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Microwave-Enhanced Thermal Decontamination of Soil

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Results of bench-scale and pilot-scale investigations on the remediation of contaminated sand and soil using microwave energy are presented. Microwave radiation penetrates the soil and heats water and contaminants. Developing vapors are withdrawn from the soil. The experiments suggest that microwave heating is an effective soil remediation technology. The process is rapid as compared to other methods, and its efficiency depends on the dielectric and physicochemical properties of the soil and the contaminant. It allows for the removal of volatile and semivolatile components, and it is especially effective in the case of polar compounds. In the presence of soil moisture, the removal of both polar and nonpolar compounds can be described quantitatively in terms of steam distillation. The remediation proceeds at a temperature not exceeding 100 °C. It is of particular importance in the case of soil rich in organic matter, where too high a temperature could result in carbonization of humic substances. The remediation of trichloroethylene-contaminated sand in a pilot-scale microwave heating system simulating the in-situ process is also reported. The profiles of temperature and contaminant concentration in the remediation zone are presented. The pilot-scale investigations indicate a possibility of the use of microwave heating as an in-situ remediation technique. The use of inexpensive low-power generators for the supply of power to individual antennas may help to reduce costs and allow for an unconstrained design of the array of antennas in the ground.

Introduction

Among the variety of the available soil decontamination technologies, thermal methods ensure the greatest efficiency of above 99%. While being relatively expensive as compared to other treatment techniques such as bioremediation or soil venting, they distinguish themselves through a very short remediation time and a possibility to treat a wide range of contaminants. The most frequently utilized solution is soil heating in closed reactors at a temperature dependent on the kind and the volatility of the contaminants. High-temperature treatment, however, results in destruction of the soil structure and the annihilation of biological activity. In recent years, a tendency has been observed to abandon off-site methods in favor of the in-situ techniques. Their advantage lies in the reduction of the excavation and transport costs, which can amount up to 50% of the total treatment costs.

One group of the thermal in-situ techniques is the one in which a heat-transporting medium like hot air or steam is introduced into the ground through injection wells. In the case of the hot air, contaminants evaporate to the air stream and are withdrawn from soil through the extraction wells. Steam stripping, which is another modification of soil vapor extraction, allows for the removal of low volatile compounds from highly permeable soils. The contaminants are steam stripped from the soil matrix and moved along with the steam condensation front. The two-phase condensate is pumped up and utilized. The consumption of steam ranges between 0.1 and 0.25 kg/kg of treated soil. The estimated operating costs are $80/m^3, and the capital costs for an installation of a capacity of 5 m^3/h of soil are ca. $0.8 million (*1*).

Another group of thermal treatment methods is techniques utilizing low- or high-frequency electrical currents to transmit heat into the ground. Electrodes emplaced in the contaminated soil supply electromagnetic radiation or force the flow of the electric current through the contaminated zone. The conversion of the radiation or electricity into heat results in a temperature rise of the soil and the evaporation of the organic contaminants. The rate of the process depends strongly on the temperature induced in the soil. The contaminant vapors generated move toward the extraction wells under the influence of the applied negative pressure (*2*, *3*).

The idea of soil heating by electrical currents and electromagnetic radiation is not new. Both electrical heating and radiofrequency radiation have been used in the 1970s for the recovery of bitumen from tar sand deposits (*4*, *5*). Bitumen was heated to the point where its viscosity was low enough to allow for its recovery. The technique was applied in the 1980s and 1990s in field tests of the remediation of airfield soils contaminated by petroleum products (*6*, *7*).

In the case of the power-line frequency currents, the heating process is based on the Joule−Thomson effect and the principle of ohmic heating. The temperature rise that can be achieved in this way is rather slow. In a field test performed by Buettner (*8*), where 90 m^3 of TCE-contaminated soil was treated, almost 10 000 kW of energy supplied to the ground during 47 days resulted in a temperature rise of only 22 °C.

In the case of radiofrequency radiation or microwaves, heat is generated simultaneously in the entire soil volume. The mechanism of heat generation is a partial dissipation of the electromagnetic field energy and its conversion into heat. The alternating electromagnetic field induces the rotation of the dipoles of water and other polar substances present in the soil. The intermolecular friction results in the generation of substantial amounts of heat (*9*).

The heating intensity is greatest in the case of polar substances, the most typical representative of which is water. The rate of heat generated per unit volume (*10*, *11*) depends directly on the frequency of the applied electromagnetic field and the dielectric properties of the treated medium:

$$P = 55.63 \times 10^{-12} f\epsilon'_{\mathrm{r}} \tan \delta E_{\mathrm{lok}}{}^2 \tag{1}$$

where P is the power dissipated in the volume V (W), f is the frequency (Hz), ϵ'_{r} is the relative dielectric constant (−), E is the local electric field intensity (V/m), and $\tan \delta$ is the loss tangent (−).

The depth of penetration, or the distance from the source, at which the amplitude of the elctromagnetic wave falls to 37% of its initial value is inversely proportional to the frequency of the wave (*10*, *11*):

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$$d = \frac{1}{2f\pi}\left[\frac{\mu\epsilon'}{2}\left(\sqrt{1 + \tan^2 \delta} - 1\right)\right]^{-0.5} \tag{2}$$

where d is the penetration depth of the electromagnetic waves (m), μ is the magnetic permeability (Tm/A), and ϵ' is the dielectric constant (F/m).

The above equations indicate that, for the most effective heating and a rapid temperature increase, the radiation of a highest possible frequency should be applied. On the other hand, with increased frequency the radiation range decreases, and for too high frequencies the heating effect is noticeable only in the immediate vicinity of an electrode.

Several field-scale investigations on the use of radiofrequency heating in the remediation of soils contaminated by fuels, oils, and solvents have been reported (*2*, *3*, *6*, *7*, *12*). In most of them a system of two or three parallel rows of electrodes was applied, which was a rectangular analogue of a cylindrical coaxial cable. An appropriate design of the electrode array made it possible to contain the electromagnetic radiation within the outer rows of electrodes. The contaminant vapors generated in the soil as a result of heating were collected by means of a hood covering the soil surface or through perforated tubular electrodes performing simultaneously a function of extraction wells. Soil volumes of several cubic meters were heated with the use of 30- and 50-kW transmitters operating in a frequency range between 6 and 13 MHz. It has been reported that over 96% of petroleum products have been removed from soil within 10−20 days and the final soil temperature reached 150 °C. An essential shortcoming of the method is high capital and operating costs of a high power transmitter. An estimated cleanup cost of 1 ton of soil ranges between \$30 and \$60, and the overall cost of a remediation system with a capacity of 35 000 m^3 of soil per year and a power of 1 MW amounts to \$1.6 million (*12*).

Microwaves are a separate band of electromagnetic radiation with frequencies in the range of 100 MHz−300 GHz. Due to higher frequencies, microwave heating is much more intensive than radiofrequency heating, and it has found many applications in industrial drying and heating operations. The best known example for the practical use of microwaves is their application in microwave ovens.

The first investigations concerning the use of microwave energy for the decontamination of soil conducted by Dauerman et al. (*13*−*15*) yielded very promising results. The soil cleanup process was based upon the principle of steam distillation. This research had a preliminary character and was carried out on a small scale. However, Dauerman and his associates attempted to compare capital and operating costs of microwave heating with those of a typical incineration process. The analysis was performed for comparable process conditions. The operating costs of the cleanup of 1 ton of soil by microwave heating was \$40, whereas the incineration costs amounted to \$175. Similarly, the overall capital cost of an installation with a capacity of 5 ton/h amounted to \$0.5 million for microwave heating and \$5.5 million for incineration. The above calculations imply that microwave heating could be a more cost-effective alternative for traditional thermal treatment technologies. Further large-scale investigations are necessary to establish the effect of basic parameters on the effectiveness of the process and to work out the engineering rules of the technique.

Bench-Scale Experiments

The aim of the preliminary laboratory-scale experiments was to investigate the effect of the dielectric properties of the contaminants and the moisture content of soil on the rate of contaminant removal.

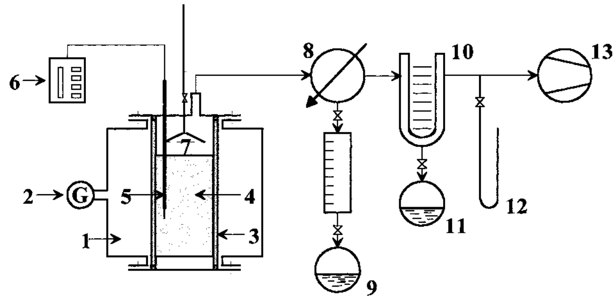

FIGURE 1. Bench-scale microwave apparatus: 1, microwave chamber; 2, microwave generator; 3, quartz column; 4, contaminated soil sample; 5, thermocouple probe; 6, electronic temperature meter; 7, sprinkler, 8, condenser; 9 and 11, distillate receivers; 10, cold trap; 12, manometer; 13, vacuum pump.

Experimental Apparatus and Procedures. The experimental setup used for the microwave heating of contaminated soil is shown in Figure 1. A 600-W generator working at the frequency of 2450 MHz was used.

The microwaves were induced inside a chamber in which a quartz column (90 mm i.d., 500 mm height) was installed. The contaminated sand or soil was placed in the column and treated by microwave heating. The top of the column was connected to a condensing system, a manometer, and a vacuum pump. The contaminant vapors generated were extracted from the column at a pressure of 0.8 atm and condensed by means of a condenser and a cold trap. The distillate was collected in a scaled container that allowed for the measurement of the contaminant volume removed during the experiment.

The microwave energy supplied to the chamber was partially absorbed by polar particles of water and the contaminants and partially suppressed by the steel walls of the chamber. The quartz column was not insulated; hence, there were heat losses to the surrounding air inside the microwave chamber. The conditions of the experiment did not allow for an accurate mass balance, but comparison of the experimental results for various conditions made it possible to examine the effect of the contaminant polarity and soil moisture on the process.

The described experiments, both in the bench and the pilot scale, were performed with the use of sand with a broad grain fraction. The density of the sand was 2.65 g/cm^3, and the porosity was 0.379. Over 61% of sand was a fraction with a diameter greater than 300 μm, while 35% was in the range between 75 and 300 μm.

The sand samples (about 2 kg) used for laboratory experiments were dried for 3 h at 150 °C and, after cooling, mixed with 100 g of a pure chemical compound. For an experiment with moist sand, distilled water was also added. The contaminated sand was then kept in a closed container for 24 h. Afterward, three 20-g samples were taken and extracted with 75 mL of *n*-hexane for 1 h in a Soxhlet extractor. After volume was adjusted, the extracts were analyzed by gas chromatography, using external standards and the peak-area calculation method. The results were expressed in terms of the contaminant's initial volume content in the soil. During the experiments, volume increase of the obtained distillate was measured. The process was terminated when no more distillate was collected.

All temperature measurements were performed using a thermocouple probe. Because the microwave field concentration around metal elements could bias the measurements, the generator had to be turned off while the temperature was measured.

Effect of Contaminant Polarity. The induction of heat resulting from the microwave radiation is determined by the dielectric properties of the heated substance. The so-called

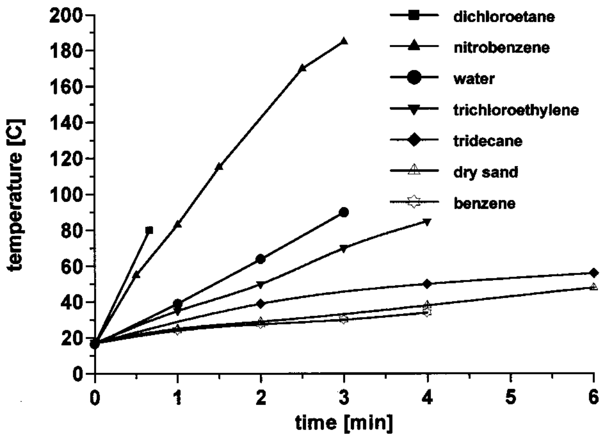

FIGURE 2. Rate of microwave heating for different substances.

TABLE 1. Boiling Points and Dielectric Properties of Selected Compounds at 20 °C

name	boiling point (°C)	dielectric constant	dipole moment (D)
	Polar Compounds		
nitrobenzene	211	35.74	4.3
1,2-dichloroetane	83.6	10.65	1.8
water	100	78.54	1.85
trichloroethylene	87	3.4	0.94
	Nonpolar Compounds		
tridecane	234	2	0
benzene	80.3	2.28	0

"lossy dielectrics" have a unique ability to suppress electromagnetic radiation and convert it into thermal energy. They are polar compounds with asymmetric structure, exhibiting a high dielectric constant and loss factor.

Figure 2 presents temperature increments of identical amounts (100 g) of polar and nonpolar substances (Table 1) placed in the microwave cavity. Such compounds as nitrobenzene, water, or 1,2-dichloroethane (displaying high dielectric constants) tend to heat much faster than nonpolar substances such as tridecane or benzene (with a symmetric structure). Sand is a weaker absorber of microwave energy, but wet sand tends to heat at the same rate as water. It was found out (*16*) that humus soil with a high organic carbon content absorbs microwave energy at a much higher rate than sand.

To determine the influence of the contaminant dielectric properties on the remediation progress, two experiments were performed at the same conditions (generator power, pressure). For each case the dry sand was contaminated with either nitrobenzene or tridecane. These semivolatile compounds exhibit a similar volatility but differ strongly in polarity (Table 1). The experimental results, including temperature profiles, are shown in Figure 3.

The final temperature of the sand after treatment was in both cases ca. 180 °C; however, the time needed for a complete removal of nitrobenzene (a polar substance) was half as long as in the case of tridecane. It may be attributed to a higher microwave absorption by polar nitrobenzene particles and the induction of greater heat amounts, according to the eq 1. In the initial period of both experiments (before the temperature of 100 °C was reached), the heating rate was greater than at their later stage. It may be associated with the presence of a small amount of moisture adsorbed on the sand grain surfaces, which intensively absorbs the microwave energy before it evaporates.

Effect of Soil Moisture. The experiments described above were performed with dry sand in order to determine the

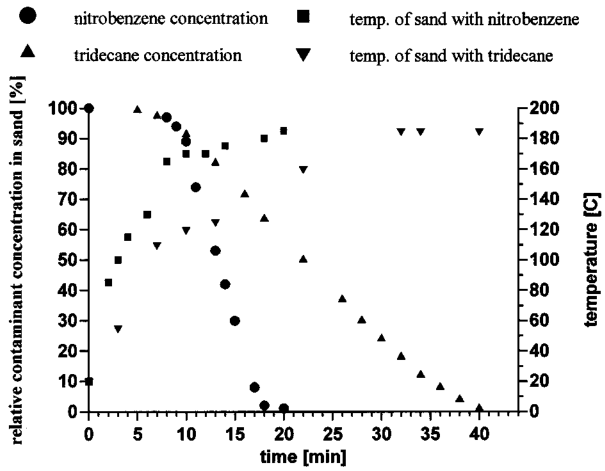

FIGURE 3. Temperature and concentration profiles during the removal of polar and nonpolar substances from sand.

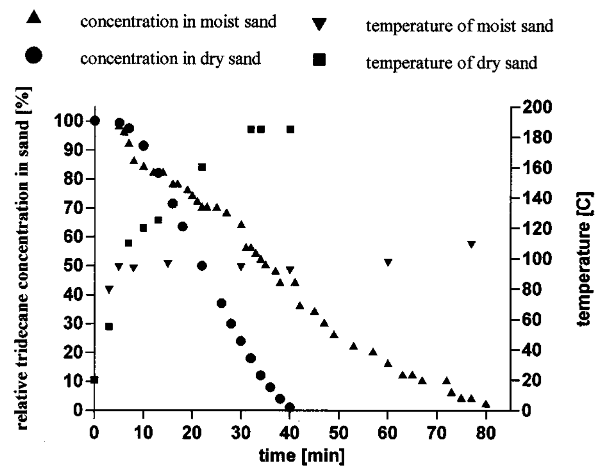

FIGURE 4. Tridecane removal from dry and moist sand.

effect of the contaminant's dielectric properties on the removal process. In reality, the water phase is always present in soil and its content frequently exceeds 30% by mass. Hence, water plays a major role in the absorption of the microwaves and in the distribution of heat. When it is present, the soil temperature quickly reaches 100 °C, provided that a sufficient amount of energy is being supplied. The contaminant removal proceeds then at a constant temperature as long as some moisture is present in the soil. The contaminant particles are removed along with the generated steam, and the process continues under the steam distillation conditions.

The concentration changes and temperature profiles during two experiments with tridecane removal from dry and moist sand (moisture kept at a constant level) are shown in Figure 4. In the second case the microwave energy is used up to a large extent for the evaporation of water. This makes the process much longer than in the case of dry sand, where the whole amount of microwave energy can be used to increase sand temperature and to vaporize the contaminant. The presence of moisture, however, allows for a complete removal of a semivolatile compound (tridecane) at a temperature lower than 100 °C. The initial moisture content of ca. 10% evaporated before tridecane was completely removed. To support the steam distillation process, the addition of water in small portions was necessary throughout the experiment.

In the steam distillation experiment, both phases (water and organic) were collected at a constant ratio. The consumption of water calculated according to the principles

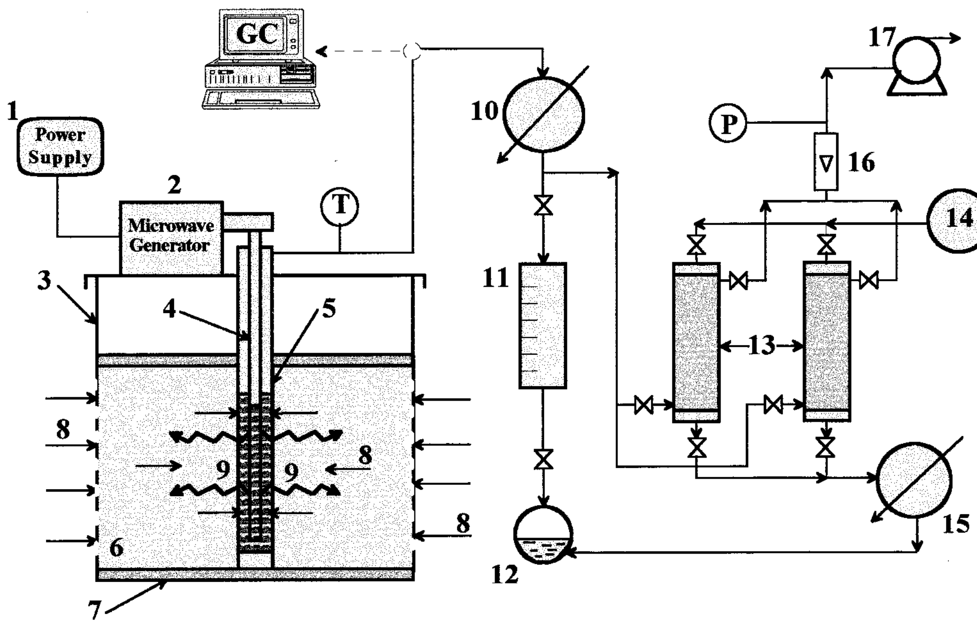

FIGURE 5. Schematic view of the pilot scale microwave apparatus: 1, power supply; 2, microwave generator; 3, cylindrical container (0.9 m height, 1 m i.d.); 4, antenna; 5, perforated PTFE vapor extraction tube; 6, contaminated sand; 7, seal; 8, vapor and air movement direction; 9, microwave radiation direction; 10 and 15, condensers; 11, scaled condensate receiver; 12, receiver; 13, activated carbon filters; 14, steam generator; 16, manometer; 17, vacuum pump.

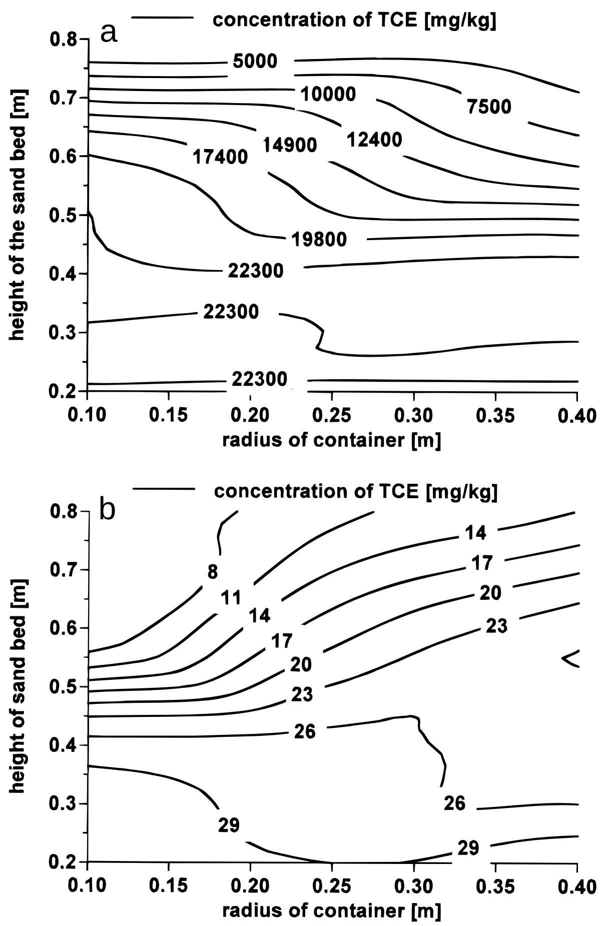

FIGURE 6. TCE concentration distribution in the sand (in mg/kg) before the experiment (a) and after 75 h treatment (b). The zone of heating was at the height between 0.30 and 0.65 m. The zone of air movement was at the height between 0.20 and 0.80 m.

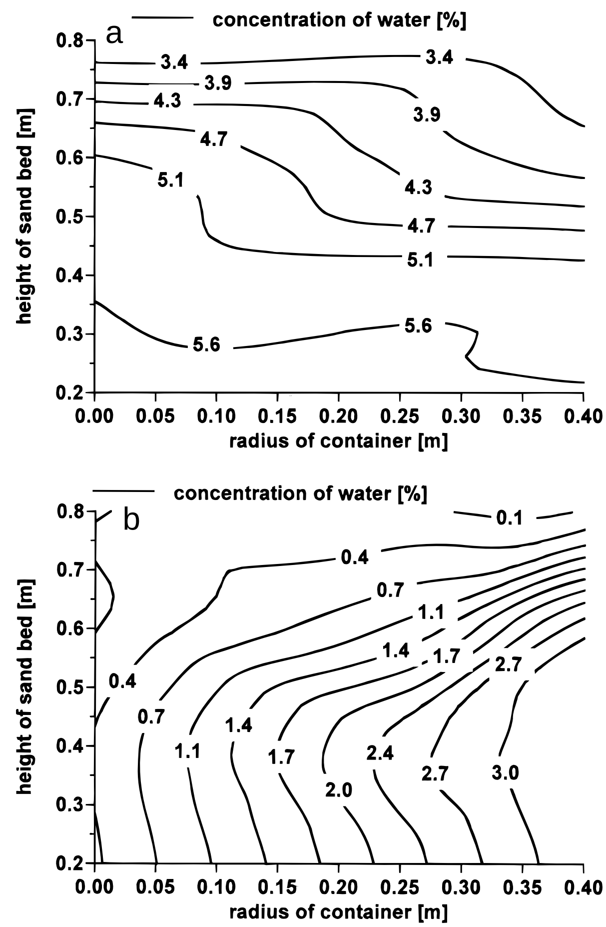

FIGURE 7. Moisture content distribution in the sand (in %) before the experiment (a) and after 75 h treatment (b). The zone of heating was at the height between 0.30 and 0.65 m. The zone of air movement was at the height between 0.20 and 0.80 m.

of steam distillation was very close to the experimental value.

In some experiments, described elsewhere (*17*, *18*), organic contaminants were removed from soil with a high humus

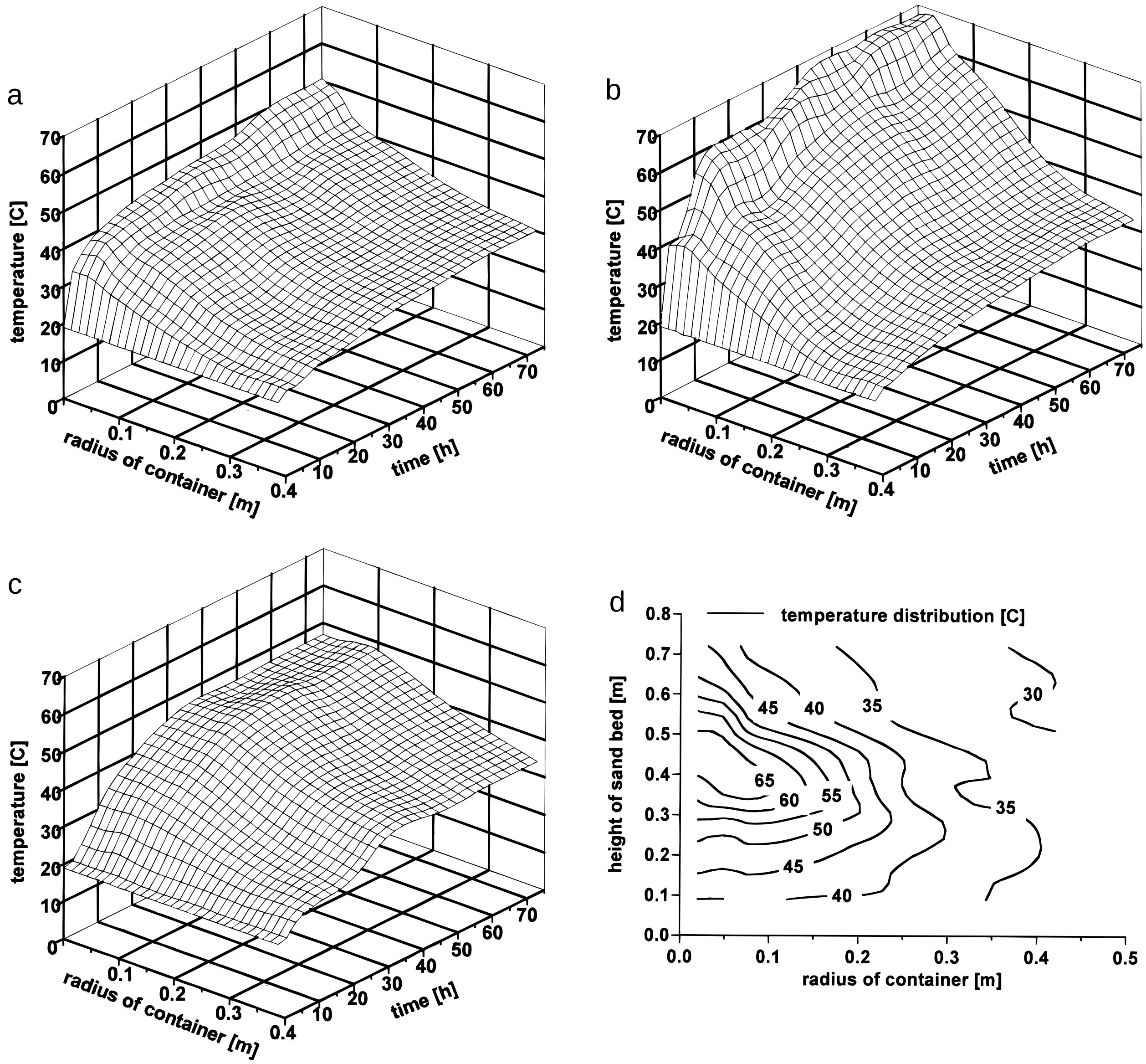

FIGURE 8. Temperature distributions at the level of 0.2 (a), 0.5 (b), and 0.7 m (c) over the container's bottom. Panel d shows the temperature distribution in the sand after the process. The initial temperature of the soil bed was 19 °C.

content. Steam distillation conditions and temperature not exceeding 100 °C prevented us from burning out the organic matter.

Pilot-Scale Experiments

The experience from the laboratory-scale experiments was used in the construction of a pilot-scale ground-heating system consisting of a single stub antenna for microwave propagation. The microwave heating system consisted of three elements: a microwave generator, a power supply system, and a stub antenna. The shell of the generator housed a typical commercial magnetron of the Toshiba 2M240 type. The power supply system was equipped with a control system allowing for a continuous or an intermittent duty. The output power of the system ranges between 550 and 650 W, and the operating frequency is 2450 MHz.

The design of the stub antenna was based on the work by Pucshner (*11*). The antenna was 0.7 m long, but the microwaves are emitted only through slots cut in its lower section of 0.35 m. The density of slots decreased toward the end of the stub in such a way that a uniform distribution of the electromagnetic field along the active section of the antenna was guaranteed.

The schematic of the pilot-scale apparatus is shown in Figure 5. The sand (1000 kg) was filled into a cylindrical container with a diameter of 1 m and a height of 0.9 m. The side wall of the container was perforated to allow clean air flow from outside. A 35 mm i.d. perforated PTFE tube was fixed vertically in the axis of the container, and the stub antenna was placed inside it. The tube was connected to a vacuum pump and acted as a vapor extraction well.

Vapors generated in the heated soil were transported together with the convective air stream to the condenser, where they were condensed and collected as a distillate in scaled receivers. Uncondensed vapors were sorbed on activated carbon. The system was connected to a manometer and a vacuum pump. One segment of the piping connecting the soil container with a condenser was provided with a heater wire to avoid vapor condensation and reflux into the container. The side wall of the container was equipped with an array of holes and openings for the introduction of a thermocouple probe and for the collection of soil samples.

In the pilot-scale study of trichloroethylene removal, a contaminant discharge into the soil was simulated by pouring 40 L of TCE onto 1000 kg of moist sand placed in a cylindrical container. After 2 days, an excess volume of ca. 18 L was collected through a valve from the container bottom. Before, after, and during the experiment, soil samples were collected using a sampler introduced radially or vertically into the container. The samples were extracted with tetrachloroethylene in a Soxhlet extractor and analyzed by gas chromatography. The water content of the samples was measured gravimetrically as a weight loss during drying at 150 °C, with a correction for the TCE content.

Remediation of TCE-Contaminated Sand. A single remediation experiment was performed in which TCE, a substance frequently identified at contaminated sites, was a contaminant. After the contamination procedure, sand samples were collected at three depths of the sand bed at various distances from the container axis. The TCE and water concentration profiles before the experiment was started are shown in Figures 6a and 7a. The microwave generator was operated intermittently, each 12 min work time being followed by a 3 min pause. The pressure inside the PTFE tube was kept at 0.9 atm. The microwave energy was supplied to the sand at the perforated part of the tube. It was obviously impossible to achieve the intensity of heating and temperature increase such as during the laboratory-scale experiments, because an identical power of 600 W was applied for the heating of a several times greater sand volume. In the first stage of the process, microwaves were reaching only the immediate vicinity of the antenna where the radiation was absorbed by water, sand, and TCE. As the sand became increasingly dry, it became more “permeable” for the microwaves, which could penetrate into more distant sand layers, in accordance with eqs 1 and 2. The induced heat was simultaneously transported to the top and the bottom soil layer, which were not penetrated directly by the microwaves. The experiment was carried out through 75 h with periodical shut-offs for sample collection and temperature measurements in various points of the soil bed. During the first several hours, mainly trichloroethylene and insignificant amounts of water were collected. At the later stage of the experiment, the water fraction in the distillate grew significantly.

After the first 24 h of the experiment, the analysis of the soil samples rendered a TCE concentration decrease in the heated zone from 2.2% to about 0.01%. The final distribution of TCE concentration and moisture content in the sand are shown in Figures 6b and 7b.

The initial TCE concentration was at its maximum of 4−5% in the bottom sand layer of 0.1 m. Although the gas movement induced by the pressure gradient was the least intensive in this zone, a reduction of the TCE content to ca. 2% could be observed there. This may be due to the evaporation and upward migration of the vapors generated.

Temperature changes of the soil bed in course of the experiment are shown in Figure 8a−c. Temperature distributions at the level of 0.2 (panel a), 0.5 (panel b), and 0.7 m (panel c) over the container's bottom are presented in Figure 8. The initial temperature of the soil bed was 19 °C. The temperature increase during the experiment was the greatest at the middle depth (a zone directly subjected to microwave heating), and the maximal local value was as high as 70 °C.

Figure 8d shows the temperature distribution in the sand after the process. The temperature in the bottom layer was slightly higher than the temperature near the surface. This may be attributed to greater heat losses to the environment from the top layer.

Heat balance is much easier to set up in the case of a pilot-scale experiment, where it can be assumed that all the energy emitted by the electrode is absorbed within the surrounding soil bed and used up in the temperature rise and evaporation of water and contaminants. During 75 h of heating, 36 kWh or 130 000 kJ of energy was supplied to the soil bed. A total of 19 kg of TCE and 11.6 kg of water were removed. The amount of energy necessary for the evaporation of TCE and water was 6000 kJ and 27 120 kJ, respectively. The remaining 97 000 kJ were used up for heating the soil bed, and a part of it was transferred to the environment through the soil bed surface or withdrawn with a stream of hot air.

Conception of Field Installation

In the pilot-scale studies of soil remediation by radiofrequency heating performed by other researchers (*2*, *3*, *6*, *7*, *12*), high-power generators were utilized, which supplied energy to the ground through an electrode array. A generator of this type is expensive and unreliable (*12*). Control and tuning instrumentation is also necessary.

The conception of the microwave energy supply to the ground would be a network of independent antennas, each powered by a separate microwave generator. A custom-built high power generator can thus be replaced by low-cost commercially available 750-W generators used in microwave ovens, working at a frequency of 2450 MHz. The wholesale cost of these generators does not exceed $15, whereas an eight times more powerful 6 kW magnetron costs as much as $3300 (*19*). A system of low power generators is very flexible, and it imposes no restrictions on the number and the arrangement of the antennas. A breakdown of a single antenna−generator cell does not require the shutdown of the entire system. Further research will be focused on determining the feasibility range of the technique and analysis of its economic aspects.

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