Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Oma, Kenton H. , Clarke, Ann N. , Megehee, M. Maria and Wilson, David J.(1993) 'Soil Clean-Up by Surfactant Washing. III. Design and Evaluation of the Integrated Pilot-Scale Surfactant Recycle System', Separation Science and Technology, 28: 15, 2319 — 2349

To link to this Article: DOI: 10.1080/01496399308019741

URL: http://dx.doi.org/10.1080/01496399308019741

SEPARATION SCIENCE AND TECHNOLOGY, 28(15&16), pp. 2319–2349, 1993

Soil Clean-Up by Surfactant Washing. III. Design and Evaluation of the Integrated Pilot-Scale Surfactant Recycle System

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ABSTRACT

A pilot-scale system for recycle and reuse of spent surfactant solution from organic-contaminated soil washing was successfully tested. The surfactant recycle system is comprised of an air-stripping column to remove volatile contaminants, a solvent-extraction column to remove nonvolatile contaminants, and a solvent-recovery system to remove and concentrate nonvolatile contaminants from the solvent. The pilot-scale recycle system was operated in conjunction with a soil test bed which was spiked with biphenyl as a representative "nonvolatile" contaminant. The individual components, which had each been tested previously, were operated together as an integrated system to produce a recycled surfactant solution which was reused during the test. The operation of this system is discussed and the results from the integrated testing are presented. Not only was 99% biphenyl removal from soil achieved in 7.7 pore volumes of 2.5 wt% sodium dodecyl sulfate surfactant solution, but there was also no decrease in the effectiveness of the recycled surfactant solution in removing the biphenyl compared to the virgin solution. Approximately 1 mg/kg biphenyl remained in the soil after surfactant washing.

INTRODUCTION

The pilot-scale surfactant recycle system developed by Eckenfelder Inc. addresses a major problem identified by the United States Environmental

Protection Agency (USEPA) which limits the successful implementation of in-situ surfactant flushing or ex-situ washing (SF/W). This is the separation of surfactant from the soil (especially clay soils) and regeneration of the surfactant for reuse (1).

Soil contaminated with organic chemicals which are considered difficult-to-treat may be treated by SF/W processes where water flushing or washing would be inadequate. The prime example of a difficult-to-treat organic chemical is polychlorinated biphenyls (PCBs). Surfactant-supplemented processes will also remove less-difficult-to-treat organic constituents such as volatile organic compounds (VOCs), polyaromatic hydrocarbons (PAHs), chlorinated and nonchlorinated aliphatic solvents, aromatic solvents, etc.

The effectiveness of SF/W is related to the ability of surfactants to solubilize "water-insoluble" compounds. The surfactant selected by Eckenfelder Inc. was sodium dodecyl sulfate (SDS) which had been used for previous laboratory-scale testing (2) and the present pilot-scale testing. SDS is a 12 carbon, straight chain, anionic surfactant that is commercially available and relatively inexpensive. In addition, SDS is biodegradable and relatively nontoxic (3). The critical micelle concentration (cmc) for SDS is 0.231 wt% or 2.31 g/L. This test employed a 2.5 wt% SDS solution which is greater than 10 times the cmc for SDS.

Initial laboratory-scale data were collected for soil spiked with biphenyl, which is a relatively nonhazardous hydrophobic compound, and toluene, which represents a class of VOCs frequently found at hazardous waste sites. Favorable results were obtained from the initial laboratory-scale testing which was conducted in 1990 under a USEPA Small Business Innovation Research Phase I (SBIR-I) research grant (2). The current pilot-scale research was conducted under a USEPA SBIR-II research grant.

The primary objectives of the pilot-scale research are divided into two phases. Phase I included the design and assembly of the pilot-scale equipment and the testing of the individual components. The results of the testing of individual components are presented in the previous paper in this series (4). The Phase II research presented in this paper involved the testing of the integrated pilot-scale unit on the removal of a "nonvolatile" component (biphenyl) in a 68.9 kg (152 pound) soil test bed and the continued treatment of the soil with the recycled surfactant solution.

Since it had been determined in the earlier Phase I research (4) that the surfactant can remove high levels of biphenyl (i.e., 1000 mg/kg) from soil, it was thought more appropriate in the Phase II research to simulate the "polishing" of the removal of biphenyl from soil. Thus, a lower initial biphenyl concentration of 100 mg/kg was selected to permit the research efforts to evaluate the behavior of the technology during the removal of low level residual concentrations. This scenario could be considered to simulate the end of a remediation.

SYSTEM DESIGN

A pilot-scale SF/W system was designed and fabricated to evaluate the scale-up feasibility of the technology and ultimately to demonstrate the integrated process on larger volumes of soils contaminated with organic compounds, specifically those that are considered difficult to treat. A complete description of the pilot-scale surfactant recycle system is presented in the preceding paper in this series (4).

The pilot-scale system was designed to simulate in-situ surfactant flushing of up to 1 ton of contaminated soil with recovery, regeneration, and reuse of the surfactant. A process schematic of the integrated pilot-scale surfactant recovery and recycle system is shown in Figure 1.

The recovery and recycle process is an integration of several unit operations: 1) an air-stripping column for removal of VOCs from the surfactant, 2) a countercurrent solvent-extraction column for removal of nonvolatile organics, and 3) a solvent-recovery system for removal and concentration of nonvolatile organics from the extractor solvent. A hyperfiltration unit (not shown in Fig. 1) was previously tested as part of the Phase I research (4) for concentrating the surfactant solution since overpumping of surfactant flushing recovery wells is required to insure that the surfactant and mobilized contaminants are completely recovered during in-situ treatment. The process for the recovery and recycle of surfactant employed for ex-situ washing is the same as for in-situ flushing with the exception

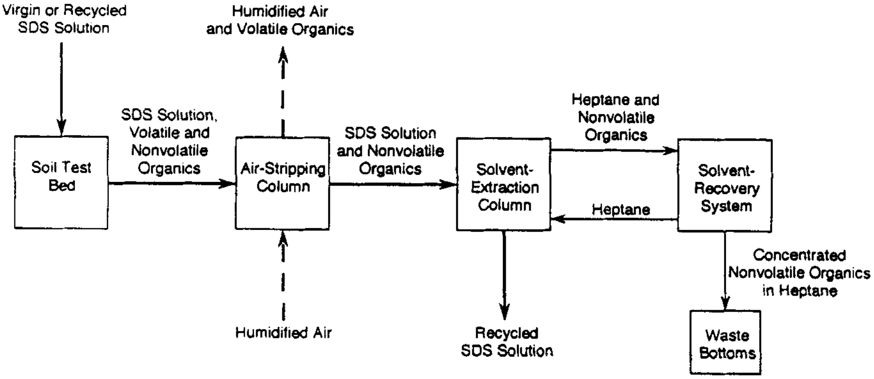

FIG. 1 Integrated pilot-scale surfactant recycle system.

of the elimination of the hyperfiltration unit. The air-stripping column, while not required when only nonvolatile compounds are present, was included in the pilot-scale system testing since it is an integral part of the process. In addition, the effects of air-stripping column operations on SDS solution with a "nonvolatile" (biphenyl) could be evaluated. Biphenyl was used as a surrogate for nonvolatile organic compounds such as PCBs. It is important to note that biphenyl has a higher vapor pressure than do PCBs; therefore, the air-stripping column operations would remove a higher percentage of biphenyl than PCBs.

The Phase II research included some modifications of the pilot-scale surfactant recycle system that was constructed during Phase I followed by integrated testing of the system with recovered surfactant solution containing biphenyl.

The following section describes the preparation of the soil test bed and modifications to the pilot-scale system design and process flow streams. The modifications were made to permit multiple aqueous surfactant processing cycles within the air-stripping column and the solvent extraction column while maintaining a continuous flow of aqueous surfactant through the soil test bed. This multiple cycling capability within a given unit process allows the residual concentrations of biphenyl to be reduced further in the surfactant solution prior to its reuse.

Soil Test Bed Configuration and Preparation

The soil mix used for the pilot-scale integrated test bed was the same mix as previously used during the Phase I testing (4). The soil test bed container was a 114 L galvanized cylindrical container that measured 57.8 cm high by 42.9 cm diameter at its widest point. The container volume was calibrated using water. A bulkhead compression fitting was installed through the side of the container flush with the bottom to allow removal of recovered surfactant solution from below the soil test bed. A single layer of 1.6 cm diameter polypropylene packing was placed across the bottom of the container, and a geomembrane was placed on top of the packing. This configuration allowed the surfactant solution to be collected and removed from within the packing material while the soil was supported above the packing by the geomembrane. Prior to preparation of the test bed soil, the container seams were sealed from the outside with silicone caulking and the container was leak tested with water.

The soil mix was prepared using 41.7 kg of surficial soil from a farm in east central Mississippi and 27.2 kg of sand from a sand mine in Camden, Tennessee, resulting in a total soil mass of 68.9 kg. The soil and sand were screened through a 4.75-mm sieve, weighed, and then mixed together in batches. Soil samples were taken for physical property measurements, and the testing results are summarized in Table 1. The average pore volume of 22.9 L presented in Table 1 was calculated from the physical property results and volume measurements of the soil test bed taken during the Phase II pilot-scale testing.

The biphenyl was next added to the test soil as a single aliquot in a heptane solution. Heptane was the solvent used in the solvent-extraction column and was used as the solvent blank and for preparation of the biphenyl standards for the ultraviolet (UV) spectrophotometric analysis. Therefore, the heptane added to the soil test bed did not interfere with subsequent UV spectrophotometric sample analysis. A 34.9-kg batch of soil mix was prepared by placing it in three equal layers and carefully applying the biphenyl/heptane solution to each layer using a pipet. A total of 3.41 g of biphenyl was added to the soil. Following the organic chemical additional, each soil batch was thoroughly mixed. The soil was then loaded into the soil test bed in layers of about 5 cm and each layer was uniformly compacted. The same procedure was used to prepare an additional 34.0 kg of biphenyl-containing soil. After addition and compaction of the total soil mass to the test bed, a lid was placed on the test bed, and tape was used to seal the lid to minimize any vapor losses from the soil prior to testing.

The soil test bed was stored in a walk-in cooler at 0 to 4°C for about 6 days to allow some "aging" of the biphenyl on the soil prior to pilot-scale testing. Three days prior to testing, the soil test bed was removed from the cooler and allowed to warm to ambient conditions. The pilot-scale soil test bed was positioned inside a larger fiberglass tank as shown in Fig. 2. A container of water and an immersion heater were also placed

TABLE 1
Physical Properties of Test Soil

Parameter	Value
Soil pH	5.9
Organic content	3.5%
Specific gravity	2.568
Percent moisture	11.8%
Mass of soil in test bed	68.9 kg (wet) 60.8 kg (dry)
Porosity (average)	49.1%
Bulk density (average)	1.48 kg/L (wet) 1.31 kg/L (dry)
Pore volume of soil in test bed (average)	22.9 L

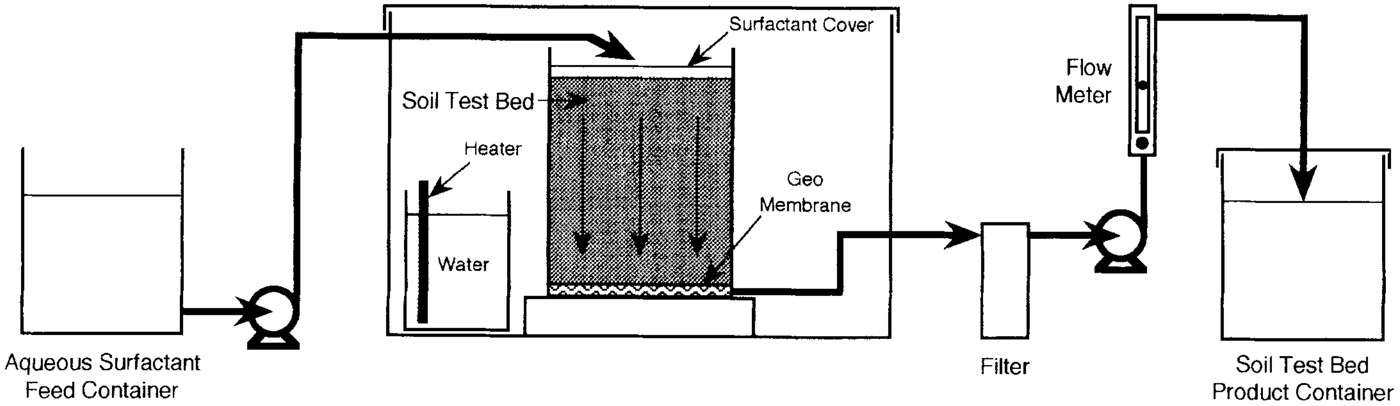

FIG. 2 Pilot-scale soil test bed schematic.

inside the fiberglass tank to provide temperature control to the soil test bed. It was necessary to maintain a temperature of about 20°C or greater to prevent the SDS from coming out of solution. (A 25 g/L SDS solution will "thicken" at temperatures lower than about 20°C. Increasing the temperature restores it to its normal viscosity with no change in its properties.) Peristaltic tube pumps were used, as shown in Fig. 2, to pump aqueous surfactant to the top of the soil test bed and to recover surfactant product from below the soil test bed. The recovered surfactant was passed through a 5-micron filter and a flowmeter and was then collected in a soil test bed product container.

Air-Stripping Column Modifications

Results of the Phase I testing provided information about the general operability and performance of the air-stripping column with SDS solutions containing toluene. During this initial testing, potential improvements to the air-stripping column were identified and were incorporated into the unit prior to the Phase II testing. A description of the air-stripping column as originally designed and fabricated is presented in the previous paper in this series (4). This section describes the modifications made to that original system. Figure 3 shows the air-stripping column as modified

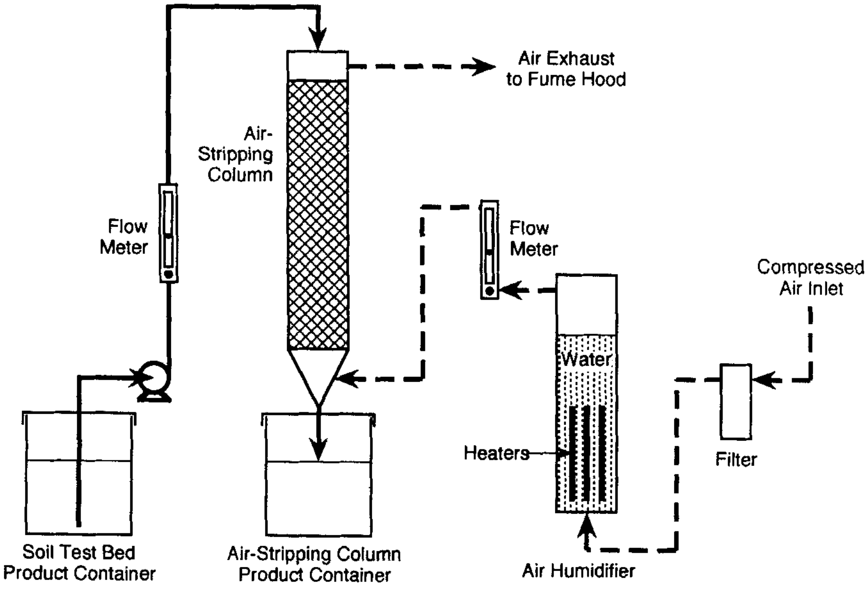

FIG. 3 Pilot-scale air-stripping column schematic.

with the addition of an air humidifier and with smaller liquid containers (replacing previously installed tanks).

The air humidifier was added to help improve the stripping efficiency of the column and to reduce (or eliminate) the buildup of SDS on the packing during air-stripping column operation. The compressed air which was supplied to the column was preconditioned by drying. As a result, the air caused the aqueous surfactant in the column to cool to about 16°C due to evaporation during previous tests with the column. This cooling has detrimental side effects in that 1) the vapor pressures (and effective Henry's law constants) of VOCs are reduced, resulting in reduced stripping efficiency, and 2) the SDS solution starts thickening and forms a buildup of SDS on the column packing.

The air humidifier has an inside diameter of 17.8 cm and an inside height of about 86 cm, and contains 1.6 cm NOR-PAC (a trademark of N.S.W. Corp.) packing to help distribute air as it flows upward through the column. Three immersion heaters of 200 W each are contained within the unit to heat the water (and thus heat the air passing through). This particular air humidifier configuration produces 150 L/min of air at greater than 95% relative humidity at 25 to 30°C.

The aqueous surfactant containers used throughout the Phase II pilot-scale research were 19 L plastic containers. As shown in Fig. 3, the soil test bed product container was used as the feed container for the air-stripping column. To achieve multiple processing cycles through the column, the surfactant solution collected in the air-stripping column product container can be fed back through the column. This was typically done four to six times for each soil test bed volume unit that was processed during the pilot-scale testing.

Solvent-Extraction Column and Solvent-Recovery System Modifications

The performances of the solvent-extraction column and solvent-recovery system were initially tested during the Part I testing using an SDS solution containing biphenyl. Potential improvements to the solvent-extraction column and solvent-recovery system were identified and were incorporated into the process prior to the Phase II testing. A description of the original solvent-extraction column and solvent-recovery system were presented earlier (4). This section describes only the modifications made to the original systems. Figure 4 shows the extraction system as modified for the Phase II testing. The modifications were minor and consisted of the elimination of the distillation unit solvent bypass line and the use of smaller liquid containers (replacing previously installed tanks).

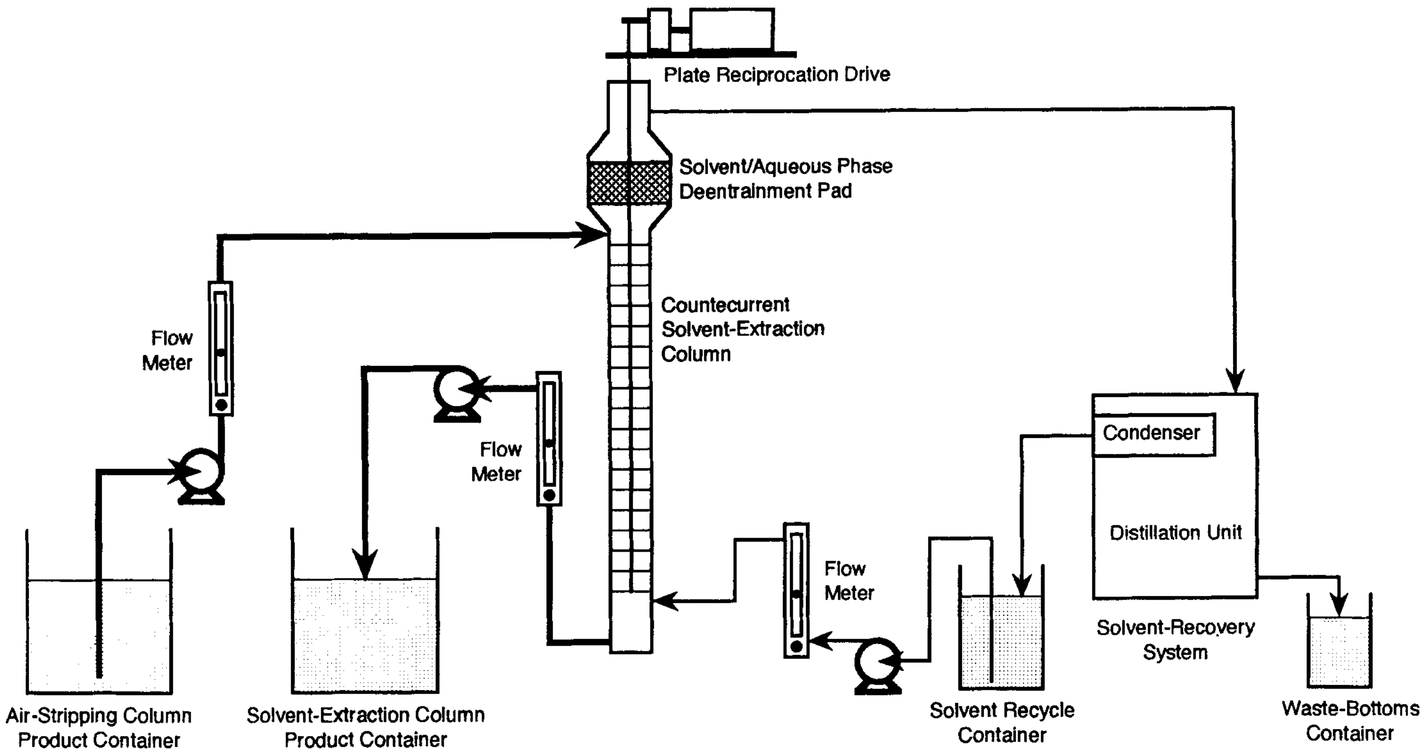

FIG. 4 Pilot-scale solvent-extraction column and solvent-recovery system schematic.

The distillation unit solvent bypass line was eliminated in an effort to reduce the biphenyl concentration in the solvent returning to the solvent-extraction column. It was previously determined that relatively low residual biphenyl concentrations in the recycle solvent limited the overall biphenyl removal efficiency of the solvent-extraction column at low biphenyl concentrations in the aqueous surfactant. This modification allows only distilled solvent to be recycled to the solvent-extraction column and requires that the heptane flow rate to the column be reduced from 100 mL/min to about 50 mL/min so that the distillation unit can process the entire recycled solvent flow.

As shown in Fig. 4, the air-stripping column product container is used as the feed container for the solvent-extraction column. The surfactant solution collected in the solvent-extraction column product container can be fed back through the column to achieve the desired degree of biphenyl removal. The volume units of SDS solution were typically cycled through the solvent-extraction column from two to four times during the Phase II testing. The term "volume unit" refers to a volume of approximately 15 L of surfactant solution which was the initial estimated pore volume of the soil test bed prior to the pilot-scale testing. The actual pore volume of the soil test bed was calculated after the test was completed by using the laboratory-determined percent moisture and specific gravity of the soil and the actual measured volume occupied by the soil in the test bed during testing. The actual pore volume was calculated to be 22.9 L, so the term "volume unit" refers to about 66% of a pore volume.

EXPERIMENTAL WORK

A description of the test operations and a presentation of the results for the pilot-scale process test with biphenyl are presented in this section. Each pilot-scale process component is treated in a separate section with conclusions from the results presented for that component. The final section contains a summary of the results for the overall pilot-scale process test and conclusions from these results.

Soil Test Bed Operations Description and Results

The pilot-scale test was initiated by applying volume unit 1 of 25 g/L virgin SDS solution onto the surface of the soil test bed from the aqueous surfactant feed container. The temperature of volume unit 1 of the SDS solution was adjusted to 27°C prior to its placement on the soil test bed; the test bed temperature was 19°C prior to contact with the warm SDS solution. The warm SDS solution was allowed to flow down into the soil test bed by gravity feed only. After approximately 1.5 hours, the soil test bed appeared to be saturated with SDS solution and had warmed to a temperature of 24°C. A second volume unit of warmed SDS solution was pumped onto the soil test bed to maintain a continuous liquid cover. Four hours after the test began, the pore volume was calculated as 24.7 L and, at the end of the test, the pore volume had been reduced to 21.0 L due to settling of the soil during the course of the test. For all data reduction calculations, an average value of the pore volume of 22.9 L was used (see Table 1).

Once the soil test bed was saturated with aqueous SDS solution, pumping of the test bed product solution from the test bed to the filter was begun. The collection of the first volume unit of the test bed product was begun as soon as the solution exited the filter. All volume units of surfactant product that were recovered from the soil test bed were collected in tared containers and were determined to be complete when their net masses reached 15 kg. Density measurements were taken on all volume units of surfactant solution, and all volume units had densities between 1.0045 and 1.0060 g/cm^3; therefore, 15 kg was equated to 15 L with two significant figures. (It should be noted that if more than two significant figures are maintained, this assumption is no longer true, i.e., 15.0 kg equals 14.9 L.)

The operation of the soil test bed continued throughout the pilot-scale test in much the manner described above. The temperature of the aqueous surfactant feed was adjusted to between 27 and 32°C and pumped onto the test bed. The surface of the soil was continuously covered throughout the test period. The actual volume of solution covering the soil surface varied but was never allowed to drop below about 0.5 L. The water and heater unit inside the fiberglass container (see Fig. 2) maintained the temperature of the soil between 20 and 27°C. The flow rate of surfactant product out of the soil test bed was regulated by the pump and monitored by the flowmeter. Average flow rate data were calculated for each volume unit by weighing the actual product and dividing the weight by the time required to collect that weight. In addition, pH and conductivity measurements were also taken for each volume unit. After each volume unit had been weighed, it was thoroughly mixed and samples were taken for chemical-specific analysis.

Table 2 gives the cumulative pore volume, mass, average flow rate, pH, conductivity, and temperature for each surfactant solution volume unit that was recovered from the soil test bed. Volume units 1 through 6 were composed of SDS solution which was freshly prepared (virgin) before entering the soil test bed. Volume units 7 through 10 were recycled SDS solution which had been processed through the entire pilot-scale

TABLE 2
Data for Volume Units of SDS Solution Removed from Soil Test Bed

Volume unit[a]	Cumulative pore volume	Mass (kg)	Average flow rate (mL/min)	pH[b]	Conductivity[b,c] (μmho/cm)	Temperature[d] (°C)
1	0.66	15.0	44.6	6.10	1700	23
2	1.31	15.0	24.0	6.49	3800	23
3	2.08	17.5	24.6	6.33	3790	22
4	2.63	12.7	22.8	6.31	3590	22
5	3.28	14.8	13.8	6.31	3500	21
6	3.94	15.0	22.8	6.26	3510	23
7	4.59	15.0	11.2	6.14	4180	22
8	5.25	15.0	10.2	6.20	3920	22
9	5.91	15.0	9.9	6.28	3950	22
10	6.56	15.0	9.6	6.06	3920	22
11	7.22	15.1	5.6	6.35	4120	21
12	7.73	11.6	1.1	6.25	2980	21
Average	—	14.7	16.7	6.26	3580	22

[a] Volume units 1 through 6 and 11 were virgin SDS solution going through the soil test bed. Volume units 7 through 10 were recycled SDS solution; volume unit 12 was tap water going through the soil test bed.

[b] Measurements were made of soil test bed product, i.e., after collection of volume unit from test bed was complete.

[c] Also called specific conductance.

[d] Average temperature of soil test bed during collection of each pore volume.

system. Volume unit 1 recovered from the soil test bed was recycled and used as volume unit 7 feed to the test bed; likewise volume units 2, 3, and 4 were recycled and used as volume units 8, 9, and 10, respectively. Volume unit 11 was virgin SDS solution which was used to further "polish" the biphenyl removal from the soil test bed. Volume unit 12 was tap water and was used to begin removing the SDS from the soil test bed. It should be noted that although the volume units are referred to as discrete units, some mixing and overlap between successive volume units, both in the soil test bed and in the filter housing, was unavoidable. The 15 kg weight per volume unit was held to as closely as possible as reflected by the data in Table 2. A total of 7.73 soil test bed pore volumes of aqueous solution had been collected by the end of the test.

Figure 5 shows that the average flow rate plotted against the cumulative number of pore volumes removed. The average flow rate decreased with each successive pore volume except for pore volume 3.94. The last volume unit recovered (volume unit 12), which had tap water as feed, had a very

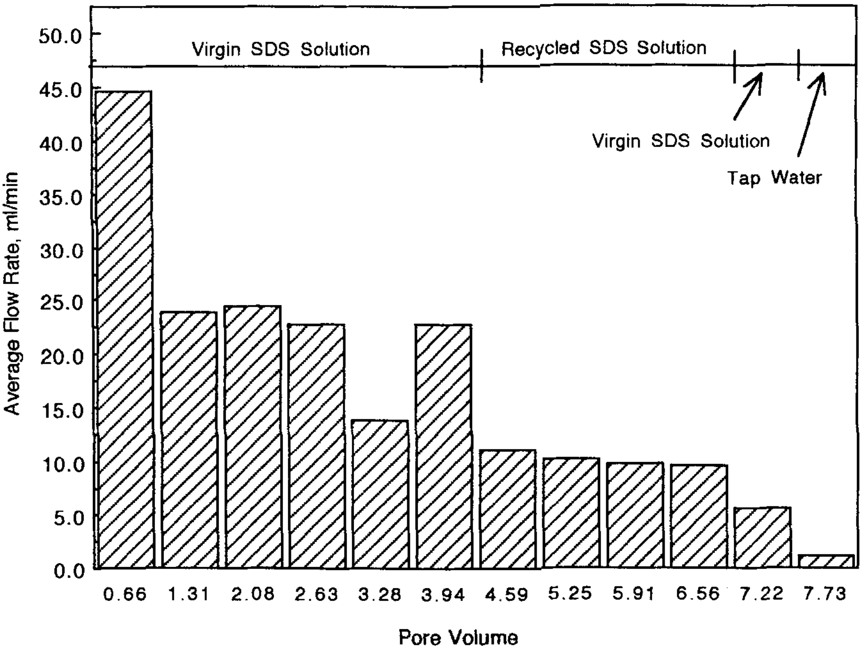

FIG. 5 Average flow rate of aqueous SDS solution removed from soil test bed.

low average flow rate of 1.1 mL/min as compared to the flow rate of 44.6 mL/min for the first volume unit and the flow rates around 23 to 24 mL/min for the second, third, and fourth volume units. The reported flow rate for the last volume unit was the maximum achievable. When the tap water was first added to the soil test bed, a white precipitate formed on the surface of the soil. The tap water gradually decreased the concentration of the SDS solution in the test bed (by dilution) as it moved downward. Previous tests with low concentration SDS solutions have resulted in a white precipitate forming in the soil. This precipitate formation within the soil test bed may be responsible for the low flow rate of the last pore volume.

The data presented in Table 2 show that the pH of the test bed product solution averaged 6.26 and varied by less than half a pH unit. The variability in the conductivity was greater. The conductivity of the first volume unit was relatively low (1700 μmho/cm). After about 1.3 pore volumes, however, the conductivity had risen to 3800 μmho/cm, and it ranged between 3510 and 4180 μmho/cm until the final measurement. The conductivity of the final pore volume (volume unit 12) was lower at 2980 μmho/

cm due to the tap water addition to the soil test bed. As a comparison, the conductivity of the tap water was measured at 210 μmho/cm.

Samples from each volume unit were analyzed for biphenyl concentration. Table 3 presents the results of the biphenyl analysis that was performed by UV spectrophotometry. The UV spectrophotometric analysis for biphenyl in SDS solution was performed by first extracting the biphenyl from the SDS solution into heptane by simple batch extraction. The heptane extract was then analyzed for biphenyl. This extraction and analysis procedure was developed as part of the Phase I testing and is described in an earlier paper (4). Figures 6 and 7 show in graphical form the mass removed results from Table 3. Figure 6 has text across the top which identifies the source for each pore volume removed from the test bed

TABLE 3

Biphenyl Data for Volume Units of SDS Solution Removed from Soil Test Bed

		Biphenyl data			
Volume unit[a]	Cumulative pore volume	Concentration[b] (mg/L)	Mass[c] (mg)	Cumulative mass removed (mg)	Cumulative percent removed[d,e] (%)
1	0.66	41	620	620	9.7
2	1.31	150	2300	2920	45
3	2.08	63	1100	4020	63
4	2.63	40	510	4530	71
5	3.28	33	490	5020	78
6	3.94	17	260	5280	82
7	4.59	15	230	5510	86
8	5.25	12	180	5690	89
9	5.91	15	230	5920	93
10	6.56	11	170	6090	95
11	7.22	7.0	110	6200	97
12	7.73	6.8	79	6279	98

[a] Volume units 1 through 6 and 11 were virgin SDS solution going through the soil test bed. Volume units 7 through 10 were recycled SDS solution; volume unit 12 was tap water going through the soil test bed.

[b] Data from UV spectrophotometric analysis. Method detection limit was 2.0 mg/L.

[c] Calculated by multiplying the concentration of biphenyl in a given pore volume by the mass of the pore volume.

[d] Based on an initial mass of 6340 mg biphenyl in soil test bed which was calculated from GC/FID data which yielded an average concentration of biphenyl in test soil of 92 mg/kg wet weight (104 mg/kg dry weight, based upon 11.8% moisture).

[e] The cumulative percent removed is calculated from the cumulative mass of biphenyl removed prior to rounding to two significant figures; therefore, the numbers in this column are not simply the cumulative mass removed shown divided by the initial mass in the soil test bed.

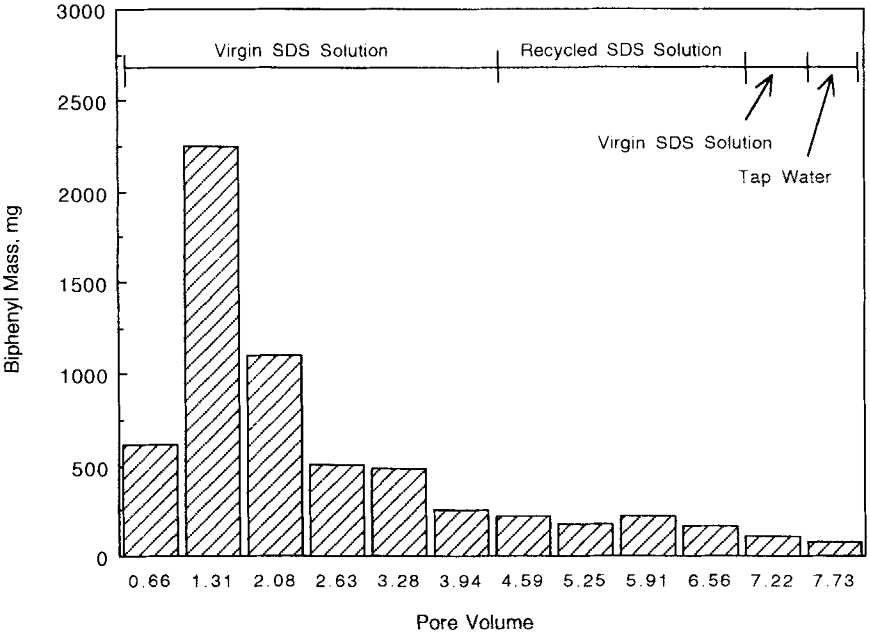

FIG. 6 Mass of biphenyl removed from soil test bed versus pore volume of SDS solution recovered.

(virgin or recycled). The plots of the mass of biphenyl removed shown in Fig. 6 exhibit the same trend that was seen in earlier column studies. Figure 7 shows the mass of biphenyl remaining in the soil test bed as a function of pore volumes removed and shows the corresponding percent biphenyl removal from the soil test bed. Both curves are based upon an initial mass of 6340 mg biphenyl in the soil test bed. This initial mass was calculated from analytical data from gas chromatograph/flame ionization detector (GC/FID) analysis of the soil prior to the beginning of the test. Two composite soil samples were analyzed by GC/FID with a method detection limit of 2.0 mg/kg and resulted in biphenyl concentrations of 89 and 95 mg/kg (wet basis). An average concentration of 92 mg/kg was used along with a test soil mass of 68.9 kg (wet) to calculate the total initial mass of biphenyl in the soil test bed. According to the data presented in Table 3 and Fig. 7, 98% of the biphenyl was removed from the test bed after 7.73 pore volumes had passed through the test bed. Eighty percent of the biphenyl had been removed after approximately 3.5 pore volumes, and 90% of the biphenyl had been removed after 5.5 pore volumes.

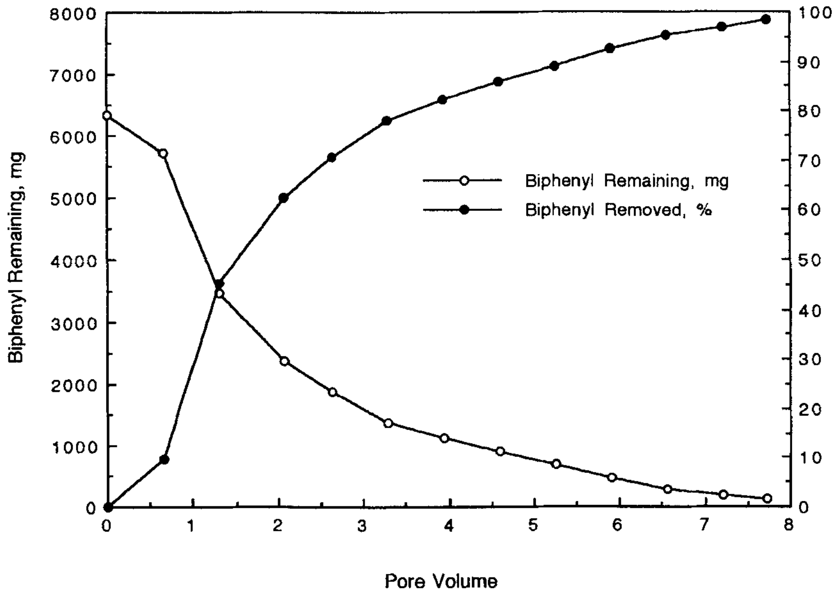

FIG. 7 Biphenyl remaining in soil test bed on a mass and a percent removed basis versus pore volume of SDS solution recovered from soil test bed.

Analysis of two composite soil samples that were taken at the end of the testing was performed by the same GC/FID method as used at the beginning of the testing. A third composite soil sample was analyzed by UV spectrophotometric analysis of a heptane extract of the soil. Both samples analyzed by GC/FID yielded values below the method detection limit (BMDL) which was 2 mg/kg. The estimated concentrations (below the detection limit) were 0.8 and 0.7 mg/kg. The extraction of two separate aliquots of soil with heptane and subsequent analysis of the heptane for biphenyl using the UV spectrophotometer yielded concentrations of 0.9 and 1.0 mg/L, both of which were also below the detection limit of 1 or 2 mg/L, respectively, set by the calibration curve used for the quantitation. If a final biphenyl concentration is conservatively assumed to be 1.0 mg/kg, a final biphenyl mass of 81.8 mg is calculated to be remaining in the soil test bed. This final biphenyl concentration is based on the final test bed volume, assuming the soil is completely saturated with water. Based upon the analysis of the soil before and after testing, 99% of the biphenyl was removed from the test bed. This corresponds well with the 98% removal calculated using the soil test bed product concentration data.

Based on the aqueous solubility of biphenyl (1 to 2 mg/L), it would require 20 to 40 pore volumes of water flushing to achieve the same level of removal. This assumes complete equilibration between the water and pure solid biphenyl, and it is therefore a very conservative lower bound. Again, there was no discernible difference in the contaminant removal between the virgin and recycled SDS solution.

Air-Stripping Column Operations Description and Results

Test bed product volume units 1 through 7 were processed through the pilot-scale air-stripping column. A description of the modifications to the air-stripping column was previously given, and Fig. 3 is a schematic of components used for this testing. The aqueous SDS volume unit feed was pumped to the top of the air-stripping column, trickled down through the packing material inside the column, and was collected in the air-stripping column product container. Both feed and product containers were kept covered as much as possible to reduce evaporation. Before introduction to the air-stripping column, each volume unit of feed was heated to between 23 and 31°C, and an antifoaming solution was added to control foaming of the solution as it moved downward through the column. The same antifoam agent was used that had been used in the previous Phase I testing with the air-stripping column. A solution of 5% GE AF9020 defoamer was prepared and added to the aqueous SDS volume units prior to the first air-stripping column feed cycle so that each volume unit had a concentration of 20 mg/L defoamer. Two exceptions to this defoamer addition and concentration occurred. First, the defoamer was not added to volume unit one until after cycle one through the air-stripping column to give a concentration of 20 mg/L defoamer for cycle two, and second, a total concentration of 60 mg/L defoamer was used for volume unit three. UV spectrophotometric analysis was performed on 25 g/L SDS solutions with 20, 40, and 60 mg/L defoamer to determine if any analytical interferences would be caused by the defoamer. The results from these analyses indicated that the defoamer does not interfere with the UV spectrophotometric analysis for biphenyl.

The air stream is passed through the air humidifier/heater into the bottom of the column, flows upward through the column countercurrent to the SDS solution, and flows out the top of the column to the fume hood. The air inlet temperature was maintained between 26 and 31°C at 95% relative humidity or greater for all air-stripping column cycles. The air flow rate was maintained at 150 L/min for all cycles. Samples of the aqueous air-stripping column product were taken for GC/FID and UV spectrophotometric analysis.

Before air-stripping column testing began, it was determined that each volume unit would be cycled through the column several times in order to evaluate the organic constituent removal. Volume units 1, 3, 4, 5, and 6 were each cycled through the air stripping column four times; volume unit 2 was cycled through five times; and volume unit 7 was cycled through six times. Table 4 gives the average operating conditions of the air-stripping column based on data from all cycles of all seven volume units that were processed.

Samples were collected of the aqueous SDS feed for the first cycle (prior to entering column) of each volume unit and from the final cycle product of each volume unit and were analyzed for biphenyl that may have occurred during the air-stripping column tests. Table 5 presents the biphenyl data from the UV spectrophotometric analysis of the samples. In addition to data on each volume unit, the cumulative masses of biphenyl in the air-stripping column feed and product are given for each volume unit. The cumulative biphenyl removal by the air-stripping column was 47% for all seven volume units.

Mathematical modeling of the air-stripping column has been performed and the results will be presented in the next paper in this series (5). The modeling results indicate that biphenyl removal efficiencies of the order of 50% in the air-stripping column are to be expected.

Solvent-Extraction Column and Solvent-Recovery System Operations Description and Results

The solvent extraction column and solvent recovery system were operated together to remove the biphenyl from volume units 1 through 5 after the volume units had been processed through the air-stripping column. A description of the modifications made to the solvent-extraction system

TABLE 4
Average Operating Conditions for the Air-Stripping Column[a]

Parameter	Value
Cycle completion time	29 min
Aqueous SDS feed mass	14.9 kg
Aqueous SDS feed temperature	26°C
Aqueous SDS product mass	14.9 kg
Aqueous SDS flow rate through column	518 mL/min
Humidified air inlet temperature	29°C
Humidified air inlet flow rate	150 L/min

[a] Based on average of all cycles of all volume units used.

TABLE 5
Biphenyl Data for Volume Units of SDS Solution Treated by the Air-Stripping Column

Volume unit	Number of cycles through column	Biphenyl data				Percent biphenyl removed (%)
		Feed for cycle 1		Product from last cycle		
		Concentration[a] (mg/L)	Mass[b] (mg)	Concentration[a] (mg/L)	Mass[c] (mg)	
1	4	41	610	11	160	74
2	5	150	2200	74	1100	50
3	4	63	1100	41	740	33
4	4	40	500	25	320	36
5	4	33	480	19	280	42
6	4	17	250	12	180	28
7	6	15	220	5.7	85	61
Cumulative[d]			5360		2860	47

[a] From UV spectrophotometric analysis of SDS solution. Data are corroborated by GC/FID QA/QC analyses.

[b] Calculated by multiplying feed biphenyl concentration by feed mass.

[c] Calculated by multiplying product biphenyl concentration by product mass.

[d] For all seven volume units; a biphenyl mass balance.

was previously given, and the modified system, as tested, is shown in Fig. 4. The test was conducted by pumping a given volume unit of aqueous SDS product from the air-stripping column product container into the top of the solvent-extraction column, allowing it to flow downward, and pumping it out the bottom of the column into the solvent-extraction product container. At the same time the extraction solvent, heptane, was pumped from the solvent recycle container to the bottom of the column, moved upward as small dispersed droplets countercurrent to the aqueous phase, passed through the solvent/aqueous phase deentrainment pad, and flowed from the top of the solvent-extraction column to the solvent-recovery system. The solvent-recovery system was operated continuously to provide distilled heptane for solvent feed for the extraction column. The reciprocating plates inside the solvent-extraction column were operated during the test at a rate high enough to significantly reduce the heptane droplet size (and improve the extraction efficiency) without limiting the aqueous SDS flow rate (either by solvent entrainment in the SDS product or by emulsion formation at the deentrainment pad). The SDS solution feed and product for each cycle through the column were sampled for biphenyl analysis. Results from these analyses were used to calculate the mass of biphenyl removed from the aqueous SDS feed which ultimately provided an overall removal efficiency and biphenyl mass balance. The heptane feed to the column (the condensate from the distillation unit) and the heptane product from the column (the feed to the distillation unit) were also sampled periodically during each processing cycle. The results of these heptane analyses for biphenyl by UV spectrophotometry were used as "real time" indicators of the process of the removal of biphenyl from the aqueous SDS solution.

After two or three cycles had been completed with the solvent-extraction column, the volume of heptane in the distillation unit was reduced so that a small volume, which had a high concentration of biphenyl, would remain in the unit. This small waste volume was then removed from the distillation unit, weighed, sampled, and stored as "waste bottoms." At the end of the testing with the solvent-extraction system, the waste bottoms for the entire test were combined, weighed, and sampled for biphenyl analysis.

Several operating conditions or parameters were held constant throughout the test: the operating rate of the reciprocating plates in the solvent-extraction column was 77.4 cycles/min (target was 75 cycles/min); the average flow rate of aqueous SDS solution into and out of the column was 104 mL/min (target was 100 mL/min); and the heptane flow rate into the column was approximately 50 mL/min (except for cycle 1 of volume unit 1 when it was set at approximately 100 mL/min). The heptane flow rate was decreased from 100 to 50 mL/min after cycle 1 of volume unit 1 because it was determined that the distillation unit could not produce heptane condensate at a rate comparable to 100 mL/min. The temperature of the aqueous SDS feed and product ranged between 20 and 25°C.

The test began with the solvent-extraction column containing 4.2 L of virgin 25 g/L SDS solution and with clean heptane flowing as dispersed droplets through the column. Volume unit 1 of aqueous SDS solution from cycle 4 of the air-stripping column was used as the feed for the beginning of the solvent-extraction column test. Therefore, as virgin SDS solution was pumped out of the column, the aqueous SDS feed was pumped onto the column. Some mixing of the virgin SDS solution and volume unit 1 did occur. The aqueous SDS product collected from this cycle was therefore a mixture of virgin SDS solution and recycled volume unit 1 solution. Also, when this product had been collected, approximately 4.2 L of volume unit 1 remained in the solvent-extraction column. Therefore, when the product from cycle one was used as feed for cycle 2, mixing again occurred between the last part of cycle 1 feed and the first part of cycle 2 feed. This mixing sequence occurred throughout the testing for volume units 1 through 5. Each aqueous SDS feed and product was weighed and sampled for biphenyl analysis.

Table 6 lists the physical parameters measured during operation of the pilot-scale solvent extraction column. Volume unit 1 was cycled through the column twice; volume unit 2 was cycled through the column four times; and volume units 3, 4, and 5 were cycled through the column three times each. The times required for each cycle as well as the masses of SDS solutions used as the feed and product for each cycle are given in Table 6 along with the averages. The product flow rate averaged 104 mL/min (slightly higher than the target flow rate of 100 mL/min) and was fairly consistent for all five volume units.

Table 7 presents the results of the biphenyl analyses of the aqueous SDS samples taken during the solvent-extraction test. All data given in this table are from UV spectrophotometric analysis of the samples. The

TABLE 6
Physical Parameters for SDS Solutions Measured During Operation of the Solvent-Extraction Column

Volume unit	Cycle	Elapsed time[a] (min)	Feed mass of SDS solution (kg)	Product mass of SDS solution (kg)	Product flow rate[b] (mL/min)
1	1	140	14.6	14.8	106
	2	133	14.8	14.3	108
2	1	140	14.4	14.4	103
	2	NA[c]	14.4[d]	14.4[d]	NA[c]
	3	135	14.4	14.2	105
	4	138	14.2	14.2	103
3	1	172	17.9	17.8	103
	2	169	17.8	17.7[d]	105
	3	179	17.7[d]	17.6	98
4	1	120	12.6	12.6	105
	2	124	12.6	12.4	100
	3	122	12.4	12.7	104
5	1	135	14.9	14.4	107
	2	141	14.4	14.5	103
	3	139	14.5[d]	14.6	105
Average		142	14.8	14.7	104

[a] Calculated based on the start and stop times for aqueous product collection.

[b] Calculated by dividing the product mass by the elapsed time with the assumption that 1.0 kg equals 1.0 L.

[c] Data not available.

[d] Data assumed as average of previous and/or next cycle mass.

TABLE 7
Biphenyl Data from Analysis of SDS Solution Samples Taken during Operation of the Solvent-Extraction Column and Solvent-Recovery System

Volume unit	Cycle	Biphenyl data: Feed concentration (mg/L)	Feed mass[a] (mg)	Product concentration (mg/L)	Product mass[b] (mg)	Percent removed per cycle (%)	Percent removed per volume unit (%)
1	1	11	160	2.0	30	81	95
	2	2.0	30	BMDL (0.6)[c]	8.6	71	
2	1	74	1100	20	290	74	98
	2	20	290	9.0	130	55	
	3	9.0	130	2.6	37	72	
	4	2.6	37	BMDL (1.6)[c]	23	38	
3	1	41	740	8.5	150	80	91
	2	8.5	150	4.3	76	49	
	3	4.3	76	3.8	67	12	
4	1	25	320	6.8	86	73	94
	2	6.8	86	3.2	40	53	
	3	3.2	40	BMDL (1.6)[c]	20	50	
5	1	19	280	5.8	84	70	86
	2	5.8	83	4.0	58	30	
	3	4.0	58	2.6	38	34	

[a] Calculated by multiplying feed concentration by feed mass, assuming 1.0 kg equals 1.0 L for SDS solutions.

[b] Calculated by multiplying product concentration mass, assuming 1.0 kg equals 1.0 L for SDS solutions.

[c] BMDL = below method detection limit. Concentration was below method detection limit of 2.0 mg/L; estimated value is listed in parentheses.

concentration of biphenyl in the initial feed cycles for each volume unit ranged from 11 to 74 mg/L. The concentration of biphenyl in the product from the final cycles of each volume unit ranged from BMDL (estimated at 0.6 mg/L) to 3.8 mg/L and averaged 2 mg/L. The mass of biphenyl in the feed and product was calculated and was used for calculation of the percent biphenyl removed per cycle and per volume unit. In general, as the biphenyl concentration decreased in the feed, so did the removal efficiency (of percent biphenyl removed). However, the overall percent removals for all cycles of each volume unit were high, ranging from 86 to 98%. The lowest overall removal efficiency was for volume unit 5 which had the lowest initial biphenyl concentration in the cycle 1 feed. The highest overall removal efficiency was for volume unit 2 which had the highest biphenyl concentration for the cycle 1 feed. Figure 8 shows, for

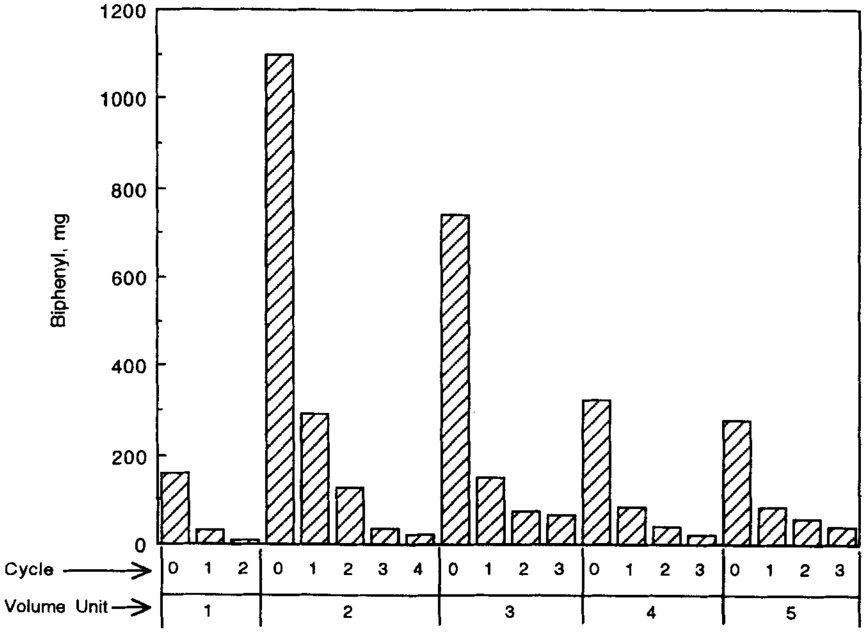

FIG. 8 Mass of biphenyl remaining in each SDS solution volume unit after each solvent extraction column cycle. (Cycle 0 shows the mass of biphenyl in the initial feed.)

each volume unit, the mass of biphenyl present in the feed for cycle 1 (listed as cycle 0 on the chart) and remaining in the product after each cycle (cycles 1, 2, etc.) through the solvent-extraction column.

The results of the biphenyl analysis of the heptane samples taken during the testing of the solvent-extraction column are shown in Table 8. Several samples of heptane feed and heptane product of the solvent-extraction column were taken during each cycle; however, the data presented in Table 8 are limited to those representative of the steady-state conditions that were established for each cycle. The concentration of biphenyl in the heptane feed to the column was below 1 mg/L for all samples analyzed except one for cycle 2 of volume unit 2 when the biphenyl was 1.8 mg/L. The biphenyl concentrations shown in Table 8 for the heptane product of the solvent-extraction column are from samples taken at the end or close to the end of each cycle. Comparison of the cycles for each volume unit show that biphenyl concentration decreased with each successive cycle. The concentrations of biphenyl in the heptane product also reflect the concentration of biphenyl in the aqueous SDS feed. For example,

TABLE 8
Biphenyl Data from Analysis of Heptane Samples Taken During Operation of the Solvent-Extraction Column and Solvent-Recovery System

Volume unit	Cycle	Biphenyl concentration: Heptane feed[a] (mg/L)	Biphenyl concentration: Heptane product[b] (mg/L)
1	1	BMDL[c]	11
	2	BMDL	1.8
2	1	BMDL	67
	2	1.8	13
	3	BMDL	6.2
	4	BMDL	2.9
3	1	BMDL	8.5
	2	BMDL	6.4
	3	BMDL	2.3
4	1	BMDL	10
	2	BMDL	4.8
	3	BMDL	2.5
5	1	BMDL	9.0
	2	BMDL	5.1
	3	BMDL	1.7

[a] Heptane from solvent-recovery system pumped into solvent-extraction column.
[b] Heptane at solvent outlet from solvent-extraction column.
[c] Below method detection limit, 1 mg/L.

volume unit 2 had the highest initial biphenyl concentration in the aqueous SDS cycle 1 feed as well as the highest biphenyl concentration in the heptane product for cycle 1.

Equilibrium distribution data for biphenyl partitioning between heptane and a 2.5% SDS solution were collected and the results are plotted in Fig. 9. The slope of the line, m, represents the biphenyl distribution coefficient which is 56.4. At lower biphenyl concentrations, the distribution coefficient is somewhat lower, 36.8, as shown in Fig. 10. The extraction factor is calculated to be from 12 to 18, which is quite high and should yield a biphenyl removal efficiency of over 90% for each theoretical stage. The actual removal efficiency per cycle through the solvent-extraction column ranged from 12 to 81% (refer to Table 7), indicating that the solvent-extraction column has less than one theoretical stage. One possible explanation for the lower than optimal removal efficiency is that the rate of mass transfer of biphenyl from the surfactact micelle to the heptane is limited by the biphenyl diffusion kinetics. Additional testing would be required to fully evaluate this possibility.

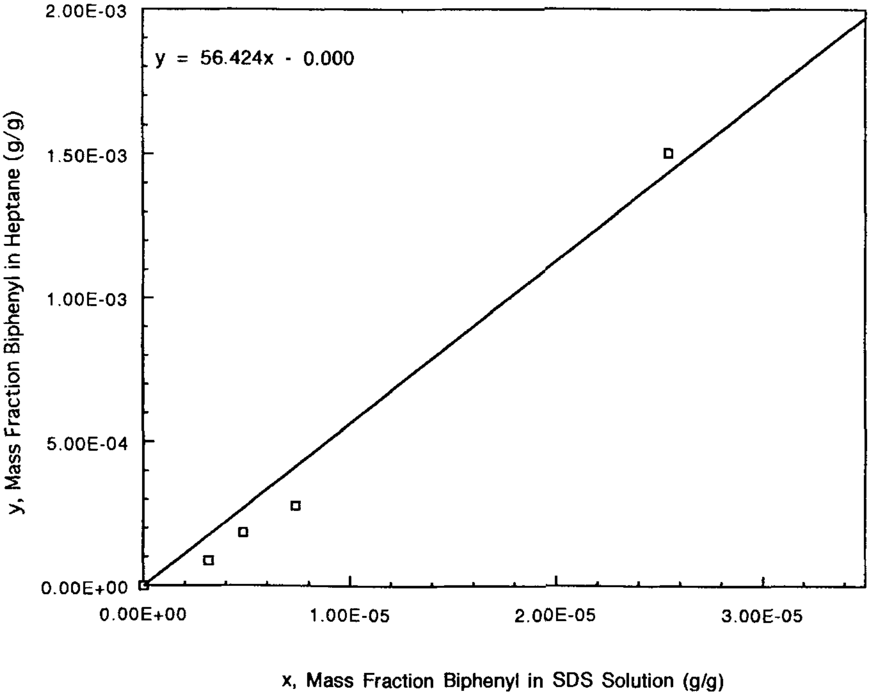

FIG. 9 Equilibrium data for biphenyl partitioning between heptane and a 2.5 wt% SDS solution.

Table 9 contains the data for the waste bottoms collected from the solvent-distillation unit during the course of the pilot-scale testing. The footnotes for the table explain when the waste bottom samples were collected. A total of 2340 mg biphenyl was collected and concentrated in the distillation unit. The composite sample was made up of the combined discrete waste bottoms after sample aliquots had been removed from each for analysis. As a result, the total mass of biphenyl calculated for the composite is less than that for the total of the individual waste bottoms samples. However, if the concentration (1100 mg/L) of the composite sample is multiplied by the total volume (2.194 L) of the waste bottoms, a mass of 2400 mg biphenyl can be reported. The total volume of 2.194 L was calculated by adding the volume of each waste bottom unit before samples were taken; the volume listed in Table 9 for the composite of all the waste bottom units was measured after all samples had been taken and is therefore less than 2.194 L. This mass (2400 mg) corresponds well

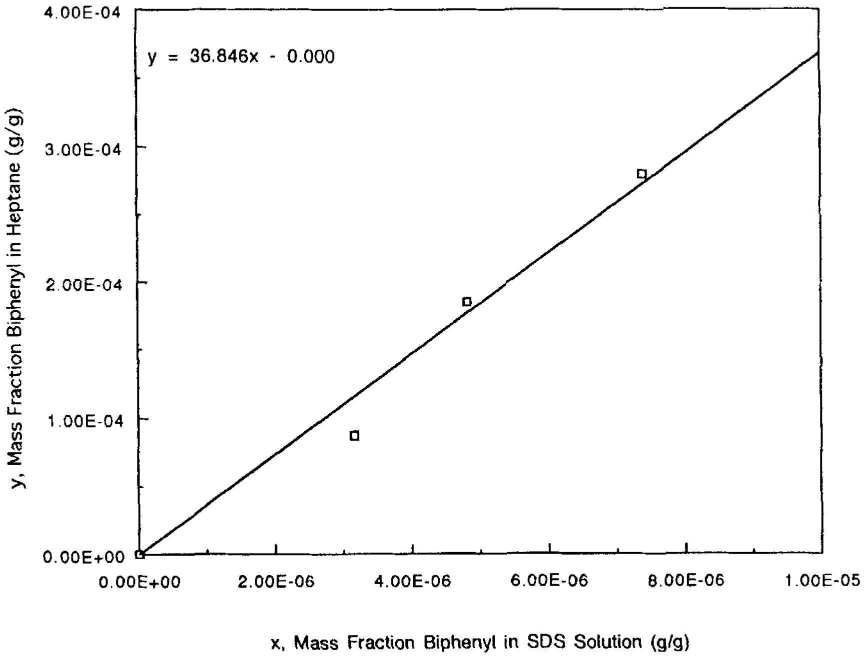

FIG. 10 Equilibrium data for biphenyl partitioning between heptane and a 2.5 wt% SDS solution at lower concentrations of biphenyl.

with the total mass of biphenyl accumulated in the waste bottoms (2340). There was approximately a 90% reduction in the volume of biphenyl-contaminated material when comparing the original soil volume which contained 2340 mg biphenyl with the volume of the final waste bottoms from the solvent-recovery system.

Mass Balance for Biphenyl

Table 10 gives the mass balance for biphenyl within the solvent-extraction column and solvent-recovery system. The total mass of biphenyl into the solvent-extraction column during the testing was 2600 mg, and the total mass of biphenyl out of the column was 2500 mg (or 2560 mg if the waste bottoms composite concentration is used). This accounts for 96 to 98% of the biphenyl in the system.

Table 11 presents detailed mass balance information for biphenyl for the entire Phase II pilot-scale test. Table 12 is a summary of the mass balance data for biphenyl as presented in Table 11. A total of 99% of the

TABLE 9
Data for Waste Bottoms Collected from the Solvent-Recovery System

			Biphenyl data	
Waste bottoms unit	Mass (g)	Volume (mL)	Concentration (mg/L)	Mass (mg)
1[a]	253	370	280	100
2[b]	141	206	3200	660
3[c]	252	368	2100	770
4[d]	237	346	1100	380
5[e]	491	718	480	340
6[f]	237	186	480	89
Total	1501	2194		2340
Composite[g]	1147	1677	1100	1800

[a] Collected after cycles 1 and 2 of volume unit 1 had gone through solvent-extraction column.

[b] Collected after cycles 1 and 2 of volume unit 2 had gone through solvent-extraction column.

[c] Collected after cycles 3 and 4 of volume unit 2 and cycle 1 of volume unit 3 had gone through solvent-extraction column.

[d] Collected after cycles 2 and 3 of volume unit 3 and cycle 1 of volume unit 4 had gone through solvent-extraction column.

[e] Collected after cycles 2 and 3 of volume unit 4 and cycle 1 of volume unit 5 had gone through solvent-extraction column.

[f] Collected after cycles 2 and 3 of volume unit 5 had gone through solvent-extraction column.

[g] Made up of combined waste bottoms units 1 through 6.

biphenyl was removed from the soil test bed based on GC/FID data from analyses of soil for initial and final biphenyl concentrations. The air-stripping column had an overall removal efficiency of 47% for biphenyl for volume units 1 through 7. The solvent-extraction column removed 94% of the remaining biphenyl from the SDS solution of volume units 1 through 5. Of this 94% biphenyl removed, 96% was recovered in the distillation unit waste bottoms. Overall, 96% of the biphenyl initially present in the soil test bed could be accounted for at the end of the test using these data. A 98% mass balance is obtained if only the surfactant solution exiting soil test bed (less the residual in the recycled solution) is compared to the initial amount of biphenyl added, i.e.,

$$\frac{6280 + 82 - 119}{6340} \times 100 = 98\%$$

TABLE 10
Mass Balance for Biphenyl in Solvent-Extraction Column and Solvent-Recovery System

		Biphenyl out:	
Volume unit	Biphenyl in: SDS solution feed to solvent-extraction column (mg)	SDS solution product from solvent-extraction column (mg)	Heptane waste bottoms from solvent-recovery system (mg)
1	160	8.6	
2	1100	23	
3	740	67	
4	320	20	
5	280	38	
Total	2600	157	2340 (2400)[a]

Mass balance:
"Biphenyl in" should equal "biphenyl out"
Biphenyl in = 2600 mg
Biphenyl out = 157 mg + 2340 mg = 2497 mg
(or = 160 + 2300 = 2460 mg)

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[a] Calculated by multiplying biphenyl concentration in composite waste bottoms by the total volume of waste bottoms collected.

Analytical Quality Control Results

Throughout the pilot-scale test, samples of the aqueous SDS solution and the heptane solvent were collected for quality assurance/quality control (QA/QC) analysis. UV spectrophotometry was used as the primary analytical tool for biphenyl analysis of both the aqueous SDS solution and the heptane solvent. Duplicate samples were analyzed for biphenyl by both UV spectrophotometry and GC/FID. The GC/FID data were used as a check for the UV data and were generated by a commercial laboratory. A number of samples were analyzed twice by UV spectrophotometry as an additional check. The QA/QC sample results for both GC/FID and UV spectrophotometry analysis show that the UV spectrophotometer produces valid results which support the overall test results and conclusions.

A total of 11 duplicate samples of aqueous SDS were extracted and analyzed for biphenyl by both GC/FID and UV spectrophotometry. The average standard deviation between the two methods was 9.2% (only results greater than the detection limit were included in the standard devia-

TABLE 11
Detailed Mass Balance for Biphenyl for Entire Phase II Pilot-Scale Test

	Mass of biphenyl in SDS solution							
	Soil test bed		Air-stripping column			Solvent-extraction column		Mass of biphenyl in heptane waste bottoms (mg)
Volume unit	Feed[a] (mg)	Product[a] (mg)	Feed[a] (mg)	Product[a] (mg)	Air exhaust (mg)	Feed[a] (mg)	Product[a] (mg)	
1	0	620	610	160	450	160	8.6	100
2	0	2300	2200	1100	1100	1100	23	660
3	0	1100	1100	740	360	740	67	770
4	0	510	500	320	180	320	20	380
5	0	490	480	280	200	280	38	429
6	0	260	250	180	70			
7	8.6[b]	230	219	85	134			
8	23[b]	180						
9	67[b]	230						
10	20[b]	170						
11	0	110						
12	0	80						
Total	119	6280	5360	2860	2490	2600	157	2340

[a] SDS solution.

[b] The SDS solution used as feed for volume units 7 through 10 were from recycled volume units 1 through 4, respectively, and are shown in solvent-extraction column product.

TABLE 12
Summary of Mass Balance for Biphenyl for Entire Phase II Pilot-Scale Test

Parameter	Value
Soil test bed:[a]	
Initial mass in soil	6340 mg
Final mass in soil	82 mg
Percent mass removed	99%
Air-stripping column:[b]	
Total mass in SDS solution feed	5360 mg
Total mass in SDS solution product	2860 mg
Percent mass removed overall	47%
Solvent-extraction column and solvent-recovery system:[c]	
Total mass in SDS solution feed	2600 mg
Total mass in SDS solution product	157 mg
Percent mass removed from SDS solution	94%
Total mass in heptane waste bottoms	2340 mg
Percent mass removed in heptane waste bottoms	96%

[a] Masses are calculated from data from GC/FID analyses performed on soil samples taken before start of test and at end of test.

[b] Masses are calculated from UV spectrophotometric analyses of SDS solutions for volume units 1 through 7.

[c] Masses are calculated from UV spectrophotometric analyses of SDS solutions and heptane for volume units 1 through 5.

tion calculation). Six replicate samples of aqueous SDS were extracted and analyzed for biphenyl by the UV spectrophotometer, and the average standard deviation between analyses was 8.4%. Nine replicate samples of heptane solvent were analyzed for biphenyl by UV spectrophotometry, and the average standard deviation between analyses was 4.9%. All data are from UV spectrophotometric analysis of samples. The QA/QC data show that there is excellent agreement between duplicate analyses by UV spectrophotometry and GC/FID as well as replicate analyses performed by UV spectrophotometry.

SUMMARY AND CONCLUSIONS

In summary, the Phase II pilot-scale testing for the removal of biphenyl from the soil test bed and subsequent recovery in SDS solution and heptane waste bottoms was successful. The recycling of the SDS solution by processing it through the air-stripping column and solvent-extraction column was also successful. Table 13 lists the primary results from this research. The major conclusions are as follows:

Soil with an initial biphenyl concentration of 92 mg/kg was effectively cleaned to approximately 1 mg/kg using 7.7 pore volumes of 2.5% SDS solution. A total of 99% of the biphenyl (mass basis) was removed from the soil. An estimated 20 to 40 pore volumes of water would be required to reach the same degree of biphenyl removal. This assumes complete equilibration between the water and pure solid biphenyl and is therefore a very conservative lower bound.

The performance of the recycled SDS solution was consistent with that of the virgin SDS solution in removing biphenyl from soil.

TABLE 13
Summary of Pilot-Scale Recycle Testing Results

Parameter	Value
Biphenyl removal from soil bed	99%
Pore volumes of SDS solution to effect removal	7.7
Removal of biphenyl from SDS solution:	
Air-stripping column	47%
Solvent-extraction column	94%
Removal of biphenyl from heptane by solvent-recovery system	96%
Residual biphenyl in SDS solution after treatment	2 mg/L (av)
Residual biphenyl in heptane after treatment	<1 mg/L
Volume reduction (in volume of biphenyl to be treated/disposed of)	~90%

The air-stripping column, while not intended for "nonvolatile" organic removal, effectively removed 47% of the biphenyl from the aqueous SDS that was processed through the column.

The solvent-extraction column was able to remove 94% of the biphenyl from the SDS solution by using two to four cycles. The residual biphenyl concentration in the recycled SDS solution averaged approximately 2 mg/L.

The recycled heptane was as effective as the virgin material in transferring the biphenyl from the SDS solution.

The volume reduction achieved by this process for the waste which would need to be treated/disposed of was approximately 90%. This volume reduction could be increased with system optimization.

REFERENCES

1. U.S. Environmental Protection Agency (USEPA), *Engineering Bulletin, Soil Washing Treatment*, EPA/540/2-90/017.
2. A. N. Clarke, P. E. Plumb, T. K. Subramanyan, and D. J. Wilson, "Soil Clean-Up by Surfactant Washing. I. Laboratory Results and Mathematical Modeling," *Sep. Sci. Technol.*, *26*(3), 301–343 (1991).
3. G. Tchobanoglous and E. D. Schroeder, *Water Quality Characteristics Modeling Modification*, Addison-Wesley, Reading, Massachusetts, 1985, p. 99.
4. A. N. Clarke, K. H. Oma, M. M. Megehee, and D. J. Wilson, "Soil Clean-Up by Surfactant Washing. II. Design and Evaluation of the Components of the Pilot-Scale Surfactant Recycle System," *Sep. Sci. Technol.*, *28*(13&14), 2103–2135 (1993).
5. M. M. Megehee, A. N. Clarke, K. H. Oma, and D. J. Wilson, "Soil Clean-Up by Surfactant Washing. IV. Modification and Testing of Mathematical Models," *Ibid.*, *28*(17&18), 2507–2527 (1993).

Received by editor March 2, 1993