Journal of Hazardous Materials, 25 (1990) 343–360
Elsevier Science Publishers B.V., Amsterdam

TREATMENT OF PHENOL AND CRESOL CONTAMINATED SOIL

ROBERT A. EVANGELISTA

CH2M Hill, 3840 Rosin Court, Sacramento, CA 95834 (U.S.A.)

HARRY L. ALLEN

Environment Response Team, U.S. Environmental Protection Agency, GSA Depot. Building 18, Edison, NJ 08837 (U.S.A.)

and

ROBERT M. MANDEL

Region IX, U.S. Environmental Protection Agency, 215 Fremont Street, San Francisco, CA 94105 (U.S.A.)

Summary

Bench-scale experiments investigated the technical feasibility of innovative treatment options to remediate soil contaminated with phenol and cresols. These experiments resulted in full-scale operations which were followed by an additional bench-scale test to remove residuals. The bench-scale treatments explored were passive evaporation, soil washing, and biodegradation. Passive evaporation reduced concentrations of phenol, ortho-cresol, and meta- and para-cresol 58 to 66%, 55 to 80%, and 36 to 43%, respectively, after 3 weeks. In the soil washing tests, alkaline water adjusted to pH 11.5 and hot water at 50 °C both showed relative cleaning efficiencies of approximately 100%. Shake-flask biotreatment experiments found that *Alcaligenes eutrophus* JMP134 degraded phenol and cresol in untreated soil. After bench-scale experiments, a full-scale soil leaching process using water as an extractant removed more than 99.9% of the phenol and 99.7% of the cresols. To degrade oil and grease remaining in the leached soil, soil column biodegradation studies were performed on washed soil from the leach field. In the presence of a nutrient solution, oil and grease degraded rapidly, and residual phenol and cresols were further reduced.

Introduction

The Poly-Carb site, located outside the town of Wells, Nevada, contained soil contaminated with spilled liquid refinery wastes, including phenol and cresols. Contaminated soil from the spill area was placed inside of two PVC-lined trenches and left on-site. This site is in the recharge zone for the aquifer used as the sole source of drinking water for the town of Wells, named for its

*Paper presented at Symposium on Characterization and Cleanup of Chemical Waste Sites. American Chemical Society 197th National Meeting, Division of Industrial & Engineering Chemistry, Dallas, TX, April 10, 1989.

dependable water. A soil decontamination study was initiated to prevent pollution of this sensitive aquifer. The objective of this study was to assess the feasibility of several treatment options for site remediation.

The 1986 Superfund Amendments and Reauthorization Act (SARA) mandates the use of innovative and alternative treatment technologies to improve current remediation practices. Therefore, the feasibility of several options to decontaminate Poly-Carb soil was explored via bench-scale experiments. Three treatment technologies explored were passive evaporation, soil washing, and biological degradation.

Passive evaporation is the transformation of soil-bound contaminants into air-bound vapor using natural conditions, such as ambient temperature, wind velocity, and contaminant vapor pressure. Meteorological and geographical conditions at the Nevada site are favorable for this treatment method: hot (in summer), dry, windy, open, and remote. The major advantages of this technique are design simplicity and low treatment cost.

Soil washing and soil leaching is the removal of soil-bound contaminants using liquid extraction. Soil washing may comprise several steps: soil classification, pretreatment, soil extraction, extractant recovery, and wastewater treatment. Extractants are sprayed onto and percolate through soils during soil leaching, a passive form of soil washing. Common extraction solutions are: basic (caustic), acidic (mineral or organic), organic (methanol, KPEG), surfactant, and chelation (see [1–13]).

In biological degradation studies, microbes acclimated to phenolic wastes recorded up to 99% destruction [14–16]. Researchers at the U.S. EPA Hazardous Waste Engineering Research Laboratory (HWERL) in Cincinnati, OH and the University of Illinois (UI) Health Sciences Center in Chigaco have used plasmid enhanced *Pseudomonas cepacia* AC1100 to degrade the recalcitrant, xenobiotic chlorinated phenoxyacetate herbicides 2,4,5-T and 2,4-D [17]. Another microbe, *Alcaligenes eutrophus* JMP134 has performed similar degradations [18,19].

The objective of the bench-scale passive evaporation, soil washing, and biodegradation studies was to evaluate their feasibility for site remediation. After these studies, a full-scale soil leaching operation was performed on-site. Following on-site treatment, an additional bench-scale biodegradation test explored the treatment of residual contaminants. This paper presents the results of all of the methods mentioned above.

Methodology

Two sampling efforts were performed: an initial sampling of two trenches for untreated soil and a later sampling of a leach field for washed soil. In the

initial sampling effort, representative soil was obtained by four vertical borings along the center line of each trench. Each borehole produced four discrete soil samples:

1 above the upper PVC cover liner,
2 just below the upper liner,
3 mid-depth (2–4 ft) and,
4 just above the lower trench liner.

To average contamination concentration, discrete samples from each trench were composited. A diagonal composite and a vertical composite were labeled: ED and EV for the east trench, and ND and NV for the north. These composites were used for the passive evaporation, soil washing, and shake-flask biodegradation studies. In the later sampling effort, the 300×75 ft leach field was divided into 7×3 grid lines, respectively, to give 21 sampling nodes. At each node, two samples were taken, surface and subsurface. The composite soil was used for the soil column biodegradation studies.

For the passive evaporation and shake-flask biodegradation studies, soil samples were analyzed for phenol, *o*-cresol and *m*- and *p*-cresol by a modified Method No. 625 using a Shimadzu GC 9-A chromatograph with a flame ionization detector (FID) [20]. Method modifications included FID capillary column (Supelco Cat. No 2-3721), a temperature program (80°C for 240 s, increased by 10°C/60 s to 230°C and held for 60 s, increased 30°C/60 s to 300°C and held 120 s), and helium carrier gas at 18 ml/60 s. For soil washing, an U.S. EPA distillation/spectrophotometric Method No. 420.1 was used [21].

For the soil-column biodegradation studies, soil samples were analyzed for oil and grease, phenol, cresols, carbon dioxide evolution, and viable cell count. Soils were analyzed for phenol and cresol according to U.S. EPA Method No. 625 with a Hewlett Packard 5995C Gas Chromatograph/Mass Spectrometer [22]. Oil and grease concentrations were determined in accordance with shaker extraction modification of Method No. 418.1 on a Perkin Elmer infrared spectrophotometer [23]. For carbon dioxide evolution, the 0.1 *N* KOH carbon dioxide absorbent in the impingers were titrated with 0.1 *N* H_2SO_4 to neutral pH. For viable cell counts, 1 g of soil was placed into 1 l of water, serially diluted, and a 1 ml aliquot was spread on a nutrient agar plate. After incubation for 5 days at room temperature (20 to 25°C), a plate count was made.

Evaporation experiments were performed without sunlight at a relatively constant ambient temperature and humidity (10 to 17°C and approximately 75%) to simulate poor evaporation conditions. This gave conservative results compared with the site's desert climate. Soil evaporation took place in plastic petri plates (88 mm diameter×18 mm high) which were placed on a canopy-covered table. A 4×4 experimental matrix was designed for each trench: four evaporation durations – 0 days (no evaporation), 7 days, 14 days, and 21 days – and four soil samples per duration.

For soil washing tests, 10 g of soil was mixed with 200 ml of extractant and

agitated for 10 min on an automatic shaker. After washing, a 100 ml aliquot was decanted into a centrifuge tube, spun for 20 min at 980 *G*, and analyzed.

Two types of biodegradation studies were accomplished: shake flask and soil column. Shake-flask studies used untreated soil; soil columns used leached soil. Shake-flask studies on untreated soil evaluated two organisms for their contaminant degradation ability: *Pseudomonas cepacia* AC1100 and *Alcaligenes eutrophus* JMP 134. *Pseudomonas cepacia* was initially grown on 2,4,5-T, harvested, resuspended in KPM 7-buffer solution (50 mmol KPO_4 and 0.1 mmol $MgSO_4$ in 100 ml adjusted to pH 7) with either 100 or 500 ppm phenol and shaken at 200 rpm and 24°C for 1 or 2 days. *Alcaligenes eutrophus* was grown but without the 2,4,5-T step. For soil biodegradation studies, 8 to 25 g of soil, 50 ml of KPM 7, 0.5 ml of 1.5 *M* $(NH_4)_2 SO_4$, and 10 ml of cell suspension (at an absorbance of 5.0 at 600 nm in KPM 7) was placed into 125 ml flasks, were shaken for up to 4 days, and the soil slurry was quickly frozen to −30°C for analysis. Cell suspension was not added to control flasks.

The soil obtained for the soil-column had been leached approximately 3 months with water to remove the target contaminants — phenol, *o*-cresol, and *m*- and *p*-cresol. Custom-made glass columns, 3 ft high×3 in. diameter, were lightly packed with leached soil. An irrigation system intermittently delivered a nutrient solution, containing 200 mg/l of nitrogen (from a common 5-10-5 fertilizer) and 30 mg/l hydrogen peroxide, into a sealed column with leachate recovery. Air drawn into the head space above the soil was scrubbed of moisture and carbon dioxide using Dririte® and Ascorite®, respectively. Next, to trap evolved carbon dioxides, the air was drawn into impinger containing 0.1 *N* KOH at 10 ml/min.

Results

Bench-scale studies

Poly-Carb soil contaminants were reduced during 21 days of passive evaporation. The reduction of phenol, *o*-cresol, and *m*- and *p*-cresol in the east trench soil was 58, 55 and 43%, respectively; for north trench soil, the reduction was 66, 80, and 36%. Although contaminant reduction between trenches differed slightly, there was little difference in the contaminant decay characteristics. The half-lives of phenol, *o*-cresol, and *m*- and *p*-cresol were 1.5, 2.0 to 2.5, and 4.2 to 4.8 weeks, respectively. These half-lives were obtained from the slopes of the log concentration of each compound versus evaporation duration (Figs. 1–3). The half-life of a contaminant is a function of its vapor pressure. A least-squares linear relationship between the target contaminants' reciprocal half-life and its vapor pressure relative to phenol shows that a contaminant's half-life increased with decreased relative vapor pressure (Fig. 4). Therefore, a compound's physical property has an effect on its half-life. In

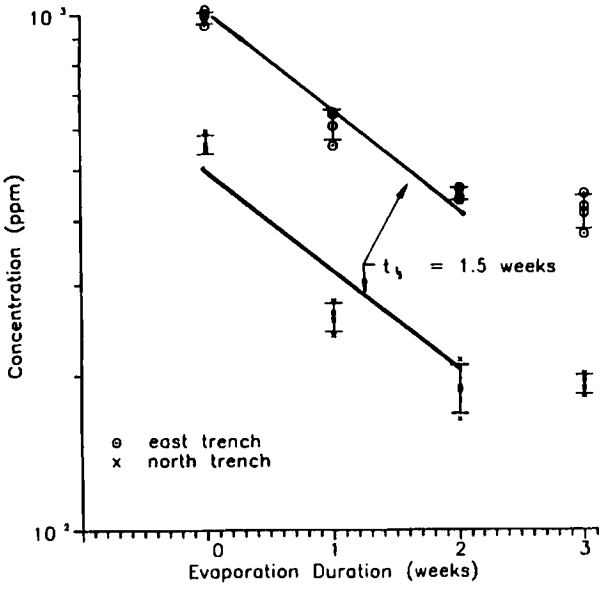

Fig. 1. Phenol evaporation.

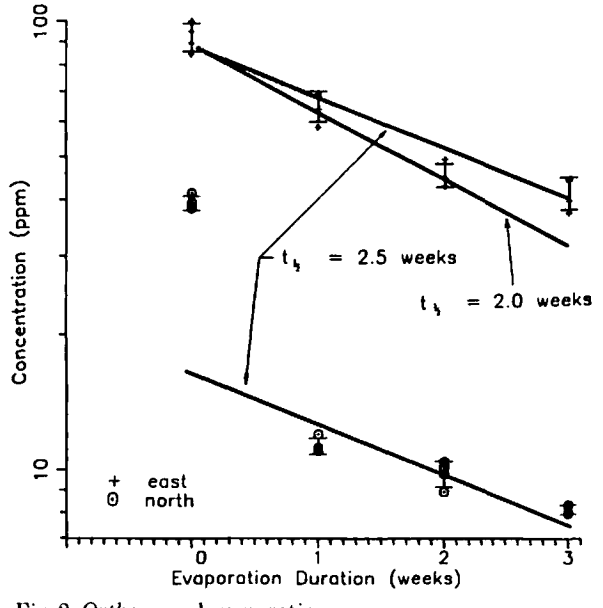

Fig. 2. *Ortho*-cresol evaporation.

addition, environmental factors, such as temperature, affect vapor pressure, thereby increasing or decreasing the evaporation duration.

Shake-flask, soil washing experiments effectively removed phenol. Alkaline

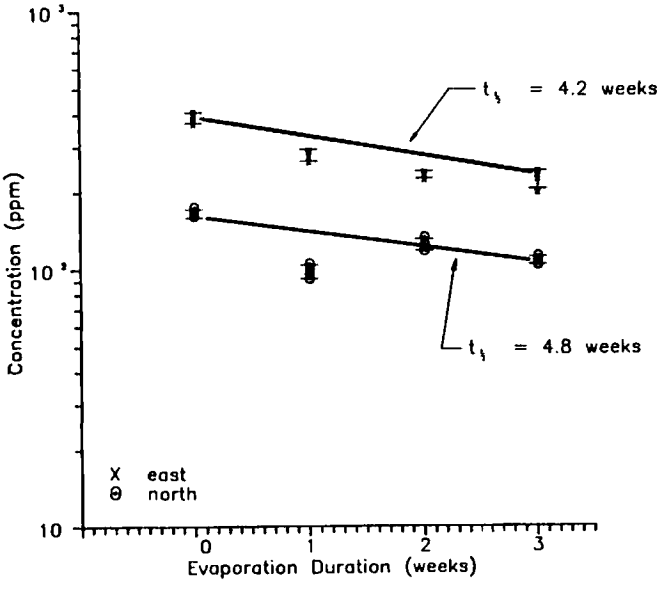

Fig. 3. *Para-* and *meta*-cresol evaporation.

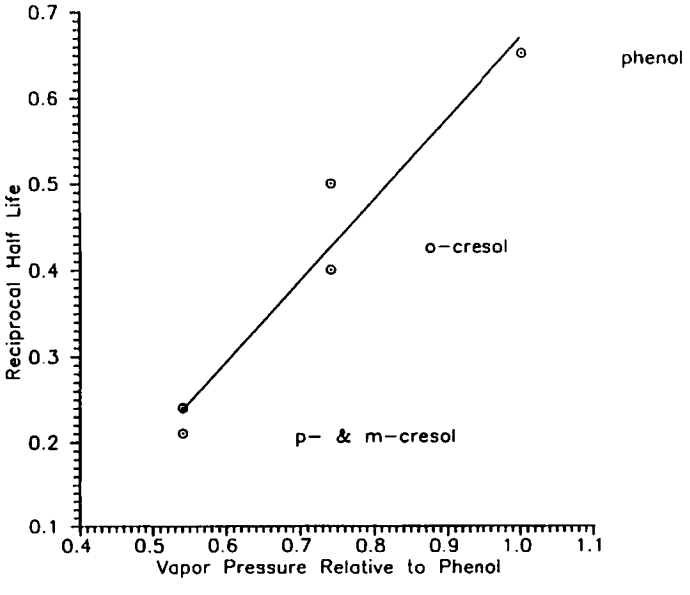

Fig. 4. Relation of relative vapor pressure to reciprocal half life.

water at pH 11.5 and hot (50°C/123°F) water were the most efficient extractants, achieving 100% of the relative cleaning efficiency relative to phenol removal by distillation [21]. Relative effectiveness of water with added surfac-

tant ranged from 72 to 97% while tap water removed 82 to 95% of the phenol (Fig. 5). Tap water was the preferred extractant because of its high removal efficiency, simplicity, and cost effectiveness.

The extraction effectiveness of water may be predicted from the contaminant's hydrophilicity or hydrophobicity. A hydrophilic contaminant has a sol-

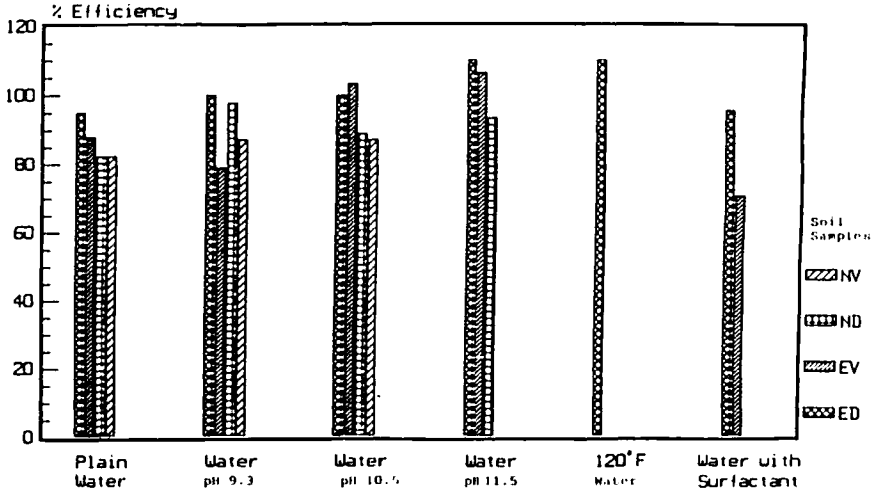

Fig. 5. Relative phenol removal efficiency of soil washing.

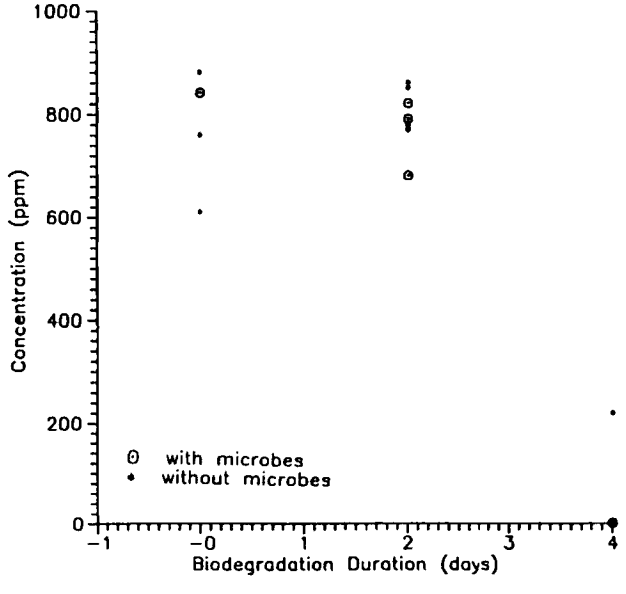

Fig. 6. Phenol biodegradation.

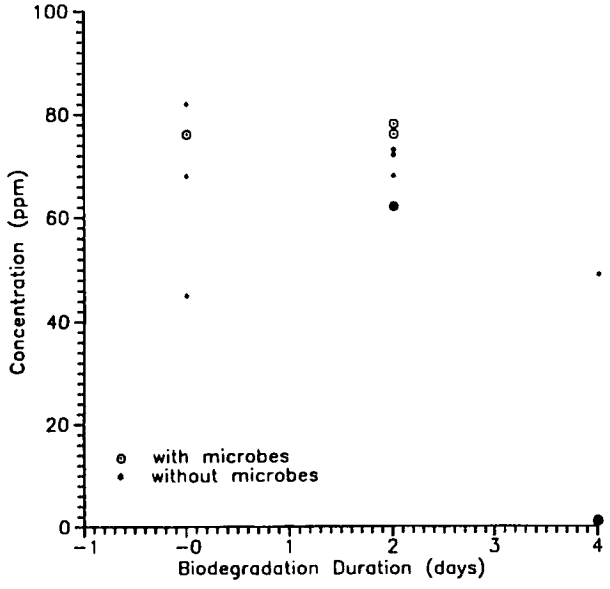

Fig. 7. *Ortho*-cresol biodegradation.

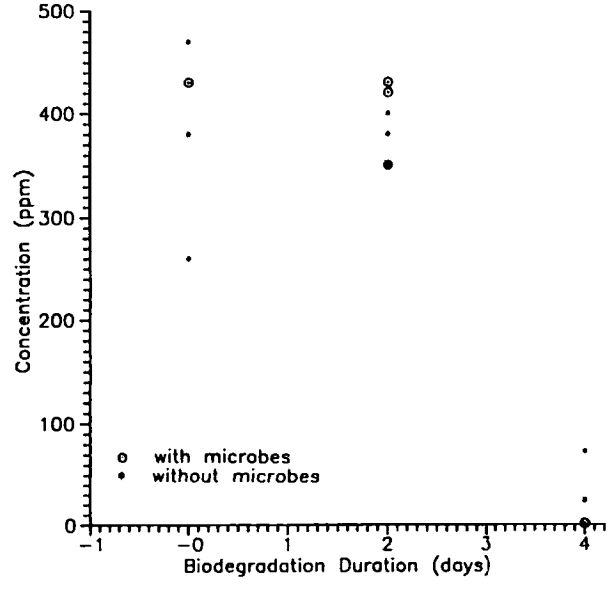

Fig. 8. *Meta*- and *para*-cresol evaporation.

ubility in water, at 25 °C, greater than 10 g/l, while a hydrophobic compound possesses an octanol/water partition coefficient (K_{ow}) greater than 100 [13]. A compound can be both hydrophilic and hydrophobic by this definition. For

aqueous extractants, the target contaminant must be hydrophilic and not hydrophobic. Phenol has a solubility of 84 g/l and a K_{ow} of 29 (a hydrophilic and not hydrophobic contaminant), making it a good candidate for soil washing with an aqueous extractant.

During preliminary soil-free shake-flask biodegradation tests, *P. cepacia* AC1100, a plasmid-enhanced organism, did not grow in buffer solution containing 100 or 500 mg/l phenol. This organism previously degraded various chlorinated phenolic compounds, such as 2,4,5-T and 2,4,5-trichlorophenol (2,4,5-TCP). *Pseudomonas cepacia* did not cross-acclimate to phenol possibly because phenol is without substituted chlorines and is not an intermediate in the degradation pathway of 2,4,5-T or 2,4,5-TCP.

Phenol, *o*-cresol, and *m*- and *p*-cresol were reduced to non-detection levels in most shake flasks containing untreated Poly-Carb soil with *A. eutrophus* JMP134. These results occurred after 4 days incubation; however, degradation was observed in flasks after 2 days (Figs. 6–8). The control flasks containing nutrient buffer and contaminated soil without *A. eutrophus*, showed parallel results.

System design and soil treatment

From the previous engineering studies, soil washing was the treatment of choice. However, due to the previous material handling and soil/liquid separation problems experienced during soil washing at other Superfund sites, a passive system, called soil leaching, was chosen. As a result, an open leach field was designed, constructed, and operated at the Poly-Carb site to water-wash and passively evaporate contaminants from soil. To contain soil contaminants, a half-acre leach field was excavated, graded and double-lined with 60 m thick high-density polyethylene (HDPE) with a 12 in. layer of pea gravel between the HDPE liners to act as leachate collection media (Fig. 9). The system contained 1500 cubic yards of contaminated soil.

The leach field extraction system contained: a water supply; an irrigation system to distribute water onto the soil; a leachate collection system above the

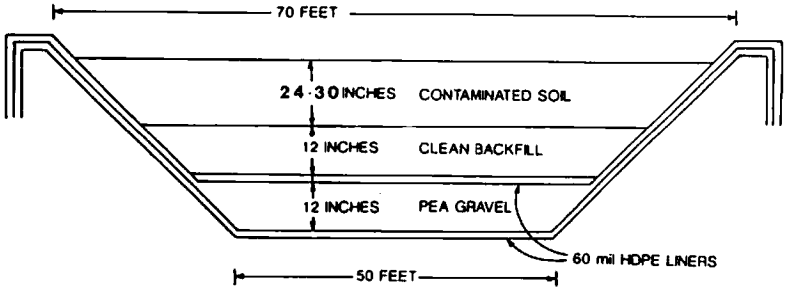

Fig. 9. Leach field cross-section.

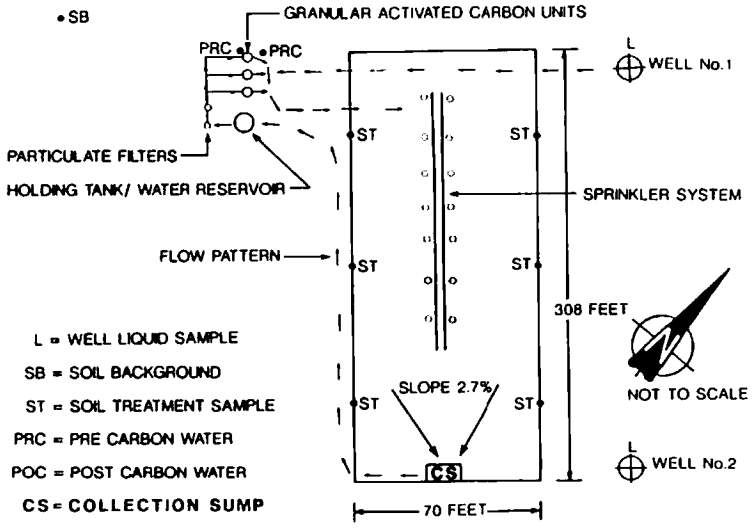

Fig. 10. Poly-Carb site.

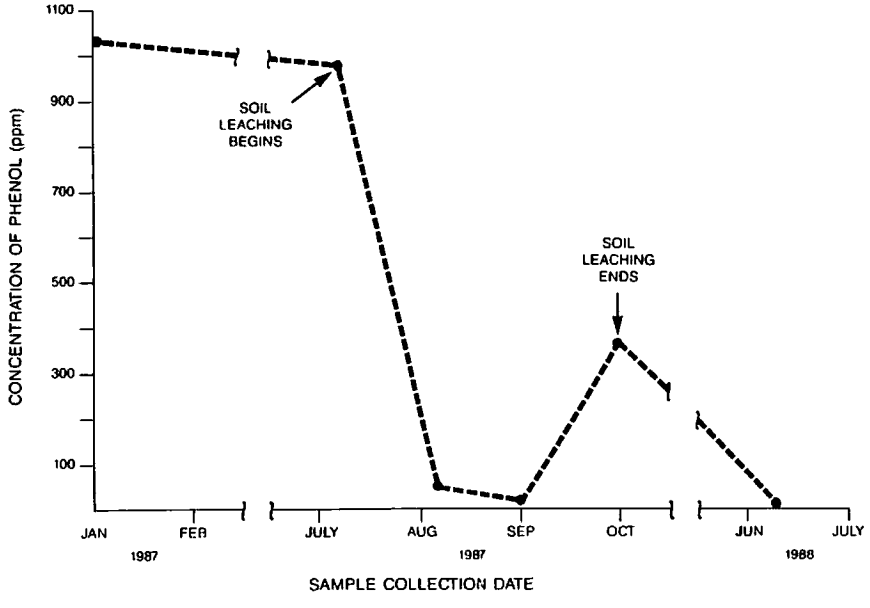

Fig. 11. Sample analysis trends in contaminated soil at Poly-Carb Site, Wells, Nevada.

top liner; a holding tank for contaminated leachate; cartridge particulate filters; disposable, 55 gal (210 l) granular activated carbon (GAC) filters; and necessary pumps (Fig. 10). Soil was irrigated by sprinklers placed in the middle of the leach field to leach contaminants. Leach water percolated through

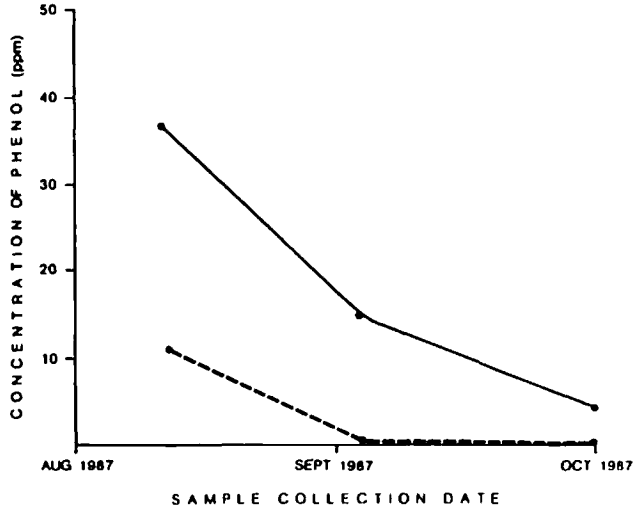

Fig. 12. Sample analysis trends in effluent (---) and influent (-) Leachate at Poly-Carb site, Wells, Nevada. (Treatment begin July 24, 1987.)

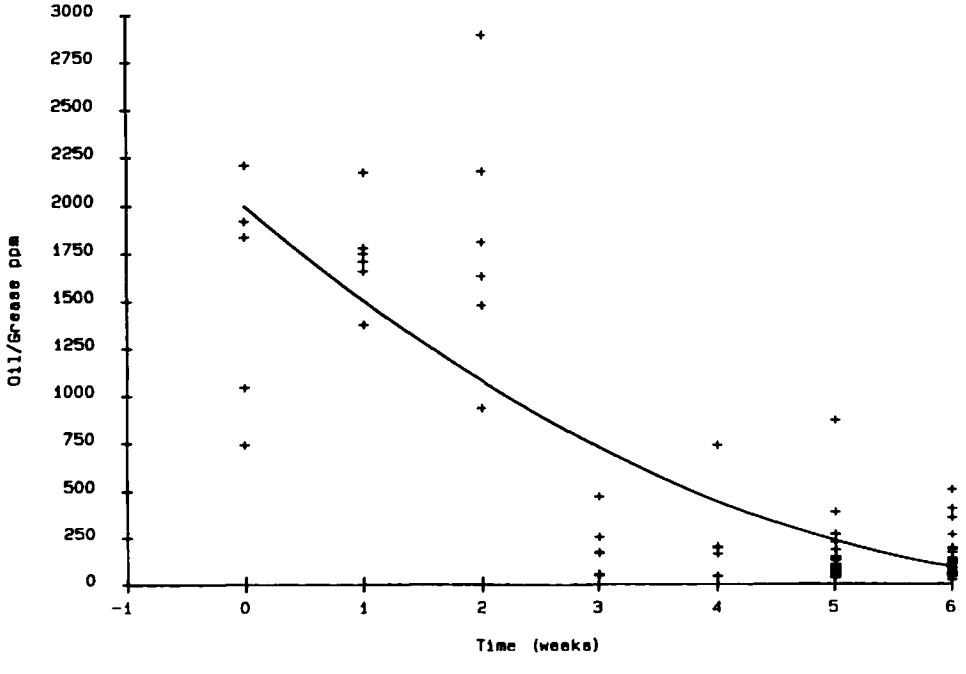

Fig. 13. Oil and grease biodegradation.

the soil, drained to the collection sump, and was transferred to the leachate holding tank where particulates and organics were removed by cartridge filters and GAC, respectively. Leach water was recycled to the irrigation system and make-up water was added to the system as needed.

Soil leaching reduced phenol concentrations in the soil 99.9 percent and lowered cresols 99.7 percent. Initial phenol concentrations in soil ranged from 543 to 1020 μg/g (Fig. 1). When this soil was excavated for deposit into the leach field during July 1987, phenol levels measured 980 μg/g. However, after treatment, phenol dropped to 1 μg/g and below. All samples analyzed for cresols were 1 μg/g and below (Fig. 11). The leachate treatment system performed well. Particulate and GAC filters removed phenol from the leachate before recycling back to the leach field to less than 1 μg/g (Fig. 12). After filtration, the red/purple leachate influent became clear. When the effluent then became yellow, corresponding to an approximate 70% removal of phenol and cresol by the GAC, the GAC filters were replaced (three 55 gal drums per week on the average).

Soil column study using leached soil

The objective of this study was to evaluate the technical feasibility of biodegradation to remove oil and grease and residual phenols and cresols remaining

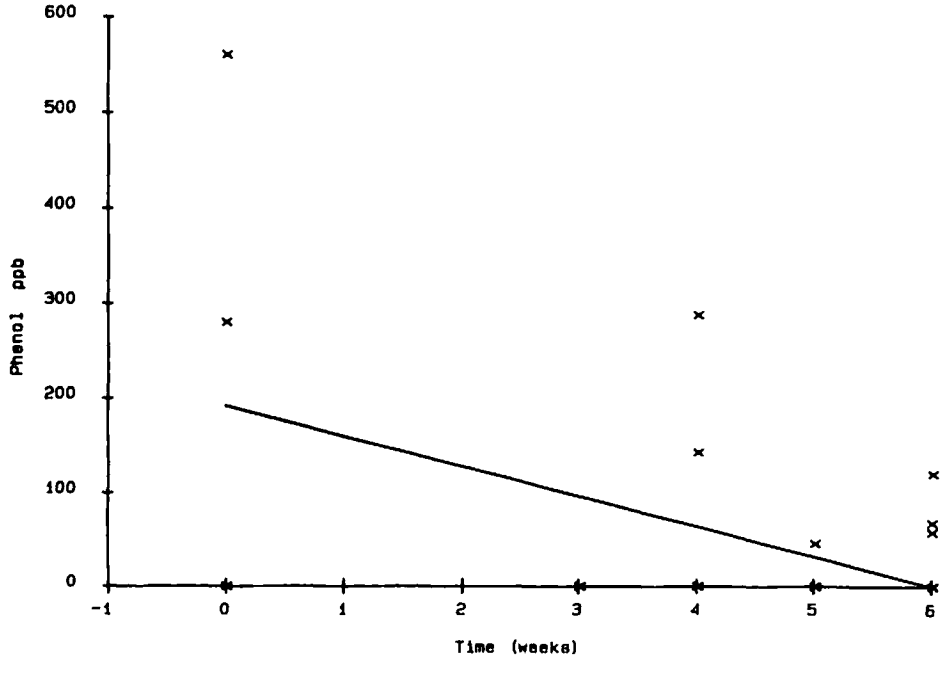

Fig. 14. Phenol biodegradation soil column study.

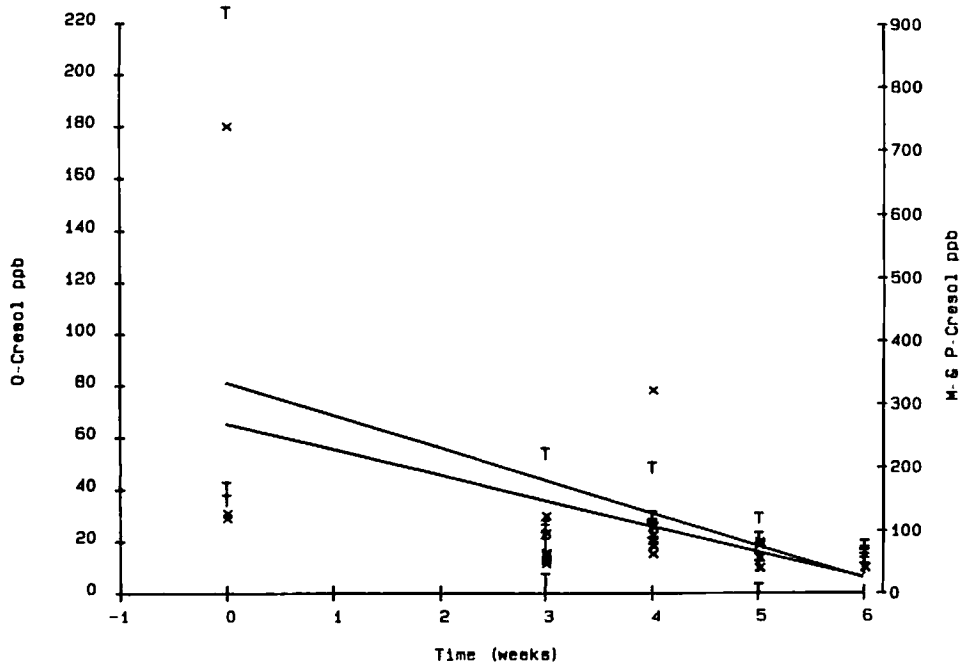

Fig. 15. *Ortho*-cresol (x) and *meta*- and *para*-cresol (T) biodegradation soil column study.

in soil after leach treatment. This study evaluated the use of enhanced biodegradation of leached soil by the indigenous microbes supported by a nutrient solution containing nitrogen, phosphorus, and hydrogen peroxide. Prior to using soil columns, a preliminary degradation feasibility was performed in shake flasks. Oil and grease removals ranged from 8.40 to 99.8% for soil enriched with nutrients and hydrogen peroxide. Based on these high removal rate, soil columns were used to replicate the 18 in. of soil remaining in the leach field.

After six weeks of biodegradation in soil columns, oil and grease removal rates were favorable. Oil and grease was reduced from a mean concentration of 1235 μg/g to a mean 138 μg/g (Fig. 13). Furthermore, phenol and cresol concentrations, in the parts per billion range before treatment, were further reduced after biodegradation (Figs. 14 and 15). Carbon dioxide evolution, a byproduct of organic-compound mineralization, was measured weekly over the course of the test. Fig. 16 shows a second order polynomial fit to carbon dioxide evolution over time. Carbon dioxide evolution increased following the initial introduction of nutrients and an oxygen enhancer, hydrogen peroxide.

Viable cells (i.e., the amount of cells able to reproduce) were counted to monitor the indigenous culture vitality. The amount of viable cells per gram of soil increased throughout the 6-week test (Fig. 17). This increase is an indication that the oxygenated nutrient solution enhanced biodegradation, since

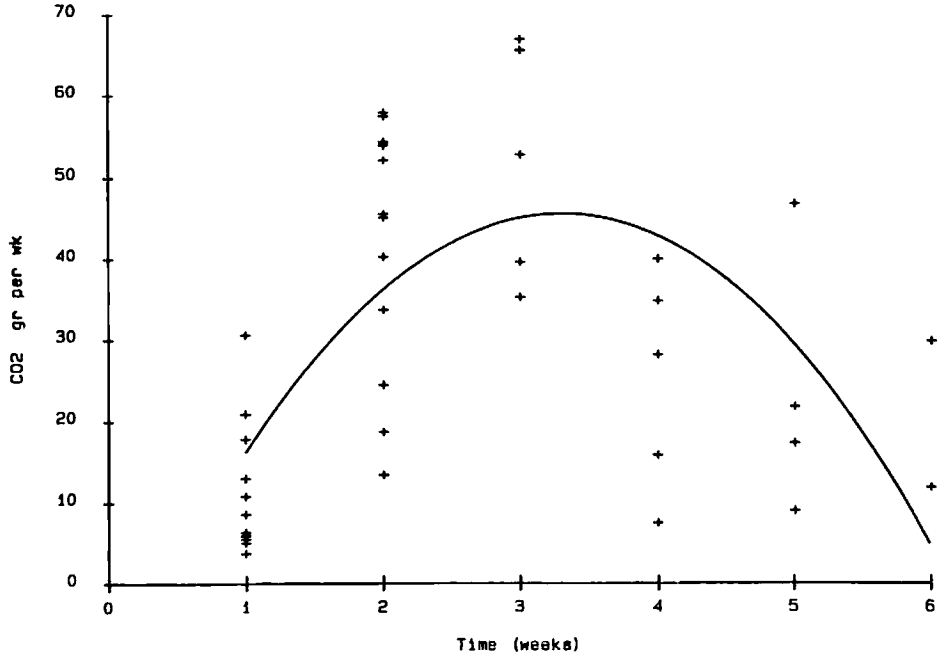

Fig. 16. Carbon dioxide evolution.

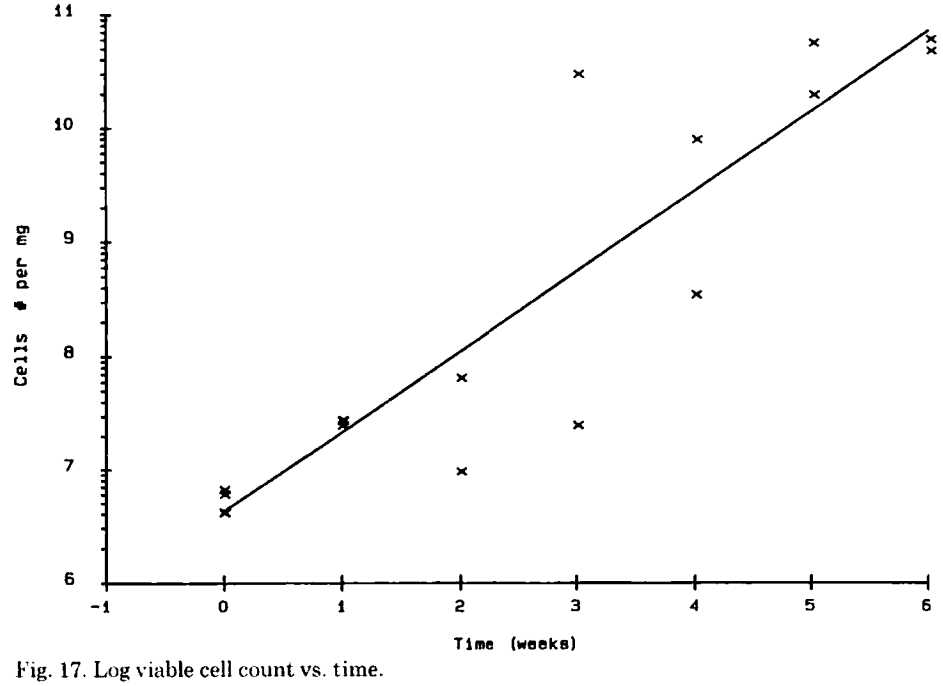

Fig. 17. Log viable cell count vs. time.

the cells must have a carbon source — oil and grease, phenol, and cresols — for energy and cell growth.

Discussion

Passive evaporation effectively reduced soil-bound phenol, *o*-cresol, and *m*- and *p*-cresol. The contaminant reduction rate (or decay) can be expressed by contaminant half-life. These values can predict the time at which a soil contaminant will reach a target concentration or the concentration of a target contaminant in the soil at some future time.

Only the initial slopes were used to determine contaminant half-lifes in Figs. 1–3. Linear decay was observed during the first two weeks; however, third week samples showed non-linearity. The absence of soil mixing during the bench-scale tests may explain this non-linearity. Regular soil mixing allows unevaporated components more contact time with air. Since only the soil surface experiences evaporation (after the components at the surface have evaporated), the limiting factor for evaporation may be internal diffusion of subsurface contaminants through the soil's interstitial space. It is believed that in the absence of mixing, evaporation occurs only at the soil surface. In an alternative evaporation mechanism, contaminants could evaporate in the interstitial space, recondense on the soil particles above it, re-evaporate, and so on until reaching the surface. In both mechanisms, a contaminant molecule faces a tortuous path to the surface which may be reduced by regular tilling and shallow soil depth.

The contaminant half-life was directly related to the contaminant's vapor pressure. A linear relationship existed between the target contaminant's reciprocal half-life and its vapor pressure relative to phenol; the half-life increased with a decreased relative vapor pressure. Therefore, a compound's vapor pressure has a marked effect on its evaporation and thereby its half-life. Consequently, in mixed waste, the duration of evaporation treatment depends upon the compound with the lowest vapor pressure. Environmental, variables which may affect passive evaporation are vapor pressure, ambient temperature, barometric pressure, and wind velocity. The vapor pressure of a contaminant may be increased by increasing soil temperature, through natural or induced methods; a reduction in barometric pressure, at high elevations; and natural or forced convection.

In the shake-flask biodegradation study using untreated soil, biodegradation occurred with or without added microbes in the presence of a nutrient solution. Four days after the addition of *A. eutrophus*, phenol and cresols were below detection limits in most flasks. Similar phenol and cresol degradations occurred in the control flasks with no added microbes. This result indicates that the indigenous soil microorganisms have adapted to the target contaminants and can degrade them. Phenol was not degraded by the plasmid enhanced *P. cepacia* even though this microorganism has successfully degraded the recal-

citrant chlorinated phenoxyacetate herbicides 2,4,D and 2,4,5-T. Apparently, the organism was not able to cross-acclimate to the simpler, unchlorinated phenol compound.

A subsequent soil column biodegradation study was initiated to explore the destruction of residual organics in soil after water leach treatment, the method utilized for full-scale remediation at the site. In the 6-week test, the concentrations of oil and grease and residual phenol and cresol (1 μg/l or below) were lowered. We concluded from these results that it is technically feasible to use biodegradation with enhanced indigenous soil microbes to reduces target organics in soil remaining in the Poly-Carb leach field. Although the final concentrations of phenol and cresol were further reduced by this technique, great care must be exercised in interpreting data containing such low contaminant concentrations in a soil matrix for a short test duration. The increase in CO_2 evolution rate corresponded to the decrease in contaminant concentration. This increase is an indication of enhanced microbial respiration due to biodegradation. The active microbes responded with an increased reproduction rate, as measured in viable cell counts.

For Poly-Carb soil, soil washing was an effective remediation technique. The alkaline water at pH 11.5 was the best extractant for phenol. Its extraction effectiveness may be due the removal of the hydroxyl proton on phenol and the subsequent greater solubility of the sodium phenate salt. The heated water was an excellent phenol extractant; however, capital and operating cost of a system using hot water may preclude its use. Furthermore, the surfactant solution did not perform as well as the previous extractants. Even though it did not have the highest extraction efficiencies, water was the extractant of choice because of the operational cost, the reduction of extractant recovery or destruction problems, and the lower treatment costs.

Because of the effectiveness of soil washing with water for removing phenol from untreated soil during bench-scale studies, the process considerations of soil washing were explored. The implementation of a soil washing system at the site would involve soil excavation, material handling, slurry mixing, soil/liquid separation, and site restoration. Historically, material handling and soil liquid separation have been problematic at several sites: Lee Farm, Woodsville, Wisconsin; Church of God, Leeds, Alabama; and, Shaffer Equipment, Minden, West Virginia [9,10,13,24]. To avoid these problems, soil leaching, a passive form of soil washing, was preferred over the previous active processes.

Conclusions and recommendations

The conclusions and recommendations based on the engineering study are:

- Bench-scale engineering studies were essential prior to soil remediation.
- These studies found soil washing, biodegradation, and passive evaporation were viable treatment options.

- Shake-flash biodegradation reduced contaminants in soil with or without added microbes.
- Soil column biodegradation studies demonstrate that residual organics, oil and grease, phenol, and cresols in leached soil can be reduced through the activity of indigenous microbes with the help of a nutrient solution.
- Soil leaching, a passive form of soil washing, was the recommended remediation technique because water was an effective extractant and materials handling and soil/liquid separation would be avoided.
- Due to the full-scale treatment success of soil leaching, additional development should be done to solve the problems of soil washing.

Acknowledgments

The authors of this paper would like to sincerely thank the following people for their special contributions: Jim Nash, Rich Hougland, Lawrence Kaelin, Howard Syvart, and Peter Schultze for their efforts with the soil washing and biodegradation studies, John Syslo, Jeff Schmidt, and Renata Wynnyk for his gas chromatograph work, his metal analysis, and her oil and grease analysis, respectively; Shekhar Iyer for his computer generated graphs; and Richelle Drummond and Eileen Hoelz for word processing. The dedication and skill of these people greatly contributed to the success of this project.

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