

Time domain reflectometry for dielectric characterization of olive mill wastewater contaminated soils

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Abstract

Olive mill wastewater (OMW) is a compound originating from oil mills during oil extraction processes. In the Mediterranean area, more than 30 million m³ of OMW are produced each year, which represents 95-97% of the world production. Such volumes of untreated OMW are usually directly disposed of into drainage systems, water bodies (such as streams, lagoons and ponds) or are sprinkled on soils, causing potentially severe environmental problems to soils and groundwater. Consequently, there is a serious waste management problem related to the olive oil industry, because these practices no longer being acceptable. In the case of on-land OMW disposal, the characterization and the identification of this contaminant in soils is a fundamental task especially with a view to maintaining the integrity and quality of agroecosystems. In recent years, soils have been extensively studied to detect contaminants by using various geophysical methods. Among such techniques, time domain reflectometry (TDR) has shown, in different contexts, evident sensitivity and resolution capability for the characterization of contaminated soil sites. In order to further exploit the potential of the TDR technique, in this study we conducted a series of laboratory-controlled tests to explore how OMW influences the dielectric response of contaminated soils. This investigation led to the development of an empirical dielectric model to estimate the presence of OMW in variably saturated-contaminated soils with different textures and pedological features. In particular, the soils selected belong to two typical pedological units in southern Italy which

account for approximately 90% of the Italian olive plantations and one of the highest concentrations of the olive oil industry in Italy. In these districts, as well as in other European and Mediterranean countries, there is the controversial habit to spread OMW on soils.

Introduction

The olive oil industry is one of the main agricultural sectors in the Mediterranean basin. Every year about 2 million tons of olive oil are produced (Piotrowska *et al.*, 2011), and this production is steadily increasing (Caputo *et al.*, 2013; Sahraoui *et al.*, 2015).

The extraction process of olive oil generates olive mill wastewater (OMW) which is a mixture of vegetation water initially present in the drupes and the water used during the different stages of oil extraction (Colarieti *et al.*, 2006; Sahraoui *et al.*, 2015). The volumes of OMW produced depend on the extraction method (*i.e.*, traditional pressing, or two-phase/three-phase centrifugation systems) and may vary between 40 and 100 liters per 100 kg of processed olives (Kavvadias *et al.*, 2014).

OMW is a waste product with a high pollution load. It is generally characterized by a low pH, high salinity and organic content, high chemical and biological oxygen demand, a high concentration of suspended solids, and abundant presence of mineral elements especially nitrogen, phosphorus, potassium, calcium and magnesium (Mekki *et al.*, 2006). Furthermore, considerable concentrations of phenolic compounds may be detectable in this wastewater with concentrations usually varying between 1.0 and 10 g/L (Capasso *et al.*, 1992; Piotrowska *et al.*, 2011).

Due to its complex composition, OMW cannot be directly added to domestic wastewater treatment plants (Caputo *et al.*, 2013), and there is a lack of practical and sustainable alternative solutions for OMW disposal. This aspect represents a potential environmental problem for olive oil-producing countries (Kavvadias *et al.*, 2014). One solution adopted for OMW discharge, which has been legally regulated in several countries, is its use for soil fertilization. However, the benefits of this practice are questionable due to its proven toxic effect on the soil biota (Isidori *et al.*, 2005). Furthermore, long-term OMW application may cause severe alteration of soil chemical and physical properties.

For all the above reasons, the problem of evaluating the spatial and temporal distribution of OMW in situ represents a research topic of paramount interest. For example, it can now be faced by using non-invasive geophysical methods (Huisman *et al.*, 2003; Robinson *et al.*, 2003).

Starting from the findings of Comegna *et al.* (2016), in this study we show the suitability of the time domain reflectometry (TDR) technique in determining the presence of OMW in a contaminated medium. Indeed, we observed that OMW affected the dielectric behaviour of the contaminated soil. A direct dependence

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of the bulk electrical conductivity (EC_b) on OMW concentration was experimentally documented. This dependence was investigated in depth and exploited to develop, calibrate and validate a dielectric logarithmic model, which provided the possibility of quantifying the presence of OMW, under different levels of soil contamination.

Dielectric permittivity and electrical conductivity determination using time domain reflectometry

TDR allows concomitant determination of soil bulk dielectric permittivity (ϵ_b) and soil bulk electrical conductivity (EC_b) on the same observation volume (Dalton *et al.*, 1984). The ϵ_b determined by TDR requires the measurement of the propagation velocity and attenuation of an applied electromagnetic wave along a transmission line in the soil (Topp *et al.*, 1980). At TDR frequencies between 200 MHz to 1.0 GHz, the dielectric losses can be assumed to be negligible, and ϵ_b along a wave-guide line of length L is a function of the propagation velocity v ($=2L/t$) according to:

$$\epsilon_b = \left(\frac{c}{v}\right)^2 = \left(\frac{ct}{2L}\right)^2 \quad (1)$$

where c ($=3 \times 10^8 \text{ m s}^{-1}$) is the velocity of an electromagnetic wave in vacuum, and t is the travel time, that is the time that the TDR signal requires to travel to and from the wave-guide.

The attenuation of the TDR signal can also be used as a measure of EC_b . According to the *thin section approach*, originally proposed by Giese and Tiemann (1974), EC_b can be calculated as follows:

$$EC_b = \frac{\epsilon_0 c Z_0}{L Z_c} \left(\frac{2V_0}{V_f} - 1 \right) \quad (2)$$

where ϵ_0 is the dielectric permittivity of free space, Z_0 is the characteristic probe impedance, Z_c is the TDR cable tester output impedance, V_0 is the incident pulse voltage, and V_f is the return pulse voltage at relatively long distances along the waveform (Or *et al.*, 2004).

Volumetric olive mill wastewater content determination in soils

The detection of contaminants in multiphase soil systems by means of geophysical methods is problematic, even if the pollutant is homogeneously distributed within the soil matrix (Redman and De Ryck, 1994; Persson and Berndtsson, 2002; Haridy *et al.*, 2004; Moroizumi and Sasaki, 2006; Francisca and Montoro, 2012). The TDR technique has the potential to reveal the presence of a contaminant in soils (Comegna *et al.*, 2013a; Comegna *et al.*, 2016; Comegna *et al.*, 2017; Comegna *et al.*, 2019). However, as the TDR waveform only returns *aggregate* information that depends on all the distinct phases involved (Comegna *et al.*, 2016), the challenge is to find a way to infer the dielectric weight of the pollutant from the whole dielectric response (Comegna *et al.*, 2013b).

In this research work, we followed the same methodological approach applied by Comegna *et al.* (2016), which was developed to detect and quantify the presence of organic contaminants, such as non-aqueous phase liquids (NAPLs) in variable saturated soils. We observed that the presence of a NAPL in the soil affected the dielectric response of the medium in terms of bulk dielectric permittivity (ϵ_b decreases as the amount of NAPL increases). The

analysis of dielectric NAPL behaviour allowed us to establish a univocal relationship between the amount of NAPL in the contaminated soil (θ_{NAPL}), the bulk dielectric permittivity of the multiphase medium (ϵ_b), and the final value of the reflection coefficient (ρ_f) which, as known, can only be determined at long TDR-travel times (Or *et al.*, 2004). Starting from these findings, we concentrated our efforts on OMWs, which are fluids with dielectric characteristics that are quite unlike those of NAPLs.

In the case of OMWs, we observed that their presence in soils scarcely alters the global dielectric response of the medium in terms of permittivity, which varies randomly with increasing amounts of OMW (see section below). By contrast, at higher propagation times (*i.e.*, those useful for TDR- EC_b calculation), a functional relationship between θ_{OMW} and EC_b can be hypothesized. Such considerations allowed us to develop, in the contaminated medium, a logarithmic relationship between EC_b , and the so-called relative volume of OMW in water (β):

$$\beta = a \ln(EC_b) + b \quad (3)$$

where a and b were the coefficients which had to be experimentally determined (*i.e.*, site specific), and the relative volume of OMW in water, β , was defined as (Rinaldi and Francisca, 2006):

$$\beta = \frac{\theta_{OMW}}{(\theta_w + \theta_{OMW})} = \frac{\theta_{OMW}}{\theta_f} \quad (4)$$

where θ_f and θ_w were respectively the volumetric content of the whole fluid phase and the volumetric water content. Values of β varied in the range between 0 for a soil-water mixture and 1 for a soil-OMW mixture.

By substituting Equation 4 into Equation 3, θ_{OMW} can be calculated as:

$$\theta_{OMW} = \theta_f [a \ln(EC_b) + b] \quad (5)$$

We observed that, for a selected soil, coefficients a and b depended on θ_f values (see section below), in the sense that for each θ_f a pair of a and b parameters can be estimated. Further data examination coupled with statistical analysis based on an ANCOVA test, which was conducted at a significance level of $\alpha=0.05$ (for more details see Comegna *et al.*, 2016), allowed us to assume the coefficient a of Equation 5 to be constant ($\alpha = \alpha = \text{cost}$, thus independent of θ_f), whereas the term b could be related to θ_f via a second-order polynomial equation:

$$b = b_1 \theta_f^2 + b_2 \theta_f + b_3 \quad (6)$$

where b_1 , b_2 and b_3 were fitting parameters of the equation.

As a result, θ_{OMW} could be finally written as follows:

$$\theta_{OMW} = \theta_f [a_c \ln(EC_b) + (b_1 \theta_f^2 + b_2 \theta_f + b_3)] \quad (7)$$

Using Equation 7, θ_{OMW} could be estimated once the bulk electrical conductivity (EC_b) and the volumetric fluid content (θ_f) of the contaminated medium were determined. It should be empha-

sized that the methodology developed can be successfully applied in those situations where the practice of OMW sprinkling on soils is a priori known. In such field conditions, OMW can indeed be considered the dominant factor that influences the dielectric behaviour of soils, mainly in terms of EC_b values, as clearly shown in the sections below.

Materials and methods

Soil and olive mill wastewater properties

The soils selected to conduct this research work were a loam *Eutric Cambisol* (IUSS Working Group WRB, 2006) and a silt-loam *Anthrosol* (IUSS Working Group WRB, 2006), both of which are found in Southern Italy. Table 1 reports the main physical and chemical properties of the two soils, while Table 2 shows a characterisation of the OMW employed in the laboratory experiments.

The total polyphenol content was obtained using Folin-Ciocalteu colorimetric method (APHA, 1995). Absorbance was measured at 760 nm with a SpectroVis Plus (Vernier Software & Technology) UV-visible spectrophotometer. Total nitrogen (TN), total organic content (TOC) and chemical oxygen demand (COD) were determined by using the IRSA-CNR 4060 method (IRSA-CNR, 2003), the IRSA-CNR 5040 method (IRSA-CNR, 2003) and the IRSA-CNR 5130 method (IRSA-CNR, 2003), respectively.

Experimental equipment

The experimental apparatus consisted of a TDR unit (Tektronix 1502C cable tester) and a three-wire TDR probe (with wave guides 14.5 cm long) connected via an RG58 coaxial cable to the tester. The TDR signals once acquired were post-processed for ϵ_b and EC_b calculation with a homemade Matlab code. The laboratory system used during the experiments is illustrated in Figure 1.

Laboratory experiments

The laboratory experiments were carried out on repacked soil samples. Simultaneous measurements of ϵ_b and EC_b were made on soil samples that were adequately prepared as a mix of known amounts of soil and volumetric water (θ_w) and OMW (θ_{OMW}) content, following the scheme reported in Table 3. Soil samples were oven dried at 105°C and sieved at 2 mm. The different combinations of soil, water and OMW were mixed and then kept for 24 h

in plastic bags to ensure that OMW and water were uniformly distributed within the soil. Since the TDR signal (hence the dielectric response of a medium) is influenced by soil porosity ϕ (Jung *et al.*, 2013), soil samples were cautiously placed in plastic cylindrical containers (15 cm high and 9.5 cm in diameter) until the bulk densities of 1.27 g cm⁻³ (*Eutric Cambisol*) and 1.13 g cm⁻³ (*Anthrosol*) were reached. Finally, a TDR probe was inserted vertically into the samples. The same procedure was replicated on a second set of samples for model validation. The laboratory tests were conducted at a constant temperature of 25°C.

Model performance evaluation

Three statistical indices were selected and calculated for evaluating model performance (equation 7): i) mean absolute percentage error (*MAE*), ii) model efficiency (*EF*), and iii) maximum absolute percentage error (*ME*), determined according to the following relations (Legates and McCabe Jr, 1999; Goovaerts *et al.*, 2005):

$$MAE(\%) = \frac{\sum_{i=1}^N |E_i - O_i|}{N} \cdot 100 \quad (8)$$

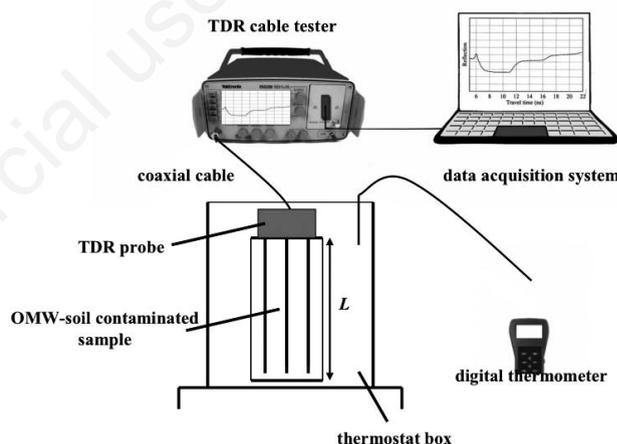


Figure 1. Experimental setup used in laboratory experiments (from Comegna *et al.*, 2016). TDR, time domain reflectometry; OMW, olive mill wastewater.

Table 1. Main physico-chemical properties of the two soils investigated.

| Soil | Depth (cm) | Soil Texture and Classification (USDA) | | | Porosity (%) | C (%) | EC_w (dSm ⁻¹) | pH | |
|------------------------|------------|--|----------|----------|--------------|-------|-----------------------------|------|------|
| | | Sand (%) | Clay (%) | Silt (%) | | | | | |
| <i>Eutric Cambisol</i> | 0-20 | 41.4 | 16.4 | 42.2 | Loam | 0.52 | 0.30 | 0.13 | 8.40 |
| <i>Anthrosol</i> | 0-20 | 15.7 | 11.6 | 72.7 | Silt Loam | 0.57 | 1.84 | 0.17 | 8.37 |

Table 2. Main physico-chemical properties of the olive mill wastewater used in the experimentation.

| Parameter | Value |
|--|-------|
| pH | 3.85 |
| Electrical conductivity at 20°C (dS/m) | 10.20 |
| Dissolved oxygen: DO (mg/L) | 0.23 |
| Total organic carbon: TOC (mg/L) | 6016 |

$$EF = 1 - \frac{\sum_{i=1}^N (E_i - O_i)^2}{\sum_{i=1}^N (O_i - \bar{O})^2} \tag{9}$$

$$ME(\%) = MAX|E_i - O_i| \cdot 100 \tag{10}$$

where E_i was the prediction (model-simulated data) and \bar{O} was the true value (observed data), was the mean of the observed data, and N was the number of observations.

Results and discussion

Dielectric characterization of olive mill wastewater-contaminated soil

Figures 2A-A' and B-B' show respectively the experimental ϵ_b vs θ_f and EC_b vs θ_f relationships, obtained for selected β values. As can be observed in Figure 2A-A', in the investigated θ_f domain (*i.e.* $0.05 \leq \theta_f \leq 0.40$), the measured dielectric permittivity of OMW-contaminated soil samples increased overall, as the volumetric fluid content increased. At the same time, for fixed θ_f values, it may be

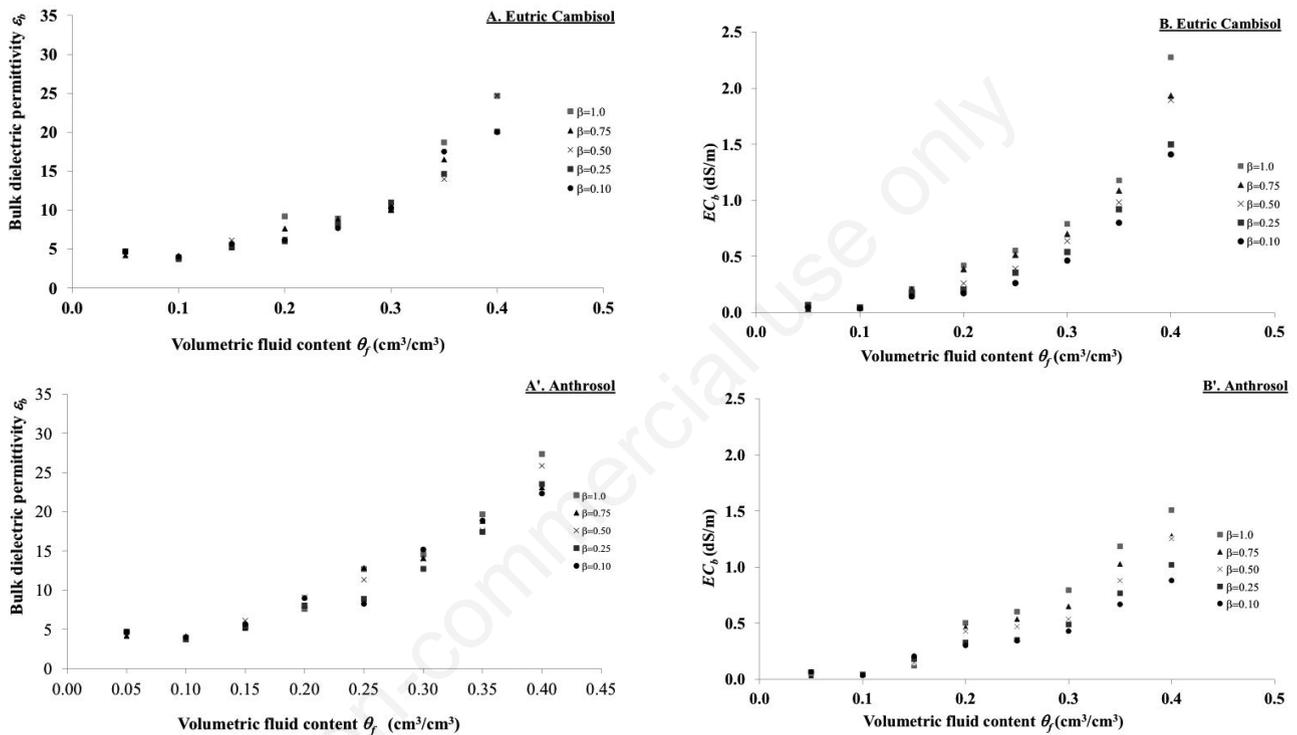


Figure 2. Effect, on the two selected soils, of volumetric fluid content (θ_f) on: A-A') bulk dielectric permittivity (ϵ_b), and B-B') bulk electrical conductivity (EC_b), of soil-water-OMW-air mixtures, for different β values.

Table 3. Combinations of moisture volume (V_w) and olive mill wastewater volume (V_{OMW}) for β and θ_f values.

| θ_f | Volume of fluids (cm^3) | Relative volume of OMW in water: β | | | | | θ_f | Volume of fluids (cm^3) | Relative volume of OMW in water: β | | | | |
|------------|------------------------------------|--|------|------|------|------|------------|------------------------------------|--|------|------|------|------|
| | | 1 | 0.75 | 0.50 | 0.25 | 0.10 | | | 1 | 0.75 | 0.50 | 0.25 | 0.10 |
| 0.05 | V_w | 0 | 13 | 27 | 40 | 48 | 0.25 | V_w | 0 | 66 | 133 | 199 | 239 |
| | V_{OMW} | 53 | 40 | 27 | 13 | 5 | | V_{OMW} | 266 | 199 | 133 | 66 | 27 |
| 0.10 | V_w | 0 | 27 | 53 | 80 | 96 | 0.30 | V_w | 0 | 80 | 159 | 239 | 287 |
| | V_{OMW} | 106 | 80 | 53 | 27 | 11 | | V_{OMW} | 319 | 239 | 159 | 80 | 32 |
| 0.15 | V_w | 0 | 40 | 80 | 120 | 144 | 0.35 | V_w | 0 | 93 | 186 | 279 | 335 |
| | V_{OMW} | 159 | 120 | 80 | 40 | 16 | | V_{OMW} | 372 | 279 | 186 | 93 | 37 |
| 0.20 | V_w | 0 | 53 | 106 | 159 | 191 | 0.40 | V_w | 0 | 106 | 213 | 319 | 383 |
| | V_{OMW} | 213 | 159 | 106 | 53 | 21 | | V_{OMW} | 425 | 319 | 213 | 106 | 43 |

OMW, olive mill wastewater.

noted that the calculated ε_b values overlapped more or less. This means that differences in β (i.e., differences in soil contamination levels) do not affect the dielectric response of the contaminated medium in terms of permittivity. In other words, ε_b was not OMW-sensitive. By contrast, as shown in the graphs in Figure 2B-B', especially in the θ_f range 0.20-0.40, a clear correlation appears between EC_b and θ_f and, for a fixed θ_f , between EC_b and β . Indeed, EC_b values increased with q_f and with β .

Model calibration and validation

In order to confirm the approach adopted, as described in section 3 above, Figures 3A and B show the experimental (colored dots) and inferred (continuous line) β vs $\ln(EC_b)$ relationships for

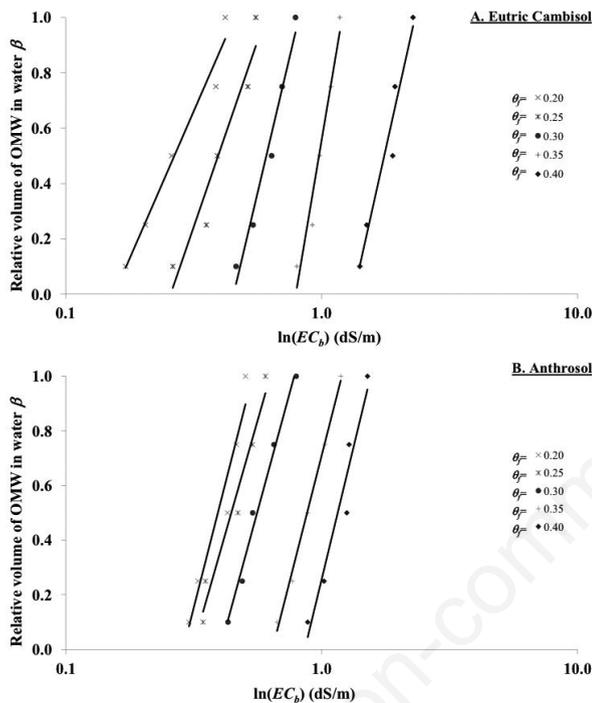


Figure 3. Experimental relationship between bulk electrical conductivity EC_b and the relative volume of olive mill wastewater in water, for constant θ_f values: A) *Eutric Cambisol* and B) *Anthrosol*.

Table 4. Estimated a_c , b_1 , b_2 and b_3 coefficients of β vs $\ln(EC_b)$ relationships at different θ_f values.

| Soil | a_c | b_1 | b_2 | b_3 |
|------------------------|-------|---------|--------|-------|
| <i>Eutric Cambisol</i> | 1.185 | -16.103 | -1.367 | 2.989 |
| <i>Anthrosol</i> | 1.569 | -22.646 | 4.7463 | 1.927 |

different values of the volumetric fluid content (θ_f). For such data, an ANCOVA analysis performed at a significance level of 0.05 confirmed a parallelism among the β - $\ln(EC_b)$ regression lines. As a consequence, a common slope a_c can be assumed for each of the tested soils. Furthermore, as demonstrated by Figure 4A and B, the intercepts b of the different β - $\ln(EC_b)$ relationships can be suitably inferred from a second order polynomial equation (R^2 is 1.0 for the *Eutric Cambisol* and 0.99 for the *Anthrosol*). Coefficients a_c , b_1 , b_2 and b_3 resulting from model calibration are shown in Table 4.

As mentioned above, the model reliability was evaluated by applying the model with the calibrated coefficients to an independent validation dataset. Figure 5 compares the computed (equation 7) and the measured volumetric OMW content. The corresponding statistical indices are reported in Table 5.

Overall, both Figure 5 and Table 5 confirmed the satisfactory agreement of the model predictions with the experimental data: the model efficiency is very close to 1 for both soils. The maximum absolute percentage error and mean absolute error were 8.8% and 3.4% for the *Eutric Cambisol* and 6.5% and 2.8% for the *Anthrosol* respectively.

Considering the complexity of the modelled process, these results are appreciable and validate the scientific consistency of the approach and its general applicability to determine volumetric OMW content in a contaminated medium by means of TDR.

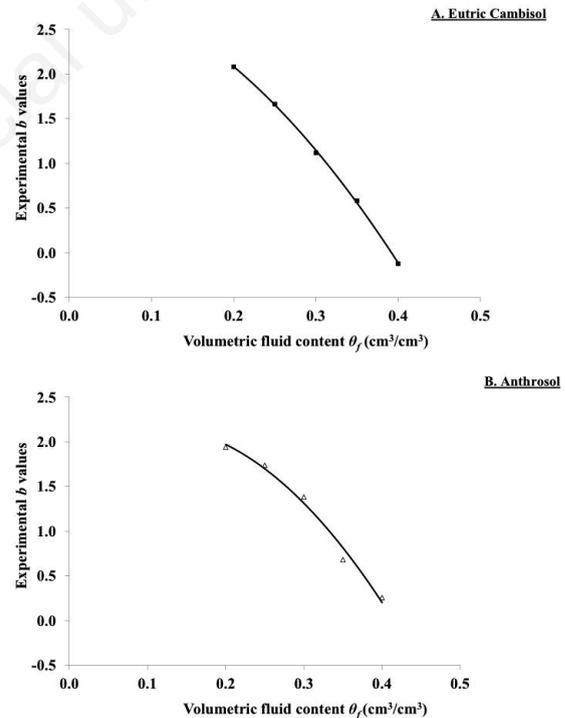


Figure 4. Experimental b values of the β - $\ln(EC_b)$ relationships versus volumetric fluid content (θ_f): A) *Eutric Cambisol* and B) *Anthrosol*.

Table 5. Range of model applicability and: i) mean absolute error (MAE), ii) maximum absolute percentage error (ME), iii) model efficiency (EF), referring to measured and predicted (Equation 7) volumetric OMW content (θ_{OMW}).

| Soil | Range of model applicability | MAE (%) | ME (%) | EF |
|------------------------|--------------------------------|---------|--------|------|
| <i>Eutric Cambisol</i> | $0.20 \leq \theta_f \leq 0.40$ | 3.4 | 8.80 | 0.95 |
| <i>Anthrosol</i> | $0.20 < \theta_f < 0.40$ | 2.8 | 6.53 | 0.96 |

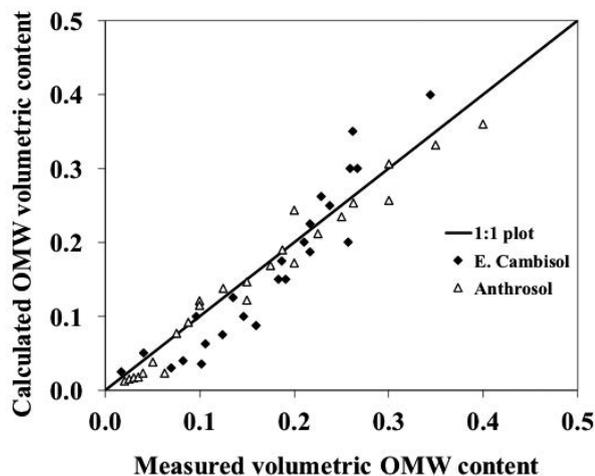


Figure 5. Calculated (Equation 7) versus measured volumetric olive mill wastewater (OMW) content (θ_{OMW}) for the two contaminated soils.

Conclusions

In this study, we conducted a series of laboratory experiments on soil samples exposed to variable degrees of OMW contamination. The measurements of soil bulk dielectric permittivity (ϵ_b) and soil bulk electrical conductivity (EC_b) were performed simultaneously via TDR within each investigated sample. The experimental framework was set up in order to accomplish, as far as possible, a full factorial plan of electromagnetic characterization of the OMW-contaminated soil samples in the $0.05 \leq q_f \leq 0.40$ domain. It was shown that the presence of olive mill wastewater in the soil had a low or null effect on ϵ_b . However, an interesting correlation between θ_{OMW} and EC_b was found. On the basis of the results attained, a dielectric model (Equation 7), which made it possible to quantify the volumetric OMW content, was developed and appropriately validated. This research work can be considered an enhancement in monitoring soil affected by OMW contamination using the time domain reflectometry technique.

The current database should be extended, selecting for example different pedological contexts in other Mediterranean countries with similar environmental problems. Full field-scale tests should also be carried out to evaluate the performance of the proposed model in real field conditions.

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