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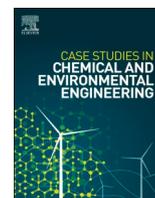
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Repeatable use assessment of silicon carbide as permanent susceptor bed in *ex situ* microwave remediation of petroleum-impacted soils

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ABSTRACT

Efficiency of microwave-enabled *ex situ* soil remediation can be improved by dielectric susceptors. Cost, and environmental burden of these susceptors can be minimized if they are used repeatedly in a permanent bed set-up. In this study, carbon nanofibers, activated carbon, magnetite, and silicon carbide were tested at the lab scale for repeated use in permanent microwave-induced thermal soil remediation. Despite their superior ability to convert microwaves into heat, carbon nanofibers experienced electrokinesis and activated carbon partially combusted in the microwave cavity, which complicates their pragmatic use in remediation applications. Magnetite was also able to convert microwaves into heat effectively and it was relatively more stable; however, repeated heating/and cooling cycles changed its physicochemical properties, which was attributed to oxidation of iron oxides at the air-soil interface. Silicon carbide, on the other hand, was an efficient heating agent and was stable during repeatable heating and cooling cycles. Through 25 heating/cooling cycles, an average peak temperature of 329 ± 55 °C was achieved for a 29 cm³ sample and analysis of dielectric properties after every 10th and 25th cycle indicated that there were no significant losses in thermal conductivity or permittivity of the material. Subsequent remediation experiments with silicon carbide demonstrated that between 89 and 97 % of the total petroleum hydrocarbons were removed from soil including a marked fraction of heavy hydrocarbons when 20.2 kJ g⁻¹ of microwave energy was introduced. In addition, post-treatment experiments demonstrated that soil conditions were capable of supporting seed germination indicating that some conditions of soil were recovered after microwave remediation.

1. Introduction

Accidental releases of long chain, heavy petroleum hydrocarbons (PHs) during exploration, production, refining, transportation, storage, and other industrial activities may pose hazards to marine and terrestrial ecosystems [1–6]. The United States (US) Environmental Protection Agency (EPA) [7] estimates there are 225,000 PH-impacted brownfields in the US. These PHs are composed of aliphatic (e.g., alkanes, alkenes) and aromatic organic compounds (e.g., benzene, toluene, ethylbenzene, xylenes). When released in the ecosystem, those petroleum hydrocarbons (PHs) can disperse, penetrate, and migrate vertically and/or laterally through soils and potentially impact surrounding air and the groundwater quality [8–10]. Exposure to these compounds may have potential health or environmental impacts [11–15] depending on a variety of factors including PH concentration, bioavailability, chemical composition, physical state, exposure path, and duration of exposure [8]. In terrestrial systems, PH spills can alter the physical, chemical, and microbiological characteristics of the native soil such as shifting C and N balance, changing pH, decreasing water holding capacity, increasing temperature, and/or decreasing soil porosity, changing the biomass density [16–19]. In addition to terrestrial spills, petroleum can also enter aquatic systems (e.g., marine environments) and can eventually impact shores and sediments [20–22].

To mitigate PH soil impacts in the environment, various *ex situ* remedial technologies have been developed to destroy or remove PH from soils and sediments. These remedial methods include biological,

chemical, physical-chemical, and thermal techniques [23] that can be deployed via *ex situ* scenarios such as landfarming and soil washing [1]. Excavation and transfer of soils to off-site facilities enables versatile control over the remediation process. To scale-up, these *ex situ* remediation technologies must be time and cost effective, deployable in remote regions, and ideally produce soils that are suitable for beneficial use after remediation. More recently, various investigations have indicated that *ex situ* remediation by microwave irradiation could be a promising alternative to conventional thermal remediation strategies [2, 24–26]. Depending on the physicochemical and dielectric properties of the soil and PH, microwave irradiation generates heat or directly interacts with PH to remove and/or destroy PH constituency [27]. The efficacy of microwave remediation can be enhanced with susceptors such as graphene, water, activated carbon, carbon fibers, and other favorable dielectric materials [2,28,29] and [30–35]. When mixed with soil, these microwave susceptors augment heating by more aggressively converting microwave energy into thermal energy [36]. Susceptor-enhanced microwave remediation is, however, constrained by the inability to recover and reuse the susceptor if blended with the soil. Especially for large amounts of soil, blending susceptors prior to microwave treatment increases the cost and raises concerns about releasing susceptors into the environment with the treated soil.

The objective of this study was to test repeated use of susceptors in a permanent-bed in microwave-induced thermal remediation (PMIT) and to determine their efficacy in removing PHs from soils using PMIT. This application is envisioned to advance the sustainable practice of

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Table 1
Characteristics of the selected dielectric susceptors.

	Bulk density (g cm ⁻³)	Moisture content (%)	Surface area (m ² g ⁻¹)
Powdered Activated Carbon	0.58	0.16	550
Carbon Nanofibers	0.05	NM	20
Magnetite	2.8	0.02	2.9
Silicon Carbide	1.7	0	0.2

NM: not measured because of the high electrostatic mobile nature of the material.

susceptor-enabled microwave remediation for petroleum impacted soils. A suite of materials that enhance microwave heating including magnetite, activated carbon, carbon nanofibers, and silicon carbide were tested to determine their suitability in PMIT by looking at heating efficacy, material longevity in terms of physical structure and dielectric properties, and their ability to assist in the thermal remediation of PHs through multiple heating cycles was demonstrated for the first time with this work. The work was coupled with subsequent soil germination tests to determine soil fertility after remediation.

2. Materials and methods

2.1. Microwave apparatus

A customized 208 V, 2.1 kW, 2.45 GHz microwave oven from Microwave Research & Applications, Inc. (Carol Stream, IL, US) with increased cavity cooling/ventilation to minimize excessive cavity heating and to prevent buildup of volatilized chemical was used for testing. The microwave oven could be operated at three power levels, 2,100 W (high power), 1,050 W (medium power), and 525 W (low power), and included 4 magnetrons, each with its own rotating antennae to distribute microwaves more effectively within the oven cavity. The oven cavity (53 cm L x 33 cm W x 27 cm H) had no rotating dolly and was equipped with an infrared (IR) temperature sensor at the center of the oven ceiling. Coupled with software, the IR temperature sensor could continuously monitor and record the temperature of the sample surface. Microwave energy inputs during operation ranged from 2.5 to 75.6 kJ g⁻¹ based on microwave power setting (e.g., low, medium, or high power), time of irradiation, and the mass of soil used. Specific energy input was calculated using the following equation:

$$E \left(\frac{\text{kJ}}{\text{g}} \right) = \frac{\text{microwave power (kW)} \times \text{time of irradiation (s)}}{\text{mass of contaminated soil (g)}}$$

2.2. Microwave susceptors

Four materials known for their microwave reactivity were screened for heat generating capability and longevity under repeated microwave heating and cooling (Table 1, Table S1). Powdered activated carbon, specifically Norit PAC 20BF (herein denoted as PAC), carbon nanofibers (CNF), magnetite, and silicon carbide (SiC; 98.5 %) were obtained from and/or manufactured by Cabot Corporation (Billerica, MA, US), Sigma-Aldrich, (Milwaukee, WI, US), Alpha Chemicals (MO, US), and Ashine Industries Inc. (Toronto, ON, Canada), respectively. Loose bulk density of the susceptors was measured by weighing 2 cm³ of each self-pressed susceptor in a disposable centrifuge tube. The moisture content of susceptors was measured in triplicate by weighing susceptors before and after drying in an oven at 110 °C for 2–4 hr. Specific surface areas of PAC and CNF were analyzed by Quantochrome Autosorb-6B (Boynton Beach, FL, USA). The analysis was conducted via 77 K nitrogen gas adsorption after 16–25 h of degassing, and the isotherms were modeled by Brunauer–Emmett–Teller (BET) to obtain the total surface area.

Preliminary tests of susceptor heating response were performed using 15 g silica sand with 1 wt % susceptor at 2100 W power. Samples

Table 2
Properties and characteristics of sand that is used to create the benchmark soil.

Composition (% w/w)	Physical properties		
Crystalline Silica (quartz)	70.0–99.5	Melting point (°C)	1,710
Aluminum Oxide	0–19	Initial boiling point (°C)	2,230
Iron Oxide	0–2	Relative density	2.65
Calcium Oxide	0–1.1	Moisture content (%)	0
Titanium Oxide	0–0.7	Loose bulk density (g/cm ³)	1.6

were heated in 5.1-cm diameter quartz petri dishes. The duration of these preliminary heating tests was for a total of 2 min with temperature measurements taken every 1 s for the entire duration. To better differentiate the heating responses of magnetite and SiC, secondary heating tests were performed using 100 g of pure susceptor in 10-cm quartz petri dishes at 2100 W power and compared to washed and dried silica sand as control (i.e., the matrix for PH impacted benchmark soil). The duration of these secondary tests was for a total of 7 min with temperature measurements taken every 10 s for the first 3 min and every 30 s thereafter.

2.3. Preparation of Synthetic Benchmark Soil

Synthetic, PH impacted soil was created as benchmark soil for testing remediation. Commercially available silica sand (Pioneer Sands LLC, Irving, Texas, US) was used as the soil medium because of its inertness and microwave transparency, providing a clearer picture of susceptor influence on sample heating. The physical properties of the sand are shown in Table 2. A heavy crude oil was used to prepare the synthetic soil samples used for testing. Crude oil aliphatic vs. aromatic composition and carbon chain length fractionation were analyzed according to MA DEP EPH 5/04 and US EPA SW-846 Test Method 8015B, by Eurofins Lancaster Laboratories i.e., certified by CA Environmental Laboratory Approval Program, No: 2792 as well as National Environmental Laboratory Accreditation Program, No: 10276CA. Benchmark soil characteristics for the benchmark soil sample are summarized in Fig. 1. All soil characterization quality was categorized as compliant based on chlorobenzene and *o*-terphenyl surrogate recovery performances. Hydrocarbon fractions were above the reported method detection limit of 3,000 mg kg⁻¹ and the reported limit of quantification of 8,900 mg kg⁻¹. Silica sand and oil were placed in a clean rotating concrete mixer for 1 hour to create a benchmark soil with a total petroleum hydrocarbon (TPH) concentration of 13,000 ± 816 mg kg⁻¹. The sample was then stored in 19-L sealed plastic buckets for future testing. Prior to each experiment, the bucket contents were rigorously stirred to ensure homogeneity of the product.

2.4. Contact dielectric measurements for microwave susceptors

Contact dielectric measurements of susceptors were performed using an Agilent 85070E open-ended coaxial probe and an Agilent E85071C Series Network Analyzer in the frequency range of 0.5 GHz–4.5 GHz (Fig. S1). Prior to each measurement, the instrument was calibrated using an electronic calibration module and reference materials. For each sample, dielectric measurements were collected by placing the probe against the specimen surface, and measurements were collected over the frequency range stated. The dielectric constant and the loss factor, ϵ_r' and ϵ_r'' , respectively, were then determined using Agilent 85071E Material Characterization Software. $\tan \delta$ (loss tangent) is the ability of a material to convert microwave energy into heat for a given frequency and temperature, which is calculated as the ratio of ϵ_r'' over ϵ_r' [37].

2.5. Repeated microwave heating of susceptors

Low-temperature (100–300 °C) and high-temperature (300–600 °C) thermal desorption remove volatile and semi-volatile organic

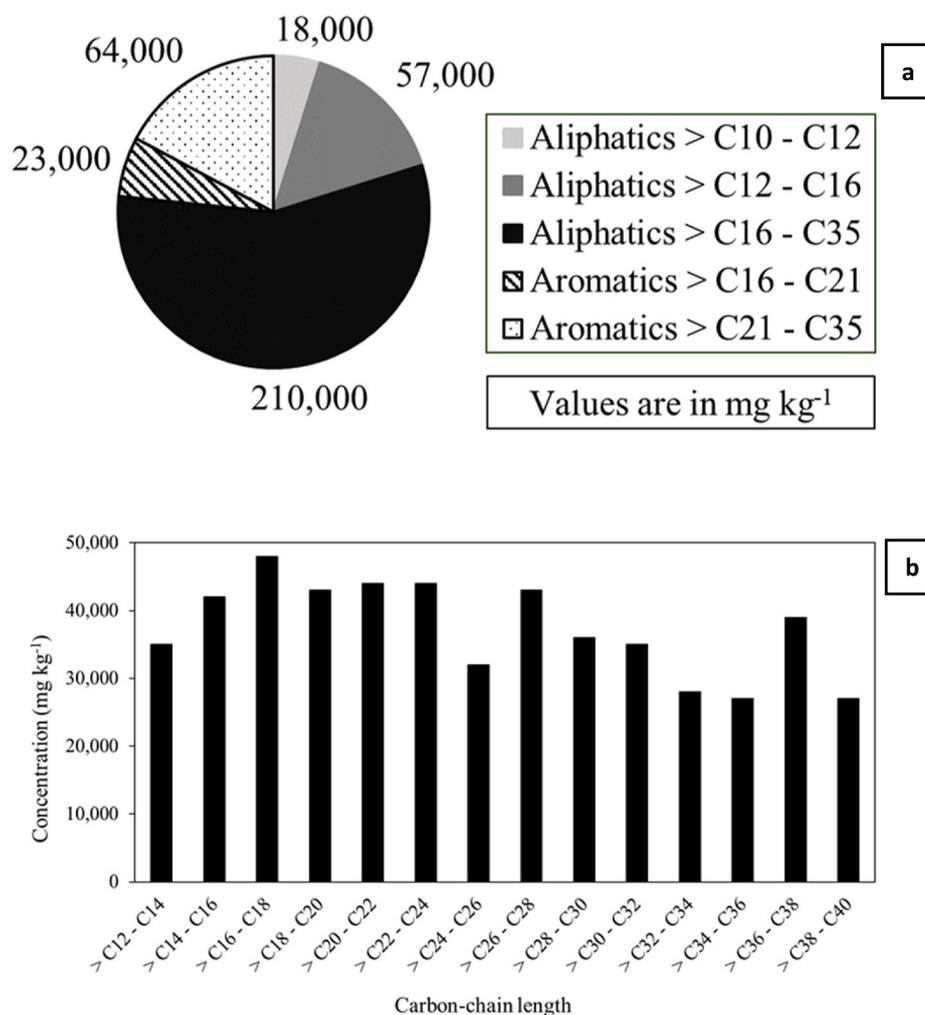


Fig. 1. a) Hydrocarbon fractionation results for the SJV crude oil that is used to prepare the benchmark soil used in this study; and b) Carbon range distribution for petroleum hydrocarbons between C₁₂ and C₄₀.

compounds from soil, respectively [38,39]. Therefore, susceptor integrity at elevated temperatures is essential to enable removal of semi-volatile PH while attaining the repeated use of susceptors in permanent bed microwave remediation configurations. The stability of susceptors for repeated use was evaluated based on their heating ability, heat retention, maintenance of physical structure, and stability of dielectric properties through 25 microwave cycles. For microwave heating response and subsequent heat retention of susceptors, 29 cm³ of each sample was heated in a 10-cm diameter quartz Petri dish and microwaved for a specified time at 2,100 W for up to 25 heating/cooling cycles. The heating time for each compound tested was designed to take that susceptor to approximately 300 °C, the minimum temperature believed to be necessary for the removal of semi-volatile PH constituency. The samples were left to cool inside the microwave oven until the surface temperature decreased to room temperature at the end of each run. Once cool down was achieved, the microwave was started for the next cycle. Samples were collected after the 10th and 25th cycle to measure dielectric properties, the dielectric constant (ϵ_r') and dielectric loss factor (ϵ_r''). The repeated heating and cooling temperature data was used to calculate statistical significance according to Student's t-test at 95 % level of significance.

2.6. Benchmark soil remediation experiments

PMIT remediation tests were conducted using a 29 cm³ SiC permanent bed in a 10-cm quartz Petri dish with 25 g PH impacted synthetic

benchmark soil. Benchmark soil was placed on top of the SiC bed (shown in Fig. S2a), treated, and replaced with untreated soil after each cycle. To prepare the PMIT treatment bed, SiC was first passed through a No. 30 sieve (0.6 mm opening) to eliminate the coarser SiC substrate and obtain a more uniform particle size distribution. The sieving did not change the heating response of SiC, but it allowed the treated soil to be separated from the SiC PMIT bed after treatment. After treatment, the remediated soil was separated by sieving and was shipped to Eurofins Lancaster Laboratories in a sealed glass vial for TPH analysis. This process was repeated 50 times using the same SiC bed. TPH of treated soils were analyzed as mentioned in the Preparation of Synthetic Benchmark Soil section. Control experiments with no SiC were conducted by replacing the SiC bed with 29 cm³ bed of inert quartz sand to maintain the same thermal mass in the microwave. All TPH analysis before and after remediation (capturing C₈-C₄₀) were conducted by Eurofins Lancaster Laboratories. The repeated remediation data was used to calculate statistical significance according to Student's t-test at 95 % level of significance.

2.7. Post-treatment germination experiment

To evaluate the effect of microwave remediation on soil fertility, 8-day plant germination tests were conducted in triplicate using benchmark soil samples under 3 different growing conditions: Condition 1) control soil (no TPH); Condition 2) benchmark soil (TPH concentration of 13,000 mg kg⁻¹); and Condition 3) microwave treated benchmark soil

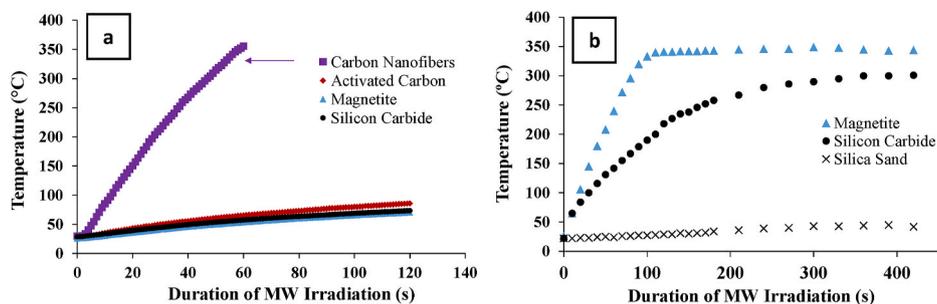


Fig. 2. a) Microwave heating comparison of 1 wt% susceptors blended in 15 g silica sand. Comparison of carbon nanofibers, powdered activated carbon, silicon carbide, and magnetite. Please note that magnetite and silicon carbide heating overlaps in the figure b) Microwave heating of 100 g pure bed of silicon carbide and magnetite compared to silica sand.

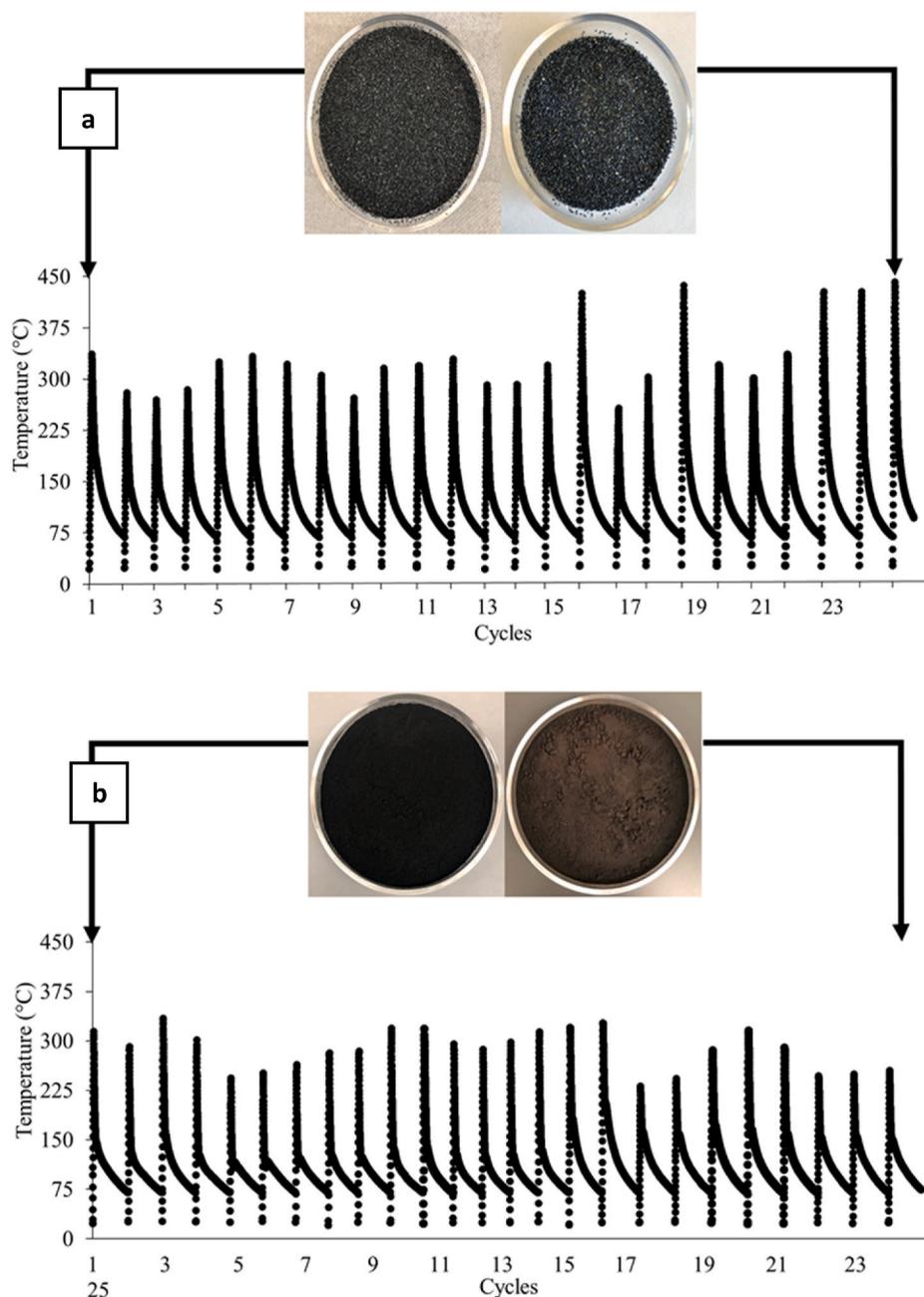


Fig. 3. Heating and cooling patterns of 29 cm³ of a) SiC and b) magnetite across 25 microwave irradiation cycles.

(medium power, 20.2 kJ g⁻¹ specific energy input, TPH concentration < 1500 mg kg⁻¹). Organic broccoli microgreens seeds (*Brassica oleracea* var. *italica*) were purchased from Back To the Roots Inc. The germination test was initiated by planting 12 seeds in each of the petri dishes that contained 25 g of test soil along with 7 mL of tap water. The amount of water was determined by preliminary germination experiments. The samples were then placed on a lab bench in an area of bright natural light incubated for 8 days. All dishes were partially covered to maintain the moisture content and allow gas exchange (Fig. S3). On days 3, 5, and 8 several drops of tap water (2–3 mL) were added to the samples. No other intervention was made (e.g., no tilling or mixing). Seedling counts and plant stem length measurements were performed at days 3, 5, and 8.

3. Results and discussion

3.1. Selection of microwave susceptors based on heating ability and practical considerations

In the past decade, a variety of susceptors have been demonstrated to successfully enhance microwave remediation of soils impacted by PH (Table S2) [23,40,41]. These studies indicated that an ideal susceptor should include a superior ability to convert microwaves into heat and not change the composition of the minerals and natural humic substances in the soil that could deteriorate the fertility of soil after treatment. For repeated use in PMIT installations, the susceptor should also show physicochemical integrity, (i.e., minimal changes in morphology such as thermal expansion and composition) and favorable heating/cooling patterns.

Preliminary testing of the four microwave susceptors was performed using 15 g of silica sand and 1 wt% of susceptor with up to 2 min of heating. These initial tests showed that CNF was the most sensitive to microwave irradiation (Fig. 2a), reaching 220 °C within 25 s. This observation agrees with literature and is attributed to extremely high electron mobility on the graphene backbone, which enables excessive heating under the incoming electromagnetic field [1,42,43]. The heating responses for PAC, SiC, and magnetite were notably lower than CNF, as observed by achieving 95 °C, 75 °C, and 75 °C, respectively, after 2 min. In our prior work, similar CNF hyper-reactivity observations were attributed to the 1D morphology, high surface area and generous electron budget of graphitic nanostructures that shows vibrational motion to align with the incoming electromagnetic field [2,44]. Separately, previous work showed 0.1 wt% activated carbon fibers in 20 g of soil could reach 700 °C within 4 min and faster than other common microwave absorbers e.g., powdered and granular activated carbon, MnO₂, an Cu₂O [31]. The ability of ‘filamentous carbon’ to convert microwaves to heat is also indicated by the dielectric loss tangent (i.e., $\tan \delta = \epsilon''/\epsilon'$) of bulk phase carbon nanotubes, which range between 0.25 and 1.14 at 2.45 GHz and room temperature, ca., 298 K [41] and the notable (~25 %) increase in imaginary part of dielectric loss from 0.8 to 1.0 when 10 wt% activated carbon is added to soil. Despite the superior performance of CNF, during irradiation in the closed oven cavity, CNF became airborne due to its low density (0.05 cm³/g), its electrokinetic characteristic, and the air movement created by cavity ventilation. An engineering solution to immobilize the materials prior to deployment would be necessary, since the PMIT application would require conservation of the susceptor mass in the permanent bed for repeatable use. PAC, the second most efficient susceptor among those tested, realized combustion after two 20 s heating cycles in the ambient oxygen environment. Oxidation of PAC was indicative of its instability under repeated use. As such, the use of PAC in PMIT would require controlled heating and a controlled atmosphere to suppress combustion. Given, the airborne mobility of CNF and combustibility of PAC, the two additives were removed from further testing in the permanent bed scenario and subsequent investigations focused on magnetite and SiC.

Based on the narrowed field of susceptors, namely magnetite and silicon carbide, subsequent testing was designed to differentiate the

performance of SiC and magnetite and to also observe their performance during a more intense heating cycle to determine if they could reach the 300 °C temperature deemed necessary for the removal of PH constituency. Fig. 2b provides the heating profiles for 100 g of each pure bed for a period of 7 min. This test was able to differentiate SiC and magnetite displaying their microwave heating performances. Magnetite was able to reach to a maximum temperature in excess of 340 °C in less than 2 min. Silicon carbide, on the other hand, while reaching temperatures in excess of 300 °C, took 6 min. The silica sand reached a maximum temperature of 45 °C within the 7 min heating period, confirming its inability to effectively convert microwaves to heat. While these results indicated the preferential heating performance of magnetite, additional testing was necessary to determine suitability for repeated use in PMIT.

3.2. Repeatable use assessment of silicon carbide and magnetite

As indicated previously, susceptor integrity at elevated temperatures is essential to enable removal of semi-volatile PH while attaining the repeated use of susceptors in permanent bed microwave remediation configurations. This section reports the heating characteristics and dielectric properties of SiC and magnetite during repeated heating/cooling cycles. The heating and cooling patterns for 25 cycles of microwave irradiation and photographs of a SiC sample before and after the 25 cycles are shown in Fig. 3a. The SiC bed reached approximately 300 °C in 40 s. After repeated 40 s microwave exposures, SiC averaged a peak temperature of 329 ± 55 °C (COV = 17 %), with maximum and minimum peak temperatures of 442 °C and 258 °C, respectively, and heating and cooling rates of 7.6 ± 1.4 °C s⁻¹ and 0.49 ± 0.08 °C s⁻¹, respectively. After 25 cycles, the SiC bed showed no visible changes in texture, shape, or color. At times, while exposed to microwave irradiation, bluish-white (micro-plasma) sparks were observed at the susceptor surface. These sparks were attributed to arc plasma discharges caused by excited electrons of SiC reacting with the ambient atmosphere. On certain occasions, sparks focused near the center of the dish, and forming glowing red hotspots up to 5 cm in diameter (Fig. S2b). The gradual formation of hotspots was random and enhanced the sample's heating rate, especially within the glowing foci. The hotspots dissipated after the microwave was shut off. No physical changes such as melting or deformation, nor emission of odor or gas were observed with the SiC samples, likely attributable to its high melting temperature of 2,700 °C and its chemical inertness [45]. Finally, the frequency-dependent dielectric constant ϵ' and dielectric loss factor ϵ'' of SiC within 1–4 GHz were measured and showed minimal change over 25 cycles (Fig. S4).

In summary, SiC showed no apparent degradation of the heating, physical, or dielectric characteristic after 25 heating/cooling cycles. These observations indicated that successful preservation of key SiC properties for microwave remediation was plausible. Prior investigations also demonstrated SiC to be an efficient microwave susceptor in a wide variety of applications because of its high melting point, thermal resistance, and low thermal expansion coefficient [35,37,45–47].

As a ferrite, magnetite is a naturally abundant material that is also used as susceptor for its magnetic and electromagnetic properties [34,40,48–50]. The performance of magnetite under 25 heating/cooling cycles and photographs before and after the experiment are presented in Fig. 3b. The magnetite bed was able to reach approximately 300 °C within 26 s, 35 % faster than SiC. Magnetite averaged a peak temperature of 283 ± 29 °C (COV = 10 %) with a maximum peak temperature of 334 °C and a minimum peak temperature of 226 °C. The average heating and cooling rates were 10 ± 1.1 °C s⁻¹ and 0.26 ± 0.05 °C s⁻¹. Compared against SiC, magnetite was heating 2.4 °C s⁻¹ faster ($p = 0.0006$) and cooling 0.23 °C s⁻¹ slower ($p < 0.0001$), indicating its favorable dielectric reactivity as well as its ability to retain heat. The slower cooling of magnetite could be attributed to smaller thermal gradient associated with the 14 % lower average peak temperatures

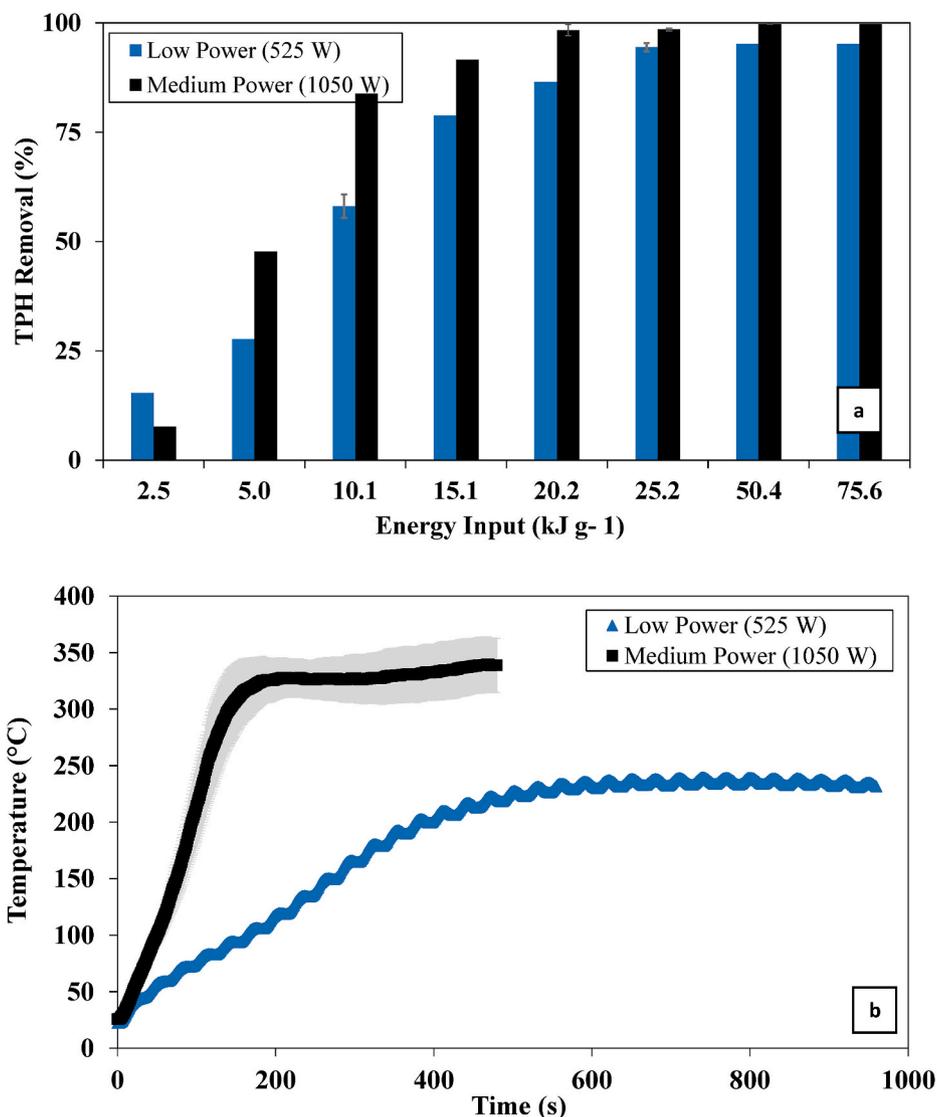


Fig. 4. a) TPH removal from benchmark soil samples as a results of various microwave energy inputs; and b) Temperature as a function of time for low and medium microwave power for 20.2 kJ g⁻¹ treatment. Shaded areas indicate one standard deviation (each way) of triplicate experiments.

when compared SiC. However, considering the one-fold higher surface area of magnetite vs. SiC, the slow cooling rate may also indicate a slightly longer retention of dielectric and orientational polarizability after microwave treatment, which is shown by the real part of the permittivity of magnetite (ϵ' of magnetite is 1.3–3.0 folds greater than SiC) [51]. It is difficult to conclude whether the cooling rate of a permanent susceptor bed in PMIT is of benefit. If treatment bed turnover requires system cooling, the faster cooling rate would enable faster turnaround. However, if system design can provide for the management and maintenance of elevated susceptor bed temperatures, then a slower cooling rate would save energy from one treatment to the next.

Magnetite's dielectric properties (ϵ' and ϵ'') within 1–4 GHz are shown in Fig. S4. The dielectric properties of magnetite changed more than SiC during 25 heating/cooling cycles. These could be attributed to oxidation of magnetite forming a layer of hematite on particle surface [52]; however, closer inspection is needed to better understand the changes in dielectric properties. The decreases, especially in the relative complex permittivity values of magnetite, may be associated with changes of iron oxide structure and formation of the hard outer layer. The gradual color change from black to brown and macrostructure changes from powder to crust after each heating/cooling cycle also indicated magnetite oxidation, especially where sample contacted air.

Although the heating performance was superior to SiC and its performance was not compromised over 25 cycles, magnetite was prone to oxidation and the heating and/or interaction with microwaves could have altered its composition to what was believed to be hematite via a metastable maghemite based on the resultant color of the material [53]. Further coordination chemistry analysis (e.g., XPS) could confirm changes in mineral composition of ferrites. The considerable physical and chemical changes caused practical difficulties in processing the soil, so, magnetite was removed from further use in PMIT remedial studies.

3.3. Soil remediation in permanent bed microwave-induced reactor bed

SiC was selected for further testing in a permanent bed PMIT configuration due to its consistent heating/cooling patterns and structural and dielectric integrity compared with the other susceptors. Fig. 4a shows TPH removal from benchmark soil at 2.5, 5.0, 10.1, 15.1, 20.2, 25.2, 50.4, and 75.6 kJ g⁻¹ at low (525 W) and medium (1050 W) power. In 7 of 8 instances, treatment at medium power removed more TPH (up to 26 %) than the same energy input under low-power treatment. A plateau in TPH removal was observed starting at 20.2 kJ g⁻¹ of energy input at medium power. After this point, the removal percentage was capped at 98 % to >99 %, which suggested that the optimum energy

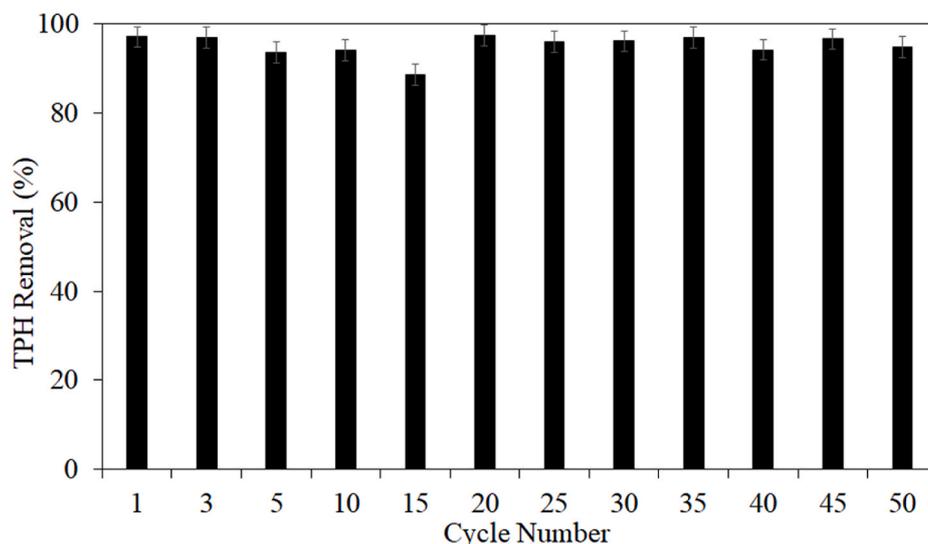


Fig. 5. TPH removal from benchmark soil using SiC permanent bed in microwave-induced remediation (SiC PMIT) for 50 cycles. Error bars indicate \pm std. dev. in each direction for the repeated experiments ($n = 12$).

input for the most efficient TPH removal at medium microwave power was within the range $15.1\text{--}20.2\text{ kJ g}^{-1}$. Other studies reported $0.45\text{--}1.56\text{ kJ g}^{-1}$ of microwave energy input to remove 16–80 % TPH from an artificially impacted soil (with diesel fuel) [54]. The lower energy requirement can be attributed to the fuel type, and associated hydrocarbon fractions, initial TPH concentration, soil mineralogical characteristics, and soil age [54].

Similarly, remediation at low power was maximized at 95 % removal after 25.2 kJ g^{-1} of energy input at low microwave power. For treatment at low microwave power, the results suggested an optimum energy input lies between 20.2 and 25.2 kJ g^{-1} . The compromised TPH removal at low power, despite the identical specific energy, was attributed to the slow heating of soil and energy losses due to the flux of air through the oven cavity for cooling and ventilation. This is supported by the temperature profiles of the samples in the microwave cavity as shown in Fig. 4b, which depict a rapid temperature rise in the beginning, followed by a constant temperature maintained at $300\text{--}350\text{ }^\circ\text{C}$ for 20.2 kJ g^{-1} . For the same energy input, remediation at low microwave power, which required twice the irradiation time, only reached a consistent remedial temperature of $200\text{--}250\text{ }^\circ\text{C}$ after 400 s of microwave exposure. This finding aligns with previously reported longer exposure times and higher microwave power requirements for TPH removal from soil [54].

Control experiments (washed silica sand as a permanent bed in place of SiC) were conducted at 10.0 and 25.2 kJ g^{-1} at low microwave power as well as 25.2 and 50.4 kJ g^{-1} at medium microwave power. Those conditions showed negligible TPH removal. The dramatic difference between the control and SiC susceptor experiments indicated that SiC-enabled microwave remediation in a permanent bed configuration could successfully remove TPH from soils in PMIT applications. Based on those findings, the repeated use of a permanent SiC bed for benchmark soil remediation in PMIT through multiple cycles was demonstrated using medium power at 20.2 kJ g^{-1} specific energy input in the next section.

3.4. Technology demonstration for permanent bed microwave induced thermal (PMIT) remediation

Following the identification of SiC as a promising susceptor for the permanent bed in PMIT, lab-scale testing of SiC bed (i.e., using the same permanent bed) was demonstrated to treat benchmark soil samples through 50 cycles with 20.2 kJ g^{-1} specific energy input at medium power. The temperature profiles through 50 cycles are presented in

Fig. S5. The treatment of samples achieved consistent TPH removal of $95\% \pm 2.4\%$ ranging from 89 % to 97 % as shown in Fig. 5. The coefficient of variation for hydrocarbon removal was 2.5 % and inter-quartile range was computed as 2.9 % with only the 15th cycle was classified as a mild outlier (89.7 % lower inner fence boundary vs. 88.6 % removal at 15th cycle) and no extreme outliers. This indicated consistently uniform performance of hydrocarbon removal during the repeated use of the same material. The GC chromatograms indicated that the treatment primarily removed $\text{C}_8\text{--C}_{26}$ hydrocarbons, with the bulk of residual, post-treatment hydrocarbons in the $\text{C}_{28}\text{--C}_{40}$ range (Fig. S6). This pattern was consistent throughout the 50 treatment cycles. Slight less removal in the $\text{C}_{28}\text{--C}_{40}$ range was attributed to the high energy demand due to strong intermolecular interactions between heavy, long-chain hydrocarbon molecules (i.e., less volatile fraction) in their bulk phase as well as their strong affinity to soil. Cho et al. [54], reported significant losses in the $\text{C}_{10}\text{--C}_{16}$ and $\text{C}_{16}\text{--C}_{22}$ ranges at ca., $130\text{ }^\circ\text{C}$, which was reached within the first 100 seconds in our study. Li et al. [31] indicated the subsequent recovery of hydrocarbons via condensation in an ice-salt bath with insignificant losses ($\sim 6\text{ wt}\%$). This implies that the low molecular weight, volatile hydrocarbons could be captured without destruction; whereas heavy hydrocarbons may require higher temperatures that can cause cracking as a result of thermolysis.

3.5. Post remediation evaluation of benchmark soils via germination experiments

The remediation of benchmark, PH-impacted soils using SiC PMIT at the lab scale was effective at reducing the levels of PHs in the soils. However, a study of post-treatment soil fertility was important to understand the viability of soils that have been thermally treated using microwaves [55]. As such, post treatment investigations examined the ability of treated soil to support plant life. Previous work showed a significant decrease in seedling survival when soil is microwaved with the seeds in it [56]. However, in this study, soil was irradiated alone, and seedlings were planted after the soil reached room temperature. Eight-day germination experiments were conducted to determine if the soils support plant growth after treatment. As previously indicated, three soil conditions were investigated: Condition 1) control (no TPH); Condition 2) benchmark soil (TPH concentration of $13,000\text{ mg kg}^{-1}$); and Condition 3) microwave treated benchmark soil (medium power, 20.2 kJ g^{-1} specific energy input, $\text{TPH} < 1,500\text{ mg kg}^{-1}$). Photographs of the germination are presented in Fig. S6, and the above-surface

Table 3

Average seedling count and length at days 3, 5, and 8 for soil conditions 1, 2, and 3. Errors indicate standard deviation of triplicate conditions. Condition 1) control soil; Condition 2) untreated benchmark soil (TPH concentration of 13,000 mg kg⁻¹); and Condition 3) SiC PMIT treated benchmark soil (TPH < 1500 mg kg⁻¹).

	Day 3	Day 3	Day 5	Day 5	Day 8	Day 8
	Seedling Count	Seedling Height (cm)	Seedling Count	Seedling Height (cm)	Seedling Count	Seedling Height (cm)
Control	10 ± 0.8	NM	10.3 ± 0.5	2.2 ± 0.24	11.3 ± 0.5	3.5 ± 0.6
No treatment	6.3 ± 1.2	NM	8.7 ± 0.5	0.8 ± 0.34	10.3 ± 0.5	1.7 ± 0.3
SiC PMIT	8.0 ± 1.6	NM	8.0 ± 1.6	1.6 ± 0.43	9.7 ± 0.9	3.4 ± 0.7

NM: not measured because the seedlings were too small to measure.

seedling counts, and their total lengths are presented in Table 3. The seedlings grew favorably, both in number and length, in control soil (Condition 1). After 8 days of cultivation, 11 of 12 seeds sprouted and grew to an average of 3.5 cm.

The seeds for untreated benchmark soil (Condition 2) showed 10 seedlings that grew to an average length of 1.7 cm, which was 50 % shorter than the control. Those seedlings sprouted and grew more slowly (6 sprouts vs. 10 for control on Day 3) than those in the control group. In short, the average number of seedlings and their growth were notably suppressed in benchmark soil before treatment. This was attributed to decreased penetration of air and water into the soil; however, additional work would be required to understand the mechanism for these results. Germination studies in treated benchmark soils (Condition 3) indicated that the sprouting rate and sprout length increased compared with the untreated benchmark soils. Sprouting rate in the treated soil was slower than in the control, but seedlings reached a similar length after 8 days. In summary, the SiC PMIT treated soil showed good viability, with an effective number of sprouts and good growth rate. This result is important because PMIT not only is effective at treating soils but also allows soils to be viable for plant growth. However, natural organic matter and soil minerals in actual soil may undergo changes when thermal remediation is applied, and future research should assess plant growth viability. Soil decomposition can remove organic carbon and water and produce char, tars, CO₂ and CO. The decomposition of humic and fulvic acids may generate small molecular weight hydrocarbons and at higher temperatures (i.e., >700 °C), carbonate minerals can decompose and increase the soil pH [38]. With all these potential, complex interactions, in addition to non-uniform microwave heating and soil heterogeneity further complicates the prediction of overall plant tolerance but seed germination and plant root growth tests could shed a light to the longer term restoration of PH-impacted soils.

4. Conclusions

This project investigated the use of susceptors deployed in a permanent susceptor bed for microwave-enabled thermal remediation of soils. Susceptor screening indicated that CNF showed superior microwave heating ability; however, electrokinesis of CNF and tendency to become airborne during microwave irradiation presented practical difficulties with their use. The microwave heating ability of CNF was attributed to its generous electron budget, 1D morphology, high surface area and it was indicated by its dielectric properties. Another graphitic susceptor, PAC, also showed relatively good heating ability, however, oxidation of the susceptor itself during microwave treatment reduced its efficacy for repeated use in ambient atmosphere. For pragmatic reasons, SiC and magnetite were selected for detailed investigation because they maintained their dielectric and thermal properties and, physical structure through 25 cycles of microwave irradiation. Magnetite was able to reach approximately 300 °C within 26 s, 35 % faster than SiC with average peak temperature of 283 ± 29 °C. In comparison to SiC, magnetite was heating significantly faster (2.4 °C s⁻¹ faster, $p = 0.0006$) and cooling significantly slower (0.23 °C s⁻¹ slower, $p < 0.0001$), indicating its favorable dielectric reactivity as well as its ability to retain heat. Despite their enhanced microwave heating ability, heat retention, and persistent heating, the physicochemical properties of magnetite

were altered unlike SiC. The magnetite bed formed a solid brown crust (vs. black magnetite) on the surface, which made its operation challenging. SiC, on the other hand, showed stable heating and physical characteristics through 25 heating/cooling cycles and was thus chosen for continued PMIT investigation.

The use of SiC as a permanent susceptor bed was further investigated for PMIT applications through 50 cycles of treatment of benchmark soils. Tests indicated that TPH concentrations in those 50 soil samples were reduced by 89%–97% when 20.2 kJ g⁻¹ of microwave energy was applied (8 min of irradiation time at 1,050 W of microwave power per 25 g of soil). At low applied energy conditions, longer irradiation times were required to reach same remedial performances. Overall, based on these findings SiC-enabled microwave remediation in a permanent bed configuration was concluded to successfully remove TPH from soils. The GC chromatograms indicated that the treatment primarily removed C₈–C₂₆ hydrocarbons, with the bulk of residual, post-treatment hydrocarbons in the C₂₈–C₄₀ range throughout the 50 treatment cycles. The strong intermolecular interactions between soil and hydrocarbon molecules were believed to suppress the removal of heavy hydrocarbons when compared to the lighter TPH fraction. The work also demonstrated that susceptor enhanced microwave-based soil remediation was not detrimental to plant growth. Further research is required to explore the engineering aspects of this technology for scaling up. Fundamental work is needed to understand the application potential of this technology for complex soil matrices for greater masses of contaminated soils (e.g., those containing humic substances, mixtures of minerals, water, and biota).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.cscee.2021.100116>.

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