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Ultrasound to decontaminate heavy metals in dredged sediments

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Abstract

Sediments contaminated with heavy metals due to past disposal practices threaten the environment and require remediation. This study was an attempt to develop a technology to decontaminate heavy metals in dredged sediments using ultrasound coupled with vacuum pressure. A set of laboratory scale experiments were conducted using dredged sediments obtained from New York/New Jersey harbor. This sediment sample is considered as category III, a material that failed to meet USEPA requirements for toxicity or bioaccumulation, and required secure disposal. Acoustic cavitation due to ultrasound energy coupled with vacuum pressure was used to facilitate the removal of chromium (the selected metal contaminant) from the sediments. Full factorial experimental designs were performed to evaluate the above treatment technique and to optimize the processes. Two coupled processes were used to separate and to treat both coarse (Process #1) and fine (Process #2) fractions of sediments. Selected variables for evaluation of Process #1 were ultrasound power, soil-to-water ratio, vacuum pressure and dwell time, and for Process #2 were ultrasound power, soil-to-water ratio and dwell time. Laboratory scale experiments were carried out with various combinations of these parameters according to the factorial design. The optimum removal was found to be 92% with the parameter levels at 1200 W power, 1:15 soil-to-water ratio, 15 psi vacuum pressure and 15 min of dwell time. After the application of Process #2 for fine sediments it was found that the fines were separated into silt and clay. Only the silt faction had a considerable metal removal while the clay fraction was insensitive to the treatment. A maximum removal of 83% was obtained for silt fraction when factor levels were at 1200 W power, 1:50 soil-to-water ratio and 90 min of dwell time. Further analysis of clay fraction showed that the chromium in clay is immobile and stable. The toxicity characteristic leaching procedure (TCLP) test on clay fraction confirmed that the treated clay fraction is safe to dispose. The study showed that the proposed treatment technique is effective and economical for sediments with lower clay contents. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Dredged sediments; Chromium; Ultrasound; Decontamination; Experiments

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1. Introduction

Harbors and waterways are routinely dredged to ensure safe navigable waters. Many waterways are located in or close to industrial and urban areas, hence sediments from surface runoffs are often contaminated with organic and inorganic contaminants. The existing disposal techniques for dredged sediments have concentrated on ocean disposal, artificial islands or disposal into lagoons for either containment or for long-term land reclamation, where the contamination levels of dredged sediments determined the end use. Ocean disposal of contaminated sediments has generated concern, as such operations may adversely affect water quality and aquatic organisms. The difficulty in finding suitable on land disposal sites for contaminated dredged sediment has contributed to partial stoppage of dredging activities at some ports. The resulting economic impacts to the regions surrounding these ports have been severe. Heavy metal contamination is also a common problem at many hazardous waste sites. Once in the soil matrix, heavy metals like chromium are absorbed making remediation difficult. Unlike many organic pollutants that can be eliminated or reduced by chemical oxidation techniques or microbial activity, heavy metals will not degrade. Heavy metals adversely affect human health and, can cause variety of ailments including brain/neurological changes, liver and kidney damage and cancer.

Two basic strategies have been adopted for the remediation of heavy metal-contaminated soils [1], namely extraction/separation of the contaminant metals (e.g. soil washing, soil flushing [2–4]) and minimization of migration potential of the contaminant metals (e.g. so-lidification, stabilization, vitrification [5]). There are a few other methods also exist to treat metal-contaminated soils other methods [6,7]. Of these two strategies, immobilization methods are more often accepted for remediation in the United States. Despite the preference for immobilization methods for remediation, a significant advantage of extraction/separation methods over immobilization methods is that contaminated soil may be reduced to below target regulatory limits and the contaminated soil may be rendered reusable for construction, fill, and other purposes. Sediment dredging and handling in these technologies represents a cumbersome and costly endeavor. Reuse of the contaminated sediments or deep sea dumping after decontamination will be the ideal solution for disposal of dredged sediments. This research is an attempt to use ultrasound to decontaminate dredged sediment, which can be ultimately developed as an in situ technology eliminating the cumbersome and costly operations in material handling in the existing methodologies.

Ultrasound is widely used in industry for a variety of applications such as in medical imaging, cleaning metal parts, removing oxide films, oil, grease and other contaminants from solid surfaces and plastic welding, and even to removing contaminants from soils. Ultrasound refers to sound waves having frequencies in the range 16 kHz to 500 MHz, in-audible to the human ear, and can be transmitted through any medium with elastic properties, including water, gas-saturated water and aqueous particle suspensions. It is transmitted in the form of mechanical energy.

Ultrasonic cleaning works by providing shear forces to remove the material adhering to a surface. This shear force is developed due to cavitation. Ultrasound causes high-energy acoustic cavitation: the formation, growth and implosive collapse of bubbles in a liquid. That is the formation of microscopic vapor bubbles in the low-pressure (rarefied) part of the ultrasonic wave. These bubbles collapse in the compression part of the wave creating



Fig. 1. A flow-chart for the technology developed at NJIT.

very minute, but high-energy movements of the solvent that results in localized high shear forces. During cavitational collapse, intense heating of the bubble occur. These localized hot spots have temperatures of roughly 5000°C, pressures of 500 atm, and a lifetime of a few microseconds (Suslick [8]). Shock waves from cavitation in liquid–solid slurries produce high-velocity inter-particle collisions, the impact of which is sufficient to melt most metals. Applications to chemical reactions exist in both liquids (homogeneous) and in liquid–solid (heterogeneous) systems. Of special synthetic use is the ability of ultrasound to create clean, highly reactive surfaces on metals. Ultrasound has also found important uses for initiation or enhancement of catalytic reactions, in both homogeneous and heterogeneous systems. These "cavities" or areas of low pressure provide a sink of low concentration or partial pressure of the contaminant into which adsorbed material will desorb. Also, cavitation is the most important factor for determining dispersion and de-aggregation of soil in ultrasonic system (Watson [9]).

The ultrasound coupled with vacuum pressure was used in this research as an integrated multi-step or multi-process technology to decontaminate the contaminated sediments. Fig. 1 shows a flow-chart of this integrated process technology consisting of three integrated steps or processes. In Process #1, ultrasound is applied to the contaminated dredged sediments to treat coarse grains, while vacuum or sub-atmospheric pressure is applied to remove fines and contaminated water from the dredged sediments. Then in Process #2, ultrasound is applied again to decontaminate the fines and sub-atmospheric pressure is applied to separate water with contaminants. In this research Process #3, to separate contaminants from water was not evaluated.

Water content (%)	225	
Loss on ignition (%)	14	
Clay content (%)	26	
Silt content (%)	45	
pH	8.0	
Total organic carbon (%)	7.5	
Pesticides (µg/kg)	~ 400	
PCB (total) (µg/kg)	$\sim \! 4000$	
Dioxins (µg/kg)	\sim 5000	
Furans (µg/kg)	$\sim \! 15000$	
PAHs (µg/kg)	$\sim \! 100000$	
Chromium (mg/kg)	~ 370	
Lead (mg/kg)	~ 600	

Table 1Properties of the dredged sediments

This paper describes a laboratory study of application of ultrasound in Processes #1 and #2 to decontaminate heavy metals (chromium) in dredged sediments. Chromium is selected to be the representative metal contaminant since it is one of the hardest metals to decontaminate. The use of chromium to explore the applicability of the technology gives an idea of the possibility of removal of other metal contaminants which are comparatively easy to remove. The dredged sediments from New York/New Jersey harbor were used in this demonstration study and chromium is selected as the metal contaminant for the quantitative evaluation of the treatment process. Table 1 provides the properties of dredged sediments used in this research.

2. Experimental design

Bench mark tests were carried out and found that the following process variables could influence the extraction of heavy metals from the sediments: ultrasound power, soil-to-water ratio, dwell time or ultrasound application time, pH of the suspension, suspension temperature, ultrasonic frequency, particle size distribution, probe insertion depth and vacuum pressure.

A probe type ultrasound source was used in this research. It is a 1500 W power probe operating at 20 kHz. Since there are no commercially available ultrasound sources at reasonable costs with the same power rating, but with different frequencies, frequency was not considered as a factor in this research. Previous study by Meegoda et al. [10,11] and Rajaratnam [12] reported that the pH of the suspension does not contribute significantly to the removal efficiency in comparison to the other factors. The disadvantage of the probe type is that the temperature of the system cannot be held constant during the experiment. It was also noted that the increase in temperature due to ultrasound was proportional to the dwell time. With the probe type ultrasound source, temperature could not be controlled, hence it was decided to run the experiments at room temperature without temperature control. Raine et al. [13] reported that with decrease in particle size there must be an increase the number of particles, which will contribute to higher cavitation. However, since the range of

particle sizes considered in this research when compared with the wavelength of ultrasound is small, this effect — the particle size distribution — will not significantly affect the energy dispersion in comparison to the other factors. Therefore, particle size was not considered as a process variable in this study. Morra et al. [14] observed that increase in energy depends on the depth of insertion, the width of the probe and the dimensions of the container. Based on that study, it was decided to keep the probe insertion depth a constant for all the experiments. Hence, by the process of elimination the critical factors that influence the process were considered to be ultrasound power, soil-to-water ratio, dwell time and vacuum pressure.

Many experiments were conducted varying the above factors to collect unbiased data that can be analyzed to obtain a valid and meaningful data for interpretation and conclusions. It was decided to perform a full factorial design for both Processes #1 and #2. Experimental procedure that was adopted in this research is shown in Fig. 2.

The four factors power, soil-to-water ratio, dwell time and vacuum pressure were chosen to be the design variables for Process #1. A series of experiments were performed with each set having one varying factor while keeping the others constant, to obtain range of probable values for the each process variable in the experimental design. It was found that beyond 15 min of ultrasound treatment, there was no significant increase in the metal removal efficiency. Hence, the three levels for the dwell time were selected as 5, 10 and 15 min. The initial test data also showed that it is reasonable and economical to have power levels of 40, 60 and 80%. Similarly, three levels of vacuum pressure were chosen as 5, 10 and 15 psi, while soil-to-water ratios were taken as 1:5, 1:10 and 1:15. Hence, four factors at three levels produced 81 combinations for a full factorial experimental design (Montgomery [15]).

Similarly for Process #2 preliminary tests that were conducted to investigate the optimum range of values to be used for the experimental design. Test results showed that vacuum pressure should be kept at the highest level (15 psi) to obtain sufficient wastewater flow rates.



Fig. 2. Flow-chart of the experimental approach.

Based on the preliminary experiments the three levels of power were chosen as 40, 60 and 80%, and three levels of soil-to-water ratios were selected as 1:10, 1:25 and 1:50 and three dwell times of 30, 60 and 90 min. Three factors at three levels produced 27 combinations for a full factorial experimental design (Montgomery [15]).

3. Experimental procedure

3.1. Sample preparation

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Sample preparation began with the wet raw sediments obtained form NY/NJ harbor. These sediments were highly contaminated with organic compounds. Higher organic concentration is expected to give background errors in flame ionization atomic absorption spectrometry (AAS), which was used for the chromium analysis. Therefore, to have a better control over chromium detection it was decided to eliminate the organics from the sediment before treating with ultrasound. This was achieved by keeping the sample in batches, in an oven at a temperature of 600°C for 36 h. Then the sample was manually crushed to homogenize, and to avoid breaking coarse particles. Dried homogenized sediments were used as the chromium contaminated sediments to evaluate Process #1.

3.2. Process #1

This process was designed to separate fines from the bulk-dredged suspension and remove the fines using sub-atmospheric pressure while treating the coarse particles. Fig. 3 shows the experimental apparatus specially designed to implement Process #1. Laboratory model was built as a cylindrical container separated into two parts with a US #200 mesh, which was used to separate the fines from coarse. The outlet of the model was connected to a vacuum supply



Fig. 3. Laboratory apparatus and set-up for Process #1.

via a sedimentation tank, which was used to collect fines and water. The ultrasound probe was dipped into the sample slurry. The total volume of sediment slurry was kept constant (500 ml) during the experiments. To find the initial chromium concentration, the prepared sediment sample was sieved using a US #200 sieve and the retained fraction, which was considered as the coarse fraction was analyzed to determine the chromium concentration. The slurry was made with the measured amount of soil knowing the soil-to-water ratio and keeping the total volume constant at 500 ml. The slurry was placed inside the container as shown in Fig. 3, and ultrasound energy with the required power setting and desired vacuum pressure were applied for given experimental condition selected from the experimental design. Water was added as needed to maintain the constant volume at 500 ml. At the end of application of ultrasound, retained coarse fraction was oven dried and measured to find the separating efficiency, and was analyzed to determine the final chromium concentration. After oven drying the separated fines were used as the feed material to evaluate Process #2.

Chromium analysis was performed by acid digestion of soil using the microwave method followed by atomic absorption spectrometry analysis. This method (EPA method SW 846-0351) provides for acid extraction of soil. The Microwave digestion system was used for metal extraction from soil. The extracted sample was then used in atomic absorption spectrometry (AAS) for chromium concentration, which is the widely used method for determination of single elements in analytical samples. This method is based on EPA SW 846-7190.

3.3. Process #2

The objective of this process was to decontaminate fines and to separate the contaminated water using a sub-atmospheric pressure. Fig. 4 gives the laboratory set-up for Process #2. A laboratory batch test module in the shape of a box was made of five porous stones as the sample container, where the ultrasound probe was dipped, and has an outer box to apply vacuum pressure to the porous box. The specially selected porous stones were used to separate the water from fines. The outlet from the outer box was connected to a retention tank to collect wastewater, which was in turn connected to the vacuum system. Collected



Fig. 4. Laboratory apparatus and set-up for Process #2.

fine sediments from Process #1 were used as the feed material to evaluate Process #2. These sediments were tested for the chromium concentration after Process #1. This concentration was considered as the initial concentration for the Process #2. The total volume of sediment slurry was maintained constant (1000 ml) during each experiment. The slurry was made with the measured amount of soil, each time according to the soil-to-water ratio, with 1000 ml as the total volume. The slurry was placed in the batch device and ultrasound energy with the required power setting and the maximum vacuum pressure was applied for a required time period selected from the experimental design. Water was added as needed to maintain the constant volume. At the end of the experiment, the retained slurry was oven dried and the final chromium concentration was determined. The chromium analysis for the separated fine fractions was performed using the procedure used for Process #1.

For Process #2, additional experiments were conducted to explore the possibility of enhancing the treatment efficiency. They are as follows:

With pH variation: Two experiments were conducted by adjusting the pH of slurry to 2 and 12. The pH of the original slurry was 8. Sodium hydroxide (NaOH) was added to make the slurry to increase the pH to 12 and nitric acid was added to lower the pH to 2. Both these samples were run with 50% power, 1:50 soil-to-water ratio and ultrasound energy applied for 60 min. The experimental procedure and the analysis were the same as the other experiments evaluated Process #2.

With chelating agents: Ethylene-diamine-tetra-acetic acid (EDTA) was used as the chelating agent. Two kinds of EDTA — with sodium and without sodium — were used in two separate experiments. The power output was 50%, soil-to-water ratio was 1:50 and the ultrasound was applied for 60 min. The experiments were carried out in the similar manner as in experimental design for Process #2 including the determination of chromium concentrations.

The environmental scanning electron microscope (ESEM) was used to observe treated sediment samples from Process #2. In ESEMs, presence of water vapor or gas molecules amplifies the electron signal and reduces the charging problem that occurs in the high vacuum SEMs. The ESEM was integrated with energy dispersive X-ray (EDX) detector. The EDX unit provided image analysis and the elemental compositions (from carbon to uranium) of treated samples using standard quantitative techniques.

Finally, the EPA toxicity characteristic leaching procedure (TCLP) test (EPA method 1311) was performed for the clay fraction after the treatments, to evaluate the possibility of land disposal of the clay fraction after treatment from Process #2.

4. Analysis of experimental results

From the particle size analysis it was found that 28.9% retained on US sieve #200. Hence, the separation efficiency was determined from Eq. (1).

Separation efficiency (%) =
$$\frac{[W_{\rm f} - (W - W_{\rm c})]}{W_{\rm f}} \times 100$$
 (1)

where W_f = weight of the fines in the sample before the treatment; W = weight of the sediments retained on the #200 mesh in the model, after treatment; W_c = weight of coarse fraction in the sample before the treatment.



Fig. 5. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 5 psi pressure and 5 min sonication time.

The removal efficiency was determined from the Eq. (2):

$$Removal efficiency (\%) = \frac{(Cr concentration before treatment - Cr concentration after treatment)}{Cr concentration before the treatment} \times 100$$
(2)

4.1. Process #1

It was found from the experimental data that extremely high separation efficiencies close to 100% were obtained for all the samples. The results of the 81 experiments with all different combinations of the parameters were shown in Figs. 5–13. Each figure shows nine tests with the variation of power and soil-to-water ratio for a specified vacuum pressure and dwell time. In general, it can be seen in all the figures that, for any given set of experiments with the increase of power, the removal efficiencies increased except for soil-to-water ratio of 1:5. Also the removal efficiencies increased with the increase of power and soil-to-water ratio removal efficiency increases except when the power and the soil-to-water ratio were ratio were ratio removal efficiency increases except when the power and the soil-to-water ratio were



Fig. 6. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 10 psi pressure and 5 min sonication time.



Fig. 7. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 15 psi pressure and 5 min sonication time.



Fig. 8. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 5 psi pressure and 10 min sonication time.

at lowest levels. Figs. 5-13 show the variation of chromium removal efficiencies with the vacuum pressure at fix dwell times. It is seen that in general, the removal efficiency is increased with the increasing vacuum pressure. In figure groups (5, 8, 11), (6, 9, 12) and (7, 10, 13) shows the variation of the removal efficiency with the dwell time at fixed vacuum pressures. It can be seen from those figures that chromium removal efficiencies have



Fig. 9. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 10 psi pressure and 10 min sonication time.



Fig. 10. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 15 psi pressure and 10 min sonication time.



Fig. 11. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 5 psi pressure and 15 min sonication time.

significantly increased with the increasing dwell time. However, the above trends are not applicable for the experiments with the lowest soil-to-water ratio or the lowest power. This could be due to the lack of water circulation and therefore improper mixing of sediments during the experiment. Mixing of sediments with water enhances the transfer of metals from sediments to water. Soil-to-water ratio and power influences the water circulation. It is seen



Fig. 12. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 10 psi pressure and 15 min sonication time.



Fig. 13. Process #1: removal efficiency of chromium for different power and water-to-soil ratio, at 15 psi pressure and 15 min sonication time.

that all the parameters have greatly influenced the removal efficiency. According to the data shown dwell time is the most influential parameter for removal efficiency. Soil-to-water ratio has a considerable influence on removal efficiency too.

The highest removal efficiency (92.2%) was obtained when all the parameters were at their highest levels. It was seen that even with power at its mid level (60%) and all the other parameters at their highest level obtained a removal efficiency of 89%, a value close to the maximum removal efficiency.

4.2. Process #2

It was found that Process #2 separated fine fraction of the bulk sediment into silt (>2 μ m) and clay (<2 μ m). For better interpretation of data final chromium concentration results of both silt and clay were determined. The separation efficiency was 100% for all the Process #2 experiments. The experimental results of chromium removal in the silt fraction are plotted in Figs. 14–16. It can be seen that the removal efficiencies of silt fraction were very low when all the factors are at the lowest levels. It is seen that in all three figures that soil-to-water ratio is the most influential parameter to the removal efficiency. It reached a maximum value of 82% when all the factors were at their highest levels. Also its seen that



Fig. 14. Process #2: removal efficiency of chromium for different power and water-to-soil ratio at 30 min sonication time.



Fig. 15. Process #2: removal efficiency of chromium for different power and water-to-soil ratio at 60 min sonication time.



Fig. 16. Process #2: removal efficiency of chromium for different power and water-to-soil ratio at 90 min sonication time.

power and time are not significantly sensitive to the chromium removal efficiency (Figs. 15 and 16). Though reasonable removal efficiencies were obtained for silt fraction, the removal efficiencies for the clay fraction were very low.

Further investigation of the possibility of removing the chromium in clay fraction produced the following results. Table 2 shows the results obtained from the pH variation. It is seen that there is no significant improvement to the removal efficiency in the clay fraction. Table 3 shows the variation of the removal efficiencies with the chelating agent (EDTA) with and without sodium. When treated sample were dried a crust was formed with silt, and the silt fraction was free of sodium. Solidified white salt particles were found through-

Table 2

Removal efficiencies of clay and silt with the pH variation		
PH	Chromium removal efficiency (

PH	Chromium removal efficiency (%)		
	Silt	Clay	
2	61.00	-5.11	
8	62.77	0.61	
12	67.00	-36.8	

EDTA	Chromium removal efficiency (%)		
	Clay	Silt	
With Na	18.69	79.34	
Without Na	22.08	92.54	

 Table 3

 Removal efficiencies of clay and silt with EDTA

out dried sample. All these samples were separately evaluated for chromium removal. The test results show that there are no significant improvement in the chromium removal efficiencies for both clay and silt fractions even at extreme pHs values or with chelating agents.

The ESEM with EDX was used to investigate the immobile nature of chromium in the clay fraction. Fig. 17 shows the ESEM micrograph of treated clay fraction of the chromium-contaminated sediments obtained after treatment Processes #1 and #2. Fig. 18 shows an elemental map of chromium in Fig. 17 from EDX. The white dots represent the locations of Cr compounds deposited on the surface of sediments. Fig. 18 shows that most of the Cr contaminants were not on the surface as depicted by only a few white dots. The low surface chromium concentration confirms the persistent stability of chromium in the



Fig. 17. ESEM micrograph of clay fraction of treated dredged sediments after Process #2.



Fig. 18. EDX results depicting the chromium distribution in the ESEM micrograph shown in Fig. 17.

treated clay fraction of the sediments. In order to confirm the above finding the EPA toxic characteristic leaching procedure (TCLP) test was run. The measured TCLP value for total chromium was 0.059 ppm and the EPA criteria for action for chromium is 5 ppm or above. Hence, the TCLP analysis confirmed that it is safe to dispose the clay that was obtained after the Process #2.

Due to the persistent immobility of chromium in the clay fraction of the sediments it appears that the proposed technology is only applicable for soils without significant proportions of clay. Also when Process #1 results are compared with those of Process #2 of the proposed technology, it shows that when the particle size decreases the removal of metal contaminants becomes more difficult. Laboratory studies on remediation of chromium-contaminated soils by Pagilla et al. [16] showed that soils with higher clay content were less amenable to chromium extraction than soils with lower clay contents. Practically, all the chromium contaminants on the tested sediments were the stable trivalent form. Neale et al. [17] showed from a study of removal of heavy metals using chelating agents, that treating clays contaminated with chromium in trivalent state may not be necessary since the leachability of the metal may be minimal.

5. Summary and conclusions

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Due to the Ocean Dumping Act and lack of containment facilities, the disposal of nearly 7 million tonnes of dredged sediments per year has become a major problem for NY/NJ metropolitan area. The dredged sediments in a slurry form need to be de-watered, remediated and disposed. The sonication coupled with extraction using sub-atmospheric pressure was investigated to remove heavy metal contaminants from dredged residues. A laboratory study was performed to evaluate the above a technology to decontaminate heavy metals in dredged sediments. Dredged sediments obtained from NY/NJ harbor were used to validate the technology. The technology based on ultrasound consisted of two coupled processes to separate and to treat both coarse (Process #1) and fine (Process #2) fractions of the sediments. Selected variables for evaluation of Process #1 were power, soil-to-water ratio, vacuum pressure and dwell time, and for evaluation of Process #2 were power, soil-to-water ratio and dwell time. Experimental data showed that the Process #1 could effectively separate and treat the coarse fraction of the sediment with 100% separation efficiency and 92% removal efficiency. The Process #2 was also quite effective for treating the silt fraction of the dredged sediments with 100% separation efficiency and 82% removal efficiency. It was concluded that the clay fraction could not be effectively treated by this technology. However, due to the distribution of chromium in the clay fraction, it was found that the chromium is quite immobile in the clay fraction of the treated sediments. Therefore, it is safe to dispose clay fraction after treatment of the sediments using Process #2 since it had low leachable chromium confirmed by the TCLP test.

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