Determination of Soil Washing Condition for Light Hydrocarbon Contaminated Soils

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Light Hydrocarbon에 오염된 토양의 용매추출조건에 관한 연구

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국문요약

본 실험은 석유의 주요성분인 benzene, toluene, p.m.o-xylene, ethylbenzene, n-propylbenzene, 1.2.4-trimethylbenzene, n-butylbenzene 등 light hydrocarbon에 오염된 토양의 회복에 필요한 최적 용매추출 조건을 밝히기 위하여 실시하였다. 토양의 수분함량이 증가할수록 추출효율은 감소하였으며, Methanol을 용매로 사용하였을때에 BTEX화합물은 1시간 추출시 최고 추출효율에 도단하였으며 고분자 hydrocarbon은 4시간이상 추출시 최고 추출효율에 도단하였다. 2-Propanol을 추출용매로 사용할때는 모든 light hydrocarbon은 추출 4시간 경과시 최고의 추출효율을 나타냈다. 추출용매와 토양의 비율이 2:1일 때 가장 경제적이고 효율적으로 추출이 이루어 졌으며, 0.4mg/g이상의 토양오염 농도에서 최적의 추출효과가 나타났다. 위의 최적조건으로 light hydrocarbon 오염토양을 batch extraction을 이용하여 추출하였을 때 평균 66%의 light hydrocarbon의 회수되었으며, 회수율은 토양의 종류, 추출용대의 종류 그리고 hydrocarbon의 종류에 따라 좌우되었다. 본 연구의 결과는 석유에 오염된 토양의 회복를 위한 토양세척에 사용될 수 있을 것으로 사료된다.

Keywords: BTX, Hydrocarbon, Soil contamination. Soil remediation, Soil washing

I. Introduction

Spills, leaks, or subsurface disposal of industrial solvents and petroleum products is known to contaminate soils and groundwater with organic compounds including aromatic, chlorinated, and polycyclic aromatic hydrocarbons. Since the 1940s, it has been a common practice to bury fuel storage tanks underground to guard against fire hazards and explosions. As these underground gasoline storage tanks near the end of their effective lifetime, leakage of hydrocarbons into soils is becoming a common problem. A portion of these spills remains trapped in the soil, immobilized by capillarity and adsorption. This frac-

tion can present a long-term threat to groundwater, as relatively cleaner recharge water may desorb or solubilize gasoline components. Remediation requires a reliable and rapid means of evaluating the total gasoline content of contaminated soils. Recent tests of public water supplies revealed that among the most frequently observed contaminants were benzene, toluene, and the xylene isomers (BTX). These light hydrocarbons are common constituents of fuels and solvents, and through the processing, handling, and storage of these products, BTX have contaminated soils and groundwater. As major components of unleaded gasoline, BTX have a ubiquitous presence throughout much of the world. Environmental re-

gulations have mandated the reduction of these compounds within soil and groundwater systems. Knowledge of the fate of these compounds in soils and aquifers is critical importance to the design of effective monitoring and remediation processes.³⁰

The soil washing process technology appears to be a viable alternative for treating some contaminated soils and sludges. As the technology continues to be applied more frequently to treat certain waste types, the various solvent extraction systems will be refined. It seems certain the technology will grow to meet the needs of the market." Water-based soil washing has been demonstrated to be ineffective in removing tar from contaminated soils.51 Other technologies, such as supercritical fluid extraction have been used in bench-scale designs to demonstrate effective removal of herbicides from soil metrices.⁶⁷ Recently, an accelerated solvent extraction system has been developed by other investigtors? for the rapid and efficient removal of organic contaminants from soils. As the technology continues to be applied more frequently to treat certain waste types, the various solvent extractionsystems will be refined. It seems certain the technology will grow to meet the needs of the market." Soil washing has several advantages over thermal treatment. Some benefits arise because it is a recovery process rather than a destructive process, and solvent extraction can often recover valuable products and reclaim some soils. Soil washing may be easier to permit, may be economically feasible for treating a wider range of hazardous wastes, and can be considerably less costly with respect to energy requirements." The other benefits often cited for soil washing is the lower energy requirements compared with, for example, distillation, which often can be used to accomplish a similar purpose. For soil wahsing, as an isolated unit operation, the energy requirements are generally limited to electricity for running feed and solvent pumps, stirrers, or column agitators. Materials requirements are essentially limited to makeup for solvent losses, since in most instances, solvent will be recovered for reuse from both the extract and the raffinate. In general, soil washing processes are most attractive relative to alternatives when the contaminant to be removed from the feed has significant material value. No Soil washing can be conducted on a large scale at costs substantially lower than other treatment methods. Typical cleaning efficiencies for soil washing ranged from 75 to 95 percent removal, depending on the type of contaminant. Atalay and Hwang in investigated that higher molecular weight compounds were recovered at greater than 80% whereas the recoveries of BTEX were generally less than 50% using methanol extraction.

Soil washing using organic solvents could be used to clean soil contaminated with high concentrations of nonvolatile hydrophobic organics. The choice of suitable solvent depends primarily on chemical structures of the contaminant, solvent extractive capacity, soil type and equilibrium characteristics. In addition to these, the solvent should be stable and must have favorable density, viscosity, and interfacial tension properties. In general, the ideal solvent for the task should not be volatile at low temperatures; should be pure, noncorrosive, nonviscous, nonfoaming, nontoxic and nonflammable; should have infinite solubility for the solute; and should be inexpensive. 111 The soil washing technology has not vet been fully developed for hazardous waste treatment applications. Dalev^{1,0} commented that soil washing has not been widely used, however, because it often cannot reach treatment quality goals, it may produce a great deal of wastewater or waste solvent, and the washing solution may carry away too much solid material. Contamination of soils from fuel spills, such as gasoline, aviation fuel, and diesel fuel are common environmental problems. Therefore, soil washing is a rapid, efficient, and environmentally friendly for remediation of contaminated sites.

II. Materials and Methods

1. Soil Preparation and Analysis

In choosing a soil for the extraction tests, the applicability of the procedure to actual field situations was a primary consideration. Soil selec-

Table	1.	Physical	and	chemical	characteristics	of	soils
used in the study.							

Parameter -	Soil Material				
rarameter	Α	В	С		
% Soil moisture	1.9	13.6	19.4		
pΗ	7.6	8.2	7.4		
4 Organic matter	0.58	1.84	5.04		
CEC.	8.34	19.33	8.40		
Inorganic separates(%)					
Sand	95.8	73.2	13.1		
Silt	1.0	14.6	57.4		
Clay	3.2	12.2	29.5		
Texture"	Sand	Sandy loam	Silty clay loam		

[&]quot;CEC: Cation exchange capacity (in meq./100 g)

tion included identification of native soils at three places near Norman in Oklahoma. Three different soils(sand, sandy loam, silty clay loam) were used in batch experiments. The soils were air-dried for 1 week and passed through a 1 mm sieve. In case of silty clay loam soil, a grinder was used to break up large clods. Soil properties such as moisture, pH, organic matter content, cation exchange capacity(CEC), and soil texture were determined by soil testing in the laboratory following the Method of Soil Analysis. The results of the soil analysis are shown in Table 1. There soils showed different moisture content, organic matter content, CEC value, and inorganic texture.

2. Soil Contamination Procedure

Nine light hydrocarbons at concentration of 400 mg/kg were used in this experiment. The compounds were benzene, toluene, o-xylene, m-xylene, p-xylene, ethylbenzene, n-propylbenzene, 1, 3.5-trimethylbenzene, and n-butylbenzene. These compounds are typical representatives for C6-C10 hydrocarbons commonly found in gasoline mixtures. They were selected, in part, because each could be resolved from the mixture by gas chromatography. A 500 mg of hydrocarbon standard chemicals were dissolved to a 25 ml volumetric flask using methanol. The concentration of the in-

dividual hydrocarbon was 20 mg/ml (grand mixture of hydrocarbon). For each experiment, 10 g of air-dry soil was weighed into glass vials. For analysis of wet soils, 0.48, 1.36, and 1.94 g of water were added to the dry soil of sand, sandy loam and silty clay loam soil respectively (4.8%, 13.6%, 19.4% moisture by weight on wet-weight basis). The soil was then injected at various depths with a total 0.2 ml grand mixture of hydrocarbon per vial. The vials were sealed with Teflon-lined screw caps and shaken for 2 min to mix completely. Soil was extracted by the solvents after 20 hr spiking.

3. Development of Soil Washing Condition

1) Extraction Solvent Selection

The choice of suitable solvent depends primarily on chemical structures of the contaminant, solvent extractive capacity, soil type and equilibrium characteristics. Methanol. 2-propanol, and water were selected for the following critical characteristics: adequate water solubility, low clay particle dispersion, good oil dispersion, and adequate biodegradability.

2) Effect of Moisture Content

The moisture content of the soil is a parameter that should be considered for good extraction efficiency. In this experiment, soil from 0 to 20 percent (maximum moisture content in a field) moisture level were chosen for a silty clay loam soil. A 10 g air-dry soil was weighed in glass vials and then 0, 0.5, 1.0, 1.5, and 2.0 ml of water were added to the dry silty clay loam soil (0, 5%, 10%, 15% and 20% moisture by weight on wetweight basis). The hydrocarbon mixture was injected at various depths in the vial containing the soil. The vials were sealed with Teflon-lined screw caps and shaken 2 min to mix completely. The vials were then allowed to equilibrate for 20 hr. Then the soil was extracted by the solvents.

3) Effect of Extraction Time

Ten grams of the hydrocarbon contaminated soil were mixed with 20 ml of the appropriate extraction solvent and shaken for 0.5, 1, 2, 4, 8, and 24 hr. The sample was then centrifuged and the supernatant was decanted. It was analyzed for its hydrocarbon content using a gas chromatograph.

⁶⁶Based on the soil texture classification system (U.S. Department of Agriculture)

4) Effect of Solvent Amount

For the purpose of this investigation, a 1 to 10 ratio (solvent/soil) was chosen. A 10 g air-dry sandy loam soil sample was weighed in glass vials and 1.36 ml of water was added. A total of 0.2 ml of hydrocarbon mixture was injected at various depths in the vial containing the soil. The vials were sealed with Teflon-lined screw caps and shaken 2 min to mix completely. To each vial either 10, 20, 40 or 100 ml of solvent were added and then shaken for 4 hr. The sample was then centrifuged and the supernatant was decanted. It was analyzed for its hydrocarbon content using a gas chromatograph.

5) Effect of Contaminant Concentration

An experiment was conducted to determine the effect of soil contamination level on extraction recovery. A range of 0.1 to 1.6 mg/g soil was used. To do this, 10 g air-dry sandy loam soil was weighed into glass vials, and 1.36 ml of water was added. At various depths in the vial, either 0.05, 0.1, 0.2, 0.4, or 0.8 ml of a hydrocarbon contaminant mixture was added. The vials were sealed with Teflon-lined screw caps and shaken for 2 min to mix completely. The soil was extracted by the solvents after 20 h equilibration.

4. Batch Tests.

Ten grams of hydrocarbon contaminated soil was mixed with 20 ml of either methanol, 2-propanol or water and shaken for 4 h. It was then centrifuged and the supernatant was decanted. This was analyzed for its hydrocarbon content using a gas chromatograph. When water was used for extraction, the extracted soil and surfactant solution were centrifuged and the combined filtrate placed in a separatory funnel containing 20 ml methylene chloride. The mixture was shaken for 3 min the phases were allowed to separate, and the aqueous phase was discarded. Methylene chloride layer was passed through a Na₂SO₄ column. These steps were repeated 3 times to obtain a final methylene chloride volume of 50 ml. A $1 \mu l$ volume of methylene chloride extract was injected in the gas chromatography. The concentration of each component was determined via an internal standard procedure.

5. Analysis of Light Hydrocarbons.

Varian gas chromatograph 3330 equipped with flame ionization detector, and 60 m×0.75 mm ID Supelco VOCAL fused-silica capillary column was used for analysis of light hydrocarbons. Mixture containing light hydrocarbons was prepared by adding 500 mg of each chemicals to 25 ml volumetric flask with optima grade methanol. All light hydrocarbon standard reagents were obtained from Chem Service, Inc., 99+% pure. A.a, a-trifluorotoluene is used for internal standard for quantitative analysis.

III. Results and discussion

The recovery of light hydrocarbons from soils using extraction methods was evaluated. With the extraction method, it is desirable to study the influence of specific factors on the analytical procedure. In this study a number of factors were evaluated including soil moisture content, extraction time, contaminant concentration, solvent amount, soil types, and solvent types. Typical chromatograms of standard hydrocarbon mixture and hydrocarbons extracted from contaminated soil samples are presented in Fig. 1.

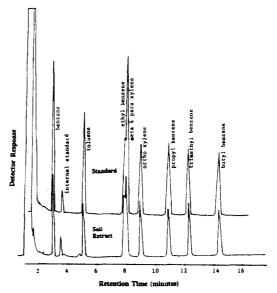
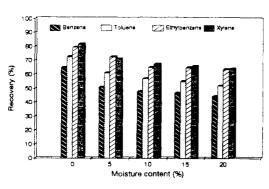


Fig. 1. Gas chromatogram of a standard hydrocarbon mixture and an extract from a contaminated soil

1. Effect of Moisture Content

It is apparent that soil moisture is a critical parameter for assessing good sample recovery. Increasing the moisture content resulted in a decreased recovery; this is demonstrated in Fig. 2. The best overall recovery was obtained when samples were extracted using dry soil. Sample moisture content may also affect analytic recovery due to variation in hydration of solids. When these highly hydrated soils are subjected to the extraction with solvents, the soil particles tend to flocculate, causing entrapment and reducing the extraction of organic matter. Higher recoveries of all individual hydrocarbons were obtained when the soil was dry rather than wet. It has been demonstrated that expansion and contraction of clays due to changes in moisture may affect sample recovery. 10 In this study the difference in recovery was proportional to the level of moisture in the soil. Benzene, toluene, ethylbenzene, and xylene (BTEX) compounds showed large variation in percent recovery with moisture content compared with the semivolatile com-



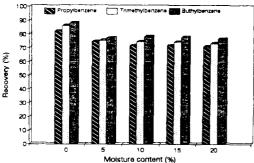


Fig. 2. Effect of moisture content on percent recovery of individual hydrocarbons

pounds. The mean percent recovery of BTEX compound was decreased about 20%, while the semivolatile compounds was decreased 10% following with increasing the moisture content. Previous work has demonstrated that water solubilities of organic pollutants are the most important physical properties controlling the transport and fate of the chemicals in soil systems.¹⁵⁰ For instance, the hydrophobicity of humic acid, the pH of the pore water, and soil/aquifer organic carbon content were three parameters that can significantly influenced the effectiveness of a humic acid solution in a decontamination scheme.11 An attempting to convey water as a chemical or biological treatment media to fuels is limited both by a low hydraulic permeability and channeling through larger pore spaces. Even in larger grained sandy soils fuel blobs can become trapped in small pores causing water to channel through only the large, unblocked pore spaces. Because soil washing is best suited for permeable soils, a sandy soil site is desirable. 160 However, environmental contamination knows no boundaries or site selection.

2. Effect of Extraction Time

This part of the study was undertaken to determine the time required to reach equilibrium using for batch extraction. Theoretically, this should indicate complete extraction of the contaminant from the soil. Two solvents: methanol and 2-propanol were used as solvents. The results are presented in Fig. 3 and 4. Batch extraction with methanol showed that percent recovery of BTEX compounds decreased after 1 hour, while recovery of semivolatile compounds increased until 8 hours. Batch extraction using 2-propanol showed that individual hydrocarbon recovery was increased until 4 hours. Using methanol as an extraction solvent, maximum recovery of BTEX components was reached quicker than when using 2-propanol. However, as the BTEX components quickly dissolved from the soil material, the extra extraction time did not improve the percent recovery. It should be noted that the total time of 8 hours should have no significant ef-

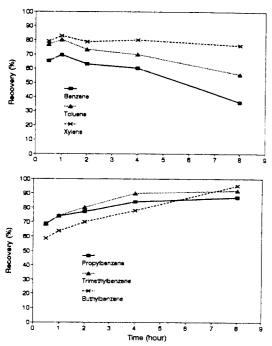


Fig. 3. Effect of extracation time on percent recovery of individual hydrocarbons after methanol extraction

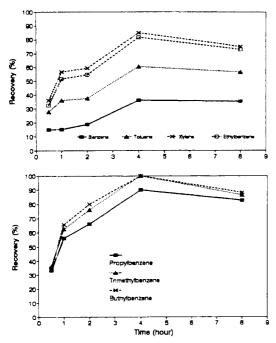


Fig. 4. Effect of extracation time on percent recovery of individual hydrocarbons after 2-propanol extraction

fect on light hydrocarbon recovery, since the 4 hours extraction period was sufficient to yield maximum recovery.

3. Effect of Solvent Quantity

Extractability of individual hydrocarbons from soils as a function of solvent quantity was evaluated. For the purpose of this investigation, a range of solvent/soil ratio from 1:1 to 10:1 was chosen as a compromise to provide more than the indicated minimum solvent for high efficiency. Increasing solvent volume resulted in a decreased component recovery, this is demonstrated in Fig. 5. When the solvent/soil ratio was 1, some semivolatile compounds, such as propylbenzene and trimethyl-benzene, showed maximum percent recovery. However, the percent recovery of benzene was very low. When the ratio was 10:1, the percent recovery of hydrocarbons decreased sharply regardless of the type of component. Percent recovery was significantly better for all components when the solvent/soil was 2:1.

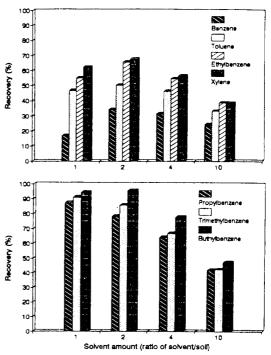


Fig. 5. Effect of solvent amount on percent recovery of individual hydrocarbons

4. Effect of Contaminant Concentration

Another evaluation of the soil washing techniques was done by comparing recovery of light hydrocarbons with different levels of soil contamination. To accomplish this, an experiment was conducted to determine the effect of different levels of soil contamination level on per-

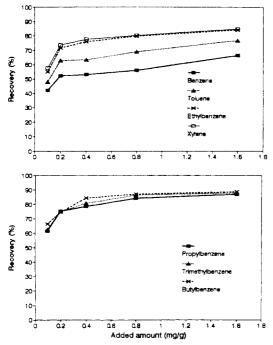


Fig. 6. Effect of contaminant added to soil on percent recovery of hydrocarbons

cent recovery. A range of contaminant level from 0.1 to 1.6 mg/g was chosen. Fig. 6 indicates that the soil is sufficiently contaminated when 0.4 mg/g of the hydrocarbon was added. Addition of contaminant greater than 0.4 mg/g did not show significant improvement on the percent recovery. Fig. 6 also shows that percent recovery values for each component were excellent, indicating that concentration, at least within the narrow range used here, was not a limiting factor in obtaining quantitative recovery. The BTEX compounds were more persistent on the soil than the semivolatile compounds at a given rate of contaminant addition. The percent recovery was greater by 20% for BTEX compounds than for semivolatiles when compared at higher contaminant concentration. No significant increase in semivolatile hydrocarbon recovery resulted greater than 0.4 mg/g contaminant addition.

5. Batch Extraction

Extraction of light hydrocarbons from three soils was studied using a batch process to assess the recoverability of these components. Three solvents: water, methanol, and 2-propanol were compared for their extractive potentials. The results were complex in that several interactions were found between soils and solvents. The data obtained is tabulated in Table 2. The data is similar in scope in that they compare the effect of soil types and solvents on recovery. It is ap-

Table 2. Percent recovery of individual hydrocarbons from different soils using three solvents for batch extraction.

		Soil type							
Components	Sand			Sandy loam			Silty clay loam		
	Water	MeOH	2-PrOH ⁵	Water	МеОН	2-PrOH	Water	MeOH	2-PrOH
Benzene	4.12	55.18	47.41	3.02	34.35	35.25	1.12	44.18	9.46
Toluene	2.34	62.84	63.24	1.75	50.26	53.33	0.57	52.01	31.24
Ethylbenzene	1.12	71.37	73.91	0.18	65.79	67.48	0.27	63.32	51.68
p+m-Xylene	1.03	72.72	66.69	1.21	67.71	67.15	0.23	64.09	58.55
o-Xylene	1.38	68.76	71.97	1.20	71.04	68.60	0.37	67.33	61.43
n-Propyl-benzene	0.32	71.74	75.48	0.33	78.47	72.15	0.06	71.23	70.84
1,2,4-Tri-methylbenzene	0.58	74.13	78.50	0.58	86.04	75.87	0.15	73.33	75.46
n-Butyl-benzene	0.14	80.29	84.50	0.55	95.70	80.77		76.63	83.82
Average	1.38	69,63	70.21	1.10	68.67	65.08	0.35	64.02	55.31

[&]quot;Methanol."2-Propanol

parent that soil type had an effect on the extent of recovery and this effect was pronounced when the soil was sandy. For light hydrocarbons, methanol and 2-propanol consistently resulted in higher extraction efficiency than water. This is due to the fact that the solubility of hydrocarbons is higher in alcohol than in water. Methanol gave superior results for contaminants removing from silty clay loam. There was a significant interaction between soils and solvents, indicating that some solvents worked better in one soil while others worked better in others. Recoveries of individual hydrocarbons varied depending on the type of soil being extracted and the solvent being used. The extraction efficiency of water varied from 4 to 0 percent depending on the soil types and contamination. Benzene was the dominant component that was removed from the soil by water. Silt and clay provide much more surface area per unit mass than sand. In addition to a higher external surface area, some of the silty and clay soils used in this experiment were sandy and porous, providing additional characteristics for adsorbing the volatile organic compounds. Soils high in clay will be particularly difficult to treat in that special provision is required to prevent agglomeration. It is also important to note that soil organic matter has some controlling influence over the removal of hydrocarbons, whereas other soil properties such as, percent clay and bulk density, do not.179 Expansion and contraction of clays due to changes in moisture may also affect recovery.110 As seen in Table 2, in general, the silty clay loam soil gave lower recoveries for both individual and average hydrocarbons compared with the other soils. This was probably due to the presence of high organic matter and moisture. Methanol gave higher percent recoveries than did 2-propanol, except in the sandy soil. This data suggests that variability in extractability of components is apparent when several vastly different extraction solvents are evaluated individually. The low organic matter content of the soil correlated with low absorbance of the pollutants spiked into the soil. Thus, the removal of contaminants from such a matrix may be expected to be relatively easy compared to a soil with a high organic matter content. In the silty clay loam, use of methanol recovered more benzene, toluene, ethylbenzene and xylene while 2-propanol was more efficient in extracting trimethylbenzene and buthylbenzene. Therefore, on the basis of the recovery results discussed above, methanol would be expected to recover greater total monoaromatic hydrocarbons than the 2-propanol. It should also be noted that methanol was used in these extraction in order to improve the recovery of the higher BTEX components.

IV. Summary

This study was conducted to establish an ideal condition for obtaining maximum extraction efficiencies using an array of soil types and under a wide variety of conditions. Nine characteristic compounds were studied; benzene, toluene, ethylbenzene, o-xylene, m-xylene, p-xylene, n-propylbenzene, 1,2,4-trimethylbenzene, and n-butylbenzene which were found in gasoline. Increasing the moisture content resulted in decreased recovery, and recovery of hydrocarbons from wet soils was significantly lower than from dry soils. For the batch extraction process, 4 hours of extraction time was sufficient to give optimum recovery of the contaminants. With methanol as an extraction solvent, maximum recovery time appeared to be reached quicker for BTEX components than with 2-propanol. The 2 to 1 ratio of solvent/soil was chosen as a compromise to provide for the indicated minimum solvent use and high extraction efficiency. The 0.4 mg/g soil contamination was adequate to show quantitative recovery. The percent recovery of BTEX was concentration dependent more than the semivolatile compounds. Methanol and 2-propanol consistently gave higher efficiency than water. Methanol was superior to 2-propanol in removing contaminants from silty clay loam soil. Using the most efficient extraction procedure, the average recovery of the light hydrocarbons from the three soils was 66 percent. Recoveries were also dependent on soil type, solvent type, extraction time, solvent amount, contaminant concentration, and compounds volatility. This study provided a useful screening technique for procedures that can be used to remediate soils contaminated with light hydrocarbons.

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