



Project Summary

Treatment of Contaminated Soils with Aqueous Surfactants

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The full report presents the results, conclusions, and recommendations of a project performed to develop a technical base for decisions on the use of chemical countermeasures at releases of hazardous substances. The project included a brief literature search to determine the nature and quantities of contaminants at Superfund sites and the applicability of existing technology to *in situ* treatment of contaminated soils. Laboratory studies were conducted to develop an improved methodology applicable to the *in situ* treatment of organic chemical contaminated soil.

Current technology for removing contaminants from large volumes of soils (too large to excavate economically) has been limited to *in situ* "water washing." Accordingly, the laboratory studies were designed to determine whether the efficiency of washing could be enhanced significantly (compared to water alone) by adding surfactants to the recharge water and recycling them continuously.

The use of an aqueous nonionic surfactant pair for cleaning soil spiked with PCBs, petroleum hydrocarbons, and chlorophenols was developed through bench scale shaker table tests and larger scale soil column tests. The extent of contaminant removal from the soil was 92 percent for the PCBs, using 0.75 percent each of Adsee® 799 (Witco Chemical) and Hyonic® NP-90 (Diamond Shamrock) in water. For the petroleum hydrocarbons, the removal with a 2 percent aqueous solution of each surfactant was 93 percent. These removals are orders of magnitude greater than obtained with just water washing and represent a significant improvement over existing *in situ* cleanup technology.

Treatability studies of the contaminated leachate were also performed to investigate separating the surfactant from the contaminated leachate to allow reuse of the surfactant. A method for separating the surfactant plus the contaminant from the leachate was developed; however, all attempts at removing the surfactant alone proved unsuccessful.

Based upon the results of the laboratory work, the aqueous surfactant countermeasure is potentially useful for *in situ* cleanup of hydrophobic and slightly hydrophilic organic contaminants in soil, and should be further developed on a larger scale at a small contaminated site under carefully controlled conditions. However, reuse of the surfactant is essential for cost-effective application of this technology in the field. Accordingly, any future work should investigate the use of other surfactants/surfactant combinations that may be more amenable to separation.

This Project Summary was developed by EPA's Hazardous Waste Engineering Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA or Superfund) recognizes the need to develop countermeasures (mechanical devices, and other physical, chemical, and biological agents) to mitigate the effects of hazardous substances that are released into the envi-

ronment and clean up inactive hazardous waste disposal sites. One key countermeasure is the use of chemicals and other additives that are intentionally introduced into the environment for controlling the hazardous substance. The indiscriminate use of such agents could, however, worsen the contamination situation.

The U.S. Environmental Protection Agency's Hazardous Waste Engineering Research Laboratory has initiated a Chemical Countermeasures Program to define technical criteria for the use of chemicals and other additives at release situations of hazardous substances such that the combination of the released substance plus the chemical or other additive, including any resulting reaction products, results in the least overall harm to human health and to the environment. Under the Chemical Countermeasures Program, the efficacy of *in situ* treatment of large volumes of subsurface soils, such as found around uncontrolled hazardous waste sites, and treatment of large, relatively quiescent waterbodies contaminated with spills of water soluble hazardous substances, will be evaluated. For each situation, the following activities are planned: a literature search to compile the body of existing theory and data; laboratory studies on candidate chemicals to assess adherence to theory and define likely candidates for full-scale testing; full-scale, controlled-condition, reproducible tests to assess field operation possibilities; and full-scale tests at a site requiring cleanup (i.e., a "site of opportunity").

This project, to develop the use of aqueous surfactants for *in situ* washing of contaminated soils, was the first technique to be developed under the Chemical Countermeasures Program. The results and conclusions from an information search formed the basis for the laboratory development work. Similarly, the results and conclusions from the laboratory work are intended to provide the basis for another project involving large-scale testing of a chemical countermeasure, either in a large test tank or under controlled conditions at a site of opportunity.

Information Search

The information search was conducted to determine the nature and quantities of hazardous soil contaminants at Superfund sites, and to assess the applicability of existing technology for *in situ* treatment of contaminated soils. To determine what

types of soil contaminants requiring cleanup were likely to be found at hazardous waste sites, a survey was made of the contaminants at 114 high priority Superfund sites. The classes of chemical wastes found at the greatest number of sites, in order of decreasing prevalence, were: slightly water soluble organics (e.g., aromatic and halogenated hydrocarbon solvents, chlorophenols), heavy metal compounds, and hydrophobic organics (e.g., PCBs, aliphatic hydrocarbons).

A variety of chemical treatment methods were considered that might prove effective in cleaning up soils contaminated with these wastes. However, methods for *in situ* chemical treatment of soils will probably be most effective for certain cleanup situations, such as those in which:

- The contamination is spread over a relatively large volume of subsurface soil, e.g., 100 to 100,000 m³, at a depth of 1 to 10 m; or
- The contamination is not highly concentrated, e.g., not over 10,000 ppm, or the highly contaminated portion of the site has been removed or sealed off; or
- The contaminants can be dissolved or suspended in water, degraded to non-toxic products, or rendered immobile, using chemicals that can be carried in water to the zones of contamination.

For contamination less than 1 m deep, other methods such as landfarming (surface tilling to promote aerobic microbial degradation of organics) would probably be more practical. For highly contaminated zones of an uncontrolled hazardous waste landfill or a spill site, methods such as excavation and removal, or excavation and onsite treatment would probably be more practical than *in situ* cleaning of the soil.

Findings under the information search indicated that aqueous surfactant solutions might be applicable for *in situ* washing of slightly hydrophilic (water soluble) and hydrophobic organics from soils. Texas Research Institute (TRI) used a combination of equal parts of Witco Chemical's Richonate®* YLA, an anionic surfactant, and Diamond Shamrock's Hyonic® NP-90, a nonionic surfactant, in several laboratory column and two-dimensional modeling studies for displacing gasoline from sand packs.

*Mention of trade names or commercial products does not constitute endorsement or recommendation for use

To further verify which organic waste chemicals should be targeted for countermeasures development, Field Investigation Team (FIT) summaries were examined for the maximum concentrations of organic contaminants in the soil and groundwater surrounding 50 Superfund sites. Results of the survey indicated that many hydrophobics were detected in the soils, mainly because hydrophilics tend to be washed from soil by infiltrating rainwater. Hydrophobics had the highest levels of all the organic contaminants, with 11 compounds averaging in the 1 to 100 ppm range, and with chlordane exceeding 1,000 ppm at one site. The soil concentrations of slightly hydrophilic compounds were in the range of 0.001 to 10 ppm.

Based on these findings, the following two hydrophobic and one slightly hydrophilic pollutant groups were chosen as model contaminants for testing and development of an aqueous surfactant countermeasure:

- High boiling point Murban crude oil fraction containing aliphatic and aromatic hydrocarbons (1,000 ppm)
- PCB mixture in chlorobenzenes (Aroclor® 1260 transformer oil) (100 ppm)
- Di-, tri-, and pentachlorophenols mixture (30 ppm)

Laboratory Studies

The laboratory research was conducted to determine whether significant improvements to the cleanup of contaminated soils with just water, the only *in situ* soil cleanup method demonstrated to date, could be obtained using aqueous surfactants. Further laboratory development of the surfactant countermeasure included optimizing the concentration of surfactant used for cleanup, and development of contaminated leachate treatment methods.

The aqueous surfactant countermeasure was tested using two basic methods: shaker table agitation, to quickly determine the soil/aqueous surfactant partitioning of the model contaminants under differing conditions; and gravity flow soil column tests to verify the cleanup behavior of the aqueous surfactant under conditions resembling field use. Besides the optimum surfactant concentration, the effects of leachate treatment and recycling were also studied.

Soil Characterization

In choosing a soil for the surfactant washing tests, the applicability of the

results to actual field situations was a primary consideration. Selection included identifying the native soils at the ten Region II Superfund sites for which data was available, determining the most commonly occurring soil type series, and locating a soil of the same soil taxonomic classification which could be excavated and used in the testing experiment. In addition to taxonomic classification, a permeability rating of 10^{-2} to 10^{-4} cm/sec was desirable since less permeable soils would take too long to test.

A Freehold soil series typic hapludult soil was chosen for the study. The total organic carbon content (TOC) of the soil was 0.12 percent by weight, implying a relatively low contribution by organic matter to the adsorption of organic contaminants. The cation exchange capacity (CEC) of the soil was determined to be 8.6 milliequivalents per 100 gms, an extremely low value, indicating an absence of mineralogic clay in the soil.

Using a percent moisture content of 11 percent and compacting the soil in the columns to a density of 1.68 g/cm^3 (105 lb/ft^3), an optimum percolation rate of 1.5×10^{-3} cm/sec was obtained under a constant 60 cm head.

Surfactant Selection

The surfactant combination used by TRI for flushing gasoline from sand, Richonate® YLA and Hynic® NP-90 (formerly called Hynic® PE-90), was screened along with several other surfactants and surfactant combinations for the following critical characteristics: adequate water solubility (deionized water), low clay particle dispersion, good oil dispersion, and adequate biodegradability. The surfactants selected for ultimate use in the laboratory studies were Adsee® 799 (Witco Chemical) and Hynic® NP-90 (Diamond Shamrock).

Soil Contamination Procedures

Soil was contaminated using an aerosol spray of the contaminant mixture dissolved in methylene chloride. The methylene chloride was allowed to evaporate, and the soil was mixed by stirring in pans. The soil was then tested in shaker or column studies.

Column Packing

The soil columns used in this study were 7.6 cm (3 in.) inside diameter by 150 cm (5 ft) long glass columns. A plug of glass wool was placed at the bottom of the column and successive plugs of contaminated soil weighing approximate-

ly 775 g were packed to a height of 10 cm (4 in.) each. To ensure better cohesion between layers, the upper 1/4 inch of each plug was scarified. The soil was packed to a total height of 90 cm (3 ft) and compacted to a density of 1.68 to 1.76 g/cm^3 (105 to 110 lb/ft^3), yielding a percolation rate which was comparable to its natural permeability.

Shaker Table Tests

Shaker table partitioning experiments were conducted to determine the minimum surfactant concentration required to accomplish acceptable soil cleanup. After spiking Freehold soil with PCBs and hydrocarbons, separately, surfactants were used to wash the soil by shaking in containers on a constantly vibrating shaker table.

One hundred grams of contaminated soil were agitated with 200 ml of the appropriate surfactant concentration on a shaker table for one hour, then centrifuged, and decanted. Both soil and leachate were analyzed to determine how much of the contaminant had been removed.

Soil Column Experiments

During the first year of study, the effect of soil washing with water, followed by 4.0 percent surfactants (2 percent each), and a final water rinse was investigated in soil column experiments using Murban distillate cut, PCBs and di-, tri-, and pentachlorophenol contaminants. Freehold soil was spiked, separately, with 1,000 ppm Murban distillate cut, 100 ppm PCB, and 30 ppm chlorinated phenols.

Results of these column experiments showed that the initial water wash had little effect; however, with surfactant washing, 74.5 percent of the pollutant was removed by the leachate after the third pore volume (i.e., volume of void space in the soil). Additional surfactant increased the removal to 85.9 percent after ten pore volumes. The pollutant concentration in the soil was reduced to 6 percent of the initial spike value after the tenth pore volume of surfactant. The final water rinse also showed only minimal effects.

Almost identical behavior was observed for the column experiments using PCB spiked soil: the initial water wash was ineffective, but the soil was cleaned substantially by the 4.0 percent surfactant solution. After the tenth pore volume, 68 percent of the PCBs were contained in the leachate, leaving only 2 percent on the soil.

Similar soil column experiments were also conducted using a mixture of di-, tri-, and pentachlorophenols, and, in contrast to the PCB and Murban distillate cut results, 64.5 percent of the chlorinated phenols were removed by the first water wash, and only 0.56 percent remained on the soil after the tenth pore volume of water.

Optimization of Surfactant Concentration

To make soil washing techniques cost effective, it was necessary to determine the minimum concentration of surfactant that would yield acceptable soil cleanup. Surfactant concentrations were varied from 0 to 1.0 percent (2 percent total surfactant) in shaker table experiments using both PCB and hydrocarbon contaminated soils. Column experiments were then undertaken to verify shaker table data and to further optimize surfactant concentrations.

Figure 1 shows the effect of surfactant concentration on PCB partitioning between soil and leachate. There was essentially no cleanup of the soil with surfactant concentrations of 0.25 percent (0.50 percent total) or below. Similar PCB partitioning was observed for 0.75 percent and 1.0 percent individual surfactant concentrations, and the most effective cleanup occurred at these levels.

As Figure 2 shows, similar soil/leachate partitioning behavior was also observed for Murban hydrocarbons with varying surfactant concentrations. Individual surfactant concentrations of 0.25 percent and below were ineffective; increased surfactant concentrations caused increased soil cleanup from 0.50 to 0.75 percent surfactant; above 0.75 percent surfactant concentration little enhancement of soil cleanup occurred.

Column Verification

To ensure that the optimum surfactant concentration under gravity flow conditions was not significantly different than under equilibrated shaker table conditions, columns packed with Freehold soil spiked with 100 ppm PCBs were also tested with varying surfactant levels.

The columns were treated with one, two, or three pore volumes of 0.50, 0.75, or 1.0 percent surfactant before sacrifice and soil analysis. The downward migration of PCBs is apparent in Figure 3, which presents the PCB concentrations in the various portions of the columns as a function of pore volume for each of the three surfactant concentrations tested. PCB mobilization was not much greater

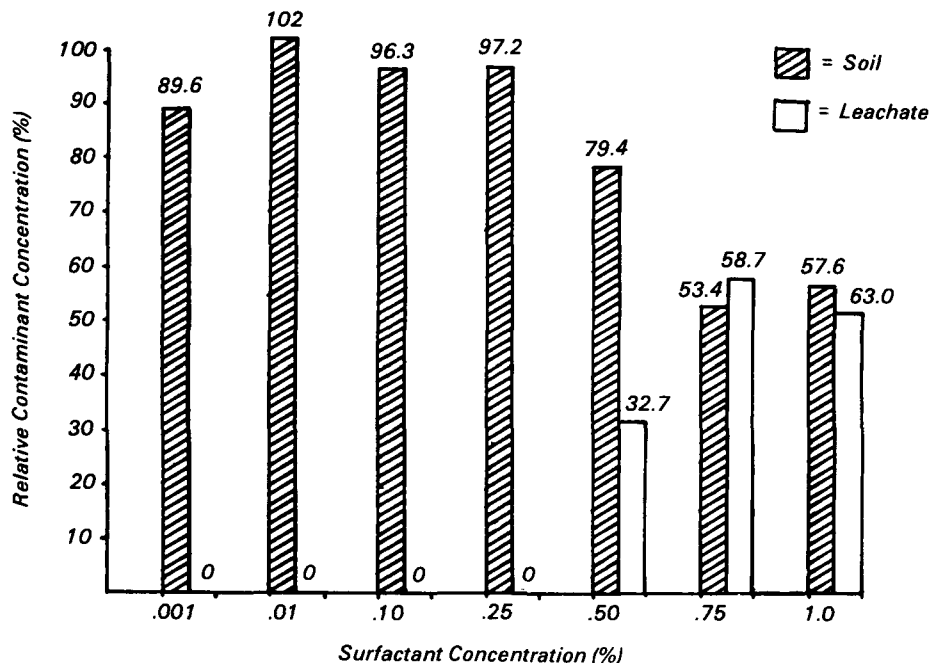


Figure 1. PCB Shaker table recoveries vs. surfactant concentration.

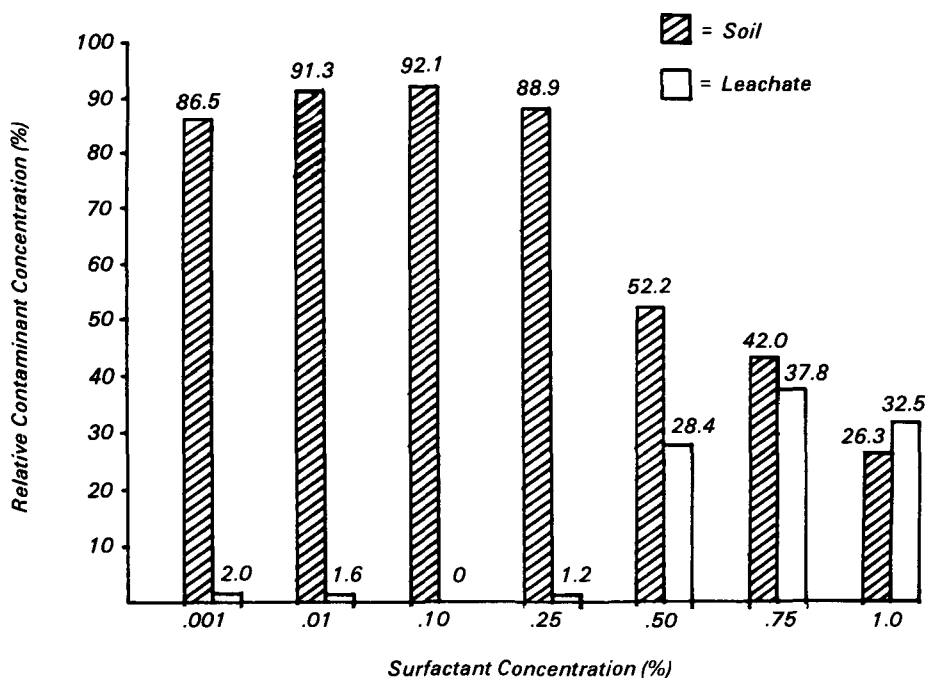


Figure 2. Murban Shaker table recoveries vs. surfactant concentration.

with 0.75 percent surfactant than with 1.0 percent surfactant, and somewhat less for the 0.50 percent surfactant concentration. After three pore volumes, the PCB concentrations at the bottom of the column were of 244 $\mu\text{g/g}$ with the 0.50 percent surfactant, compared with 405 $\mu\text{g/g}$ using 0.75 percent surfactant and 562 $\mu\text{g/g}$ using the 1.0 percent surfactant.

Results of the column experiments, coupled with the results of the shaker table experiments, indicate that the optimum surfactant concentration for soil cleanup is about 0.75 percent of each surfactant or 1.5 percent total surfactant.

Evaluation of Leachate Treatment Techniques

Large amounts of surfactants and wash water are required for field application of this countermeasure technology. Surfactants are expensive, and for this technology to be cost effective, surfactant recycling is an important consideration. Accordingly, various leachate treatment techniques were evaluated for their ability to remove and concentrate the contaminants, while leaving the surfactants behind for further use. All treatment methods evaluated were ineffective in separating the contaminants from the surfactant. However, several leachate treatment techniques were able to (1) concentrate the contaminants to facilitate disposal, and (2) clean the water enough that it could be sent to a publicly owned treatment works (POTW) or reused.

Four treatment alternatives were tested, and the conditions for efficient leachate treatment were optimized in preparation for large-scale field testing. Foam fractionation, sorbent adsorption, ultrafiltration, and surfactant hydrolysis were subjected to preliminary laboratory tests using simulated leachate.

The results of the foam fractionation tests showed that good cleanup of the leachate was achieved if the concentration of surfactant was below about 0.1 percent, while no significant reduction in surfactant occurred at starting concentrations above that.

Eleven solid sorbents were tested for their efficiency in removing PCBs and the surfactants from an aqueous solution. None of the sorbents was very efficient in removing PCBs from a surfactant solution. The most efficient sorbent for PCB removal was the Filtrol XJ-8401, with an efficiency of 0.00045 g/g; WV-G 12x40 Activated Carbon, and Celkate magnesium silicate were most efficient in overall surfactant and PCB removal (0.195 g/g).

Hydrolysis treatment of the surfactant and contaminant-containing leachate was also tested. Adsee® 799, a fatty acid ester, formed a separate organic phase upon hydrolysis that contained both the surfactants and 95 percent of the organic contaminants.

Further treatment of the aqueous surfactant solution with a column of activated carbon (Westvaco Nuchar WV-B 14x35) yielded a solution containing only 0.01 ppm of PCBs. Foam fractionation was also used as a polishing method for removing traces of surfactants from aqueous solutions. A four-column series of foam fractionation columns operating in a continuous countercurrent flow mode was used. The test results demonstrated that the residual PCB level (0.0036 ppm) should be low enough to allow disposal to a POTW, and low enough to permit reuse of the leachate water for soil cleaning. However, the use of hydrolysis was necessary for the higher surfactant concentrations found in the raw leachate.

Evaluation of Leachate Recycling

To evaluate the effect of recycling the untreated aqueous leachate on soil cleanup, column experiments were conducted. The results showed that leachate recycling—without some sort of treatment—is not an acceptable method, as contaminants become redistributed back onto the soil with each successive pass. However, a column experiment in which the recycled leachate was treated between each pass showed very effective cleanup of soil.

Between passes, fresh surfactant was added to the treated leachate prior to recycling, and the soil in the column received four passes of fresh surfactant; only the water was recycled. After four passes, less than 1.0 percent of the original soil contamination remained.

Conclusions and Recommendations

Effectiveness of the Surfactants

Based on bench-scale tests designed to screen potential surfactants for use as *in situ* soil washing enhancers, a 1:1 blend of Adsee® 799 (Witco Chemical Corp.) and Hyonic® NP-90 (Diamond Shamrock) was chosen because of adequate solubility in water, minimal mobilization of clay-sized soil fines (to maintain soil permeability), good oil dispersion, and adequate biodegradability.

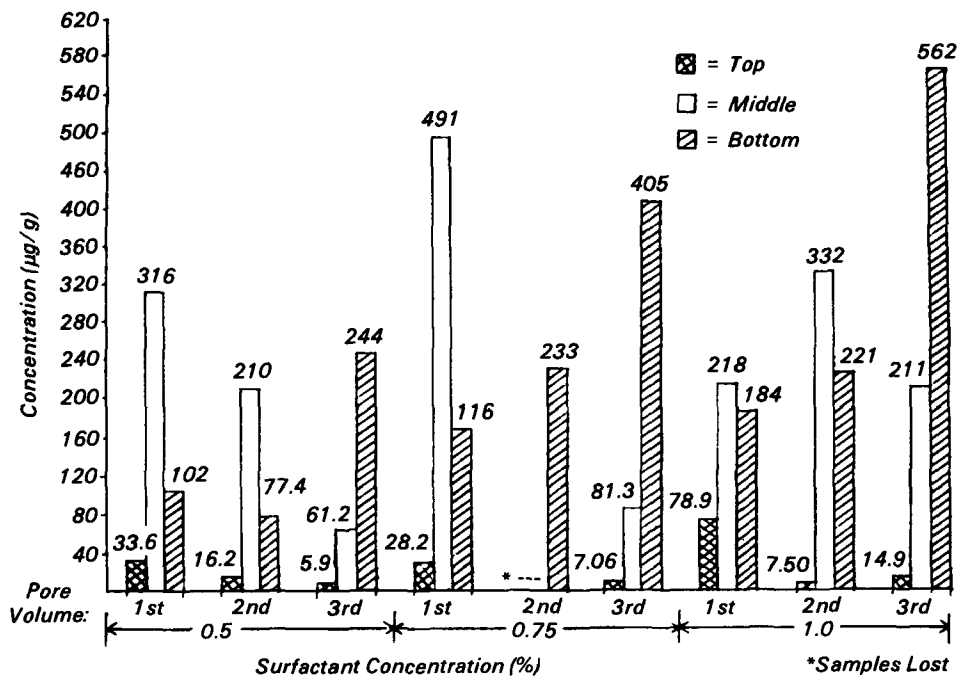


Figure 3. PCB soil column cleanup vs. surfactant concentration.

Shaker table and column experiments show that 4.0 percent of this blend of surfactants in water removed 93 percent of the hydrocarbon and 98 percent of the PCB pollutants from contaminated soil. These removals are orders of magnitude greater than those obtained with just water washing and represent a significant improvement to the efficiency of existing technology. Chlorinated phenols were readily removed from the test soil by water washing alone.

Shaker table experiments conducted to determine the optimum surfactant concentration for soil cleanup, with PCB and petroleum hydrocarbon (Murban) contaminated soils, showed the optimum concentration to be 1.5 percent total surfactant. Individual surfactant concentrations of 0.25 percent or less were unacceptable for effective soil washing, and individual surfactant concentrations above 0.75 percent (1.5 percent total) were excessive, since no significant enhancement of cleanup resulted. In addition, similar partitioning between soil and surfactant solution by the two pollutant types suggests that the results which would be obtained in further large-scale experiments with the low toxicity hydrocarbons in a fuel oil like Murban might reliably model the behavior of other more toxic hydrophobic pollutant groups, such as PCBs.

The experiment which evaluated the effect of leachate recycling, with treatment applied to the PCB leachate between cycles, showed that:

- Soil cleanup with 1.5 percent total surfactant is good, with less than 1 percent of the PCB remaining on the soil.
- The product of hydrolysis represents a relatively small volume (about 12 percent of the total mass of leachate) of highly contaminated material, which can be further treated by incineration, or disposed of for a minimal cost.
- The use of the same water for repeated cycles precludes the generation of large volumes of waste leachate.
- The final treated water after four cycles contains less than 0.0005 percent of the initial contamination encountered in the soil.

Additional surfactant tests are warranted before this technology can be applied in the field. The surfactant combination used was water soluble, and effective in soil cleanup, and allowed good soil percolation rates, as the mixture did not resuspend a significant amount of the clay-sized particles in the soil, thereby inhibiting flow. These characteristics are

definitely important; however, for this technology to be cost effective, reuse of the surfactant is equally important. Accordingly, it is recommended that other surfactants/surfactant combinations be evaluated that have the same "flushing" characteristics but are also more amenable to separation for reuse. The surfactant should be screened for solubility, clay dispersion, and oil dispersion, and should also be screened by mutagenicity tests to avoid the distinct possibility that the release situation could be made worse by the application of a toxic chemical or other additive.

Effects of the Test Soil

The efficiency of cleanup of the hydrophobic organic contaminated Freehold soil by the aqueous surfactant solution was directly affected by the low natural organic carbon content of the soil. The low TOC (0.12 percent) represented little organic matter in the soil to adsorb the organic pollutants spiked onto the soil, so the contaminant removal could be expected to be relatively easy compared to a soil with, for example, a 1 percent TOC. The removal of hydrophobic organics from a 1 percent TOC soil using the Adsee® 799 - Hyonic® NP-90 surfactant pair would require more surfactant solution. Also, the surfactants would become necessary for removing chlorophenols from a 1 percent TOC soil; water alone would not be very effective.

If additional laboratory or pilot-scale testing were undertaken, a second soil type with greater percentages of organic carbon should be considered for testing to expand the overall applicability of the program results to a broader variety of soil matrices.

The hydraulic conductivity of the Freehold soil packed in the soil columns, which was measured at 1.05×10^{-3} cm/sec, would be practical for field implementation of the countermeasure. However, the time required for surfactant solution to flow through the soil should be considered. With this hydraulic conductivity, if surface flooding were used to obtain saturated conditions from the surface to a groundwater depth of 10 m (33 ft), and assuming a porosity of 50 percent, it would take 5.5 days for one pore volume of solution to flow through the soil from surface to groundwater. A flow rate under similar conditions, with a soil permeability of 1×10^{-4} cm/sec, would yield flow rates of about 1.2 m/wk, which is probably a practical lower limit for the method.

Potential Target Contaminants

The types of hazardous chemicals for which the surfactant countermeasure was more effective than water without surfactant, included hydrophobic organics (PCBs and aliphatic hydrocarbons in the Murban fraction) and certain slightly hydrophilic organics (aromatic hydrocarbons in Murban). The chemicals for which the method is probably not applicable are heavy metal salts and oxides, and cyanides. For soils with low TOC values, chlorophenols and certain other slightly hydrophilic organics can be removed with water alone. However, for soils with high TOC values, the use of aqueous surfactants would significantly improve the removal efficiency of slightly hydrophilic organics.

Effective Treatment Methods

A need to conserve both water and surfactant prompted the investigation of leachate reuse or recycling. Recycling of the untreated leachate is unacceptable because portions of the soil that have been previously cleaned are recontaminated rapidly by the introduction of spent leachate. The ideal treatment method removes and concentrates contaminants while leaving the surfactants behind for further use. However, the same chemical and physical properties of the surfactant mixture that help to extract the pollutants from the soil also inhibit separation of the contaminants from the surfactants. Due to the high (percentage) level of surfactant contained in the leachate, most of the treatment methods evaluated were ineffective. The best treatment that could be obtained removed both surfactants and pollutants, leaving clean water for possible reuse or easy disposal.

Additional efforts should be directed toward optimizing feasible and cost-effective methods of leachate treatment and in particular separation of the surfactant for reuse. Ultrafiltration appears promising and warrants further investigation along with foam fractionation. The use of already existing equipment and technologies should be examined in greater detail to minimize scale-up costs.

Further Countermeasure Development Before Field Use

The testing of a new technique, in which hazardous contaminants are rendered more mobile, presents a potentially greater environmental threat unless the tests can be readily stopped at any point as required to permit the immediate remedy of any failure by established

techniques. Because the aqueous surfactant countermeasure is still developmental, the field tests should be conducted on a reduced scale until the procedures are proven workable and the important parameters are understood and controlled.

The laboratory tests have established that the technique of *in situ* washing with aqueous surfactants is sufficiently effective for soil cleanup to justify tests on a larger scale. Pilot-scale testing requires the use of disturbed soil, and will probably not further the development of the method as much as controlled-condition field testing at a site of opportunity. An appropriate site for field testing should have the following characteristics:

- Moderate to high permeability (coefficient of permeability of 10^{-4} cm/sec or better)
- Small size (e.g., 30 m x 30 m x 10 m deep)
- Minimal immediate threat to drinking water supplies
- Hydrophobic and/or slightly hydrophilic organic contaminants
- Concentrated contamination source removed or controlled
- Low to moderate natural organic matter content in soil (TOC 0.5 to 2 percent).

If either small sites, or physically separated sections of a large site (e.g., with a slurry or grout wall) were selected, the aqueous surfactant countermeasure described in this report could be applied, tested further, and improved to a point of full field countermeasure applicability. However, future work should evaluate other surfactants that have the same cleanup characteristics as those used in the laboratory studies but are more amenable to separation for reuse. Also, prior to any larger scale/site of opportunity studies, the toxicity of the surfactants should be ascertained.

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Anthony N. Tafuri is the EPA Project Officer (see below).

The complete report, entitled "Treatment of Contaminated Soils with Aqueous Surfactants," (Order No. PB 86-122 561 / AS; Cost: \$11.95, subject to change) will be available only from:

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