



Evaluation of soil washing process to remove mixed contaminants from a sandy loam

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Abstract

Soil washing is a remediation process which is primarily used to treat soils and sludges which are contaminated with only one or two groups of contaminants (e.g., metals and/or volatile organic compounds). This process, however, has not been extensively employed on soils that are contaminated with pesticides in addition to metals and volatile organic compounds. This paper describes the development of a new soil washing process which is used to remove these mixed pollutants from soils. First, an overview of the soil washing process and its previous applications for soil remediation is presented. Then, an extensive experimental program which determined the most effective wash solution for removing mixed pollutants from a sandy loam is described. The sandy loam soil used for this experimentation was poorly graded, containing 66% sand and 34% silt/clay. The contaminants remediated in the experiments were: (1) metals (cadmium, silver, and copper), (2) volatile organic compounds (ethyl benzene, methyl iso-butyl ketone), (3) halogenated compounds (chloroethene, tetra chloroethylene), and (4) pesticides, herbicides, and insecticides (lindane, methoxychlor, and endrin). The experimentation results determined that a combination of 2.5 *N* sulfuric acid and iso-propyl alcohol in a 4:9 ratio and with a dilution of 5:1, solution to soil, would form as effective wash solution. The paper also describes the development of a bench-scale model and the results obtained from these bench-scale tests. These tests confirmed that the new soil washing process can remove the mixed pollutants efficiently and economically.

Keywords: Soil; Mixed pollutants; Remediation; Washing

1. Introduction

Numerous hazardous waste sites exist in the United States today because of accidental surface spills, leaking underground storage tanks and landfills, uncontrolled waste disposal, industrial and military activities, and other man-made events.

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Significant efforts are now underway to clean up these sites in order to prevent any adverse effects to both human health and the surrounding environment. Currently, many treatment processes have been developed or are now being developed which can remediate contaminated soils in an effective and economic manner [1–3]. The most popular remediation processes for these soils include acid extraction, bioremediation, and thermal desorption. However, these processes have been found to be successful only for the removal of specific groups of contaminants from soils; either metals, volatile organic compounds, or pesticides and herbicides, and are not effective for the removal of mixed pollutants which can include a combination of these contaminants. As a result, two or more remediation processes are used in combination to remove the mixed pollutants from the soils, resulting in expensive remedial operations. This paper presents a new soil washing technique which can remove metals, volatile organic compounds, and pesticides and herbicides, all in a one step process. The new process is easy to implement, efficient, and economical.

The paper first describes the development of the new soil washing process which included performing laboratory tests on a sandy loam soil sample under a variety of environmental conditions. Following the laboratory testing, a bench-scale soil washing unit was fabricated and used for evaluating the remediation process. The bench-scale tests confirmed that this new process is efficient in removing the combination of metals, volatile organic compounds, and pesticides and herbicides from the contaminated soil.

2. Background

Soil washing is a treatment process used for remediating both organic and inorganic chemical constituents from contaminated soils, sludges, and sediments [3–5]. This process involves high energy contact between the contaminated soils and an aqueous based washing solution. Soil washing can be a physical and/or chemical process which results in the separation, segregation and volume reduction of hazardous materials and/or the chemical transformation of contaminants to nonhazardous, unregulated substances.

The steps involved in soil washing are: (1) excavation of the contaminated soil; (2) remedial treatment of the contaminated soil; (3) solid/liquid separation of all contaminants; (4) treatment or disposal of all residues; and (5) final soil deposition. Large debris or soil particles coarser than 2 in. are separated out before treatment begins. The soil then enters a soil scrubbing unit. The soil portion containing sand needs only initial rinsing treatment because contaminants do not strongly adhere to the sand particles. The silt/clay fraction needs more extensive remedial treatment because contaminants are easily sorbed by this fine-grained soil fraction, and consequently, are more difficult to desorb. The solid/liquid separation process then takes place, and the contaminated wastewater containing residues is treated in a conventional wastewater treatment system and recycled. At this stage, the contaminants have either been destroyed or are isolated and removed. The last step in the soil washing process is the redeposition of the remediated soil in the appropriate location [4, 5].

The effectiveness of soil washing is dependent on the soil type and contaminants that it contains. Soil washing is most effective for removing halogenated volatiles, nonhalogenated volatiles, and metals contained in sandy and gravelly soils. However, the applicability of this procedure must be tested when the soils are classified as silt or clay because soil washing is not considered effective when soils contain more than 20–30% silt/clay [3–5].

The type of extractive agent needed for the specific soil washing process depends on the class of contaminant and the soil type. Water washing with a basic or surfactant agent is used to remove organics. The types of basic aqueous solutions used as extractive agents include caustic lime, slaked lime, or industrial alkali-based washing compounds. An acidic or chelating agent is used to remove organics and heavy metals. Acidic aqueous solutions used as extractive agents include sulfuric, hydrochloric, nitric, phosphoric, and carbonic acids. Oxidizing agents, such as hydrogen peroxide and sodium hypochlorite, chemically change the contaminants and often facilitate the washing process. Organic-solvent washing agents are used to remove hydrophobic organics and PCBs [3–6].

There are numerous advantages to soil washing as a remediation technique. First, the actual process takes place in a closed system which permits control of the ambient environmental conditions. Secondly, the process can result in a significant volume reduction of the contaminant mass. Also, soil washing has extensive applications for varied waste groups, and the hazardous waste can remain on site due to mobile technology. The cost of soil washing is relatively low compared to other multi-contaminant technologies and significantly lower than the cost of landfilling.

Soil washing also has disadvantages as a remediation procedure. When the soil washing treatment is only a physical process, there is little reduction in the toxicity of the contaminants. If chemical processes are involved, potentially hazardous chemicals that are used in the remediation process may then be difficult to remove from the treated soil and may remain on site. The effectiveness of soil washing is also limited by the following factors: (1) complex waste mixtures, (2) high humic content of soils, (3) inhibiting solvent–soil reactions, and (4) high fine-grained clay content of soils.

Soil washing has been previously used to remove various specific contaminants which include oil, heavy metals, cyanide, and bromide. A few of the past applications of soil washing are listed below:

(1) In 1988, the Soil Cleaning Company of America remediated bulk soil of oil and grease using hot water with surfactants [7]. The removal efficiencies of both the oil and grease was 50–83%, with 250–600 ppm residual. The residue was wet oil.

(2) The EPA's Mobile Counter-Current Extractor used various solvents, additives, surfactants, redox acids, bases and the chelating agent EDTA to clean soluble organics and heavy metals [8]. Soil ranging in size from 2 to 25 mm was cleaned in a drum washer and soil less than 2 mm was cleaned in a four stage extractor. The removal efficiency for phenol was 90% from inorganic soil and 80% from organic soil. The As_2O_3 was removed with an efficiency of 50–80%. The residue was the clay fraction, the recovered organics, and the spent carbon.

(3) Beginning in 1985, the Heijman Milieutechniek Company in the Netherlands used soil washing to clean cyanide, heavy metals, and mineral oil from soil which was greater than 10 mm in size and contained no more than 30% soil particles which were less than 63 μm in size [3, 5]. Proprietary extraction agents were used. Hydrogen peroxide was added to react with extracted CN to form CO_2 and NH_3 . The cyanide was removed to an efficiency of 93–99%, while the heavy metal cations were removed approximately 70%. The sludge residue was composed of flocculated fines.

3. Objective and scope of present study

The objective of this study was to develop a soil washing process which would effectively remove mixed contaminants from a sandy loam. To achieve this objective, the scope of work included the following tasks:

- (1) performing laboratory tests to determine the feasibility of using soil washing to remove mixed contaminants from a sandy loam;
- (2) determining the most efficient wash solution for removing these mixed contaminants;
- (3) designing and fabricating a bench-scale soil washing unit that could process up to 5 kg of contaminated soil at a time;
- (4) performing bench-scale tests to investigate the remediation efficiency of the soil washing process.

The results of these tasks are presented and discussed in the following sections.

4. Laboratory investigation

The laboratory investigation was performed in order to determine the feasibility of using soil washing as an effective remediation technique for removing the mixed contaminants from a soil classified as sandy loam. The laboratory investigation also determined the most efficient wash solution for removing the contaminants. The laboratory test variables, procedures, results, and discussion of results are presented below.

4.1. Soil type

The soil used for this study was a brown, sandy silt. The grain size distribution of the soil was determined by performing sieve analysis in accordance with the ASTM Standard D 442. The results of this analysis are presented in Table 1 and Fig. 1. From this analysis, the soil was found to consist of 66% sand and 34% silt/clay. Based on the Unified Soil Classification System (USCS), the soil is classified as silty sand (SM), while according to the US Department of Agriculture (USDA) Classification System, the soil is classified as sandy loam. For the purpose of this paper, the soil type is referred to as sandy loam.

Table 1
Grain size distribution of sandy loam soil

Component	Size (mm)	Percent retained	Percent finer
Sand	4.0	0	100
	2.0	20	80
	1.0	14	66
	0.5	14	52
	0.25	10	42
	0.106	8	34
Silt and clay	0.05	34	~

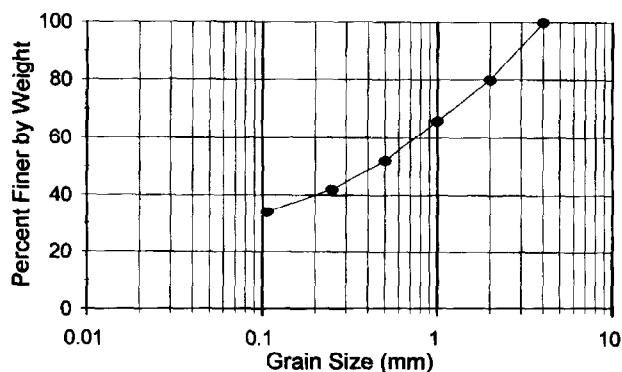


Fig. 1. Grain size distribution of sandy loam.

4.2. Contaminants

The contaminants considered for this study are representative of actual contaminants which could be encountered at a hazardous waste site. These contaminants include various hazardous substances from the following categories: (1) pesticides, herbicides, insecticides, (2) metals, (3) volatile organic compounds, and (4) halogenated compounds. The compounds from the first category included lindane, methoxychlor, and endrin. The metals studied were cadmium, silver, and copper. The volatile organic compounds examined were ethyl benzene, and methyl iso-butyl ketone. The halogenated compounds included chloroethene and tetra chloroethylene. Table 2 summarizes the concentrations of these different chemicals which were contained in the soil. This table also presents the desired remediation levels and the desired removal efficiencies for this study.

4.3. Sample preparation

To simulate a possible field contamination as given in Table 2, soil samples were mixed with the appropriate amounts of different chemical contaminants, based on the results of stoichiometric analysis. The chemicals were first solubilized in the

Table 2
Soil contamination levels and desired remediation levels

Contaminant	Concentration in soil (mg/kg)	Remediation criteria (mg/kg)	Desired removal efficiency (%)
Pesticides, herbicides, insecticides			
Lindane	150	10	93.3
Methoxychlor	150	10	93.3
Endrin	150	10	93.3
Metals			
Cadmium	350	15	95.7
Silver	100	15	85.0
Copper	100	15	85.0
Organic compounds			
Ethyl benzene	75	10	86.7
Methyl iso-butyl ketone	100	10	90.0
Halogenated compounds			
Chloroethene	75	14	81.3
Tetra chloroethylene	100	14	86.0

appropriate solvents to ensure uniform contaminant concentration levels. To minimize losses due to volatilization, the volatiles were stored in the freezer. The soil was contaminated in the laboratory with these chemicals immediately preceding each trial. On most occasions, several contaminated soil samples were prepared simultaneously. The differences in contamination levels of the samples were minimized by contaminating a single batch of soil, and then placing equal amounts of the contaminated soil into individual sample containers. Each container held 10 g of soil. One batch sample was not contaminated and was used as a control. The appropriate wash solutions were then added to the other experimental samples in the amount of 50 ml of solution per each sample. The wash solutions used were hydrochloric acid, nitric acid, sulfuric acid, and a combination of sulfuric acid and isopropyl alcohol.

4.4. Testing procedure

The soil solution was placed in a rotary apparatus (Millipore) which tumbled the sample at 30 revolutions per minute for the specified time period. Experiments were conducted by varying the type of acid used for the solution, the normality of the acid solution, and the treatment time. Treatment times ranged from 1 to 24 h. One hour tests aided in establishing the relative removal capacities of the extractive agents. The initial testing utilized a closed system (no head space in the container), while all other tests were conducted using an open system (head space in the container) to assure that the volatiles would have room to escape.

After the allotted treatment time, the sample was removed from the rotary apparatus. The effluent was separated from the soil using a 5 μm pore size pressure filter

(Millipore). The soil remained in a wet condition and was removed from the filter paper, placed in a sample container and preserved.

4.5. Analytical testing

The samples were analyzed using the test procedures set forth by the Environmental Protection Agency. The pesticides were evaluated using EPA Standard SW-846 Method 8080-Standard Method for Analyzing Organochlorine Pesticides. Samples were prepared for analysis using SW-846 Method 3550. Volatiles were tested using SW-846 Method 8240, while metals were tested in accordance with the procedures described in USEPA [9]. The complete details on these analytical test procedures are also given in USEPA [10] and Keith [11].

4.6. Test results and discussion

Acid type: The test results using the different acid extraction solutions are summarized in Table 3. From these results, it can be seen that, overall, the sulfuric acid was better than either the hydrochloric or the nitric acid in removing both the volatiles and the pesticides. The 1 N sulfuric acid was more effective than either the 5 N or the 0.5 N sulfuric acid in removing pesticides. The hydrochloric acid was the most efficient wash solution for the removal of the metals. Generally, the stronger the acid, the greater the percentage of metal removal.

Use of isopropyl alcohol: Soil samples contaminated with pesticides were analyzed after a 1 h treatment time. One batch of samples was treated with a 1 N sulfuric acid solution, while the other batch was treated with a 1 N sulfuric acid and isopropyl

Table 3
Evaluation of different extraction solutions (tumbling time = 1 h)

Chemical contaminant	HCl (% removal)			H ₂ SO ₄ (% removal)			HNO ₃ (% removal)			Required (% removal)
	4 N	1 N	0.5 N	5 N	1 N	0.5 N	5 N	1 N	0.5 N	
Methyl iso-butyl ketone	92.0	98.1	95.2	98.5	98.0	97.8	97.6	98.3	^a	90.0
Tetra chloroethylene	49.3	90.4	75.6	92.0	86.8	87.7	83.0	89.0	^a	86.0
Ethyl benzene	76.9	94.8	89.5	94.7	93.0	93.2	91.4	93.8	^a	86.7
Chloroethene	ND ^a	ND	ND	ND	ND	ND	ND	ND	ND	81.3
Lindane	58.0	26.0	66.0	63.0	72.0	55.0	51.0	57.0	69.0	93.3
Methoxychlor	56.0	7.0	61.0	60.0	65.0	45.0	40.0	53.0	66.0	93.3
Endrin ^b	99.0	38.0	70.0	76.0	83.0	56.0	84.0	79.0	84.0	93.3
Cadmium	97.2	96.9	97.1	95.7	85.4	81.3	97.3	21.5	88.0	95.7
Silver	99.0	98.0	78.8	62.1	78.8	84.3	86.6	86.6	79.8	85.0
Copper	87.5	78.9	72.1	73.2	39.6	24.0	59.4	49.9	44.6	85

^aND – not detected – chloroethene volatilizes below room temperature and proved to be difficult to stabilize in the soil.

^bEndrin was partially converted into endrin ketone, another hazardous substance.

Table 4
Removal efficiencies using 1 N sulfuric acid and isopropyl alcohol (tumbling time = 1 h)

Contaminant	1 N sulfuric acid (% removal)	1 N sulfuric acid and isopropyl alcohol (% removal)
Lindane	37.0	54.0
Endrin	39.0	48.0
Methoxychlor	34.0	43.0

Table 5
Removal efficiencies using sulfuric acid (tumbling time = 24 h)

Contaminant	Sulfuric acid (1.5 N) (% removal)	Desired (% removal)
Methyl iso-butyl ketone	64.0	90.0
Tetra chloroethylene	38.0	86.0
Ethyl benzene	46.0	86.7
Chloroethene	62.0	81.3
Lindane	96.5	93.3
Methoxychlor	92.3	93.3
Endrin	95.6	93.3
Cadmium	91.9	95.7
Silver	81.5	85.0
Copper	NA ^a	85.0

^aNA - The test failed to pick up the copper in the untreated sample.

alcohol combination solution. These test results are summarized in Table 4. The percentage of contaminant removed was greater for all pesticides when the combination of sulfuric acid and isopropyl alcohol was used as the wash solution. The sulfuric acid and isopropyl alcohol combination removed 17% more lindane, 9% more endrin and 9% more methoxychlor than the solution which contained only the sulfuric acid.

Closed versus open system: Testing was performed using 1.5 N sulfuric acid in a closed system (i.e., with no head space) for 24 h, and the test results obtained are shown in Table 5. From these results, it can be seen that only 38-64% of the volatiles were removed after a 24 h treatment time. It should be noted that the test results shown in Table 3 were all based on experiments which were performed in an open system (i.e., with headspace). From Table 3, it can be seen that the open system allowed for almost complete removal of the volatiles after 1 h treatment time.

Treatment time: One normal HCl was tested after 2, 4, 12, and 18 h to determine the effect of treatment time. These test results are shown in Table 6. The best results for pesticides were obtained after 18 h. The 24 h test using the sulfuric acid and isopropyl alcohol combination removed over 92% of the pesticides.

The pesticides which were treated with the combination of 1 N sulfuric acid and the isopropyl alcohol solution were analyzed after each hour of tumbling for a total of 3 h. These test results are shown in Table 7. All three pesticides showed linear relationships between removal rates and time. A 90% lindane removal efficiency was

Table 6
Effect of treatment time on removal efficiencies (1 N HCl for pesticides)

Contaminant	Percentage of contaminant removal after indicated time			
	2 h	4 h	12 h	18 h
Lindane	0	49	54	60
Endrin	63	92	97.6	98.6
Methoxychlor	0	12	7	37

Table 7
Removal efficiencies using 1 N sulfuric acid and isopropyl alcohol (three 1 h cycles vs. one 3 h cycle)

Contaminant	Three 1 h cycles (% removal)			One 3 h cycle (% removal)
	1	2	3	
Lindane	30.0	54.0	76.0	52.0
Endrin	15.0	26.0	38.0	36.0
Methoxychlor	16.0	27.0	39.0	40.0

obtained after approximately 6 h, while a 90% endrin removal efficiency was obtained after approximately 8 h. Seven hours were required for a 90% methoxychlor removal efficiency.

Discussion of test results: The test results showed that hydrochloric acid is an effective remediation agent for the removal of metals from the contaminated soil sample. Sulfuric acid was found to be more effective than hydrochloric acid in removing pesticides from the soil, however. The experimentation determined that isopropyl alcohol enhances the effectiveness of sulfuric acid in the removal of pesticides. Treatment time was found to be significantly longer for pesticide removal than for the removal of volatiles or metals. The results indicated that the most effective soil washing technique would be to treat the contaminated soil with the combination of sulfuric acid and isopropyl alcohol for a time period ranging from 12 to 24 h, depending upon the desired removal level.

In previous applications, soil washing has not been recommended for use if the soil consists of more than 20–30% fines [3–5]. Although the soil used for this study contained 34% fines, higher than desired remedial efficiencies were obtained by using the selected wash solution.

Based on this investigation, the different steps involved in a typical on-site soil washing process are shown in Fig. 2. The process consists of tumbling the contaminated soil with the sulfuric acid and isopropyl alcohol combination solution. After the adequate treatment is achieved, the soil is filtered and the effluent is separated. The soil pH is adjusted by adding the appropriate basic substance such as lime and the soil is then returned to the excavation site. Laboratory tests performed on the effluent collected after soil washing with sulfuric acid and isopropyl alcohol showed only metals to be present, while the organics and pesticides were not detected. In

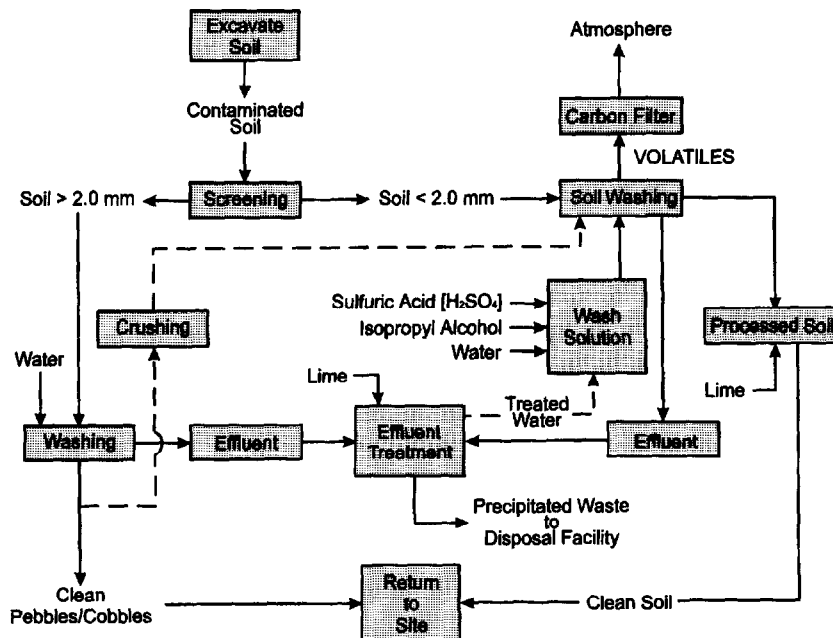


Fig. 2. Typical on-site soil washing process.

order to precipitate out the metals, an appropriate basic substance such as lime could be added which would raise the pH of the effluent. The supernatant resulting from this can be recycled for subsequent soil washing, while the precipitated material can be either salvaged or disposed of at a hazardous waste facility. A treatability study should be conducted to fully evaluate the soil washing process for any given site specific conditions [12, 13].

5. Bench-scale testing

5.1. Model design and construction

A bench-scale model was designed and fabricated at the University of Illinois at Chicago. This model was used to verify the remedial efficiency of the soil washing process on a larger scale. The bench-scale model was able to process up to 5 kg of contaminated soil at a time. A schematic of the bench-scale model is shown in Fig. 3. The model is very simple, easy to operate, and cost efficient. A brief description of the model is given below.

The model consists of a tumbler composed of PVC material. The tumbler is 12 in. in diameter and 3 ft in length, and can be capped at both ends. The bottom cap is permanently sealed and has a PVC valve attached to it for effluent removal. The top cap is removable so that the soil and wash solution can be placed into the tumbler. A vinyl hose is connected to the top cap which allows the volatiles to escape during

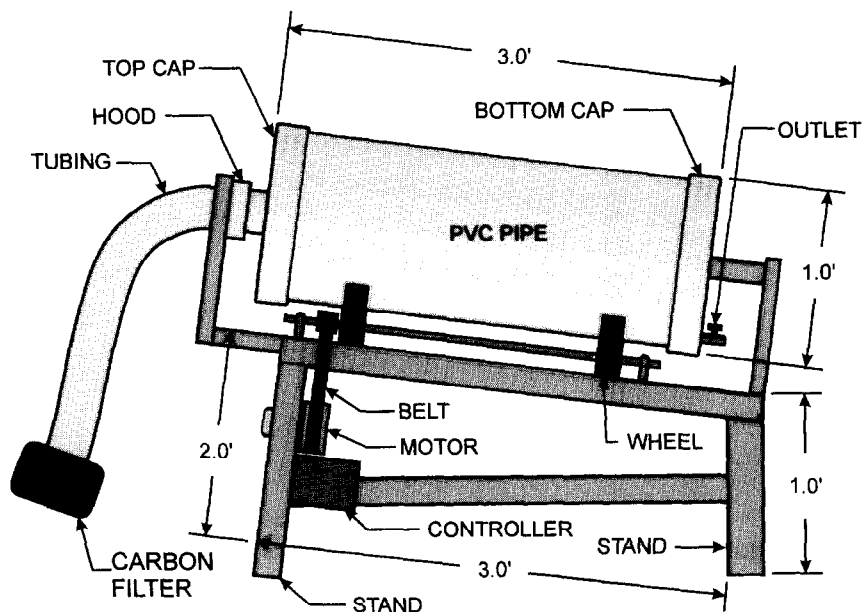


Fig. 3. Schematic of bench-scale model.

the machine operation. The other end of the hose is connected to a carbon filter for the safe collection of these volatiles (Fig. 3).

The tumbler is placed on an inclined stand for actual operation. The stand is constructed of aluminum perforated angles with bolted connections. Additional tumbler support is supplied by a rotating wheel that is rigidly attached to the frame. The tumbler itself rests on 4 in. rubber wheels connected at the ends of two 0.5 in. steel rods. The rods are attached to the structure by pylon blocks so that they can rotate freely. One rod has a 2 in. pulley, facilitating a belt which is driven by a variable speed DC motor. The speed variable control of the motor allows the speed of the mixing tumbler to be adjusted during operation.

5.2. Testing procedure

The bench-scale testing procedure consisted of first placing 5 kg of contaminated soil in the tumbler. Next, 25 l of wash solution was added to the soil. The wash solution used for this testing consisted of a mixture of 2.5 *N* sulfuric acid and isopropyl alcohol in a ratio of nine parts alcohol to four parts acid. The top cap of the tumbler was then secured. It is noted that the tumbler size was specifically designed to accommodate 5 kg of contaminated soil and 25 l of wash solution as well as to provide adequate headspace to allow the volatiles to escape.

The tumbler was rotated at approximately 20 revolutions per minute for approximately 20 h. This tumbling time ensured a thorough interaction of the wash solution with the contaminated soil. Any volatiles emitted from the soil during operation were able to exit through the vinyl hose into a carbon filter. At the end of the

Table 8
Summary of bench-scale test results

Contaminant	Concentration prior to soil washing (mg/kg)	Concentration after soil washing (mg/kg)
Methyl iso-butyl ketone	100	ND
Tetra chloroethylene	100	ND
Ethyl benzene	75	ND
Chloroethene	75	ND
Lindane	150	6
Methoxychlor	150	5
Endrin	150	ND
Cadmium	350	11
Silver	100	29
Copper	100	5

^aND: not detected.

operation, the effluent was separated and the soil was filtered. The soil was then dried and tested to determine the final concentrations of chemical constituents.

5.3. Results and discussion

Ninety five percent of the original soil was recovered after the bench-scale testing. The concentrations of chemical constituents found in the soil after this process are summarized in Table 8. The results show that all of the volatiles as well as the endrin were removed to trace levels. The removal efficiencies of copper, silver, and cadmium were 95%, 71%, and 97%, respectively. Lindane was removed to 96%, and methoxychlor was removed to 97%. The contaminant removal efficiency of this soil washing process exceeded the desired remedial levels for all contaminants except silver.

6. Summary and conclusions

Soil washing can be an efficient and effective remediation treatment for soils containing mixed pollutants. In this study, the feasibility of using the soil washing process to remediate a soil classified as sandy loam contaminated with mixed pollutants was investigated. The mixed pollutants contained in the soil included lindane, methoxychlor, endrin, cadmium, copper, ethyl benzene, methyl iso-butyl ketone, chloroethene, and tetra chloroethylene. Based on the laboratory and bench-scale test results, the following conclusions can be drawn:

(1) The combination of sulfuric acid and isopropyl alcohol was determined to be the most appropriate wash solution among the different wash solutions investigated.

(2) Soil treatment time will vary based upon the types of contaminants it contains, the desired remediation efficiencies, soil type, and the wash solution used. A total treatment time of 20 h was found adequate for the sandy loam tested in this study.

(3) Pesticide treatment is determined to be rate limiting factor in soil washing treatment of mixed waste.

(4) Fines contained in the soil do not necessarily have to be discarded as waste to obtain high contaminant removal rates. Discarding fines could shorten the treatment time, but would add to the by-products generated from the remediation process.

(5) The experimental soil washing process utilized only one wash solution and generated minimal waste bi-products. This process was, therefore, determined to be an efficient, simple and cost effective means of treatment.

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