Affordable Time-Domain Reflectometry System for Rapid Food Analysis

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Affordable Time Domain Reflectometry System for Rapid Food Analysis

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Abstract—The development of an inexpensive instrumental chain based on Time Domain Reflectometry is presented. The attention paid to the choice of the components allowed purchase costs reduction compared to the instruments available on commerce. The chain is composed by a hand-made 30 mm needle probe, a square wave fast risetime pulses generator on board, and a digital sampling USB oscilloscope with bandwidth of 11 GHz. Stability measurements and sodium chloride, sucrose, and cow milk were conducted to assess device repeatability and evaluate the predictive potential of estimation for some food parameters. The traditional geometrical-mathematical approach was replaced by multivariate investigation of the time domain signal. Good results in terms of coefficient of determination up to 0.983 were obtained. The results disclosed show how a cheap and easy to use instrumental chain can be very promising for attributes determinations of liquid materials, such as foodstuff.

I. INTRODUCTION

TIME Domain Reflectometry (TDR) is a well-established technique for characterizing numerous attributes of materials (e.g. moisture content), depending on their dielectric properties. It is based on the generation of a fast rise time step, covering different frequencies, and on the time domain analysis of the signal reflected by the materials under test. This last causes an impedance mismatch, along the transmission line; consequently, a fraction of the incident wave will be reflected along the line [1]. The wave travel time is represented by the time required to the signal to travel back and forth through the probe inserted into samples under test. Reflected pulse amplitudes are displayed in a time scale, so discontinuities are spatially assessed [1].

The pioneering work conducted by Fellner-Feldegg [2], suggested the use of the technique for permittivity measurements through the assessment of time dependence of the voltage reflection coefficient (ratio of the reflected wave to the incident ones), starting from step pulses from 1 MHz to 5 GHz, in a cylindric waveguide. The same information obtained by frequency domain measurement becomes available in a fraction of second simply analyzing the signal, which contains all frequencies of interest, in time domain [2]. Examples of successful applications of TDR technique are represented by the numerous devices, set up and commercialized in the soil science panorama [3,4]. Literature on this topic is mainly addressed to permittivity determination to indirectly estimate volumetric soil water content, soil dry density and soil bulk electrical conductivity [5, 6, 7].

Starting from some parameters related to soil physical characteristics such as soil bulk density, a huge amount of inferential fitting models was developed taking into consideration different typologies of soil and chemical compositions, in addition to deeply explored deterministic and probabilistic models [8, 9, 10, 11].

Concerning food applications, microwave dielectric properties in terms of complex permittivity were measured starting from TDR waveforms by exploring a wide frequency range, from 100 kHz to 20 GHz, on vegetables (carrot, radishes and potatoes), rice, milk and cheese, egg yolk and albumen, fish, chicken, alcoholic beverages (whiskey, beer and wine) and aqueous gels of ovalbumin, gelatin and agar [12]. Examples of works were conducted on mono-, and di-polysaccharides solutions [13], different granular materials such as cereals and legumes (corn, corn flour, soybean, bran,), and coffee [14, 15, 16, 17]. TDR techniques were applied for moisture determination in construction materials [18] or monitoring of cracks in concrete structures [19]. Cataldo et al. suggested a TDR method for the measurement of liquid levels, multiple interfaces in stratified liquids, and the determination of the dielectric permittivity and conductivity in petrol and water-based substances [20, 21]. A specific study was addressed to estimate the uncertainty of measurements [22].

The analysis in the time domain of the reflected signal was also promising as a rapid analytical approach for the assessment of extra virgin olive oils quality parameters. Water content, distributed in the form of a stabilized micro-dispersion, was assessed by analyzing the reflectometric signals with a classical

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TDR approach. In addition, the potentiality of the multivariate Partial Least Square (PLS) regressions was explored [23]. Main results evidenced that the multivariate analysis could overcome the limits represented by the traditional approach where the identification of the reflection starting point and the interpolation curves should be taken into consideration. Regarding the complexity and difficulty of the time domain determination, a study aimed at automating calculation of the time of flight was conducted by Giaquinto et al. [24]. Other study was addressed to set up models of multiple reflection analysis that, starting from TDR waveforms, calculate the complex spectral dielectric permittivity from 10 MHz to1 GHz [25]. The multivariate approach (Principal Component Analysis and PLS regression) of the TDR signal was also used by Berardinelli [26] for a rapid screening of fatty acid alkyl esters ranging from 3 to 100 mg kg^{-1}; these compounds represent important parameters related to the quality of fruit in-postharvest stage and oil storage processes. TDR combined with PLS regression was also considered in the work conducted by Fulladosa [27] aimed at non-destructively characterization of differences in dry-cured ham composition by means of an open-ended coaxial line probe.

TDR instruments for small sized samples measurement, because of the shortness of the probe, need to operate with steps of few pico-seconds and require acquisition systems (e.g. a digital oscilloscope) in the GHz range. Such devices are rather expensive and packed with features often not needed for TDR analysis.

Table I reports a comparison among the TDR devices developed for food analysis and related cost ranges. Several works were conducted to developed devices able to predict different quality food parameters, but the high cost of the instrumentations limits the application. Some devices concern also low cost of components; however, it does not specify the entity of such cost. Furthermore, the low-cost devices comprise low frequency range instrumentation characterized by slow step rise time and long probes, that are intrinsically economic.

By considering the above-described successful applications and the need of industry and research labs to find reliable tools to rapidly assess and screen chemical-physical properties of materials, the affordability of TDR technique could be increased by using assemblies of low-cost devices without losing the necessary performances and functionality.

The present work aims at analyzing the potentiality of a rapid and inexpensive instrumental chain for TDR spectroscopic technique in high frequency range. Furthermore, a short length probe (3.5 cm) compared to most of the presented in literature for low cost devices was developed. The choice of the suggested components makes it possible to reduce purchase costs by an order of magnitude. After assessing the stability of the instrumental chain and the repeatability of the measurements, the TDR technique and analysis method

performances are conducted by assessing increasing percentages of sodium chloride and sucrose in samples of deionized water and different cow milk, this last characterized by different fat content. The information contained in the TDR signal are evaluated for predicting concentration by using two different approaches, simple linear regression, and partial least square regression (PLS). These approaches are proposed in alternative to the traditional TDR elaboration based on the geometrical determination of the reflection signal travel time.

TABLE I
COMPARISON OF TIME DOMAIN REFLECTOMETRY MEASUREMENT

Sample	Range	Godness of Estimation	Error	Device and probe	Rise time (ps)	Cost of the device	Author
Salt and distilled water solutions	0-35%	N.R.	N.R.	HP4191A analyzer of impedance	17.5 (acquisition) 35 (reflected)	high cost (but N. R.)	[29]
Alcohols	Pure pentanol, butanol, propanol and ethanol	N.R.	N.R.	Tektronix 11802 with a Sampling Head SD 24	17.5 (acquisition) 35 (reflected)	high cost due to sampling head and oscilloscope (but N. R.)	[26]
Fatty acids alkyl esters in olive oils	3-100 mg kg^{-1}	R^2 values of 0.905, 0.923 and 0.927 (test set)	RMSE 2.4, 6.5, and 7.9 mg kg^{-1}	two copper wire probe (3.5 cm lenght), sampling head (Tektronix, SD-24) and oscilloscope (Tektronix, 11801B)			
Acqueous solution of sucrose	32-53,04% mass fraction	Adj. R^2 values up to 0.991	0.3%	coaxial probe (lenght N.R.) + HL 1500 TDR unit	200	low cost (but N. R.)	[30]
Sliced of tomatoes in glucose syrup	67.4-63.8 °brix	Adj. R^2 values up to 0.907	N.R.	metallic wire (5 m lenght or more), lance (2 m), TDR unit (N. R.)	N. R. (but in the order of ns)	low cost (but N. R.)	[28]
Water content in hops	9-80%	N.R.	N.R.	three rod probes (15 cm lenght), TDR unit (N. R.)			
Water content in corn flour	0-25%	N.R.	N.R.				
milk, cheese, egg white, egg yolk, ovalbumin gel, agar gel, gelatin gel, carrots, radishes, potatoes, rice, fish, and chicken	several (e. g. 10% to 75% for gelatine)	N.R.	N.R.	cell probes (up to 2.1 mm lenght) with sampling head (Agilent 54121A) and digitized by the sampling scope (Agilent 54121B)	35	high cost (but N. R)	[12]

N.R (NOT REPORTED)

II. Material and methods

A. TDR instrumental chain

The instrumental chain (figure 1) was set up by assembling the following main components:

PG, fast risetime pulse generator
SP, power splitter
OSC, digital sampling oscilloscope
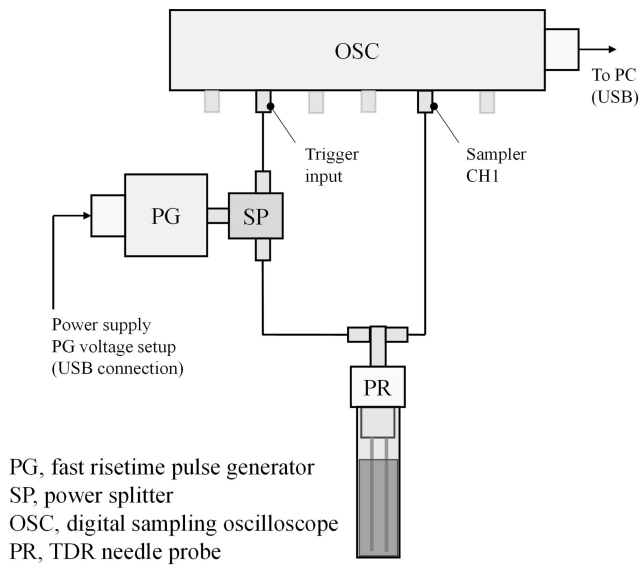PR, TDR needle probe

Fig. 1. Layout of the TDR instrumental chain

The probe is made by two chromed steel needle of 30 mm length, with a diameter of 1 mm, spaced 5.0 mm inserted in a nylon support. The sample holder is a cuvette and an SMA type socket connect the probe to the circuit. To guarantee a constant volume filling of the sample holder cuvette a stainless-steel device with a needle is made with a pre-set height correspondent to 3.5 ml of liquid, as previously reported by [23].

The pulse generator (Leo Bodnar Electronics, UK) allows rise times of approximately 30 ps for square wave (negative) pulses, at a frequency of 10 MHz, with a 50% duty cycle, the amplitude of the output signal ranging from 50 mVpp to 1.2 Vpp (selectable via USB), the generator requiring 200 mA at 5V. The oscilloscope used (IC227, IC Haus Integrated Circuits, Germany), with two channels, is suitable for fast sampling of repetitive signals, with bandwidth up to 11 GHz and DC coupled inputs. The device is connected to the PC via USB, and the software has an intuitive graphic interface. The oscilloscope used is inexpensive compared to the usual counterparts. The power splitter (Weinschel model 15151, USA) is used to distribute the impulse signal both to the trigger of the oscilloscope and to the TDR probe.

An estimation of the cost for the whole instrumental chain is around 3500 euro, as the sum of single components purchase at retail.

B. Examined materials

Analyses were conducted on several solutions of sodium chloride and sucrose in deionized water and on cow milk to assess lipid percentages. Per each constituent concentration three replications were acquired. All concentrations have been expressed in % of mass/volume (% m/v).

Different percentages of sodium chloride and deionized water solutions (0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 1.9, 2.0, 2.1, 2.2, 2.3, 2.4 and 2.5 % m/v) were tested.

Several sucrose concentrations (0.5, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24 and 25 % m/v) were examined.

Finally, cow milk (0.00, 0.10, 0.30, 0.50, 0.75, 1.00, 1.25, 1.30, 1.55, 1.60, 1.75, 2.00, 2.10, 2.25, 2.4, 2.6, 2.7, 2.9, 3.00, 3.30, 3.60, 3.65, 3.70 and 3.80 % m/v) was investigated among specified increasing percentages.

C. Repeatability of the measurement

To evaluate the stability of the instrumental chain and the repeatability of the measurements, tests were carried out with standard fluids, such as water and air. Signal were then processed and discussed in terms of waveforms differences, to underline the instrumental accuracy. Signals were acquired in air, and with deionized water. Ten repetitions for each component were acquired. In order to assess the possible, additional effect of the filling on the repeatability, measurements were also acquired with deionized water by empty and refilling the cuvette with water with ten repetitions (filling mode). The measurements were carried out with pulses of about -800 mV (in air). The maximum standard deviation and the maximum difference between values relating to each of the 626 measurement points of the TDR waveform (time resolution = 8 ps, total time = 5 ns) were selected as characteristic parameters for stability and repeatability assessment. The ratio between maximum difference and maximum signal amplitude (%) was also calculated, by considering each point, as additional index of the measurement variability (about 800 mV). Measurements were conducted at room temperature (23°C).

D. Data modelling

To estimates sodium chloride, sucrose, and lipid percentages simple linear model were built. Linear regressions were calculated between each of the 626 signal points at different concentrations. Coefficient of determination and related Root Mean Square Error (RMSE) of the best points, as well as p-level values of the estimated coefficient, are reported to evaluate the goodness of fitting [31]. Adjusted R^2 was also calculated to give a predictive feature with potential new data.

Multivariate analysis was performed to exploit the whole information contained in the spectra accounting for latent

information and possible predictive zones. Multivariate approach on this kind of signal elaboration is quite innovative, while traditional statistical estimation is widely applied. Partial least square regression was performed on the averaged spectra for each concentration and material, and random segmented cross validation was used to validate the calibration model by using Unscrambler software (version 9.7, CAMO, Oslo, Norway). Each segment contains six samples randomly selected. Therefore, the model performance was evaluated in terms of determination coefficient of cross-validation, and root mean square error of cross-validation (RMSECV).

III. RESULTS AND DISCUSSION

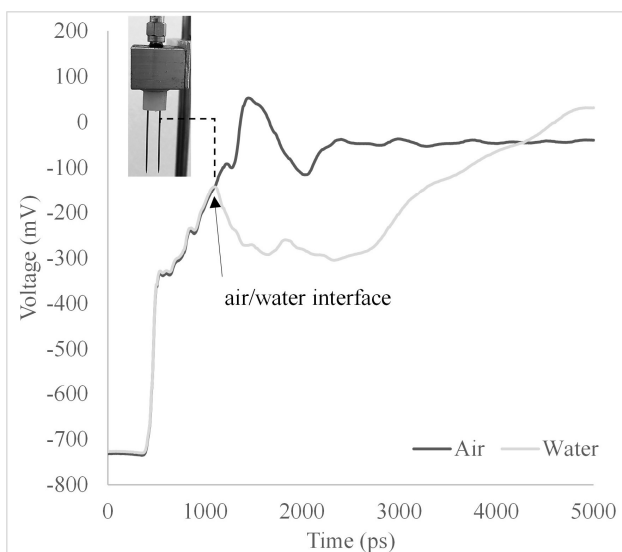Waveforms in air and deionized water are shown in Fig. 2.

Fig. 2. TDR waveforms obtained with a probe in air and deionised water (voltage equal to about 800 mV, averages of ten repetitions).

The impedance mismatch due to the interface air/water is shown in Fig. 2. The traditional approach exploits the calculation of the travel time with mathematical approach to estimate the dielectric constant from the reflection coefficient. However, previous work reports traditional mathematical approach in case of irregular waveforms showing relevant imprecisions and sometimes unsuccessful application [23, 26].

In this work air/water interface point is used as the start of the signal to be statistically processed, without calculation of the travel time. Standard deviation and maximum difference between single measurements relating to all points of the waveform acquired with a cuvette as sample holder empty (air), with water, and filled with water each of the ten repetition are shown in figure 3). In order to evaluate the accuracy of estimation a similar approach was presented by Cataldo et al. [16].
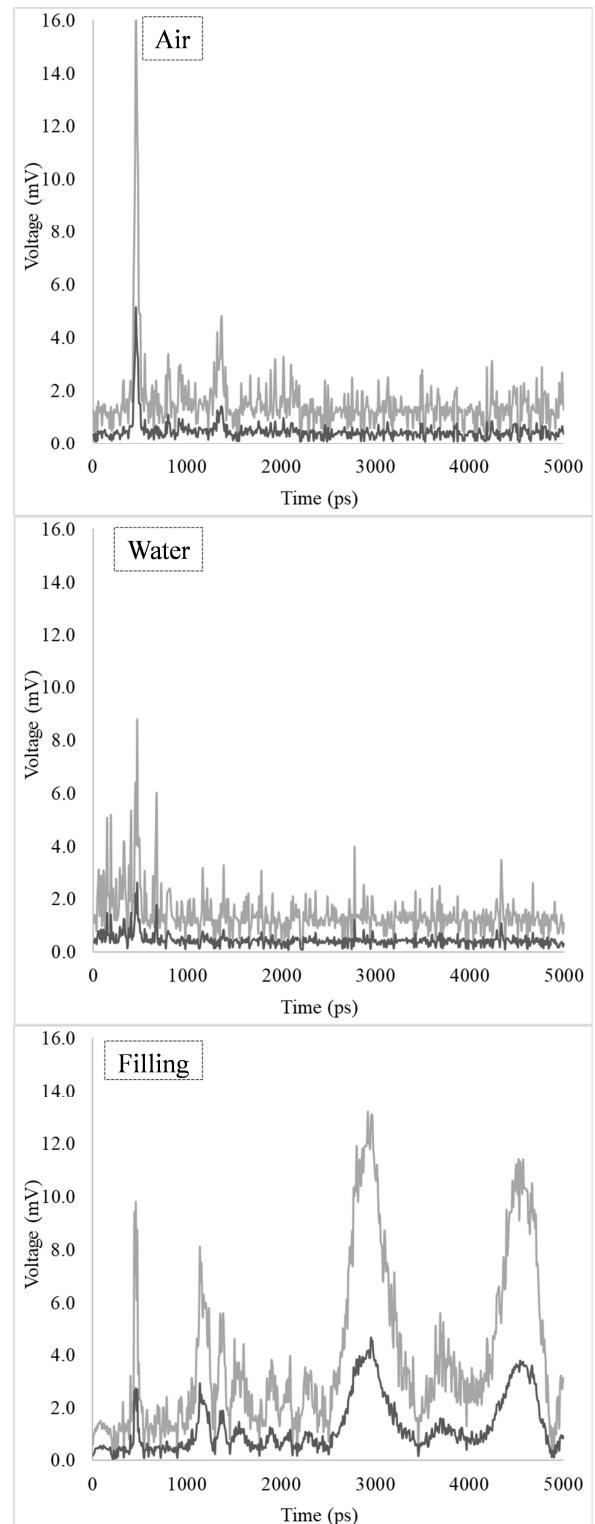

Fig. 3. Standard deviation (dark grey) and maximum voltage difference (light grey) calculated between ten repetitions, with probe in air and immersed water, and filling and emptying the cuvette for each repetition.

Table II resumes the parameters obtained from the repeatability tests.

TABLE II
PARAMETERS CHARACTERIZING THE STABILITY AND REPEATABILITY OF THE
MEASUREMENTS FOR TESTS IN AIR AND WATER

Parameters	Air	Water	Filling
Maximum standard deviation (mV)	2.3	2.6	4.7
Maximum difference (mV)	7.0	8.8	13.2
Maximum difference/signal range (%)	0.9	1.2	1.7

Table II reports good stability and repeatability of the signal for the selected parameters. In the examined conditions, a maximum standard deviation, in water filling mode (sample holder was filled for each acquisition), of around 4.7 mV with a maximum difference and maximum signal amplitude of 1.7% were obtained. It should also be considered that these values represent, as mentioned, the maximum recorded on the 626 points acquired by the oscilloscope. In this way it represents the worst condition. Furthermore, the highest signal instrumental variability was mostly present in the first part of the waveforms, before 1 ns, and the part mentioned as discarded for the statistical analysis.

A worsening of repeatability is due to the filling/emptying of the cuvette from 2.5 ns, but this does not seem to affect too much the statistical estimation of the concentration, as shown below.

Figure 4 shows the waveforms of the solutions of water/sodium chloride, water/sucrose, and milk at different concentrations. Each spectrum is an average of three sample replications.

As it can be seen, sodium chloride concentrations produce a huge spectrum variation in the time domain comparing the solution of water/sucrose and milk. However, in the square on the right down corner, a spectral variation with clear trends for each considered matrix is observable.

The spectral points were used as variables for the development of simple linear regression models.

Figure 5 shows the determination coefficients for each spectral point together with an example of waveform for sucrose.

It is possible to observe which portion of the spectrum most contributes to the estimation of the parameters examined.

Signals up to 3000 ps and the range from 3000 to 4000 ps give highest R^2 values, for sodium chloride and both sucrose and fat, respectively. Within this ranges, the model's parameters having the highest determination coefficients for sodium chloride, sucrose, and milk are reported in table III.
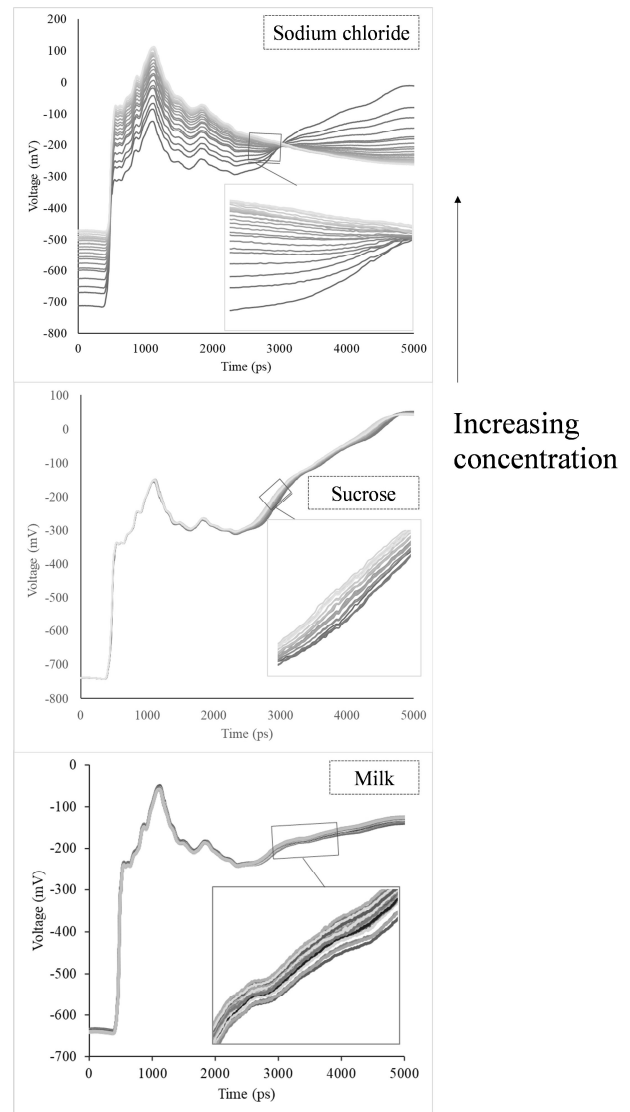

Fig. 4. Averaged waveforms (mV) acquired in the time domain (ps), for different concentrations of sodium chloride and sucrose in water, and lipid in milk.

The suggested TDR technique quite well estimates the concentration for all the examined liquids, with values of R^2 adjusted up to 0.952, and a RMSE equal to 1.15% (sucrose). RMSE value is strictly related to the measurable solution concentrations, it is a measure of the difference between predicted and observed values. For example, considering the selected sugar range 0.5-25%, the accuracy is equal to 1.15%. Other technical solutions, given in the literature, are quite difficult to compared, as several authors propose calibration models without coefficient of determination nor index related to the goodness of fitting.
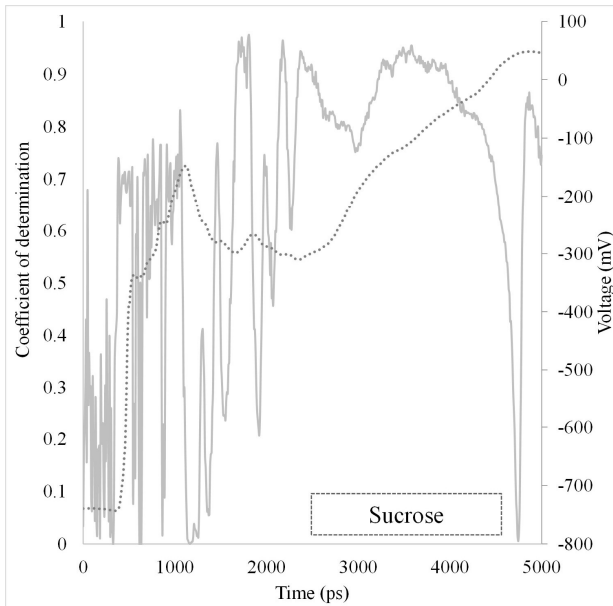
Fig. 5. Trend of the coefficient of determination of the linear regression for all spectral points (continuous line), and signal waveform (dashed line) for sucrose, shown as an example.

TABLE III

EQUATIONS AND VALUES OF THE ESTIMATED EQUATION COEFFICIENTS WITH RELATIVE RMSE (ROOT MEAN SQUARE ERROR).

Sample	R^2	R^2 adjusted	RMSE	Equation	Coefficient	Standard error
Sodium chloride	0.942	0.940	0.18 (%)	y = 0.115x + 24.084	x	0.006
					intercept	1.153
Sucrose	0.954	0.952	1.15 (%)	y = 1.340x + 146.3	x	0.061
					intercept	6.153
Milk	0.939	0.936	0.27 (%)	y = 0.3888x + 74.269	x	0.022
					intercept	4.110

Estimated coefficient p-level <0.05

However, some authors [15] reported similar coefficient of determination for agri-food materials, such as 0.930 for corn, 0.994 for corn flour, and 0.967 for soya bean, and other [27] showed calibration model for salt, water, water activity and fat estimation in dry cured ham with R^2 of 0.94, 0.95, 0.98, and 0.62 respectively. Moreover, R^2 higher than 0.93 were obtained for fructose, glucose and saccharose with a third order polynomial model [13]. For water content estimation in construction materials (sand, gray and white cements) R^2 from 0.976 to 0.989 were obtained for linear or cubic calibration curves [18]. Other Authors report uncertainty not more than 2% [20, 22].

The whole signals were then elaborated with multivariate PLS regression. Cross segmented validation gives a higher R^2

value than that of simple linear regression. The results of PLS analysis were summarized in table IV.

TABLE IV

PLS REGRESSION COEFFICIENT OF DETERMINATION (R^2), ROOT MEAN SQUARE ERROR (RMSE, % M/V) AND LATENT VARIABLES (LVS).

Sample	PLS model	R^2	RMSE	LVs
Sodium chloride	Calibration	0.989	0.07 (%)	
	Validation	0.982	0.11 (%)	4
Sucrose	Calibration	0.981	0.98 (%)	
	Validation	0.974	1.16 (%)	3
Milk	Calibration	0.989	0.12 (%)	
	Validation	0.964	0.21 (%)	5

PLS analysis confirmed a good ability of the TDR instrumental chain to estimates the different parameters, with R^2 values up to 0.982 in validation and RMSE of 0.11% (sodium chloride). Calibration and validation models present similar RMSE values, suggesting good model performances.

Literature [3] reported TDR performance evaluation on cheap commercial instrument. It is shown that permittivity estimation performances are related to the probe delay time. The high-frequency spectrum portion is well conditioned only if the probe time delay is equal or less than that of the dielectric relaxation time [3].

Cataldo [16] describes the time domain reflectometry (TDR) for both in-line and off-line moisture content measurement of agri-food materials. The research implemented an empirical approach as simple and accurate, as the herein presented. As the Authors reiterated [28], calibration curves are specific for each foodstuff; hence, different curves must be acquired for different materials. Furthermore, extensive research has been dedicated so far to TDR and to its use on a plurality of materials, but availability of very low-cost TDR systems for food quality parameter estimation, with very short probe (pulse rise time of around 30 ps), is extended with the present paper even further increasing the interest of agri-food industry. The measurement accuracy of the system appears to be in line with studies reported in literature.

IV. CONCLUSION

Technical performances of the suggested TDR instrumental chain were evaluated with air and water measurements. Measurement repeatability was remarkable, with maximum

standard deviation (considering the filling variability), around 4.7 mV and a maximum variability of 1.7 % (out about 800 mV of max amplitude). The overall results adequately demonstrated that different solution concentrations of sodium chloride, sucrose and fat in milk could be determined by TDR signal coupled with a simple linear regression. Better results were obtained with PLS regression improving a segmented cross validation helpful also for further investigation on more complex matrix.

Finally, the easy to set up device, appears very promising for rapid, non-destructive determinations, accounting very short acquisition time, such as one minute per each sample, often resulting less complicated than that of chemical-physical analysis. The choice of cheap components for the suggested instrumental chain allowed a cost reduction of around one order of magnitude if compared with many traditional solutions such as multi-function wideband oscilloscopes integrating the module for pulse generation. This last aspect could be crucial for a greater diffusion of the TDR technique.

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