

STUDY OF HYDROCARBON SATURATION BY GEOPHYSICAL METHODS

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ABSTRACT

The contamination of soil and groundwater by LNAPLs is a problem that has been treated over several years, because, they are used in the industrial worldwide. Its location and behavior in the subsurface has been monitored through invasive methods that disturb the environment and they are costly. In this sense, we integrated a noninvasive hydrogeophysical method as Ground Penetrating Radar (GPR) with chemical analysis (Infrared Spectrophotometry) to localize contaminant distribution and quantify TPH concentrations. For this purpose, we made a 2D hydrogeophysical experiment in a sand box saturated by diesel in which we validated this methodology.

Keywords: LNAPL, TPH, IR, GPR, hydrogeophysics.

1 INTRODUCTION

The contamination of soil and groundwater is produced by human activity mainly from spills, leaks and discharges of industrial processes. This implies a high risk to health and the environment. In this context, the pollution effect is produced by two types of organic and inorganic contaminants. Within the group of organic contaminants there are light non-aqueous phase liquids (LNAPLs) such as gasoline and diesel.

Because of their density and chemical composition, LNAPLs tend to move toward the upper aquifer causing the formation of spots along the same, so making difficult their monitoring. Usually, direct chemical methods such as infrared spectrophotometry (IR) and gas chromatography (GC) are used to quantify the LNAPLs concentrations expressed in Total Petroleum Hydrocarbons (TPHs). Moreover, the indirect geophysical methods usefully applied for hydrogeological characterization and monitoring in contaminated area, are Ground Penetrating Radar (GPR), Electrical resistivity (ER), Induced Polarization (IP), and Self-potential (SP). They are sensitive to physical and chemical subsurface property variations due to the contaminants in the porous media.

The aim of this work is to identify a relationship between LNAPLs saturation degree and the dielectric permittivity by means of an integrated chemical and geophysical approach. In order to estimate the diesel content in groundwater by GPR measurements, a hydrogeophysical laboratory experiment was developed merging GPR and chemical IR data.

2 METHODS

2.1 Hydraulic theoretical background

The two-phase flow numerical model establishes the governing equations and constitutive relationships that define fluid retention and permeability in the natural media. The governing equations for two-phase flow in porous media follow separate mass conservation equations for the wetting and

non-wetting fluids. Water is considered to be the wetting fluid since water favors contact with the solid matrix (i.e. mineral grains making up the aquifer). LNAPL is the non-wetting fluid, which means it has a lower tendency to interact with the solid matrix as compared to water (Charbeneau, 2000). The governing equations for multiphase flow are coupled nonlinear partial differential equations (PDEs). Constitutive relationships are also integrated into the PDEs to account for fluid retention and aquifer permeability. The following equations are based on Mualem (1976) and Van Genuchten (1980). The mass conservation equations for the wetting (w) and non-wetting (nw) fluids, assuming two incompressible fluids, are:

$$\theta_s \frac{\partial S_{e,w}}{\partial t} + \nabla \cdot \left(-\frac{K_0 K_{r,w}}{\eta_w} \nabla (p_w + \rho_w g z) \right) = 0 \quad (1)$$

$$\theta_s \frac{\partial S_{e,nw}}{\partial t} + \nabla \cdot \left(-\frac{K_0 K_{r,nw}}{\eta_{nw}} \nabla (p_{nw} + \rho_{nw} g z) \right) = 0 \quad (2)$$

where θ_s is the porosity of porous medium, S_e is the saturation degree of wetting and non-wetting phase, k_0 is the intrinsic permeability of porous medium [L^2], k_r is the relative hydraulic conductivity of wetting and non-wetting phase [LT^{-1}], η is the dynamic viscosity of wetting and non-wetting phase [$ML^{-1}T^{-1}$], p is the hydraulic pressure of wetting and non-wetting phase [$ML^{-1}T^2$], ρ is the density of wetting and non-wetting phase [ML^{-3}], g is the gravity acceleration [LT^{-2}], and finally z is the depth [L]. Equations (1) and (2) are subject to the constraint:

$$S_{e,w} + S_{e,nw} = 1 \quad (3)$$

This constraint assumes that the void space of the porous media is completely filled by water and/or LNAPL. The saturation of either fluid phase can range from 0 to 1. Capillary pressure (p_c) is the pressure difference between the non-wetting and wetting phase interfaces and is mathematically defined as:

$$p_c = p_{nw} - p_w \quad (4)$$

Capillary pressure results from the density difference between two fluids and is a function of the fluid phase saturations. Effective saturation changes with capillary pressure. This relationship is quantified as:

$$C_{p,w} = -C_{p,nw} = \theta_s \frac{\partial S_{e,w}}{\partial p_c} \quad (5)$$

where C_p is the specific capacity of the wetting and non-wetting phases at a given pressure. In order to simplify the model, Equations (3), (4) and (5) are substituted in Equations (1) and (2), so that the governing equations become:

$$C_{p,w} \frac{\partial}{\partial t} (p_{nw} - p_w) + \nabla \cdot \left[-\frac{k_0 k_{r,w}}{\eta_w} \nabla (p_w + \rho_w g z) \right] = 0 \quad (6)$$

$$C_{p,w} \frac{\partial}{\partial t} (p_w - p_{nw}) + \nabla \cdot \left[-\frac{k_0 k_{r,nw}}{\eta_{nw}} \nabla (p_{nw} + \rho_{nw} g z) \right] = 0 \quad (7)$$

2.2 GPR theoretical background

Ground penetrating radar (GPR) is an electromagnetic technique designed primarily to investigate the shallow subsurface of the earth, building structures, roads, and bridges. GPR utilizes the transmission and reflection of high frequency electromagnetic (EM) waves in the range between 10MHz to 2GHz. The propagation of the radar signal depends on the frequency-dependent electrical properties of the ground. When the radiated energy encounters an inhomogeneity in the electrical and

magnetic properties of the subsurface, part of the incident energy is reflected back to the radar antenna and part is transmitted through the inhomogeneity (Daniels, 2004).

The GPR data are presented as a two or three dimensional depth profile along a single or more scanned traverse line in which the vertical axis corresponds to the two-way travel-time measured in nanoseconds. If the propagation velocity of the electromagnetic waves is known, the depth of the reflector (d) can be determined from:

$$d = \frac{vt_v}{2} \quad (8)$$

where t_v is the two-way travel time, and v is the EM velocity through the subsurface material. The EM wave velocity of propagation in a medium is equal to:

$$v = \frac{c}{\sqrt{k}} \quad (9)$$

where c is the light velocity in free space (0,3 m/ns) and k is the relative dielectric permittivity which is a measure of the capacity of a material to store a charge when an electric field is applied to it relative to the same capacity in a vacuum (Sheriff, 1984).

The GPR is one of the techniques that has seen increasing use in the investigation of both unsaturated and saturated hydrogeology. Greaves et al. (1996) used GPR interval velocities gathered from surface common midpoint (CMP) surveys, and coupled their results with Topp's equation (Topp et al., 1980) to estimate moisture content in saturated sediments. Due to the success achieved in the field of hydrogeology, in recent years GPR was used effectively for the study of large areas to search of anomalies into the electromagnetic behavior attributable to the presence of pollutants. GPR is able to detect the level of pollution in vadose zone and to identify the plume in groundwater (Atekwana et al., 1998; Godio et al. 1998). Furthermore, several researches were conducted aimed at identifying mainly the correlation between soils electromagnetic properties and water and hydrocarbons content. Laboratory experiments have allowed to verify the complex interaction between electromagnetic properties, conductivity and dielectric permittivity (Santamarina et al., 1997). Most of the studies were controlled injections of contaminants in which GPR surveys were performed before, during and following the injections. In addition to controlled injections of LNAPLs there have been several GPR studies of accidental spill sites (Bermejo et al., 1997; Sauck et al., 1998).

In presence of contaminants in the subsurface, it is possible to record changes in the electrical behavior of contaminated rocks and soils. However, for a good understanding of the pollution phenomenon the use of GPR in the analysis and monitoring of contaminants in the environment cannot be separated from knowledge of the chemical and physical dispersed contaminants. Even though, GPR is able to provide relatively high spatial resolution, on the other hand its penetration depth will in many cases be insufficient because of attenuation related to clays or high pore water conductivity.

The physical properties of the most common contaminants have been tabulated in a rigorous manner by Lucius (1992). From the study of these tables has been possible to obtain reference parameters for the characterization of LNAPL used in laboratory. The electrical properties are defined in terms of dielectric permittivity, magnetic permeability, and electrical conductivity. The attenuation of the signal generated by the EM antennas is particularly affected by both the dielectric permittivity and electrical conductivity. For this reason, the estimation of these parameters is very important for the understanding of the investigated problem.

Dielectric permittivity for the GPR frequencies is determined by the physical properties of the materials of the medium, that usually consists of a matrix of air, water, minerals and NAPL. The mechanism that most influences GPR measurement is characterized by displacement of the water ($k=80$) naturally contained in the matrix when is replaced by NAPL characterized by permittivity values much lower ($k=2-10$). In general, when NAPL replaces water in the pore space of rock or soil,

the relative dielectric permittivity of the media decreases, thereby increasing the wave velocity. The electrical properties of rock and soil, containing mixtures of air, water and NAPL have been estimated using volumetric mixing laws such as the complex refractive index model (CRIM) (Birchak et al., 1974) and effective media theory (Sen et al., 1981). The accuracy of quantitative estimates of the LNAPL content affected significantly the specific distribution of the voids and the volume fraction of variable liquid and gaseous substances present in the medium. Overcoming this difficulty is probably the most important challenges in the field of environmental research conducted in geophysics.

2.3 Chemical background

The Spectrophotometry InfraRed (IR) methodology is used to investigate the composition of a sample or to identify a compound. This method analyze the electromagnetic spectrum, i.e. the absorbed infrared spectrum of a material within a whole frequency range of the spectrum. Because each chemical element has its own absorption band or wavelength associated with the energy differences of its different atomic orbitals, knowing the absorbed infrared spectrum it is possible to measure the sample composition. For the TPHs, this method measures the absorption caused by changes in vibration-rotation of the C-H bonds of hydrocarbons. In particular, in our experiment we used the 8440 U.S. EPA (1996) method in a wavelength range of 3200-2700 cm^{-1} and with detection limit of 10 to 600 mg kg^{-1} of TPH.

The quantification is obtained by comparing the sample absorption against a calibration curve made with a reference oil, and in case of more concentrated extracts it is advisable to make the necessary dilutions to obtain measurements of absorbance between 0.1 and 0.8. (Fernández et al., 2006). The TPHs signal in IR method is represented by three characteristic peaks at 2956, 2926 and 2855 cm^{-1} ; however, in this study to calculate the TPHs concentration we only consider the peak at 2956 cm^{-1} . The TPH values are calculated from the extrapolation of the absorbance value obtained for samples within the calibration curve. Values are expressed in mg L^{-1} and converted to mg kg^{-1} of dry soil (DS), considering the amount of soil removed, the amount of solvent that was used to make a solution, and the amount of sample taken from this solution, as follows:

$$TPHs = \frac{(Abs-b)}{m} \quad (10)$$

where $TPHs$ is the Total Petroleum Hydrocarbons (mg L^{-1}), Abs is the absorbance of the sample 2956 cm^{-1} , b is the intercept of the calibration curve and m the calibration curve slope.

This value can be expressed in mg kg_{DS}^{-1} by:

$$TPHs \left(\frac{\text{mg}}{\text{kg}_{DS}} \right) = \left[\frac{TPHs \left(\frac{\text{mg}}{\text{L}} \right) * VT}{FC_1} \right] * \left[\frac{VD}{VE} \right] * \left[\frac{FC_2}{P} \right] \quad (11)$$

where VD is the total volume of solution (dichloromethane), VE is the volume taken from VD and began to evaporation, VT is the volume of tetrachlorethylene where the sample was dissolved or evaporated, FC_1 is the correction factor for the TPH (mg) in VT equal to 1000, P is the weight (g of dry soil extracted equal to $P_H * FH$), FC_2 is the correction factor to obtain mg of TPH in kg of dry solid, P_H is the amount of moisture soil extracted and, finally, FH the correction factor of humidity equal to:

$$FH = 1 - \frac{\text{humidity}(\%)}{100} \quad (12)$$

In this sense, the TPH quantitative analysis with infrared spectroscopy is a relatively fast method for determining the approximate LNAPL amount in the soil, in fact the main advantage of the technique is the simplicity, speed and low cost. However, especially for heterogeneous samples it shows limited accuracy and precision and it does not give information concerning the type of hydrocarbons exist in the sample or the presence or absence of toxic molecules (Weisman, 1998).

3 EXPERIMENTS SET-UP

The experiments were performed at the hydrogeophysical Laboratory of University of Calabria (Cosenza, CS, Italy) in collaboration with the Hydrogeosite Laboratory (Institute of Methodology for Environmental Analysis of CNR, Marsico Nuovo, PZ, Italy). The model is a 2D sand-box in plexiglass with the following dimensions: $100 \times 60 \times 10 \text{ cm}^3$.

On the front of the Sand-Box twenty-one holes with a diameter of 16 mm were drilled, the holes were ordered in three different levels each having seven holes; the first level is located at 15 cm, the second at 30 cm and the third at 45 cm from the bottom. Into holes are embedded samplers of the same material and the ring has double function: as a guide for the set-up a hydraulic seal with the insertion of an O-ring gasket (see Figure 1).

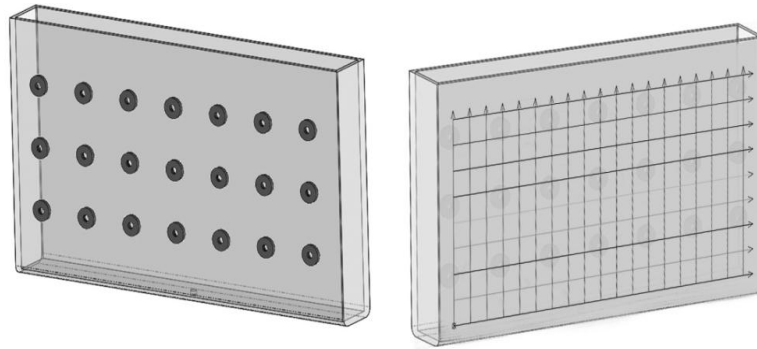


Figure 1. Picture of the 2D sand box in plexiglass. On the left, the front side where twenty-one extraction holes of 16 mm are placed in 3 rows and 7 columns separated by 15 cm from the bottom and each. On the right, the backside of the sand-box where GPR measurements are carried out.

The Sand-box was filled by a natural silicic sand rich of quartz coming from Maranello (Italy), with grain size ranged from 0.15 to 1.2 mm and a characteristic diameter (d_{30}) equal to 0.3 mm. The non-compacted sand had a total porosity of about 0.37 as determined from dry and water-saturated weighting. A hydraulic conductivity of $4.51 \pm 0.23 \cdot 10^{-4} \text{ m/s}$ has been obtained by means of a constant head permeameter tests. In order to avoid the entrapment of air bubbles, the sand was saturated with tap water from the bottom up to a level of around 55 cm. Successively 2000 ml of diesel was injected from the bottom by means of a peristaltic pump.

During the experiment, GPR measurements on the backside of the sand-box were performed. Data were acquired in reflection mode and in continuous by means of the GPR SIR-3000 (GSSI-Instrumentation), with antenna with survey-wheel incorporated. Due to the limited size of the box, the experiments were performed by a high resolution antenna with central frequency of 2Ghz. The used antenna has a survey wheel system, which is important to have the position of each recorded data. The data were acquired along parallel and perpendicular profiles spaced 5 cm from each other. GPR measurements were carried out in saturated conditions before and after the injection, in order to have reference data during the unpolluted state. The post-processing data allowed to obtain the best signal/noise ratio and to estimate the distribution of the permittivity values by EM velocity analysis. The permittivity distribution maps allowed to highlight clearly the contaminated area during the hydrocarbon transfer from the bottom to the top. The dielectric constant of a porous material is highly sensitive to its volumetric water content θ_w because the relative dielectric constant of water is much higher than the dielectric constant of most minerals forming a rock matrix and of air (Topp et al., 1980). Topp et al. (1980) identified an empirical relationship between the dielectric constant k and the volumetric water content θ_w :

$$\theta_w = -5.3 \times 10^{-2} + 2.92 \times 10^{-2} k - 5.5 \times 10^{-4} k^2 + 4.3 \times 10^{-6} k^3 \quad (13)$$

Moreover the water content (θ_w) is equals to the product of porosity (Φ) and water saturation (S_w).

$$\theta_w = \Phi S_w \quad (14)$$

In our case of study, using Topp's empirical relationship, the GPR data allowed to estimate the LNAPL saturation degree using equations 13 and 14.

The three different datasets were processed by removing the effect connected to the coupling between the antenna and the box and the information contained in radargrams outside the area of interest. From processed-data, "significant traces" (one profile every 5 cm) were extracted. They describe the propagation mode on the high-frequency electromagnetic waves generated in the sand-box. Analyzing the propagation velocity of the waves and knowing the sand box geometry it was possible to estimate the relative dielectric permittivity.

When the steady-state conditions were reached, twenty-one samples were collected, in order to chemically quantify the hydrocarbon content. All samples were analyzed according to the methods 3541 and 3540C for extraction, and 8440 U.S. EPA (1996) for determination of Hydrocarbons (g/kg) of TPH by Infrared Spectrophotometry. The validation of the results was previously performed by means of three benchmarks with known concentrations of hydrocarbons (0.5, 1, 2 mL) giving an error of 2.5% in concentration values: this result shows the effectiveness of the IR method for quantifying TPH. The chemical analysis has the aim to obtain the distribution of the hydrocarbon content and to validate the LNAPL saturation degree determined by Topp's formula from the GPR data.

4 RESULTS AND DISCUSSIONS

The first GPR acquisition was performed at the end of water saturation phase, where it was possible to identify a mean baseline value of the dielectric constant $k > 20$ and an homogeneous distribution of EM waves velocities. The dielectric permittivity map, shown in Figure 2, was obtained using a geostatistical approach based on kriging contouring. In the figure 2 it is possible to notice a decrease in the values of k near the bound borders of the box where there are minimum effects due to the bulging box.

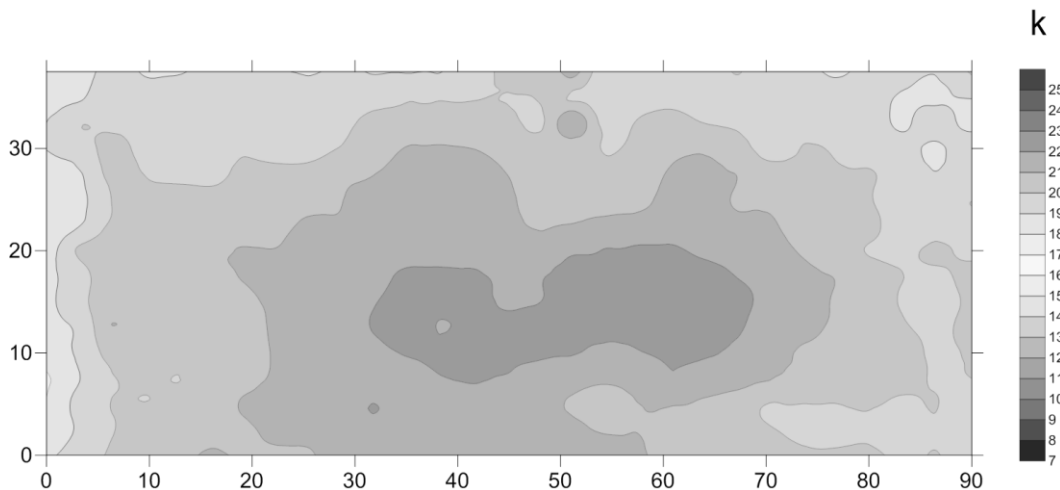


Figure 2. Permittivity map of investigated Sand-box in water saturated conditions.

The figure 3 shows the permittivity map obtained from GPR data acquired during (fig. 3a) and after six day (fig. 3b) the fuel-injection (diesel-LNAPL).

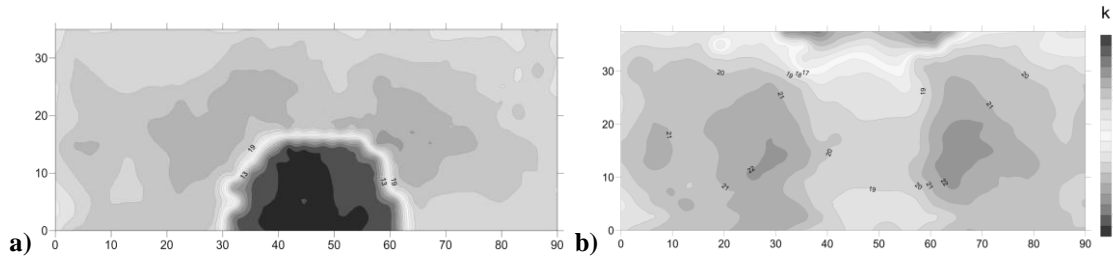


Figure 3. a) Permittivity map during fuel infiltration, b) Permittivity map after six days from diesel infiltration

To better highlight the variations induced by the contaminant, a “contrast map” (fig. 4.b) has been realized where the differences in dielectric behavior between the two scenarios just discussed have been reported. The use of this type of representation allowed to eliminate the effects induced by the bulging of the box.

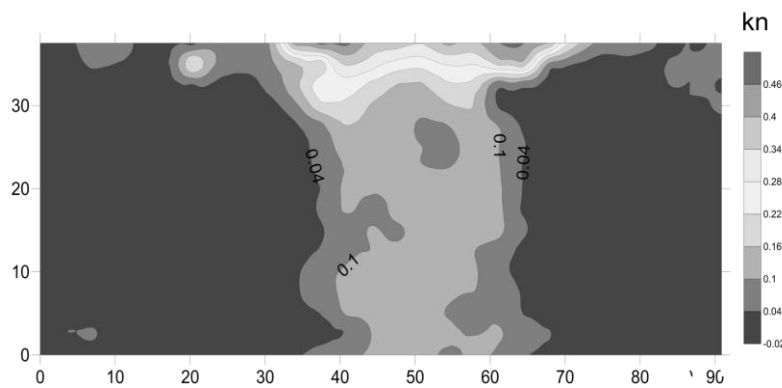


Figure 4. Interpolated contrast permittivity map between the scenarios in water saturated conditions and the scenarios obtained after six-day from diesel infiltration.

The final step consisted in the implementation of Topp’s equation (13) to quantify LNAPL saturation degree from measured dielectric permittivity values. The results are shown in figure 5.

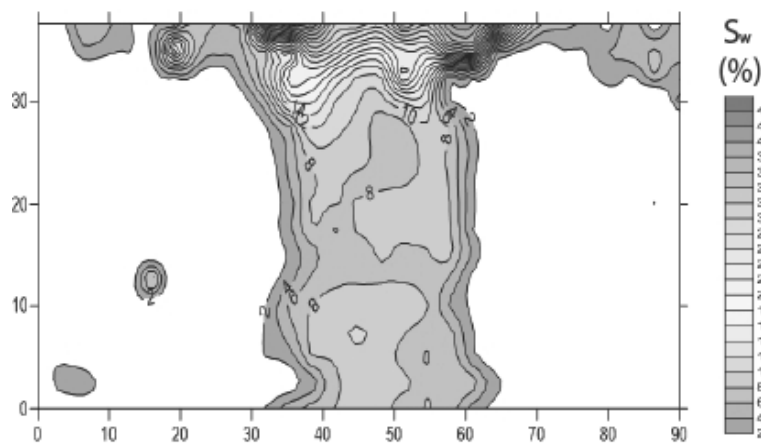


Figure 5. Contrast map expressed in percent saturation S_w .

Results are extremely interesting and allow a clear distinction between an area characterized by low dielectric permittivity values (and thus high speed of EM waves propagation), where diesel saturation is probably greater, as well as two lateral areas and a shallow layer where great variations in the electromagnetic behavior of sand are not detectable, due to absence of contaminant dispersion. In order to find the correlation between the dielectric permittivity measures and the LNAPL degree of

saturation in the ground, a series of sampling of the sand were carried out through the holes, with the samplers previously described. The samples located in the middle zones of the box were characterized by higher LNAPL concentration values of 31,664 ($\text{g kg}_{\text{DS}}^{-1}$), while, in the lateral zones of the box we found lower TPH concentration of 0,109 ($\text{g kg}_{\text{DS}}^{-1}$). These values converted in the form of TPH saturation are equal respectively to 11.86% and 0.2%, so we found a good correlation between LNAPL saturation degree obtained by chemical analysis and geophysical data.

5 CONCLUSIONS

The reliability of GPR as a tool to detect near-surface contaminants is illustrated through a test, representing by diesel fuel infiltration in a saturated soil, carried out under controlled conditions in a hydro-geophysical laboratory of the University of Calabria. The several tests carried out show that GPR may provide a guess of NAPL saturation degree in the saturated zone. In fact, results show significant changes in the responses of electromagnetic measurements in presence of fuel contamination. Moreover, GPR can provide high-density, quantitative data for investigations of vadose zone contaminant hydrogeology. The experimental results show the capability of GPR to identify the contaminant dispersed in the subsoil.

On the other hand, chemical analysis carried out on samples can validate the technique of GPR in the study of infiltration process into the soil as well as provide basic data for the construction of empirical relationships to the estimation of the real amount of contaminants in the subsurface. For this reason, the future of this research will be the evaluation of the capability to estimate the real content of LNAPL from the values of permittivity by geophysical techniques.

ABBREVIATIONS

LNAPLs Light Non-Aqueous Phase Liquids

TPHs Total Petroleum Hydrocarbons

C-H Functional group: carbonyl

IR InfraRed spectroscopy

GPR Ground Penetrating Radar

U.S. EPA U.S. Environmental Protection Agency

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