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Treating contaminated sediment with a two-stage base-catalyzed decomposition (BCD) process: bench-scale evaluation

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Abstract

The New York/New Jersey (NY/NJ) Harbor estuary sediment contains polychlorinated dibenzo-*p*-dioxins/furans (PCDDs/PCDFs), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and chlorinated pesticides. The U.S. EPA-developed two-stage base-catalyzed decomposition (BCD) process was tested for removal and decomposition of these organic contaminants of concern (OCCs) from the sediment. The Stage 1 process removed OCCs to below the limits of analytical quantitation (LAQs) or the limits of the potentially applicable requirements (LPARs), and concentrated OCCs into a small volume of organic condensate for Stage 2 chemical destruction. The aqueous condensate fractions contained some nondestroyed contaminants, either at levels below LPARs or at levels that could be treated by conventional treatment processes. Factors such as chemical addition, water content, and residence time were examined for their effects on the treatment process. Mass balances were calculated. The distribution of metal contaminants was determined. © 1997 Elsevier Science B.V.

Keywords: Base-catalyzed decomposition (BCD) process; Contaminated marine sediments; PCDDs/PCDFs; PCBs

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1. Introduction

The New York/New Jersey (NY/NJ) Harbor sediment contains high concentrations of polychlorinated dibenzo-*p*-dioxins/furans (PCDDs/PCDFs) [1–3], polychlorinated biphenyls (PCBs) [2,4–7], polycyclic aromatic hydrocarbons (PAHs) [8], chlorinated pesticides [3,5,9], petroleum hydrocarbons [10,11], and heavy metals [12–16]. These toxic contaminants enter the estuary through municipal and industrial discharges, urban run-off, landfill sites, and accidental spills [1,17–20]. Some of these contaminants have been detected in fish and shellfish tissue, and fishing advisories have been issued [21].

In response to the concerns over the degradation of sediment and marine resources, the U.S. Environmental Protection Agency (U.S. EPA) and the Army Corps of Engineers (COE) initiated a congressional-mandated program to select decontamination technologies for treating dioxin- and PCB-contaminated dredged materials [17]. Base-catalyzed decomposition (BCD) was one of the four technologies selected for bench-scale testing.

The BCD process was originally known as a base-catalyzed dehalogenation process which was developed in 1978 to destroy PCBs in soils [22]. Since then, a group of reagents called 'APEGs' (alkali metal polyethylene glycolates) have been developed by reacting sodium (Na) or potassium (K) or their hydroxides with polyethylene glycols or their derivatives between 75 and 120°C [23,24]. Proposed mechanisms involved nucleophilic substitution and oxidative dehalogenation of halogenated aromatics. However, increased moisture content may reduce APEGs' effectiveness and increase reagent consumption. Any by-products produced will remain in the treated matrices with unknown long-term effects.

A two-stage BCD process was later developed by the U.S. EPA [25]. In Stage 1, contaminated materials were mixed with $NaHCO_3$ and heated to about 340°C. In Stage 2, the resulting organic condensates were dehalogenated at about 340°C using a reagent mixture containing a hydrogen-donor oil, NaOH, and a catalyst. The Stage 1 removal efficiency for contaminated soils ranged to 99.9997% and to $> 99.99\%$ for PCBs [26] (B.C. Kim, Decontamination of a PCB-Contaminated Soil: Results of a Pilot-Plant Study at Guam, Personal communication, Battelle, Columbus, OH, 1994) and PCDDs/PCDFs [27], respectively. The effectiveness for marine sediments containing large amounts of water has not been fully demonstrated; only a few bench- and pilot-scale studies have been carried out [17]. One study using a one-stage process similar to the Stage 2 treatment removed PCDDs/PCDFs and PCBs in a Newark Bay sediment to below the limits of analytical quantitation (LAQs) [28].

Most BCD investigations have focused on Stage 1 treatment efficiency; little is known about the removal mechanisms. Most researchers attributed removal to dehalogenation or chemical destruction [17,26–29], although volatilization appeared to predominate. Very few studies have examined the mass balance and appropriate reagent proportions in both stages. Partial oxidation and pyrolysis may produce more toxic by-products than their precursors [30]. The contaminants and their by-products may volatilize and partition into various condensate fractions, which must be treated before disposal. The treated oily residue in Stage 2 may contain dehalogenated products and other organic contaminants originally present in the contaminated matrix.

No information is available on the effects of metals on BCD and their behavior during treatment. Mercury (Hg), if present, most likely will volatilize and partition into the water and organic condensates during the Stage 1 reaction. Arsenic (As) and elemental cadmium (Cd) may partially transfer to the vapor phase and condense before entering the Stage 2 reactor. Metalloid and metallic contaminants in off-gas, condensates, and treated oily fluids could require additional air and water treatment, increasing the complexity and expense of disposal. Nonvolatile metals remaining in the sediment could cause the treated sediment to exhibit a Resource Conservation and Recovery Act (RCRA) leachable toxicity characteristic, thus, requiring additional treatment before disposal.

This paper reports the results of a bench-scale study evaluating the effectiveness of the two-stage BCD process for treating sediment and examining the partitioning of chlorinated organic and metal contaminants during each treatment stage [31]. The objectives were to optimize $NaHCO_3$ usage in Stage 1 while tracking the partitioning of contaminants, and to evaluate the effectiveness of Stage 2 for destroying the contaminants in condensates from Stage 1.

2. Materials and methods

2.1. Sampling locations and sample characterization

A black, clayey silt sediment was sampled off the Newtown Creek at the entrance to Dutch Kills, which represents some of the most contaminated areas in the NY/NJ estuary. The sediment was collected with a modified Van Veen sampler, stored in a labeled 11.4-l tin container, and shipped overnight to Battelle's Columbus Laboratories. Upon arrival, free water was skimmed off and the sediment was thoroughly mixed, dispensed into three 3.8-l amber glass bottles, and stored in the dark at 4°C. Appropriate aliquots of the sample were analyzed for the analytes listed in Table 1. Because PCDDs and PCBs were critical to process validation, they were the main targets of this study.

2.2. Reactor systems

Fig. 1 presents the schematic of the process evaluated. The Stage 1 reactor system consisted of a 1-l stainless steel (SS) reactor capped with a three-port SS reactor head, a mixing assembly, a temperature monitoring/control system, a condensate receiver (connected to two jacketed condensers in series), and an off-gas granular-activated carbon (GAC) trap packed with 20 × 60 mesh U.S. standard mesh Darco 3000 GAC (NORIT, Atlanta, GA). Heating mantles and heating tapes were used on the reactor, reactor head, and the nonreceiving side of the condensate receiver (to prevent vaporized gases from being condensed back into the reactor). Thermocouples were used to monitor the temperatures in the reactor and the vapor prior to the condenser. A closed-system vacuum seal adaptor was used to seal the leaks in and around the mixing assembly. Tap water at 4°C was used to cool the jacketed condensers.

Table 1
List of analytes and analytical methods

Analyte	Untreated sediment	Treated sediment	Treated and untreated aqueous and yellow condensates	Organic condensate and rinsate	Oily residue	GAC trap
Dioxins/furans	Modified EPA 8290	Modified EPA 8290	Modified EPA 8290	Modified EPA 8290	Modified EPA 8290	N/A
PCBs/pesticides	Modified EPA 8080, SOP 5-128-02,SOP 5-192, SOP 5-191	Modified EPA 8080, SOP 5-128-02,SOP 5-192,	Modified EPA 8080, SOP 5-128-02,SOP 5-191	Modified EPA 8080, SOP 5-128-02,SOP 5-191	Modified EPA 8080, SOP 5-128-02,SOP 5-191	N/A
Chlorinated herbicides	EPA 8150	N/A	N/A	N/A	N/A	N/A
Ag/Cd	SOP MSL-M-024	SOP MSL-M-024	SOP MSL-M-024	SOP MSL-M-024	N/A	N/A
As/Cr/Cu/Ni/Pb/Zn	PNL-SP-19	PNL-SP-19	SOP MSL-M-024	SOP MSL-M-024	N/A	N/A
Hg	SOP MSL-M-031	SOP MSL-M-031	SOP MSL-M-027	SOP MSL-M-027	N/A	N/A
Percent solids	EPA 2540	N/A	N/A	N/A	N/A	N/A
Grain size distribution	Folk, 1974 [32]	N/A	N/A	N/A	N/A	N/A
TOC	Modified EPA 9060	Modified EPA 9060	Modified EPA 9060	N/A	N/A	N/A
EOX	See text	See text	See text	See text	See text	N/A
AOX	N/A	N/A	N/A	N/A	N/A	ASTM D4744-87
pH	EPA 9045	N/A	EPA 150	N/A	N/A	N/A

N/A = not applicable.

SOP 5-128-02. 1992. Identification and Quantification of Polychlorinated Biphenyls (by Congener and Aroclor) and Chlorinated Pesticides by Gas Chromatography/Electron Capture Detector (GC/ECD). Battelle Ocean Sciences, Duxbury, MA.

SOP 5-192. 1991. Sediment Extraction for Trace Level Semi-Volatile Organic Contaminants. Battelle Ocean Sciences, Duxbury, MA.

SOP 5-191. 1993. HPLC Cleanup of Sample Extracts for Semi-Volatile Organic Pollutants. Battelle Ocean Sciences, Duxbury, MA.

SOP MSL-M-024. 1994. Determination of Elements in Aqueous and Digested Samples by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS). Battelle Pacific Northwest Laboratories, Marine Sciences Laboratory, Sequim, WA.

PNL-SP-19. 1986. Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF) Using the Backscatter/Fundamental Parameter (BFP) Approach with the Kevex D810 System. Battelle Pacific Northwest Laboratories, Richland, WA.

SOP MSL-M-031. 1992. Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption (CVAA). Battelle Pacific Northwest Laboratories, Marine Sciences Laboratory, Sequim, WA.

SOP MSL-M-027. 1993. Total Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence (CVAF). Battelle Pacific Northwest Laboratories, Marine Sciences Laboratory, Sequim, WA.

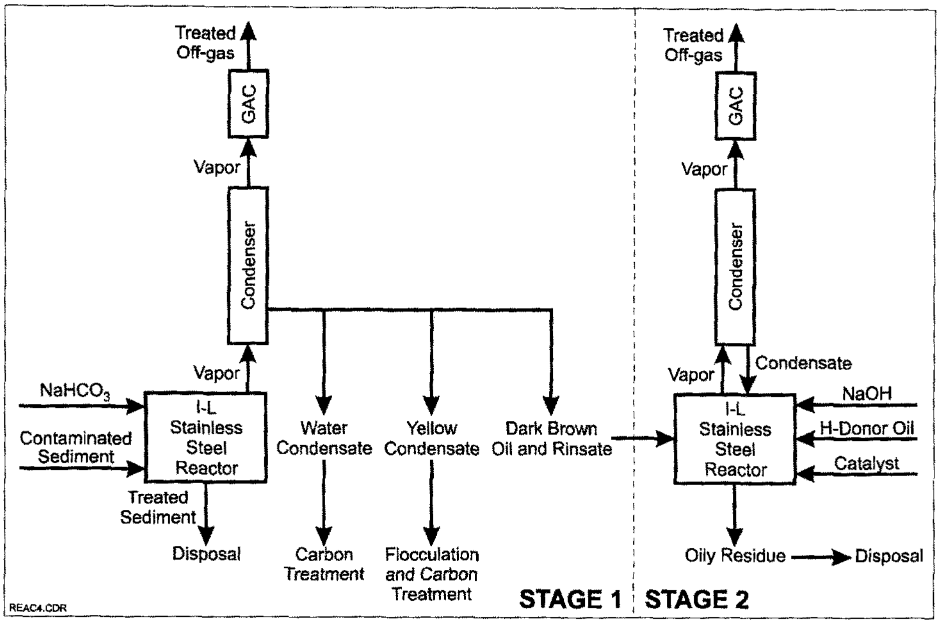


Fig. 1. Schematic of two-stage BCD process.

The Stage 2 reactor system was constructed similarly, except that a four-port glass reactor head, a No. 121 Vigruex-style condenser (VSC) (to hold up the oily residue vaporized during heating), and a syringe needle (to provide an N_2 blanket in the reactor) were included in the system. Two jacketed condensers in series were connected to the exit side of the VSC and exited to a condensate receiver held at ambient temperature. Both reactor systems were leak-proof, as indicated by $> 99.3\%$ water recovery in the respective condensate receivers.

2.3. Stage 1 process

Three Stage 1 process variables were examined: $NaHCO_3$ dosage (0, 5, and 10% of sediment dry weight), contact time (1 and 2 h), and water content (wet and dry). (Experiments also were conducted using NaOH; however, during processing, the sediment/NaOH mixture hardened into a cementatious mass, causing handling problems and mechanical failure of the reactor mixer.) For the wet runs, 600 g of the as-received sediment and a weighed dose of $NaHCO_3$ were measured into and thoroughly mixed in the reactor, and heated under hood to between 100 and 110°C (for initial drying) and then to 340°C with a 1- or 2-h hold. For the dry runs, $NaHCO_3$ was added after the initial drying and subsequent cooling of the sediment to about 25°C (to avoid volatilization of the contaminants when opening the reactor). Once the $NaHCO_3$ was mixed into the dried sediment, the Stage 1 process was resumed by rapidly ramping the temperature from the ambient to 340°C. Runs with no $NaHCO_3$ served as controls.

The condensate fractions generated during heating included a clear water condensate, a cloudy yellow emulsion, and a dark brown oil. Collection of the clear water condensate began between 85 and 98°C and ceased as it started to turn yellow between 140 and 274°C. Collection of the yellow emulsion continued until the end of the run. The oil started to emerge on top of the water condensate at about 100°C. The pale-white oily layer gradually increased in volume (to about 4 ml) and turned dark brown between 211 and 274°C. The water condensate and yellow emulsion were withdrawn intermittently from the condensate receiver; the oil was removed at the end of the run. The condensates were collected separately in glass bottles, capped with Teflon™-lined lids, and stored in the dark at 4°C.

All glassware was rinsed sequentially with toluene, acetone, and hexane and then washed with a chlorine-free Alconox™ detergent and distilled deionized water (DDW). The solvent rinsates were combined and stored as described above for the condensates. The reactor was rinsed with acetone, washed sequentially with Alconox™ and DDW, and rinsed again with acetone.

The treated sediment was either disaggregated using a SS spatula or pulverized using a Nasco soil grinder (Ft. Atkinson, WI). Pulverization significantly improved the precision of extractable organic halides (EOX) measurements, as indicated by the reduction of relative standard deviation (RSD) values from 27.5–104.5% to 2.3–38.3%. Therefore, only pulverized materials were used for chemical analyses.

The treated sediment thus obtained was analyzed for EOX and/or PCDDs/PCDFs. The experimental run, judged to have produced the most favorable treatment results, was repeated four more times to yield sufficient process sidestreams for follow-on treatments. Appropriate aliquots of treated sediment and each process sidestream were analyzed for the analytes listed in Table 1.

2.4. Condensate treatment

The clear water condensate and yellow emulsion were treated by oil extraction, flocculation, and/or GAC adsorption. Oil extraction was carried out on three different aliquots, corresponding to a basic (pH 12.5 by NaOH), a mildly basic (pH 8.6, no pH adjustment), and an acidic (pH 1.5 by H_2SO_4) pH value. A 70/30 (by vol.) Sunpar LW110/104 oil mixture (Sunoco, Trenton, NJ) was used at a 1:1 volume ratio for 1 h. After phase separation for 30 min, both fractions were analyzed for EOX.

Six flocculation tests were performed using alum, $FeSO_4$, and three commercially available cationic polyelectrolytes (i.e. Clarifloc® C-9420, C-9455, and C-9535 [Rhône-Poulenc, Parsippany, NJ]). C-9455 and C-9535 are more highly charged than C-9420. Stock solutions of 1000 mg/l and 0.25% (by vol.) were prepared for the inorganic salts and the polyelectrolytes, respectively. The test procedures followed those of Dempsey et al. [33]. The solutions before and after treatment were analyzed for EOX and total organic carbon (TOC).

The batch adsorption experiments used 100 × 200 U.S. Standard mesh Calgon F-400 GAC. After 2 days, the solutions were filtered through 0.45-mm glass fiber filter membranes (Millipore, Bedford, MA) and analyzed for EOX and TOC.

The dark brown oil and solvent rinsate from Stage 1 was treated in the Stage 2 reactor. About 6.4 ml of the liquid was mixed with 2 g of a carbonaceous catalyst, 5 g of

NaOH, and 100 ml of an 80/20 (by vol.) Sunpar LW110/104 oil mixture, and heated to 340°C at 13°C/min for 3 h. The resulting black oily residue and the solvent rinsate were analyzed as indicated in Table 1.

2.5. Analytical procedures

The analytical methods and process matrices are listed in Table 1. Modified U.S. EPA standard methods which adhered to EPA's requirements for marine sediment matrices [34], were used in most cases. Methods involving significant modifications had already been published [35] or documented as standard operating procedures (SOPs). Internal standards, cleanup standards, and/or recovery standards were added to sample matrices and solvent extracts when preparing samples for compound-specific analyses. Some sample extracts had to be diluted or refractionated by high-performance liquid chromatography (HPLC) for reanalyses because of background interference. Solvent extracts of oily residue samples had to be digested with concentrated H_2SO_4 to improve the chromatographic resolution. Solids in the oily residue were Soxhlet-extracted to remove any organic contaminants of concern (OCCs) and the resulting extracts were combined with the respective solvent extracts for processing. Quality control samples including method blanks, blank spikes, sample replicates, matrix spikes, and standard reference materials were used to ensure data quality.

EOX samples of the untreated sediments were prepared by centrifuging known amounts of samples at 10 000 rotations/min (rpm) for 5 min, extracting the partially dewatered materials with 10.0 ml of ethyl acetate (EtAc) at room temperature for 24 h, purifying the EtAc extracts by repetitive centrifugation, and concentrating the purified extracts to known volumes. Treated sediment preparation differed only by initially wetting the samples with 10 ml of DDW. The water condensates and their treated counterparts were prepared by extracting 10 ml of the samples with 2 ml of EtAc and purifying and concentrating the extracts as described above. The solvent rinsate, dark brown liquid, and oily residue were analyzed by direct injection (with or without dilution with EtAc). Adsorbable organic halides (AOX) in GAC from the off-gas traps were measured using a Dohrmann DX-2000 total halide analyzer through a direct-burn process of accurately weighed samples. TOC in sediment and aqueous condensate samples was analyzed using a UIC Model 5012 carbon analyzer equipped with a coulometric/photometric detector. All samples were extracted/analyzed in triplicate.

3. Results and discussion

3.1. Sediment characterization

Table 2 presents the contaminant concentrations in the sediment and each process sidestream. Over 17.7 and 4 ng/g of PCDDs and PCDFs, respectively, were measured in the sediment. The 2,3,7,8-tetrachlorodibenzo-*p*-dioxin/furan (TCDD/TCDF) concentrations were low, ranging from 10 to 130 pg/g. The TCDD concentration was about

Table 2
Contaminant concentrations[a] in untreated sediment and process sidestreams

Contaminants			Stage 1				Stage 2	
	Untreated sediment	Treated sediment	Water condensate	Yellow emulsion	Dark brown oil	Rinsate	Oily residue	Rinsate
PCDDs	*(pg / g)*	*(pg / g)*	*(pg / ml)*	*(pg / ml)*	*(ng / ml)*	*(pg / ml)*	*(pg / g)*	*(pg / ml)*
2,3,7,8-TCDD	9.8	< 2.0	< 0.34	< 0.55	46.7	11.5	< 7.6	< 0.59
1,2,3,7,8-PeCDD	28.3	1.9	< 0.62	ND	58.4	< 0.98	< 9.4	< 0.98
1,2,3,4,7,8-HxCDD	36.9	< 1.9	< 0.39	< 0.47	18.0	< 0.74	< 15.9	< 0.74
1,2,3,6,7,8-HxCDD	97.7	2.5	< 0.32	< 0.44	49.7	21.9	< 10.1	< 0.65
1,2,3,7,8,9-HxCDD	96.4	7.4	< 0.37	< 0.74	107.9	37.0	< 13.6	< 0.73
1,2,3,4,6,7,8-HpCDD	2133	22.9	< 0.16	< 0.30	294.4	88.7	68.6	< 0.24
OCDD	15 369	48.8	0.5	9.3	516.2	146.9	234.1	< 0.42
Total PCDDs	17 771	83.5	0.5	9.3	1091.3	306.0	302.7	ND
PCDFs								
2,3,7,8-TCDF	129.9	< 2.2	< 0.36	< 0.75	76.2	14.2	< 8.6	< 0.53
1,2,3,7,8-PeCDF	111.9	< 2.9	< 0.49	< 0.20	29.1	< 0.94	< 9.2	< 0.94
2,3,4,7,8-PeCDF	78.9	< 1.3	< 0.39	< 0.71	19.3	< 0.75	< 6.2	< 0.75
1,2,3,4,7,8-HxCDF	398.9	< 1.5	< 0.08	< 0.31	47.4	14.4	< 6.9	< 0.34
1,2,3,6,7,8-HxCDF	141.5	< 0.8	< 0.07	< 0.22	26.4	5.7	< 6.1	< 0.32
1,2,3,7,8,9-HxCDF	95.2	< 0.9	< 0.09	< 0.46	< 0.20	< 0.38	< 4.4	< 0.38
2,3,4,6,7,8-HxCDF	5.9	< 1.1	< 0.07	< 0.66	9.4	4.7	< 5.4	< 0.33
1,2,3,4,6,7,8-HpCDF	1844	4.8	< 0.07	< 0.22	60.3	18.5	< 8.5	< 0.20
1,2,3,4,7,8,9-HpCDF	71.4	< 1.2	< 0.16	< 0.43	< 0.45	< 0.27	< 9.8	< 0.27
OCDF	1170	< 2.5	< 0.26	< 0.82	8.4	3.8	< 13.5	> 0.50
Total PCDFs	4047	4.8	ND	ND	276.5	61.3	ND	ND
PCBs	*(ng / g)*	*(ng / g)*	*(ng / ml)*	*(ng / ml)*	*(μg / ml)*	*(ng / ml)*	*(ng / g)*	*(ng / ml)*
di-CB	59.8	9.3	0.54	34.6	8.1	0.11	< 18.8	1.7
tri-CB	216.4	1.1	0.19	38.4	4.4	0.43	< 9.2	6.6
tetra-CB	403.3	8.0	1.32	32.5	10.1	0.56	< 8.4	26.6
penta-CB	397.6	1.7	0.11	11.5	7.1	0.38	< 7.1	17.5
hexa-CB	211.4	1.2	0.28	6.8	6.4	0.40	< 6.9	3.0
hepta-CB	220.7	0.4	0.06	3.7	2.1	0.12	< 5.4	0.3
octa-CB	< 0.05	< 0.05	< 0.002	< 0.002	< 2E-06	< 0.002	< 4.7	< 0.002
nona-CB	< 0.08	< 0.08	< 0.001	< 0.001	< 1E-06	< 0.001	< 8.1	< 0.001
deca-CB	< 0.09	< 0.09	< 0.002	< 0.002	< 2E-06	< 0.002	< 8.5	< 0.002
Total PCBs	1509.2	21.7	2.50	127.5	38.2	2.0	ND	55.7
Pesticides	*(ng / g)*	*(ng / g)*	*(ng / ml)*	*(ng / ml)*	*(μg / ml)*	*(ng / ml)*	*(mg / g)*	*(ng / ml)*
Lindane	< 0.095	< 0.095	< 0.007	< 0.007	0.42	0.24	NQ	< 0.007
Heptachlor	< 0.094	0.44	< 0.005	28.6	5E-06	0.11	NQ	< 0.005
Aldrin	< 0.091	< 0.091	< 0.002	< 0.002	2E-06	< 0.002	NQ	< 0.002
Heptachlorepoxide	< 0.061	0.37	< 0.006	< 0.006	6E-06	< 0.006	NQ	0.13
Endosulfan I/2,4-DDE	< 0.200	< 0.200	< 0.004	2.4	4E-06	0.08	NQ	< 0.004
α-Chlordane	51.94	2.91	0.07	1.2	0.19	0.01	NQ	< 0.005
t-Nonachlor	32.28	0.16	< 0.005	< 0.005	5E-06	0.04	NQ	< 0.005
Dieldrin	35.71	0.31	0.08	< 0.002	0.51	< 0.002	NQ	0.10
4,4-DDE	63.43	1.23	0.18	2.4	1.09	0.04	NQ	1.07
2,4-DDD	47.82	< 0.213	< 0.005	< 0.005	5E-06	< 0.005	NQ	0.14
Endolsulfan II	35.38	< 0.200	< 0.002	1.2	2E-06	0.06	NQ	< 0.002

Table 2 (continued)

Contaminants	Stage 1						Stage 2	
	Untreated sediment	Treated sediment	Water condensate	Yellow emulsion	Dark brown oil	Rinsate	Oily residue	Rinsate
4,4-DDD	120.57	2.13	0.05	2.6	1.27	0.12	NQ	0.71
2,4-DDT	9.07	< 0.137	< 0.004	2.6	4E-06	< 0.004	NQ	< 0.004
Endosulfan sulfate	23.75	0.43	0.03	< 0.003	1.48	< 0.003	NQ	< 0.002
4,4-DDT	< 0.137	< 0.137	0.04	1.2	4E-06	0.15	NQ	< 0.004
Total pesticides	419.95	7.98	0.45	42.2	4.96	0.85	NQ	2.15
Metals	*(μg/g)*	*(μg/g)*	*(ng/ml)*	*(ng/ml)*	*(ng/ml)*			
Ag	14.4	12.5	1.5	< 0.3	0.7			
As	33.1	29.1	< 20.0	456	49.4			
Cd	19.9	16.1	< 0.3	< 0.3	< 0.3			
Cr	305	321	< 6.0	< 6.0	< 6.0			
Cu	769	731	8.4	3.2	40.0			
Hg	2.8	0.13	5.4	13.1	3.0			
Ni	140	139	2.4	1.5	10.2			
Pb	554	530	1.9	3.0	9.4			
Zn	1260	1260	< 10.0	< 10.0	50.8			
Surrogate parameters								
Solids (%)	33.40							
Grain-size distribution (%)								
Gravel	0.1							
Sand	35.1							
Silt	43.4							
Clay	21.5							
Fine[c]	64.9							
TOC[d] (% or μg/ml)[e]	7.89		59.1	1282				
EOX[d] (μg/g or μg/ml)[f]	36.31	0.40	0.51	1.73	1150.9	3.61	0.24	3.61
pH[b]	7.76		8.6	9.6				

[a] Dry weight basis.

[b] Average of 3 to 5 replicate analyses.

[c] Sum of % silt and % clay.

[d] Average of 2 or 3 replicate extractions/analyses.

[e] %, unit for sediments; μg/ml, unit for condensates.

[f] μg/g, unit for sediments and oil residue; μg/ml, unit for condensates and rinsates.

ND, None detected.

NQ, Not quantifiable.

three orders of magnitude lower than the highest concentration (21 ng/g) ever detected in the NY/NJ Harbor estuary [1]. The concentrations of heptachlorodibenzo-*p*-dioxin (HpCDD) and octachlorodibenzo-*p*-dioxin (OCDD) were significantly higher. Although these congeners usually are less toxic, they might be converted to more toxic

TCDD/TCDF by BCD [27]. Therefore, the presence of the more highly chlorinated congeners should not be overlooked.

The total PCB concentration measured was 1509 ng/g. Of nine PCB homologs, five had concentrations over 216 ng/g. Chlorinated pesticides totaled 420 ng/g. Chlorinated herbicides were below the LAQs. Among the seven RCRA metals detected, As, Cd, and Hg could be transferred partially to the vapor phase at elevated temperatures. In general, the contaminant concentrations in the sediment were within two orders of magnitude of the reported data [1,16,21,28].

The sediment pH was 7.8, comparable with reported values [16]. Because the sediment comprised 66.6% water, removal of interstitial water before heating would be desirable. Centrifugation reduced the water content only to about 51%, largely due to its high silt and clay contents. The sediment was very rich in TOC (i.e., 7.9%), about 3.5 times more than that of a Newark Bay sediment [16]. A small fraction of the TOC was halogenated organic compounds, as indicated by the small amount of EOX measured.

3.2. Stage 1 process

Fig. 2 shows two temperature curves, depicting a wet and a dry test run (Table 3 has process conditions). The differences between the curves represent $NaHCO_3$ effects. $NaHCO_3$ apparently had elevated the boiling temperature and the heat of evaporation of the interstitial water in the wet runs, thereby changing the drying temperature and drying rate. For example, water began to condense between 85 and 93°C for all dry and all control runs, but between 96 to 98°C for all wet runs. The corresponding drying time at 100 to 110°C decreased from 80–130 min to 35–50 min. The ramping rate from 110 to

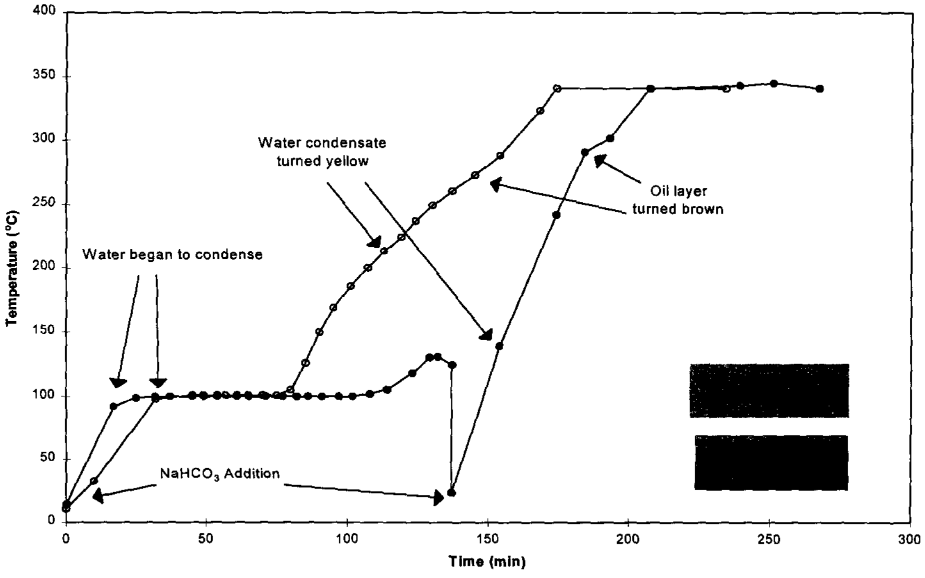


Fig. 2. Temperature curves of a wet and a dry run.

340°C also changed from 4.5–4.7°C/min (for wet control runs) to 2.5–3.5°C/min (for wet runs) or to 4.3–6.3°C/min (for dry runs). The hump and the dip on the dry-run curve indicated cooling of the reactor just after its temperature had started to ramp upwards; the actual time elapsed during cooling was not plotted on the curve.

$NaHCO_3$ also significantly changed the amounts of water condensate produced. For the runs dried with no $NaHCO_3$, 351 to 387 ml was collected, equivalent to 88 to 94% of the total condensate generated. In contrast, the wet runs produced 270 to 330 ml, equivalent to only 67 to 78% of the total. Less water condensate meant more yellow emulsion, which required more complex post-treatment. Therefore, the sediment should be dried before adding $NaHCO_3$.

Table 3 shows the EOX concentrations of the treated sediment. Thermal desorption alone removed 93.47 to 99.42% of EOX. $NaHCO_3$ increased EOX removal to as high as 99.89%. However, the increase in EOX removal was marginal regardless of $NaHCO_3$ dosage or contact time. For PCDDs, 10% $NaHCO_3$ and 1 h contact time (Run 11) attained complete removal of all PCDDs except traces of HpCDD and OCDD. Run 9, using 5% $NaHCO_3$ and 1 h contact time, left residues of TCDD and most other congeners, totaling 29.8 pg/g. PCDD removal by Run 8, a predried control run, was more complete (totaling 8.25 pg/g) than that by Run 9, which was inconsistent with the EOX results or with the expected $NaHCO_3$ effects. Additional studies are needed to examine the mechanistic role of $NaHCO_3$. Although it was difficult to determine the best process conditions based on the EOX results, the most complete removal of PCDDs/PCDFs was achieved during Run 11, so its process conditions were used to produce quantities of process sidestreams for follow-on treatments.

Table 3
EOX in untreated and treated sediment

Run no.	$NaHCO_3$ dosage (%)	Contact time (h)	Water content	EOX^{ab} ($\mu g/g$)	Removal (%)
0^c	0	0	AR	36.31	–
1	0	1	AR	2.37	93.47
2	0	2	AR	0.68	98.13
3	5	1	AR	0.17	99.53
44	5	2	AR	0.20	99.45
5	10	1	AR	0.04	99.89
6	10	2	AR	0.29	99.20
7	0	1	PD	0.21	99.42
8	0	2	PD	0.62	98.29
9	5	1	PD	0.10	99.72
10	5	2	PD	0.08	99.78
11	10	1	PD	0.09	99.75
12	10	2	PD	0.08	99.78

[a] Dry weight basis.
[b] Average of three replicate extractions.
[c] Untreated sediment.
AR = As received.
PD = Predried.

3.3. Stage 1 treatment effectiveness

The Stage 1 treatment removed 99.53% of PCDDs and 99.88% of PCDFs from the sediment (Table 2). TCDD was reduced to below the LAQ (2.3 pg/g), but trace amounts of pentachloro- to octachloro-congeners (1.9 to 48.8 pg/g) were detected. The treatment also removed 98.56% of PCBs. Three of the nine pesticides were removed to below the LAQs; the others were reduced by 94.4 to 99.5%. Overall removal exceeded 98%. Of the nine metals, five (Cr, Cu, Ni, Pb, and Zn) remained relatively unchanged; three (Ag, As, and Cd) lost about 12 to 19%; and one (Hg) almost completely disappeared. Hg removal by heat, a common treatment [36], was consistent with its vapor pressure data. The treated material exhibited nonleachable characteristics of all eight RCRA metals.

The universal treatment standards for the Land Disposal Restrictions [37] require the total concentrations of each PCDD/PCDF congener and total PCBs in a waste matrix to be reduced to 1 ng/g and 10 mg/g, respectively, before land disposal may be considered. Further, a landfill facility permitted under RCRA Subtitle C in NY would require the waste to contain < 2 ng/g of dioxins and < 500 mg/g of PCBs, with disposal evaluated on a case-by-case basis [31]. Disposal in a landfill permitted under RCRA Subtitle D for nonhazardous solid waste would require nondetectable dioxins and < 50 mg/g of PCBs. Therefore, the treated material potentially could be disposed of in a RCRA Subtitle C facility.

For ‘unrestricted’ ocean disposal, the treated matrix must be tested for water column toxicity, benthic toxicity, and bioaccumulation, and must be ‘clean enough’ to pass these tests when compared to clean reference sediments [34]. If the sediments were to show reduced toxicity but failed to pass these tests, capping of the treated matrix with clean sediment may be required. Materials that fail these tests cannot be returned to the ocean even if treatment were to reduce the contaminant levels to nondetectable levels.

3.4. Yellow emulsion / water condensate treatment

Neither yellow emulsion nor water condensate exceeded the LAQs for PCDDs (except for OCDD) or PCDFs (Table 2). The yellow emulsion was turbid and had higher concentrations of other OCCs, making it desirable to segregate and treat this condensate fraction separately. Among the three methods tested, oil extraction removed only 35.6 to 66.5% of EOX. The extraction was more efficient at acidic pH, probably due to protonation of some organic moieties in the matrix. The treated liquid remained turbid.

Table 4 summarizes the results of the flocculation tests for yellow emulsion. Test 1 used alum, $FeSO_4$, and C-9420. Tests 2 to 4 used $FeSO_4$ and C-9420. Light flocs formed only after adding C-9420 and adjusting the solution pH values to near neutral. Tests 5 and 6 used only C-9455 or C-9535. Without pH adjustments, dense flocs formed immediately after adding the polyelectrolytes. About 14 to 18% of EOX and 29 to 47% of TOC were removed by Tests 1 to 4. Up to 62% of EOX and 90% of TOC were removed by Tests 5 and 6, indicating effective treatment by both C-9455 and C-9535. GAC adsorption removed $> 97\%$ of EOX and $> 98\%$ of TOC and the sharp odor associated with the yellow emulsion.

Table 4
Flocculation tests for yellow emulsion

		pH				Mixing		EOX			TOC		
Test no.	Condensate volume (ml)	Before[a]	After[b]	Coagulant	Coagulant dosage (ml)	Rapid (min)	Slow (min)	Untreated (mg C/l)	Treated (ml Cl/l)	Removal (%)	Untreated (mg C/l)	Treated (mg C/l)	Removal (%)
1	73	9.42	7.60	Alum/$FeSO_4$/ C-9420	5.7/4.4/0.5	1	> 120	1.37	1.12	18.3	1485	1048	29.4
2	71	9.26	7.57	$FeSO_4$/C-9420	5.9/1.0	1	45	1.37	1.17	15.2	1485	982	33.9
3	70	9.36	7.62	$FeSO_4$/C-9420	8.9/0.5	1	20	1.78	1.52	14.6	1551	822	47.0
4	30	9.31	7.30	$FeSO_4$/C-9420	3.0/1.0	1	20	1.78	1.54	13.4	1551	873	43.7
5	70	9.68	N/A	C-9455	1.0	1	< 1	1.85	0.72	61.4	1506	184	87.8
6	70	9.68	N/A	C-9535	1.0	1	< 1	1.85	0.70	62.1	1506	149	90.1

[a] Before flocculation test.

[b] After flocculation test; H_2SO_4 was used for pH adjustments.

N/A = No pH adjustment.

Table 5
Water condensate and yellow emulsion treatment results

		Water condensate		Yellow emulsion		
Contaminant	Concentration unit	No treatment	After GAC treatment	No treatment	After polyelectrolyte treatment	After polyelectrolyte and GAC treatment
PCDDs	ng/l	0.5[a]	1.3[a]	9.3[a]	0.1[a]	ND
PCDFs	ng/l	ND	ND	ND	ND	ND
PCBs	ng/l	2497	ND	127,600	9870	ND
Pesticides	ng/l	445	ND	42,240	3030	ND
EOX	μg/l	509	144	1726	1007	54
TOC	mg/l	59.1[b]	10.4[b]	1282.1[b]	80.8[b]	22.2[b]

[a] Less than method blank.
[b] Average of two measurements.
ND = None detected.

Based on these results, the yellow emulsion was treated with C-9535 followed by GAC, and the water condensate was treated only with GAC. The treated solutions contained below the LAQs of all OCCs (Table 5). GAC removed 94.6% of EOX and 75.7% of TOC from the polyelectrolyte-treated yellow emulsion, and 71.7% of EOX and

Table 6
Partitioning and mass balance in Stage 1 reactor system

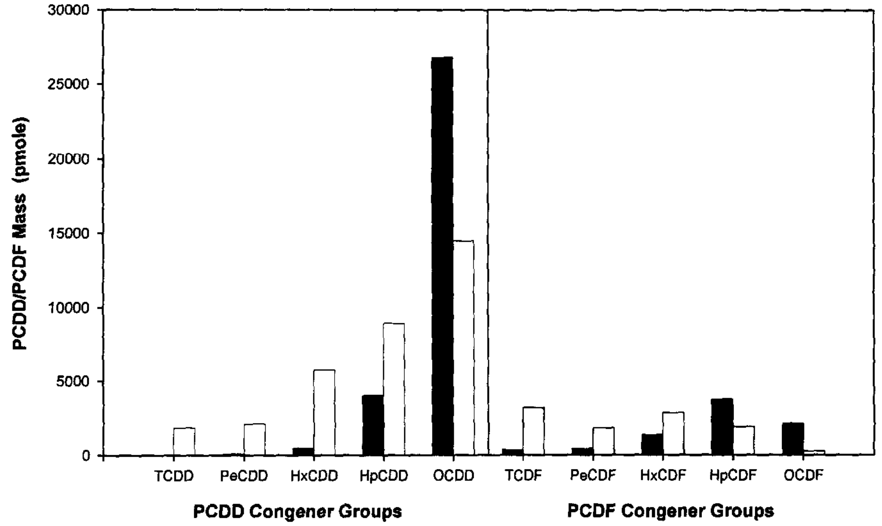
Mass flow	Matrix	Weight or volume (g[a] or ml[b])	PCDDs		PCDFs		PCBs		Organic halides	
			Total mass (pmole)	Mass balance (%)	Total mass (pmole)	Mass balance (%)	Total mass (nmole)	Mass balance (%)	Total mass (mgCl)	Mass balance (%)
In	Untreated sediment	801.9	31 388.0	100.00	8029.0	100.00	3897.2	100.00	29.28	100.00
Out	Treated sediment	791.5	150.4	0.48	9.50	0.12	64.9	1.67	0.31	1.07
	Aqueous condensate	165.0	12.65	0.04	ND	0.00	11.3	0.29	0.64	2.20
	Yellow emulsion	373.0	0.75	0.002	ND	0.00	176.4	4.53	0.64	2.20
	Dark brown liquid	12.8	32 938	104.94	9979.0	124.29	1697.9	43.57	14.73	50.32
	Rinsate	896.0	641.5	2.04	148.7	1.85	5.9	0.15	3.23[d]	11.04
	GAC trap[c]	39.8	N/A	N/A	N/A	N/A	N/A	N/A	5.32[e]	18.16
Recovery (%)				107.50		126.26		50.2		84.99

[a] Dry weight of sediments and GAC.
[b] Volume of condensates and rinsate.
[c] Because of their low vapor pressures, PCDDs/PCDF, and PCBs were very unlikely to partition to the GAC trap.
[d] Corrected with blank, 0.98 μg Cl/ml.
[e] Corrected with blank, 315.3 μg Cl/g.
ND = None detected.
N/A = Data not available.

83.8% of TOC from the water condensate, indicating the presence of less adsorbable organic moieties in both condensate fractions.

3.5. Partitioning and mass balance in Stage 1 system

The OCCs partitioned primarily to the dark brown oil; very few were found in the water condensate (Table 2). Some OCCs were recovered into the yellow emulsion and solvent rinsate. Molar concentrations of most OCCs (see Table 6) were used to calculate



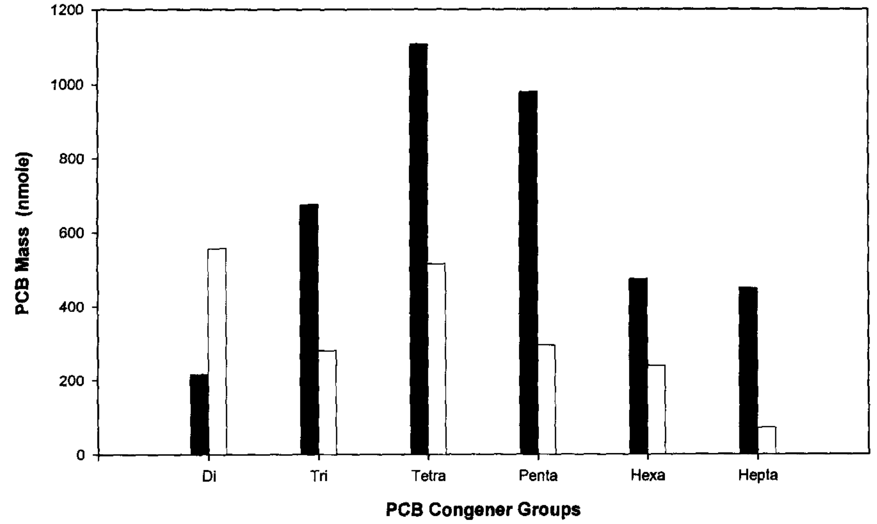


Fig. 3. Congener shifting following Stage 1 treatment. (a) PCDDs and PCDFs; (b) PCBs. Filled bars represent before treatment, hollow bars represent after treatment.

total masses in each process sidestream which, in turn, were used to calculate the mass balance. The congener-specific data indicated that partial dechlorination occurred in Stage 1, as evidenced by increased amounts of less-chlorinated congeners and decreased amounts of more-chlorinated congeners (see Fig. 3). For example, following treatment, TCDD and pentachloro-dibenzo-*p*-dioxin (PeCDD) masses increased over 77 and 33 times, respectively, whereas, OCDD mass decreased almost 2 times. A similar dechlorination trend also is shown in Fig. 3 for PCDFs and PCBs and was demonstrated for PCDDs/PCDFs by Tiernan [27].

Totaling the masses of TCDDs and PCDFs in each congener group resulted in a total recovery of 107.5 and 126.3%, respectively. This high conservation of mass across the congener groups supports the evidence of partial dechlorination shown in Fig. 3. Analysis of the di-chlorinated through hepta-chlorinated PCB congeners resulted in recovery of only 50.2% of the initial mass of PCBs. The loss in PCB mass could be due to dechlorination beyond the di-chlorinated level or, volatilization to the GAC trap, which was less likely because of the low vapor pressure of PCB congeners. In a previous BCD study by Battelle on a contaminated soil, dechlorination of PCBs to mono-chlorinated biphenyl and biphenyl was observed (data unpublished).

Of the nine chlorinated pesticides, eight lost over 68% and one, endosulfan sulfate, remained unchanged. Again, the loss could be due to decomposition or adsorption to GAC. Volatilization of organic halides during treatment did occur, as evidenced by the recovery of about 18% of organic halides from the GAC trap. Chemical compositions of these organic halides were not analyzed because of the lack of an efficient extraction technique. The adsorbed organic halides most likely were derived from more highly volatile compounds removed during the drying stage, which the condensers were less effective in removing from the vapor phase.

The occurrence of dehalogenation in Stage 1 must be further verified using more carefully designed studies. Chemical destruction may be verified through radiolabel techniques or analyses of nonchlorinated biphenyl and other by-products. The mechanism through which $NaHCO_3$ effects removal must be elucidated; a possible explanation is localized sweeping of volatilized contaminants by CO_2 resulting from $NaHCO_3$ decomposition.

Table 7 presents the metal mass balance. Only 5% of Hg was recovered; the rest either was swept into the GAC trap or was not accurately measured. Analysis of the GAC trap may assist in closing the mass balance. The measured amounts of Ag, As, and Cd in the treated matrix could indicate partial removal but were within the range of possible experimental variations. Two condensate fractions had a small amount of As; vaporization of As was mechanistically plausible if As_2O_3 or other species were present [38]. Physical carryover of some sediment particulates during treatment also could cause the detection of some of the metals in the various condensate fractions.

3.6. Stage 2 treatment effectiveness and mass balance

The Stage 2 process effectively treated PCDDs (except HpCDD and OCDD), PCDFs, and PCBs to below the LAQs (Table 2). Chlorinated pesticides were not quantified, because, if present, they were destroyed by the H_2SO_4 used to remove the matrix effects

Table 7
Metal mass balance in Stage 1 reactor system

Mass flow	Matrix	Weight or volume (g^a or ml^b)	Metal mass (mg)								
			Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
In	Untreated sediment	801.9	11.5	26.5	16.0	245	617	2.2	112	444	1010
Out	Treated sediment	791.5	9.9	23.0	12.7	254	579	0.10	110	419	1010
	Aqueous condensate	1265.0	0.002	ND	ND	ND	0.011	0.007	0.003	0.002	ND
	Yellow emulsion	373.0	ND	0.17	ND	ND	0.001	0.005	0.001	0.001	ND
	Dark brown liquid[c]	908.8	0.001	0.045	ND	ND	0.036	0.003	0.009	0.009	0.046
Recovery (%)			85.6	87.5	79.6	103.9	93.8	4.9	98.0	94.4	100.0

[a] Dry weight of sediments.
[b] Volume of condensates.
[c] Including rinsate.
ND = None detected.

that, otherwise, would have prevented the samples from being analyzed for PCBs. Trace levels of PCBs and chlorinated pesticides were detected in the rinsate, indicating attachment of some of these OCCs to the reactor apparatus. Table 8 shows the mass balance of the OCCs. Assuming no volatilization, > 99.65% of PCDDs/PCDFs and > 97.8% of PCBs, could have been destroyed in Stage 2. Some organic halides had been swept into the off-gas GAC trap, as evidenced by the adsorption of over 8% of the

Table 8
Partitioning and mass balance in Stage 2 reactor system

Mass flow	Matrix	Weight or volume (g^a or ml^b)	PCDDs		PCDFs		PCBs		Organic halides	
			Total mass (pmole)	Mass balance (%)	Total mass (pmole)	Mass balance (%)	Total mass (nmole)	Mass balance (%)	Total mass (μg Cl)	Mass balance (%)
In	Dark brown liquid	6.4	16,469	100.00	4989	100.00	848.9	100.00	7365.8	97.43
	Oil (blank)	86.3	N/A	N/A	N/A	N/A	N/A	N/A	17.7	0.23
	Catalyst	2.1[c]	N/A	N/A	N/A	N/A	N/A	N/A	176.6	2.34
Out	Oily residue	86.3	57.8	0.35	1.4	0.03	ND	0.00	23.36	0.31
	Rinsate	100.0	ND	0.00	ND	0.00	18.7	2.20	360.9	4.77
	GAC trap	9.6[c]	N/A	N/A	N/A	N/A	N/A	N/A	619.7	8.20
Loss (%)				99.65		99.97		97.8		86.72

[a] Weight of oil, oil residue and GAC.
[b] Volume of condensate and rinsate.
[c] Dry weight.
ND = None detected.
N/A = Data not available or not applicable.

organic halides originally present in the reactor. Because the GAC was not analyzed for the OCCs, the extent of OCC volatilization could not be verified.

4. Conclusions

The Stage 1 treatment achieved its goal by reducing all OCCs in the treated matrix to either the LAQs or the LPARs. The treated material also passed the toxicity characteristics leaching procedure (TCLP) test. The clear water condensate and yellow emulsion generated contained some OCCs at levels either below the LPARs or that could be effectively removed by coagulation and/or GAC. Segregation of the clear water condensate from the yellow emulsion would allow treatment of up to 94% of the total condensate produced with GAC alone, which would greatly simplify the treatment process and reduce costs. Except for metals, all OCCs partitioned primarily to a small volume of a dark brown oil (about 4 ml from treating 600 g of sediment with 66.6% water content, or 6.7 l per 1 ton of a similar sediment). Therefore, the Stage 1 treatment also achieved its goal by concentrating OCCs into a small volume of a condensate fraction for subsequent chemical destruction.

The Stage 2 treatment reduced the OCCs (except chlorinated pesticides) to either a nondetectable level (for TCDD) or to levels below the LPARs. Because the Stage 2 treatment process was not optimized, the amount of oily residue produced could not be quantified. Additional studies will be needed to attain this information. Table 9 summarizes the overall mass balance of the OCCs in the two-stage system.

Most metals were retained in the treated matrix, although trace quantities were detected in some condensate fractions, perhaps due to physical carryover of some sediment particulates. There was evidence of Hg and As volatilization, but these metals were most likely captured by the GAC trap. GAC has a strong affinity for Hg vapor and can serve as a filter to remove any As fume. A pilot-scale test may allow a more complete characterization of the final disposition of these metals.

Table 9
Overall mass balance

Contaminant	Total mass partitioned to Stage 1 process sidestreams disposal (%)	Total mass remaining in oily residue (%)	Total mass decomposed by treatment		Total mass partitioned to off-gas GAC trap (%)
			Stage 1 (%)	Stage 2 (%)	
PCDDs	0.5	0.4	0	105	0[a]
PCDFs	0.1	Trace	0	124	0[a]
PCBs	6	0	50	43	0[a]
Pesticides	3	N/A	80	N/A	0[a]
EOX	5	16	15	43	22

[a] Assuming negligible partitioning.
N/A = Data not available.

Although the removal of OCCs has been demonstrated, much more needs to be done before the BCD process can be practically used for sediment treatment. More information is needed to determine if the process can be used cost effectively, especially when used in conjunction with processes that would be required for condensate and off-gas treatment. A preliminary economic analysis indicated a unit cost of about US$100/ton of sediment treated (including capital and operating costs for treating 158,400 tons of sediment per year for 10 years), which is competitive with reported costs of soil washing and the KPEG (potassium polyethylene glycolate) process [39]. The unit costs may increase significantly when treating a smaller amount of sediment.

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