Enhanced reflectometry measurements of permittivities and levels in layered petrochemical liquids using an “in-situ” coaxial probe

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Abstract

Measurement and control of the fluid parameters play a very important role in industry applications such as in the petrochemical industrial processing, where there is an increasing demand for real-time determination of dielectric parameters in relation to the product quality. Furthermore, in such a context, additional stringent requirements frequently deal with the spatial localization of non-miscible-layered liquids, like water layers often present in the bottom of tanks during the refinery industrial processing. Recently, reflectometry has become a very attractive method for monitoring applications, thanks to its accuracy and flexibility performance. In this paper, the design of a suitable probe configuration and an associated calibration structure, both leading to an optimal experimental set-up for practical reflectometry measurements in petrochemical industrial applications, are illustrated. Moreover, starting from frequency-domain reflectometry data, a robust optimization procedure is implemented and experimentally tested, thus allowing the accurate evaluation of the frequency-dependent dielectric properties and of multiple levels in different stratified liquids.

Results derived through the simple time-domain technique are compared with those achieved through two different frequency-domain approaches, involving the Fast Fourier Transformation (FFT) of time-domain reflectometry (TDR) data and direct vector network analyzer (VNA) measurements, respectively. It is demonstrated that the frequency-domain approaches can significantly enhance the measurement accuracy, allowing the estimation of fuel level with an uncertainty lower than 0.5 mm. Furthermore, it is also shown that a low-cost TDR system, combined with an appropriate FFT-based algorithm, can be successfully adopted for the simultaneous measurement of permittivity and levels, without substantially affecting the measurement accuracy performance when compared to the direct frequency-domain VNA measurements.

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1. Introduction and motivation of the work

In recent years, dielectric measurements have become an effective tool for extracting significant information related to the structure and to the quality-status of various materials. In this regard, relevant scientific efforts have been largely devoted to the dielectric spectroscopy of liquids [1–5], since several industrial applications require an on-line continuous monitoring of the processing quality-status. In fact, different techniques allowing the measurement of the permittivity spectrum can be successfully implemented for characterizing various liquid products from a qualitative point of view. Particularly, in the petrochemical industrial processing, there is an increasing demand for real-time determination of dielectric parameters related to the product quality [6,7]. As an example, the oil density, the amount of heavy polar components,
the viscosity, the size distribution of the molecules, are strictly correlated to the Cole & Cole dielectric parameters [8].

Furthermore, in such a context, additional stringent requirements frequently deal with the spatial localization of non-miscible-layered liquids and with the depth estimation of different layers by remote sensing. For example, during the refinery industrial processing, a portion of water is often present in the bottom of the tanks, as a non-miscible fraction, and its continuous monitoring plays a crucial role [7,9,10].

Typically, various flow meters are used for fiscal and quality purposes [9]; nevertheless, the a-priori knowledge of the permittivity of the involved fluid components and the associated sample calibration are mandatory for the practical implementation of such methods.

Due to its accuracy, flexibility, large application conditions and potential for automated measurement, reflectometry has become one of the most attractive methods for various monitoring applications [11–14]. Reflectometry can be approached both in the time-domain and in the frequency-domain, depending on the instrumentation availability and cost limits, on the sensitivity and accuracy requirements, as well as on the required resolution and on the frequency bandwidth range. Measurements based on Frequency-Domain Reflectometry (FDR) can be performed through dedicated instrumentation, such as Vector Network Analyzer (VNA), Radar, and Spectrum Analyzer (SA); however, despite the wider frequency range capability, the potential application area is commonly limited by the high costs and reduced portability of the involved instrumentation. On the contrary, Time-Domain Reflectometry (TDR), which is definitely less expensive, is a well-established technique for the dielectric characterization of materials. In fact, thanks to the relation existing between the reflection coefficient and the dielectric constant, TDR is particularly adopted for real-time measurements of soil moisture and water content, as well as for determining the spatial location and nature of various objects, the localization of faults, different interfaces, or discontinuities [15–18]. This renders the TDR an appealing method for a variety of environmental and industrial applications.

As reported in [17,18] TDR-based techniques can simultaneously measure both the level and the static dielectric constant of different liquids. However, in order to enhance the measurement accuracy, the evaluation of frequency-dependent permittivities of liquids is a key issue. As well-known, in fact, permittivity is a frequency-dependent parameter, and appropriate numerical models, such as the Cole & Cole formula or the Havriliak–Negami model, need to be adopted, particularly when dispersive or lossy media are considered [19–22].

Reflectometry measurements commonly involve the insertion of a specific cell into the samples under test. Thanks to their characteristic impedance design, variously-shaped metallic coaxial probes can be typically used for this purpose.

The overall characteristic impedance matching of the probe plays a crucial role in terms of reflectometry measurement accuracy. In this regard, different strategies can be adopted, both in the probe design and in the calibration procedures, in order to simultaneously optimize the impedance matching, the mechanical stability of conductors, the practical realization and the adaptability to “in-situ” applications [22–25].

Furthermore, at the state of the art, no work proposes a combined approach for the simultaneous detection of levels, of multiple interfaces in layered media, and of the related dielectric properties, thus approaching both the quantitative and the qualitative monitoring.

On the bases of the discussed arguments, two are the main aims that motivate the present work. Firstly, to define a suitable probe configuration and an associated calibration structure both leading to an optimal experimental set-up useful for practical industrial applications. Secondly, to assess an optimization procedure that can ensure, in one shot, the accurate evaluation of the frequency-dependent dielectric properties and of the multiple levels in different stratified liquids.

Experimental measurements have been performed considering different levels of fuel–water layered samples. Since both fuel and water exhibit a strong dispersive behaviour, (especially with reference to the imaginary part of the complex permittivity), a complete spectral picture of the frequency-dependent dielectric properties can be obtained adopting the five-parameter Cole & Cole formula. In this regard, a robust optimization routine is established with an objective function that minimizes the sum of squared differences between the measured scattering parameter $S_{11}(f)$, and the one evaluated through a multi-section transmission line model. This model includes the Cole & Cole parameters of the considered liquids whose values are thus extrapolated through the minimization procedure.

The direct advantage of the proposed approach lies in the possibility of evaluating an enhanced model that suitably describes the frequency spectrum of the complex permittivity of the liquid materials under test.

To assess the method, results obtained starting from the scattering parameter, $S_{11}(f)$, derived from VNA measurements are compared with those derived from the frequency-domain transformation of TDR data. This way, it is demonstrated that the two methods show similar performances and that the measurement accuracy is substantially improved with respect to the simple time-domain approach.

2. Theoretical background

In this section, the theoretical aspects related to the extraction of frequency-dependent dielectric properties and of levels in liquid materials are analyzed. In particular, the proposed approach leads to the simultaneous evaluation of the Cole & Cole parameters and of the liquid levels, in the case of multi-layered liquids.

Typically, reflectometry measurements are based on the analysis of the signal that is reflected from the sample under test. The electromagnetic (EM) signal interacts with the investigated material that can be contained, for example, into a metallic coaxial probe. Both the dielectric properties of the material and the probe geometry affect
the reflected signal. In particular, the signal velocity is a function of the frequency-dependent complex permittivity of the samples under test, while the significant reflections are related to the difference in complex permittivity between the different media involved (i.e., air and liquids). This way, the reflected signals can be used to determine the permittivity frequency spectrum and the effective levels of the investigated materials [17–22].

The work is particularly focused on the case of fuel samples with a non-negligible fraction of water at the bottom; this is a relevant test case since this experimental condition is of great interest in the field of petrochemical industry application. The final aim is to simultaneously evaluate both the fuel dielectric properties and the liquid levels.

As described in [17,18], the simple time-domain approach to this problem involves two steps: first, the apparent liquid level is evaluated from the derivative of the TDR measured data, later on, the reflection coefficient value allows calculating the dielectric constant value of the considered medium, as well as its effective level. The main limitation of the above described time-domain approach is related to the estimation of the TDR dielectric constant, which does not take into account the frequency spectrum components of the actual liquid permittivity. This limitation can be overcome adopting a frequency-domain approach that mainly considers the scattering parameter $S_{11}(f)$. Such parameter may be obtained through direct frequency-domain measurements with a VNA or through an appropriate Fast Fourier Transform (FFT) processing of TDR time-domain data [19–22].

The measured scattering parameter is influenced by the probe geometry, by the liquid levels and by the permittivity spectra. The probe configuration impacts on the scattering parameter in two ways. On one side, the radius dimensions of both the inner conductor and the external shield influence the probe characteristic impedance and consequently $S_{11}(f)$. On the other one, the finite length of the probe causes the well-known multiple-reflection phenomenon induced by the signal propagating back and forth in the probe. As for the liquids filling the probe, they modify the propagation velocity and the coaxial probe impedance, according to their complex permittivity $\varepsilon_r(f)$. Moreover, the fuel-to-water interface causes additional reflections, together with that originating at the probe termination. Consequently, $S_{11}(f)$ is to be seen as the resultant of infinite contributes, that can be mathematically represented through the multiple-reflection theory [10,26]. The above description implies that the $S_{11}(f)$ spectrum contains all the information related to the liquid levels and to the permittivity, which are the unknowns to be determined. Such parameters can be estimated by an appropriate analytical model for $S_{11}(f)$, involving the specific liquid dielectric parameters and levels, and minimizing the sum of the squared differences between the analytical model and the measured $S_{11}(f)$.

More specifically, the five-parameter numerical model adopted for the complex relative permittivity of fuel and water, $\varepsilon_r(f)$, is derived by the Cole & Cole formula, according to [10,19]:

$$\varepsilon_r(f) = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{1 + \left(j \frac{f}{f_{\text{rel}}}ight)^{(1-\beta)}} - j \frac{\sigma_{dc}}{2\pi f \varepsilon_0},$$  \hspace{1cm} (1)

where $f_{\text{rel}}$ is the dielectric relaxation frequency, $\varepsilon_s$ is the dielectric permittivity at infinite frequency, $\varepsilon_\infty$ is the static dielectric permittivity, $\beta$ is a parameter characterizing the spread in relaxation frequencies and $\sigma_{dc}$ is the dielectric permittivity of vacuum ($8.854 \times 10^{-12}$ [F m$^{-1}$]). Such a function, both for fuel and water, is incorporated in the $S_{11}(f)$ multiple-reflection model described by the following equation [10,26]:

$$S_{11}^k(f) = \frac{\rho^k(f) + S_{11}^{k-1}(f)}{1 + \rho^k(f) S_{11}^{k-1}(f)} e^{-2j(f)kL}$$  \hspace{1cm} (2)

where:

$$\rho^k(f) = \frac{Z_{k-1}(f) - Z_k(f)}{Z_{k-1}(f) + Z_k(f)}$$  \hspace{1cm} (3)

is the reflection coefficient between section $k$ and section $k-1$ of the considered transmission line, $L^k$ is the length of the $k$th probe-section and $Z_k(f)$ is the frequency-dependent impedance of the section $k$ and can be estimated as:

$$Z_k(\omega) = \frac{Z_{0,k}}{\sqrt{\varepsilon_r^k(\omega)}}$$  \hspace{1cm} (4)

in which $Z_{0,k}$ is the characteristic impedance of the section $k$ when no filling liquid is present.

The frequency-dependent electrical length $\gamma^k(f)L^k$ is given by:

$$\gamma^k(f)L^k = 2\pi f L^k \sqrt{\varepsilon_r^k(f)/\varepsilon_0},$$  \hspace{1cm} (5)

where $j = (-1)^{1/2}$, $c$ is velocity of light in free space (2.99792458 $\times 10^8$ m/s); the relative magnetic permeability is unity in most liquids.

In particular, the considered multi-section model comprises three sections: section 0 is the probe termination, section 1 is the probe portion filled with water, and section 2 is the portion filled with fuel.

In order to estimate the input scattering parameter $S_{11}^{k=0}(f)$ of this multi-section transmission line, the value of the reflection coefficient at the termination has to be assumed. As clarified in the next section, the used probe adopts an open-ended configuration and therefore $S_{11}^{k=0} = 1$ has been assumed. Starting from this value and using Eq. (2), firstly for section 1 and then for section 2, the input scattering parameter can be obtained. It is worth noting that $\rho^{k=1}$, due to the open-ended termination, while, due to the specific probe design, $Z_{0,1} = 50 \Omega$ for all the remaining sections.

In order to assess the validity of the proposed model, it has been implemented within the commercial microwave circuit simulator MicroWave Office (MWO), which also makes optimization routines directly available. In particu-
lar, the chosen objective function to be minimized is the sum of the squared magnitude of the differences between the measured and the MWO-modeled scattering parameter.

This way, since the Cole & Cole formula is included in the modeled scattering parameter, along with the liquid level, the minimization of the objective function successfully provides an accurate estimation of the five dielectric parameters and of the levels of liquids, over the considered frequency range.

It must be pointed out that the optimization is rather complex because of the large number of parameters involved: the fuel level (the water level is constrained to the difference between the known probe length and the fuel level), four Cole & Cole parameters for the fuel (the static conductivity is constrained to 0 S/m), and five Cole & Cole parameters for the water. Therefore, the optimum Cole & Cole parameters and levels for fuel and water are evaluated through the following three-step optimization procedure:

1. The Cole & Cole parameters for water and fuel are fixed at reasonable initial guess values (the dielectric properties of water are well-known once the temperature is measured [27]), thus the fuel level is optimized. This gives an initial reasonable guess for the fuel level.
2. Starting from the previous values, all parameters are optimized through a random optimization procedure that should take the objective function close to the global minimum and should guarantee that the subsequent optimization step does not fall into a local minimum.
3. Using the previous results as the new starting inputs, a simplex optimization procedure is applied [28], which forces the objective function to the final global minimum.

The robustness and efficiency of the proposed procedure will be shown in Section 4.

It should be noted that, after this initial test phase is carried out with the aid of the circuit simulator, the whole procedure can be completely implemented through a customized software routine based on (2). This way, the possible variation of the liquid characteristics and the presence of undesired water layers can be promptly revealed and monitored in real-time. In a realistic monitoring application, after the first run of the optimizer has been completed and initial Cole & Cole parameters and liquid levels have been estimated, the continuous monitoring simply requires the optimizer to track the small changes in dielectric parameters and level that occur in time. This means that the further optimizations will only require a few steps of the simplex algorithm and will therefore be definitely suitable for a real-time application.

Finally, on a side note, it is worth pointing out that the overall computational time would be, in theory, reduced, by considering a simplification of the model described by Eq. (2). In fact, the reflection scattering parameter for the water portion could be removed, including in its place, a simple equivalent load impedance. Such a simplification is valid only if the round trip attenuation of such a layer is high enough to make reflections by the open-ended termination of the probe negligible. However, this is not the case of the presented experimental conditions, where the water level (about tens of centimetres) causes an attenuation far less than 1 dB, particularly in the lower frequency range.

3. Coaxial probe design and measurement set-up

As aforementioned, the probe configuration design plays a crucial role in terms of measurement accuracy. In fact, any impedance mismatch in the transmission line may seriously affect the evaluation of the liquid dielectric properties and levels. Although different calibration strategies can be adopted to minimize the systematic errors introduced by the mismatched components that are included in the test set-up (i.e.: connectors, cable, probe-head sections, etc.), such parasitic effects cannot be perfectly compensated for [23–25]. Particularly, when long coaxial probes are used, as for the considered application purposes, the sole SOL (Short-Open-Load) calibration measurements are not sufficient to eliminate all the parasitic effects, hence the residual errors affect the uncertainty in the frequency response estimation. To circumvent this problem the optimal strategy to adopt should simultaneously involve a suitable calibration procedure and a good impedance matching of each component of the measurement set-up. In this regard, the most critical component to be designed is the probe-head section, which should guarantee a perfectly matched cable-to-probe connection, as well as a robust mechanical stability of the probe central conductor. Additionally, the probe-head structure should be included in the SOL calibration measurements, in order to de-embed its effect and to accurately derive the response related only to the probe-section filled with the sample under test. As a matter of fact, all these requirements have been considered in the design of the coaxial probe configuration. Fig. 1 shows the 433-mm long coaxial probe, the 160-mm long probe-head portion as well as the calibration structure that exactly reproduces the internal probe-head portion: all these components are 50 Ω-matched, as explained in the following. The designed coaxial probe is made up of a central cylindrical conductor and of a coaxial stainless steel conductive shield, while its termination is open-ended. The shield is adequately perforated, in order to allow fluid circulation when the coaxial line is immersed, for instance, in industrial tanks. The inner and outer diameters of the probe conductors (a = 9 mm and b = 21 mm, respectively) have been chosen in order to have a 50 Ω-matched characteristic impedance when the probe is in air (Z0), according to the following formula [29]:

\[ Z_0 = 60 \ln(b/a) \]  

The chosen dimensions limit the frequency bandwidth of the probe to about 6 GHz, when the dielectric material considered is air. However, when a filling liquid is present this limit decreases proportionally to the square root of the liquid permittivity. In particular, for the application of interest, the liquid with the highest permittivity is water,
which limits the useful bandwidth of the probe to approximately 700 MHz.

The probe-head internal portion is made up of a 50 Ω foam-dielectric coaxial cable (Cellflex LCF78-50JA), contained in the stainless steel external shield that is ground contacted. The 433-mm long inner conductor of the probe is obtained removing the same length of the external shield and foam-dielectric of the Cellflex cable. The adopted design strategy is the key to ensure the overall impedance matching of the measurement set-up. Furthermore, the calibration structure reproducing the internal probe-head portion can ensure the physical connection to the HP 85032 B N-type SOL standard calibration kit, used to perform the calibration measurements exactly at the probe-head to sample reference plane.

For the time-domain measurements, the TDR experimental set-up consists of the TDR commercial unit (TDR100, Campbell Scientific) and of the processing control software. The excitation TDR signal is a step-like electromagnetic (EM) impulse, characterized by a time-duration of 14 μs and a rise time (t_r) of about 200 ps, corresponding to a frequency bandwidth of approximately 1.7 GHz. Frequency-domain measurements have been performed using the Agilent E8363B VNA, whose upper operating frequency is 40 GHz. The probe-head has been connected to the TDR and to the VNA instruments through a standard 3-m long RG213 coaxial cable. The HP 85032 B N-type SOL (Short-Open-Load) standard calibration kit has been used for calibration measurements, both for VNA and TDR measurements. In particular, such optimization procedure is applied both to the scattering parameter derived through the FFT-transformation algorithm of TDR waveforms and to the one achieved through direct frequency-domain measurements performed with the VNA. The accuracy of results obtained with the different approaches is compared.

Three samples have been considered in the experiments. All of them were contained inside a beaker with a height equal to the probe length. All measurements have been performed at a room temperature of 26 ± 1 °C. The three samples differed in the relative level of fuel and water, as shown in the first row of Table 1. The reported reference levels have been measured through a millimetre-graduated tape applied at the surface of the glass bea-

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fuel level [mm]</th>
<th>Water level [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>283.0 ± 0.5</td>
<td>150.0 ± 0.5</td>
</tr>
<tr>
<td>Sample 2</td>
<td>219.0 ± 0.5</td>
<td>214.0 ± 0.5</td>
</tr>
<tr>
<td>Sample 3</td>
<td>335.0 ± 0.5</td>
<td>98.0 ± 0.5</td>
</tr>
</tbody>
</table>
ker, with an uncertainty of ±0.5 mm, paying particular attention to avoid parallax errors.

Before performing measurements on the samples, for all the TDR and VNA measurements reported in Section 4.2, the SOL calibration procedure (with the calibration structure connected at the end of the feeding coaxial cable) has been performed, according to the calibration error model reported in [22]. Additionally, also the TDR measurements described in Section 4.1 have been calibrated, according to the time-domain calibration procedure described in [17,18].

4.1. Time-domain approach

Table 2 shows the results, in terms of fuel relative dielectric constant ($e_{TDR}$) and fuel level, obtained through direct processing of the time-domain TDR waveforms [17,18,30].

As mentioned in Section 2, $e_{TDR}$ is extrapolated from the amplitude of the first reflected pulse, caused by the air-to-fuel impedance change. Based on the $e_{TDR}$ evaluation and measuring the apparent distance associated to the fuel, the effective fuel level is determined from the ratio between the apparent fuel distance and $\sqrt{e_{TDR}}$. The water level is therefore simply derived from the difference between the overall probe length and the estimated effective fuel level. As an example, Fig. 2 shows the TDR waveform together with its derivative recorded with the probe immersed in sample 1. As highlighted in the figure, the significant reflection points are associated to the level estimation derived from the distance difference between peaks of the derivative curve [17,18,30]. The comparison between the TDR estimated levels and the reference values reported in Table 1 is summarized in Table 2: the maximum deviation for fuel and water level estimation is 4 mm.

4.2. Frequency-domain approach

After applying the time-domain approach, data measured on the three samples under test have been processed through the frequency-domain optimization routine.

As discussed in Section 2, the frequency-domain approach is theoretically able to simultaneously extrapolate the Cole & Cole parameters and the levels of both fuel and water. However, this theoretical capability is limited by the actual bandwidth of the measurement system. In fact, as aforementioned, the dimensions of the probe and the permittivity of water limit the system bandwidth to about 700 MHz, which is far below the relaxation frequency of water and fuel. In order to investigate the accuracy of the optimization procedure under these frequency bandwidth limitations, the theoretical multi-section model (2) has been applied to generate the “ideal” scattering parameters of the three samples having the nominal characteristics reported in Table 3. The computed $S_{11}(f)$ parameters correspond to those that would be measured under ideal conditions, that is without any uncertainty due to imperfect calibration, imperfect equipment, environmental noise, etc. These ideal scattering functions have been given as an input to the frequency-domain optimization routine, obtaining the extrapolated results reported in Table 3 (the subscripts ‘f’ and ‘w’ indicate the Cole & Cole parameters for fuel and water, respectively). The deviations of

Table 2
Fuel $e_{TDR}$, fuel level, and water level extrapolated for the three samples using time-domain approach. Deviations from reference levels are also reported.

<table>
<thead>
<tr>
<th></th>
<th>Fuel $e_{TDR}$</th>
<th>Fuel level [mm]</th>
<th>Water level [mm]</th>
<th>Deviation [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>2.23</td>
<td>281</td>
<td>152</td>
<td>±2</td>
</tr>
<tr>
<td>Sample 2</td>
<td>2.22</td>
<td>219</td>
<td>214</td>
<td>0</td>
</tr>
<tr>
<td>Sample 3</td>
<td>2.23</td>
<td>331</td>
<td>102</td>
<td>±4</td>
</tr>
</tbody>
</table>

Fig. 2. TDR waveforms recorded with the probe immersed in sample 1.
Table 3
Fuel and water levels and Cole & Cole parameters extrapolated from "simulated" frequency-domain measurements.

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\varepsilon_{sf})</td>
<td>2.35</td>
<td>2.35</td>
</tr>
<tr>
<td>(\varepsilon_{sw})</td>
<td>2.02</td>
<td>2.03</td>
</tr>
<tr>
<td>(f_d [\text{GHz}])</td>
<td>13.0</td>
<td>12.1</td>
</tr>
<tr>
<td>(\beta_i)</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>(h_i [\text{mm}])</td>
<td>283.0</td>
<td>283.0</td>
</tr>
<tr>
<td>(\varepsilon_{sw})</td>
<td>79.8</td>
<td>79.8</td>
</tr>
<tr>
<td>(\varepsilon_{sw})</td>
<td>5.2</td>
<td>6.1</td>
</tr>
<tr>
<td>(f_w [\text{GHz}])</td>
<td>17.3</td>
<td>17.1</td>
</tr>
<tr>
<td>(\beta_w)</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>(\sigma_{sw} [\text{S/m}])</td>
<td>2.58 \times 10^{-3}</td>
<td>2.58 \times 10^{-3}</td>
</tr>
<tr>
<td>(h_w [\text{mm}])</td>
<td>150.0</td>
<td>150.0</td>
</tr>
</tbody>
</table>

Table 4
Fuel static permittivity, fuel level, and water level extrapolated for the three samples using frequency-domain approach considering VNA measurements. Deviations from reference levels are also reported.

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuel static permittivity</td>
<td>2.37</td>
<td>2.24</td>
</tr>
<tr>
<td>Fuel level [mm]</td>
<td>283.5</td>
<td>219.5</td>
</tr>
<tr>
<td>Water level [mm]</td>
<td>149.5</td>
<td>213.5</td>
</tr>
<tr>
<td>Deviation [mm]</td>
<td>± 0.3</td>
<td>± 0.5</td>
</tr>
</tbody>
</table>

Table 5
Fuel static permittivity, fuel level, and water level extrapolated for the three samples using frequency-domain approach considering FFT of TDR measurements. Deviations from reference levels are also reported.

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuel static permittivity</td>
<td>2.21</td>
<td>2.18</td>
</tr>
<tr>
<td>Fuel level [mm]</td>
<td>282.7</td>
<td>219.3</td>
</tr>
<tr>
<td>Water level [mm]</td>
<td>150.3</td>
<td>213.7</td>
</tr>
<tr>
<td>Deviation [mm]</td>
<td>± 0.3</td>
<td>± 0.3</td>
</tr>
</tbody>
</table>

As a matter of fact, different minimization methods, such as the globalized bounded Nelder-Mead (GBNM), can be alternatively used. Nevertheless, this would probably improve the efficiency of the first run, but should not have any relevant impact in the subsequent continuous monitoring, when the optimization routine should just track smooth temporal variations in liquid levels and permittivities.

The obtained results are reported in Table 4 and Table 5 for VNA and FFT data, respectively. Analysis of the two tables demonstrates that the optimization procedure can suitably estimate fuel and water levels with a maximum deviation within the accuracy of the measuring tape used to evaluate the reference levels. Surprisingly, the FFT-processed data derived from TDR calibrated measurements do not cause substantial accuracy decrease with respect to the VNA measurements.

This demonstrates that a low-cost TDR system, associated to the proposed procedure, is the key issue for the practical implementation in industrial-related applications.

Furthermore, on a side note, the decreasing trend of static permittivity from sample 1 to sample 3 can be explained by the steady increase of room temperature during the experimental session. However, temperature increase was so small to cause only minimal changes in static permittivity, not detectable in the TDR traces, which only allow an approximate average permittivity (\(\varepsilon_{avg}\)) to be estimated. As a matter of fact, the proposed frequency-domain approach confirms its appropriateness also in suitably sensing small deviations from the standard qualitative status even when they are caused by the temperature variation.

As an example, Figs. 3 and 4 show the magnitude (a) and phase (b) of the scattering parameter measured for
sample 1, together with the one evaluated through the optimization procedure. Figs. 3 and 4 refer to VNA measurements and FFT of TDR data, respectively. Comparison of the two figures clearly shows that the data deriving from TDR measurements are much more noisy, especially at low frequencies, because the time-domain truncation of the acquired TDR waveform is intrinsically associated to the spectral dispersion of the reflected signal. Nonetheless, as evidenced in Table 5, this does not have any relevant impact in terms of accuracy in level estimation. Similarly, Figs. 5 and 6 show the magnitude (a) and phase (b) of the scattering parameter measured for sample 2, together with the one evaluated through the optimization procedure (Fig. 5 refers to VNA measurements while Fig. 6 refers to FFT of TDR data). Figures concerning sample 2 have been included because this sample proved to be the most critical in terms of relaxation frequency estimation. However, comparison of the two figures clearly shows that the fitting of optimized data is excellent. In fact, the investigated frequency range is far below the relaxation frequencies of both fuel and water. Once again, as evidenced in Table 5, the uncertainty in relaxation frequency estimation does not have any relevant impact in terms of accuracy in the level evaluation.
It is important to point out that the adopted optimization procedure converges to a final solution that depends on the initial estimates and on the intermediate convergence point of the random part of the optimization procedure itself. In order to assess the robustness of the overall procedure, it has been repetitively applied to the same sample, slightly varying the initial guesses for the Cole & Cole parameters of water and fuel. The obtained results, in terms of extrapolated fuel levels, show dispersion within 0.1 mm, thus demonstrating the robustness of the procedure.

To summarize the obtained results and to compare the different methods, Table 6 compares the fuel levels extrapolated from the time-domain and from the two frequency-domain approaches, with the reference values. Comparison of data clearly shows the relevant improvement achievable through the proposed frequency-domain approach.

5. Conclusion

In this work a robust investigative procedure for enhancing the simultaneous measurements of complex permittivity and levels in layered petrochemical liquids is presented. It is demonstrated that time-domain reflectometry measurements, combined with appropriate FFT-
based processing transformation, are definitely suitable to characterize multi-section layered media both from a qualitative and a quantitative point of view. Furthermore, the low-cost TDR system, associated to the FFT-based algorithm, can be successfully adopted for this purpose, without substantially affecting the measurement accuracy performance when compared to direct frequency-domain VNA measurements. This way, the possibility of extending the classical TDR approach to the case of the dielectric spectroscopy monitoring of layered liquids is assessed.

In particular, results deriving from simple TDR technique are compared with those achieved, in frequency-domain, both through the FFT-processing of TDR data and through direct VNA measurements. The comparison between the reference and the frequency-domain extrapolated values of permittivity and levels shows a very good agreement. Particularly it is worth noting that the uncertainty in fuel level estimation is under 0.5 mm. These promising results are achieved thanks to the combination of some novel strategies, such as an appropriate probe design, a suitable calibration procedure and, most importantly, a targeted optimization routine, thus rendering the method extremely robust.

The results lead to consider the proposed procedure as an excellent candidate for qualitative and quantitative li-

**Fig. 5.** Frequency-domain scattering parameter for sample 2 as obtained from VNA measurements and evaluated through the optimization procedure: (a) Magnitude, (b) phase.
quid monitoring applications, with relevant perspectives in the field of petrochemical industry monitoring, which not only do require accuracy and reliability, but also low-cost and portability.

Table 6
Reference and extrapolated fuel levels, in time-domain and frequency-domain approaches, for the three samples.

<table>
<thead>
<tr>
<th>Fuel level [mm]</th>
<th>Reference</th>
<th>Time-domain</th>
<th>Frequency-domain (VNA)</th>
<th>Frequency-domain (FFT of TDR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>283.0</td>
<td>281</td>
<td>283.5</td>
<td>282.7</td>
</tr>
<tr>
<td>Sample 2</td>
<td>219.0</td>
<td>219</td>
<td>219.5</td>
<td>219.3</td>
</tr>
<tr>
<td>Sample 3</td>
<td>335.0</td>
<td>331</td>
<td>335.1</td>
<td>334.7</td>
</tr>
</tbody>
</table>

References