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# 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.



## 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,

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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