

REMEDICATION OF ORGANICS-CONTAMINATED SANDY SOIL BY *IN-SITU* WASHING

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Abstract

A new method of in situ soil remediation called in situ washing by sedimentation (IWS) was introduced, by injecting a high air-pressure into a mixture of saturated water-sandy soil and hydraulically separating the soil particles based on their particle size and density. This physical separation exploits the distribution of contaminant in the soil by physically separating a selected contaminant-rich fraction. The effect of soil-water ratio and diameter geometry of the column on the effectiveness of separation by IWS was investigated. Soil-water ratio 1:2 (v/v) was found to be optimum for particle segregation produced by IWS, however, generally the diameter geometry column was not effect on the particle separation. The suitability of IWS for organics remediation was investigated by batch column experiment. The laboratory experiment was effective to produce a distinct size separation of the contaminated soil into the coarse and fine fractions, as well as the wash water, and indicating that a significant reduction in organics contaminant level (90%) may be achieved.

Keywords: *In situ* washing, Sedimentation method, Soil-water ratio, PAH contamination

Introduction

Several techniques have been developed for soil remediation. Basically, they can be divided in the these categories, *ex situ* methods where the contaminated soil is removed and treated away from the site that has to be cleaned up and *in situ* methods, where the contaminated soil is cleaned on site.

There are some options in each of these methods. *Ex situ* methods include incineration, extraction, *ex situ* washing, and *in situ* methods include soil vapor extraction, soil flushing, bioremediation and phytoremediation. The advantage of *ex situ* methods is that it takes relatively little time to remove the contaminated soil. The soil is excavated, treated or replaced and then filled in again. By comparison with in situ methods, however, method of ex situ remediation has many disadvantages. They are expensive, large quantities of soil have to be transported often in residential areas, there are risks to buildings and other structures especially with major excavations, pollutants can be released during excavation, and it causes major disruption of daily life in the area to be cleaned.

Nowadays, in situ techniques are used for sustainable remediation of contaminated sites [1]. Air and water extraction have in practice proved to be reliable methods for various types of soil remediation [2,3,4]. Although bioremediation and phytoremediation was also used, this technology works slowly to be fully effective as a remediation technique [5,6].

Soil washing was conventionally performed *ex situ* in treatment plants that employ extracting chemical to remove contaminant from soil into aqueous solution [7,8,9,10]. Few studies of *in situ* soil washing have been conducted, even though *in situ* soil washing could be suitable for certain contaminated soil in the field [11,12,13].

In this paper, a new method of *in situ* soil treatment called *in situ* washing by sedimentation (IWS) was proposed, by injecting a high air-pressure into a mixture of water-sandy soil column and hydraulically separating the soil particles based on their particle size and density, as shown in Figure 1 [14,15,16,17]. This physical segregation exploits the distribution of contaminant in the soil by physically separating a selected contaminant-rich fraction. Several researches indicate that the finest parts of soil are particularly active in the sorption processes of organic as well as inorganic contaminant [18,19,20,21]. For the *in situ* application, the physical segregation by IWS and on-site wash water treatment happen as an integrated process and it is important to isolate the site to protect the leakage of the aqueous solution used (Figure 1). The advantage of IWS was that the washing and segregation processes take place simultaneously during the remediation process, quick, effective and cheap since there are no costs for excavation of contaminated soil from the site. The fine fraction is recovered for further treatment or disposal. The wash water is completely collected, treated and recycled.

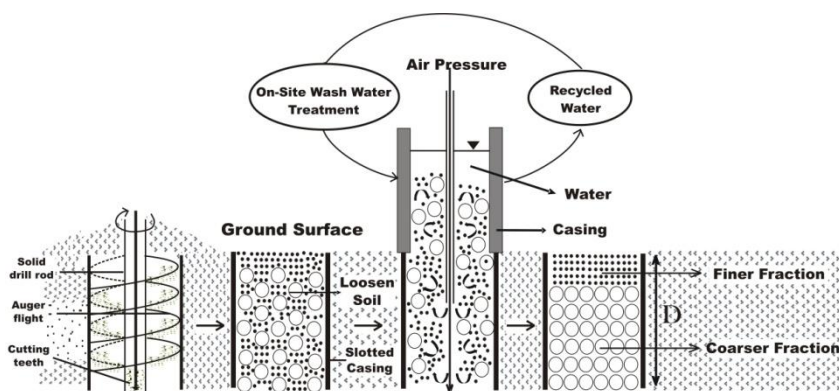


Figure 1. In situ washing by sedimentation method (IWS) [16]

In our earlier laboratory work on simulation of IWS, simple batch laboratory scale experiment was conducted on metal-contaminated soil, such as Cu, Pb, Zn and Cd indicated that a very high reduction in Cu, Pb, Zn and Cd level (up to 90%) may be achieved consistently over a wide range of initial Cu, Pb, Zn and Cd concentrations (200-4000 mg/kg) [16].

On the other hand, it is important to consider organics contaminant as a major source on soil contamination instead of heavy metals. Frostner (1989) demonstrated that heavy metal and polycyclic aromatic hydrocarbons (PAHs) show parallel evolution pattern with heavy metals and it is concluded that both contaminants could originate from a common source [22]. PAHs as representative of organic contaminant, which contain more than two benzene rings, are refractory organic compounds commonly produced by incomplete combustion of fossil fuels [23]. Remediation processes for PAH-contaminated soil have been studied extensively over the last two decades, most of them being bioremediation processes based on microbial degradation [24,25,26]. Unfortunately, however, these processes are not only time-consuming, but are of limited value.

Theoretical Background of Sedimentation

A conceptual model developed on IWS was based on sedimentation process. Typically, the settling and sedimentation behavior of particulate suspension was assumed to be governed by Stokes' Law. This based on early work conducted by Terzaghi (1925) through series of simple column sedimentation test [27]. For the most part, dilute slurries with low solid concentration settle according to Stokes' Law, resulting in the segregation of coarse and fine particles.

However, Stoke's equation is valid only for dilute suspension where the effect of neighboring particles on the movement of particle under consideration can be neglected, i.e., for discrete particle settling. At a high sediment concentration, the interfering force of adjacent particles has a pronounced effect on the settling velocity to establish relationship between sedimentation velocity and fluid volumetric concentration for suspended solid particles in water.

Related to sediment concentration or the soil-water ratio, several works has shown that as the solid concentration of a particulate suspension increase, the resulting sedimentation rates occur at a slower rate and do not follow Stokes' Law [28,29,30,31]. Kynch (1952) determined that the settling velocity of a particulate suspension was dependent on the suspension solid concentration and termed this behavior hindered settling [28]. Hindered settling occurs when the suspension concentration increases, causing interference between particles, which affect the settling velocity. An important characteristic of hindered settling was determined by McRoberts and Nixon (1976), in which suspension with varying size of particles could be deposited without significant segregation under hindered condition [29]. Segregation is minimized under hindered settling condition as the suspension settles at approximately the same velocity.

In IWS, when the air pressures become sufficiently high to counteract the gravitational pull on the soil-water column, the soil particles effectively float, or suspended as hindered settling. One promising method to enhance the efficiency of the washing processes under consideration was based on creation of a suspended soil for accomplishing the interaction of the liquid-gaseous medium with the ground solid materials. Air pressure velocity will be effective when the upward velocity of air pressure through a porous solid mass reaches a condition whereby the frictional pressure drop across the solid becomes equal to the buoyant weight per unit area of the solid.

The main advantage of IWS is to produce vertical column sedimentation which separates contaminated soil particle into coarse and fine fraction as well as wash water. For practical purposes, the two soil fractions are considered in this study. We used the terminology "fine" and "coarse" particles as the results of segregation of particles by the difference in settling velocity. The top layer was assumed to be finer fraction (silts and clays) and the bottom layer was assumed to be coarser fraction (fine-coarse sands and gravels) (Figure 1).

Objective of the Study

The objective described in this paper was two folds. Firstly, our earlier work on the simple-batch laboratory-column experiment of IWS [16] was extended by a systematic investigation on the evaluation of the effect of soil-water ratio and diameter geometry of column on the effectiveness of particle segregation by IWS in laboratory scale. Secondly, our second goal was to observe the removal of organics-contaminated sandy soil by IWS in batch laboratory experiment.

Soil Sample Characteristic

Experiment were conducted on the uncontaminated soil were collected in 1.5 m depths. The result of grain-size distribution indicated that the original soil sample contained

approximately 10-20% clay-silt size particles and the remaining was sand (sandy soil). The main properties measured in this uncontaminated soil were pH, particle size distribution, the organic content, the cation exchange capacity, the specific surface area and the density. Selected properties of this soil are given in Table 1.

Table 1. Properties of Soil Sample

Properties	Value
pH	6.65
Effective Diameter, d_{10} (mm) ¹⁾	0.063
Median Diameter, d_{50} (mm) ¹⁾	0.24
Uniformity Coefficient d_{60}/d_{10} ¹⁾	4.25
Mineralogical Composition ²⁾	Quartz, Feldspar, Albite, Kaolinite, Illite, Chlorite
Clay Mineral Composition in Fine Fraction ^{2) 6)}	Kaolinite, Illite, Chlorite
Cation Exchange Capacity (meq/100g) ³⁾	6.8
Carbon Content (%) ⁴⁾	2.81
Coarse Fraction ⁶⁾	0.30
Fine Fraction ⁶⁾	3.31
Specific Surface Area (m ² /g) ⁵⁾	8.007
Coarse Fraction ⁶⁾	4.964
Fine Fraction ⁶⁾	27.831
Density (g/cm ³)	2.710
Coarse Fraction ⁶⁾	2.768
Fine Fraction ⁶⁾	2.650

¹⁾ Analyzed by using sieving and hydrometer test

²⁾ Analyzed by using X-ray Diffraction

³⁾ Analyzed by using BaCl₂.2H₂O compulsive exchange method

⁴⁾ Analyzed by using Total Organic Carbon (TOC) Analyzer

⁵⁾ Analyzed by using Brunauer, Emmitt and Teller (BET) method

⁶⁾ Fraction after separated by 90 mm sedimentation column (see later description)

The Effect of Soil-water Ratio and Geometry Diameter of Column on the Effectiveness of Particle Segregation by IWS

The Effect of Soil-Water Ratio

The objective of this experiment was to evaluate the effect of a soil-water ratio on the effectiveness of particle segregation by IWS. Environmental and economic concern required that the volume of water solution used on IWS in order to obtain sufficient particle segregation should be kept to a minimum. Generally, one of the main drawbacks of the washing method on soil remediation is the vast consumption of water required to make up the washing solution for the removal of the contaminants that have been retained in the contaminated soil. In IWS, we propose for washing solution which must be subsequently be on-site treated before it can be re-use. A series of laboratory experiment were carrying out to optimize the soil-water ratio for sufficient particle segregation.

Experiment was conducted in the cylinder tube of 2000 ml in volume with 90 mm inside diameter. For constant mass of 810 g dry soil, different volumes of water solution were used and were described in Table 2. The value of 300 ml volume of dry soil sample was divided by 2.710 g/cm³ measured density (see Table 1).

After the soil sample and the water solution was prepared into the cylinder tube, the column then was stirred for one minute and let sedimentation occur for 30 minutes. Theoretically, the soil particle in the column will settle in a descending order of particle sizes with the top part of the soil layer consisting of smaller particle.

The segregation of the soil particle into nominal size fraction in this experiment depends on the sedimentation process on hindered settling in the high sediment concentration.

Table 2. The Water Solution and the Dry Sample Soil Used

No. Tube	Soil Sample Gram (milliliter)	Volume of Water (ml)	Soil-Water Ratio (v/v)
1	810 (300)	300	1:1.00
2	810 (300)	400	1:1.33
3	810 (300)	500	1:1.67
4	810 (300)	600	1:2.00
5	810 (300)	700	1:2.33
6	810 (300)	800	1:2.67

The next step, all the cylinder tubes containing sedimentation soil column was kept in a refrigerator for -18⁰ C temperature, in order to obtain an undisturbed frozen soil column sample. After 24 hour, the frozen soil column samples then were taken out and marked. The purpose and the advantage of this freezing were to obtain a selected frozen column soil sample accurately by slicing the soil column without disturbing the sedimentation column. These undisturbed frozen soil column samples then were cut into several certain thicknesses and were analyzed for particle size distribution (Figure 2).

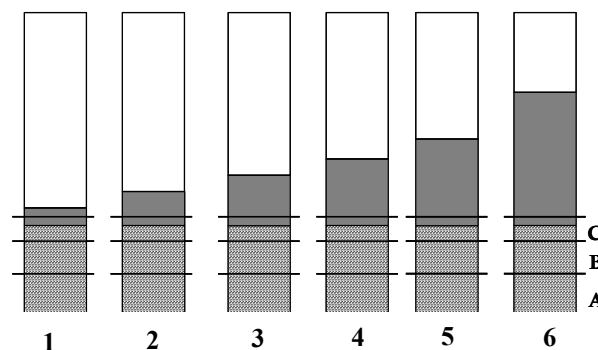


Figure 2. An illustration of undisturbed frozen soil column (not to scale or actual size)

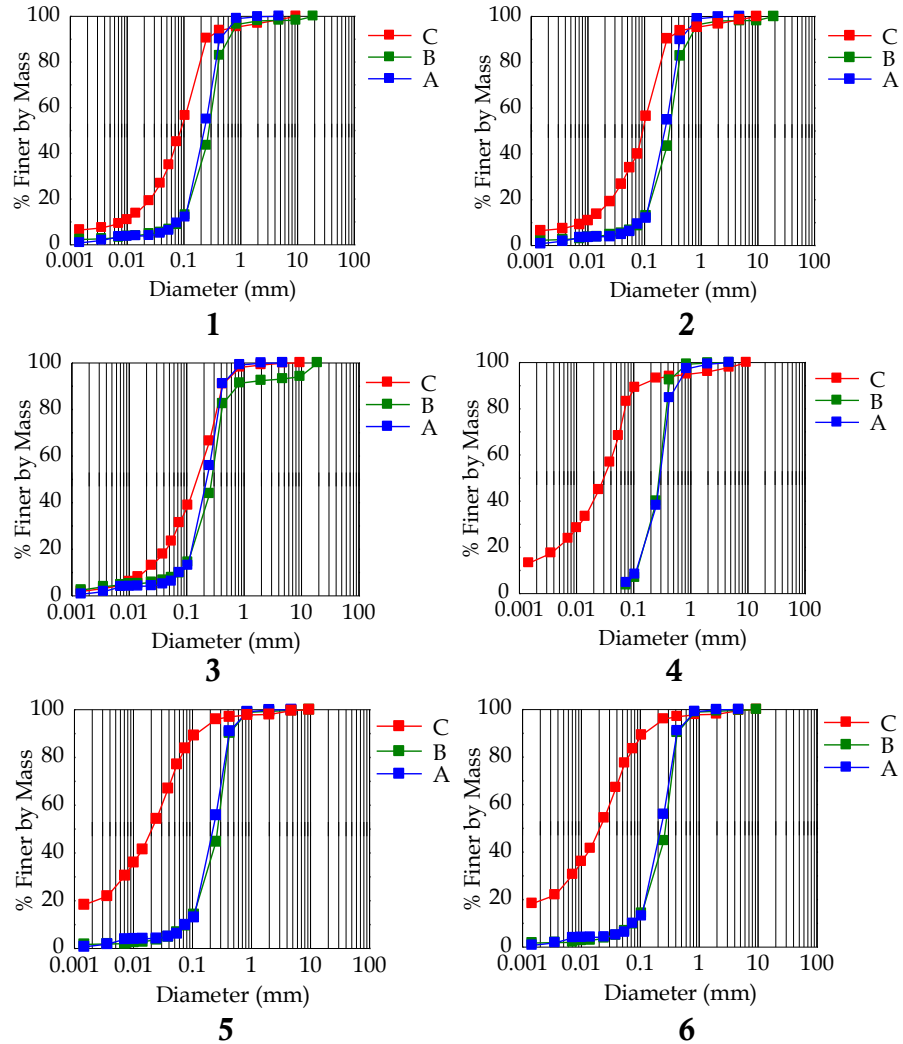


Figure 3. Particle size distribution analysis of selected frozen sedimentation column in each tube

The experiment was continued by observing more accurately the two soil fractions considered in this study. The terminology “fine” and “coarse” particles was used as the results of particle segregation by IWS. As previously stated, the top layer was assumed to be fine fraction and the bottom layer was assumed to be coarse fraction assumed reflects the high and low content contaminant in each fraction. Similar to previous experiment which conducted in the cylinder tube with 90 mm inside diameter, the optimized the soil-water ratio 1:2 (v/v) consist of 810 g dry soil and 600 ml volumes of water solution were used.

After sedimentation column was created, the top layer consisting of fine fraction was sampled very carefully by using small spoon, after the wash water was removed by suctioning. The fine fraction was then determined for particle size distribution analysis. Corresponding to the fine fraction, the remaining coarse fraction in the bottom part of the sedimentation column was also determined for particle size distribution analysis. The result of particle size distribution analysis was shown in Table 3. The fine fraction separated in the study was labeled as Clay and the coarse

fraction was labeled as Fine Sand. These two fractions were then dried and weighed. The result can be seen in Table 3. The percentage of saturated volume was obtained by measured the height of each fraction in the sedimentation column.

In this stage, these two fractions separated (coarse and fine) was also determined for several parameter as described in Table 1. It shows that the fine fractions have the high specific surface area, the high organic content, and containing of 1:2 clay minerals such as illite. It can be understood that the fine fraction separated by our sedimentation column particularly active in the sorption processes of contaminant.

Table 3. Particle Size Distribution Analysis of Fine and Coarse Fraction

Fraction Observed (Size Diameter)	Fine	Coarse
Gravel 2-75 mm %	0.0	0.9
Sand 0.075-2 mm %	1.0	43.8
Fine Sand 0.075 mm %	6.9	48.5
Silt 0.005-0.075 mm %	37.0	5.1
Clay <0.005 mm %	55.1	1.6
Uniformity Diameter (mm)	-	2.59
	Clay	Fine Sand
Percentage Dry Weight	4.5	94.5
Percentage Saturated Volume	10	90

The Effect Diameter Geometry of Column

Following the previous experiment, by using the optimized soil-water ratio 1:2 (v/v), the effect of diameter geometry of sedimentation column on the effectiveness of particle segregation by IWS was investigated. The diameter of the borehole, as sedimentation column, was critical in determining the volume of each column, the number of borehole to be drilled and the relationship of the sedimentation column to each other.

Four different tubes of varying diameter were used in this experiment. In detail, the volume of the water solution and the weight of the soil sample used in this experiment were described in Table 4.

Similar to the previous experiment, after creating the sedimentation column in each cylinder tube and were kept in a refrigerator, the undisturbed frozen soil column samples then was taken out, marked and sliced into several certain thickness, and in each selected thickness was analyzed for particle size distribution (Figure 5). The result of the particle size distribution analysis of a selected frozen sedimentation column in the each cylinder tube is shown in Figure 6.

Table 4. The Water Solution and the Dry Soil Sample

No. Tube	Inside Diameter (mm)	Soil Sample Gram (milliliter)	Volume of Water (ml)	Soil Water Ratio (v/v)
1	30	270 (100)	200	1:2.00
2	60	405 (150)	300	1:2.00
3	70	540 (200)	400	1:2.00
4	90	810 (300)	600	1:2.00

By using the optimized soil-water ratio 1:2 (v/v), it showed that there was no significant difference on the particle segregation by observing the presence of fine particle in the top layer of the frozen sedimentation column of each different diameter geometry tube (section C). Generally, it can be concluded that the diameter geometry column does not need to be considered for the effectiveness of particle segregation by IWS.

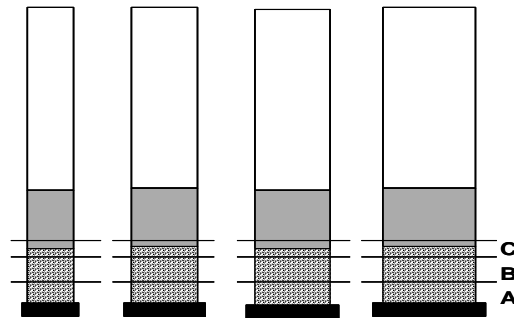


Figure 5. An illustration of undisturbed frozen soil column sample (not to scale or actual size)

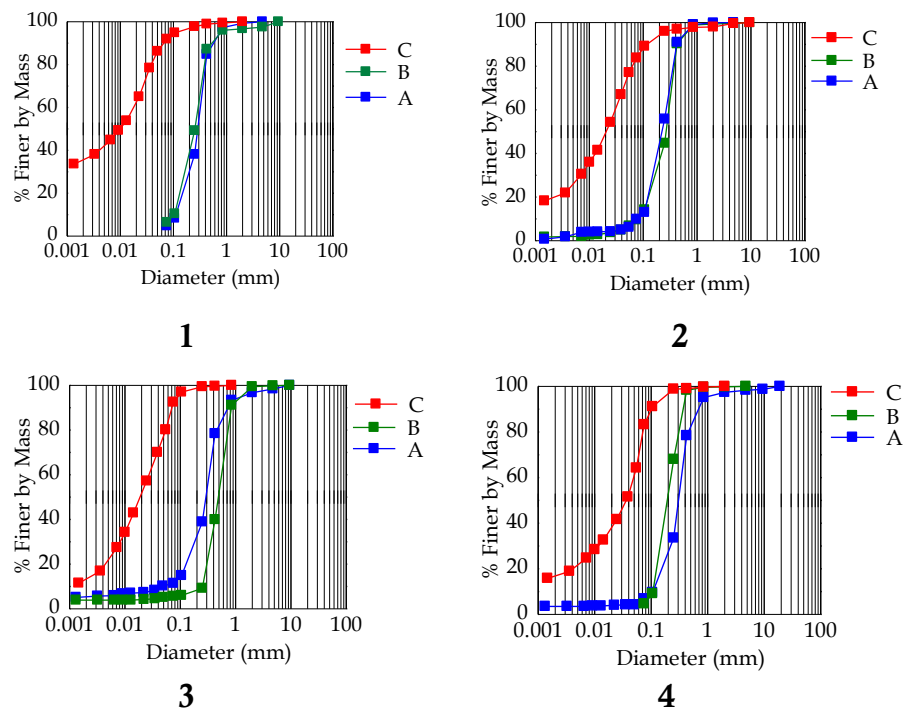


Figure 6. Particle size distribution analysis of selected frozen sedimentation column in each tube

Removal of the Organics Contaminant on the Sandy Soil by IWS

Batch Sedimentation Column Experiment

Naphthalene (NAP), phenanthrene (PHE) and pyrene (PYR) were selected as example of PAHs representing organics contaminant. Two artificial contaminated soils were prepared by dissolving uncontaminated soil which collected from Ota District Tokyo (see Table 1 for detail) with an

appropriate quantity of NAP, PHE and PYR solution as described by Sawada, et al., 2004 [32]. Briefly, uncontaminated soil sample was spiked with solution of NAP, PHE and PYR for three days to allow the dispersion and sorption of the contaminant in the soil matrix. (A) Soil with low concentration of PAH; (B) Soil with high concentration of PAH, by spiking uncontaminated soil sample with 500 mg/kg of NAP, PHE and PYR solution for soil A and 1000 mg/kg for soil B. All samples then were determined for PAH concentration after ethanol digestion by using a Gas Chromatography Mass Spectrometry (GC-MS) under optimized operating condition. The resulting of the artificial contaminated soils had a final concentration of 30, 75 and 50 mg/kg of NAP, PHE and PYR on Soil A and 250, 490 and 350 mg/kg of NAP, PHE and PYR on Soils B.

Experiment was conducted by using air pressure created by air pump injected into the soil-water column on 90 mm internal diameter cylinder tube (Figure 7). A 0.5 kg dry PAH-contaminated soil sample was used and 370ml water was added based on the optimized soil-water ratio 1:2 (v/v) obtained from previous experiment. Seven similar tubes were constructed and the air pressure was introduced into each tube for 1, 2, 3, 4, 5, 10 and 15 minutes to observe the effect of washing duration.

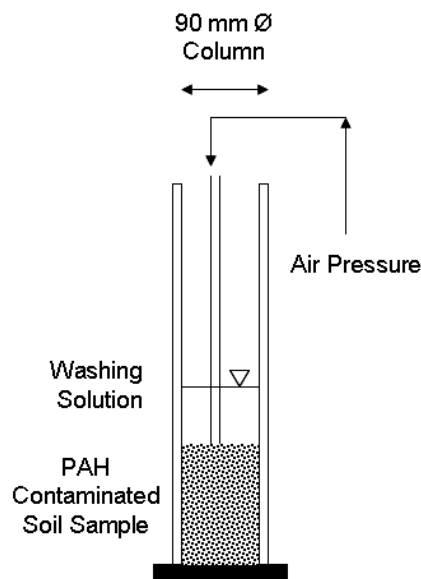


Figure 7. Laboratory experimental setup

The air flow started with the position of the pipe reaching the surface of the soil sample inside the tube. The air pressure rate was increased as the inlet pipe penetrated the soil sample. After the final depth was reached, air flow rate was kept constantly until the inlet pipe almost reached the bottom of the cylindrical tubes.

The soil inside the tube was allowed to settle to obtain clear water above the settled solids. The coarse fractions separated in this batch sedimentation experiment were analyzed for their particle size distribution. The wash water and the fine fraction were sampled through pipe by suctioning and the coarse fraction was sampled using a small spoon. All samples then were determined for PAH concentration by using a Gas Chromatography Mass Spectrometry (GC-MS) under optimized operating condition. The wash water was determined after filtered by using 0.45 μ m filter.

The segregation of coarse and fine fractions produced by IWS was investigated as a

function of the washing duration, obtained by introducing air pressure into each tube for 1, 2, 3, 4, 5, 10 and 15 minutes. The result shown in Table 4 indicated that the result of segregation in our batch sedimentation experiment was reliable, and the accuracy of segregation increase depending on the washing duration. After 5 minutes washing, 92.2% of particles in the coarse fraction were separated as >0.075mm particles size diameter (fine to coarse sand) and fine fraction separated as <0.075mm particles size diameter (clay-silt). After 5 to 15 minutes, it seems that no significant difference occurred in the results. Perfect segregation was expected in this method, but it showed that only about 90% of its grains were separated.

Table 5. Values Obtained by Particles Size Analysis of Coarse Fraction Separated by IWS

Sample (observed fraction)	Washing Duration	Sand % (0.850 mm-0.250 mm)	Fine Sand % (0.250 mm-0.075 mm)	Silt % (0.075 mm-0.005 mm)	Clay % (<0.005 mm)
Original	0	42.1	33.7	12.8	11.4
Coarse	1	44.7	47.6	5.0	2.6
Coarse	2	44.8	47.5	5.0	2.5
Coarse	3	43.5	49.1	4.9	2.3
Coarse	4	44.8	48.2	4.7	2.1
Coarse	5	44.2	49.7	4.0	2.0
Coarse	10	44.8	48.2	4.7	2.1
Coarse	15	44.2	49.7	4.0	2.0

The inaccurate result is attributed by the lack homogeneity in the particle size distribution of the coarse fraction. The sedimentation process is more complicated if several particles are present and the system becomes a sediment suspension in hindered settling reflected when the concentration of the suspension decrease, the homogeneity of the separated fraction will increase and therefore impossible to exclude small amounts of finer particles. Although the result of segregation by IWS was not perfect, the first important point of this study was that the process succeeded to separate the soil sample into a coarse fraction and a fine fraction. The segregation into coarse and fine fraction will affect the high and low concentration of the contaminant in each fraction.

Removal of PAHs in Contaminated Soil

As shown in the result of the previous experiment, the accuracy of particle segregation was influenced by the washing duration, and consequently it will affect the percentage removal of contaminant. In this experiment, the coarse fraction sampled was a representation of a clean fraction.

Figure 8 shows the experimental data by using the de-ionized (DI) water and DI water with addition of biosurfactant as washing solution. In the case of DI water washing, the result show that the removal process rapidly reaches equilibrium, at approximately 10 minutes for each PAH; after this period no considerable changes in the removal rates were observed. The PAH in the contaminated soil sample was mostly must found in the wash water and the fine fraction.

The fine fraction in the seven tubes was collected, combined and analyzed for PAH

concentration. The results of the GC-MS analysis on the fine fraction showed that the concentrations of the PAH were very high. The accumulations of PAH in the fine fractions are attributed to the high specific surface area, the presence of clay minerals such as illite, and the high organic content (see Table 1).

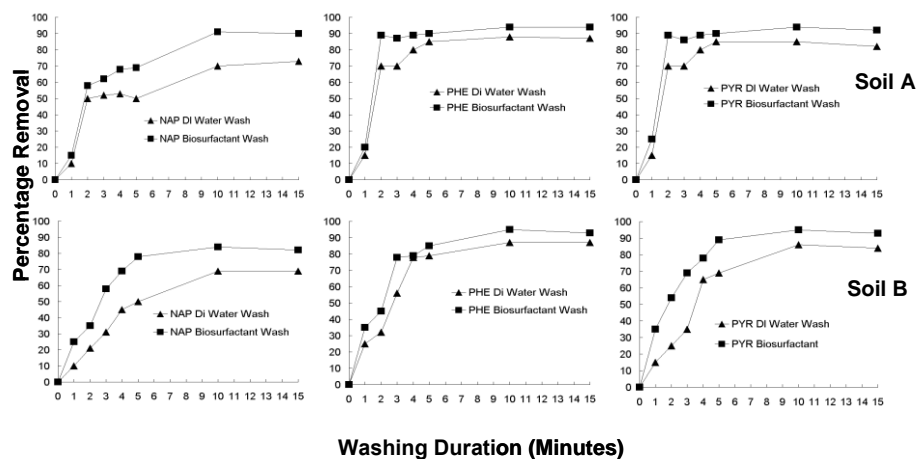


Figure 8. Percentage removal PAH vs washing duration for soil A and B

The addition of biosurfactant (saponin) in order to enhance the percentage removal was also observed, by adding 0.25% by weight of saponin in the washing solution (Figure 8). In this stage, after fine fraction was removed, the wash water used was returned to the cylinder tube and saponin was added. The air pressure was then introduced into each tube for 1, 2, 3, 4, 5, 10 and 15 minutes to observe the effect of washing duration, the same as in the previous experiment. As shown in Figure 9, the removal percentage increased after the addition of saponin. The addition of saponin as an anionic biosurfactant was effective to assist in the solubilisation, dispersal and desorption of PAH from the contaminated soil fraction [33].

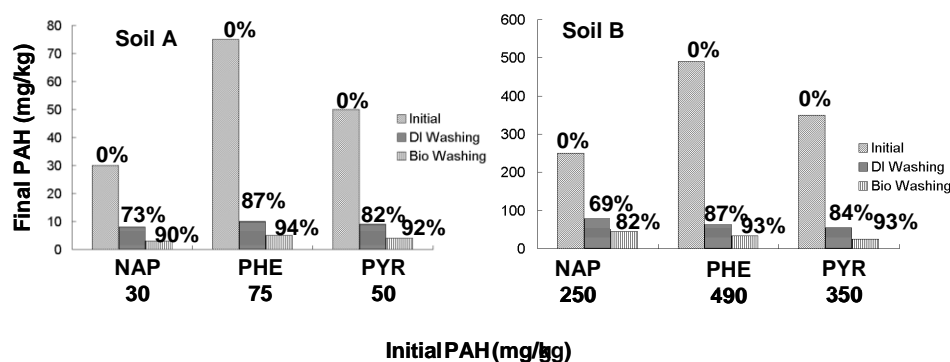


Figure 9. Removal efficiency of IWS on Soil A and Soil B

The result of the removal efficiency of PAH from soil A and B is shown in Figure 9. It shows that by using DI water only, approximately 80% of PAH retained in the soil sample was removed. The addition of saponin has proven to be effective to enhance the removal efficiency up to more than 90%.

Fractionation Efficiency

To be effective on removing PAH, it is imperative that clean fractionation of the coarse, fine particles and the wash water be obtained. Figures 10 and 11 illustrate the result of fractionation efficiency after 15 minutes washing with DI water and continued by 15 minutes washing with DI water with the addition of biosurfactant to the soil sample obtained from the Ota District area, Tokyo.

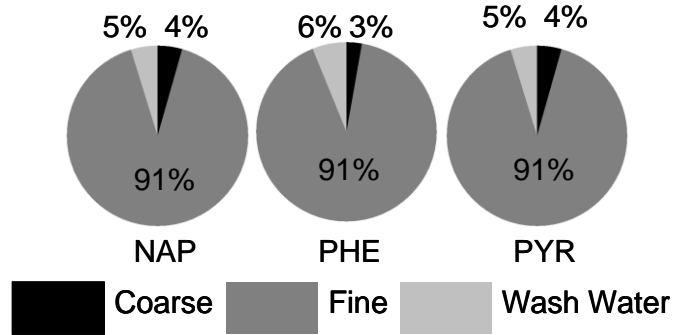


Figure 10. Distribution PAH among various soil fraction and wash water in Soil A

The effectiveness of IWS was indicated by the washing result for coarser fraction containing <5% of NAP, PHE and PYR. On the other hand, >90% of the original PAH contamination in soil sample was associated with fine particles and transferred into the wash water.

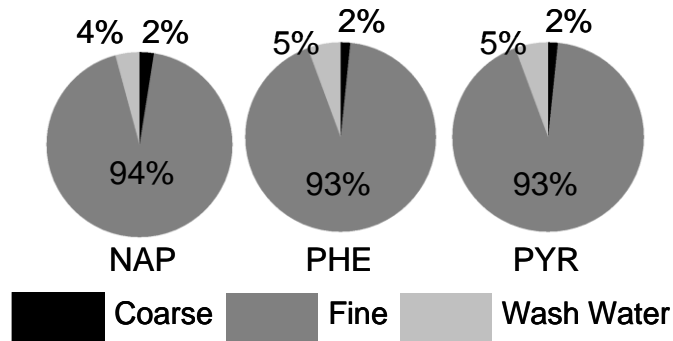


Figure 11. Distribution PAH among various soil fraction and wash water in Soil B

Generally, the washed coarse fraction contained a small amount of the initial PAH contamination while the fine particles contained higher amount. The coarse particles resulting from the washing process may be returned to the site without any treatment when applicable remediation objective are met.

Conclusions

This study has addressed the *in situ* soil remediation with an emphasis on the washing and segregation of soil particles by IWS. By using Ota District sandy soil as an object of this experiment, the following initial conclusions can be drawn from the results in this study:

1. The results of laboratory study on the effect of soil-water ratio on the effectiveness of particle segregation by IWS show that by using soil-water ratio 1:2 (v/v), the optimum for particle segregation on IWS was found. However, the result of laboratory study on the effect of diameter geometry of column on the effectiveness of particle segregation shows that generally the diameter geometry column will not affect the soil particle segregation.
2. The laboratory scale of *in situ* washing apparatus on IWS was able to produce a distinct size separation of the soil into coarse and fine and a significant reduction of Polycyclic Aromatic Hydrocarbon (PAH) such as Naphthalene, Phenanthrene and Pyrene level (90%) was achieved.
3. The concentration of PAH contaminant was found to be a function of particle size; the coarse fraction were the cleanest and the fine fraction contained the highest PAH contaminant and a very small amount of the original contaminant was retained in the coarse fraction.
4. The removal efficiencies of remediation method proposed in this study depend on initial PAH concentration, the addition of biosurfactant in the washing solution and the duration of washing. Further treatment for the fine fractions and the wash water containing suspended solid particle and dissolve PAH contaminant need to be further investigated.

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