

An Overview on Microwave Heating Application for Hydrocarbon-contaminated Soil and Groundwater Remediation

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Abstract

In the last decades, the world's dependence on oil and the growing demand for its final products have been caused a massive amounts of soils and water contaminated with hydrocarbon compounds, including fuel and petrochemical products. This resulted in the search of cost-effective techniques for remediation applications. Several chemical-physical or biologic treatments have been proposed to decontaminate hydrocarbon-polluted articles, however these alternatives may be ineffective, expensive or too lengthy. Recently, microwave (MW) heating has been identified as a potential tool for hydrocarbon-polluted soil and groundwater remediation due to several advantages including simplicity, safety, flexibility, short treatment times and low risk. The present work reports MW heating theoretical background and the related techno-economic features.

Keywords: Hydrocarbon; Petro-chemical; Chemical-physical; Thermal desorption; Groundwater.

Introduction

The world's dependence on oil has created in the last decades a continuous and growing demands for hydrocarbon products [1]. Their improper management or accidental spills have caused a discharge in the environment of massive amounts of organic contaminants in soils, also resulting in groundwater contamination phenomena [2]. Contamination process is often dynamic, hidden, cumulative and not reversible. Generally, the common volatile and/or soluble organic contaminants include aromatic compounds, polychlorinated organic compounds, herbicides and pesticides. Most of these contaminants are low-soluble, toxic, mutagenic and even carcinogenic, and may be of great risk to human health and ecosystems [3]. In recent years, a variety of chemical-physical or biologic treatments have been investigated to remediate hydrocarbon-polluted soils and groundwater [4-6]. However, these techniques have been often demonstrated as ineffective, expensive or too lengthy [5], while literature reports that thermal technologies could be very effective due to their high versatility, removal efficiency and required time [7-10]. On the other hand, they may be expensive especially due to fuel costs, making the need of novel alternative cost-effective remedial solutions a key factor in the territory restoration strategies. Since the late 1990s, microwave (MW) heating has been proved to be capable of remediating various types of hydrocarbon contaminated-soils or organic by-products [11-13], whereas, more recently it has been successfully tested for in situ groundwater treatment. The great attention in MW application in environmental field depends on its high potential to significantly reduce treatment times and costs due to the direct interaction of electromagnetic waves with the contaminated matrix and their ability to overcome the heat transfer limitations. In fact, heating time is about three orders of magnitude lower than with conventional heating [14]. Other advantages are the possibility to achieve it, [13] told that homogeneous heating of the contaminated materials; a low energy consumption jointly with short remediation times, a direct and instantaneously controlling of the power-temperature response and a selective heating in the presence of high-polarity (dielectric) contaminants.

Mechanisms of Organic Contaminant Removal by MW Irradiation

Microwave (MW) is a term loosely applied to those radio frequency wavelengths which are sufficiently short to exhibit some

of the properties of light. MWs are a part of the electromagnetic spectrum occurring in the frequency range of 300 MHz to 300 GHz with wavelengths between those of infrared radiation and radio-waves. MW power irradiation of dielectrics is nowadays well recognized and extensively used as an exceptionally efficient and versatile heating chemical processes, such as polymerization, organic synthesis, ceramics processing, and plasma chemistry. In environmental field, MWs ability to heat rapidly and selectively the contaminated matrices leads to the possibility of significant energy savings and the need of much smaller process equipment by which strictly control in the automatic the implementation of heating processes suggested [14,15]. The remediation process is based on the mechanism of partial dissipation of the MW electromagnetic field energy into the irradiated matrix and its conversion into the heat needed for the activation of the organic contaminant removal, mainly thermal desorption. Differently to conventional thermal desorption of solid matrix, the soil temperature increase depends mainly on conduction process, whereas in the case of MW radiation, the penetration of the generated alternating electric field into the contaminated soil induces the rotation of the dipoles of polar or semi-polar substances constituting the soil mineral or present in its surface or surrounding space such as water and hydrocarbon-contaminants. The intermolecular friction generated by ipole rotation results in the generation of heat. Specifically, the internal electric field (E) variation, generated by MW irradiation, into the contaminated region decreases with the distance from the irradiating source (d), and its dependence on d (m) is expressed by Eq. 1 according to the law of Lambert and Beer [16,17].

$$E_d = E_0 \cdot e^{-\frac{d}{D_p}} \quad (1)$$

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Where: E_0 is the incident electric field ($V\ m^{-1}$); D_p is the penetration depth (m); it represents the ability of the electromagnetic waves to penetrate into the medium. In particular, D_p is defined as the distance from the emission point at which E drops to 0.37 from its value at the emission point. For low loss dielectric materials (i.e.: soil) ($\epsilon''/\epsilon' \ll 1$), D_p is given by the simplified form expressed in the Eq 2

$$D_p = \frac{\lambda_0}{2\pi} \frac{\sqrt{\epsilon'}}{\epsilon''} \quad (2)$$

Where λ_0 is the wavelength of the irradiation (m), whereas ϵ' (-) and ϵ'' (-) are the real part (dielectric constant) and the imaginary parts (dielectric loss factor) of the complex permittivity, respectively. The power Q dissipated into heat per unit of volume depends on the frequency of the applied electromagnetic field and the dielectric and thermal properties of the medium. The dissipation is assessable by Falciglia et al.[7],

$$\dot{Q} = \frac{1}{2} \omega \epsilon_0 \epsilon'' |E_{\max}^2| = \omega \epsilon_0 \epsilon'' |E|^2 \quad (3)$$

where

ω is the angular frequency ($\omega = 2\pi f$); 85

ϵ_0 is the permittivity of free space ($8.85 \cdot 10^{-12}\ F\ m^{-1}$)

E_{\max} is the electromagnetic field peak value ($V\ m^{-1}$);

E is electromagnetic field effective value ($V\ m^{-1}$).

Consequently, the increase in temperature (T) with time (t) ($^{\circ}C\ min^{-1}$) can be quantified by the Eq. 4, as 89 follows Falciglia [7]

$$\frac{\Delta T}{\Delta t} = \frac{P}{c_p \rho} = \frac{\omega \epsilon_0 \epsilon'' |E|^2}{c_p \rho} \quad (4)$$

where c_p is the heat capacity of the irradiated medium ($KJ\ kg^{-1}\ ^{\circ}C^{-1}$) and ρ is its density ($kg\ m^{-3}$). In particular, the dielectric constant ϵ' represents the electric energy storage capacity of the medium, while the dielectric loss factor ϵ'' can be considered as the ability of the medium to convert electromagnetic energy into heat due to the dielectric polarization of the particles in an alternating electric field. Substances which exhibit a large value of loss factor are good microwave absorbers, whereas substances whose loss factor is close to zero can be considered to be microwaving transparent Metaxas [18]. MWs are highly absorbed by materials with a high dielectric loss factor (absorbing), while passing through the low loss (transparent) material, resulting in a selective, uniform and rapid heating. For most hydrocarbon contaminants such as diesel or gasoline, which are classified as semi-polar compounds, with ϵ' and ϵ'' values similar to those of soils, the main removal mechanism is the contaminant thermal desorption and it depends on the final temperature that the contaminated matrix can reach with reference to the contaminant boiling point. Soil moisture content also increases the removal efficiency due to the water high dielectric properties and, consequently, its ability in energy absorbing Falciglia [19]. Furthermore, in the case of irradiation of compacted matrices, the increase of the temperature (T) also depends on the conduction phenomena, as expressed by the Eq.5 [7]

$$\rho c_p \frac{\partial T}{\partial t} = -k \nabla^2 T + Q \quad (5)$$

where k is the thermal conductivity ($W\ m^{-1}\ ^{\circ}C^{-1}$). Overall, hydrocarbon compound removal could be due to four different mechanisms: (i) vaporization due to MW remediation of soils contaminated with low polar compounds where treatment temperature is higher than the contaminant boiling point. The second process refers to the disintegration of the original contaminant molecules and the production of by-products as observed for example in irradiating process of PAHs [20] or PAH derivatives [21]. On the other hand, selective heating/vaporization vaporization occurs when a significant hydrocarbon removal takes place even if the global soil temperature is significantly lower than the contaminant boiling point. Finally, in the presence of water as soil moisture, the soil temperature increase induces the physical process of evaporation-contaminant stripping phenomena. This last mechanism can be relevant in irradiation treatment of hydrocarbon contaminated aquifers, which are systems generally characterized by more complex dynamic mechanisms. The simultaneous treatment of both solid (grains of the aquifer skeleton) and interstitial water (groundwater) phases, makes the problem more complex, this also due to the different abilities of the two aquifer phases to absorb and convert MW energy into heat [15]. In this case, it was shown that, although the total mean temperature of the irradiated matrix influences the contaminant removal process, in the presence of a high water content, it does not represent the principal factor in the remedial process and that residual contaminant concentration does not strictly depend on the maximum temperature that the system can reach. When aquifer grains and groundwater are mixed together and irradiated by MW, grain temperature are cooled down by interstitial water, however grains are able to absorb concentrated MW energy and act as temperature "hot spot". The main removal mechanism is the water evaporation that in vapour phase produces contaminant stripping phenomena. MW energy adsorbed by solid grains and interstitial water results in a global increase of the aquifer temperature leading to the evaporation of contaminants adsorbed onto the solid matrix and dispersed in water jointly with dissolved hydrocarbon. Hydrocarbons, initially dissolved in water, are removed with vapour whereas different hydrocarbon fractions, initially adsorbed onto solid phase, were first moved into water and after removed by evaporation phenomena. The evaporation of the lightest TPH fractions produced a solubilisation and a consequent extraction/removing of the heaviest fractions. Hence, lighter hydrocarbon fractions act as solvents for those that are heavier. In addition, the presence of a vapour stream leads to an increase of vapour pressure resulting in a reduction of the temperature needed for the \contaminant desorption and consequently in an increase in removal efficiency [15].

Contaminant Removable and Parameters Influencing the MW Removal Effectiveness: Findings from Lab-scale Experiments

In the last few years, several experimental works on contaminated soil remediation by MW irradiation have been performed in order to understand the fundamentals of the treatment of hydrocarbon-contaminated soils or to investigate the effectiveness of the remedial process. First studies that proposed MW remediation technique as a promising treatment of hazardous wastes were proposed by Dauerman [22], respectively. Removal of PCBs from contaminated soil was performed by several researchers. Liu et al. investigated the effect of a 10 min-MW remediation at 750 W on 20 g PCB-polluted soil samples. They showed an increase in contaminant removal with increasing soil moisture content, to a maximum value close to 100% at a moisture content of 20% (dry basis). Huang [23] reported a

maximum removal efficiency of ~95%, for a soil polluted by PCBs at 5 mg kg⁻¹ and treated at 800 W for a period of 45 min. Removal efficiency of crude oil of ~95% was observed for a 15 min at 800 W treatment enhanced by different microwave absorbers such as activated carbon powder or graphite fibres. Yuan et al. investigated the remediation of soil contaminated with hexachlorobenzene (HCB) using a domestic microwave oven and powdered MnO₂ as a microwave absorber. Their results showed that a complete removal of HCB was obtained with 10 min microwave treatment. Similar results in terms of removal efficiency were also obtained by Kawala [24] in a pilot-scale study for the remediation of a TCE polluted soil where a microwave power of 600 W was supplied intermittently for 75 h. After the irradiation, the contaminant concentration decreased from 5000 - 22300 mg kg⁻¹ to 8 - 29 mg kg⁻¹, confirming the possibility of the use of microwave heating as a remediation technique of volatile and semi-volatile-compound polluted sandy soils, and that the use of low power generators for the supply of microwave energy may help to reduce the costs of the full-scale remediation interventions. In the case of diesel-contaminated soils, Falciglia [19] reported a maximum soil temperature achievable of 260 °C corresponding to contaminant removal of ~95% for soil samples treated at 1000 W for 60 min. Microwave treatment was also shown to be efficient in a short time for the remediation of soil polluted by PAHs Robinson, PCPs by Di [25], Lin [26]. Antibiotics, oils, the presence of water into the soil as soil moisture was shown to significantly increase the final temperature reachable during MW heating, and consequently increase the contaminant removal efficiency. Falciglia [19] reported that a significant difference in soil temperature up to 40 °C was observed between soils with 0 and 12% water content, considering a 1000 W-application. This specific behaviour was shown to be dependent on the dielectric properties of the centric properties than for dry soils. Furthermore, the presence of a vapour stream leads to an increase of vapour pressure resulting in a reduction of the temperature needed for the contaminant desorption and consequently in an increase in removal efficiency. Similarly to the importance of soil moisture, also soil texture has been shown to play a major role in contaminant removal processes, specifically, under two major points: the maximum soil temperature achievable and contaminant adsorption/desorption phenomena occurring during irradiation Falciglia [7,16]. MW penetration into the soil and soil temperature variation strictly depend on the dielectric and thermodynamic properties of the irradiated soils that, in turn, significantly change depending on the texture of the soil. Specifically, ϵ' and ϵ'' values increased with increasing the soil texture having a significant effect on the temperature variation. On the other hand, contaminant thermal desorption also depend on different contaminant adsorption processes that are strictly influenced by the specific surface area, lowest for sandy soil and highest for clayey ones. Difference in dielectric and thermodynamic properties results in a significant variation of final temperature (T) that was shown to follow the order: medium sand > fine sand >> silt > clay) Falciglia [16]. Specifically, a marked difference of temperature in a wide range 24-65°C was observed between sandy and fine texture soils depending on the operating power applied during the treatment. It is important to highlight how soil texture influences in a different way MW and conventional thermal heating processes. It was shown that when different soils were heated using conventional ex-situ thermal heating (T oven=100-300°C) the highest soil temperature was reached in clayey soil at every temperature of treatment. This due to conduction and convection represent the main heat transfer processes occurring during the treatment Falciglia [16]. Lab-scale experiments on hydrocarbon contaminated aquifer treatment by MW were recently performed by Falciglia [15]. Results revealed that a maximum TPH removal of 88 or 80% was observed for

the W treatment of the solid or water phase, respectively. Specifically, a minimal residual diesel concentration adsorbed on the solid phase of about 100 mg kg⁻¹ was achieved by applying a power of 500 W for a treatment time of 60 min, whereas the contaminant concentration was only halved (about 400 mg kg⁻¹) by using a power of 160 W. In the case of the water phase treatment, the application of a 500 W power for treatment times higher than 5 min resulted in a significant reduction of contaminants in water from 575 to about 100 mg L⁻¹. Overall, MW heating resulted in preferential effects of the thermal treatment on the removal of different TPH molecular weight fractions and their moving between the two phases.

Ex-situ and in situ technologies for soil and groundwater remediation

Technologies for MW heating application include both ex-situ and in situ applications. A schematic ex-situ MW treatment plant generally includes that polluted soil is loaded into a hopper and by means of a screw belt passes through the cavity of the plant where it is cleaned by MW irradiation (Figure 1) Buttress and Falciglia [17,27]. The volatile organic compounds (VOCs) produced are treated in a dedicated off-gas treatment line. The treating capability and the effectiveness of MW penetration and contaminant removal depend on the maximum thickness of the soil layer. Overall, for an effective remediation, soil layer values in the range 30 - 70 cm should be considered for the plant design activities Falciglia [15]. Treatment times required to reach a specific remediation target are strictly influenced by the contaminant removal kinetics achievable during the MW irradiation. It was experimentally shown that MW heating is characterized by an exponential decay kinetic:

$$C = C_0 e^{-kt^n} \quad (6)$$

where t (min) is the heating time, k (min⁻¹) is the decay rate and n is the shape term. For the remediation of hydrocarbon-polluted soils, k and n values as a function of the operating condition applied and soil moisture have been reported by Falciglia [15]. For any initial contaminant concentration (C₀) given, the knowledge of k and n parameters allows the calculation of the residual concentration values (C). A continuous conveyor-belt processing concept using MW heating was evaluated by Buttress [27] considering a residence time of the soil within the applicator of 4 s at a constant throughput of 150 kg

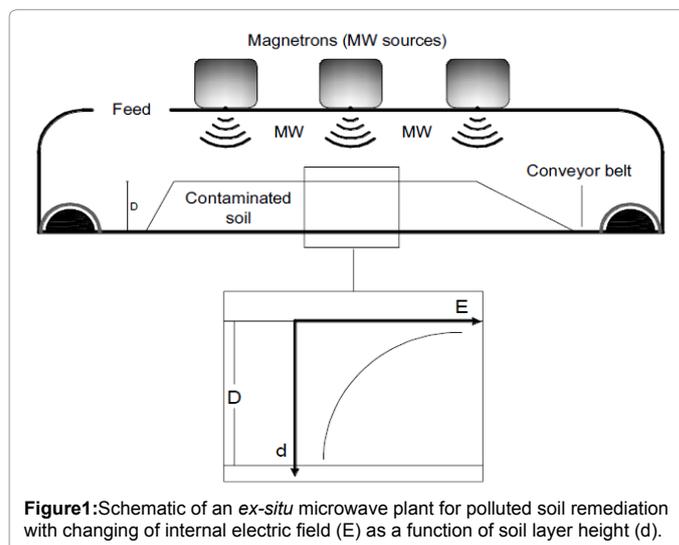
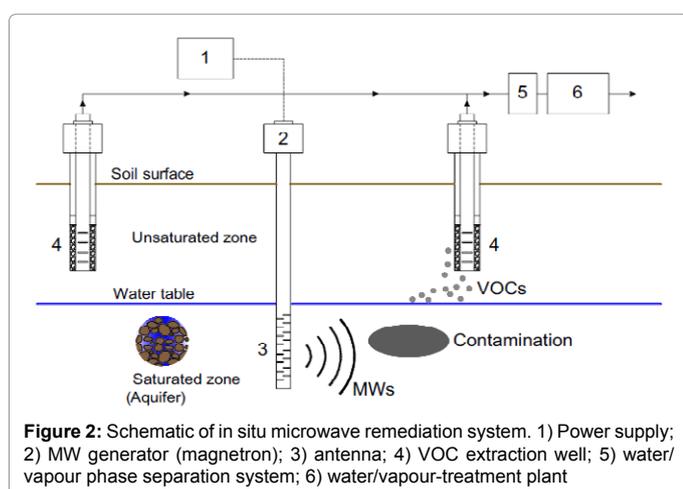


Figure 1: Schematic of an ex-situ microwave plant for polluted soil remediation with changing of internal electric field (E) as a function of soil layer height (d).

h⁻¹. Authors found for the remediation of hydrocarbon-contaminated soils that applying the power in a single stage was 20-30% more energy efficient but with removals lower than. A maximum contaminant removal of 75% was achieved using two processing steps in series, but its application is limited by the amount of power that can be safely and reliably delivered to the process material. A potential scheme of in situ-MW treatment includes the irradiation of the contaminated soil by means of specific antennas connected to a MW generator and a power supply. The heat generated by the interaction of the electric field with the soil results in a separation through vaporization of the hydrocarbon contaminants from the solid matrix. The irradiation system must be coupled with conventional extraction for volatile compound (VOC) capture and treatment. Falciglia et al. [7] recently reported results from a modelling and experimental study aimed at investigating the evaluation of the temporal and spatial effects of the in situ application of a diesel-contaminated MW irradiation on the electric field penetration, temperature profiles and contaminant removal. Their findings better defined the limits of the in situ MW treatment. Results showed the synergic progressive soil temperature increasing and the electric field penetration into the soil. At the beginning, when soil temperature values were close to 20°C, electric field dropped rapidly with the distance from MW source. After 12 h of treatment, a slight increase of the electric field penetration and a marked change of the electric field curve shape were observed due to the initial small temperature variation observed. At the end (6 d-treatment), MW electric field (E) was able to penetrate up to a maximum distance of about 80 cm from the source and its value was reduced by about one third of its initial value. A maximum final temperature of about 500 °C was recorded at a distance of 10 cm. Overall, the thermal effect of the MW treatment was observed to affect a maximum distance of about 120 cm. In terms of contaminant removal, a maximum distance of 80 cm can be considered also for removals up to 99%. In situ MW application for hydrocarbon contaminated-aquifer treatment includes the simultaneous treatment of both solid and water phases. Schematic of the plant is similar to that needed for in situ soil treatment. Differently, in this case, the antennas must be able to irradiate the saturated zone as shown in Figure 2. Similarly to the previous case, extraction well for volatile compound (VOC) capture are inserted in the unsaturated zone. Obtained findings showed the applicability of MW energy in in situ hydrocarbon contaminated aquifer treatment, however further scaling-up investigations needed in order to define the real scale applicability of the treatment and related costs.

Economic Analysis for MW Applications and Comparison to other Hydrocarbon-removal Treatments

Very few information on MW heating costs are available in scientific and technical literature. Specific costs for ex situ applications in the range 20 - 160 € ton⁻¹ were found by Falciglia [15] for sandy soils. However, it was found that the presence of humidity in the soil decreased the cost up to about 70 € ton⁻¹. The possibility of using very short times for sandy soils resulted in a most economical treatment whereas double costs are required for the treatment of a clayey soils (maximum cost of 160 € ton⁻¹) respect to a medium sandy soil. This makes the remediation of fine texture soils unsuitable. More recently, Buttress [27], evaluating a continuous microwave processing system for hydrocarbon removal from contaminated soils (energy dose of 150 kWh t⁻¹), reported an average operating cost of about 20 € ton⁻¹ to reach a maximum contaminant removal of 75%. Detailed costs for in situ application for both soil or aquifer remediation have not yet exhaustively discussed in literature and they can be assessable only by means of further scaling-up studies. Hydrocarbon contaminant removals reported in literature jointly with times are hardly achievable by other remediation treatments, especially if bioremediation- or bio-augmentation processes are involved. In this case, hydrocarbon removals lower than about 80% and remediation times up to 300 days were generally observed. Lee [28] found a maximum biodegradation ratio of hydrocarbon of ~60% for a remediation time of 12 days by applying an enhanced bioremediation treatment of a low permeability soil artificially contaminated with diesel (2500 mg kg⁻¹). Li [29] showed that, for a sandy soil artificially contaminated with diesel fuel at different rates (from 500 to 50000 mg kg⁻¹), the natural biodegradation reached a maximum value in the range of 70-73% for a total incubation period of 110 days. Łebkowska reported an 80% removal for a sandy soil polluted by TPHs at 5300 mg kg⁻¹ of treated by ex-situ bio pile remediation for a period of about 2 months. Lower hydrocarbon removals (37-50%) were achieved by Fernandez, Liu [30] reported findings from a lab-scale bio augmentation of diesel- contaminated soils artificially contaminated with diesel at a maximum rate of 12000 mg kg⁻¹. Authors observed, degradation efficiency in the range 59 - 73% considering a treatment period of 300 d. More recently, Mena [31] showed data regarding the application of coupled electro kinetic soil flushing (EKSF) and bioremediation through innovative biological permeable barriers (Bio-PRBs) for the removal of TPH from spiked clay soils. Authors reported a low contaminant removal of 30% in a period as short as two weeks. Da Rosa [32] showed results from an experimental study aimed at removing petroleum (5% w/w) and diesel oil (5% w/w) from soil using micro foams of biological and chemical surfactants. They found a removal efficiency of about 45%. Also the employment of chemical treatments could likewise lead to low hydrocarbon-removals. Do et al. [33] showed that a chemical oxidation treatment of a diesel polluted soil at 5000 mg kg⁻¹, using peroxy monosulphate/cobalt, was characterized by a maximum degradation of 47% and that a sequential injection treatment using a large quantity of chemicals was needed to reach a contaminant degradation of 88%. High performances of MW treatment can be reached by using conventional thermal desorption remediation or a limited number of chemical-physical treatments which are generally characterized by higher costs. Falciglia et al. [15] found that, applying a conventional ex-situ thermal desorption (TD) technique, a soil temperature in the range of 175-250 °C was sufficient to completely decontaminate a TPH-polluted fine sandy soil with costs up to 560 US\$ ton⁻¹. The US-EPA (2004) reported an average cost of fuel consumption ranging from 200 to 400 € ton⁻¹ for hydrocarbon polluted soil remediation by conventional ex-situ thermal desorption



with a rotary kiln system. Islam et al. [34,35] found that subcritical water extraction (SCWE) treatment application resulted in a 99% of hydrocarbon removal with costs in the range of US\$ ton⁻¹ 250 - US\$ ton⁻¹ 733. Khalladi et al. [36] reported for a diesel extraction treatment for soils using an ionic surfactant sodium dodecyl sulfate column process, an elimination rate of 97% was achieved after a soil washing process. In terms of cost, the Federal Remediation Technologies Roundtable (FRTR) reports a minimal cost of US\$ 361 per m³ for a typical ex-situ chemical extraction process. Other researchers have shown electro kinetic (EK) decontamination to be somewhat ineffective for diesel removal from soils thereby suggesting that only enhanced processes should be used in order to reach contaminant removal higher than 95% Pazos and Tsai [37,38] Therefore MW costs jointly with very short remediation times make MW technique a suitable alternative to conventional thermal or physical-chemical treatments for the remediation of hydrocarbon-polluted soils. Also in the case of aquifer remediation, high TPH removal observed for MW heating Falciglia et al. [15] are hardly achievable in very short times using different biological or chemical treatments. Hess refers simulating an in situ bioremediation of aquifers contaminated with diesel based on the infiltration of groundwater supplemented with oxidants and nutrients, reported a TPH-removal ranging from 53 to 86% after a treatment of 96 d. Furthermore, TPH removals drop down to ~20% for the heaviest hydrocarbon fractions [39,40]. A study on the bio-remediation of a diesel fuel contaminated aquifer in Switzerland treated by injecting aerated groundwater supplemented with KNO₃ and NH₄H₂PO₄ was performed by Hunkeler. Authors found a contaminant degradation of ~25% within 2 years of engineered bioremediation. Van Stempvoort studied the enhanced solubility of petroleum-derived compounds in humid acid solutions during a 5 year-treatment of a diesel contaminated-aquifer with in situ flushing technology. Authors showed that during aqueous transport, biodegradation of the BTEX and PAHs occurred, limiting the lateral and longitudinal extent of the diesel contaminant plume in the model aquifer [41-43]. Lee reported results from a pilot-scale study aimed at investigating the effect of a surfactant-enhanced in situ flushing treatment on the removal of TPH from soil and groundwater contaminated with diesel fuel. Authors observed a TPH (C₈-C₄₀) removal of about 88% flushing a sorbitol Manolete solution for approximately 3 months [44,45].

Future Trends

Based on the above considerations, it is clear that MW technology is a potential economically and powerful remediation technique with high contaminant removals and short remediation times, representing a suitable alternative to conventional thermal or physical-chemical treatments for the remediation of hydrocarbon-polluted soils and groundwater. However, at the moment specific literature reports limited data regarding full scale MW treatments and this makes their real applicability very limited despite the several advantages. The need of further scaling-up and pilot studies aimed at better knowing the real scale processes and improving the overall ex-situ and in situ application performance represents at the moment the main challenges of future research activities on MW heating remediation.

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