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LIQUIDS CONTAMINATED WITH CHLORINATED ORGANICS

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PERFORMANCE CHARACTERISTICS OF A MULTI-STAGE PROCESS FOR EX-SITU TREATMENT OF SOLIDS OR LIQUIDS CONTAMINATED WITH CHLORINATED ORGANICS

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SUMMARY

The US Environmental Protection Agency has sought to develop soil decontamination technologies which are effective, but which do not involve incineration. This paper presents the results of a study in which a steam-assisted, thermodesorption system utilized on Guam was characterized in terms of its effectiveness for removing PCBs and PCDD/Fs from soil. Data are also presented which are indicative of the performance of the air pollution control system which is directly coupled to the thermodesorber. The potential for formation of PCDD/Fs during thermodesorption is discussed.

KURZFASSUNG

Die US-Behörde fuer Umweltschutz hat nach einer Entwicklung einer wirkungsvollen Technologie zur Bodenaufbereitung gesucht, ohne eine Verbrennung des Bodens herbeizufuehren. Der vorliegende Artikel praesentiert die Ergebnisse einer Studie, in der ein dampfunterstuetztes Thermodesorbitionssystem, angewendet auf Guam, auf seine Effektivitaet untersucht wurde, PCBs und PCDD/Fs aus dem Erdreich zu entfernen. Es werden auch Daten vorgestellt, die fuer die Leistung des an den Thermodesorber, unmittelbar angeschlossenen Luftverschmutzungs-Kontrollsystems kennzeichnend sind. Die Moeglichkeit einer Bildung von PCDD/Fs waehrend der Thermosorbition wird diskutiert.

RE'SUME'

L'Agence Americaine de Protection de l'Environnement (US EPA) cherche a developper des technologies efficaces de decontamination des sols sans incineration. Cet article presente les resultats d'une etude dans laquelle un systeme de thermodesorption assiste par vapeur utilise a Guam est evalue en terme de d'enlevement (removal) des PCBs et PCDD/Fs contenus dans le sol. Des donnees sont aussi presentees et indiquent la performance du systeme de controle de la pollution de l'air. Ce systeme est directement couple a celui de thermodesorption. La possibilite de la formation de PCDD/Fs durant la thermodesorption est discutee.

INTRODUCTION

The United States Environmental Protection Agency's (USEPA's) National Risk Management Research Laboratory (NRMRL) has been involved in developing chemical dechlorination technology for nearly 20 years. Initial efforts focused on developing a process which utilized a reagent consisting of an alkali metal salt of a polyethylene glycol. This process was successfully applied to treat 1-ton batches of soil which was contaminated with several hundred parts-per-million of polychlorinated biphenyls (PCBs)¹. Subsequently, NRMRL personnel developed and patented another chemical dechlorination process termed Base Catalyzed Decomposition (BCD)^{2,3,4}. For treating solids (e.g. soil, sand) the BCD process involves two steps. In the first stage of the process, soil [to which sodium bicarbonate (NaHCO_3) has been added in concentrations ranging from 2% to 12% on a weight:weight basis] is fed into a thermodesorption unit. The soil is heated to 340°C. During heating, the NaHCO_3 may react with halogenated organics such as chlorophenols and PCBs to form partially or totally dechlorinated organic reaction products, plus inorganic chlorides such as sodium chloride. The NaHCO_3 may also assist in breaking up agglomerated soil particles, thus facilitating volatilization of contaminants. The volatilized contaminants and entrained fine particles emanating from the thermodesorption treatment must be captured and subsequently treated in the Second Stage of the BCD Process. In the Second Stage of the BCD process, the collected chlorinated organics are treated using a mixed reagent which includes a base, a hydrogen donor and a catalyst.

In April 1996, the US Navy began utilizing a technology - the Base Catalyzed Decomposition Process (BCDP) - on Guam for treating soil contaminated with polychlorinated biphenyls (PCBs) as well as polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/Fs). This technology is similar to the BCD process developed by the USEPA. The BCDP technology entails heating soil (to which 1% NaHCO_3 has been added) to 300°C in a rotary calciner while injecting steam. The volatilized organics and fine particulates produced during the 1-hour thermodesorption are swept into an Air Pollution Control System (APCS) which removes particulates and organics from the flue gas stream prior to venting to the atmosphere. The treated solids from the thermodesorber are returned to the site or deposited in a landfill. The particulates and liquids collected in the APCS must be subjected to further treatment since these materials typically are contaminated with halogenated organic residues including PCDD/Fs. The Second Stage of the BCD process is not presently used in Guam.

It is generally recognized that thermodesorption can remove many types of organics from soil and similar solids (sand, gravel, sediments) and the effectiveness of the BCDP technology for removing PCBs from Guam soil has been reported^{5,6}. PCDD/Fs are often found in association with PCBs and these compounds, especially the 2,3,7,8-substituted congeners, have been identified as being particularly toxic. PCDDs and more likely PCDFs can be generated during the manufacture of PCBs; in addition, PCDD/Fs can be formed from PCBs under certain conditions^{7,8}. The BCDP system operating on Guam presented an opportunity to not only assess the overall performance of the BCDP system but to obtain data regarding the fate and formation of PCDD/Fs during implementation of the BCDP system. With the cooperation of the US Navy, a plan was formulated for sampling the various process streams associated with the BCDP unit followed by subsequent analyses of these samples to obtain a more complete understanding of the chemistry of soil-bound PCBs and PCDD/Fs during BCDP treatment.

The study had two objectives:

1. Determine the efficiency of the thermodesorption process for removing PCBs and PCDD/Fs from Guam soil
2. Concurrent with Objective 1, determine the location in the APCS where PCBs and PCDD/Fs are being removed.

In the sections which follow, the BCDP Demonstration Unit located in Guam will be described and the results of the sampling and analysis program will be discussed.

DESCRIPTION OF THE BCDP TECHNOLOGY

The BCDP Demonstration Unit^{5,6} includes the Rotary Kiln Reactor (RKR) which is directly coupled with the Air Pollution Control System (APCS). The BCDP unit comprises several modules which are coupled in series. These modules are described below.

Rotary Kiln Reactor System.- The reactor is a rotary calciner consisting of two concentric carbon steel shells. Soil containing 1% sodium bicarbonate is fed into the calciner's inner shell at the rate of 900 to 1800 kg per hour. The inside shell rotates and is heated externally to 315°C to 485°C. Steam, at a flow rate of 48 kg/hr., is injected countercurrently to the soil flow. Volatilized organics and entrained particulates are swept into the APCS by the flow of steam and the slight negative pressure created by the in-line blower positioned just prior to the carbon column in the APCS.

Multiclone (MC).- This unit removes particles by centrifugal force and is electrically heated to minimize condensation.

Wet Electrostatic Precipitator (WESP).- Additional steam, at a flow rate of 265 kg/hr, is injected to prevent formation of an explosive mixture. A charged grid imparts electrostatic energy to suspended particles to promote particle agglomeration and precipitation.

Primary Condenser (PC).- This is a shell-and-tube condenser which cools the gases from 100°C to 30°C. The flow of gases entering the condenser is 3.68 cubic meters per minute and, due to condensation, the gas flow leaving the condenser is 0.85 cubic meters per minute.

High Efficiency Mist Eliminator (HEME).- This module consists of a tightly woven fiberglass pad about 7.6 cm thick. The HEME is designed to remove nearly 100% of the particles >3 micrometers and 99% of the <3 micrometer particles.

Carbon Column.- After the HEME, the gas passes through an induced draft fan. The exhaust is routed through a carbon column to remove residual organic vapors, prior to exhausting to the atmosphere.

FIELD SAMPLING AND LABORATORY ANALYSES

The sampling and analysis program was designed to provide data to be used in assessing the performance of the RKR to remove PCBs and PCDD/Fs from the Guam soil, and to determine the location in the APCS where these compounds are being removed. The various process streams which were sampled and the analyses which were performed are discussed in the paragraphs which follow.

Feed Soil (RKRf), Treated Soil (RKRp), Breach Soil, and Cyclone Fines.- The concentrations of PCBs, PCDD/Fs in the feed soil, treated soil and cyclone fines were quantitated using gas chromatographic/mass spectrometric analytical methods [EPA Method 680 for PCBs⁹ and EPA Method 8290 for PCDD/Fs¹⁰]. The concentrations of these compounds in the treated soil were compared to the concentrations of these compounds in the untreated soil to calculate percentage removals achieved during each of three 6-hour tests. During each test, samples of the treated and untreated soil were taken each hour, then composited to obtain a single sample representing the soil before and after treatment during the six-hour test. Similarly, PCBs and PCDD/Fs were quantitated in the cyclone fines. The breach soil is a mixture of feed soil which drops out of the front end of the kiln and cyclone fines which are collected and ultimately recycled back through the process. The concentration of PCBs in the feed soil and in the cyclone fines were quantitated and the ratio of soil to cyclone fines was calculated and utilized to determine the concentrations of PCDD/Fs in the breach soil.

WESP Blow-down, WESP Makeup Water, and Primary Condensate.- Each of these streams was sampled hourly during each 6-hour run and the samples were then composited to obtain one sample for each stream. The composited liquid samples were analyzed by EPA Method 680 for PCBs and EPA Method 8290 for PCDD/Fs. It was necessary to analyze the WESP makeup water because this is recycled process water and contains the analytes of interest. These data, in conjunction with the other data, provided an indication of the performance of the WESP in removing PCBs and PCDD/Fs from the gas stream.

Gas Sample Before HEME and Gas Sample After HEME.- The gas streams entering the HEME and exiting the HEME were simultaneously sampled and the samples subsequently analyzed using the methodology described in USEPA Method 23¹¹. Gas sampling was performed throughout the duration of each of the three six-hour runs. PCBs and PCDD/Fs were quantitated using gas chromatographic/mass spectrometric methods. The resulting data were used to calculate the efficiency of the HEME for removing the target compounds from the gas stream.

Rationale for Not Assessing the Performance of the Carbon Column.- The efficiency of the carbon column in the APCS was not assessed due to the fact that stack sampling data were available and funding for the present project was limited.

RESULTS AND DISCUSSION

PCBs and PCDD/Fs in Soil Samples Before and After Thermodesorption

Table 1 lists the concentrations of PCBs, PCDDs, and PCDFs found in feed soil (untreated soil) and treated soil for each of the three tests. It should be noted that the table includes the limits of detection for those cases where no PCBs were found and includes the number of isomers in those cases where PCBs were detected. In some cases, the values in the table are flagged due to the fact that high concentrations proved difficult to quantitate accurately.

For PCDDs, comparatively low levels of PCDDs are present in the soil before treatment (values range from 3 ppb to 7 ppb) and concentrations were below detection limits after treatment (refer to RKRp values). Detection limits, however, were not much lower than the range of concentrations before treatment.

Table 1. Concentrations of PCBs, PCDDs, and PCDFs in Untreated (RKRF) and Treated (RKRP) Soil Samples from Base Catalyzed Decomposition Process Operating on Guam^{1,3}

Date Sample Collected in Guam	Sample Number	PCB Concentration in ppb (nanograms per gram)										Total PCB Concentration (ppb)
		MONO	DI	TRI	TETRA	PENTA	HEXA	HEPTA	OCTA	NONA	DECA	
2/25/97	RKRP-1	RO ¹	10(6) ²	40(10)	160(18)	480(22)	1300(23)	460(15)	400(5)	ND[1.0] ³	ND[2.0]	2490
2/25/97	RKRF-1x	ND[20]	ND[20]	670(4)	2600(8)	21,500(15)	94,000(17)	51,700(15)	9800(9)	280(1)	ND[100]	180,550
2/26/97	RKRP-2	ND[1.0]	2(3)	9(7)	40(13)	240(19)	1400(23)	770(18)	110(9)	2(1)	ND[1.0]	2573
2/26/97	RKRF-2x	ND[20.0]	ND[20.0]	80(1)	3400(8)	61,200(16)	283,000(20) y	164,000(19)	30,100(9)	990(2)	ND[90]	543,000
2/27/97	RKRP-3	ND[1.0]	3.0(3)	20(7)	110(18)	780(24)	3600(24) y	1900(18)	270(9)	7.0(2)	ND[1.0]	6700
2/27/97	RKRF-3x	ND[20]	ND[20]	80(1)	3200(7)	58,700(15)	273,000(21) y	158,000(18)	30,000(9)	820(1)	ND[90]	524,000
Date Sample Collected in Guam	Sample Number	PCDDs Concentration in ppb (nanograms per gram)					Total PCDD Concentration (ppb)					
		TETRA	PENTA	HEXA	HEPTA	OCTA						
2/25/97	RKRP-1	ND [1.5]	ND [1.8]	ND [1.8]	ND [2.8]	ND [4.0]	ND					
2/25/97	RKRF-1	0.049 (1)	RO	0.44 (4)	1.42 (2)	4.51	6.42					
2/26/97	RKRP-2	ND [1.5]	ND [2.3]	ND [1.9]	ND [3.4]	ND [4.7]	ND					
2/26/97	RKRF-2	ND [0.02]	ND [0.03]	0.30 (4)	0.81 (2)	2.48	3.58					
2/27/97	RKRP-3	ND [3.6]	ND [4.3]	ND [4.0]	ND [5.2]	ND [5.7]	ND					
2/27/97	RKRF-3	ND [0.02]	ND [0.03]	0.19 (3)	0.74 (2)	2.27	3.20					
Date Sample Collected in Guam	Sample Number	PCDFs Concentration in ppb (nanograms per gram)					Total PCDF Concentration (ppb)					
		TETRA	PENTA	HEXA	HEPTA	OCTA						
2/25/97	RKRP-1x	18.6 (3)	RO B	0.83 (1) B	ND [1.7]	ND [3.5]	19.4					
2/25/97	RKRF-1	32.4 (14)	21.1 (11)	6.76 (7)	2.03 (4)	1.63	63.9					
2/26/97	RKRP-2x	29.9 (4)	21.6 (2)	4.33 (2) B	ND [2.1]	ND [4.1]	55.8					
2/26/97	RKRF-2	16.7 (13)	12.9 (11)	5.56 (8)	7.06 (4)	7.05	49.3					
2/27/97	RKRP-3x	104 (5)	85.4 (5)	22.9 (3)	ND [3.3]	ND [4.9]	212					
2/27/97	RKRF-3	9.64 (16)	9.13 (9)	9.59 (9)	5.07 (3)	5.0	38.4					

¹ RO = peak(s) detected but ratio out (indicating ratios of either the M⁺/M⁺² peaks or the M⁺²/M⁺⁴ peaks fall outside acceptable limits) B = analyte found in blank.

² () = No. of isomers observed

³ [] = detection limit

x - reported data for the sample not recovery corrected

y - concentration of analyte in sample exceeds the upper calibration limit and is therefore considered an estimate

The concentrations of PCDFs are significantly higher than that of PCDDs in all samples. For example, the PCDFs in the reactor feed (RKRF) ranged from 38 to 64 ppb whereas the PCDDs in the same samples ranged from 3 ppb to 7 ppb. Therefore, the ratio of PCDF to PCDD is approximately 10:1 in the RKRF samples. This is as expected since the Guam soil was contaminated with discarded transformer oil, and PCBs are more likely to be converted to PCDFs than PCDDs during heating of transformer oil.

Concentrations of PCDFs in the treated soil deserve special comment. On February 25, the first day of this testing program, the levels of PCDFs in the treated soil were found to be 30% of the concentration of PCDFs in the before-treatment soil. However, in the second test, PCDF concentrations in the treated soil were approximately equal to the levels in the untreated soil. In the third test, PCDF concentrations in the treated soil were 5.5 times the level of PCDF in the untreated soil. Also, the ratio of concentrations of the tetra-octa congener groups changed during treatment. The concentrations of hepta-octa chlorinated PCDFs decreased and the concentrations of tetra-, penta-, and hexa-congeners generally increases after treatment. These results suggest that PCDFs are formed during treatment using stage 1 of the BCD process, i.e., the thermal treatment stage. This result is not surprising considering the reports in the literature^{7,8} which indicate PCBs can give rise to PCDFs especially when air is present, at or near 250°C and up to around 650°C. Air leakage was a problem during these test runs due to worn knife gate valves.

Additional Data

Although a large amount of additional data were obtained, it cannot be included in this paper and will be published elsewhere. Some of the data are summarized in the following table. The International Toxicity Equivalent Values for 2,3,7,8-substituted PCDD/Fs published in 1989¹² were used to calculate Toxicity Equivalent (TEQ) values for feed soil, treated soil, and air samples collected before and after the HEME (and upstream of the final carbon column). These values are shown in Table 2.

Table 2. Toxicity Equivalent Values Calculated on the Basis of PCDD/F Concentrations

Date Sample Collected in Guam	Sample Number	Sample Type	TEQ
25 Feb 97	RKRF-1	Feed soil	3.15 ng/g
25 Feb 97	RKRP-1	Treated soil	1.01 ng/g
25 Feb 97	HEME Outlet	BCDP Exhaust	12.9 ng/m ³
26 Feb 97	RKRF-2	Feed soil	1.75 ng/g
26 Feb 97	RKRP-2	Treated soil	2.02 ng/g
26 Feb 97	HEME Outlet	BCDP Exhaust	1.36 ng/m ³
27 Feb 97	RKRF-3	Feed soil	1.50 ng/g
27 Feb 97	RKRP-3	Treated soil	15.3 ng/g
27 Feb 97	HEME Outlet	BCDP Exhaust	1.72 ng/m ³

The increase in TEQ values for treated soil is due to the increased concentrations of residual PCDFs.

The overall performance of the thermodesorption unit and the APCS are discussed in the section which follows.

Treatment Efficiency of the BCDP Thermodesorption Unit

The performance of the thermodesorption unit of the BCDP during each of the six-hour test runs was characterized by determining the percentage of removal by BCDP treatment of the PCBs, PCDDs, and PCDFs originally present in the Guam soil.

To determine the treatment efficiency, the following formula was developed:

$$\text{Efficiency of Treatment (lb/hr basis), \%} = 100 \times (\text{Target input in feed soil} - \text{target returned}) \div \text{Target input}$$

where:

target = total PCBs, PCDDs, PCDFs, or total 2,3,7,8-substituted PCDD/Fs

target returned = amount of target in treated soil + amount of target in breach soil

The BCDP treatment efficiency for PCBs averaged 96 percent for the three six-hour test runs. This means that 96 percent of PCBs initially present in the soil were removed by BCDP treatment.

In the case of PCDDs, however, the available data could not be utilized to calculate treatment efficiencies. This was due to the fact that no PCDDs were detected in the samples of treated soil and the limits of detection for the PCDDs were higher than the concentration of PCDDs found in samples of the feed soil.

The treatment efficiency values calculated for PCDFs and for total 2,3,7,8-substituted isomers were lower than the values for PCBs and these values decreased from run-to-run to the extent that the third run demonstrated that PCDFs were being formed instead of removed (as evidenced by negative treatment efficiency values). The concentration of PCDFs in the treated soil (product) was over five times higher than the level in the feed for the third test run. Since the furan isomers contributed the most to the total 2,3,7,8-PCDD/F values, the total PCDF and total 2,3,7,8-PCDD/F results follow a similar pattern throughout the data.

Control Efficiency of Air Pollution Control System (prior to the Carbon Column)

Table 3 summarizes the control efficiency of the APCS based on the test results. The control efficiency values shown in this table indicate the fraction of the target compounds entering the unit (total PCBs, PCDDs, PCDFs, or total 2,3,7,8-substituted PCDD/Fs) which were removed by that unit of the APCS expressed as a percentage. For example, a 4 percent control efficiency value for total PCBs on 2/25/97 (see top panel of Table 3) indicates that the multi-cyclone removed 4 percent of the total PCBs emitted by the reactor and entering the multi-cyclone during the 6-hour test run. Similarly, on that same day, the WESP removed 24 percent of the total PCBs which were not captured by the multi-cyclone and therefore entered into the

Table 3. Summary of Control Efficiency by Pollutant

Total PCBs	Unit Air Pollution Control Efficiency %				Overall Removal
Date	Multi-Cyclone	WESP	Primary Condenser	HEME	
2/25/97	4%	24%	99.9%	72%	99.98%
2/26/97	6%	74%	99.9%	85%	99.995%
2/27/97	3%	84%	99.9%	78%	99.996%
Average	4%	61%	99.9%	78%	99.99%
Total PCDD	Unit Air Pollution Control Efficiency %				Overall Removal
Date	Multi-Cyclone	WESP	Primary Condenser	HEME	
2/25/97	25%	65%	98%	73%	99.7%
2/26/97	34%	99%	60%	97%	99.97%
2/27/97	17%	99%	65%	93%	99.95%
Average	25%	88%	74%	88%	99.9%
Total PCDF	Unit Air Pollution Control Efficiency %				Overall Removal
Date	Multi-Cyclone	WESP	Primary Condenser	HEME	
2/25/97	10%	33%	98%	74%	99.7%
2/26/97	59%	95%	81%	98%	99.98%
2/27/97	24%	99%	79%	97%	99.98%
Average	31%	76%	86%	90%	99.9%
Total 2,3,7,8 PCDD/F	Unit Air Pollution Control Efficiency %				Overall Removal
Date	Multi-Cyclone	WESP	Primary Condenser	HEME	
2/25/97	17%	39%	98%	76%	99.7%
2/26/97	62%	97%	81%	98%	99.99%
2/27/97	28%	99%	79%	97%	99.99%
Average	35%	78%	86%	91%	99.9%

WESP. The total target input to the WESP also included the quantities of targets present in the makeup water. The removal efficiencies shown for each unit of the APCS indicate that except for the multi-cyclone, each unit removes a significant percentage of the target species, with the APCS exhibiting excellent overall control efficiencies. In calculating overall control efficiencies, uncontrolled target emissions from the reactor were compared with HEME outlet emissions.

As stated earlier in this report, the carbon column was not included in this study. Terres et al reported⁵⁶ that the dioxin/furan stack emissions (expressed as Toxicity Equivalents) were 0.18 ng per cubic meter.

SUMMARY/CONCLUSIONS

The BCDP technology was found to effectively remove PCBs and achieve the clean level of 2 ppm per PCB congener group using the system operating on Guam. The data for PCDD/Fs indicate that PCDFs apparently form under certain conditions. Although this study did not provide definitive data indicating the cause of formation of PCDD/Fs in the third test, published reports indicate that oxygen levels may play a role.

The APCS was found to perform very well and PCDD/Fs were effectively lowered to concentrations which would readily be removed by carbon column technology. The stack emissions were found to fall well within proposed U.S. EPA guidelines.

Clearly there is a need to treat the solids and liquids removed from the exhaust gas stream by the APCS. This latter treatment can probably be accomplished in one of several ways including the patented Second Stage of the BCD process, incineration, landfilling, or other physical/chemical processes.

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16. ABSTRACT The US Environmental Protection Agency has sought to develop soil decontamination technologies which are effective, but which do not involve incineration. This paper presents the results of a study in which a steam-assisted, thermodesorption system utilized on Guam was characterized in terms of its effectiveness for removing PCBs and PCDD/Fs from soil. Data are also presented which are indicative of the performance of the air pollution control system which is directly coupled to the thermodesorber. The potential for formation of PCDD/Fs during thermodesorption is discussed.					
17. KEY WORDS AND DOCUMENT ANALYSIS					
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