



## Research article

# Lab-scale investigation on remediation of diesel-contaminated aquifer using microwave energy



Pietro P. Falciglia<sup>a, \*</sup>, Riccardo Maddalena<sup>a</sup>, Giuseppe Mancuso<sup>b</sup>, Valeria Messina<sup>a</sup>, Federico G.A. Vagliasindi<sup>a</sup>

<sup>a</sup> Department of Civil Engineering and Architecture, University of Catania, Via S. Sofia, 64, 95125 Catania, Italy

<sup>b</sup> Department of Civil, Environmental and Mechanical Engineering, University of Trento, Via Mesiano, 77, 38123 Trento, Italy

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## ABSTRACT

Aquifer contamination with diesel fuel is a worldwide environmental problem, and related available remediation technologies may not be adequately efficient, especially for the simultaneous treatment of both solid and water phases. In this paper, a lab-scale 2.45 GHz microwave (MW) treatment of an artificially diesel-contaminated aquifer was applied to investigate the effects of operating power (160, 350 and 500 W) and time on temperature profiles and contaminant removal from both solid and water phases. Results suggest that in diesel-contaminated aquifer MW remediation, power significantly influences the final reachable temperature and, consequently, contaminant removal kinetics. A maximum temperature of about 120 °C was reached at 500 W. Observed temperature values depended on the simultaneous irradiation of both aquifer grains and groundwater. In this case, solid phase heating is limited by the maximum temperature that interstitial water can reach before evaporation. A minimal residual diesel concentration of about 100 mg kg<sup>-1</sup> or 100 mg L<sup>-1</sup> was achieved by applying a power of 500 W for a time of 60 min for the solid or water phase, respectively. Measured residual TPH fractions showed that MW heating resulted in preferential effects of the removal of different TPH molecular weight fractions and that the evaporation-stripping phenomena plays a major role in final contaminant removal processes. The power law kinetic equation shows an excellent fit ( $r^2 > 0.993$ ) with the solid phase residual concentration observed for all the powers investigated. A maximum diesel removal of 88 or 80% was observed for the MW treatment of the solid or water phase, respectively, highlighting the possibility to successfully and simultaneously remediate both the aquifer phases. Consequently, MW, compared to other biological or chemical-physical treatments, appears to be a better choice for the fast remediation of diesel-contaminated aquifers.

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## 1. Introduction

Hydrocarbon-pollution in soil and groundwater is usually related to accidental leaks during handling, transport or storage activities, all of which constitute a serious environmental issue worldwide (Careghini et al., 2015; Mena et al., 2015). In most cases, released petroleum products form non-aqueous phase liquids (NAPLs), which then become the long-term source of aquifer contamination. Among hydrocarbons, diesel fuel is a complex mixture of saturated and polycyclic-aromatic-hydrocarbons (PAHs) which, due to its extensive use worldwide, is a permanent source of

contamination (Jagtap et al., 2014; Pazos et al., 2012; Silva-Castro et al., 2013; Tatáno et al., 2013).

It is well known that conventional methods such as pump-and-treat are ineffective for treating the contamination source or in removing total petroleum hydrocarbons (TPHs) from the aquifer due to the hydrocarbon low solubility in water and the heterogeneity of the sites (Liang et al., 2011). Regarding alternative methods, there are a number of in situ biological/chemical techniques such as air- or bio-sparging (AS/BS), in situ chemical oxidation (ISCO) or permeable reactive barriers (PRBs). In situ biodegradation is classified as a cost-effective and eco-friendly treatment to remove TPHs from contaminated aquifers due to its ability to produce minimal amounts of toxic secondary by-products (Hunkeler et al., 2002). However, the effectiveness of the process depends on many factors such as contamination nature, presence of microorganisms able to

\* Corresponding author.

E-mail address: [ppfalcic@dica.unict.it](mailto:ppfalcic@dica.unict.it) (P.P. Falciglia).

biodegrade the contaminants or the absence of toxic substances, all of which generally result in very long remediation times (Garoma et al., 2008). Moreover, in situ biodegradation is neither suitable in cases of high levels of hydrocarbon contamination or free phase due to its toxicity to microorganisms (Oya and Valocchi, 1998). ISCO involves the application of various combinations of agents such as  $O_3$  or  $H_2O_2$  and is able to oxidize a wide variety of organic pollutants into intermediates and end-products. Despite its advantage of a rapid reaction rate, the chemical oxidation process may create some toxic or mobile by-products that can potentially limit its in situ application especially in the presence of groundwater (Kim and Lee, 2012). PRBs have received a great deal of attention as an innovative method for in situ contaminated groundwater remediation (Bortone et al., 2013; Careghini et al., 2015; Xin et al., 2013); however, these allow for only a passive plume control and are unsuitable for the direct treatment of the contamination source or the remediation of small sites such as petrol stations (Liang et al., 2011). Therefore, it is necessary to widen the boundaries related to alternative remediation methods for TPH removal from aquifers and to diesel fuel in particular (Fan et al., 2014).

In recent years, microwave (MW) ( $f = 2.45$  GHz) heating remediation has attracted great attention in the environmental field. MW energy adsorbed by a contaminated matrix is converted into heat and, consequently, into a temporal increase ( $\Delta T \Delta t^{-1}$ ,  $^{\circ}C \text{ min}^{-1}$ ) of the matrix temperature as defined in the Eq. (1) (Falciglia et al., 2013)

$$\frac{\Delta T}{\Delta t} = \frac{P}{c_p \cdot \rho} = \frac{\omega \cdot \epsilon_0 \cdot \epsilon'' |E|^2}{c_p \cdot \rho} \quad (1)$$

where  $P$  is power absorbed per unit volume ( $W \text{ m}^{-3}$ ),  $\omega$  is the angular frequency,  $\epsilon_0$  is the permittivity of free space ( $8.85 \cdot 10^{-12} \text{ F m}^{-1}$ ),  $E$  is the internal electric field ( $V \text{ m}^{-1}$ ),  $c_p$  is the heat capacity of the matrix ( $KJ \text{ kg}^{-1} \text{ }^{\circ}C^{-1}$ ) and  $\rho$  is its density ( $kg \text{ m}^{-3}$ ).  $\epsilon''$  is defined as dielectric loss factor, and it represents the ability of a material to convert the MW energy into heat. MW energy was shown to be effective as a remedial alternative for contaminated soils (Falciglia et al., 2013; Robinson et al., 2012), sludge (Barba et al., 2012; Tyagi and Lo, 2013), drilled cuttings (Pereira et al., 2014), or electric arc furnace dust (Al-harrahseh et al., 2014).

MW treatments were reported to be efficient in a short time for the removal of a large number of volatile and semi-volatile contaminants such as polychlorobiphenyls (PCBs) (Gomes et al., 2013), hexachlorobenzene (HCB) (Yuan et al., 2006), polycyclic aromatic hydrocarbons (PAHs) (Robinson et al., 2009), crude oil (Li et al., 2009), antibiotics (Lin et al., 2010) or diesel-fuel (Falciglia et al., 2013; Falciglia and Vagliasindi, 2014, 2015a). Several studies, aimed at investigating pilot-scale in situ or ex-situ MW application, have been recently performed by Barba et al. (2012), Chien (2012) and Buttress et al. (2016). The overall advantages of microwave heating are: short time, effective and homogeneous heating, high flexibility, improved ease of risk control, automatic control of the heating processes (Benedetto and Calvi, 2013). In fact, MW heating is based on the direct interaction of the microwave with the contaminated matrix. This consequently allows conventional heating limitations to be overcome, which in turn leads to a reduction of treatment time, risk of further contamination and costs (Falciglia and Vagliasindi, 2014).

In addition, as the amount of water increases, so does MW removal efficiency due to its MW absorbing features and the possibility to activate contaminant-stripping phenomena (Falciglia et al., 2013; Robinson et al., 2014). This highlights the possibility of reaching a high MW absorbing phase for a contaminated aquifer

for which water is highly present. Despite the MW heating ability and current knowledge about its environmental implications, studies on the potential in situ MW applicability for the treatment of hydrocarbon-contaminated aquifers have not been undertaken. The necessity of the simultaneous treatment of both solid (grains of the aquifer skeleton) and interstitial water (groundwater) phases, makes the problem more complex, this also being due to the different abilities of the two aquifer phases to absorb and convert MW energy as well as their physical interactions with contaminants. This study was performed to fill this knowledge gap.

The main objective of the work is to study the contaminant removal processes and the potential MW treatability of an aquifer contaminated with diesel fuel by assessing the influence of power and heating time on the temperature profiles and the TPH fraction contaminant removal kinetics of solid and water phases. Lab-scale experiments were conducted with the purpose of fulfilling the objective.

## 2. Materials and methods

### 2.1. Materials

Diesel fuel (Esso, Italy) was used to artificially contaminate the lab-scale aquifer. Contaminant properties are reported in Falciglia and Vagliasindi (2015a). All chemicals used in this work were of analytical reagent quality. n-Hexane ( $C_6H_{14}$ , purity 99%) was purchased from Merck KGaA (Darmstadt, Germany). The lab-scale aquifer was simulated using a model silica sand (0.05–1.00 mm) free of anthropogenic contamination (Table 1) and deionised water. Activated carbons used for the volatile compounds (VOCs) capture system present in the experimental setup were supplied by Norit Italia S.p.A. (Italy).

### 2.2. Contamination procedure

The contamination procedure was performed by mixing a diesel-hexane solution with selected sand samples. Sand and pollutant solution were shaken for 48 h using an orbital shaker, then the solvent was removed in 1 h applying a slight vacuum by means a rotary evaporator. The contaminated sand was kept in a closed vessel and stored for 1 month, then was saturated with deionised water and stored in a dark room at  $4 \text{ }^{\circ}C$  for 4 months in

**Table 1**  
Properties and characteristics of selected medium sand.

Parameter	Method	Value
Mineral		Silica sand
Texture ( $\mu\text{m}$ )		50–1000
pH	ASTM D4972-13 <sup>a</sup>	8.73
Bulk density ( $\text{g cm}^{-3}$ )	ASTM D7263-09 <sup>b</sup>	1.42
Specific surface area ( $\text{m}^2 \text{ g}^{-1}$ )	EGME <sup>c</sup>	2.11
Organic matter ( $\text{g kg}^{-1}$ )	UV-VIS <sup>d</sup>	0.88
Specific heat capacity ( $\text{KJ kg}^{-1} \text{ }^{\circ}C^{-1}$ )	ASTM E1269-11 <sup>e</sup>	932
Dielectric constant ( $\epsilon'$ )	cavity perturbation <sup>f</sup>	7.21
Loss factor ( $\epsilon''$ )	cavity perturbation <sup>f</sup>	0.78

<sup>a</sup> ASTM D4972-13. Standard Test Method for pH of Soils.

<sup>b</sup> ASTM D7263-09. Standard Test Methods for Laboratory Determination of Density (Unit Weight) of Soil Specimens.

<sup>c</sup> Simplified Ethylene Glycol Monoethyl Ether (EGME) procedure for assessment of soil surface area (Cihacek and Bremner, 1979).

<sup>d</sup> UV-VIS Method - Test procedure for determining organic matter content in soils (Harvey et al., 2010).

<sup>e</sup> ASTM E1269-11. Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry.

<sup>f</sup> Cavity perturbation method for dielectric properties measurements (Sheen, 2005).

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