Washing studies for PCP and creosote-contaminated soil

Richard J. Tobia[a], JoAnn M. Camacho[b], Patrick Augustin[c],
Richard A. Griffiths[c],*, Raymond M. Frederick[c]

[a] *Roy F. Weston, Inc., Building 209 Annex, 2890 Woodbridge Avenue,
Edison, NJ 08837, USA*
[b] *Environmental Response Team, US Environmental Protection Agency, 2890 Woodbridge Avenue,
Edison, NJ 08837, USA*
[c] *Risk Reduction Engineering Laboratory, US Environmental Protection Agency, 2890 Woodbridge Avenue,
Edison, NJ 08837, USA*

Abstract

The Environmental Protection Agency has conducted a series of bench-scale and pilot-scale studies to evaluate the feasibility of washing pentachlorophenol (PCP) and creosote from the soil at an abandoned wood-treatment Superfund site in Pensacola, FL. The high sand content and low silt/fines content of the soil made soil washing a promising alternative to incineration. The bench-scale tests confirmed the feasibility of washing the PCP and the carcinogenic creosote compounds from the soil using a nonionic surfactant at a pH of nine to ten and a water temperature of approximately 120 °F. The target concentrations for total creosote were not achieved, but the results were sufficiently close to warrant further testing. The pilot-scale tests using the EPA's mobile Volume Reduction Unit produced residual PCP, carcinogenic creosote, and total creosote levels below the target levels. The tests also produced comparison data on the effects of surfactant concentration, pH, temperature, and liquid:solid ratio.

1. Introduction

This paper presents the results of bench- and pilot-scale tests of the feasibility of washing the contaminated soil at a former wood-treating site in Pensacola, FL. The site is one of several formerly operated by the Escambia Treating Company. Pentachlorophenol (PCP) and creosote are the primary contaminants.

The US Environmental Protection Agency (EPA) began a time-critical removal in 1991 to prevent further migration of the contaminants, which had already reached the

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* Corresponding author. Fax: (+1-908) 321-6640.

groundwater in several places. By July 1992, over 180 000 cubic yards of soil had been excavated and stockpiled on plastic tarps. The excavation was planned to continue to 250 000 cubic yards.

The EPA's Environmental Response Team (ERT) assisted EPA Region 4 with the removal action. Incineration of PCP and creosote-contaminated soil is an accepted cleanup method that was considered. However, among other cleanup technologies proposed, ERT personnel decided to evaluate the effectiveness of soil washing for the Escambia site.

Since the soil is very sandy, with less than 5% silt- and clay-sized particles, ERT personnel expected soil washing to be effective in removing the comparatively-soluble PCP. The oily/gummy creosotes were expected to be more difficult to remove.

Bench-scale tests conducted in December 1991 and January 1992 by Roy F. Weston personnel under the Response Engineering and Analytical Contract (REAC) showed that PCP and carcinogenic creosote compounds could be reduced to target levels. Total creosotes could not be reduced to target levels, as expected. However, the results showed sufficient promise that the ERT decided to proceed with pilot-scale tests to obtain better performance and cost information.

During this same period, the EPA's Releases Control Branch (RCB) completed development of the Volume Reduction Unit (VRU), a mobile pilot plant that has subsystems for soil washing, soil classification by particle size, wash-water preparation and treatment for recycling, and fugitive air-emission control.

The VRU is described in more detail by Masters et al. [1] and in the section of this paper on pilot-scale testing.

The VRU was used to conduct pilot-scale tests at the Escambia site in July 1992 by a team of ERT, RCB, R.F. Weston, and Foster Wheeler Enviresponse personnel. These tests produced residual PCP, carcinogenic creosote, and total creosote levels below the target levels using a variety of process conditions. The tests also produced comparison data on the effects of surfactant concentration, pH, temperature, and liquid:solid ratio.

It is commonly believed that a treatment that does not work on the laboratory bench cannot work in the field. This is not necessarily true for the treatability tests commonly used to evaluate soil washing. A bench-scale process generally cannot properly simulate a full-scale process, and often no attempt is made to do so. Bench-scale tests (including those reported here) are normally batch-type tests. Each stage of a batch-type test progresses toward an equilibrium partitioning of the contaminants among the various soil components and the wash water. Between stages, the slurry of wash water and soil fines (presumably highly contaminated) must be separated from the coarser soil. Mere decanting will not effect a good separation, nor will sieving unless additional water is used, constituting an additional step in the process. In continuous-flow extraction and separation, the soil and contaminants are always progressing toward an equilibrium distribution with clean wash water, and the separation of the fines from the coarse tends to be more thorough. In the tests reported here, the pilot-scale process removed the contaminants better in one pass than the bench-scale process did in three sequential washings.

2. Bench-scale testing

REAC personnel conducted a literature search to identify what surfactants had been previously used for washing creosote-contaminated soils. Many of the surfactants described in the literature were no longer available, so other current products had to be substituted.

In a previous soil-washing treatability study conducted by Weston REAC on creosote-contaminated soils from the GCL Tie and Treating Site [2], seven surfactants, a caustic washing solution, and water alone were evaluated. The results of this study were used to select three biodegradable surfactants for the Escambia soil-washing study: Triton X-100 and Tergitol NP-10, two nonionic surfactants manufactured by Union Carbide, and Dowfax 8390, an anionic surfactant manufactured by Dow Chemical Company.

Two separate sets of bench-scale tests were performed. The objectives of the first set were to evaluate the potential effectiveness of the surfactant solutions to solubilize and remove creosote compounds and PCP from the Escambia site soil. The effects of temperature, pH, surfactant type and concentration, and time were to be studied. The required effectiveness of the washing was based on EPA cleanup criteria of 30 parts per million (ppm) in soil for PCP, 50 ppm for carcinogenic creosote compounds, and 100 ppm for total creosote compounds. The specific compounds are listed in Table 4.

The objectives of the second set were to confirm the results of the first set and to evaluate the effectiveness of three sequential washes. In addition, because of the poor performance of the Dowfax 8390 in the first set, Tween 80, another nonionic surfactant manufactured by ICI America, was substituted in the second set.

Two 5-gallon samples were taken of the surface and near-surface soil. These soils also contained substantial quantities of small wood chips. Each sample was sieved to remove rocks and debris larger than 0.25 in. The two samples were then mixed to provide one composite source.

Samples of the composite were wet-sieved with deionized water to determine the particle size distribution. The results are shown in Table 1. The samples contained less

Table 1
Bench-scale particle-size distribution

Sieve No.	Particle size (mm)	Weight (%)
5–3	4–6	0.6
10–5	2–4	3.2
18–10	1–2	17
35–18	0.5–1	43
60–35	0.25–0.5	31
120–60	0.125–0.25	5
pan	< 0.125	0.6

Table 2
Distribution of contaminants by particle size before washing

Particle size (mm)	Weight (%)	PCP (mg/kg)	Creosote (mg/kg)
1–6	20	2200	15400
0.5–1	43	320	2230
0.25–0.5	31	310	2060
< 0.25	6	610	3930

Table 3
Distribution of contaminants by particle size after washing with Triton X-100

Particle size (mm)	Weight (%)	PCP (mg/kg)	Creosote (mg/kg)
1–6	20	2400	16800
0.5–1	43	90	890
0.125–0.5	37	120	900

than 1% of their weight in particles less than 0.125 mm diameter. The larger-size fractions also contained an undetermined amount of small wood chips, as noted earlier.

Table 2 shows the gross contaminant levels in four particle size ranges of the wet-sieved soil samples. Contamination decreased with decreasing particle size. This is in contrast to the findings at other sites, where contamination increased with decreasing particle size. The unexpected results may be due to the wood chips that were sieved out with the larger-size soil particles. A similar result due to wood and other coarse organics in samples was found by Scholz and Milanowski [3].

These wood chips may have absorbed large amounts of the contaminants and would be very difficult to wash. This has not been conclusively verified, but Table 3 presents data that support this hypothesis. A single-stage wash with Triton X-100 reduced the contamination on the sizes smaller than 1 mm but did not affect the larger sizes.

Contaminant levels in all four particle size ranges were above the cleanup criteria set for the site (30-ppm PCP, 50-ppm carcinogenic creosotes, 100-ppm total creosote). Dioxin/furan analyses were also performed, and toxicity equivalency factors (TEFs, 1987) were calculated to be 0.3 ppb for dioxins and 5.2 ppb for furans.

Table 4 shows the results of a scan for PCP and creosote compounds on the untreated composite soil samples. As indicated in Table 4, not all of these compounds were included in later evaluations. Naphthalene, for example, could not be tracked reliably because of its volatility, and the larger ring compound concentrations were below method detection limits.

Table 4
Raw waste analyses for the bench-scale tests

Compound	December (mg/kg)	January (mg/kg)
Naphthalene[a]	100	31
Acenaphthylene[a]	14	14
Acenaphthene	490	450
Fluorene	520	470
Phenanthrene	1400	1030
Anthracene	610	580
Carbazole	140	180
Fluoranthene	680	740
Pyrene	460	460
Benzo(a)anthracene[b]	120	100
Chrysene[b]	130	130
Benzo(b)fluoranthene[a,b]	32	39
Benzo(k)fluoranthene[a,b]	nd	nd
Benzo(a)pyrene[a,b]	22	20
Indeno(1,2,3-cd)pyrene[a]	nd	nd
Dibenzo(a,h)anthracene[a]	nd	nd
Benzo(g,h,i)perylene[a]	nd	nd
Total creosote	4700	4200
Total carcinogens	300	280
Pentachlorophenol	690	420
TPH	5900	
Percent solids	92	

[a] These compounds were not included in later evaluations during the soil washing experiments because of their low concentrations or problems with their volatility.
[b] Carcinogenic creosote compounds.
nd = compound below method detection limit.

Note: The tables in this paper show some concentrations below the lowest linear calibration standards. These, therefore, are estimated values. Concentrations below the method detection limits are shown as nondetect (nd), which is not necessarily equal to zero. In addition, values have been rounded to two significant digits to reflect the estimated overall accuracy of the analyses. However, all computations used full precision before rounding, so a total or subtotal may not exactly equal the sum of the items in the column. Also, there are no 100% removals, even where nondetect is shown in the residuals column. Removal is computed as (feed conc. − residual)/feed conc.

The washing tests were performed with a Phipps and Bird 400 Series Flocculation Test Stirrer and 1-liter wide-mouth glass jars. Chemical measurements were performed with a Hewlett-Packard Model 5890 gas chromatograph equipped with a flame ionization detector (GC/FID), Model 7673 automatic liquid sampler, and

Model 3396A electronic integrator. Confirmation analyses were performed on a minimum of 10% of the samples at another laboratory using a Hewlett-Packard Model 5995 GC/MS equipped with a 7673 automatic sampler and an HP-1000 RTE-6/VM computer controller. All soil concentrations are shown on a dry weight basis.

A summary of the results of the initial bench-scale experiments is shown in Table 5. The data show that use of a nonionic surfactant, increased temperature, and increased pH improved the removal of the PCP and creosote. Surfactant increased the removal of the contaminants 55–820%, elevated temperatures accounted for a 37–540% increase, and elevated pH accounted for a 5–410% increase in contaminant solubility.

Temperature and pH did not have as great an effect on the solubility when surfactants were used as compared with when water alone was used. Triton X-100 and Tergitol NP-10 surfactants performed better by removing more contaminants than the Dowfax 8390 surfactant.

The results of the confirmation soil-washing tests are presented in Table 6. The data confirm that use of a nonionic surfactant, increased temperature, and increased pH improve the removal of the PCP and creosote. Surfactant increased the removal of the contaminants up to 130%, elevated temperatures accounted for a 20–80% increase, and elevated pH accounted for a 26–90% increase in contaminant solubility.

Table 7 presents the results of the sequential runs using an initial prewash at ambient temperature and pH, followed by two stages of washing with surfactant. PCP removal is markedly greater in these three-step washes. The target level of 30 ppm was achieved in all tests, including the one using three stages of water alone. Creosote

Table 5
Summary of results from the December bench-scale tests

Test conditions			Residuals		Removals	
Surfactant	Temp. (°F)	pH	PCP (mg/kg)	Total creosote (mg/kg)	PCP removed (%)	Creosote removed (%)
None	68	7.6	600	4300	13	6
Triton	68	7.6	290	2000	58	55
Tergitol	68	7.6	300	2200	57	51
Dow	68	7.6	400	2800	42	38
None	120	7.6	420	2800	39	39
Triton	120	7.6	140	1100	80	76
Tergitol	120	7.6	130	1200	81	74
Dow	120	7.6	520	3500	25	23
None	68	9	400	3000	42	34
Triton	68	9	240	2000	65	55
Tergitol	68	9	240	2100	65	54
Dow	68	9	340	2600	51	42

Feed soil concentrations: 690 ppm PCP, 4550 ppm total creosote.

Table 6
Summary of results from the January bench-scale tests

Test conditions			Residuals		Removals	
Surfactant	Temp. (°F)	pH	PCP (mg/kg)	Total creosote (mg/kg)	PCP removed (%)	Creosote removed (%)
None	68	7.6	170	2500	60	40
Triton	68	7.6	150	2300	64	45
Tergitol	68	7.6	170	2700	60	36
Tween	68	7.6	160	2500	62	39
None	120	7.6	220	3000	48	28
Triton	120	7.6	97	1500	77	65
Tergitol	120	7.6	82	1500	80	64
Tween	120	7.6	98	1700	77	60
None	68	9	66	2100	84	50
Triton	68	9	nd	1500	> 93	64
Tergitol	68	9	nd	1300	> 93	68
Tween	68	9	59	1100	86	72

Feed soil concentrations: 420 ppm PCP, 4130 ppm total creosote.
nd = compound below method detection limit.

Table 7
Summary of results from the three-step bench-scale tests

Test conditions		Residuals		Removals	
Treatment	Surfactant	PCP	Total creosote	PCP removed (%)	Creosote removed (%)
Feed	—	420	4100	—	—
Prewash	None	170	2500	60	40
Wash 1	None	nd	2300	> 91	44
Wash 2	None	nd	1400	> 96	66
Wash 1	Triton	nd	900	> 98	78
Wash 2	Triton	4	440	99	89
Wash 1	Triton	23	910	95	78
Wash 2	Triton	nd	550	> 98	87
Wash 1	Tergitol	19	1100	95	73
Wash 2	Tergitol	nd	310	> 99	92
Wash 1	Tween	120	990	71	76
Wash 2	Tween	nd	510	> 98	88

Wash 1 and wash 2 were conducted at 120 °F, pH 9, and surfactant concentration of 0.4%.
nd = compound below method detection limit.

Table 8
Mass balance estimates for selected runs

Contaminant: wash reagents	Mass of contaminant in sample (mg)			Percent recovered
	Feed soil	Output water	Output soil	
PCP				
Water wash	48	27	nd	56
Triton	48	29	1	62
Triton dup.	48	61	nd	126
Tergitol	48	41	nd	84
Tween	48	54	nd	112
Total creosote				
Water wash	474	27	162	40
Triton	474	53	51	22
Triton dup.	474	65	61	27
Tergitol	474	42	36	16
Tween	474	114	59	37

nd = compound below method detection limit.

removal also improved, but the target level of 100 ppm total creosote was not achieved with any of the wash solutions.

Mass balances were examined for the three-step tests, and the results are shown in Table 8. The concentrations of the contaminants were converted to total mass of contaminant in order to perform the mass balance. The total mass of contaminant in a soil sample was calculated by multiplying the soil concentration by the (undried) mass of soil used in the study (125 g) and by the solids content of the soil (92%). The total mass of contaminant in a wash-water sample was calculated by multiplying the extract concentration by the volume of water used in the study (3 washes × 750 ml = 2250 ml).

The mass balances are acceptable for PCP (56–126%). That is, this range is acceptable, considering measurement variability within the QA acceptance limits. However, the mass balances for creosote (16–40%) are poor. The low recoveries may have been due to matrix interference problems encountered with the water samples.

The following can be concluded from the two bench-scale studies:

1. Adding surfactant to the wash water, increasing the wash-water temperature, and increasing the wash-solution pH increases the removal of contaminants from the soil.

2. The nonionic surfactants tested performed equally well and better than the anionic surfactant.

3. Cleanup targets for carcinogenic creosote compounds and PCP were achieved after three consecutive washes of the Escambia soil. Dioxin and furan levels were reduced 95% on average.

4. The cleanup target for total creosote compounds was not met, even after three consecutive washes.

5. The coarser 'particles' of a soil may be as contaminated as the finer particles. The coarse material at wood-treating sites may be wood chips or other organic materials that can absorb large amounts of contaminant.

3. Pilot-scale testing

A pilot-scale washing study was conducted to evaluate the most effective bench-scale conditions using equipment that would provide better information about the probable success and cost of full-scale soil washing. This objective included the following subobjectives:
– investigate the effects of temperature and a higher ratio of soil to water,
– investigate the effects of pH more fully at the pilot scale,
– demonstrate the capabilities of the mobile soil washing unit,
– evaluate the costs and effectiveness of increasing surfactant concentration, pH, and temperature versus using additional wash stages,
– provide data for scale-up of the process.
The objectives did not include particle size separation because of the small percentage of fines present in the soil. The objectives also did not include evaluating the water recycling system of the VRU. Process slurry was collected in plastic tanks for later tests of commercially available UV/peroxide, chemical, thermal, and biological wastewater treatment systems. Tergitol NP-10 was selected as the single surfactant to use in the pilot-scale tests based on its effectiveness in the bench-scale tests and the desire to examine the effect of surfactant concentration more fully.

3.1. Apparatus

The VRU is a mobile, pilot-scale soil washing system. The system consists of process equipment and support systems mounted on two trailers (Fig. 1). Only those subsystems shown in Fig. 2 process schematic were used in this effort.

The feed hopper can hold up to 500 lb of soil. The screw feeder has a variable-speed drive and can convey solids to the miniwasher at up to 200 lb/h. In the miniwasher, soil is mixed with process water using a ribbon blender. Surfactant and caustic solutions may also be added at this point. The surfactant and caustic solutions are premixed in two plastic 55-gallon tanks and are fed to the miniwasher by metering pumps. The flows are adjusted to provide the proper water/soil consistency (about 1:2) for good attrition. The residence time in the miniwasher is about 30 min. A small feed screw conveys the soil from the miniwasher into the trommel.

The trommel is 15 in. diameter by 40 in. long and has 2-mm (10-mesh) slots. Process water is sprayed onto both the inside and outside of the trommel. The flow is measured by four rotameters and controlled manually using four valves to achieve up to a 10:1 weight ratio of water to soil.

The underflow from the trommel (finer than 10 mesh) is fed to the first of two vibrating screens. The screen inserts in these units can be changed to provide separations from 10 to 200 mesh. The first unit is typically set to 40 or 60 mesh, and the
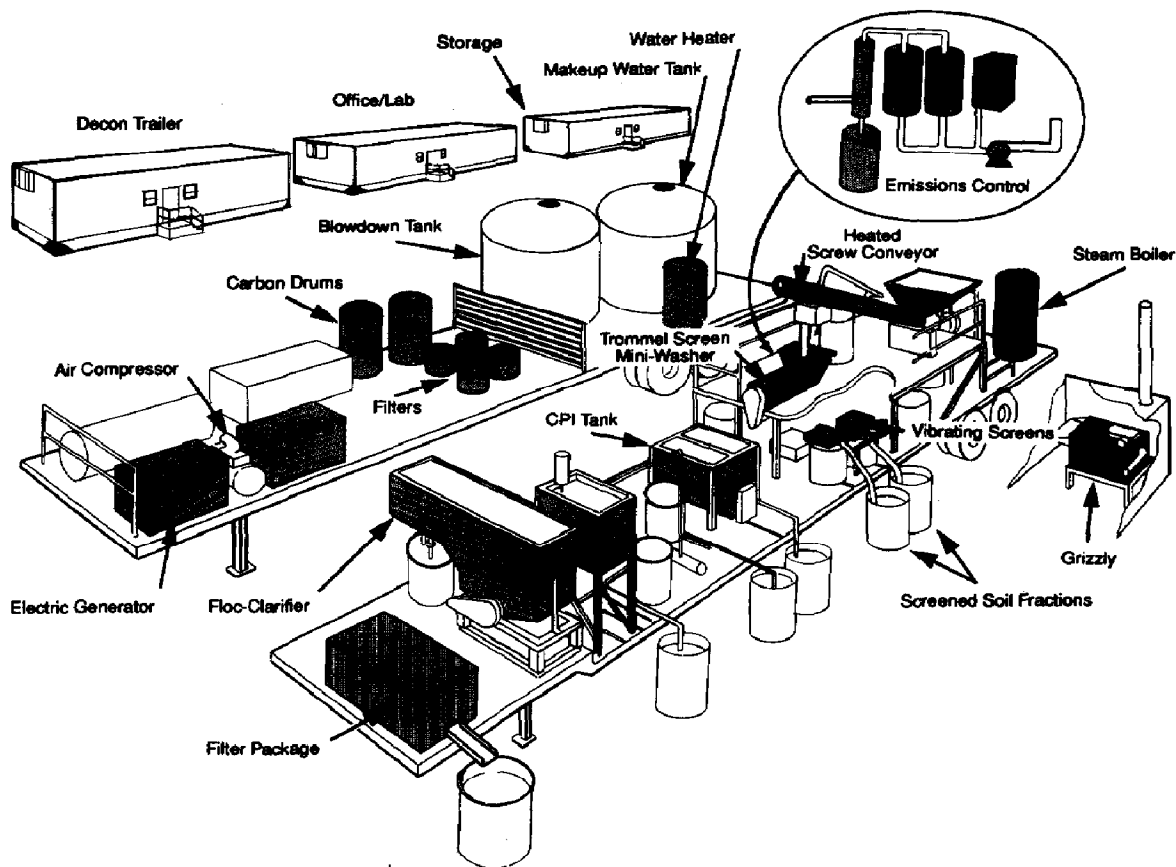
Fig. 1. The process and utility trailers of the VRU.

second unit is typically set to 100 or 140 mesh. The mesh sizes are selected to match the particle-size distribution of the feed soil. The overflows from the trommel and each vibrating screen are discharged to separate 55-gallon drums, and the final slurry underflow from the second vibrating screen drains into a stirred tank.

3.2. Procedures

Six cubic yards of soil from the excavation pit were placed on plastic near the VRU and mixed using a Bobcat and shovels. Two composite samples were sent for PCP/creosote analysis. The pile was then bermed with lumber and covered with plastic. A second batch of soil was later excavated from a vein of more heavily contaminated soil for use in runs 19 and 20.

The VRU was operated over a 10-day period at a nominal 100 lb/h. The unit needed approximately 30–60 min to achieve steady-state operation after the process conditions were changed to new settings. In order to avoid biasing the results, the unit was shut down on several occasions so that the residual soil remaining in the miniwasher, the trommel, and the vibrascreens could be removed before the start of the next run.
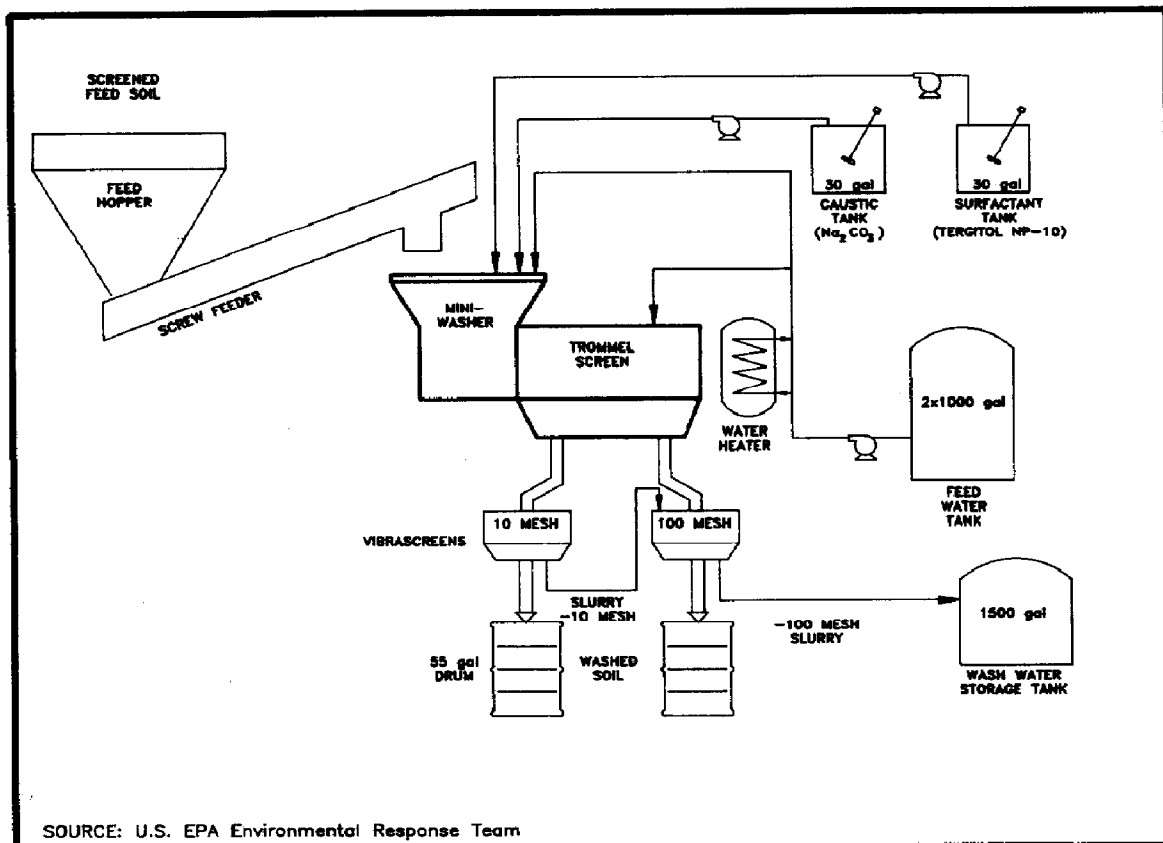
Fig. 2. Schematic of the soil washing and particle-size separation systems of the VRU.

The ERT and RCB team initially planned pilot-scale tests that would simulate the bench-scale studies previously performed in Brunswick. Runs during the first five days consisted of two single-pass tests per day at different values of pH, surfactant concentration, and temperature. Runs during the second five days were to consist of triple-pass tests. However, residual concentrations were so low during the first runs that the triple-pass tests were replaced with additional single-pass tests that explored additional process conditions.

Each run required approximately 300 lb of soil and 240 gallons of water. Soil from the premixed pile was screened by hand through 0.25-inch mesh hardware cloth into plastic 5-gallon pails. Lids were placed on the pails to minimize loss of contaminants by volatilization. The pails were weighed and dumped manually into the soil feed hopper. The approximate weight of a full pail was 50 lb.

The two vibrascreens contained screens with mesh sizes of 10 and 100 mesh. Due to the characteristic of the soils at the Escambia site, 98% of the soil was distributed in the −10 mesh to +100 mesh stream, with the remainder in the −100 mesh stream.

Each run lasted two hours. The start of the run was determined by the project manager once the proper slurry consistency was obtained in the miniwasher. Proper

consistency was determined by visual observation. Process readings were taken every 15 min during the run.

All the process wastewater was collected in a 1500-gallon container for later use in testing other processes.

The washed soil from several selected runs was rinsed with clean water to see if this additional step would improve the removal. The test team members speculated that most of the contaminant remaining in the soil was 'solubilized' in the interstitial water rather than bound to the soil. Thus it could be removed easily by simple flushing. To rinse, samples were placed on a 120-mesh screen over a beaker, and approximately one liter of clean water was poured through the sample.

Eight-ounce samples of the washed and screened soils (the 10-mesh to 100-mesh fraction), the feed soils, and 1-liter samples of the −100 mesh slurry were collected every 30 min during the run after allowing 30 min for stabilization. The samples were later composited and analyzed for PCP and creosotes. No samples were collected from the +10 mesh stream, because such a small portion of the soil was in that stream. The washed soil stream was collected in 5-gallon buckets and weighed every 30 min. Accumulated water in these buckets was then decanted and the bucket reweighed.

The samples taken from the feed piles during soil preparation were analyzed using the procedure described under bench-scale testing. The process slurry and wipe-sample analyses were analyzed using Method 8100 found in SW-846. Compounds analyzed were identical to those analyzed in the creosote/PCP soil analysis. Dioxin/furan analyses of the untreated soil, treated soil, and process slurry extracts were performed using Method 8290 of SW-846.

3.3. Results

The particle size distributions of the two feed soils used in the pilot-scale study are shown in Table 9, and the average feed-soil contaminant levels are shown in Table 10. Both soils had approximately the same particle size distribution. The first soil was red in color, and the second was red-brown. The soil used in runs 1–18 was typical for the site. Since the results were favorable, the second soil, which has a higher level of creosote contamination, was obtained for runs 19 and 20.

For both soils, the blending produced a very consistent feed, reducing the likelihood of misinterpretations when comparing the results of one run with another.

Table 11 summarizes the results for the pilot-scale experiments. The contaminant removals were generally high, and the cleanup criteria were easily met using several different sets of conditions.

The target residual of 30 ppm for PCP was achieved in all runs. Most results are lower than 4 ppm.

The target residuals of 100 ppm total creosote and 50 ppm carcinogenic creosotes were achieved in many runs. The maximum residual of total creosotes was 200 ppm, and most were less than 90 ppm. The maximum residual of carcinogenic creosotes was 12 ppm, and most were less than 5 ppm.

Table 9
Pilot-scale particle size distribution

Sieve No.	Particle size (mm)	Runs 1–18 Weight (%)	Runs 19 and 20 Weight (%)
5–3	4–6	0	0
10–5	2–4	1	3
18–10	1–2	10	12
35–18	0.5–1	60	59
60–35	0.25–0.5	23	21
120–60	0.125–0.25	5	4
Pan	< 0.125	1	1

Table 10
Average feed soil contaminant levels (mg/kg)

Percent solids	Runs 1–18	Runs 19 and 20
	93	90
Naphthalene	180	980
Acenaphthene	130	230
Fluorene	130	240
Phenanthrene	340	570
Anthracene	59	110
Carbazole	17	23
Fluoranthene	170	160
Pyrene	100	97
Benzo(a)anthracene	24	39
Chrysene	23	36
Benzo(b)fluoranthene	9	14
Benzo(k)fluoranthene	8	14
Benzo(a)pyrene	7	12
Total creosotes	1200	2500
Carcinogenic creosotes	71	110
Pentachlorophenol	150	130

Of the primary independent process variables, the concentration of surfactant had the greatest effect on removal of creosotes. The effect was moderate but important, in that it brings most residual levels of total creosote below the target concentrations. The pH of the wash solution had the next greatest effect on removal of creosotes. The effects of temperature and liquid:solid ratio were minor.

The pH of the wash solution had the greatest effect on removal of PCP, but the effect was not strong. The other variables had insignificant effects on the removal of PCP.

Table 11
Summary of results from the pilot-scale tests

Test conditions					Residuals (mg/kg)			Removals (%)		
Run No.	Comment	Surfactant (%)	Temp. (°F)	pH	PCP	Total creosote	Carcinogenic creosote	PCP	Total creosote	Carcin. creosote
1	Wash	0.4	120.0	10.0	2.9	48	2.3	98	96	97
3	Wash	0.4	85.0	10.0	2.0	60	3.3	99	95	96
2	Wash	0.2	120.0	10.0	1.7	120	8.2	99	89	90
11	Wash	0.2	120.0	10.0	3.8	80	3.4	98	94	95
11	W & R	0.2	120.0	10.0	nd	17	0.0	100	99	100
20	Wash	0.2	120.0	10.0	1.6	66	2.4	99	97	98
20	W & R	0.2	120.0	10.0	nd	1.6	0.0	100	100	100
4	Wash	0.2	85.0	10.0	2.0	69	3.5	99	95	96
4	W & R	0.2	85.0	10.0	nd	32	0.0	100	97	100
17	Wash	0.2	100.0	8.5	2.2	56	2.7	99	96	97
9	Wash	0.2	120.0	7.0	9.6	150	6.5	94	89	91
10	Wash	0.2	85.0	7.0	6.6	94	4.0	96	93	94
12	Wash	0.1	120.0	10.0	3.3	76	3.7	98	94	95
14	Wash	0.1	120.0	10.0	4.5	99	4.4	97	92	94
15ª	Wash	0.1	120.0	10.0	2.8	66	3.1	98	95	96
13	Wash	0.1	85.0	10.0	3.7	99	4.1	98	92	94
16	Wash	0.1	100.0	8.5	3.8	86	4.1	98	93	95
5	Wash	0.0	120.0	10.0	2.8	110	5.1	98	91	93
6	Wash	0.0	85.0	10.0	2.5	130	6.0	98	90	92
6	W & R	0.0	85.0	10.0	nd	92	4.7	100	93	94
18	Wash	0.0	100.0	8.5	2.1	150	8.8	99	88	88
8	Wash	0.0	120.0	7.0	7.4	170	9.6	95	86	85
7	Wash	0.0	85.0	7.0	10.3	200	12	93	85	84
19	Wash	0.0	85.0	7.0	2.4	110	3.7	98	96	97
19	W & R	0.0	85.0	7.0	nd	36	0.0	100	99	100

ªA higher liquid:solid ratio (8.2:1) was used in this run (versus 6:1 in all other runs).
nd = nondetect.
W & R = wash followed by rinse.

Rinsing after washing proved to be highly beneficial, reducing residual PCP below detection limits in all five cases and reducing carcinogenic creosotes below detection limits in four out of five. Total creosotes were also significantly reduced (to below target levels) in all five cases.

The improvements due to rinsing were probably caused by displacing residual contaminated wash water from the spaces between the soil particles. Run No. 15, which is similar to run No. 12 but had a higher liquid:solid ratio, produced a higher removal efficiency. However, the difference is small and probably not statistically significant. No replicate runs were performed to prove this.

The process slurry samples were collected from the underflow outlet of the second vibrascreen. One-liter samples were collected every 30 min and were composited at the end of the run. This stream was found to contain 1–2% solids.

Process slurry contaminant levels ranged from 0.94 to 23 mg/L of PCP and 5.7 to 42 mg/L of total creosote compounds. Most values are nondetect or are below linear detection limits. These levels are one to two orders of magnitude lower than expected. Matrix spikes of the soil samples showed no analytical interference from the surfactant. For the soil samples, recoveries of surrogates were 70–126% and recoveries of matrix spikes were 69–117%. So we speculate that the contaminants were tightly bound to the fines in the slurry and were not easily extractable.

To evaluate this possibility, the process slurry from run No. 16 was flocculated by reducing the pH to 3.5, and the resulting suspended solids were removed using filter paper. Surrogate recoveries for the unfiltered samples were 34–38%, and recoveries for the filtered samples were 61–74%. These results support the hypothesis.

Checks with matrix spikes add further evidence. Matrix spike recoveries for the slurry samples ranged from 0 to 110%, with 11 out of 29 exceeding QC limits. This provides further evidence that the contaminants were associated with the settled fines. It also means, unfortunately, that good mass balance of the contaminants could not be conclusively demonstrated for the process.

The VRU ran smoothly with only a few operational problems during the course of the study.

The soil, although dry, tended to bridge or to 'rat-hole' in the soil hopper. An operator had to stir the soil periodically to minimize this problem.

The soil flow rate was somewhat erratic from run to run, even when the same feed-screw settings were used. For the same screw setting, soil flow varied between 83 and 128 lb/h. The variability of the soil flow rate resulted in variability of the liquid:solid ratio (L/S) from 4.9:1 to 7.6:1 around the intended value of 6:1.

Only one run intentionally used a different L/S ratio. Run No. 15 used the same settings as run No. 12 except for an L/S ratio of 8.2:1 (targeted to be 10:1) to evaluate the effect of a high L/S. The higher L/S ratio seems to have had no effect on the extraction efficiency. We speculate that an L/S of 6:1 is much greater than the minimum L/S needed for good washing, so little improvement results from increasing the L/S.

The vibrating screens were less efficient at dewatering when 0.2% or more surfactant was used at ambient temperature. Under these conditions, 50–100% by weight of excessive water accumulated in the 5-gallon buckets used to capture the solids, and

foaming was evident. Generally, during well-dewatered runs, the buckets contained approximately 10% by weight excess water. Foaming and dewatering problems were less severe at higher slurry temperatures, probably due to the fact that higher temperatures reduce the surface tension, which reduces the formation of bubbles.

3.4. Conclusions

1. The pilot-scale tests have shown that soil washing can be an effective means of removing PCP and creosotes from sandy soil.

2. Surfactant was the most important process factor for reducing the creosote below the target levels.

3. Increasing the wash water pH using sodium carbonate had a slight additional positive effect on reducing the PCP and creosote levels.

4. Increasing the wash water temperature had little effect within the range from 85 to 140 °F.

5. Changing the liquid:solid ratio had little effect over the range from 4.9 to 8.2.

6. The pH of the wash water was the most important process factor for reducing PCP levels. A pH of 8.5 produces almost complete removal.

7. Rinsing with clean water after washing can be highly effective in reducing residual contamination levels.

8. The majority of the contaminants apparently end up associated with the fines in the process slurry. The binding is apparently very tight. Good mass balance cannot be demonstrated for the washing process as a result.

3.5. Recommendations

1. A final water rinse unit should be added to the VRU. A final rinse should be included in a full-scale remediation using soil washing. A two-stage counter-current operation should be extremely effective.

2. The washwater for a full-scale remediation at the Escambia site should have a pH of 8.5 and a nonionic surfactant such as Tergitol NP-10 at 0.1% by weight.

3. Standardized bench-scale test procedures should be developed that will closely approximate what is achievable at field and pilot scale.

4. The suspended solids in the process slurry should be removed by coagulation, flocculation, filtration, centrifugation, or some combination of these. This step could substantially reduce the volume of contaminated material and allow recycling of the wash water.

5. Effective analytical methods should be developed for measuring the amount of contaminant in the fines.

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