} and D_i^{meas} are derived according to:

$$u_{\epsilon_i^{meas}} = \left| \frac{\partial \epsilon_i^{meas}}{\partial \rho_i^{corr}} \right| u_{\rho_i^{corr}} = 4 \frac{(1 - \rho_i^{corr})}{(1 + \rho_i^{corr})^3} * u_{\rho_i^{corr}} \quad (12)$$

$$u_{D_i^{meas}} = \sqrt{\left[\left(\frac{\partial D_i^{meas}}{\partial D_{a(i)}} * u_{D_{a(i)}} \right)^2 + \left(\frac{\partial D_i^{meas}}{\partial \epsilon_i^{meas}} * u_{\epsilon_i^{meas}} \right)^2 \right]} = \\ = \sqrt{\left[\left(\frac{1}{\sqrt{\epsilon_i^{meas}}} * u_{D_{a(i)}} \right)^2 + \left(-\frac{D_{a(i)}}{2 \epsilon_i^{meas} \sqrt{\epsilon_i^{meas}}} * u_{\epsilon_i^{meas}} \right)^2 \right]} \quad (13)$$

Table II: Summarized results for 20 de-ionized water sample series: expected dielectric constant, reflection coefficient, measured dielectric constant, and measured level. For each parameter respective estimated standard uncertainties are also reported.

Number of sample	ϵ_i^{exp}	$u_{\epsilon_i^{exp}}$	ρ_i^{meas}	ρ_i^{corr}	ϵ_i^{meas}	$u_{\epsilon_i^{meas}}$	D_i^{meas} (cm)	$u_{D_i^{meas}}$ (cm)
1	79.15	0,31	-0.75	-0.80	78,98	3,70	41,76	0,46
2	79.19	0,32	-0.75	-0.80	79,62	3,71	41,77	0,46
3	79.09	0,31	-0.75	-0.80	79,50	3,82	41,74	0,46
4	78.97	0,31	-0.75	-0.80	79,74	3,76	41,71	0,46
5	78.95	0,31	-0.75	-0.80	80,56	3,85	41,70	0,46
6	78.88	0,31	-0.75	-0.80	80,08	3,75	41,69	0,46
7	79.02	0,31	-0.75	-0.80	80,30	3,86	41,72	0,46
8	79.03	0,31	-0.75	-0.80	81,26	3,66	41,73	0,46
9	79.62	0,32	-0.76	-0.80	81,34	3,67	41,88	0,46
10	79.23	0,32	-0.76	-0.80	81,49	3,59	41,78	0,46
11	79.07	0,31	-0.76	-0.80	81,43	3,89	41,74	0,46
12	78.94	0,31	-0.76	-0.80	83,04	3,95	41,70	0,46
13	79.68	0,32	-0.76	-0.80	81,48	4,39	41,90	0,46
14	78.87	0,31	-0.75	-0.80	81,15	3,64	41,68	0,46
15	79.40	0,32	-0.75	-0.80	80,78	3,87	41,82	0,46
16	79.42	0,32	-0.76	-0.80	81,42	3,62	41,83	0,46
17	79.45	0,32	-0.75	-0.80	81,08	3,60	41,84	0,46
18	78.85	0,31	-0.75	-0.80	80,67	3,63	41,68	0,46
19	78.97	0,31	-0.75	-0.80	80,61	3,71	41,71	0,46
20	79.28	0,32	-0.76	-0.80	82,38	3,68	41,79	0,46

Table III: Summarized results obtained by calculating the weighted mean for de-ionized water, acetone, fuel and diesel oil: expected dielectric constant, measured dielectric constant and measured level. For each parameter respective estimated standard uncertainties are also reported.

Fluid under test	$\bar{\varepsilon}^{\text{exp}} \pm u_{\varepsilon^{\text{exp}}}$	$\bar{\varepsilon}^{\text{meas}} \pm u_{\varepsilon^{\text{meas}}}$	Relative error (%)	$\bar{D}^{\text{meas}} \pm u_{D^{\text{meas}}} \text{ (cm)}$	Relative error (%)
De-ionized water	79.15±0.14	80.84±1.62	2.1%	41.76±0.21	1%
Acetone	21.71±0.14	21.31±0.63	1.8%	42.8±0.64	0.9%
Fuel	2.17±0.05	2.21±0.03	1.8%	41.74±0.79	0.8%
Diesel oil	2.19±0.05	2.17±0.03	0.9%	42.28±0.81	0.4%

Table II summarizes the results. For each sample of water we report: the expected dielectric constant and respective standard uncertainty, the measured reflection coefficient, the corrected reflection coefficient according to Eq. (10), the measured dielectric constant and respective standard uncertainty and, finally, the calculated effective liquid length and respective standard uncertainty.

Through the same procedure used for de-ionized water samples, we evaluated the relative dielectric constant, level, and the associated uncertainty, for all the others liquids under test. As in section 4.1, we present results obtained by calculating the weighted mean for the dielectric constant value and for the effective liquid level. The weighted mean is calculated on twenty samples for de-ionized water, and on five samples for acetone, fuel and diesel oil. Summarized results are reported in Table III.

The results in Table III indicate that, for all the liquids under test, the relative uncertainty in both the relative dielectric constant and in the effective length estimation, associated to a 95% confidence level, are in the range of 2%. Furthermore, the results show a very good agreement between the theoretical and the estimated dielectric constant values, and between the effective and the estimated liquid level. In fact, the relative error calculated for ε_r as $\Delta\varepsilon = |\varepsilon^{\text{exp}} - \varepsilon^{\text{meas}}| / \varepsilon^{\text{exp}}$ is under 2.1%, and for D ($\Delta D = |D - D^{\text{meas}}| / D$) is even under 1%. Looking to these values we can notice that, for all the considered liquid samples, the relative errors are included in the measured uncertainty range. On such bases, we demonstrated that the experimental results validated the qualitative and quantitative monitoring method developed by the Authors in [1].

5. CONCLUSION

Basing on the technique developed for simultaneous detection of dielectric properties and levels of liquids, previously proposed by the Authors [1], in the present work an assessment procedure on the measurement uncertainty estimation of the method is developed. Since the depicted TDR method is an excellent candidate for quantitative and qualitative liquid monitoring applications, particularly for industrial and environmental control purposes, the results deriving from this analysis can be useful for a rigorous metrological characterization of TDR systems especially in the field of liquid sample monitoring. Starting from the evaluation of experimental uncertainty in the apparent distance estimation, both the uncertainties in relative dielectric constant measurements and effective liquid levels are investigated. In order to extend the accuracy estimation of the method to a wider range of liquids, the tests are

performed on four different fluids, which are representative of “low” (diesel and fuel), “medium” (acetone) and “high” (de-ionized water) permittivity values. The results confirm that the system is definitely adequate for simultaneously measuring liquid dielectric constants and levels: the associated uncertainty is under 2% for liquids with a wider dielectric constant. Moreover, a comparison between the expected and the estimated values of liquid dielectric constant and level show a very good agreement, thus validating the measurement method proposed by the Authors.

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