Decontamination Systems Information and Research Program

**Quarterly Report
January 1 - March 31, 1997**

Work Performed Under Contract No.: DE-FC21-92MC29467

For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
P.O. Box 880
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EXECUTIVE SUMMARY

The projects for the 1997 WVU Cooperative Agreement are categorized into the following five focus areas:

- FOCUS AREA 1: Subsurface Contaminants, Containment, and Remediation
- FOCUS AREA 2: Mixed Waste Characterization, Treatment, and Disposal
- FOCUS AREA 3: Decontamination and Decommissioning
- FOCUS AREA 4: Cross-Cutting Innovative Technologies
- FOCUS AREA 5: Outreach

Summaries of the accomplishments for the subtasks reporting under these categories during the first quarter, 1 January 97 through 31 March 97 are presented and discussed below.

FOCUS AREA 1: Subsurface Contaminants, Containment, and Remediation

Subtask 1.1 Characteristics Evaluation of Grout Barriers in Grout Testing Chamber

Progress this quarter continued with testing of urethane and vinyl monomer grouts. Research into the urethane grouts focused on shrink/swell testing and permeability testing. The results have led to modeling the shrink/swell behavior in a grouted sand, and flexible-wall permeability results show no variation in hydraulic conductivity (k) with variation in sample thickness from 1 cm ($k=7 \times 10^{-10}$ cm/s) to 2 cm ($k=9 \times 10^{-10}$ cm/s). This implies that under similar installation conditions the thickness of the barrier in the field can be small, while still functional.

Results of the vinyl monomer grout testing enabled researchers to narrow the field of eight mix ratios to two. Testing will continue with the two mix ratios: Mix 1) 98% gel, 2% accelerator, 89% water, 11% hardening agent, and 2) Mix 2) 97% gel, 3% accelerator, 98% water, 2% hardening agent. From these mix ratios it will be determined which one is more economically feasible for the injection process.

Subtask 1.2   *Development of Standard Test Protocols and Design Models for Desiccation*

Barriers:

The three activities focused on this period consisted of: 1) SARS submission for Phase II experiments, 2) Sand Pack Assembly Characteristics Evaluation, and 3) Sand Pack Drying Characteristics Determination. The SARS for Phase II experimentation in FETC Building B-17 has been prepared. Testing on the Sand Pack Assembly continued with compaction and permeability testing of various size sand and water content combinations. Results of this testing have shown that the air flow rate controls the evaporation rate. This generally occurs within the evaporation front is close to the bottom of the sand pack and sufficient distance exists before exiting the sand pack. The initial water saturation determines how the evaporative front moves through the sand pack. Research will continue during the second quarter focusing on sand pack drying to evaluate the effect of initial water saturation on the drying profile.

Subtask 1.3   *Development of Standard Test Protocols & Design Models for In Situ Formed*

Barriers - Technical Support

This support effort is directed toward establishing DOE/FETC Building B-17 as a center for laboratory, bench-scale, and large-scale experiments to evaluate *in situ* formed barrier systems. The focus of this work is to (1) Support operation of the Circulating air barrier experiments currently installed in B-17, (2) complete scale-up of experiments and support planning for additional barrier systems in 1997. During the first quarter of 1997 work was initiated on development of an operating procedure and permit for the scaled-up circulating air barrier studies in a larger test vessel. Progress is reported with locating the experiments on the ground floor of B-17 and with electronic instrumentation packages used for data collection and verification. The Quality Assurance and Quality Control Guidelines for the scaled-up desiccation barrier were initiated during this quarter. Work planned for the next quarter involves purchasing of new equipment and installation of the large-scale experimental apparatus for the desiccation barrier tests.

Subtask 1.4   *Laboratory Studies and Field Testing at the DOE /RMI Extrusion Plant*
(Ashtabula, Ohio)

This project is part of the on-going efforts in support of the proof-of-concept for the Drain Enhanced Soil Flushing (DESF) technology. Work performed this quarter included completion of the OSHA 1910.120 40 Hour Hazardous Waste Basic Worker Course for nine WVU CEE students and faculty who are involved with the field research activities at the RMI site. Two faculty members from WVU visited the RMI facility in February to observe their soil washing process for removal of uranium particles from site soils.

Efforts on the groundwater modeling have resulted in characterization of the RMI site by completing calibration of the MODFLOW numerical model. Comparisons of the actual observed field recorded data of the groundwater well measurements with the model generated results are presented and discussed. Investigation into the treatability study for surfactant use to remove TCE from contaminated soils have progressed to identification of suitable food-grade surfactants for use at the RMI site. For the next reporting period key areas of research include: contaminant transport modeling of the TCE plume, and design of the TCE treatability experimental apparatus. It is expected that physical sampling of the RMI site soil will take place and for WVU to take possession of the soils for experimental research purposes.

Subtask 1.5   *Drain Enhanced Soil Flushing (DESF) for Organic Contaminants Removal*

Work this period focused on the primary goal of adsorption and desorption kinetic laboratory testing of soil found at the Weston, WV site. This research was conducted to determine how effective the soil was in the adsorption of the chosen model contaminant, ethyl benzene. A secondary goal was initiated to investigate the prefabricated vertical drain performance at the Weston site under liquid injection/extraction operating

conditions. For this series of tests, the drains were installed in site soils and free product, obtained from the site, was cycled through the soil. The drains were operated under injection and extraction modes to determine the degree of filter clogging. This testing is long-term and results are expected to be reported next quarter.

FOCUS AREA 2:   Mixed Waste Characterization, Treatment, and Disposal

Subtask 2.1   Analysis of the Vortec Cyclone Melting System (VMS) for Remediation of PCB Contaminated Soils Using CFD

During this quarterly period the recuperator component of Vortec's Cyclone Melting System was analyzed to determine the influence radiation heat transfer under various flow conditions. Research efforts were focused on the radiation heat transfer analysis of the recuperator component under various flow conditions. Various radiation models implemented in the FLUENT computer code were investigated and the heat transfer rates resulting from different models were evaluated. Calculations were performed to determine the heat transfer rates for two cases of flue gas flow. The results showed that with radiation the heat transfer rate from the flue gas to the air stream increases by approximately 30% with a clear gas and 200% with the gray gas assumptions. The Chemkin II chemical kinetic code has been studied and made operational with test cases being studied. Work planned for the current quarter includes comparison of equilibrium concentrations computed via the Chemkin code.

FOCUS AREA 3:   Decontamination and Decommissioning

Subtask 3.1   Production and Evaluation of Biosorbents and Cleaning Solutions for Use in Decontamination and Decommissioning

During the past quarter surfactant samples were obtained from Witco Chemical Co. And

evaluated for their ability to desorb PAHs from contaminated soil. Several of these commercially available surfactants exhibited good ability to desorb PAHs and show enhanced detergency in the presence of ethanol as a co-solvent. A meeting was held with scientists from Witco to review all of the data which we had obtained regarding the performance of various surfactant products and to make selections of specific surfactant products to be the focus of future work. Samples of lignin products were obtained from Lignotech Co. for evaluation as biosorbents for removal of metals and radionuclides from wastewater. Evaluation tests will continue during the next quarter on actual samples of wastewater obtained from the West Valley Nuclear facility.

Subtask 3.2   *Use of SpinTek Centrifugal Membrane Technology and Sorbent/Cleaning Solutions in the Decontamination and Decommissioning of DOE Facilities*

During this period experimental runs using the SpinTek ultrafiltration system for separating organic contaminant-micelle complex and sorbent-metal complexes were performed. The purpose of these runs were to evaluate the feasibility of using the SpinTek technology and operational conditions for treatment of actual D&D wastewater.

A commercial surfactant manufactured by Witco Co. , Witcamide 5085, was selected as a cleaning agent and naphthalene was selected as a hydrophobic contaminant. The surfactant solution was first treated by the SpinTek system to get a baseline operation. Naphthalene was added to the feed solution and the effect of separation was studied by measuring naphthalene content in permeate and concentrate using the spectrophotometric method. Naphthalene removal of 87% to nearly 100% was achieved when the naphthalene concentration in the feed solution ranged from 190 mg/L to 34 mg/l. As these test results have shown promising results, further experimentation using Witcamide 5085 and naphthalene as the primary surfactant/contaminant combination will be performed.

FOCUS AREA 4:   *Cross Cutting Innovative Technologies*

Subtask 4.1   Use of Centrifugal Membrane Technology with Novel Membranes to Treat Hazardous/Radioactive Wastes

During the first quarter, the effects of permeate back pressure due to membrane rotation on the permeate flux using clean water and oil contaminated feed solutions were examined. A mathematical model used to correct for the average permeate back pressure was tested. It was demonstrated that this correction is an adequate initial model to predict the transmembrane pressure as a function of membrane rotation. These experiments provided baseline membrane performance data over a pressure range of 25 to 75 psig, temperature range of 110 and 135 °F, and rotational speeds of 250, 1250, and 1750 rpm. Permeate flux and turbidity, and permeate/concentrate pH and conductivity were periodically measured.

Experimental results have shown that when the feed water was contaminated with 10 wt. Machine oil, a steady pronounced decrease in the average flux was observed at each gauge pressure and temperature condition. This flux decline with decreasing rotation was consistent with previously observed flux response as a gel layer forms on the membrane surface. Technical difficulties were encountered this period with performance of the system pumps, these included worn mechanical seals and a broken pump impeller. The components were repaired and the system brought back on line.

Subtask 4.2   Environmental Pollution Control Devices Based on Novel Forms of Carbon

Continued work focused on removal of cadmium and lead from aqueous solutions, removal of nickel from an industrial plating rinse solution, and removal of strontium and uranium was demonstrated. Removal efficiencies of over 90% were achieved for the cadmium and lead experiments, and a removal efficiency of 89% was obtained for the

nickel rinse solution. Removal of strontium was demonstrated using carbon nanofibers with efficiencies of 34% being reported. Optimization of the strontium removal increased with the use of magnesium oxide coatings to efficiencies of 68%.

The removal of Uranium was investigated using ASI oxidized nanofibers. This work showed high removal efficiencies due to the production of a precipitate on the electrode surface of approximately 99%. Research continues on this project with further examinations of the cleanup of lead from soil at Fort Benjamin Harrison. On this project, the aqueous waste was purified using the electrolytic cell to remove 95% of the lead from the soil washed solution.

Subtask 4.3   *Design of Rotating Membrane Filtration System for Remediation Technologies*

This project involves the design analysis of the SpinTek Filtration apparatus and to optimize the design in order to enhance the flow rate capacity of the system. During the first quarter several design base models were created and the Finite Element Model software IDEAS was used to analyze the relevant stresses on the disks and the modal vibrations of the entire system. Results indicated that the vibrational stresses developed in the filter apparatus are within the allowable design limits of the material. Continued modeling will be performed to examine all critical areas of the filter apparatus.

FOCUS AREA 5: Outreach

Subtask 5.1 Small Business Technology Base Support

During this reporting period association with RMI Environmental Services and the US DOE Ashtabula Project Office continued. Drs. Cook and Quaranta performed a site visit to RMI Ashtabula in February to observe first-hand demonstration of the RMI soil washing operation. Meetings were held with RMI engineers to continue discussions of where WVU research technologies may be inserted into the RMI remediation processes. Follow-up visits were made by representatives from RMI and DOE - Ashtabula to WVU and FETC in March. The purpose of this visit focused on observing operation of the SpinTek filtration apparatus and meeting with researchers to obtain information regarding this technology and the Prefabricated Vertical Drain operation. Meeting were held with INTERA Corp. and WVU for responding to a Research Opportunity Announcement from FETC.

TABLE OF CONTENTS

Executive Summary

Project Titles with Principal Investigators

Administrative Summary

Budget Updates

Schedule Updates

Quarterly Technical Progress Reports

FOCUS AREA 1.0: SUBSURFACE CONTAMINANTS, CONTAINMENT AND REMEDATION

- 1.1 CHARACTERISTICS EVALUATION OF GROUT BARRIERS IN GROUT TESTING CHAMBER (GTC)
- 1.2 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR DESICCATION BARRIERS
- 1.3 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR IN-SITU FORMED BARRIERS - TECHNICAL SUPPORT
- 1.4 LABORATORY STUDIES AND FIELD TESTING AT THE DOE/RMI EXTRUSION PLANT (ASHTABULA, OHIO)
- 1.5 USE OF DRAINED ENHANCED SOIL FLUSHING (DESF) FOR CONTAMINANTS REMOVAL

TABLE OF CONTENTS (cont.)

FOCUS AREA 2.0: MIXED WASTE CHARACTERIZATION, TREATMENT AND DISPOSAL

- 2.1 ANALYSIS OF THE VORTEC CYCLONE MELTING SYSTEM (CMS) FOR REMEDIATION OF PCB CONTAMINATED SOILS USING COMPUTATIONAL FLUID DYNAMICS (CFD)

FOCUS AREA 3.0: DECONTAMINATION AND DECOMMISSIONING

- 3.1 PRODUCTION AND EVALUATION OF BIOSORBENTS AND CLEANING SOLUTIONS FOR USE IN DECONTAMINATION & DECOMMISSIONING
- 3.2 USE OF SPINTEK CENTRIFUGAL MEMBRANE TECHNOLOGY AND SORBENTS/CLEANING SOLUTIONS IN THE D & D OF DOE FACILITIES

FOCUS AREA 4.0: CROSS-CUTTING INNOVATIVE TECHNOLOGIES

- 4.1 USE OF CENTRIFUGAL MEMBRANE TECHNOLOGY WITH NOVEL MEMBRANES TO TREAT HAZARDOUS/RADIOACTIVE WASTES
- 4.2 ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON
- 4.3 DESIGN OF ROTATING MEMBRANE FILTRATION SYSTEM FOR REMEDIATION TECHNOLOGIES

FOCUS AREA 5.0: OUTREACH

- 5.1 SMALL BUSINESS TECHNICAL BASED SUPPORT

PROJECT TITLE WITH PRINCIPLE INVESTIGATORS

FOCUS AREA 1.0: SUBSURFACE CONTAMINANTS, CONTAINMENT AND REMEDATION

1.1 CHARACTERISTICS EVALUATION OF GROUT BARRIERS IN GROUT TESTING CHAMBER

M. Gabr, Department of Civil & Environmental Engineering

1.2 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR DESICCATION BARRIERS

S. Ameri and K. Aminian, Department of Petroleum Engineering

1.3 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR IN-SITU FORMED BARRIERS - TECHNICAL SUPPORT

B. Overbey and D. Locke, BDM Federal

1.4 LABORATORY STUDIES AND FIELD TESTING AT THE DOE/RMI EXTRUSION PLANT (ASHTABULA, OHIO)

E. Cook and J. Quaranta, Department of Civil & Environmental Engineering
W. Best, US DOE/RMI Decommissioning Project
J. Henderson, RMI Environmental Services, Inc.

1.5 USE OF DRAINED ENHANCED SOILED SLUSHING (DESF) FOR CONTAMINANTS REMOVAL

M. Gabr, Department of Civil & Environmental Engineering
J. Cramer, Nilex Corporation
R. Freemant & G. Smith, Ashland Petroleum

PROJECT TITLES WITH PRINCIPAL INVESTIGATORS (cont.)

FOCUS AREA 2.0: MIXED WASTE CHARACTERIZATION TREATMENT AND DISPOSAL

- 2.1 ANALYSIS OF THE VORTEC CYCLONE MELTING SYSTEM (CMS) FOR
REMEDICATION OF PCB CONTAMINATED SOILS USING COMPUTATIONAL
FLUID DYNAMICS (CFD)

I. Celik, Department of Mechanical & Aerospace Engineering
J. Gnat and J. Santoriani, Vortec Corporation

FOCUS AREA 3.0: DECONTAMINATION AND DECOMMISSIONING

- 3.1 PRODUCTION AND EVALUATION OF BIOSORBENTS AND CLEANING
SOLUTIONS FOR USE IN DECONTAMINATION & DECOMMISSIONING

J. Kilbane and V. Srivastava, IGT

- 3.2 USE OF SPINTEK CENTRIFUGAL MEMBRANE TECHNOLOGY AND
SORBENTS/CLEANING SOLUTIONS IN THE D & D OF DOE FACILITIES

B. Reed and W. Lin, Department of Civil & Environmental Engineering
W. Green and R. Hayes, SPINTEK

PROJECT TITLES WITH PRINCIPAL INVESTIGATORS (cont.)

FOCUS AREA 4.0: CROSSCUTTING INNOVATIVE TECHNOLOGIES

4.1 USE OF CENTRIFUGAL MEMBRANE TECHNOLOGY WITH NOVEL MEMBRANES TO TREAT HAZARDOUS/RADIOACTIVE WASTES

B. Reed and W. Lin, Department of Civil & Environmental Engineering
W. Green and R. Hayes, SPINTEK

4.2 ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON

J. Zondlo, A. Stiller, and A. Brennsteiner
Department of Chemical Engineering
B. Alig, Applied Sciences, Inc.

4.3 DESIGN OF ROTATING MEMBRANE FILTRATION SYSTEM FOR REMEDIATION TECHNOLOGIES

V. Mucino, Department of Mechanical & Aerospace Engineering

FOCUS AREA 5.0: OUTREACH

5.1 SMALL BUSINESS TECHNICAL BASED SUPPORT

E. Cook, Department of Civil & Environmental Engineering

ADMINISTRATIVE ISSUES

Administrative Summary

Background

West Virginia University (WVU) and the US Department of Energy, Federal Energy Technology Center (DOE/FETC) entered a Cooperative Agreement in August 1992 titled "Decontamination Systems Information and Research Programs" (DOE Instrument No.: DE-FC21-92MC29467). Requirements stipulated by the Agreement require WVU to submit quarterly Technical Progress reports. This report contains the efforts of the research projects comprising the Agreement for the first calendar quarter of 1997.

Administrative Issues

Currently, twelve active projects make up the 1997 WVU Cooperative Agreement. They are divided into the following five WVU focus areas:

Focus Area 1.0: Subsurface Contaminants. Containment and Remediation

Focus Area 2.0: Mixed Waste Characterization, Treatment and Disposal

Focus Area 3.0: Decontamination & Decommissioning

Focus Area 4.0: Cross Cutting Innovative Technologies

Focus Area 5.0: Outreach

All of the 1997 projects, except Task 1.4 (Laboratory Studies and Field Testing at the DOE/RMI Extrusion Plant) and Task 4.3 (Design of Rotating Membrane Filtration System for Remediation Technologies), are a continuation from 1996. Those projects from 1996 not selected for continued funding submitted their final draft reports this quarter.

The submittal for this quarter includes the project reports in PDF format for FETC's use and is submitted on an attached diskette in ZIP file compression format.

Accounting Issues

A semi-accrued accounting system has been adopted for reporting expenditures to reflect a timely accounting of the awarded funds. Each project's report contains a column for accrued expenses and obligated amounts that have not been disbursed by the WVU controller's office. All accrued adjustments, except subcontractor expenses, are made in this column. Subcontractor expenses are reported on a cash basis when received by our office.

For the first calendar quarter of 1997 the following accrued adjustments were made to the financial status report prepared by the WVU Research Corporation's - Sponsored Research Accounting Department:

Total Outlays to Date:	\$ (5,038)
Unliquidated Obligations:	296,224

TOTAL ADJUSTMENT: \$ 291,186

Approximately 1.6 million is budgeted for calendar year 1997. This includes the rollover balance from 1997. A detailed summary of the funds allocated per project is included in the budget section of this report.

BUDGETS

ACCRUED ACCOUNTING OF AWARDED FUNDS

Period Covered 1/1/97 - 3/31/97

REPORT DATE: April 30, 1997

OUTLAYS**PREVIOUS OUTLAYS & ADJUSTMENTS (Grant Start - 3/31/96)****FUND 7631 - NRCCE**

Previous Outlays	3,525,462	
Current Adjustments	<u>0</u>	3,525,462

FUND 8527 - WVU

1995 Outlays	2,594,205	
Current Adjustments	<u>(160)</u>	2,594,045

1996 Outlays	2,311,735	
Current Adjustments	<u>36,851</u>	2,348,586

TOTAL PREVIOUS OUTLAYS	8,468,093

1997 CURRENT OUTLAYS (1/1/97 - 3/31/97)	<u>148,144</u>

<u>TOTAL OUTLAYS TO DATE 3/31/97</u>	8,616,237

<u>UNLIQUIDATED OBLIGATIONS</u>	<u>430,013</u>

<u>TOTAL SHARE OF OUTLAYS & UNLIQUIDATED OBLIGATIONS</u>	9,046,250

FUNDS AUTHORIZED

FUND 7631 (Prior to 1995)	4,749,048

FUND 8527

1995 Award	1,547,000	
Amendment No. A013	<u>75,950</u>	1,622,950

1996 Award	1,900,000	
Amendment No. A015	<u>361,605</u>	2,261,605

1997 Award	<u>1,500,000</u>	1,500,000	5,384,555

<u>TOTAL FUNDS AUTHORIZED TO DATE</u>	<u>10,133,603</u>

<u>AMOUNT REMAINING</u>	<u>\$1,087,353</u>

1997 TASK #	PROJECT TITLE	PLANNED ACTUAL	TOTAL PLANNED	TOTAL ACTUAL	Obligated Encumbrances	AMOUNT REMAINING
FOCUS AREA 1.0: SUBSURFACE CONTAMINANTS, CONTAINMENT & REMEDIATION						
1.1	Characteristics Evaluation of Grout Barriers in Grout Testing Chamber (GTC)	PLANNED ACTUAL	61,522	11,821	4,983	44,718
1.2	Development of Standard Test Protocols and Barrier Design Models for Desiccation Barriers	PLANNED ACTUAL	154,998	15,361	21,310	118,327
1.3	Development of Standard Test Protocols and Barrier Design Models for In Situ Barriers - Technical Support	PLANNED ACTUAL	283,485	30,108	242,368	11,009
1.4	Laboratory Studies and Field Testing at the DOE/EMI Extrusion Plant (Ashtabula, Ohio)	PLANNED ACTUAL	150,000	2,236	34,109	113,655
1.5	Use of Drained Enhanced Soil Flushing (DESF) for Contaminants Removal	PLANNED ACTUAL	148,461	18,016	23,678	106,767
		PLANNED ACTUAL	0	0	0	0
TOT 1.0	TOTAL FOCUS AREA 1.0	PLANNED ACTUAL	798,466	77,542	326,448	394,476
FOCUS AREA 2.0: MIXED WASTE CHARACTERIZATION, TREATMENT AND DISPOSAL						
2.1	Analysis of the Vortec Cyclone Melting System (CMS) for Remediation of PCB Contaminated Soils Using Computational Fluid Dynamics (CFD)	PLANNED ACTUAL	76,994	9,295	4,719	62,980
		PLANNED ACTUAL	0	0	0	0
TOT 2.0	TOTAL FOCUS AREA 2.0	PLANNED ACTUAL	76,994	9,295	4,719	62,980
FOCUS AREA 3.0: DECONTAMINATION AND DECOMMISSIONING						
3.1	Production and Evaluation of BioSorbents and Cleaning Solutions for Use in D & D	PLANNED ACTUAL	61,851	4,926	46,620	10,305
3.2	Use of SpinTek Centrifugal Membrane Technology and Sorbents/Cleaning Solutions in the D & D of DOE Facilities	PLANNED ACTUAL	65,353	8,656	4,705	51,992
		PLANNED ACTUAL	0	0	0	0
TOT 3.0	TOTAL FOCUS AREA 3.0	PLANNED ACTUAL	127,204	13,582	51,325	62,297
FOCUS AREA 4.0: CROSS CUTTING INNOVATIVE TECHNOLOGIES						
4.1	Use of Centrifugal Membrane Technology with Novel Membranes to Treat Hazardous/Radioactive Waste	PLANNED ACTUAL	180,775	11,503	5,684	163,588
4.2	Environmental Pollution Control Devices Based on Novel Forms of Carbon	PLANNED ACTUAL	133,875	25,632	24,930	83,313
4.3	Design of a Rotating Membrane Filtration System for Remediation Technologies	PLANNED ACTUAL	65,906	6,176	3,345	56,385
TOT 4.0	TOTAL FOCUS AREA 4.0	PLANNED ACTUAL	380,556	43,311	33,959	303,286
FOCUS AREA 5.0: OUTREACH						
5.1	Small Business Technical Based Support	PLANNED ACTUAL	272,094	4,414	3,505	264,175
Unsettled Accounts						139
GRAND TOTALS			PLANNED ACTUAL	1,655,314	148,144	419,956
						1,087,353

Summary of Planned Versus Actual Expenses

1997 WVU Cooperative Agreement

■ Planned Expenditures * Actual Expenditures

[illegible]

1997	PLANNED	1	2	3	4	5	6	7	8	9	10	11	12	TOTAL	TOTAL	
TASK #	ACTUAL	JAN	FEB	MAR	APR	MAY	JUNE	JULY	AUG	SEPT	OCT	NOV	DEC	PLANNED	ACTUAL	
FOCUS AREA 4.0: CROSS CUTTING INNOVATIVE TECHNOLOGIES																
4.1	PLANNED	4,648	4,648	8,885	66,107	10,073	14,754	17,654	13,688	10,437	9,722	9,722	10,437	180,775	11,503	
	ACTUAL	3,790	3,923	3,790												
4.2	PLANNED	8,701	13,861	12,882	10,131	10,812	12,958	13,908	11,798	9,724	9,688	9,688	9,724	133,875	25,632	
	ACTUAL	8,236	8,883	8,513												
4.3	PLANNED	1,287	3,287	3,555	2,287	5,773	11,526	12,909	8,424	4,491	3,938	3,938	4,491	65,906	6,176	
	ACTUAL	1,287	1,287	3,602												
TOT	PLANNED	14,636	21,796	25,322	78,525	26,658	39,238	44,471	33,910	24,652	23,348	23,348	24,652	380,556	43,311	
4.0	ACTUAL	13,313	14,093	15,905	0	0	0	0	0	0	0	0	0			
5.1	PLANNED	1,582	3,012	1,653	18,898	16,332	15,612	56,637	47,682	91,189	9,517	7,969	2,011	272,094	4,414	
	ACTUAL	1,349	1,635	1,430												
Unsettled Accounts																
GRAND TOTALS																
	PLANNED	48,103	70,477	97,051	178,844	224,731	189,708	219,509	179,802	180,373	92,601	88,148	85,967	1,655,314	148,144	
	ACTUAL	40,874	47,781	59,489	0	0	0	0	0	0	0	0	0			

General Note: The above monthly numbers are rounded to the nearest dollar.
Detailed information per project is included on the following pages.

FOCUS AREA 1.0

Subsurface Contaminants,
Containment & Remediation

PROJECT: Characteristics Evaluation of Grout Barriers in GTC

1997 TASK NO.: 1.1

*** Summary ***
on/off Campus

PRINCIPAL INVESTIGATOR: M.A. Gabr

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
M.A. Gabr	planned	0	0	0	0	1,050	2,100	2,100	1,050	0	0	0	0	\$6,300			
	actual	0	0	0											\$0	0	6,300
Other	planned	0	0	0	0	0	0	0	0	0	0	0	0	\$0			
	actual	0	0	0											\$0	0	0
BENEFITS																	
29% of Full Time Salaries	planned	0	0	0	0	305	609	609	305	0	0	0	0	\$1,827			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		\$0	0	1,827
STUDENTS																	
Graduate	planned	2,550	2,550	2,550	2,550	2,125	1,700	1,700	1,700	1,700	1,700	1,700	1,700	\$24,225			
	actual	2,550	2,550	2,550											\$7,650	3,825	12,750
Undergraduate	planned	375	375	375	375	375	375	375	375	375	375	375	375	\$4,495			
	actual	270	180	0											\$450	0	4,045
SUPPLIES	planned	300	300	300	300	700	650	650	650	650	650	650	700	\$6,500			
	actual	0	189	611											\$800	0	5,700
TRAVEL	planned	0	1,000	0	0	1,000	0	0	500	0	0	0	0	\$2,500			
	actual	0	0	0											\$0	0	2,500
OTHER	planned	0	0	25	25	75	50	50	50	50	50	75	50	\$500			
	actual	0	0	0											\$0	0	500
EQUIPMENT < \$1,000	planned	0	0	0	0	0	0	0	0	0	0	0	0	\$0			
	actual		710	0											\$710	227	(937)
* > \$1,000	planned	0	1,000	1,000	1,000	0	0	0	0	0	0	0	0	\$3,000			
	actual	0	0	0											\$0	0	3,000
INDIRECT COSTS																	
Variable 23% / 43%	planned	742	972	747	747	1,520	1,617	1,617	1,285	723	723	734	744	\$12,171			
	actual	649	835	727											\$2,211	932	9,028
PLANNED TOTALS: PER MONTH																	
		3,967	6,197	4,997	4,997	7,149	7,101	7,101	5,914	3,498	3,498	3,534	3,569	\$61,518			
ACTUAL TOTALS: PER MONTH																	
		3,469	4,464	3,888	0	0	0	0	0	0	0	0	0		\$11,821	4,983	44,713
CUMULATIVE																	
PLANNED TOTALS		3,967	10,163	15,160	20,156	27,305	34,406	41,507	47,421	50,918	54,416	57,949	61,518	\$61,518			
ACTUAL TOTALS		3,469	7,933	11,821	11,821	11,821	11,821	11,821	11,821	11,821	11,821	11,821	11,821		\$11,821		44,713
Budget Variance		\$498	\$2,230	\$3,339	\$8,335	\$15,484	\$22,585	\$29,685	\$35,599	\$39,097	\$42,595	\$46,128	\$49,697				

1997 Task 1.1: Evaluation of Grout Barriers in GTC

PRINCIPAL INVESTIGATOR: M.A. Gabr

**PROJECT: Development of Standard Test Protocols and Design Models
for Desiccation Barriers**

1997 TASK NO.: 1.2

***Summary on/off campus

PRINCIPAL INVESTIGATOR (S): K. Aminian & S. Ameri

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
K. Aminian	planned actual	0 0	0 0	0 0	0 0	2,568 0	5,135 0	5,135 0	2,568 0	0 0	0 0	0 0	0 0	15,406 0	0	0	15,406
S. Ameri	planned actual	0 0	0 0	0 0	0 0	2,246 0	4,493 0	4,493 0	2,246 0	0 0	0 0	0 0	0 0	13,478 0	0	0	13,478
Technician	planned actual	2,571 0	2,571 0	2,571 3,750	2,571 0	2,571 0	2,571 0	2,571 0	2,571 0	2,571 0	2,571 0	2,571 0	2,571 0	30,846 3,760	3,760	7,500	19,596
BENEFITS																	
29% of Full Time Salaries	planned actual	745 0	745 0	745 1,088	745 0	2,142 0	3,538 0	3,538 0	2,142 0	745 0	745 0	745 0	745 0	17,322 1,088	1,088	2,175	14,059
STUDENTS																	
Graduate	planned actual	2,550 2,550	2,550 2,550	2,550 2,550	2,550 0	2,550 0	2,550 0	3,450 0	3,450 0	3,450 0	3,450 0	3,450 0	3,450 0	36,000 7,650	7,650	7,650	20,700
SUPPLIES																	
	planned actual	25 0	25 0	25 0	75 0	50 0	50 0	50 0	50 0	50 0	50 0	25 0	25 0	500 0	0	0	500
TRAVEL																	
	planned actual	0 0	1,000 0	0 0	1,000 0	0 0	1,000 0	0 0	1,000 0	0 0	1,000 0	0 0	0 0	5,000 0	0	0	5,000
OTHER																	
	planned actual	0 0	0 0	25 0	50 0	50 0	50 0	50 0	50 0	50 0	50 0	50 0	75 0	500 0	0	0	500
EQUIPMENT																	
< \$1,000	planned actual	0 0	1,000 0	1,000 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	2,000 0	0	0	2,000
* > \$1,000	planned actual	0 0	1,500 0	1,000 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	2,500 0	0	0	2,500
INDIRECT COSTS																	
Variable 23% / 43%	planned actual	1,355 587	1,816 587	1,591 1,699	1,623 0	3,394 0	5,636 0	5,613 0	3,831 0	1,590 0	1,820 0	1,584 0	1,594 0	31,448 2,872	2,872	3,985	24,589
PLANNED TOTALS: PER MONTH		7,246	11,206	9,507	8,614	15,570	25,022	24,899	17,907	8,456	9,686	8,425	8,460	154,998			
ACTUAL TOTALS: PER MONTH		3,137	3,137	9,087	0	0	0	0	0	0	0	0	0	15,360	15,360	21,310	118,329
CUMULATIVE																	
PLANNED TOTALS		7,246	18,452	27,959	36,573	52,143	77,165	102,064	119,971	128,427	138,113	146,538	154,998	154,998			
ACTUAL TOTALS		3,137	6,273	15,360	15,360	15,360	15,360	15,360	15,360	15,360	15,360	15,360	15,360	15,360	15,360		118,329
Budget Variance		4,109	12,179	12,599	21,213	36,783	61,805	86,705	104,612	113,068	122,754	131,179	139,639				

1997 Task 1.2: Standard Test Protocols for Desiccation Barriers

PRINCIPAL INVESTIGATOR (S): K. Aminian & S. Ameri

◆ PLANNED SPENDING * ACTUAL SPENDING

PROJECT: Technical Support for In Situ Formed Barriers

1997 TASK NO.: 1.3

PRINCIPAL INVESTIGATOR: E. Cook - WVU
B. Overby & D. Locke (BDM Federal)

WVU ACCOUNT NO.: 8527-090-3278

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Management	planned	5,609	5,609	5,609	5,609	5,609	5,609	1,171	1,171	1,171	1,171	1,171	1,171	40,677			
	actual	5,609	5,609	5,103											16,321	18,388	5,968
BENEFITS																	
29% of Full Time Salaries	planned	1,627	1,627	1,627	1,627	1,627	1,627	339	339	339	339	339	339	11,796			
	actual	1,627	1,627	1,480	0	0	0	0	0	0	0	0	0		4,733	5,333	1,731
SUB-CONTR. > \$25,000	planned	0	0	0	0	86,852	17,371	17,371	17,371	17,371	17,371	17,371	17,371	208,447			
	actual														0	208,447	0
INDIRECT COSTS																	
43% of Direct Costs except	planned	3,111	3,111	3,111	3,111	3,111	3,111	649	649	649	649	649	649	22,564			
	actual	3,111	3,111	2,831	0	0	0	0	0	0	0	0	0		9,053	10,200	3,310
PLANNED TOTALS: PER MONTH		10,347	10,347	10,347	10,347	97,199	27,718	19,530	19,530	19,530	19,530	19,530	19,530	283,484			
ACTUAL TOTALS: PER MONTH		10,347	10,347	9,414	0	0	0	0	0	0	0	0	0		30,107	242,368	11,009
CUMULATIVE																	
PLANNED TOTALS		10,347	20,694	31,041	41,388	138,587	166,304	185,834	205,364	224,894	244,424	263,954	283,484	283,484			
ACTUAL TOTALS		10,347	20,694	30,107	30,107	30,107	30,107	30,107	30,107	30,107	30,107	30,107	30,107		30,107		11,009
Budget Variance		0	0	933	11,280	108,479	136,197	155,727	175,257	194,787	214,317	233,847	253,377				

1997 Task 1.3 - Technical Support for In Situ Formed Barriers

PRINCIPAL INVESTIGATOR (S): B. Overby & D. Lock (BDM Federal), E. Cook (WVU)

◆ PLANNED SPENDING * ACTUAL SPENDING

PROJECT: Field Testing at the DOE/RMI Extrusion Plant (Ashtabula, Ohio)

1997 TASK NO.: 1.4

*** Summary
on/off Campus

PRINCIPAL INVESTIGATOR: J. Quaranta

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Faculty	planned actual	0 0	0 0	0 1,125	2,642	2,642	2,642	2,642	2,642	2,642	0	0	0	15,852			
															1,125	7,875	6,852
Other	planned actual	0 0	0 0	0 0	0	0	0	0	0	0	0	0	0	0	0	0	0
BENEFITS																	
29% of Full Time Salaries	planned actual	0 0	0 0	0 326	766	766	766	766	766	766	0	0	0	4,597			
															326	2,284	1,987
STUDENTS																	
Graduate	planned actual	0 0	0 0	1,800 306	1,450	1,750	2,050	2,050	2,050	2,900	2,900	2,900	2,900	22,750			
															306	2,394	20,050
Undergraduate	planned actual	0 0	0 0	0 0	130	130	130	130	234	234	234	234	234	1,690			
															0	0	1,690
SUPPLIES	planned actual	0 0	0 0	750 61	2,250	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	15,000			
															61	4	14,935
TRAVEL	planned actual	0 0	500 0	500 0	500	500	500	500	500	500	500	250	250	5,000			
															0	175	4,825
OTHER	planned actual	0 0	0 0	100 0	200	200	200	200	200	200	200	200	300	2,000			
															0	0	2,000
EQUIPMENT < \$1,000	planned actual	0 0	0 0	1,000 0	1,000	1,000	1,000	0	0	0	0	0	0	4,000			
															0	0	4,000
* > \$1,000	planned actual	0 0	0 0	0 0	0	0	0	0	0	0	0	0	0	0			
															0	0	0
SUB-CONTR. < \$25,000	planned actual	0 0	0 0	0 0	10,000	10,000	5,000	0	0	0	0	0	0	25,000			
															0	15,000	10,000
> \$25,000	planned actual	0 0	0 0	0 0	0	0	5,000	10,000	10,000	875	0	0	0	25,875			
															0	0	25,875
INDIRECT COSTS																	
Variable 23% / 43%	planned actual	0 0	115 0	955 418	5,212	4,958	3,877	2,497	2,521	2,887	1,762	1,705	1,748	28,237			
															418	6,378	21,441
PLANNED TOTALS: PER MONTH		0	615	5,105	24,150	23,446	22,655	20,285	20,413	12,504	7,096	6,789	6,932	150,001			
ACTUAL TOTALS: PER MONTH		0	0	2,236	0	0	0	0	0	0	0	0	0		2,236	34,109	113,655
CUMULATIVE																	
PLANNED TOTALS		0	615	5,720	29,870	53,316	75,982	95,267	116,680	129,184	136,280	143,069	150,001	150,001			
ACTUAL TOTALS		0	0	2,236	2,236	2,236	2,236	2,236	2,236	2,236	2,236	2,236	2,236		2,236		113,655
Budget Variance		0	615	3,484	27,634	51,080	73,745	94,030	114,443	126,948	134,044	140,833	147,765				

1997 Task 1.4: Lab & Field Studies - DOE/RMI Extrusion Plant

PRINCIPAL INVESTIGATOR (S): E. Cook & J. Quaranta

PROJECT: Use of Drained Enhanced Soil Flushing (DESF) for Contaminant Removal

1997 TASK NO.: 1.5

PRINCIPAL INVESTIGATOR: M.A. Gabr

***Summary on/off Campus

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
M.A. Gabr	planned actual	0 0	0 0	0 556	0	2,040	4,080	4,080	2,040	0	0	0	0	12,240	556	7,816	3,869
Research Associate	planned actual	0 0	0 0	2,000 2,084	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	2,000	20,000	2,084	3,126	14,790
BENEFITS																	
29% of Full Time Salaries	planned actual	0 0	0 0	580 765	580 0	1,172 0	1,763 0	1,763 0	1,172 0	580 0	580 0	580 0	580 0	9,350	765	3,173	5,411
STUDENTS																	
Graduate	planned actual	3,400 3,400	3,400 3,400	5,100 3,400	5,100	3,400	2,550	2,550	2,125	1,700	1,700	1,700	1,700	34,425	10,200	5,100	19,125
Undergraduate	planned actual	0 0	0 0	246 0	246	246	246	246	246	755	755	755	755	4,496	0	0	4,496
SUPPLIES																	
	planned actual	750 0	750 644	1,650 10	1,650	1,650	1,650	1,650	1,650	1,650	1,650	1,650	1,650	18,000	654	0	17,346
TRAVEL																	
	planned actual	0 0	1,000 0	0 389	1,000	0	1,000	0	1,000	0	1,000	0	0	5,000	389	35	4,577
OTHER																	
	planned actual	0 0	0 0	50 0	50	50	50	50	50	50	50	50	50	500	0	0	500
EQUIPMENT																	
< \$1,000	planned actual	0 0	500 0	500 0	200	0	0	0	0	0	0	0	0	1,200	0	0	1,200
> \$1,000	planned actual	0 0	1,000 0	1,000 0	1,000	0	0	0	0	0	0	0	0	3,000	0	0	3,000
Rent																	
	planned actual	0 0	0 0	500 0	500	500	500	500	500	500	500	500	500	5,000	0	0	5,000
INDIRECT COSTS																	
Variable 23% / 43%	planned actual	955 782	1,300 930	3,276 1,657	3,437	3,639	4,542	4,312	3,575	2,496	2,726	2,496	2,496	35,250	3,369	4,428	27,453
PLANNED TOTALS: PER MONTH		5,105	7,950	14,902	15,763	14,697	18,381	17,151	14,358	9,731	10,961	9,731	9,731	148,461			
ACTUAL TOTALS: PER MONTH		4,182	4,974	8,860	0	0	0	0	0	0	0	0	0		18,017	23,678	106,766
CUMULATIVE																	
PLANNED TOTALS		5,105	13,055	27,957	43,720	58,417	76,798	93,949	108,307	118,038	128,999	138,730	148,461	148,461			
ACTUAL TOTALS		4,182	9,156	18,017	18,017	18,017	18,017	18,017	18,017	18,017	18,017	18,017	18,017		18,017		106,766
Budget Variance		923	3,899	9,940	25,703	40,400	58,781	75,932	90,290	100,021	110,982	120,713	130,444				

1997 Task 1.5: DESF for Contaminants Removal
PRINCIPAL INVESTIGATOR: M.A. Gabr

FOCUS AREA 2.0

Mixed Waste Characterization,
Treatment & Disposal

**PROJECT: Analysis of the Vortec Cyclone Melting System (CMS) for
Remediation of PCB Contaminated Soils Using CFD: Phase II**

1997 TASK NO.: 2.1

WVU ACCOUNT NO.: 8527-093-3576

PRINCIPAL INVESTIGATOR: I. Celik

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
I. Celik	planned	0	0	0	0	3,454	6,908	6,908	3,454	0	0	0	0	20,724			
	actual	0	0	0											0	0	20,724
Management	planned	0	0	3,910	1,303	1,303	1,303	0	0	0	0	0	0	7,820			
	actual	0	0	0											0	0	7,820
BENEFITS																	
29% of Full Time Salaries	planned	0	0	1,134	378	1,380	2,381	2,003	1,002	0	0	0	0	8,278			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0	0	8,278
STUDENTS																	
Graduate	planned	2,100	2,100	2,100	1,050	1,050	1,050	1,050	1,050	1,050	0	0	0	12,600			
	actual	2,100	2,200	2,200											6,500	3,300	2,800
Undergraduate	planned	0	0	0	0	0	0	0	0	0	307	307	307	920			
	actual	0	0	0											0	0	920
SUPPLIES	planned	0	40	140	140	140	140	140	140	140	140	140	100	1,400			
	actual	0	0	0											0	0	1,400
TRAVEL	planned	0	500	0	500	0	500	0	500	0	0	0	0	2,000			
	actual	0	0	0											0	0	2,000
OTHER	planned	0	0	25	0	0	25	0	0	25	0	0	25	100			
	actual	0	0	0											0	0	100
INDIRECT COSTS																	
43% of Direct Costs except *	planned	903	1,135	3,143	1,450	3,151	5,292	4,344	2,643	522	192	192	186	23,152			
	actual	903	946	946	0	0	0	0	0	0	0	0	0		2,795	1,419	18,938
PLANNED TOTALS: PER MONTH																	
		3,003	3,775	10,452	4,821	10,478	17,600	14,445	8,788	1,737	639	639	617	76,994			
ACTUAL TOTALS: PER MONTH																	
		3,003	3,146	3,146	0	0	0	0	0	0	0	0	0		9,295	4,719	62,980
CUMULATIVE																	
PLANNED TOTALS																	
		3,003	6,778	17,230	22,051	32,528	50,128	64,573	73,362	75,099	75,738	76,376	76,994	76,994			
ACTUAL TOTALS																	
		3,003	6,149	9,295	9,295	9,295	9,295	9,295	9,295	9,295	9,295	9,295	9,295		9,295		62,980
Budget Variance																	
		0	629	7,935	12,756	23,233	40,833	55,278	64,067	65,804	66,443	67,081	67,699				

1997 Task 2.1: Vortec CMS for Remediation of PCB Soils

PRINCIPAL INVESTIGATOR: I. Celik

◆ PLANNED SPENDING * ACTUAL SPENDING

FOCUS AREA 3.0

Decontamination & Decommissioning

**PROJECT: Production and Evaluation of BioSorbents and Cleaning Solutions
for Use in Decontamination and Decommissioning**

1997 TASK NO.: 3.1

WVU ACCOUNT NO.: 8527-090-32

**PRINCIPAL INVESTIGATOR: J. Kilbane (IGT)
B. Reed (WVU)**

SUMMARY

Sub Contractor detail follows

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Management	planned	0	0	0	621	621	621	621	621	621	621	621	621	5,586	0	0	5,586
	actual	0	0	0											0		
BENEFITS																	
29% of Full Time Salaries	planned	0	0	0	180	180	180	180	180	180	180	180	180	1,620	0	0	1,620
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0		
SUB-CONTR.																	
* >\$25,000	planned	0	2,362	4,366	6,021	3,804	4,678	4,678	4,678	4,678	4,678	4,678	6,925	51,546			
	actual	0	2,909	2,017											4,926	46,620	0
INDIRECT COSTS																	
43% of Direct Costs except *	planned	0	0	0	344	344	344	344	344	344	344	344	344	3,099			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0	0	3,099
PLANNED TOTALS: PER MONTH																	
		0	2,362	4,366	7,166	4,949	5,823	5,823	5,823	5,823	5,823	5,823	8,070	61,850			
ACTUAL TOTALS: PER MONTH																	
		0	2,909	2,017	0	0	0	0	0	0	0	0	0		4,926	46,620	10,304
CUMULATIVE																	
PLANNED TOTALS		0	2,362	6,728	13,894	18,843	24,666	30,489	36,312	42,135	47,958	53,781	61,850	61,850			
ACTUAL TOTALS		0	2,909	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926		4,926		10,304
Budget Variance		0	(547)	1,802	8,968	13,917	19,740	25,563	31,386	37,209	43,032	48,855	56,924				

**PROJECT: Production and Evaluation of BioSorbents and Cleaning Solutions
for Use in Decontamination and Decommissioning**

1997 TASK NO.: 3.1

*****Subcontractor
Detail**

PRINCIPAL INVESTIGATOR: J. Kilbane

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Amount Remaining
SALARIES																
Professional Level III	planned actual	0 0	642 932	642 323	642	500	500	500	500	500	500	500	1,500	6,926		
Professional Level II	planned actual	0 0	0 0	0 0	0	0	0	0	0	0	0	0	0	0	1,255	5,671
Technician Level I	planned actual	0 0	0 0	642 323	642	604	884	884	884	884	884	884	605	7,797	323	7,474
LABOR OVERHEAD																
138% of Salaries	planned actual	0 0	886 1,286	1,772 891	1,772 0	1,524 0	1,910 0	1,910 0	1,910 0	1,910 0	1,910 0	1,910 0	2,905 0	20,318	2,178	18,140
Materials & Service	planned actual	0 0	273 0	273 0	273 0	273 0	273 0	273 0	273 0	273 0	273 0	273 0	270 0	3,000	0	3,000
Travel	planned actual	0 0	0 0	0 0	1,262	0	0	0	0	0	0	0	0	1,262	0	1,262
Other	planned actual	0 0	0 0	0 0	0	0	0	0	0	0	0	0	0	0	0	0
G & A (22%)	planned actual	0 0	396 488	732 338	1,010 0	638 0	785 0	785 0	785 0	785 0	785 0	785 0	1,162 0	8,647	826	7,820
Fee	planned actual	0 0	165 203	305 141	421 0	266 0	327 0	327 0	327 0	327 0	327 0	327 0	484 0	3,600	344	3,256
PLANNED TOTALS: PER MONTH																
		0	2,362	4,366	6,021	3,804	4,678	4,678	4,678	4,678	4,678	4,678	6,925	51,549		
ACTUAL TOTALS: PER MONTH																
		0	2,909	2,017	0	0	0	0	0	0	0	0	0		4,926	46,623
CUMULATIVE																
PLANNED TOTALS																
		0	2,362	6,728	12,750	16,554	21,232	25,911	30,589	35,267	39,946	44,624	51,549	51,549		
ACTUAL TOTALS																
		0	2,909	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926	4,926		4,926	46,623
Budget Variance																
		0	(547)	1,802	7,824	11,628	16,307	20,985	25,663	30,342	35,020	39,698	46,623			

1997 Task 3.1: Evaluation of BioSorbents/Cleaning Soln's for D&D

PRINCIPAL INVESTIGATOR: J. Kilbane (IGT), B. Reed (WVU)

**PROJECT: Use of SpinTek Centrifugal Membrane Technology and Sorbents/Cleaning Solutions
in the Decontamination and Decommissioning of DOE Facilities**

1997 TASK NO.: 3.2

WWU ACCOUNT NO.: 8527-090-3276

PRINCIPAL INVESTIGATOR: B. Reed

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
B. Reed	planned	0	0	0	0	925	1,850	1,850	925	0	0	0	0	5,550			
	actual	0	0	0											0	0	5,550
W. Lin	planned	0	0	0	0	688	1,375	1,375	688	0	0	0	0	4,125			
	actual	0	0	0											0	0	4,125
Management	planned	0	0	3,352	1,117	1,117	1,117	0	0	0	0	0	0	6,703			
	actual	0	0	0											0	0	6,703
BENEFITS																	
29% of Full Time Salaries	planned	0	0	972	324	792	1,259	935	468	0	0	0	0	4,750			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0	0	4,750
STUDENTS																	
Graduate	planned	1,450	1,450	1,450	1,450	1,450	1,450	1,450	1,450	1,450	1,450	1,450	1,450	17,400			
	actual	1,450	1,450	1,450											4,350	2,175	10,875
Undergraduate	planned	0	0	0	0	0	25	0	0	25	0	0	25	75			
	actual	0	0	0											0	0	75
SUPPLIES																	
	planned	100	100	100	100	100	100	100	100	100	100	0	0	1,000			
	actual	0	701	0											701	1,115	(816)
TRAVEL																	
	planned	0	0	500	0	500	0	500	0	500	0	0	0	2,000			
	actual	0	0	1,001											1,001	0	999
OTHER																	
	planned	0	0	200	200	200	200	200	200	200	200	200	200	2,000			
	actual	0	0	0											0	0	2,000
EQUIPMENT																	
< \$1,000	planned	0	0	0	0	0	0	0	0	0	0	0	0	0			
	actual	0	0	0											0	0	0
* > \$1,000	planned	0	1,000	1,000	1,000	0	0	0	0	0	0	0	0	3,000			
	actual	0	0	0											0	0	3,000
INDIRECT COSTS																	
43% of Direct Costs except *	planned	667	667	2,827	1,372	2,482	3,172	2,756	1,647	978	763	710	720	18,749			
	actual	624	925	1,054	0	0	0	0	0	0	0	0	0		2,603	1,415	14,732
PLANNED TOTALS: PER MONTH																	
		2,217	3,217	10,400	5,563	8,253	10,548	9,167	5,477	3,253	2,503	2,360	2,395	65,352			
ACTUAL TOTALS: PER MONTH																	
		2,074	3,076	3,506	0	0	0	0	0	0	0	0	0		8,655	4,705	51,992
CUMULATIVE																	
PLANNED TOTALS		2,217	5,433	15,833	21,396	29,649	40,198	49,364	54,841	58,094	60,597	62,956	65,352	65,352			
ACTUAL TOTALS		2,074	5,149	8,655	8,655	8,655	8,655	8,655	8,655	8,655	8,655	8,655	8,655		8,655		51,992
Budget Variance		143	284	7,178	12,741	20,994	31,543	40,709	46,186	49,440	51,942	54,302	56,697				

1997 Task 3.2 - SpinTek Technology w/ Sorbents/Cleaning Soln's
PRINCIPAL INVESTIGATOR: B. Reed

◆ PLANNED SPENDING * ACTUAL SPENDING

FOCUS AREA 4.0

Cross Cutting Innovative Technologies

**PROJECT: Use of Centrifugal Membrane Technology w/ Novel Membranes
to Treat Hazardous/Radioactive Wastes**

1997 TASK NO.: 4.1

WVU ACCOUNT NO.: 8527-090-3275

PRINCIPAL INVESTIGATOR: B. Reed and W. Lin

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
B. Reed	planned	0	0	0	0	1,233	2,467	2,467	1,233	0	0	0	0	7,400			
	actual	0	0	0											0	0	7,400
W. Lin	planned	0	0	0	0	917	1,833	1,833	917	0	0	0	0	5,500			
	actual	0	0	0											0	0	5,500
Management	planned	0	0	1,677	559	559	559	2,518	2,518	2,518	2,518	2,518	2,518	18,462			
	actual	0	0	0											0	0	18,462
BENEFITS																	
29% of Full Time Salaries	planned	0	0	486	162	786	1,409	1,977	1,354	730	730	730	730	9,095			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0	0	9,095
STUDENTS																	
Graduate	planned	2,650	2,650	2,650	2,650	2,650	2,650	2,650	2,650	2,650	2,650	2,650	2,650	31,800			
	actual	2,650	2,650	2,650											7,950	3,975	19,875
Undergraduate	planned	600	600	600	600	600	600	600	600	600	600	600	600	7,200			
	actual	0	0	0											0	0	7,200
SUPPLIES																	
	planned	0	0	200	200	200	200	200	200	200	200	200	200	2,000			
	actual	0	93	0											93	0	1,907
TRAVEL																	
	planned	0	0	500	0	0	500	0	0	500	0	0	500	2,000			
	actual	0	0	0											0	0	2,000
OTHER																	
	planned	0	0	100	100	100	100	100	100	100	100	100	100	1,000			
	actual	0	0	0											0	0	1,000
EQUIPMENT																	
* > \$1,000	planned	0	0	0	60,000	0	0	0	0	0	0	0	0	60,000			
	actual	0	0	0											0	0	60,000
INDIRECT COSTS																	
43% of Direct Costs except *	planned	1,398	1,398	2,672	1,836	3,029	4,437	5,309	4,116	3,138	2,923	2,923	3,138	36,317			
	actual	1,140	1,180	1,140	0	0	0	0	0	0	0	0	0		3,459	1,709	31,149
PLANNED TOTALS:																	
PER MONTH		4,648	4,648	8,885	66,107	10,073	14,754	17,654	13,688	10,437	9,722	9,722	10,437	180,773			
ACTUAL TOTALS:																	
PER MONTH		3,790	3,923	3,790	0	0	0	0	0	0	0	0	0		11,502	5,684	163,587
CUMULATIVE																	
PLANNED TOTALS		4,648	9,295	18,180	84,287	94,360	109,115	126,769	140,456	150,893	160,615	170,337	180,773	180,773			
ACTUAL TOTALS		3,790	7,712	11,502	11,502	11,502	11,502	11,502	11,502	11,502	11,502	11,502	11,502		11,502		163,587
Budget Variance		858	1,583	6,678	72,785	82,858	97,613	115,267	128,954	139,391	149,113	158,835	169,272				

1997 Task 4.1: Centrifugal Membrane Technology to Treat Waste

PRINCIPAL INVESTIGATOR (S): B. Reed and W. Lin

◆ PLANNED SPENDING * ACTUAL SPENDING

PROJECT: Environmental Pollution Control Devices Based on Novel Forms of Carbon

1997 TASK NO.: 4.2

WVU ACCOUNT NO.: 8527-089-2413

PRINCIPAL INVESTIGATOR (S): A. Brennsteiner, J.W. Zondlo & A. Stiller

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
A. Brennsteiner	planned	2,500	2,500	2,500	2,500	2,500	2,500	2,835	2,835	2,835	2,835	2,835	2,835	32,011			
	actual	2,500	2,500	2,500											7,500	7,500	17,011
J.W. Zondlo & A. Stiller	planned	0	0	0	0	1,144	2,288	2,288	1,144	0	0	0	0	6,864			
	actual	0	0	0											0	0	6,864
Management	planned	1,151	1,151	1,151	1,151	1,151	1,151	1,234	1,234	1,234	1,234	1,234	1,234	14,312			
	actual	1,151	1,151	1,151											3,453	3,453	7,406
BENEFITS																	
29% of Full Time Salaries	planned	1,059	1,059	1,059	1,059	1,391	1,722	1,844	1,512	1,180	1,180	1,180	1,180	15,424			
	actual	1,059	1,059	1,059	0	0	0	0	0	0	0	0	0		3,176	3,176	9,071
STUDENTS																	
Graduate	planned	1,050	1,050	1,050	1,050	1,050	1,050	1,200	1,200	1,200	1,200	1,200	1,200	13,500			
	actual	1,050	1,050	1,050											3,150	3,150	7,200
SUPPLIES																	
	planned	325	325	325	325	325	325	325	325	325	325	325	325	3,900			
	actual	0	452	0											452	154	3,294
OTHER																	
	planned	0	0	25	0	0	25	0	0	25	0	0	25	100			
	actual	0	0	193											193	0	(93)
EQUIPMENT																	
< \$1,000	planned	0	2,000	1,500	1,000	0	0	0	0	0	0	0	0	4,500			
	actual	0	0	0											0	0	4,500
* > \$1,000	planned	0	2,300	2,000	0	0	0	0	0	0	0	0	0	4,300			
	actual	0	0	0											0	0	4,300
INDIRECT COSTS																	
43% of Direct Costs except *	planned	2,616	3,476	3,272	3,046	3,251	3,896	4,182	3,548	2,924	2,913	2,913	2,924	38,963			
	actual	2,477	2,671	2,560	0	0	0	0	0	0	0	0	0		7,708	7,496	23,759
PLANNED TOTALS: PER MONTH																	
		8,701	13,861	12,882	10,131	10,812	12,958	13,908	11,798	9,724	9,688	9,688	9,724	133,874			
ACTUAL TOTALS: PER MONTH																	
		8,236	8,883	8,513	0	0	0	0	0	0	0	0	0		25,632	24,930	83,312
CUMULATIVE																	
PLANNED TOTALS		8,701	22,562	35,444	45,576	56,387	69,345	83,253	95,052	104,775	114,463	124,151	133,874	133,874			
ACTUAL TOTALS		8,236	17,119	25,632	25,632	25,632	25,632	25,632	25,632	25,632	25,632	25,632	25,632		25,632		83,312
Budget Variance		465	5,443	9,812	19,944	30,755	43,713	57,621	69,419	79,143	88,831	98,518	108,242				

1997 Task 4.2 - Pollution Control Based on Novel Forms of Carbon

PRINCIPAL INVESTIGATOR (S): A. Brennsteiner, J.W. Zondlo & A. Stiller

PROJECT: Design of Rotating Membrane Filtration Systems for Remediation Technologies

1997 TASK NO.: 4.3

WVU ACCOUNT NO.: 8527-093-3577

PRINCIPAL INVESTIGATOR: V. Mucino

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
V. Mucino	planned actual	0 0	0 0	0 0	0 0	2,432 0	4,863 0	4,863 0	2,432 0	0 0	0 0	0 0	0 0	14,590 0	0	0	14,590
Clerical	planned actual	0 0	0 0	300 0	0 0	0 0	300 0	0 0	0 0	300 0	0 0	0 0	300 0	1,200 0	0	0	1,200
Management	planned actual	0 0	0 0	0 0	0 0	0 0	0 0	1,437 0	1,437 0	1,437 0	1,437 0	1,437 0	1,437 0	8,622 0	0	0	8,622
BENEFITS 29% of Full Time Salaries	planned actual	0 0	0 0	87 0	0 0	705 0	1,497 0	1,827 0	1,122 0	504 0	417 0	417 0	504 0	7,079 0	0	0	7,079
STUDENTS Graduate	planned actual	900 900	900 900	900 900	900 900	900 900	900 900	900 900	900 900	900 900	900 900	900 900	900 900	10,800 2,700	2,700	1,350	6,750
TRAVEL	planned actual	0 0	0 0	500 0	0 0	0 0	500 0	0 0	0 0	0 0	0 0	0 0	0 0	1,000 0	0	0	1,000
EQUIPMENT < \$1,000	planned actual	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0	0	0
* > \$1,000	planned actual	0 0	2,000 0	1,000 2,315	1,000 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	0 0	4,000 2,315	2,315	1,414	271
INDIRECT COSTS 43% of Direct Costs except *	planned actual	387 387	387 387	768 387	387 0	1,736 0	3,466 0	3,882 0	2,533 0	1,351 0	1,184 0	1,184 0	1,351 0	18,615 1,161	1,161	581	16,874
PLANNED TOTALS: PER MONTH		1,287	3,287	3,555	2,287	5,773	11,626	12,909	8,424	4,491	3,938	3,938	4,491	65,907			
ACTUAL TOTALS: PER MONTH		1,287	1,287	3,602	0	0	0	0	0	0	0	0	0	6,176	6,176	3,345	56,386
CUMULATIVE																	
PLANNED TOTALS		1,287	4,574	8,129	10,416	16,190	27,716	40,625	49,049	53,540	57,478	61,416	65,907	65,907			
ACTUAL TOTALS		1,287	2,574	6,176	6,176	6,176	6,176	6,176	6,176	6,176	6,176	6,176	6,176	6,176	6,176		56,386
Budget Variance		0	2,000	1,953	4,240	10,014	21,540	34,449	42,873	47,364	51,302	55,240	59,731				

1997 Task 4.3: Design of a Rotating Filtration System

PRINCIPAL INVESTIGATOR: V. Mucino

TASK 5.0

Outreach

PROJECT: Small Business Technical Based Support

1997 TASK NO.: 5.1

PRINCIPAL INVESTIGATOR: E. Cook

WVU ACCOUNT NO.: 8527-090-3274

PERIOD ENDING: March 31, 1997

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Management	planned	0	0	0	821	205	205	5,278	4,797	4,309	3,488	3,488	870	23,461			
	actual	0	0	0											0	0	23,461
BENEFITS																	
29% of Full Time Salaries	planned	0	0	0	238	59	59	1,531	1,391	1,250	1,012	1,012	252	6,804			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		0	0	6,804
STUDENTS																	
Graduate	planned	906	906	906	906	906	906	906	906	906	906	906	234	10,200			
	actual	850	850	850											2,550	1,527	6,123
SUPPLIES																	
	planned	200	200	200	200	200	200	200	200	200	200	117	0	2,117			
	actual	94	294	150											537	75	1,505
TRAVEL																	
	planned	0	1,000	0	1,000	0	1,000	0	1,000	0	1,000	0	0	5,000			
	actual	0	0	0											0	849	4,151
OTHER																	
	planned	0	0	50	50	50	50	50	50	50	50	50	50	500			
	actual	0	0	0											0	0	500
SUB-CONTR.																	
* > \$25,000	planned	0	0	0	0	0	5,000	9,498	0	81,587	0	0	0	96,085			
	actual	0	0	0											0	0	96,085
< \$25,000	planned	0	0	0	10,000	10,000	5,000	25,000	25,000	0	0	0	0	75,000			
	actual	0	0	0											0	0	75,000
INDIRECT COSTS																	
43% of Direct Costs except *	planned	476	908	497	5,682	4,911	3,191	14,175	14,338	2,887	2,862	2,396	605	52,925			
	actual	406	492	430	0	0	0	0	0	0	0	0	0		1,328	1,054	50,544
PLANNED TOTALS: PER MONTH																	
		1,682	3,012	1,653	18,898	16,332	15,612	66,637	47,682	91,189	9,517	7,969	2,011	272,092			
ACTUAL TOTALS: PER MONTH																	
		1,349	1,635	1,430	0	0	0	0	0	0	0	0	0		4,415	3,505	264,173
CUMULATIVE																	
PLANNED TOTALS																	
		1,682	4,593	6,246	25,144	41,475	57,087	113,724	161,406	252,595	262,113	270,081	272,092	272,092			
ACTUAL TOTALS																	
		1,349	2,985	4,415	4,415	4,415	4,415	4,415	4,415	4,415	4,415	4,415	4,415		4,415		264,173
Budget Variance																	
		232	1,608	1,831	20,729	37,061	52,672	109,310	156,992	248,181	257,698	265,667	267,678				

1997 Task 5.1: Small Business Technical Based Support

PRINCIPAL INVESTIGATOR: E. Cook

◆ PLANNED SPENDING * ACTUAL SPENDING

SCHEDULES

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997													J
					J	F	M	A	M	J	J	A	S	O	N	D		
1	Task 1-1 Grout Testing Chamber	52w	1/1/97	25%														
2	NEPA Information	4w	1/1/97	100%														
3	Grout Testing Chamber (GTC) Set Up	12w	1/15/97	100%														
4	Performance Testing	40w	1/1/97	40%														
5	Injectability	8w	1/1/97	100%														
6	Permeability	16w	4/1/97	25%														
7	Shear Strength	8w	4/15/97	10%														
8	Compresibility Characteristics	8w	5/15/97	0%														
9	Data Reduction and Development of a Field Implementation Plan	8w	10/1/97	0%														
10	Quarterly Report #1	1w	3/31/97	0%														
11	Quarterly Report #2	0.2w	7/1/97	0%														
12	Quarterly Report #3	0.2w	10/1/97	0%														
13	Quarterly Report #4	0.2w	12/25/97	0%														
14	Final Report	4w	12/1/97	0%														
15																		

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
16	Task 1-2 Desiccation Barriers	52w	1/1/97	25%													
17	NEPA & SARS Information	12w	1/1/97	100%													
18	CATC Assembly, Calibration and Characterization	24w	1/1/97	50%													
19	CAB Deployment	12w	7/1/97	0%													
20	CAB Integrity Verification	12w	10/1/97	0%													
21	Design and Experimental Plans for the Dual Barrier Tesing	12w	7/1/97	0%													
22	Quarterly Report #1	1w	3/31/97	100%													
23	Quarterly Report #2	0.2w	7/1/97	0%													
24	Quarterly Report #3	0.2w	10/1/97	0%													
25	Quarterly Report #4	0.2w	12/25/97	0%													
26	Final Report	4w	12/1/97	0%													
27																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
28	Task 1-3 In Situ Formed Barriers	52w	1/1/97	25%													
29	NEPA & SARS Information	24w	1/1/97	65%													
30	Planning and Technology Transfers	0.2w	1/1/97	0%													
31	Bench Scale Test Plans	24w	1/1/97	65%													
32	Plan Reactive Bench Tests	20w	4/1/97	10%													
33	Design Dual Barrier Pilot Tests	16w	6/2/97	0%													
34	Design Reactive Barrier Pilot	16w	9/1/97	0%													
35	Technology Transfer CAB	32w	5/1/97	0%													
36	Technology Transfer CTC	32w	5/1/97	0%													
37	CAB Pilot Scale Assistance	0.2w	1/1/97	0%													
38	Install CATC Vessel in B-17	12w	2/3/97	100%													
39	Design & Test Instrumentation	8w	1/1/97	100%													
40	Test & Install Instrumentation	8w	2/3/97	100%													
41	Pilot Scale Test Support	36w	4/1/97	10%													
42	GTC Pilot Scale Assistance	0.2w	1/1/97	0%													
43	Install GTC in B-17	8w	1/1/97	100%													
44	Chemical Grout Material Support	40w	3/3/97	20%													
45	Dual Barrier Test Support	0.2w	8/1/97	0%													
46	Design Bench Scale Experiments	12w	8/1/97	0%													
47	Design Pilot Scale Test Series	12w	10/1/97	0%													
48	Quarterly Report #1	1w	3/31/97	100%													
49	Quarterly Report #2	0.2w	7/1/97	0%													
50	Quarterly Report #3	0.2w	10/1/97	0%													
51	Quarterly Report #4	0.2w	12/25/97	0%													
52	Final Report	4w	12/1/97	0%													
53																	

Quarterly Report January - March 1997

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
54	Task 1-4 DOE/RMI Field Testing	52w	1/1/97	25%													
55	Phase I - Modeling & Lab Research	38w	1/1/97	40%													
56	Site Data Evaluation	7w	1/1/97	80%													
57	Predictive Modeling	20w	2/17/97	45%													
58	RMI Field Sampling	1.6w	5/16/97	0%													
59	Lab Scale Physical Testing	20w	5/26/97	0%													
60	Lab Scale Chemical Testing	30w	5/19/97	0%													
61	RMI Site Support	20w	5/5/97	0%													
62	RMI Site Selection	2w	7/7/97	0%													
63	Phase II - Feild Demonsration	25w	7/7/97	0%													
64	Quarterly Report #1	1w	3/31/97	100%													
65	Quarterly Report #2	0.2w	7/1/97	0%													
66	Quarterly Report #3	0.2w	10/13/97	0%													
67	Quarterly Report #4	0.2w	12/25/97	0%													
68	Final Report	4w	12/1/97	0%													
69																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
70	Task 1-5 Drain Enhanced Soil Flushing	52w	1/1/97	25%													
71	NEPA Information	4w	1/1/97	100%													
72	Site Specific Batch Studies	12w	1/1/97	100%													
73	PVD Performance; Geotex. Selection	8w	1/1/97	100%													
74	Engineering Design & Drafting	8w	1/1/97	100%													
75	Permit Application Process	8w	1/1/97	100%													
76	Procurement of Equipment & Supplies	2w	5/28/97	0%													
77	Field Layout & Equipment Mobilization	6w	4/1/97	50%													
78	Field Construction; Equipment Set Up	7w	4/7/97	30%													
79	Experimental Testing Phase	32w	4/15/97	5%													
80	Chemical Lab Analysis	19w	7/15/97	0%													
81	Completion of Testing	2w	8/15/97	0%													
82	Abandonment, Site Closure	2w	9/22/97	0%													
83	Equip Final Disposition	2w	10/6/97	0%													
84	Data Reduction	8w	10/1/97	0%													
85	Quarterly Report #1	1w	3/31/97	100%													
86	Quarterly Report #2	0.2w	7/1/97	0%													
87	Quarterly Report #3	0.2w	10/1/97	0%													
88	Quarterly Report #4	0.2w	12/25/97	0%													
89	Final Report	4w	12/1/97	0%													
90																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
91	Task 2-1 Vortec Cyclone Melting System	52w	1/1/97	25%													
92	Equilibrium Calculation Recuperator	12.4w	4/1/97	0%													
93	Chemical Kinetics Calculations Residence Chamber	16.6w	7/1/97	0%													
94	Recuperator Analysis	24w	1/1/97	50%													
95	Recuperator Design Criteria	15.8w	6/1/97	10%													
96	Transient Calculations	12w	10/1/97	0%													
97	Quarterly Report #1	1w	3/31/97	100%													
98	Quarterly Report #3	0.2w	7/1/97	0%													
99		0.2w	10/1/97	0%													
100	Quarterly Report #4	0.2w	12/25/97	0%													
101	Final Report	4w	12/1/97	0%													
102																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
103	Task 3-1 Biosorbents & Cleaning Solutions	52w	1/1/97	25%													
104	Development of Waste Streams	8w	1/1/97	100%													
105	Metal/Radionuclide Binding Tests	24w	1/1/97	50%													
106	Desorption Testing of Surfactants	24w	6/2/97	0%													
107	Industrial Partner Development	52w	1/1/97	25%													
108	Management and Reporting	52w	1/1/97	25%													
109	Quarterly Report #1	1w	3/31/97	100%													
110	Quarterly Report #2	0.2w	7/1/97	0%													
111	Quarterly Report #3	0.2w	10/1/97	0%													
112	Quarterly Report #4	0.2w	12/25/97	0%													
113	Final Report	4w	12/1/97	0%													
114																	

ID	Task Name	Dur	Start	% Comp.	J	F	M	A	M	J	J	A	S	O	N	D	J
115	Task 3-2 Spin Tek Membrane	52w	1/1/97	25%													
116	NEPA Information	4w	1/1/97	100%													
117	Characterize IGT Sorbents, Surfactants, & Cleaning Solutions	44w	1/1/97	25%													
118	Identify Additional DOE Wastes	44w	1/1/97	25%													
119	Technology Testing	44w	2/3/97	25%													
120	Quarterly Report #1	1w	3/31/97	100%													
121	Quarterly Report #2	0.2w	7/1/97	0%													
122	Quarterly Report #3	0.2w	10/1/97	0%													
123	Quarterly Report #4	0.2w	12/25/97	0%													
124	Final Report	4w	12/1/97	0%													
125																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997											
126	Task 4-1 Use of Centrifugal Membrane Technology	52w	1/1/97	25%												
127	NEPA Information	4w	1/1/97	100%												
128	Membranes Acquisition	44w	1/1/97	100%												
129	Waste Development	44w	1/1/97	25%												
130	Standardized Test Development	12w	2/3/97	75%												
131	Quarterly Report #1	1w	3/31/97	100%												
132	Quarterly Report #2	0.2w	7/1/97	0%												
133	Quarterly Report #3	0.2w	10/1/97	0%												
134	Quarterly Report #4	0.2w	12/25/97	0%												
135	Final Report	4w	12/1/97	0%												
136																

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997													J
					J	F	M	A	M	J	J	A	S	O	N	D		
137	Task 4-2 Environmental Pollution Control Devices	52w	1/1/97	25%														
138	Industrial Contacts	52w	1/1/97	20%														
139	Define Industrial Needs	52w	1/1/97	20%														
140	Test Removal Efficiencies Using Small Cell	52w	1/1/97	20%														
141	Optimize Experimental Parameters	52w	1/1/97	30%														
142	Study Affect of Interfering Ions	16w	6/2/97	50%														
143	Design and Construct Large Prototype	12w	1/1/97	100%														
144	Apply Large-Scale Prototype	24w	7/1/97	20%														
145	Tailor Carbon Materials	52w	1/1/97	0%														
146	Develop Recomendations	4w	11/3/97	0%														
147	Quarterly Report #1	1w	3/31/97	100%														
148	Quarterly Report #2	0.2w	7/1/97	0%														
149	Quarterly Report #3	0.2w	10/1/97	0%														
150	Quarterly Report #4	0.2w	12/25/97	0%														
151	Final Report	4w	12/1/97	0%														
152																		

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												J
					J	F	M	A	M	J	J	A	S	O	N	D	
153	Task 4-3 Rotating Membrane Filtration System	52w	1/1/97	25%													
154	Parametric CAD Representation	3.5w	1/1/97	90%													
155	Modelling	4.5w	2/3/97	90%													
156	System Response	5.5w	2/19/97	90%													
157	Post-Processing Results	4.5w	4/1/97	50%													
158	Validation of Results	3.5w	4/22/97	0%													
159	Parametric Variations	3.5w	5/21/97	0%													
160	Design Definition	6.5w	6/18/97	0%													
161	System Analysis	4.5w	7/16/97	0%													
162	Fluid Flow Modelling	5.5w	8/13/97	0%													
163	Presentation	37.2w	2/26/97	0%													
164	Quarterly Report #1	1w	3/31/97	100%													
165	Quarterly Report #2	0.2w	7/1/97	0%													
166	Quarterly Report #3	0.2w	10/1/97	0%													
167	Quarterly Report #4	0.2w	12/25/97	0%													
168	Final Report	4w	12/1/97	0%													
169																	

1997 Tasks

West Virginia University Cooperative Agreement

ID	Task Name	Dur	Start	% Comp.	1997												
					J	F	M	A	M	J	J	A	S	O	N	D	J
170	Task 5-1 Small Business Technology Support	52w	1/1/97	25%													
171	Small Business Contact Development	52w	1/1/97	25%													
172	Problem Identification	52w	1/1/97	25%													
173	Team Researchers & Funding Allocation	52w	1/1/97	25%													
174	Quarterly Report #1	1w	3/31/97	100%													
175	Quarterly Report #2	0.2w	7/1/97	0%													
176	Quarterly Report #3	0.2w	10/1/97	0%													
177	Quarterly Report #4	0.2w	12/25/97	0%													
178	Final Report	4w	12/1/97	0%													

Quarterly Report January - March 1997

TECHNICAL PROGRESS

FOCUS AREA 1.0

Subsurface Contaminants, Containment & Remediation

TASK 1.1

CHARACTERISTICS EVALUATION of GROUT BARRIERS

FETC Task No. 1.1

PROGRESS REPORT

Reporting Period: January 1 - March 31, 1997

Work Performed Under Contract No.
DE-FC21-92MC29467

Principle Investigator: M.A. Gabr, Ph.D., P.E.

Graduate Research Assistants:

Jason Lowther
Nancy York

For:

U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
Morgantown, WV

Department of Civil and Environmental Engineering
West Virginia University
Morgantown, WV 26506

Evaluation of Acrylate Grouts for Subsurface Containment of Wastes

Abstract: A significant effort in the U.S. and the world is undertaken to develop remediation technologies for contaminated soils and waters. However, in some cases a permanent technology in response to some urgent environmental problems is yet to be developed. For this situation, a temporary barrier may be needed to retard the advancement of the plumes until a permanent remediation technology is implemented. As a result, extensive research is being conducted to evaluate the feasibility of using permeation grouts to develop subsurface containment/confinement systems to retard, or even prevent, the advancement of subsurface contaminant plumes.

A research program was conducted to assess the effectiveness of a urethane (Scotch-Seal (TM) Chemical Grout 5610 Gel manufactured by 3M Company) and a vinyl monomer (Duroseal^{TM} manufactured by Beton-Bau-Zubehor (BBZ) of Germany) grout in controlling the subsurface migration of waste. The study was carried out on the laboratory scale, and was intended to evaluate the chemical grouts ability to create a low-permeability barrier when injected into the soil and develop standard grout evaluation and testing protocols.

The experimental program was implemented in four phases: a) Grout Development, b) Performance Testing, and c) Grout Testing Chamber. The first phase included the evaluation of the grouts' properties and development of viable mix ratios. The second phase of the experimental program aimed at examining the feasibility of injecting the urethane grout into a uniformly graded Ottawa sand (i.e., groutability) in large diameter (29.2 cm), acrylic columns. Samples were collected from the grout bulb and subjected to additional testing which yielded results on the grouted-sands hydraulic conductivity, compressive strength, and

durability under permeation and immersion with various classes of chemicals. Phase 3 involves the design and construction of the Grout Testing Chamber (GTC) (essentially a 91.4 cm flexible-wall permeameter) and manufacture of a sand rain device to prepare uniform specimens for the GTC. The first phase of the study resulted in four urethane mixes and one vinyl monomer mix being developed for the next phase. All the urethane mixes were 10:1 (activator:gel) ratios; however, the mix additives varied. The vinyl monomer mix was comprised on 50% water and 50% gel concentrate.

Results from the second phase indicated that two of the urethane grout mixes could be easily injected into the Ottawa sand at its porosity extremes. Additionally, the larger the perforation size and perforation length on the injection probe, the larger the resulting grout bulb and bulb radius. Samples collected from the injected bulbs yielded hydraulic conductivities (k) of 2.4×10^{-7} cm/s with water and significantly increased when using chemical permeants. Grouted-sand samples from both of the grout mixes swelled excessively and decreased in strength when immersed in water, bases, and ethylene glycol solutions. Cupric sulfate, methanol, acetone, and acids appeared to have few observable affects on the samples.

The GTC was designed and is currently under construction. Additionally, a sand rain device was designed and manufactured to produce uniform density specimens for the GTC. It is currently being calibrated.

Within this paper, testing methodologies, data, and results from each phase of the experimental program are presented.

TABLE OF CONTENTS

1.0	INTRODUCTION	1
2.0	EVALUATION OF A URETHANE GROUT AND VINYL MONOMER GROUT FOR CONTAINMENT OF WASTES	
2.1	Purpose	3
2.2	Background	3
2.3	Methodology	3
2.4	Work Performed this Period	4
2.5	Results and Discussion	4
2.6	Conclusions	6
2.7	Work Planned for Next Period	6
	APPENDIX A. BIBLIOGRAPHY	7

1.0 INTRODUCTION

There is a significant effort in the U.S. and the world to develop remediation technologies for contaminated soils and waters. For some contaminated medias, no technologies are feasible or fully developed to meet today's environmental problems. For example, radioactive contaminated soils are impossible to treat with current technologies. Only waste minimization/segregation techniques are applicable which results in a highly concentrated (i.e., radioactive) residual material. Because of the lack of treatment methods, confinement and containment technologies are needed to retard, or even prevent, the advancement of subsurface contaminant plumes into the environment until the development of appropriate remediation technologies are identified and implemented.

There are numerous confinement/containment (C/C) methods available in today's market. Though these techniques are fully capable of preventing lateral migration of contaminants in soil and water, longitudinal migration of contaminants is generally not suppressed or retarded. One method that appears suitable for developing both vertical cut-off walls and/or horizontal bottom barriers is permeation (injection) grouting utilizing chemical grouts which is defined as the process of filling small interstitial voids and fissures in rock or soil (Karol 1990). If permeation grouting is proven to be preventative measure (i.e., secondary containment), as well as a post-release alternative, it could potentially be utilized at numerous sites (Superfund and RCRA) throughout the country. Because of the potential benefits of this technology and the large number of sites where it could be implemented, research was conducted at West Virginia University to evaluate two chemical grout's effectiveness and applicability as a subsurface barrier. Unlike prior and other current studies, instead of immediately advancing from the lab-scale grout investigation to field scale injection testing, a meso-scale testing program was developed to investigate the material Injectability. Two types of chemical grouts were evaluated for barrier use: Duraseal™ (a vinyl monomer compound from Germany) and Scotch-Seal (TM) Chemical Grout 5610 (a urethane gel product manufactured by 3M). Both grout materials were evaluated on their ability to form in-situ barriers (i.e., injectability/viscosity), toxicity, resistance to chemical degradation, strength properties, and permeability. The research work was divided into the following tasks:

- I. Grout Development
- II. Performance Testing
- III. Grout Testing Chamber (GTC)

Grout Development involves the identification of the grout mix ratio which exhibits the optimum properties for use as a subsurface barrier. Performance Testing utilizes large diameter acrylic columns to investigate the injectability of the grout into Ottawa sand using different grout mixes and varying injection strategies. The structural and hydraulic performance of the formed barrier is also evaluated by performing immersion/strength testing (triaxial and unconfined compression) and permeability testing on samples collected from the grouted-sand bulb. Task III involves the design and construction of the Grout Testing Chamber (GTC), essentially a large-scale flexible wall permeameter with 3 feet diameter specimens which will be capable of simulating in-situ stress conditions. A sub-task was the development of a device and method for

preparing specimens of a uniform density which can be accomplished through the use of a sand rain device.

2.0 EVALUATION OF A URETHANE GROUT AND VINYL MONOMER GROUT FOR CONTAINMENT OF WASTES

2.1 Purpose

This research presented in this section was performed to successfully fulfill the requirements of Task I (Grout Development) and Task II (Performance Testing). Only urethane grout is performance is currently being pursued. The objectives of this research program are to establish grout testing protocol, evaluate grout performance, and determine grout characteristics for several injection strategies.

2.2 Background

Although the principles of permeation grouting were developed in the 1940's and numerous companies are in existence which specialize in this practice, none have utilized permeation grouting for waste containment. Past field studies (May et al. 1986, Dwyer 1994, Moridis 1995) conducted to evaluate subsurface barrier characteristics have identified the need for a barrier verification technique. A limited number of research programs (Bodocsi et al. 1988, Bodocsi et. al. 1991, Carson 1988, Heiser et al. 1992, Sherer 1986) have investigated the properties of various types of chemical grouts which make them amenable as a subsurface barrier. At present, there is a considerable amount of research being conducted on this topic at government facilities.

The urethane grout evaluated in this research was supplied by the 3M Company under the trade name Scotch-Seal (TM) Brand Chemical Grout 5610 (Gel). This was chosen after reviewing the product literature and past studies as well as discussing the grout's capabilities with various distributors and manufacturers with research objectives in mind. Furthermore, 3M defines Scotch-Seal (TM) as groutable. The 5610 gel is a hydrophilic material most commonly used for sewer and water line rehabilitation projects.

2.3 Methodology

The experimental program of this investigation was performed to develop a standard testing protocol to evaluate the performance (i.e., durability, hydraulic conductivity, and strength) of permeation grouts. Experimental testing was implemented in two phases: a) Materials Testing Phase and b) Performance Testing Phase. The first phase consisted of testing material properties which included: moisture content (ASTM D 2216), specific gravity (ASTM D 1429), minimum and maximum densities (ASTM D 4254 83 and D 4253 83), and triaxial compression (ASTM D 2850). Additionally, the permeability the material was determined using a rigid wall permeameter conducted in accordance with ASTM Draft Standard Test Method for Measurement of Hydraulic Conductivity Using a Rigid Wall, Compaction-Mold Permeameter. Performance testing examined the injectability (groutability) of the grout in large diameter columns and Post-grout testing (permeability testing, immersion testing, and strength testing).

2.4 Work Performed This Period

Urethane Grout:

During this work period, shrink/swell testing using water as the fluid was continued. As reported last period, the amount of swelling that was experienced during the first hydration cycle was approximately 68% of the initial height. Upon reaching this plateau, the fluid was drained from the consolidometer and the sample was allowed to shrink (applied vertical stress remained constant). Once there was no change in sample height, the sample was again saturated for the second hydration cycle. In order to observe if this behavior was consistent, another shrink/swell test was performed.

The questions of the permeability variance as a function of layer thickness and sample degradation (internal fissures or cracks) upon drying still remained unanswered. To resolve this issue, two flexible wall permeability tests were performed, using water as the permeant, with a sample thickness of 1 cm and 2 cm. Once the hydraulic conductivity was obtained, the permeameters were disassembled and the samples were allowed to dry. In order to replicate the drying process in the shrink/swell tests, the membrane was left on the samples and the lines to the caps of the consolidometers were opened. The samples will again be tested for hydraulic conductivity, after the drying process is complete.

Vinyl Monomer Grout:

During this work period, six characteristics were determined for each mixture of 20/30 Ottawa sand and kaolinite. These six characteristics are the specific gravity, liquid limit, plastic limit, plastic index, void ratio, and porosity. Also, out of the original eight mix ratios of the vinyl monomer grout, two were chosen because the gel time was in between twenty and thirty minutes. The two mix ratios chosen were 98% gel, 2% accelerator, 89% water, 11% hardening agent, and 97% gel, 3% accelerator, 98% water, 2% hardening agent.

2.5 Results and Discussion

Urethane Grout:

The results from the shrink/swell testing, presented in Table 2.1, indicate an total, first hydration swelling of 65% and 73% of the initial sample height. A problem encountered in the first test was a hinderence of the sample swelling and shrinking behavior due to the filter paper. In the second test, the filter paper was trimmed to minimize interference between the paper and the walls of the consolidometer. As a result, the data from Test #2 is probably the most representative. After the first drying cycle, it was noted that the sample recovered all but 2% of the initial swelling deformation. The swelling during the second hydration cycle was approximately 6% less than the initial swelling deformation in Test #1.

Table 2.1. Results from Shrink/Swell Testing

	Swelling Deformation after 1st Hydration Cycle, % of initial Height	Shrinking Deformation after 1st Drying Cycle, % of initial Height	Swelling Deformation after 2nd Hydration Cycle, % of initial Height
Test #1	65	6	59
Test #2	73	2	not yet available

The results from the flexible wall permeability tests are presented in Table 2.2. It should be noted that, using water as the permeant, there was no affect on the hydraulic conductivity of the grouted sand with variation in the sample thickness from 1 cm to 2 cm.

Table 2.2. Results from Flexible-Wall Permeability Tests

Sample Thickness, cm	Hydraulic Conductivity, cm/s
1	7×10^{-10}
2	9×10^{-10}

Vinyl Monomer:

The specific gravity, liquid limit, plastic limit, plastic index, void ratio, and porosity that were determined for each mixture of 20/30 Ottawa sand and kaolinite are shown in Table 2.3.

Table 2.3. Results from Specific Gravity, Liquid Limit, and Plastic Limit Tests

Soil Mixture sand / kaolinite	Specific Gravity	Liquid Limit(%)	Plastic Limit(%)	Plastic Index(%)	Void Ratio	Porosity (%)
50% : 50%	2.67	25.4	14.75	10.65	.47	31.97
60% : 40%	2.67	22.6	12.35	10.25	.382	27.6
70% : 30%	2.66	18.5	9.05	9.45	.36	26.5
80% : 20%	2.68	13.5	NA	NA	.29	22.5
90% : 10%	2.67	8.6	NA	NA	.43	30

2.6 Conclusions

Urethane Grout:

The testing performed this period has lead to the modeling of the shrink/swell behavior of the grouted sand at simulated depth of approximately 0.3 ft (based on a $\gamma_{dry} = 102$ pcf). From the flexible-wall permeability results, it can be seen that there is no variation in the hydraulic conductivity with a variation in the sample thickness from 1 cm to 2 cm. This implies that, under similar conditions, the thickness of the barrier in the field can be small, while still functional.

Vinyl Monomer Grout:

The tests performed this period have allowed for a better understanding of the sand/kaolinite mixtures being used. Taking eight mix ratios and narrowing them down to two has also been accomplished. From these two mix ratios it will be determined which one is more economically feasible to run the test with and that will be the one chosen for the injection process.

2.7 Work Planned for Next Period

Urethane Grout:

- The shrink/swell testing will continue using water as the fluid, but simulating depths of 3 ft and 30ft (based on a $\gamma_{dry} = 102$ pcf)
- Shrink/swell testing will also be performed using TCLP#2 as the fluid, defining the extent of shrink/swell deformation as a function of confining stress
- Permeability testing will resume on the samples that are currently drying
- Undrained, unconsolidated triaxial tests will be performed on the samples that have been saturating for a period of time equal to the total hydration time in the shrink/swell tests
- With the arrival of the Grout Testing Chamber (GTC), preliminary setup and testing is hopeful for the next reporting period

Vinyl Monomer:

- Rigid Wall permeability test will be performed on the five sand/kaolinite mixtures to determine the flow rate in which to inject, and to find the hydraulic conductivity
- Injection process should begin by the end of the month of May
- Flexible wall permeability test will be performed on the injected soil to determine the hydraulic conductivity

BIBLIOGRAPHY- CHEMICAL GROUTS

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TASK 1.2

**DEVELOPMENT OF STANDARD TEST PROTOCOLS AND DESIGN MODELS FOR
DESICCATION BARRIER
FETC Task No. 1.2**

Quarterly Technical Progress Report
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For:
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ABSTRACT

Significant research effort is currently being conducted in the United States and the world for remediation of contaminated soils and groundwater. In parallel to remediation efforts, confinement technologies are needed to retard, or even prevent, the advancement of contamination plumes into the environment until the implementation of remediation efforts is completed. Research proposed herein aims at developing and investigating containment/confinement technology using desiccation for in situ formation of barriers.

The desiccant barrier proposed to be investigated will be the recently developed circulating air barrier (CAB). The CAB utilizes the injection of dry gas (air, nitrogen, CO₂, etc.) to create an ultra dry zone in situ; beneath or adjacent to the zone of contamination. The CAB relies on the removal (drying) of in situ liquids (water) so that any contaminating liquids entering the zone of desiccation must replace the previously removed liquids before the contaminants can migrate further. In field application, the CAB may be established by means of specific patterns of vertical or horizontal (or a combination of vertical and horizontal) injection and withdrawal wells which enable the injected dry gas to travel through and dry the formation prior to exiting via a withdrawal well.

This research will be conducted using laboratory and meso-scale setups. In addition to conventional laboratory equipment proposed research will utilize a large scale reactor for simulating in situ conditions. Conducting the research based on a meso-scale setup will minimize problems associated with scale effects. Such problems are usually encountered when a method is developed in the laboratory with no considerations given to correlation of laboratory scale results to field implementation. The B-17 facility in FETC with its infrastructure will be utilized to conduct the proposed work. The proposed experimental methodologies will simulate field conditions and should provide laboratory data to test and enhance predictions of CAB performance made by existing models and computer simulations.

TABLE OF CONTENTS

	Page No
Abstract	ii
Table of Contents	iii
List of Tables	iv
List of Figures	v
Executive Summary	vi
1.0 Introduction	1
2.0 Purpose	1
3.0 Background	1
4.0 Methodology	2
5.0 Work Performed this Period	4
5.1 Task 1: SARS Activity	4
5.2 Task 2.1: Literature Review	4
5.3 Task 3: Laboratory Equipment and Preliminary Testing	4
5.3.1 Sand Pack Assembly Characteristics Evaluation	5
5.3.2 Sand Pack Drying Characteristics Determination	5
5.3.3 Design and Experimental Plans for Scaled Models	5
6.0 Results and Discussion	5
6.1 SARS Activity	5
6.2 Sand Pack Assembly Characteristics Evaluation	6
6.3 Sand Pack Drying Characteristics determination	9
6.4 Design and Experimental Plans for Scaled Models	9
7.0 Conclusions	12
7.1 SARS Activity	12
7.2 Sand Pack Assembly Characteristics Evaluation	12
7.3 Sand Pack Drying Characteristics Determination	12
8.0 Work Planned for Next Period	12
8.1 SARS Activity	12
8.2 Sand Pack Assembly Characteristics Evaluation	12
8.3 Grain Size and Sorting Influence on Sand Pack Characteristics	13
9.0 Technical/Administrative Difficulties	13
10.0 References	14

LIST OF TABLES

TABLE 1.	Sandpack Characteristics for various Size Sandpacks	Page No 6
TABLE 2.	Sandpack Characteristics for Sand Mixtures	6

LIST OF FIGURES

	Page No
FIGURE 1. Results of Compaction Test for various Sandpacks	7
FIGURE 2. Average Measured Porosity for Various Sandpacks	7
FIGURE 3. Average Measured Permeability for Various Sandpacks	8
FIGURE 4. Results of Drying Experiments for Various Sandpacks	10
FIGURE 5. Results of Mixture 1 Drying Experiments	11
FIGURE 5. Results of Mixture 2 Drying Experiments	11

EXECUTIVE SUMMARY

This study aims at developing and investigating containment/confinement technology using a "Circulating Air Barrier (CAB)". The CAB utilizes the injection of dry air to create an ultra dry zone in situ; beneath or adjacent to the zone of contamination. The CAB system can provide many advantages, however, there are no laboratory data or CAB field performance data available in the literature. There are however are a number of issues that must be settled before it can be decided if CAB technology is applicable. Key among these issues are the influence of the soil characteristics (such as porosity, permeability, water saturation, grain size distribution, and clay content) on the drying profile. This research will be conducted using laboratory and meso-scale setups to evaluate the soil characteristics influence on CAB performance and will determine the effects of flow and sweep efficiency for fixed and mixed grain size soils. These experiments will validate and/or improve model predictions of system behavior and drying profiles of different soil types.

Sand-packs are being utilized for bench-scale testing to determine the relationship between soil characteristics and in situ barrier quality. Sand-packs are being calibrated to determine porosity, permeability, and saturation characteristics. The soil characteristics will be varied by changing grain size and sorting of the pack materials. A detailed CAB vessel assembly has been designed and been approved for experimental studies at B-17 facilities. The procurement of materials and fabrication of the assembly is in progress by EG&G at FETC. Additional laboratory tests on core samples have been undertaken to evaluate the influence of porous media on air-water equilibrium and mass transfer.

1.0 INTRODUCTION

Many contaminated subsurface sites can not be economically decontaminated because of the limitations in technologies for decontamination. In such cases, containment becomes the primary technology for environmental protection. For non-excavatable sites, in situ containment is the most viable approach for preventing further spread of contamination. Different approaches for achieving in situ containment have been proposed, however, the study herein is focused on the evaluation of in situ barriers created by soil desiccation.

The desiccant barrier proposed to be investigated is the recently developed (1,2) Circulating Air Barrier (CAB). The CAB utilizes the injection of dry gas into a porous media to create an ultra dry zone in situ by removing water and reducing the water saturation below the level required for liquids to flow in order to prevent migration of underground contaminants. The CAB, once established, will also provide a means of monitoring and removing contaminants entering the zone. The proposed experimental methodology should provide laboratory data to test and enhance predictions of CAB performance made by existing models and computer simulations.

2.0 PURPOSE

The concept of the Circulating Air Barrier was conceived (1,2) to solve the subsurface contamination problems at the Hanford site used by the former Atomic Energy Commission. The references to CAB (1,2,3) are mainly related to CAB as a concept, and no field or laboratory data is available in the literature. The previous studies (1,2) concluded that a CAB system provided many advantages, however, the concept needs to be demonstrated to develop data necessary for scale-up and regulatory acceptance.

Among issues that must be settled before it can be decided if CAB technology is applicable to all DOE sites are the influence of the soil characteristics (such as, porosity, permeability, water saturation, grain size distribution, and clay content) on the drying profile and CAB performance. The laboratory study to be undertaken will evaluate the soil characteristics influence on CAB performance and will determine the effects of flow and sweep efficiency for fixed and mixed grain size soils. These experiments will validate and/or improve model predictions of system behavior and drying profiles of different soil types.

3.0 BACKGROUND

The Circulating Air Barrier (CAB) system is a desiccant-type barrier designed to prevent the movement of liquid contaminants toward groundwater, using air circulation and a processing system to lower water saturation in a targeted subsurface zone. The concept of

Circulating Air Barrier was introduced in a study (1) that evaluated the barrier systems that could be installed beneath and around the tank farms with a minimum of excavation at DOE's Hanford Site. The CAB can be installed using either vertical or horizontal wells, establishing a pattern of air injection and extraction wells. The moving air vaporizes water in the zone and carries the water vapor to an extraction well. In time, circulation of the air reduces the water saturation in the swept interval, and continues to remove, by evaporation, liquids that move into the zone such as a leaked plume. No liquids can flow through this interval until a critical saturation is achieved, a saturation level that is well above the initial saturation. In the event that a leak occurs, the CAB system serves as a tool for early leak detection and provides a means to withdraw volatile contaminants for surface treatment. The study concluded that CAB system provided many advantages including non-physical confinement, active monitoring and leak detection, commercially available oil and gas technologies and equipment, emergency response and rapid deployment capability, and high potential for integration with other remediation technologies. However, the concept needs to be demonstrated to develop data needed for scale-up and regulatory acceptance.

A follow up study by K&M/BDM (2) which addressed modeling, cold test design, and surface processing and control design for the CAB demonstration at Hanford concluded that several issues must be settled before it can be decided if CAB technology is applicable to all DOE sites. Key among these issues is the influence of the soil characteristics such as porosity, permeability, water saturation, grain size-distribution, and clay content on the drying profile and CAB performance. This report further recommended a laboratory study to be undertaken to evaluate the soil characteristics influence on CAB performance and to find out the effects of flow and sweep efficiency for fixed grain-size and mixed grain-size soils. This would also serve to validate and/or improve model predictions of system behavior and drying profiles of different soil types. In addition, a range of geologic and climatic environments within which the CAB could successfully operate, baseline equilibrium moisture curve for a specific grain-size, and relative humidity for a specific temperature could be determined.

4.0 METHODOLOGY

Experimental procedures will be developed to address the problem of evaluating a desiccant barrier, i.e., a barrier based on creation of a dry zone of earth beneath a contaminated zone to store contaminating liquids, thus preventing their further migration. Two phases of experimental research are proposed in this study. The B-17 facility in FETC with its infrastructure will be utilized to conduct the proposed work. The first phase deals with bench-scale experiments using laboratory setups. The second phase will deal with a meso-scale setup that will utilize large tanks for simulations of in situ conditions in order to minimize problems associated with scale effects.

Bench-scale testing of the CAB process parameters will utilize sand packs to determine the relationship between soil characteristics and in situ barrier quality. Sand packs

will be calibrated to determine porosity, permeability, and saturation characteristics. The soil characteristics will be varied by changing grain size and sorting of the pack materials. Residual water saturation of the pack will be established by first saturating the pack with water and then desaturating the pack by injection of water-saturated air. The pack will then be dried at various rates of air injection to determine drying characteristics as a function of rate, remaining water saturation, and soil characteristics. The final phase of sand pack testing will be to resaturate the packs with simulated contaminant fluids injected at low pressure (near atmospheric) at the top while monitoring any effluent at the bottom. This will allow determination of contaminants migration rate as well as total storage capacity of the packs.

Meso-scale performance testing will be used to investigate the effectiveness of the CAB system in containment/confinement of the study waste under simulated field conditions. The meso-scale tests of the CAB system will utilize large rectangular tanks in DOE/METC's B-17 building to create a three-dimensional test bed to verify, refine, and extrapolate the results of sand pack experiments to field like conditions. The tanks will be filled to a desired depth by slurring sand of predetermined grain size range into the tanks with water. All or part of bed will then be desaturated, dried, and "contaminated" with a safe liquid to simulate the CAB process on the meso-scale. Instrumentation and sampling ports and tubes will be placed in strategic locations in or at the tanks to monitor the status of the CAB.

5.0 WORK PERFORMED THIS PERIOD

5.1 Task 1: SARS Activity

A preliminary 'Operating Permit' for larger scale experiments in FETC Building B-17 has been prepared.

5.2 Task 2.1: Literature Review

A comprehensive literature review relative to air-water mass transfer in porous media has been conducted. Considerable work has been reported in the literature on drying of porous materials, and related heat and mass transfer processes. Most of the published studies were conducted under conditions that are more representative of the industrial applications such as adsorption of gases in granular beds or drying of solids by through circulation of air. The information relative to drying of naturally formed unsaturated porous media is very limited and are limited only to the case of steady-state liquid infiltration(22,23,24). More recently, Rogers and Kaviani(25,26) distinguished two distinct regimes, funicular and pendular, in convective drying process in granular beds. Funicular regimes occurs when the liquid phase is continuous. Pendular regime occurs when liquid phase is immobile. They concluded that evaporation rate during funicular regime was constant and decreased substantially during pendicular regime indicating surface drying.

A review of literature relative to influence of textural parameters on the rock properties was initiated. Grain size, sorting, shape, roundness, and packing are textural parameters that influence the rock properties. Of these, grain size and sorting are believed to be of major importance, whereas grain shape and roundness are of minor importance with respect to porosity and permeability. The grain size has been found to have little influence on porosity of well sorted sands (27,28). According to Rogers (27), well sorted sands have higher porosities than poorly sorted sands. Fraser (29) theoretically demonstrated that well rounded grains of high sphericity will pack with a minimum pore space and as the angularity increases porosity increases. Predicting permeability from theoretical considerations has been undertaken by many investigators However, the complexity of the problem made theoretically determined permeability unreliable.

5.3 Task 3: Laboratory Equipment and Preliminary Testing

The results of our previous experiments were found to be unreliable. The problem was investigated and it was discovered that sand had entered into the air flow manifold. As a result, the CAB vessel assembly was disassembled and cleaned and assembled again. The CAB vessel assembly was then tested and calibrated. The results of the tests on the CAB vessel assembly were found to be consistent and reliable after clean up. It was decided to periodically clean the assembly to assure the accuracy of the measurements.

5.3.1 Sand Pack Assembly Characteristics Evaluation

The influence of the particle size on sand pack characteristics has been under study. Compaction tests coupled with porosity and permeability measurements were performed with 20/30, 30/40, 60, 80, 120 mesh sizes sands. Table 1 summarizes the characteristics of each sand pack. In addition, two sand mixtures were prepared to study the influence of size distribution. The compositions of the mixtures are given in Table 2. The previously developed protocols for determining sand pack characteristics were implemented, using the CAB vessel assembly, to evaluate the porosity and permeability of various size sand compacted at the optimum water content.

5.3.2 Sand Pack Drying Characteristics Determination

To study the influence of sand size on drying of the unsaturated unconsolidated porous media, drying experiment for various size sand packs were performed. In addition, the drying experiments were performed for the two sand mixtures.

5.3.3 Design and Experimental Plans for the Scaled Models

The design of the scaled model, which is referred to here as the Circulating Air Test Cell (CATC) has been completed. The monitoring system, and experimental procedures for Phase II experiments are being further evaluated.

6.0 RESULTS AND DISCUSSION

6.1 SARS Activity

A preliminary 'Operating Permit' for larger scale experiments in FETC Building B-17 has been prepared.

6.2 Sand Pack Assembly Characteristics Evaluation

The results of the compaction tests, as well as porosity and permeability measurements for various size sand packs as well as the sand mixtures are illustrated in Figures 1 through 3. Tables 1 and 2 summarizes the results of permeability and porosity measurements for various sand packs. As it can be seen, the sand size has minor effect on porosity while it has a significant effect on permeability. The properties of the mixture 1, as expected, falls between 20/3 and 30/40 sand packs properties. The properties of mixture 2, with the exception of porosity, falls between 80 and 120 sand packs properties.

TABLE 1. PROPERTIES OF VARIOUS SAND PACKS

SAND SIZE	20/30	30/40	60	80	120
Average Grain Diameter (mm)	0.7	0.56	0.24	0.18	0.13
Optimum Water Content (mass %)	2.4	2.1	1.8	1.7	1.6
Dry Density (g/ml)	1.70	1.67	1.62	1.59	1.57
Effective Porosity (%)	30.1	31.5	33.2	34.1	34.8
Total Porosity (%)	34.6	35.0	36.1	36.8	37.3
Initial Water Saturation (%)	12.4	10.0	7.7	7.2	6.6
Initial Permeability (D)	222	185	88	56	17
Permeability After Drying (D)	290	245	114	72	21

TABLE 2. PROPERTIES OF SAND MIXTURES

MIXTURE	1	2
Composition	20/30 : 50% 30/40 : 50%	>45: 3.5% 45/60: 16% 60/80: 27% 80/120: 33% 120/170: 17% <170 : 3%
Optimum Water Content (mass %)	2.1	1.6
Dry Density (g/ml)	1.73	1.61
Effective Porosity (%)	31.5	37.3
Total Porosity (%)	35.2	40.4
Initial Water Saturation (%)	10.3	6.6
Initial Permeability (D)	176	22
Permeability After Drying (D)	210	28

FIGURE 1. Results of Compaction Tests for Various Sandpacks

FIGURE 2. Average Measured Porosity for Various Size Sandpacks

Figure 3. Average Measured Permeability for Various Sandpa

6.3 Sand Pack Drying Characteristics Determination

The results of the sand pack drying experiments for different size sands for the same air flow rate (8 liter/min) are illustrated in Figure 4. The results in Figure 4 indicate that initially, the evaporation rate are approximately the same for various size sands. This mainly due to the fact that over this early period (less than 1000 pore volumes), the air becomes fully saturated with water and the only controlling factors is the air flow rate. This generally occurs when the evaporation front is close to the bottom of the sand pack and air has sufficient distance to travel through the sand pack before it exits the sand pack. The evaporation rate begins to decline after this early period. The sand size appears to have some influence on the decline period. This decline is generally function of Reynolds Number which depends on sand grain diameter. Finally, the end of the sand pack is approached and the final decline occurs. As it can be observed in Figure 4, the final decline begins at different pore volumes for different sand sizes. The start of the final decline, which is the result of dry air break-through, mainly depends on the initial water saturation in the sand pack. The initial water saturation determines how fast the evaporative front moves through the sand pack. At low water initial water saturation, the front moves faster and the first decline is steeper and has a shorter duration. Since the packs with different size sands are prepared at optimum water content, the initial water saturations are different (see Table 1). This results in the somewhat of different shapes for rate evaporation curves for different size packs.

Figures 5 and 6 illustrate the results of drying experiments for two sand mixtures. Figures 5 illustrates the results for the sand mixture 1 and compares it with the results of 20/30 and 30/40 sands. As it can be seen from Figure 5 , the evaporation rate for mixture 1 is similar to uniform size sand packs with exception of the final decline period. The final decline period appears to initiate earlier but it is less steeper. This might indicate a partial and earlier break-through of the dry air. Figures 6 illustrates the results for mixture 2 and compares it with the results of 60 and 120 sands. Similar conclusions can be reached as those from Figure 5.

6.4. Design and Experimental Plans for the Scaled Models

A preliminary design of the CATC consist of a large tank which accommodates the sand bed and the necessary plumbing to simulate various well configurations. The construction of CATC will be undertaken upon availability of funds to purchase the tanks and other necessary parts and approval of the construction permit. Phase II experiments will be initiated by installation of CATC and assembly of the necessary the monitoring system at B-17 at FETC.

FIGURE 4. Results of the Drying Experiments for Various Size Sandpacks

FIGURE 5. Results of Mixture 1 Drying Experiment

FIGURE 6. Results of Mixture 2 Drying Experiment

7.0 CONCLUSIONS

7.1 SARS Activity

The next phase of SARS activity is submission of the 'Operating Permit' for Phase II experiments and presentation to FETC for approval.

7.2 Sand Pack Assembly Characteristics Evaluation

The results of porosity and permeability measurements confirm the established sand pack tests have been useful in establishing experimental protocols and have provided baseline data for the larger scale experiments.

7.3 Sand Pack Drying Characteristics Determination

The results of the drying tests indicate that during early drying period (less than 1000 pore volumes), the air flow rate controls the evaporation rate. This generally occurs when the evaporation front is close to the bottom of the sand pack and air has sufficient distance to travel through the sand pack before it exits the sand pack. The sand size appears and as a result Reynolds Number influences the decline period that follows the initial period. Finally, the end of the sand pack is approached and the final decline occurs as the result of dry air break-through. The initial water saturation determines how the evaporative front moves through the sand pack. At low water initial water saturation, the front moves faster and the decline is steeper and has a shorter duration. In sand mixtures, a partial and earlier break-through of the dry air appears to be present.

8.0 WORK PLANNED FOR NEXT PERIOD

8.1 SARS Activity

Prepare 'Operating Permit' for larger scale experiments in FETC Building B-17.

8.2 Grain Size and Sorting Influence on Sand Pack Characteristics

Developing protocols for sand compaction, porosity, and permeability measurements relative to CATC.

8.3 Sand Pack Drying Characteristics Determination

Continue drying tests on sand packs to evaluate the effect of initial water saturation on drying profile.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

Due to delays in receiving fund, we have not been able to proceed with ordering the large tanks. Therefore, the assembly of CATC has been delayed. As result, we anticipate delays in conducting large scale experiments.

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TASK 1.3

**DEVELOPMENT OF STANDARD TEST PROTOCOLS AND BARRIER
DESIGN MODELS FOR IN SITU FORMED BARRIERS**
FETC Task No. 1.3

Quarterly Technical Progress Report
Reporting Period:
Jan.1, - Mar. 30, 1997

Work Performed Under Contract
No. 96-MO44-1

For:

West Virginia University

Under Contract
No: DE-FC21-92MC29467

For:
US Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
Morgantown, West Virginia

By:
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ABSTRACT

This work is designed to support the on-going research at DOE/METC aimed at the development of standard test protocols for insitu formed barriers. The immediate support effort is directed toward establishing DOE/METC B-17 Building as a center for laboratory , bench scale, and large scale experiments to evaluate in-situ formed barrier systems. Two systems are being evaluated initially: (1) a grout barrier system, and (2) a circulating air or desiccant barrier system. The grout and desiccant barrier systems currently were identified previously by DOE researchers as having high potential for containing the movement of liquid contaminants which may have leaked from underground storage tanks existing at various DOE former weapons sites.

Other barrier approaches are being independently evaluated under the DOE program, such as the freeze wall systems and systems that completely encapsulate the tank. The contractor will continue to monitor these programs for the development of standard test protocols in the B-17 facility. The goal is to establish the B-17 facility as the DOE/EM Focus Group Test and evaluation facility for the entire DOE program.

The focus of the CY 1997 work is to (1) Support the operation of the circulating air barrier and grout laboratory experiments to be moved into the B-17 facility; (2) complete the scale -up of these experiments in 1997, and (3) support the planning for testing additional barrier systems in 1997 and beyond.

Documents will be developed for METC's Lab Safety Committee to obtain construction and/or operating permits for the B-17 experiments and will include, but are not limited to, line drawings, and definition of operating parameters. The contractor will plan and coordinate the installation of equipment and utilities to support the experimental work to be conducted in B-17, with a special focus on using the existing equipment for research. The contractor will also assist principal investigators with the planning, hands on operation, and analysis of experimental operations.

In the conduct of this support, the contractor personnel will meet with the principal investigators and review the key design elements. Operational procedures, quality assurance and control procedures, and initial sketches for experimental equipment. After collecting the required supporting data, the contractor will prepare draft drawings and operational procedures for construction and subsequent operation of the test cells. Application for construction permits and operating permits will be prepared and submitted to the DOE/METC Lab Safety Committee.

TABLE OF CONTENTS

<u>DESCRIPTION</u>	<u>PAGE NOS.</u>
Executive Summary	v
1.0 Introduction	1
2.0 Purpose	1
3.0 Background	1
4.0 Methodology	2
5.0 Work Performed this Period	2
6.0 Results and Discussion	3
7.0 Conclusions	3
8.0 Work Planned for Next Period	3
9.0 Technical/Administrative Difficulties	4
Appendix	4
References	4
List of Acronyms	4
Glossary	5
List of Tables	
List of Figures	

Executive Summary

The purpose of the current studies being conducted by West Virginia University is to develop protocols for testing and evaluating barrier materials and barrier deployment processes that may be useful in either saturation (partially or completely saturated) condition.

The DOE in response to requirements of congressional and executive orders has developed a program designed to first contain and then remediate hazardous and mixed waste materials which have moved or are moving towards points where the potential environmental impacts are not acceptable according to federal law. DOE managers have established a policy moving forward the time of completion of most of the remediation work in the next ten years. A draft ten year plan has been drawn up to accomplish the many tasks remaining. A new sense of urgency for the completion of key research elements to support the remediation effort has been imparted to the DOE Complex. The primary thrust is to complete the remediation using current off-the-shelf technology, and conduct research to further understand the science behind some of the technologies being employed. The Federal Energy Technology Center (FETC) of the DOE has been assigned the tasks of managing one of the five focus areas for the remediation efforts. That focus area is Decontamination and Decommissioning of buildings, reactors, and other contaminated infrastructure on site. FETC also provides project management for procurements for research conducted in the Plume Containment and Remediation Focus area. FETC also in cooperation with West Virginia University has developed a Decontamination and Containment Test Center to examine proof of concept or technical feasibility of new technologies being developed in the Subsurface Containment and Decontamination and Decommissioning focus areas. The center is located in building B-17 on the FETC/DOE Morgantown WV Campus.

The purpose of the current studies being conducted by West Virginia University is to develop protocols for testing and evaluating barrier materials and barrier deployment processes that may be useful in either saturation (partially saturated vadose zone or completely saturated groundwater aquifer) condition. The application of the testing results will be in moving potential barrier materials or processes to the next phase of demonstration before deployment. The current thrust is to use containment systems that will not permanently alter the vadose zone or prevent the movement of groundwater in its normal process once the containment system is decommissioned.

The Desiccation barrier work has completed most of phase one studies which were proof of concept studies. Results show that water in the vapor phase can be removed from the subsurface as projected. The studies were conducted with a single grain size and the tests on other grain sizes and mixed grain sizes are in progress. Studies to investigate the effects of crystalline materials deposited in the pore space as the water is removed from liquid contaminants during the desiccation phase will be made to determine the impact on native permeability. Phase II is directed at conducting similar tests on a larger scale where the effects of horizontal well injection and production operations can be simulated and verified in simple models before testing operations in chaotically bedded mixtures of grain sizes, including cobbles and boulders.

1.0 Introduction

This report discusses the progress made during the 1st, quarter of 1996 by BDM in providing technical support to the WVU departments of PNGE and CEE on their tasks to develop test protocols for barrier materials, barrier deployment processes, and barrier design functions

2.0 Purpose

The purpose of the current studies being conducted by West Virginia University is to develop protocols for testing and evaluating barrier materials and barrier deployment processes that may be useful in either saturation (partially saturated vadose zone or completely saturated groundwater aquifer)condition. The application of the testing results will be in moving potential barrier materials or processes to the next phase of demonstration before deployment. Industry partnerships with companies that will seek to commercialize the technology are solicited and suggested to stakeholders where the logical application of the technology is likely to occur.

3.0 Background

Across the Department of Energy's (DOE) Nuclear Weapons manufacturing plant complex, most sites disposed of hazardous and mixed waste liquids by dumping them into pits or placing them in specially manufactured steel and concrete tanks with a design life of 30 years. Most of the tanks have exceeded their design life but are still in service. Almost half of them are suspected of leaking contaminated liquids, or are known to have leaked. In some cases the leaked contaminant material has already reached the groundwater and is approaching a point of contact with the biosphere (river bed). The use of barriers to halt movement into the biosphere where humans and animal life forms can contact the materials is an important strategy developed by DOE officials until remediation technologies are developed and implemented.

The DOE has supported testing and evaluation studies of various barrier materials over the past 20 years. Many materials and deployment processes have been tested during this period, but DOE scientists are not convinced that they have found the right barrier material and deployment process for broad scale application to the former weapons sites. A reason for this is the geographic and climatic differences in the sites. Western sites are mostly in semi-arid climates with sediment deposition by mass wasting or catastrophic depositional modes. Eastern sites are located in high rainfall types of climates where sediment cover on bed rock is thin and usually contains more fine grained sediments. Vadose zone soils have higher water saturations in the east than dry soils of the west. Barriers are generally considered as containment devices without any particular use as a remediation unit. There are potential processes and materials that can participate in the remediation processes, and these are the next step in the evaluation of the materials currently being tested.

The particular materials being tested by West Virginia University are low viscosity permeation grout materials, the desiccation barrier process, and in the near term reactive barrier materials.

4.0 Methodology

BDM reviews state-of-the-art barrier materials and processes and evaluates the potential for application at the DOE Sites. Evaluation is based on suggested approaches by the national Academy of Science Audit Teams which review DOE approaches to containment and remediation. Screening criteria are used to determine if a specific material or process will meet all of the functional requirements that have been developed to insure the safety of the general population. BDM prepares drawings and procedures for operation of different types of barrier processes it examines, and prepares reports recommending a material or process and suggesting how the material or process may be tested and verified.

5.0 Work Performed this Period

Work was initiated on the development of an operating procedure and operating permit for the scaled-up CAB studies to be conducted in an order of magnitude larger test vessel.

Discussions were held with design engineers with the company that will build the containment vessels for the scaled-up studies that will be conducted in the north bay on the ground floor of B-17.

Discussions were held with electronic design engineers who will design the Conductivity based instrumentation package to be used for data collection and process verification at the scaled-up level.

Revision of a draft report submitted during the previous quarter was initiated.

The Construction Permit was received in January, 1977, some three months after initial request submission.

BDM initiated development Quality Assurance and Quality Control Guidelines for the scaled-up Desiccation barrier project during the quarter.

A second report which deals with dual barrier system utilizing combinations of materials and processes was nearly completed during the month of January until available funds were depleted and work on the project halted until funds could be added.

6.0 Results and Discussion

One of the problems that surfaced during the review of the surface barrier, is that even when applied in a vadose zone application with a natural saturation of approximately 30 %, additional water often needs to be added during the development of the freeze barrier to insure integrity. This may result in the transport and inclusion of contaminants into the freeze barrier structure. The contaminant remains immobilized until the barrier is removed when it will again become mobile and move toward groundwater inclusion.

Discussions held with RKK Inc. the holder of patents for the Cryocell or "Freeze" barrier, claims in a recent patent that was issued, that the contaminants are excluded from the ice structure and the material is piled up in front of the barrier where it can be removed by drilling a wellbore to intercept the material and pumping it to the surface.

Dual barrier systems composed of a desiccation barrier and a freeze barrier seem to be the ideal combination of non-permanent barrier systems. The Circulating air associated with the desiccation barrier can be monitored for presence of contaminants, and will be the prime mover in the decommissioning process for the freeze barrier when it is no longer needed.

The concept of a desiccation barrier and a reactive barrier also has merit. A postulated application is to install a reactive barrier (which could be a liquid or a solid material) above a CAB to catch and adsorb contaminants while allowing water to continue to move downward where it will be removed by the CAB in its normal function.

7.0 Conclusions

As DOE moves forward with the concept of developing non-permanent non-polluting barrier materials, the concept of utilizing two (Dual Barrier System) non-permanent barriers is one that needs to be pursued further with additional research.

A potential area of research associated with the freeze barrier is to examine the types of materials that could be included in the barrier which will adsorb contaminants and prevent the decommissioned barrier water from polluting the groundwater aquifer. Such a material is by definition a reactive barrier material.

8.0 Work Planned For Next Quarter

Work planned for next quarter is the purchasing of equipment and installation of the large scale experimental apparatus for the desiccation barrier tests (CATC).

After the equipment has been built, an operating permit will be submitted to the DOE/FETC lab

Safety Committee for review and approval.

9.0 Technical/Administrative Difficulties

BDM was not funded during the current quarter. BDM thinking that funding would be forthcoming in early January, accepted a risk that funding would be supplied and authorized the expenditure of \$34,500.00 dollars or 16 percent of the project total funds for the contract to keep the project moving. To date funding has not been received, although WVU contracts officials advise that funding is eminent. The loss of 2 months out of the quarter and a reduced level of funding will have a deleterious effect on the completion of the proposed work on time.

Funding levels may impact the completion of the desiccant barrier project. The budget awarded was approximately fifty percent of the budget requested. Cuts in the instrumentation system may impact the quality of data collected during the scale-up operations.

Appendix

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List of Acronyms

CAB - Circulating Air Barrier

DOE -Department of Energy

METC-Morgantown Energy Technology Center

FETC -Federal Energy Technology Center (Combining of METC & PETC)

RKK -Ronald K. Kreig, Inc

UST -Underground Storage Tank

CATC -Circulating Air Test Cell

Glossary

Vadose zone - Soil zone in which there is a mixed saturation of fluids in the pore space which includes water and air.

TASK 1.4

**Laboratory Studies & Field Testing at
the DOE/RMI Extrusion Plant
1997 FETC Task No. 1.4**

Quarterly Technical Progress Report

Reporting Period:

January - March 1997

Work Performed Under Contract

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For:

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Office of Fossil Energy

Federal Energy Technology Center

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ABSTRACT

Continuing development of the use of Prefabricated Vertical Drains for Enhanced Soil Flushing technology is progressing with application of the technology at a private-sector site that was a former uranium processing facility for the US Department of Energy. The site, located in Ashtabula Ohio, is owned by the RMI Titanium Corporation. It has both radiological and hazardous materials contamination in soil and groundwater mediums which are limited to a 7 acre area of a 26 acre site. The facility terminated all production operations in 1988 and since then have focused efforts on the decontamination and decommissioning of the facility which includes remediation of the soil and groundwater contaminated by low-level uranium and TCE.

WVU's Civil and Environmental Engineering Department work with RMI Environmental Services is made possible through support from DOE's Federal Energy Technology Center - Morgantown and DOE's Ashtabula Area Office - RMI Decommissioning Project. WVU, DOE-Ashtabula, and RMI are working together in problem identification and with bringing WVU's technical resources in technology development to bear on several of RMI's decontamination and decommissioning projects. Current work for 1997 consists of performing a groundwater modeling study of the site and a treatability study using surfactants to remove TCE from contaminated fine-grain fraction soil and groundwater.

TABLE OF CONTENTS

Disclaimer	ii
Abstract	iii
Executive Summary	1
Introduction	1
Purpose	2
Background	2
Methodology	3
Work Performed this Period	3
Conclusions / Next Period Work Plan	9

List of Tables		
Table 1	Food Grade Surfactants	8
Table 2	Non-Food Grade Surfactants	9

List of Figures		
Figure #1	Groundwater Contour - Plan	4
Figure #2	Cross Section - Elevation	5
Figure #3	Cross Section - Velocity Profile	5
Figure #4	Calibration Curve	5

EXECUTIVE SUMMARY

Work performed this quarter included completion of the OSHA 1910.120 40 Hour Hazardous Waste Basic Worker Course for nine WVU CEE students and faculty who are involved with the field research activities at the RMI site. Two faculty members from WVU visited the RMI facility in February to observe their soil washing process for removal of uranium particles from site soils. Subsequent to this meeting, representatives from RMI and DOE's Field Office visited WVU to observe operation of the SpinTek ultra-filtration apparatus and to further discuss application of the PVD remediation technology.

Efforts on the groundwater modeling have resulted in characterization of the RMI site by completing calibration of the MODFLOW numerical model. Comparisons of the actual observed field recorded data of the groundwater well measurements with the model generated results are presented and discussed. Investigation into the treatability study for surfactant use to remove TCE from contaminated soils have progressed to identification of suitable food-grade surfactants for use at the RMI site.

INTRODUCTION

The research activities presented in this report are part of the WVU Cooperative Agreement's initiative for bringing innovative environmental remediation technologies to field-scale demonstrations. This project is a concerted effort between WVU's Civil and Environmental Engineering Department, U.S. Department of Energy's (DOE) Federal Energy Technology Center - Morgantown and DOE's Ashtabula Area Office - RMI Decommissioning Project, and RMI Environmental Services. The goal of the work during Phase I efforts consist of performing preliminary engineering studies necessary for subsequent design and installation of the Prefabricated Vertical Drain Enhanced Soil Flushing technology at the RMI facility located in Ashtabula, Ohio. This report discusses the progress made for the current reporting period.

The Phase I research includes: an information review of previous engineering studies/reports performed at the RMI facility regarding the site corrective measures; groundwater modeling to characterize a TCE contaminant plume; laboratory bench-scale testing to investigate performance requirements of the prefabricated vertical drains in site soil, and chemical laboratory analysis as part of a treatability study addressing the site contaminated soil. Results of the Phase I efforts will produce: identification of candidate locations for installation of a PVD test pad area; characterization of the PVD performance requirements necessary for operation at the site; and determination of contaminant clean-up levels achieved using RCRA approved, food-grade surfactants on TCE contaminated site soils.

PURPOSE

As environmental remediation technologies progress through the research process, it is important to continually evaluate the research goals and determine the value-added of the developing technology. For this project, the research objectives of Phase I are two-fold: The first is to perform a limited groundwater model of the RMI Ashtabula site for identifying candidate locations suitable for the Phase II field test pad research; The second objective is to perform laboratory bench-scale experiments for the design of the PVD system used in the field application, and to perform treatability studies to determine clean-up levels achieved using RCRA approved, food-grade surfactants on the TCE contaminated soils at the RMI facility. This research is specific to addressing key issues necessary for the design, installation, and operation of the test pad experiment, and remediation of the TCE contaminated soils proposed for a subsequent Phase II.

BACKGROUND

The RMI Titanium Company (RMI) Extrusion Plant is located in Ashtabula County, Ohio. The facility consists of 25 buildings and occupies 7 acres of a 26 acre site. The facility is contaminated with both radiological and hazardous materials resulting from previous operations for the US Department of Energy. The primary function of RMI, since 1962, has been the extrusion and closed-die forging of metallic depleted, natural, and slightly enriched uranium (U) used in the production of nuclear fuel elements for defense production reactors, extrusion operations for other government agencies, and the private-sector. Extrusion operations ceased in 1990, since then the Nuclear Regulatory Commission (NRC) has required the facility to implement accelerated site clean-up pursuant to its Site Decommissioning Management Plan. The US Environmental Protection Agency has issued RMI a Resource Conservation Recovery Act (RCRA) Hazardous Waste Management Permit with conditions mandating clean-up of an existing Corrective Action Management Unit. DOE's office of Environmental Restoration and Waste Management (EM) has contracted RMI to conduct the RMI Decommissioning Project by removing all radiological and hazardous contaminants to levels which permit the facility and adjacent areas to be released for unrestricted use.

The principal contaminant on equipment, buildings, and surrounding open space areas is uranium. In addition to the uranium, soil and groundwater are contaminated with Trichloroethylene (TCE) and Technetium 99 in limited areas of the site. Clean-up of the facility is slated to continue for the next several years due to technology limitations and the unique challenges which face RMI in handling the large physical size of the manufacturing equipment and facility.

METHODOLOGY

The project objectives for Phase I are two fold: The first is to perform a limited groundwater model of the RMI Ashtabula site for identifying candidate locations suitable for the Phase II field test pad research; The second objective is to perform laboratory experiments for the design of the PVD system for the field application, and to perform treatability studies to determine clean-up levels achieved using RCRA approved, food-grade surfactants on the TCE contaminated soils at the RMI facility. This research is specific to addressing key issues necessary for the design, installation, and operation of the test pad experiment, and remediation of the TCE contaminated soils proposed for Phase II. The work scope of the first phase activities is broken down into the following subtasks:

Subtask 1.1 Site Data Evaluation

Subtask 1.2 Predictive Modeling

Subtask 1.3 Lab Scale Physical Testing

Subtask 1.4 Lab Scale Chemical Testing

Subtask 1.5 RMI Environmental Site Support

WORK PERFORMED THIS PERIOD

Accomplishments made during this period are reported by subtask in the following discussions.

Subtask 1.1 Site Data Evaluation

This work focuses on obtaining and correlating existing site information pertinent to the fate of the subsurface contaminants and groundwater as reported previously by RMI in documents spanning the years from 1980 to 1996. The information obtained from this review was used to establish groundwater modeling parameters with respect to soil borings and groundwater monitoring well information that reports piezometric and contaminant concentration data. A trip to the field site was performed by Drs. Cook and Quaranta to observe the RMI soil washing process, meet with RMI engineers and administrators to discuss subcontracting issues, and further define site support activities.

Subtask 1.2 Predictive Modeling

Work in this subtask focuses on development of a groundwater model for the site and performing numerical simulations for prediction of subsurface contaminant transport. During this reporting period the following accomplishments were made. New computer software and hardware was purchased and received for modeling purposes. The Visual Modflow groundwater modeling package sold by Waterloo Hydrogeologic was purchased and installed on a new Pentium based computer. This software uses the US Geological Survey program MODFLOW and includes the MODPATH and MT3D shell programs for contaminant transport computations.

Modeling of the RMI site has progressed with the calibration of the MODFLOW simulations for five time periods as steady-state events, and a transient simulation having five time step intervals. Representative results of the hydraulic contour modeling for a 1986 time period output are shown in Figures 1 through 4. The calibration effort is a trial and error evaluation process. It is used to verify the model calculations with field observed data for exacting the model's performance in predicting transient groundwater conditions and for simulating impacts or effects of remediation options.

Figure #1 shows the model output as piezometric head contours of the groundwater flow within the confines of the RMI facility. Figure #2 shows a representative cross section of Figure #1 and outlines the equipotential contours. Figure #3 illustrates the velocity profile of the groundwater movement occurring through a cross-section of the aquifers. Figure #4 is a representative calibration curve of the numerical model showing comparison between the calculated heads versus the observed head values. The closeness of the data points to the forty-five degree line reflects the precision of the model solution.

Figure 1: Groundwater contour - Plan

Figure 2 : Cross Section - Elevation

Figure 3: Cross Section : Velocity Profile

Figure 4 : Calibration Curve

Subtask 1.4 Lab Scale Chemical Testing

During this reporting period progress advanced with the literature review. Several journal and conference proceedings papers were obtained addressing the TCE degradation using food-grade surfactants applied in clay and sand soils. The literature review has identified several of the following food-grade surfactants as candidates for column testing. Discussions of these topics are presented below.

Properties of TCE as a Contaminant

TCE is a colorless liquid at room temperature, is less viscous than water, will sink in water, and will dissolve slightly in water. Additionally, this chemical is carcinogenic, and as a result, has very low minimum contaminant limits. Microorganisms are active in the decomposition of organic matter, and further study of the organisms indicated the successive transformation of PCE to TCE, and of PCE and TCE to other compounds. The results of this research have several unfortunate implications. First, the identification of the source of contamination becomes much more difficult. As a result, the abatement of the source of discharge takes more time, during which additional contaminants may be introduced into the environment. Second, treatment methods developed to remove one compound may prove ineffective in removing the breakdown compounds. Third, TCE may be transformed into contaminants that have lower MCLs or higher MCLs, perhaps making remedial activities more difficult to accomplish.

Column Tests

Column tests are used in the search to figure out the solubilization efficiency and applicability of surfactants. Various column dimensions, surfactant concentrations, flow rates, soil amounts, etc. are utilized, depending on the contaminant, surfactant, and soil characteristics. The soil to be used in these tests are characterized by grain size; percent moisture; C,H,N analysis; phosphorus content; TOC; density; and permeability. The soil, prior to being placed in the columns, is dried, homogenized, kept in an oven overnight at 105 C, and then passed through the appropriate sieve.

Various column studies accomplished by researchers can be summarized as follows:

- * Dipak Roy, et al. attained 42% removal of automatic transmission fluid with 8 mM of the surfactant SDS in a sandy-silt soil.
- * Abdul S. Abdul, et al. observed an 86% removal of PCB from a sandy soil by the surfactant Witconol SN70.
- * Kurt D. Pennell, et al. removed dodecane from a silica sand with the help of the surfactant POE(20) sorbitan monooleate.
- * David A. Sabatini, et al. accomplished an 87% removal of PCE from alluvium (72.4% sand) with 15-21 pore volumes of 5 wt% POE(20) sorbitan monostearate.

- * Medha M.Joshi, et al. attained a 60-70% removal of polycyclic aromatic hydrocarbons (PAHs) from sand with 4% of the surfactant Igepal CA-720.

Surfactants Reviewed

Food Grade (edible) Surfactants Acting on TCE: A restriction of using surfactant enhanced subsurface remediation is obtaining regulatory approval for the injection of surfactants. This obstacle is common to all chemicals being considered for use in subsurface remediation. However, the use of surfactants with U.S. Food and Drug Administration direct food additive status (food grade or edible surfactants), increases the likelihood of gaining regulatory approval. Another advantage of using food grade surfactants is that they are biodegradable. A disadvantage, though, is that these surfactants suffer losses due to phase separation (precipitation, formation of liquid crystals) and adsorption. In one of the studies done, David A. Sabatini, et al. found the following results:

- * On a molar basis, T-MAZ-20 and T-MAZ-80 showed slightly higher adsorption than the AOT and much higher adsorption than the DOWFAX disulfonates.
- * On a weight basis, T-MAZ-20 and T-MAZ-80 showed even higher adsorption losses than the anionic surfactants (due to the high molecular weight for nonionic surfactants).
- * T-MAZ-60 and T-MAZ-28 showed much greater adsorption at higher concentration due to liquid crystal formation.

From the literature review, 14 food grade surfactants that can be used on TCE have been identified. These surfactants are listed in Table 1.

Table 1-Food Grade Surfactants

SURFACTANT NAME	HYDROPHILE-LIOPHILE BALANCE (HLB)	MOLECULAR WEIGHT (gr/mole)	TYPE
Sodium dodecyl sulfate (SDS)	40.0	288	A*
POE (20) sorbitan monolaurate (T-MAZ 20)	16.7	1266	N*
POE (20) sorbitan monostearate (T-MAZ-60)	14.9	1310	N
Sodium mono and dimethyl naphtalene sulfonate (SMDNS)	NA	260	A
POE (80) sorbitan monolaurate (T-MAZ-28)	19.2	3866	N
POE (20) sorbitan tristearate (T-MAZ-65)	10.5	1842	N
POE (20) sorbitan monooleate (T-MAZ-80)	15.0	1308	N
Bis-2-ethylhexyl sodium sulfosuccinates (Aerosol OT)	NA	445	A
Sodium stearyl-2-lactylate	10.0	450	A
Calcium stearyl-2-lactylate	NA	894	A
Triglycerol monooleate (MAZOL-PGO-31K)	6.2	536	N
Sorbitan monooleate (S-MAZ-80K)	4.6	428	N
Glycerol monooleate (MAZOL-300K)	3.8	356	N
Sorbitan trioleate (S-MAZ-85K)	2.1	956	N

*A=anionic; N=nonionic

The first three surfactants in this table (SDS; T-MAZ-20; and T-MAZ-60), have been selected by researchers as the most effective food grade surfactants, and therefore will be used in the laboratory experiments of this research.

Other Types of Surfactants Acting on TCE:

Surfactants that are not food grade and active on TCE have been identified, as shown in Table 2. Even though these surfactants have the advantage of less loss due to precipitation and adsorption, they have the disadvantage of being harder to gain regulatory approval for use in subsurface remediation.

Table 2-Non-Food Grade Surfactants

SURFACTANT NAME	TYPE
FC-760	N*
Igepal CO-730	N
Monawet MM-80	A*
Rexophos 25/97	A
Triton X-114	N
Witconol H-31A	N

*N=nonionic; A=anionic

Subtask 1.5 RMI Environmental Site Support:

During this period work on the subcontract for services with RMI Environmental was developed and submitted to WVU Sponsored Programs for processing. The process has been initiated for WVU to release a subcontract to RMI for the site support services. The work performed under this release deals with site construction operations for obtaining soil and groundwater samples from the RMI site for subsequent laboratory testing at WVU.

CONCLUSIONS/WORK PLANNED FOR NEXT PERIOD

No final conclusions of this work are made as the project continues to unfold. For the next reporting period key areas of research include: contaminant transport modeling of the TCE plume, and design of the TCE treatability experimental apparatus. It is expected that physical sampling of the RMI site soil will take place and for WVU to take possession of the soils for experimental research purposes.

TASK 1.5

Use of Drain Enhanced Soil Flushing (DESF) for Contaminants Removal 1997 FETC Task No. 1.5

Quarterly Technical Progress Report
Reporting Period:
January - March 1997

Work Performed Under Contract
No. DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
Morgantown, West Virginia

By:
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Department of Civil and Environmental Engineering
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April 1997

TABLE OF CONTENTS

Abstract	1
Introduction	2
Purpose	2
Background	2
Work Performed This Period	3
Conclusions	5

ABSTRACT

The main objective of this research is to investigate major parameters affecting the efficiency of contaminant extraction using PVD systems. The concept aims at enhancing the effectiveness of the soil flushing process by installing the PVD to shorten the drainage path of the flow and provide redundancy and wide coverage to capture the subsurface pollutants. The laboratory phase and pilot scale field test have proven the feasibility of the concept. The objective of this proposed phase is to prove and evaluate the technology performance in the field on a full scale. Hence, the overall cleanup of a chosen site in accordance with acceptable groundwater standards. In addition, a comparison with the conventional vapor extraction system, proposed at the same test site, will be conducted.

Work conducted so far on the project included laboratory tests, pilot scale testing and analytical model development. Laboratory tests included flexible wall permeability tests on soil specimens with and without a PVD installed. Bench-scale tests using large scale reactors, referred to as Smear Cells, were conducted to evaluate the equivalent diameter, long term performance, and zone of influence of the PVDs. In addition, several pilot scale Contaminant Recovery Cells (CRCs) were used to evaluate the flow characteristics of the soil samples and the concept of soil flushing using the PV drains in various installation arrangements. Sodium chloride (NaCl) tracer (100 and 1,000 mg/l) was used for discerning the concept of contaminant recovery using the PVDs system. Soil samples were prepared from sand and a mixture of 80% Ottawa sand and 20% kaolinite. An analytical model for the design of PVD systems for soil remediation was also developed. The field site selection and investigation was done following procurement of a site in Weston, WV. This site is owned by Ashland Petroleum Co. and has LNAPL and DNAPL contamination present in the groundwater. A Pilot Scale field experiment was performed to find air and fluid flowrates per PVD. Also, several grab samples were acquired during testing and tested for BTEX concentrations.

1.0 INTRODUCTION

This project researches the use of Prefabricated Vertical Drain (PVD) systems for remediation of contaminated sites with fine-grained soils. PVD systems are relatively simple in operation and installation. This type of system can be expected to reduce remediation costs while at the same time increase the efficiency of the remediation effort.

Currently, PVDs are used for soil improvement and consolidation. Other than the work performed in the first two phases of this project, no research has been done to test the feasibility of using PVDs for enhanced soil flushing.

2.0 PURPOSE

The primary goal of this quarter was the continuation of laboratory work on the chemical aspect of the site remediation technology. Also, to begin the implementation of the full scale test. This included:

- 1) The continuation of adsorption and desorption batch testing, using water as the flushing solution.
- 2) The collection of liquid samples from the chosen site. These samples will be used in a bench scale test to determine the clogging potential of the PVD.
- 3) The collection of liquid samples to be used for gas chromatograph analysis. This will yield the extent of contamination at the site before the full scale is implemented.
- 4) The graduated researchers, (Williamson and Sabodish) also attended the 40-hour HAZWOPER training course.

3.0 BACKGROUND

The PVD system is used to shorten the drainage path of the groundwater flow and therefore promote subsurface liquid movement and expedite the soil flushing process in low permeability soils. Proved technologies exist for the in situ soil flushing for sites with granular soils (hydraulic conductivity approximately 1×10^{-2} cm/s). However in the case of sites where the soil is fine-grained with corresponding hydraulic conductivity ranging 1×10^{-3} to 1×10^{-7} cm/s, implementation of the traditional soil flushing technology is ineffective and cost prohibitive.

Previous work conducted on the project included laboratory tests, pilot scale testing and analytical model development. Laboratory tests included flexible wall permeability tests on soil specimens with and without a PVD installed. Bench-scale tests using large scale reactors, referred to as Smear Cells, were conducted to evaluate the equivalent diameter, long term performance, and zone of influence of the PVDs. In addition, several pilot scale Contaminant Recovery Cells (CRCs) were used to evaluate the flow characteristics of the soil samples and the concept of soil flushing using the PV drains in various installation arrangements. Sodium chloride (NaCl) tracer (100 and 1,000 mg/l) was used for discerning the concept of contaminant recovery using the PVDs system. Soil samples were prepared from sand and a mixture of 80% Ottawa sand and 20% kaolinite. An analytical model for the design of PVD systems for soil remediation was also developed. The field site selection and investigation was done following procurement of a site in Weston, WV. This site is owned by Ashland Petroleum Co. and has LNAPL and DNAPL contamination present in the groundwater. A Pilot Scale field experiment was performed to find air and fluid flowrates per PVD. Also, several grab samples were acquired during testing and tested for BTEX concentrations.

4.0 WORK PERFORMED THIS PERIOD

The primary objective during this quarter where extensive research was done on the adsorption and desorption kinetics of the soil found at the Weston site. This research was conducted to determine how effective the soil was in the adsorption of the chosen model contaminant, in this case, ethyl benzene. From the enclosed figures, (figs 1-4) one can see the isotherms which are achieved by batch testing. Figures 1-4 show the adsorptive capacity of the soil. Figures 5-8 show the desorptive capacity of the soil while using water as the flushing solution. There were two distinct tests performed to determine the adsorption and desorption kinetics of the soil. Trials 1a and 1b show the adsorptive capacity of the soil while allowing the soil to equilibrate with the contaminant for a period of 19 days. Trials 3a and 3b show the adsorptive capacity of the soil while allowing the soil to equilibrate with the contaminant for a period of 10 days.

The procedure for this type of testing is as follows. Samples are prepared using a known amount of soil and a known concentration of ethyl benzene. The contaminant is in solution with water. The solution of ethyl benzene and water is then placed in a test tube with the known amount of soil. The test tubes are then placed on a shaker table for a known amount of time. This time (in days) is referred to as the equilibrium concentration. After the samples are removed from the shaker table, the soil needs to be removed from the solution. This was accomplished using a refrigerated centrifuge. The samples are centrifuged at 4°C for a time of 20 minutes. After the samples are centrifuged, the supernatant is removed from the test tube and placed in a 30ml separatory funnel. In order to analyze the samples, the water must be removed from the supernatant. This is accomplished by the addition of a solvent to the funnel. In this case, the selected solvent was carbon disulfide. Approximately 2ml of carbon disulfide is added to the funnel. Then, the funnel is shaken, by hand, for a period of five minutes. This is done to ensure total extraction of the ethyl benzene by the solvent. The funnels are then placed in a rack to allow the carbon disulfide to separate from the water. The density of carbon disulfide is heavier than

that of water, so it will sink to the bottom of the funnel, and two distinct layers will be visible. The solution which remains at the bottom of the funnel is then drained from the bottom of the funnel and is placed in 4ml glass vials. These samples are then injected into a gas chromatograph fitted with the appropriate column for the detection of BTEX. Injection was done via manual injection. The report achieved gave the concentration of ethyl benzene in solution reported in parts per million (ppm). With the information now available, the adsorption isotherm could now be constructed. Figures 1-4 show these isotherms using the equilibrium concentration (mg/l) vs. The mass adsorbed (mg/kg) on the axes. From these plots, information regarding the adsorptive capacity of the soil can be achieved. These constants are K_p , sorption capacity, $1/n_p$, sorption intensity. From these constants, the partitioning coefficient and Freundlich isotherm equations can be determined. These values can be seen on the enclosed figures. From the reviewed literature on adsorptive and desorptive kinetics, it has been determined that the higher the values of K_f and $1/n_f$, the greater the sorption capacity for the medium. Results achieved here are very comparable to results found by Zytner, 1994. He found the value of $1/n_f$ and K_f for clay to be 1.01 and 1.64 respectively. The results achieved from this research show a $1/n_f$ value and K_f value of approximately 1.02 and .512. The differences in the K_f values may be attributed to the different soil used in this research than in Zytner's.

The same procedure outlined above was performed to determine the desorptive capacity of the soil. This was accomplished by filling the test tubes which contained soil with an amount of water which was equal to amount of supernatant that was removed. The same procedure of placing the tubes on the shaker table, and method of extraction and analysis was followed. From these results obtained, it was then possible to construct desorption isotherms for the site soil. These isotherms can be seen in figures 5-8. Trials 1a and 1b were allowed to equilibrate for a period of 19 days, while Trials 3a and 3b were allowed to equilibrate for a period of 10 days. The resulting values of $1/n_f$ and K_f could then be determined. These values were determined to be 1.01 and .411 respectively. These values cannot be compared to results found by Zytner's results because no desorptive values were determined by his testing. It can be seen from the enclosed figures, that the time the samples are allowed to equilibrate on the shaker table seems negligible after 10 days. Tests will be conducted next quarter to verify this assumption.

A secondary goal was started late in this quarter. The secondary goal employs the use of smear cells and a laboratory scale extraction PVD system. Smear cells have been used previously to determine zone of influence of the PVDs. The smear cells are setup to simulate groundwater and an unconfined aquifer. The secondary goal, termed PVD Performance, operates under the same functions excluding the water. A large amount of contaminated groundwater along with free product was extracted from the Weston site via monitoring wells. The contaminated water and free product is then cycled through the system to determine the effect the contaminant has on the geotextile filter jacket encompassing the flow channels of the PVD. An in-line electronic flowmeter connected to the extracting PVD shows fluctuations in the flow from the PVD at a constant vacuum pressure. These fluctuations determine points of interest. If the flow decreases sharply, then the PVD is assumed to be clogged. The test is long term, therefore it is run constantly. Data thus far collected is not ample to reduce conclusive results. Next quarter results

from the PVD Performance testing should be available.

The third objective is the testing of liquid samples which were extracted from monitoring wells was done. Samples were extracted from monitoring wells at the Weston location. This was accomplished by inserting a rubber hose into the well, and applying a vacuum to the other end. Two samples were taken from each monitoring well, resulting in 8 samples. The samples were then packed in ice to prevent volatilization and tested for concentration of ethyl benzene. The results of the testing showed no traces of ethyl benzene. This may be attributed to the inaccuracy of the gas chromatograph in detecting concentrations which are less than 1 ppm. Research is still being conducted as to why this may have occurred.

Also this quarter, the two graduate researchers, (Williamson and Sabodish) attended the 40-hour OSHA Hazardous Waste Operations and Emergency Response(HAZWOPER) course. This course was offered by the West Virginia University Safety and Health Extension. The course was successfully passed by both graduate students and are now HAZWOPER certified.

5.0 CONCLUSIONS

Several tasks that were vital to the implementation of the full scale project were completed or will be completed in the near future. This includes the continuation of batch testing, the determination of the PVD Performance, and the determination of the extent of contamination which is now present at the site. Results from the clogging potential of the geotextile will be available next quarter along with continual results from batch .

6.0 WORK PLANNED FOR NEXT PERIOD

Tasks to be performed next period involve the set-up of the piping network for the full scale test. Also, large scale testing to determine the smear effect of PVD installation will be examined. The testing to determine the clogging potential of the PVD will also be completed, and results will be presented next quarter. As for the chemical side of the project, tests will be done to determine the CMC (critical micelle concentration) and the MSR (molar solubility ratio) of the selected surfactant, Tergitol. Also, testing will be done to determine the extent of volatilization and biodegradation of ethyl benzene. In addition, Permeability testing will be performed in order to determine the hydraulic conductivities of the soil using water as the permeant, and to determine the hydraulic conductivity using varying amounts of Tergitol as the permeant. In addition to the above, column test will be performed to determine the number of flushes needed to remediate a contaminated soil sample.

Adsorption Isotherm (Trial #1a) 19 Days with water

Figure 1

Adsorption Isotherm (Trial #1b) 19 Days with water

Figure 2

Adsorption Isotherm (Trial #3a) 10 Days with water

Figure 3

Adsorption Isotherm (Trial #3b) 10 Days with water

Figure 4

Desorption Isotherm (Trial #1a) 19 Days with water

Figure 5

Desorption Isotherm (Trial #1b) 19 Days with water

Figure 6

Desorption Isotherm (Trial #3a) 10 Days with water

Figure 7

Desorption Isotherm (Trial #3b) 10 Days with water

Figure 8

FOCUS AREA 2.0

Mixed Waste Characterization, Treatment & Disposal

TASK 2.1

**ANALYSIS OF THE VORTEC CYCLONE MELTING SYSTEM (CMS™)
FOR REMEDIATION OF PCB CONTAMINATED SOILS USING CFD**

FETC Task No. 2.1

Quarterly Technical Progress Report
Reporting Period: January 1 - March 30, 1997

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For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
Morgantown, West Virginia

by

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ABSTRACT

In this report, the results of flow and heat transfer analysis for selected retrofits of a cyclone melting system (CMS) are presented. The analysis is based on computational fluid dynamics (CFD) simulations. The assumptions, approximations, and the implications of the results with respect to the gas residence time, flow patterns, and temperature distribution, and conversion and destruction rates for selected chemical species are discussed. Problem specific mathematical models are presented. Recommendations are made for improved flow conditions that will lead to longer residence times, and higher conversion and destruction efficiency. Specific application target is the CMS^{TM} of Vortec Corporation. The results are summarized in the executive summary section as they become available for each quarterly reporting period.

TABLE OF CONTENTS

Abstract	iii
List of Tables	v
List of Figures	vi
Executive Summary	viii
1.0 Introduction	1
2.0 Purpose	2
3.0 Background	3
4.0 Methodology	3
5.0 Work Performed this Period	4
6.0 Results and Discussion (Current Period)	5
7.0 Conclusions (Current Period)	7
8.0 Work Planned for Next Period	7
Appendices	
A. References	8
B. Tables	10
C. Figures	16

LIST OF TABLES

- 6.1 Calculated Outlet Temperatures with No Radiation
- 6.2 Calculated Outlet Temperatures with the P1 model with Varying Properties
- 6.3 Calculated Outlet Temperatures with the WSGG added to the P1 model
- 6.4 Preliminary Results from the Case Where Particulate Matter was Included as Soot
- 6.5 Summary of Total Heat Transfer Rates

* All tables are given in Appendix B unless otherwise noted.

LIST OF FIGURES: Appendix B

- 6   Vortec CMS™ System Sketch
- 6.1a   Temperature Variation Along Axial Direction
Center of Flue Gas
P1 Model w/Varying Properties at .6kg/s
- 6.1b   Temperature Variation Along Axial Direction
Center of Preheated Air
P1 Model w/Varying Properties at .6kg/s
- 6.2a   Temperature Variation Along Axial Direction
Center of Flue Gas
WSGG Added to the P1 Model
- 6.2b   Temperature Variation Along Axial Direction
Center of Preheated Air
WSGG Added to the P1 Model
- 6.3a   Temperature Variation Along Axial Direction
Center of Flue Gas
Soot Model Added to WSGG and P1 Model
- 6.3b   Temperature Variation Along Axial Direction
Center of Preheated Air
Soot Model Added to WSGG and P1 Model

*   All figures are given in Appendix C unless otherwise noted.

EXECUTIVE SUMMARY (current period)

Most of the research effort in this quarter was focused on the radiation heat transfer analysis of the recuperator which is in design stage. Calculations were performed with combustion air flow rate ranging from 0.2 to 1.0 kg/s, and the flue gas stream at 1.0 kg/sec. The inlet temperature of the flue gas was assumed to be 1636 K (2485 degree F). The combustion air stream was assumed to be at 300 K (80 degrees F). The outer wall of the recuperator was assume to be perfectly insulated. The heat transfer rates with and without radiation are compared. The two extreme cases, namely, assuming the flue gas as a clear gas, and as a gray gas with steam and carbon dioxide being the major components were studied. Preliminary calculations were also made including the particulate matter as soot. The results show that with radiation the heat transfer rate form the flue gas to the air stream increases by about 30% with a clear gas, and about 200% with the gray gas assumption. Radiative heat transfer accounts for 22% and 65% of the total heat transfer for the later two cases, respectively. The computations including particulate matter is not conclusive at this stage. However, we believe that the case with the gray gas assumption can be taken as the worst case for analysis of species concentrations.

1.0 INTRODUCTION

The low level radioactive waste and other hazardous waste materials such as Polychlorinated Biphenyls (PCB) has become an important risk factor in endangering human health and environment. Each year millions of tons of contaminated soil is identified and classified for clean up only in the United States. An efficient and safe way to reduce and in most cases eliminate this risk factor to health and environment is the so called vitrification and remediation process via thermochemical treatment at high temperatures. In this process the hazardous waste and the contaminated residuals are heated, where most organic contaminants are vaporized and oxidized, while the inorganic contaminants are immobilized within a glassy, vitrified matrix. One such system has been developed by Vortec Corporation (here and after referred as Vortec) which has been demonstrated to be flexible in processing a wide variety of contaminated soils, sludges, and other hazardous materials. The system consists of a counter rotating combustor (or melter), a separation unit where liquid phase is separated from the gas phase, a residence chamber to provide reaction time to complete the conversion processes, and a recuperator which provides further residence time in addition to providing hot air/gas to be used as a heating source for the combustor (see Figure 1.0). The design, refinement, and retrofits of such systems require calculation of optimum design parameters such as residence time, heat loss, pressure drop, mixing rates, and the flow field dependent conversion and destruction (C&D) rates.

In practice where detailed flow and temperature distributions are not available, usually a plug flow assumption is used to calculate the gas residence time from empirical relations (see e.g. Van Dell et al., 1994a, 1994b; Van Dell and Mahle, 1992; Duvall and Rubey, 1977). This is a crude assumption and it may lead to significant errors. For example Van Dell and Mahle's calculations show a two-three fold difference in residence time even in a controlled, bench-scale laminar flow reactor due to temperature differences within the same reactor. Such errors directly translate to the calculation of species concentrations, hence may lead to quite erroneous information for design of PCB destruction systems. Celik and Chattree (1988) developed a rigorous method for calculating mean gas and particle residence times in an entrained flow reactor. This method (which will also be used for the present analysis) takes into account the gas temperature and density changes locally in the reactor as well as the recirculation zones. Their results also showed significant differences between the particle and gas residence time due to inertia and gravitational effects.

Furthermore, the destruction rates of PCB's are usually modeled by a simple Arrhenius type rate expressions where the gas temperature is assumed to be constant. To obtain more accurate results the gas temperature needs to be varied locally within the reactor and the reaction rates should be generalized to account for reformation of the compounds. Thus the resulting rate expression should include both the coefficients for the rate of formation as well as for the rate of destruction. Such a model has been proposed by Van Dell et al. (1994a) and it is a good candidate to be used in the present analysis.

The computation and optimization of the above mentioned design parameters can be accomplished in a cost effective way using Computational Fluid Dynamics (CFD) analysis. With CFD codes the flow field, temperature distribution, and species concentrations can be calculated at many discrete spatial points inside the reactor as well as temporal (or transient) variations. These primitive flow variables, in turn, can be used, by appropriate post processing to calculate integral design parameters of interest. The calculated design parameters along with the detailed information and virtual visualization of flow patterns can increase our understanding of the physical processes involved, and hence reduce the risk and the cost of experimental testing and/or validation.

2.0 PURPOSE

The primary objective of the proposed research work is to provide via computer simulations, needed design information such as gas residence time, temperature distribution, species concentrations, and heat and pressure losses for refining the proposed design and/or validation of the assumptions made during the design of sub-component of a cyclone melting system (CMS) currently under development by the Vortec Corporation. The analysis is to focus on specific needs for remediation of PCB contaminated soils. A secondary objective is to develop and implement a global kinetic model for the formation and destruction of selected PCB's such as *o*-dichlorobenzenes in conjunction with a readily available computational fluid dynamics (CFD) code. This model will rely upon experimental information available in the literature (see. e.g. Van Dell et al., 1994a, 1994b; Sethuraman et al., 1992; Ritter and Bozzelli, 1990; Tsang, 1990;). If successful, the proposed analysis will allow important enhancements for processing capabilities, and it will further reduce the development risk associated with commercial implementation of the CMS^{TM} technology.

3.0 BACKGROUND

An extensive research and development (R&D) program is being implemented by the Vortec Corporation with considerable funding from the Department of Energy(DOE) to provide waste processing equipment and services for industrial and institutional waste generators, and to manufacture glass and ceramic products from these waste materials. In particular, Vortec is planning to modify their existing high temperature waste processing and recycling units and retrofit it with appropriate units so that the system can be used for efficient remediation of PCB (Polychlorinated Biphenyls) contaminated soils. This requires determination of optimum design parameters, such as residence times, heat loss, pressure drop, and size for each subcomponent of the system. The conceptual system consists of four interconnected sub-components, namely, a cyclone melting system (CMS^{TM}), separation reservoir, recuperator base (or residence chamber), and recuperator (see Figure 1.0). The gas residence time in each of these components is strongly dependent on the degree of turbulent mixing and the presence of recirculation zones. The time required for complete conversion of the PCB compounds is, in turn, depends on the residence time as well as the temperature distribution. The very complex nature of the particle laden flow within the above mentioned units requires the use of sophisticated computer simulations to obtain a detailed and accurate description of the flow field, temperature distribution, species concentrations as well as calculating the total heat loss and the overall pressure drop within each unit.

Although this study specifically targets the CMS^{TM} of Vortec Corporation, the end results could also be used, with minor modifications, in assessment of other type of incinerators for waste material. More importantly, this analysis can be extended to such cases where severely toxic and radioactive materials can be tested with computer simulations which would otherwise be impossible.

4.0 METHODOLOGY

The difficulties mentioned above can be resolved effectively using a readily available and extensively tested computational fluid dynamics (CFD) code. In this way, the PCB tracking can be done dynamically during the computation of the flow field where the local variations in the gas properties are automatically taken into account. Heat transfer and flow analysis, and PCB tracking will be performed using a commercial CFD code to numerically simulate the processes in the above mentioned units with the objective of providing supplemental information to improve the conceptual designs towards the most efficient system. Once such a code is reconfigured to simulate a certain unit, the key parameters can then be easily changed, and results can be obtained within a matter of days to determine optimum operating conditions.

The present computations are performed using the commercial code FLUENT (FUG-V4.4, 1996) This is a general purpose, multidimensional CFD code suitable for simulating flows in complex geometries. However, application to turbulent reacting flows

is not trivial. To achieve properly converged solutions, and to avoid misinterpretation of large amounts of numerical data, extreme caution and expertise both in physics of fluids and in computational fluid dynamics is necessary. Moreover, the chemistry sub-model for PCB tracking is not available in any of these codes. Such a model shall be developed and attached to the main code as a subroutine. It may be necessary to use the well known chemical kinetics code, CHEMKIN (Kee et al., 1980) and/or the general purpose chemical equilibrium code, CET (Gordon et al., 1984) in an interactive manner with the hydrodynamic data that will be obtained from the CFD code.

5.0 WORK PERFORMED THIS PERIOD

In this quarterly period the recuperator that is under design consideration by Vortec Corp. is analyzed to determine the influence of radiation heat transfer under various flow conditions. In particular, various radiation models implemented in FLUENT code were investigated and the heat transfer rates resulting from different models are compared with each other. The Chemkin II chemical kinetic code has been studied and made operational on our computers. Some test cases are running. A simplified kinetic mechanism for the conversion of dichloromethane is being developed. In the next quarter this mechanism will be implemented via Chemkin and the results will be compared with equilibrium concentrations.

6.0 RESULTS AND DISCUSSION

6.1 Analysis of the Recuperator:

The analysis of the recuperator has continued by investigating the effects of radiation heat transfer using an axisymmetric flow case, see Figure 6.1a in Appendix C, with a parallel heat exchanger with two conducting and radiating walls. The inner wall is made of Inconel 800H, outer wall is made of T304 stainless steel. The outer wall was assumed to be completely insulated. The flue gas flow rate was kept constant at 1.0 kg/s. The preheat air stream flow rate was varied in the range of 0.2 to 1.0 kg/s. The 0.6 kg case was checked for accuracy in detail and sample results are presented for this case. The following runs were made: (i) A base case without any radiation, (ii) A clear gas with the P1 Model (Method of Spherical Harmonics), (iii) A Weighted Sum of Gray Gases (WSGG) model added to the P1 model, and (iv) a Generalized Soot Radiation model added to the P1 with WSGG model. Energy balances shown in Tables 6.1 to 6.4 show that all errors are mostly on the order of 100 Watts which is considered to be small (less than about 0.5 % of the total enthalpy change of the flue gas). Computed outlet temperatures are also given in Tables 6.1-6.4 and axial variations of temperature along the centerlines of Zone 1 (the flue gas stream), and Zone 3 (combustion air stream) are shown in Figures 6.1-6.3.

The P1 model (Method of Spherical Harmonics) was implemented with variable properties (viscosity, specific heat, etc.). The participating medium was assumed to be a clear gas (air). It is seen from Table 6.2 that this case gives the lowest of the outlet temperatures predicted for the air stream. This is to be expected, because the gas is not directly transferring energy to the walls of the recuperator via radiation. The heat transfer to the inner wall in the recuperator is due mainly to convection. The radiation part is much smaller because of the clear medium assumption. The only radiation effect in the flue gas region is due to radiation reflecting from the pipe wall.

A significant change in energy transfer modes is predicted when Weighted Sum of Gray Gases model was added to the P1 model. This is due to the flue gas going from a clear medium to a radiating medium. The Weighted Sum of gray Gases in Fluent 4.4 accounts for H_2O and CO_2 only. But, this is probably not a bad assumption for the Vortec recuperator. Equilibrium analysis shows that the major products of combustion that exist in the recuperator are N_2 , H_2O , CO_2 . The mole fractions of CO_2 and H_2O were respectively 0.124 and 0.157 according to our equilibrium analysis presented in the previous quarterly report. The rest of the gases was assumed to be N_2 , which is not a radiating medium. It is seen from Table 6.3 that there is an increase in outlet temperature of the air stream of about 350 K on the average.

As a final case run, a generalized soot model was added to the WSGG and the P1 model. This was done by adding a concentration of soot 0.00289 kg/m^3 in the flue gas stream. It was assumed that the particulate matter present in the flue gas could be modeled as soot. The concentration of particulate matter in the flue gas was specified

according to the information obtained from Vortec engineers. Because of the two flow regions in the recuperator model, care had to be taken not to include the soot in the combustion air stream. This was accomplished by adding a single step soot production / combustion model. The parameters were then set equal to very small numbers so that a constant concentration of soot was present only in the flue gas stream. Table 6.4 shows that, in this case, the outlet temperatures of air stream are predicted to be even higher. This is to be expected since soot is assumed to be a black body in the radiation model. It is interesting to note that in this case the total change in the enthalpy of the flue gas decreases in this final case compared to the previous case without the soot (or the particulate matter). The increase in the sensible enthalpy of the airstream (corresponding to an increase in the air temperature) then must be compensated via a change in the enthalpy of the particulate matter. As this was constant, these results need to be investigated further.

The sample set of temperature profiles given in Figures 6.1 to 6.3 show that without the radiation the change in temperature with axial distance is approximately linear. When the radiation effects are added, the profiles are of power law type.

The overall effect of radiation is summarized in Table 6.5. It is seen that the total heat transfer rate to the air stream increases between 30 to 200% with radiation present. In each zone the mode of heat transfer may differ between convection/conduction and radiation modes. However, the overall influence of the radiation heat transfer can be calculated by subtracting the total heat transfer with clear gas (Table 6.1) from the total heat transfer with radiation. The difference must be due to radiative heat transfer effects. The percent radiation heat transfer with respect to the total heat transfer for each case is computed in this manner and shown in Table 6.5. In each individual case the radiation heat transfer constitutes between 25 to 65 percent of the total heat transfer. Considering the uncertainties involved in the species concentrations at the inlet of the recuperator, we anticipate that the actual numbers should be somewhere between the clear gas and gray gas cases (Tables 6.2 and 6.3, respectively).

6.2 Chemical Kinetics

In this period, the initial work was performed on the chemical kinetic simulation of the residence chamber. Chemkin II (Kee et al., 1980) was studied and made operational on our work stations. It was determined that the Senkin part of Chemkin II would be suitable to simulate the residence chamber assuming that it operates at constant pressure. A kinetic scheme of the combustion of hydrogen was used to evaluate the accuracy of Senkin. A reduced chemical kinetic scheme for the combustion of dichloromethane, a major incineration waste species is currently under development. Chemical kinetic simulations will be performed with this reduced mechanism using Chemkin and the results will be compared with equilibrium chemistry predictions. The equilibrium chemistry calculations will also be extended to the recuperator section after selecting a base case including variable temperature at fixed pressure.

7.0 CONCLUSIONS (*for the current period of performance*)

Most of the research effort in this quarter was focused on the radiation heat transfer analysis of the recuperator which is in design stage. Calculations were performed with combustion air flow rate ranging from 0.2 to 1.0 kg/s, and the flue gas stream at 1.0 kg/sec. The inlet temperature of the flue gas was assumed to be 1636 K (2485 degree F). The combustion air stream was assumed to be at 300 K (80 degrees F). The outer wall of the recuperator was assume to be perfectly insulated. The heat transfer rates with and without radiation are compared. The two extreme cases, namely, assuming the flue gas as a clear gas, and as a gray gas with steam and carbon dioxide being the major components were studied. Preliminary calculations were also made including the particulate matter as soot. The results show that with radiation the heat transfer rate form the flue gas to the air stream increases by about 30% with a clear gas, and about 200% with the gray gas assumption. Radiative heat transfer accounts for 22% and 65% of the total heat transfer for the later two cases, respectively. The computations including particulate matter is not conclusive at this stage. However, we believe that the case with the gray gas assumption can be taken as the worst case for analysis of species concentrations.

8.0 WORK PLANNED FOR NEXT PERIOD

In the next quarterly period the particulate matter present in the flue gas will be included in the radiation heat transfer model. Then, chemical kinetic calculations will be performed for the residence chamber, and these will be compared with the previously reported equilibrium calculations. A reduced multi-step mechanism will also be considered for major PCB compounds. The work on recuperator will continue with equilibrium calculations focusing on possibility of condensation in case of certain species.

APPENDIX A

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APPENDIX B: TABLES

Table 6.1 Calculated Outlet Temperatures with No Radiation					
	Inlet				
	Flue Gas	Air			
	K	K			
	1,636	300			
	outlet				
Mass Flow Rate	Flue Gas	Air			
kg/s					
0.6	1,503	607			
Heat Balance for a air mass flow = .6 kg/s					
	Conductive & Convective	Sum Heat Transfer Rates	Absolute Value Sum Heat Transfer Rates	Change in Enthalpy	Sum
zone	W	W	W	W	W
zone1 (flue gas)	-2.81E+04	-2.81E+04	2.81E+04	-2.81E+04	8.00E+00
zone2 (inner wall)	-1.51E+02	-1.51E+02	1.51E+02	0	-1.51E+02
zone3 (air)	2.81E+04	2.81E+04	2.81E+04	2.83E+04	-1.45E+02
zone4 (outer wall)	1.12E+02	1.12E+02	1.12E+02	0	1.12E+02

Table 6.2 Calculated Outlet Temperatures with the P1 model with Varying Properties

Table 6.2 Calculated Outlet Temperatures with the P1 model with Varying Properties								
	Inlet							
	Flue Gas	Air						
	K	K						
	1,636	300						
	outlet							
Mass Flow Rate	Flue Gas	Air						
kg/s								
0.2	1,510	1,036						
0.4	1,478	801						
0.6	1,460	681						
0.8	1,451	607						
1	1,444	556						
Heat Balance for a air mass flow = .6 kg/s								
	Conductive & Convective	Radiative	Sum Heat Transfer Rates	Absolute Value Sum Heat Transfer Rates	% Conductive & Convective	% Radiative	Change in Enthalpy	Sum
zone	W	W	W	W			W	W
zone1 (flue gas)	-2.91E+04	-6.87E+03	-3.59E+04	3.59E+04	80.88	19.12	-3.62E+04	-2.96E+02
zone2 (inner wall)	-7.26E+03	7.05E+03	-2.03E+02	1.43E+04	50.71	49.29	0	-2.03E+02
zone3 (air)	3.57E+04	-1.71E+02	3.56E+04	3.59E+04	99.52	0.48	3.59E+04	6.28E+01
zone4 (outer wall)	1.39E+04	-1.41E+04	-1.53E+02	2.80E+04	49.73	50.27	0	-1.53E+02

Table 6.3 Calculated Outlet Temperatures with the WSGG added to the P1 model								
	inlet							
	Flue Gas	Air						
	K	K						
	1,636	300						
	outlet							
Mass Flow Rate	Flue Gas	Air						
kg/s								
0.2	1,418	1,403						
0.4	1,320	1,239						
0.6	1,212	1,109						
0.8	1,142	1,013						
1	1,091	921						
Heat Balance for a air mass flow = .6 kg/s								
	Conductive & Convective	Radiative	Sum Heat Transfer Rates	Absolute Value Sum Heat Transfer Rates	% Conductive & Convective	% Radiative	Change in Enthalpy	Sum
zone	W	W		W	W			W
zone1 (flue gas)	-3.68E+03	-7.83E+04	-8.20E+04	8.20E+04	4.49	95.51	-8.18E+04	1.86E+02
zone2 (inner wall)	3.83E+04	-3.87E+04	-4.49E+02	7.70E+04	49.71	50.29	0	-4.49E+02
zone3 (air)	8.25E+04	-8.68E+02	8.16E+04	8.33E+04	98.96	1.04	8.29E+04	4.61E+02
zone4 (outer wall)	4.05E+04	-4.05E+04	3.60E+01	8.10E+04	50.02	49.98	0	3.60E+01

Table 6.4 Preliminary Results from the Case Where Particulate Matter was Included as Soot								
	Inlet							
	Flue Gas	Air						
	K	K						
	1,636	300						
	outlet							
Mass Flow Rate	Flue Gas	Air						
kg/s								
0.2	1,433	1,429						
0.4	1,324	1,277						
0.6	1,228	1,139						
0.8	1,144	1,048						
1	1,091	964						
Heat Balance for a air mass flow = .6 kg/s								
	Conductive & Convective	Radiative	Sum Heat Transfer Rates	Absolute Value Sum Heat Transfer Rates	% Conductive & Convective	% Radiative	Change in Enthalpy	Sum
zone	W	W		W	W			W
zone1 (flue gas)	-2.15E+03	-7.90E+04	-8.12E+04	8.12E+04	2.65	97.35	-8.03E+04	8.92E+02
zone2 (inner wall)	4.00E+04	-4.05E+04	-4.84E+02	8.04E+04	49.70	50.30	0	-4.84E+02
zone3 (air)	8.39E+04	-3.24E+03	8.07E+04	8.72E+04	96.28	3.72	8.44E+04	2.75E+03
zone4 (outer wall)	4.18E+04	-4.18E+04	-1.00E+00	8.36E+04	50.00	50.00	0	-1.00E+00

Table 6.5 Summary of Total Heat Transfer			
	No Radiation	P1 w/ Varying Properties	P1 & WSGG
Change in Enthalpy from Flue Gas	-2.81E+04	-3.62E+04	-8.18E+04
Change in Enthalpy from Air	2.83E+04	3.59E+04	8.29E+04
Percentage of Radiative Heat Transfer	0.00	22.33	65.61
Percentage Enthalpy Change Due to Radiation (Flue Gas Only)	0.00	28.75	190.80

APPENDIX C: FIGURES

VORTEC'S CMS SYSTEM

Figure 1. Vortec CMS™ System Sketch

Figure 6 Computational Domain

Figure 6.1a Temperature Variation Along Axial Direction
Center of Flue Gas
P1 Model w/Varying Properties at .6 kg/s

Apr 03 1997
Fluent 4.44
Fluent Inc.

Figure 6.1b Temperature Variation Along Axial Direction
Center of Preheated Air
P1 Model w/Varying Properties at .6 kg/s

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Fluent Inc.

y
z x

Figure 6.2a Temperature Variation Along Axial Direction
Center of Flue Gas
WSGG added to the P1 Model

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Fluent 4.44
Fluent Inc.

Y
Z x

Figure 6.2b Temperature Variation Along Axial Direction
Center of Preheated Air
WSGG added to the P1 Model

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Fluent 4.44
Fluent Inc.

y
z x

Figure 6.3a Temperature Variation Along Axial Direction
Center of Flue Gas
Soot Model Added to WSGG and P1 Model

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Fluent Inc.

y
z x

Figure 6.3b Temperature Variation Along Axial Direction
Center of Preheated Air
Soot Model Added to WSGG and P1 Model

Apr. 03 1997
Fluent 4.44
Fluent Inc.

FOCUS AREA 3.0

Decontamination & Decommissioning

TASK 3.1

**PRODUCTION AND EVALUATION OF BIOSORBENTS AND
CLEANING SOLUTIONS FOR USE IN
DECONTAMINATION AND DECOMMISSIONING
FETC Task No. 3.1**

Quarterly Technical Progress Report
Reporting Period:
January 1, 1997 to March 31, 1997

Work Performed Under Contract
No. DE-FC21-92MC29467

For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
Morgantown, West Virginia

By
Institute of Gas Technology
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ABSTRACT

The objectives of this project are to prepare and to evaluate biosorbents and cleaning solutions that may be useful in the decontamination and decommissioning (D&D) of U.S. Department of Energy facilities. In this collaborative project, the Institute of Gas Technology will make available to researchers at West Virginia University (WVU) biosorbents and cleaning solutions that are useful for D&D activities, but require filtration/liquid processing in order to be used in a practical process. Filtration tests will be performed by WVU.

During the past quarter additional surfactant samples were obtained from Witco Chemical Co. and evaluated for their ability to desorb PAHs from contaminated soil. Several of these commercially available surfactants exhibited good ability to desorb PAHs and to show enhanced detergency in the presence of ethanol as a co-solvent. A meeting was held with scientists from Witco to review all of the data which we had obtained regarding the performance of various surfactant products and to make selections of specific surfactant products to be the focus of future work. Based on cost and technical performance two surfactant products were selected as preferred products for our applications in D & D and environmental remediation: Witco DXP-5344-135 and Witco 2722. The first product is intended for use in the presence of ethanol while the 2722 product is intended to stimulate the in situ biodegradation of organic contaminants. Samples of each of these products will be forwarded to WVU so that they can be included in ultrafiltration testing.

Additional samples of lignin products were obtained from Lignotech Co. so that they could be evaluated as biosorbents for the removal of metals and radionuclides from wastewater. An actual sample of wastewater was obtained from the West Valley Nuclear facility for use in biosorbent evaluation tests which will be performed during the next quarter.

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
1.0	Introduction	1
2.0	Purpose	2
3.0	Background	2
4.0	Methodology	2
5.0	Work Performed this Period	3
6.0	Results and Discussion	3
7.0	Conclusions	13
8.0	Work Planned for Next Period	13

1.0 Introduction

The decontamination and decommissioning (D&D) of the Department of Energy facilities can result in the production of large volumes of wastewater containing metals, radionuclides, organic contaminants, and particulate material. Treatment of these wastewater streams is highly problematic because of their complexity and toxicity. Treatments appropriate for the removal of particulate material, such as filtration, are generally ineffective for the removal of soluble contaminants in D&D wastes such as metals and radionuclides. Techniques commonly used for the removal of metals and radionuclides from wastewater include the use of ion exchange resins; however, particulate material and organic contaminants present in D&D waste are incompatible with the use of ion exchange columns because of plugging/fouling. Biosorbents are biologically derived substances that can be used to sorb metals/radionuclides in a manner analogous to ion exchange resins, and biosorbents may be inexpensive in comparison with ion exchange resins.

The approach that is being tested in this project is the use of biosorbents, in a powdered form, added directly to D&D wastewater for the removal of metals and radionuclides. The use of biosorbents as powders rather than as ion exchange columns avoids the plugging/fouling problems associated with ion exchange columns. Also the powdered biosorbent should provide better contact and therefore more rapid metal/radionuclide binding than with ion exchange columns or immobilized biosorbent columns. D&D wastewaters require filtration in order to remove particulates and the same filtration step can be used to remove biosorbent particles and metals/radionuclides bound to them. It is expected that this approach will simplify and decrease the cost of treatment of D&D wastewater.

A related issue in D&D activities is the use of effective cleaning solutions, and the ultimate fate of used/spent cleaning solutions. The Institute of Gas Technology (IGT) has developed novel surfactant formulations that are highly efficient in the removal of organic and inorganic contaminants from solids. Ordinarily surfactants are discarded in spent D&D solutions. However, these surfactants can inhibit the subsequent treatment of these solutions and if not completely destroyed can contribute to the mobility of contaminants and thereby pose an environmental risk. This project will examine the possibility of using filtration to remove

surfactants from D&D solutions, and possibly aid in the recovery/reuse of those surfactants, as well as aid in the treatment of D&D wastewater.

2.0 Purpose

The purpose of this project is to evaluate the use of biosorbents in conjunction with filtration to treat D&D wastewater. An additional purpose of the project is to evaluate the removal of surfactants from D&D solutions.

3.0 Background

IGT has experience in the production and use of biosorbents, and in the production and use of surfactants for removing contaminants from solids. The West Virginia University (WVU) has experience in filtration and possesses a bench-scale model of a novel centrifugal filtration device developed by SpinTek Corporation. IGT and WVU are collaborating on this project to combine their areas of expertise to develop improved processes for the treatment of D&D wastewater.

4.0 Methodology

Bacterial Strains and Culture Media

Escherichia coli (ATCC 23724) was grown in nutrient broth (Difco) while *Zoogloea ramigera* 115 (ATCC 25935) was grown in nutrient broth supplemented with 25 g glucose per liter. Cultures were incubated at 30°C and the production of exopolysaccharide by *Z. ramigera* was monitored spectrophotometrically, and by measuring the viscosity of the medium. *Z. ramigera* cultures were harvested when viscosities exceeding 600 centipoise were achieved.

Immobilization of Dried Biomass

Biomass (cells plus exopolysaccharides if present) was harvested by centrifugation at 10,000 X g for 10 minutes and dried under vacuum in a rotary evaporator at 65°C. The dried biomass was ground to a uniform fine powder using a mortar and pestle. Dried, powdered biomass was used as is for metal-binding experiments.

Analytical Procedures

Atomic Absorption Spectrophotometry (AAS) (Model: 403, Perkin-Elmer Co.) Inductively Coupled Plasma Emission Spectroscopy (ICPAES) (Model: Atom Scan 25, Thermo Jarrell Ash Co.) and ion exchange chromatography (Model DX-100, Dionex Corp.) were used for metal concentration analyses. Samples were diluted to 0.1-1.0 ppm to ensure accurate analysis. To remove the interference from suspended particles, samples were centrifuged (600 rpm, 5 min) and filtered by a 0.45- μ m filter before injection into the AAS, ion exchange chromatography, or ICPAES apparatus.

Metal Adsorption Studies

Batch studies were performed using 100 mg of biomass in a 250-ml Erlenmeyer shaker flask containing 100 ml of 10 to 100 ppm metal solution (adjusted to pH 6.0 or 4.0 using HCl) and shaken for 15 minutes to 24 hours at a speed of 160 rpm at room temperature. Biomass was removed by centrifugation, the supernatant was decanted, filtered, and metal ions were then analyzed by AAS, ion exchange chromatography, or ICPAES.

Surfactant Solutions

Surfactants were obtained from Witco Chemical Corp. or were prepared according to proprietary procedures.

5.0 Work Performed This Period

During this quarter biosorbent samples were obtained from Lignotech Co. and a wastewater sample was obtained from the West Valley Nuclear facility. This wastewater will be used to evaluate the performance of lignin products to act as biosorbents and remove inorganic contaminants. Additionally, surfactant samples were obtained from Witco Corp. and evaluated for use in environmental remediation. Several good surfactant products were identified

6.0 Results and Discussion

Witco Corporation is a major worldwide supplier of surfactants and their collaboration was sought to assist us in identifying commercially available surfactants which may be used in our environmental remediation applications and to assist us eventually in commercialization. Witco

provided IGT with several surfactant samples to be evaluated for their ability to desorb PAH from Vandalia 1 MGP soil.

Desorption experiments were performed by placing 10 g (wet weight) Vandalia 1 soil in 50 ml conical centrifuge tubes along with 40 ml of surfactant solution. The surfactants were present at 4% in solutions comprised of 40% ethanol and the remainder water. A few surfactant solutions were prepared in water without any ethanol and those solutions were designated with a (W). Solutions of 40% ethanol, 100% ethanol and water only were included as controls. All soil slurries were then sealed and tumbled at room temperature for 16 hrs, centrifuged at 10,000 x g for 10 min, the supernatant was removed by aspiration, then the PAHs remaining in the soil were analyzed. Experiments were performed last quarter to examine all of the Witco samples available at that time. The untreated control samples (triplicates) had an average of about 1300 ppm total PAHs. Water resulted in no detectable desorption of PAHs, and pure ethanol resulted in nearly quantitative removal of PAHs with only 137 ppm remaining. The various surfactant solutions either in 40% ethanol or in water (W) were all similarly successful in reducing the PAH concentration in this soil to levels ranging from 324 to 507 ppm.

These results suggested that some of the surfactants were performing better than others and that there may be a correlation between surfactant structure and PAH desorption. This quarter additional surfactant samples were obtained from Witco and new PAH desorption experiments were performed with 2 MGP soils: Vandalia 1 and Vandalia 2.

The Vandalia 1 and Vandalia 2 MGP soils were collected from two different locations at the same site. These samples differ radically as regards PAH contamination. The amount of PAHs present in Vandalia 1 and Vandalia 2 soils are shown in Figures 1 and 2 respectively. The total amount of PAHs in Vandalia 1 soil is about 820 ppm. (A previous subsample of this soil contained about 1300 ppm total PAHs.) And as Figure 1 shows, Vandalia 1 soil is nearly devoid of low molecular weight PAHs. This is in sharp contrast to Vandalia 2 soil which contains about 32,000 total PAHs of which the low molecular weight PAHs are the most abundant.

Based on the results obtained with previous PAH desorption experiments, Witco provided additional surfactants for evaluation: DXP-5344-205 (#205), DXP-5344-206 (#206), and DXP-5344-207 (#207). These samples, as well as some of the previously tested surfactant solutions,

were tested for PAH desorption using Vandalia 1 soil. Since Vandalia 1 soil was used in previous tests of Witco samples, these tests should facilitate a comparison of all of the commercial surfactants under similar conditions. The results obtained with Vandalia 1 soil in recent tests are summarized in Figure 3 which illustrates the percentage of total PAHs removed from this soil with various solutions. The new samples (#205, #206 and #207) appear to perform equal to or better than other ethanol surfactant solutions, and all of the ethanol surfactants solutions performed significantly better than 40% ethanol alone. The desorption efficiency of individual PAH compounds in Vandalia 1 soil by various ethanol surfactant solutions is shown in Figure 4. The greatest differences in performance are seen with the high molecular weight compounds such as benzo(g, h, i)perylene where the new surfactants #205, #206 and #207 exhibit somewhat better desorption than the other surfactants.

PAH desorption tests with Vandalia 2 soil are summarized in Figure 5 which illustrates the total PAHs desorbed by various solutions. The Vandalia 2 soil is predominately contaminated with two and three ring PAHs and has a total PAH concentration of 32,000 ppm which is about 30 times higher than Vandalia 1 soil. Nonetheless, from 70% to 90% of the total PAHs were desorbed from Vandalia 2 soil by most of the ethanol surfactant solutions. The results are also somewhat puzzling as they indicate that water can desorb 45% of total PAHs while a 40% ethanol solution only exhibited about 10% desorption of total PAHs. This is primarily due to the fact that 40% ethanol does not desorb low molecular weight PAHs well in comparison with water and Vandalia 2 soil predominately contains low molecular weight PAHs. The new commercial surfactants #205, #206 and #207 perform well but are not clearly better than other surfactant products. To examine desorption efficiency further, the percentage desorption of selected individual PAHs from Vandalia 2 soil by various ethanol surfactant solutions and controls are shown in Figure 6. Only a portion of the data is shown in Figure 6 so that the figure does not contain so much information that it becomes illegible, but it still illustrates the trends observed in the experiment. The 100% ethanol solution desorbed nearly 95% of total PAHs but does less well on higher molecular weight than lower molecular weight PAHs. The ethanol surfactant solutions uniformly perform better than 40% ethanol only and generally improve the desorption efficiency of higher molecular weight PAHs relative to pure ethanol but do somewhat less well than pure ethanol for the desorption of low molecular weight PAHs.

Taken altogether, the results of all of the PAH desorption experiments indicate that the newest samples supplied by Witco (#205, #206 and #207) perform well but are not clearly better than previously tested surfactants such as Witcamide-5085 (W85) Witco 2722, and DXP-5344-135. Because of variations in soil type, contaminant concentration, and the relative abundance of various contaminants in a mixture, there can be no single surfactant solution which yields optimum performance for all MGP soils. The available data suggest that Witcamide-5085, Witco 2722, DXP-5344-135 (#135) and DXP-5344-207 (#207) would all be appropriate choices for desorbing PAHs from MGP soils. The PAH desorption data was discussed with scientists from Witco who also contributed information about the cost and the relative toxicity of these surfactants. While W85 exhibits good PAH desorption performance it has relatively high toxicity in comparison with some other surfactants and therefore would not be the best choice for environmental remediation applications. On the other hand, 2722 is completely non-toxic and is used as a food grade additive in some applications. 2722 was recommended for use in the future research, but because this surfactant does not perform well in the presence of ethanol another product was also selected for future work: #135. Both #135 and #207 showed good ability for PAH desorption and are similarly non-toxic (although neither are food grade products), but #135 is less expensive. Consequently the surfactants 2722 and #135 will be used in future work and samples of these surfactants will be sent to WVU for filtration testing.

FIGURE 1. PAH CONCENTRATION IN VANDALIA 1 SOIL.

FIGURE 2. PAH CONCENTRATION IN VANDALIA 2 SOIL.

FIGURE 3. PAH DESORPTION FROM VANDALIA I SOIL: SUMMARY OF RESULTS WITH COMMERCIAL SURFACTANTS

FIGURE 4. PAH DESORPTION FROM VANDALIA 1 SOIL: EFFICIENCY OF DESORPTION OF VARIOUS COMPOUNDS

FIGURE 5. DESORPTION OF TOTAL PAHS FROM VANDALIA 2 SOIL: SUMMARY OF RESULTS WITH COMMERCIAL SURFACTANTS

FIGURE 6. PAH DESORPTION FROM VANDALIA 2 SOIL: EFFICIENCY OF DESORPTION OF SELECTED COMPOUNDS

Previous tests of biosorbents for the removal of metals/radionuclides from wastewater indicated that lignin byproducts of the paper industry could be useful and cost effective materials. To further explore this possibility several additional lignin products were obtained from Lignotech Co.: Norlig A, Norlig TSFL, Marasperse AG, and Borresol HA-1. These lignin products have somewhat different properties and are calcium-based, ammonium-based, sodium-based, and potassium-based respectively. They vary in molecular weight from 10,000 to over 100,000. These products will be evaluated for their ability to sequester metals/radionuclides as well as to bind/sorb organic contaminants. If they perform well they could be added to wastewater to bind both organic and inorganic contaminants, then these lignins can subsequently be removed by ultrafiltration. Testing of these biosorbents will occur next quarter, and industrial wastewater obtained from the West Valley nuclear facility will be included in those tests.

7.0 Conclusions

The project is proceeding as scheduled. Filtration tests using biosorbents and surfactant solutions should proceed as planned as should the evaluation of biosorbents to bind metals using an industrial wastewater sample obtained from the West Valley Nuclear facility.

8.0 Work Planned for Next Period

Metal binding experiments to test the ability of biosorbents to remove metals from wastewater are planned.

TASK 3.2

**USE OF SPINTEK CENTRIFUGAL MEMBRANE TECHNOLOGY AND
SORBENT/CLEANING SOLUTIONS IN THE DECONTAMINATION AND
DECOMMISSIONING OF DOE FACILITIES**

FETC Task No. 3.2

Quarterly Technical Progress Report
Reporting Period: 1/1/97-3/31/97

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ABSTRACT

The use of sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelating agents) to decontaminate surfaces such as pipes, walls, *etc.* represents a unique and cost-effective method for remediating DOE facilities. As envisioned, the sorbent or cleaning agents would be added to a liquid (most likely water) and the solution would be contacted with the contaminated surface. For example, a pipe that is contaminated with inorganic and organic contaminants can be contacted with a solution containing a surfactant (for organics) and a chelating agent (for inorganics). After removal of the surface-bound contamination by the cleaning agents, a solid sorbent can be added to the liquid to bind the cleaning agent-contaminant compound. A major problem associated with this approach is the efficient separation of the sorbent/cleaning agent from the liquid phase using conventional separation techniques.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance and increasing the volume of waste treated per area of membrane.

The objective of this Task 2.6B research is to investigate the feasibility of using centrifugal membrane technology to separate sorbents/cleaning agents used in the decontamination/decommissioning of DOE facilities. The result of this effort will be the development of an efficient cost-effective method for the decontamination/decommissioning of DOE facilities. An additional benefit of this research is the application of the experimental results to the treatment of other DOE wastes such as contaminated groundwater, mixed-waste process water, and existing waste residuals (*e.g.*, Hanford Tank Farm Waste). It is envisioned that the volume reduction factors for this technology will be high and a relatively low amount of residual requiring additional treatment will be produced.

Work on the project will be performed by West Virginia University's Department of Civil and Environmental Engineering (WVU-CEE) and the Institute of Gas Technology (IGT). The following tasks will be completed by WVU-CEE: 1) selection and procurement of membrane(s) and 2) bench-scale testing of centrifugal membrane process. The following tasks will be completed by IGT: 1) identification and formulation of synthetic wastes and 2) selection and evaluation of sorbents and cleaning solutions. WVU-CEE and IGT will submit separate quarterly reports. A joint proposal will be prepared to obtain funding for D&D research through existing DOE ROAs and PERDAs.

TABLE OF CONTENTS

Item	Page
Title Page	i
Disclaimer	ii
Abstract	iii
Table of Contents	iv
List of Tables	v
List of Figures/Illustrations	vi
Executive Summary	vii
Introduction	1
Purpose	2
Background	3
Methodology	6
Work Performed During This period	7
Results and Discussion	8
Conclusions	45
Work Planned for Next Quarter	46
Technical/Administrative Difficulties	47

LIST OF TABLES

Table #		Page
1.	Average Surfactant Concentration and Rejection For Surfactant Run 9	16
2.	Average Naphthalene and Rejection For Surfactant Run 9	17

LIST OF FIGURES/ILLUSTRATIONS

Figure #		Page
1.	Schematic of SpinTek Centrifugal Membrane Pressure Chamber	5
2.	Schematic of Experiment Apparatus	18
3.	Absorption Spectrum for Naphthalene	19
4.	Spectrophotometry Calibration for Naphthalene Analysis	20
5.	Concentration Versus Exposure Time for Solubilized Naphthalene	21
6.	Spectrophotometry Calibration for Surfactant Analysis	22
7.	Permeate and Concentrate Temperature Versus Operation Time for Surfactant Run 9	23
8.	Permeate Flux Versus Operation Time for Surfactant Run 9	24
9.	Permeate and Concentrate Naphthalene Concentration Versus Operation Time for Surfactant Run 9	25
10.	Permeate and Concentrate Turbidity Versus Operation Time for Surfactant Run 9	26
11.	GC Calibration for Naphthalene Analysis	27
12.	Extraction Recovery of Naphthalene Versus Salt Dosage	28
13.	Extraction Calibration for Naphthalene in 0.02% Surfactant Solution	29
14.	Extraction Calibration for Naphthalene in 0.5% Surfactant Solution	30
15.	COD Calibration for Surfactant Analysis	31
16.	Permeate Flux Versus Operation Time for Sorbent Run 6	32
17.	Permeate and Concentrate Conductivity Versus Operation Time for Sorbent Run 6	33
18.	Permeate and Concentrate pH Versus Operation Time for Sorbent Run 6	34
19.	Permeate and Concentrate Turbidity Versus Operation Time for Sorbent Run 6	35
20.	Permeate Sr Concentration Versus Operation Time for Sorbent Run 6	36
21.	Permeate and Filtrate Concentrate COD Versus Operation Time for Sorbent Run 6	37
22.	Permeate Flux Versus Operation Time for Sorbent Run 7	38
23.	Permeate and Concentrate Temperature Versus Operation Time for Sorbent Run 7	39
24.	Permeate and Concentrate Conductivity Versus Operation Time for Sorbent Run 7	40
25.	Permeate and Concentrate pH Versus Operation Time for Sorbent Run 7	41
26.	Permeate and Concentrate Turbidity Versus Operation Time for Sorbent Run 7	42
27.	Permeate Sr Concentration Versus Operation Time for Sorbent Run 7	43
28.	Permeate and Filtrate Concentrate COD Versus Operation Time for Sorbent Run 7	44

EXECUTIVE SUMMARY

One surfactant run and two sorbent runs were completed in this quarter to study the SpinTek centrifugal ultrafiltration system's effectiveness at separating organic contaminant-micelle complex and sorbent-metal complex. The purpose of these runs are to evaluate the feasibility of using the SpinTek technology and operational conditions for treatment of actual D&D wastewater.

In surfactant run 9, Witcamide 5085, a commercial nonionic surfactant manufactured by Witco Company was selected as a cleaning agent and naphthalene was selected as a hydrophobic contaminant. The surfactant solution was first treated by the SpinTek system to get a baseline operation. Then naphthalene was added to the feed solution and the effect of separation was studied by measuring naphthalene content in permeate and concentrate using the spectrophotometric method. Naphthalene removal of 87% to nearly 100% was achieved when naphthalene concentration in the feed solution ranged from 190 mg/L to 34 mg/L. Because of the promising results from this run, Witcamide 5085 and naphthalene were retained as the primary surfactant/ contaminant combination for further studies.

Spectrophotometry analysis, however, is not an accurate method in determining naphthalene and surfactant concentration. To obtain more reliable naphthalene and surfactant measurements, sampling and analytical procedures were developed. A gas chromatography (GC) method was developed for naphthalene analysis and chemical oxygen demand (COD) was used for the measurement of surfactant concentration.

In the two sorbent runs, Indulin AT, an industrial byproduct provided by IGT was used as a sorbent for the removal of dissolved Sr. In both runs, Sr removal rate of over 70% was achieved. COD of the permeate was also studied to investigate the effect of disassociation of sorbent on the permeate quality. Results showed that permeate COD remained stable throughout the runs.

1.0 INTRODUCTION

The use of sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelating agents) to decontaminate surfaces such as pipes, walls, *etc.* represents a unique and cost-effective method for remediating DOE facilities. As envisioned, the sorbent or cleaning agents would be added to a liquid (most likely water) and the solution would be contacted with the contaminated surface. For example, a pipe that is contaminated with inorganic and organic contaminants can be contacted with a solution containing a surfactant (for organics) and a chelating agent (for inorganics). After removal of the surface-bound contamination by the cleaning agents, a solid sorbent can be added to liquid to bind the cleaning agent-contaminant compound. A major problem associated with this approach is the efficient separation of the sorbent/cleaning agent from the liquid phase using conventional separation techniques.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance, increasing the volume of waste treated per area of membrane, and increasing the contaminant/solids concentration in the residual (*i.e.* increases volume reduction factor). SpinTek's centrifugal membrane technology represents an improvement over conventional ultrafiltration technology and increases the number of waste streams that can be treated using membrane treatment.

2.0 PURPOSE

The purpose of this research is to investigate the feasibility of using sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelating agents) to decontaminate surfaces (*e.g.*, pipes) and then to separate the sorbents/cleaning solutions from the aqueous phase using the SpinTek centrifugal membrane technology. The result of this work will be an effective decontamination/decommissioning process that can be used for inorganic (*e.g.* radioactive nuclides and heavy metals) and organic contaminants.

3.0 BACKGROUND

Background information contained in this report pertains only to membrane technology and surfactant. The Institute of Gas Technology (IGT) will provide, in a separate report, the required background information on the development/use of sorbents/cleaning solutions.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The fluid stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate ("clean" liquid) is forced through the membrane and is collected in the hollow shaft and then discharged. The concentrate (liquid containing the contaminants) exits at the edge of the membrane packs. A schematic of the SpinTek centrifugal membrane pressure chamber is presented in Figure 1.

With time, the membrane surface will become fouled resulting in a decrease in the permeate flow rate. To reduce fouling in conventional membrane systems, a large portion of the concentrate is recycled back to the membrane unit producing large liquid velocities near the membrane surface. The large velocities increase turbulence which reduces the thickness of the fouling layer. In centrifugal membrane systems, the rotation of the membrane disk induces the turbulence (*i.e.*, shear force near membrane surface) required to minimize the thickness of the fouling layer. In conventional membrane systems, maximum surface velocities of about 15 ft/s are possible while with centrifugal systems surface velocities of 60 ft/s are typical. As the concentrate thickens with treatment time, conventional systems are not able to maintain the high velocities because of the difficulty in pumping viscous material at large flow rates. Because the centrifugal membrane system does not rely on pumping to produce the required surface velocities, extremely concentrated wastes can be treated.

Membrane systems are typically operated in either the semi-batch mode or the batch mode. In the semi-batch mode, fresh waste is added to the system at a rate equal to the permeate production rate. In the batch-mode, which normally occurs at the end of semi-batch operation, no fresh waste is added to the system and the concentrate remaining from semi-batch operation is concentrated further ("batch down"). During batch-down, large concentration factors are achieved (permeate is still being produced reducing the concentrate volume).

All membrane technologies are volume reduction technologies because contaminants are not degraded or destroyed. Thus, an important measure of the efficiency of membrane technology is the concentration factor (CF). The hydraulic-based CF during semi-batch and batch-down operation are calculated using the following two equations:

Semi-Batch Operation

$$CF_{SB} = 1 + V_{perm}/V_{feed\ tank}$$

Batch-Down Operation

$$CF_{BD} = CF_{SB} \times [V_{feed\ tank}/(V_{feed\ tank} - V_{perm})]$$

where, CF_{SB} = hydraulic-based concentration factor during semi-batch operation.
 V_{perm} = volume of permeate produced, gal.
 $V_{feed\ tank}$ = volume of the feed tank, gal.
 CF_{BD} = hydraulic-based concentration factor during batch-down operation.

Concentration factors are expressed as 1X, 2X, *etc.*, and increase with treatment time. CFs in excess of 100X are obtainable with the membrane technology. Another measure of treatment efficiency is the permeate flux (volume of permeate/membrane area per time). The permeate flux for a given membrane is dependent on the waste's viscosity, solids content, and temperature. These waste characteristics can also change during treatment.

Membranes that are used for ultrafiltration are characterized by the molecular weight of a molecule that is not allowed to pass through the membrane. For example, the membrane used in this quarter's research had a molecular weight cut-off (MWCO) of 100,000. In theory, compounds having a molecular weight greater than the MWCO would be retained by the membrane and compounds with molecular weights less than the MWCO would pass through the membrane and be contained in the permeate. It should be noted that the MWCO designation is somewhat misleading because of molecule having a molecular weight less than the membrane's MWCO may still be retained by the membrane because of its unique geometry.

Surfactants, or surface active agents, are substances that have amphiphilic molecules which have both hydrophilic head groups and hydrophobic hydrocarbon chain. When dissolved in water, the amphiphilic surfactants have a tendency to collect at any interface where the hydrophobic groups can be partially or completely removed from the contact of water and the hydrophilic groups can remain wetted. The same dual tendencies of the molecules will cause the formation of particles of colloidal dimensions which are termed micelles when the surfactant concentration is in exceed of a certain value. In micelle formation, the molecules undergo self-association in solution to produce aggregates in which the hydrocarbon chains are put together so that the total contact area of the hydrophobic groups of the surfactant molecules with water is reduced. The typical aggregation number of micelles is in the range of 50-300. The concentration at which micelles form is termed the critical micelle concentration (CMC). The CMC of a surfactant is determined by its molecular structure and also affected by additives and temperature. When the surfactant concentration is beyond its CMC, the concentration of monomers - single molecules in surfactant solution may stay relatively constant at the CMC value for some types of surfactant and also may increase for other types of surfactant.

In surfactant solutions, the hydrocarbon core of micelles provide a hydrophobic pseudophase in a polar environment. When organic substances insoluble or sparingly soluble in water are mixed with surfactant solution, their hydrophobic molecules will be repelled by water molecules and they tend to be incorporated by the hydrocarbon core of micelles. As a result, apparent solubility of these substances are increased. This phenomenon is termed solubilization. Because the solubilized organic substances will reside within the micelles, they can be separated together with micelles from water when the solution is passed through a ultrafiltration membrane since most of surfactant micelles are large enough in size to be retained by ultrafiltration membranes.

Figure 1. Schematic of SpinTek Centrifugal Membrane Pressure Chamber

4.0 METHODOLOGY

The centrifugal membrane technology was tested for separating surfactant micelles in surfactant run and sorbents in sorbent run. Polyvinylidene fluoride membranes with MWCO of 5k were used in the experiments. For each experiment, the clean water flux (CWF) was determined for the virgin membrane and after the system was cleaned. The CWF served as a baseline indicator to determine if the membrane is 1) permanently fouled and 2) sufficiently cleaned. The following parameters were monitored during technology testing:

1. Permeate flux;
2. Feed and permeate temperatures;
3. System pressure; and
4. Turbidity, light absorbance and conductivity of the feed and permeate.
5. Contaminant (naphthalene or Sr) concentration in the permeate

Turbidity of all samples was measured using a *LaMOTTE* Model 2008 turbidimeter and the turbidimeter was calibrated daily during the experiment. All the turbidity measurement was presented in the nephelometric turbidity unit (NTU).

A *Milton Roy SPECTRONIC® GENESIS™ 5* Spectrophotometer was employed to estimate the concentrations of surfactants and contaminants by measuring the light absorbance of the substances at specific wave lengths. The wavelength for each substance was determined by doing a wavelength scanning and finding the wavelength which gave a maximum absorbance. After the wavelength was determined, a calibration curve was developed for each substance to correlate its concentration and absorbance.

The conductivity of samples was measured using a *TRACEABLE™* Conductivity meter and it was calibrated daily during the experiment. All the conductivity measurement was presented in the unit of micromhos/cm ($\mu\text{S}/\text{cm}$).

Naphthalene content was analyzed using the above mentioned spectrophotometer at a wavelength of 275 nm. Sr content was analyzed quantitatively using atomic adsorption (AA) spectrophotometry.

Permeate flux, contaminant(s) concentration in the permeate, concentration factor, operational ease, and the membrane's physical/chemical resistance and cleanability were used to judge the effectiveness of the process.

To establish analytical methods for naphthalene and surfactant, gas chromatography (GC) and COD analyses were conducted. An HP 5890 GC equipped with a 25 m x 0.2 mm x 0.33 μm film thickness HP 5 (crosslinked 5% Ph Me Silicone) column and a FID detector was used. The oven temperature was set at 150 °C and the helium carrier gas flow rate was set 1 mL/min. The close reflux, colorimetric method was used for COD analysis.

5.0 WORK PERFORMED DURING THIS PERIOD

During the period 1/1/97 to 3/31/97, the experimental studies were focused on the separation of surfactant micelle-contaminant complex and sorbent-metal complex. In Surfactant Run 9, Witcamide 5085, a nonionic surfactant was tested and naphthalene was selected as a model organic contaminant. To obtain more reliable sample analysis results, analytical methods were developed for measurement of naphthalene and surfactant concentration in samples collected from ultrafiltration runs. Removal of Indulin AT, an industrial byproduct provided by IGT, and strontium (Sr) was studied in two sorbent runs.

6.0 RESULTS AND DISCUSSION

In this chapter, results from one surfactant run and two sorbent runs are presented and discussed. The surfactant run was conducted using Witcamide 5085 surfactant. The contaminant in this run was naphthalene. After it was proved that this surfactant/contaminant combination could be successfully treated by the SpinTek system, analytical methods were developed for accurate measurement of naphthalene and surfactant concentration. The two sorbent runs were conducted using Indulin AT sorbent and strontium (Sr) as a contaminant.

For all the 3 runs, PV-5k membranes were employed. Recycle operation was conducted in the surfactant run and Sorbent Run 6. Both recycle and batchdown operations were conducted in Sorbent Run 7. A schematic of the SpinTek centrifugal membrane system is presented in Figure 2. (Figures are included at the end of the text portion of Section 6.0).

Surfactant Run 9: Witcamide 5085, PV-5k membrane

In Surfactant Run 6 through Run 8 (see report of the last quarter), several surfactants had been tested for separation of phenol from water but none yielded satisfactory removal. In this run, a more hydrophobic compound, naphthalene, was selected as the contaminant to study its separation by the surfactant enhanced ultrafiltration. Witcamide 5085 surfactant was selected to solubilize the contaminant.

Witcamide 5085 is a commercial amide-based surfactant manufactured by Witco Company. In Surfactant Run 7, the CMC of this surfactant was determined to be 0.02 wt. % and an overall rejection of 90.1% was obtained in the ultrafiltration test.

Prior to the run, a bench scale test was carried out to study the solubilization of naphthalene by Witcamide 5085 surfactant at room temperature. In the test, naphthalene was first dissolved into methylene chloride and then a known volume of the solution was mixed with surfactant solution to be dissolved. The solubility of naphthalene in pure water was reported to be 31 ppm¹. Up to 100 ppm naphthalene was dissolved in 0.5 wt. % Witcamide 5085 surfactant solution. After naphthalene was dissolved in surfactant solution, the naphthalene concentration was analyzed using a Milton Roy Genesys® 5 spectrophotometer. The absorbance of a sample was measured at different wavelengths, a peak in absorbance was found at 275 nm, as shown in Figure 3. Therefore, the wavelength of 275 nm was selected for naphthalene analysis. Standards containing 20 ppm, 30 ppm, 40 ppm, 50 ppm and 60 ppm were prepared and their absorbance against a blank of surfactant solution were measured. A calibration curve for measuring naphthalene in Witcamide 5085 surfactant solution was obtained and presented in Figure 4.

In ultrafiltration tests, the SpinTek system is typically operated at a temperature of 110 °F. To study the effect of evaporation loss of naphthalene at this temperature, a sample containing naphthalene dissolved in surfactant solution was heated to 110 °F and kept open to atmosphere under a hood for 5 hours. Samples were taken at each hour and absorbance of each sample was

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¹ The Soil Chemistry of Hazardous Materials by James Dragun, Hazardous Materials Control Institute, 1988

analyzed at the wavelength of 275 nm. The results of this test, as shown in Figure 5, indicate that little naphthalene evaporation loss occurred in five hours at temperature of 110°F.

A virgin PV-5k membrane pack was installed into the system and a clean water flux of 401 gal/ft²-day was determined. The system was then started with 5 gallons of surfactant solution with an approximate concentration of 0.3% under the following operational conditions: vessel pressure = 42 psi, rotational speed = 1750 rpm, return flow = 1-1.2 gal/min, feed temperature = 110±2 °F and operational mode = recycle. The purpose of running surfactant alone was to get a baseline performance of the SpinTek system before introducing naphthalene into the system. The absorbance of the permeate and concentrate was measured to determine their surfactant concentration. The wavelength of 260 nm which was selected in Surfactant Run 7 was used for the surfactant analysis. The calibration for surfactant analysis at this wavelength is presented in Figure 6. An surfactant rejection rate of 78% was calculated using the first 2-hour experimental data. The results are presented in Table 1. Samples of concentrate and permeate was collected to use later as blanks in naphthalene analysis.

After two hours of operation, 50 ppm naphthalene was added to the system with the operation conditions remaining the same. The system was kept running for three hours and then another naphthalene dosage was added to make the naphthalene content of 100 mg/L. Three hours later, the last dosage was added to the system to increase naphthalene concentration in the feed tank to 300 ppm. The system was allowed to run for another three hours and then shut down and cleaned. Five gallons of distilled water was used to clean the system. After cleaning, a CWF was determined to be 417 gal/ft²-day. This CWF was slightly higher than the CWF of the virgin membrane. During the run, permeate samples were taken every half hour and naphthalene concentration was measured using the spectrophotometric method at a wavelength of 275 nm. One concentrate sample was collected after each naphthalene dosage.

The permeate and feed temperature versus operational time is presented in Figure 7. The feed temperature was controlled well at 110±2 °F. The permeate temperature was typically one to two degrees higher than the feed. The permeate flux versus operational time is presented in Figure 8. The permeate flux fluctuated between 270 to 340 gal/ft²-day during the entire run and no obvious impact of naphthalene dosage on permeate flux was observed. The naphthalene concentration was calculated from the absorbance measurements and the results are presented in Figure 9. The naphthalene concentration in the feed solution was determined to be 34 ppm, 76 ppm and 190 ppm after each naphthalene dose. These concentrations are lower than the actual doses, indicating that only part of the naphthalene added was dissolved into the solution. This was confirmed by the fact that a second phase was observed at the bottom of the feed tank when the system was shut down and cleaned. The permeate naphthalene concentration was in the range of 0-26 ppm during the run and it increased at each naphthalene dosage but was fairly constant during the three hours between naphthalene dosages. The average concentration of naphthalene in the permeate at each dosage and the rejection rates are presented in Table 2. The results of turbidity measurements for the entire run are presented in Figure 10. Turbidity of the concentrate kept increasing after the first dose of naphthalene. A possible explanation for the increase of concentrate turbidity at higher naphthalene concentration is that the size of surfactant micelles would increase after naphthalene was dissolved into them. The permeate turbidity decreased

sharply during the first two hours of recycling, which was typical in all the ultrafiltration runs. Then it dropped at a slower rate in the following 3 hours and remained stable regardless of the naphthalene dosage in the remaining of the run. The result showed that naphthalene dosage had no effect on permeate turbidity.

Results from this run demonstrate that micelles of Witcamide 5085 surfactant are capable of solubilizing naphthalene and micelle-naphthalene complex was further retained by the PV-5k membrane. The spectrophotometric analyses, however, was not able to generate accurate results in measuring naphthalene and surfactant concentration because of the interference's between naphthalene and surfactant measurements. More reliable analytical methods for naphthalene and surfactant measurement need to be developed.

Development of Naphthalene and Surfactant Analytical Methods

The purpose of developing these methods is to provide more accurate measurements of naphthalene and surfactant concentrations in permeate and concentrate samples from ultrafiltration tests. To measure naphthalene content, a liquid-liquid extraction and gas chromatography (GC) method was developed. In this method, naphthalene was first extracted out from aqueous sample by methylene chloride and then the extract was injected into GC to determine naphthalene concentration. For Witcamide 5085 surfactant, because its composition is unknown, COD analysis was selected to determine its concentration. Because both surfactant and naphthalene will be present in the samples obtained from ultrafiltration experiment, the COD measured would be the total COD of naphthalene and surfactant. Theoretical oxygen demand (ThOD) of naphthalene in the sample can be estimated by using the GC results. Surfactant COD can be calculated by subtracting naphthalene ThOD from the total COD. Then, surfactant concentration can be determined using its COD calibration curve.

Naphthalene Analysis

A method for measurement of the naphthalene concentration in surfactant solutions was developed based on EPA Method 8100. The Method 8100 was modified so that more effective extraction of naphthalene from surfactant solutions can be achieved. The modified method also requires smaller sample volume and less amount of organic solvent. The method employed liquid-liquid extraction followed by direct injection of the extract into GC to determine naphthalene concentration in surfactant solution.

1. Gas Chromatography (GC) Analysis of Naphthalene

A Hewlett Packard (HP 5890) GC with a FID detector and an HP-5 (crosslinked 5% Ph Me Silicone) capillary column (0.2 mm ID and 25 m long) was employed for the analysis of naphthalene. Samples with naphthalene dissolved in methylene chloride were injected directly into the GC column. Sample volumes varied from 5 μ L to 10 μ L depending on naphthalene concentration in the solution. Naphthalene concentration was determined from the peak area. The detection limit of this GC method was found to be 2 mg/L. A calibration curve which correlated peak area and naphthalene concentration was developed, as shown in Figure 11. A

linear correlation between peak area and concentration was found in the naphthalene concentration range of 2 mg/L to 200 mg/L.

2. Naphthalene Extraction

Methylene chloride was used to extract naphthalene from surfactant solutions. Extraction was performed in 8 mL Teflon lined vials with 4 mL of naphthalene/surfactant sample and 4 mL of methylene chloride. The vials were shaken vigorously to allow well mixing of the two liquid phases. After shaking, the vials were put on a rack to let the two phases separate. Approximately 1.5 mL of the methylene chloride phase was withdrawn from each extraction vial and transferred to a 2 mL Teflon lined vial. In each 2 mL vial, approximately 10 mg of sodium sulfate was added to absorb remaining moisture in the sample. Samples from the 2 mL vials were then injected into the GC column for naphthalene analysis.

Initial results of this extraction study, however, showed that the method was not effective in naphthalene extraction because some of the methylene chloride was emulsified by surfactant micelles. As a result, low naphthalene recovery was achieved and recovery rate varied as a function of surfactant concentration. To solve this problem, sodium chloride (NaCl) was added to the extraction vials to break the emulsion. To determine the effectiveness of NaCl addition on naphthalene extraction, a series of extraction was conducted with different amount of NaCl added. The results of this study are shown in Figure 12. Naphthalene recovery rate increased as NaCl dose increased in a range of 20 mg to 200 mg. Since only slight increase in naphthalene recovery rate was observed when NaCl dose increased from 150 mg to 200 mg, it was determined that approximately 200 mg NaCl was required to break the emulsion.

To investigate the interference of surfactant in liquid-liquid extraction and to determine the recovery (the ratio of measured concentration versus actual concentration) of naphthalene by the extraction, extraction calibration at two surfactant concentrations were made. Extraction calibrations at the surfactant concentration of 0.02% and 0.5% are presented in Figure 13 and Figure 14. The surfactant concentration of 0.02% was selected because it is the CMC of the surfactant and the permeate surfactant concentration is expected to be close to this value. The concentration of 0.5% represents the surfactant concentration in the feed. The calibration for 0.02% surfactant was made in the range of 2 ppm to 30 ppm naphthalene and the average recovery was 82%. The calibration for 0.5% surfactant was made in the range of 5-300 ppm naphthalene and the average recovery was 87%. Results show that recovery for the two surfactant concentration is close, indicating surfactant will not significantly interfere with the analysis in the concentration range 0.02% to 0.5%.

Correlation between COD Measurements and Surfactant Concentrations

COD was employed for the measurement of surfactant concentration. Because the permeate quality is the major concern in ultrafiltration experiment, a calibration was made to measure surfactant concentration in the permeate. The range 0 to 0.1% wt. was selected because permeate surfactant concentration was expected to be in this range. The calibration curve is presented in Figure 15. A linear relationship between COD measurements and surfactant concentrations

indicates that it is feasible to determine the surfactant concentrations based on the COD values of the permeate samples.

Sorbent Run 6: Indulin AT, PV-5k Membrane

An industrial byproduct, Indulin AT, obtained from IGT was used as a sorbent for removal of dissolved metals. In this run, non radioactive strontium (^{88}Sr) was selected to simulate the removal of radioactive ^{90}Sr . The purpose of this run was to study the performance of SpinTek membrane system on separating Indulin AT sorbent from water and Sr binding by the sorbent. PV-5k membrane was selected because its pore size was small enough to retain sorbent particles. The following procedure was designed for this experiment:

1. Run the system with sorbent/water mixture for 24 hours at the temperature 85 °F;
2. Add Sr and run for 24 hours without changing operation conditions;
3. Raise temperature to 110°F and run for 24 hours; and
4. Reduce the waste volume to 12 L by batchdown operation.

Residual sorbent level in the permeate was determined by monitoring the turbidity of the permeate and COD measurements. Sr concentration was measured using a atomic adsorption (AA) spectrophotometer.

Prior to the run, a CWF of 368 gal/ft²-day was measured for the PV-5K polymeric membrane. Sr stock solution of 1 g/L was prepared by dissolving 211.6 g of $\text{Sr}(\text{NO}_3)_2$ in 1 liter of D.I. water. A 13.2 gallon (50 L) mixture of 0.1% (wt.) Indulin AT sorbent in distilled water was treated using PV-5K membrane under following conditions: vessel pressure = 42 psi, rotational speed = 1750 rpm, feed temperature = 85°F, and operational mode = recycle. The system was started at room temperature and the feed tank temperature was allowed to rise to 85°F and it was kept constant at this temperature using a heat exchanger. During the run, permeate flux, turbidity, pH and conductivity of both permeate and concentrate were measured frequently. COD samples of both permeate and concentrate were taken frequently. After 24 hours operation, Sr stock solution was added to obtain Sr concentration of 10 mg/L in the mixture. The same operational conditions were kept. Concentrate and permeate samples for AA were taken frequently for Sr measurements. After another 24 hours, the feed tank temperature was allowed to rise to 110°F and was kept constant at this value. After 20 hours of operation at temperature 110 °F, the system was shut down because the bypass pipeline of the system was broken and leakage occurred. Step 4 was not conducted as planned. The system was cleaned by running the cleaning solution (30 mL *Dawn* dishwasher detergent in 5 gallons of distilled water) through the system at 110°F, then the membrane was taken out and cleaned manually with a sponge soaked in a solution of *Dawn* dishwasher detergent in tap water. After cleaning, a CWF of 230 gal/ft²-day was measured.

The permeate flux versus operational time is presented in Figure 16. The permeate flux decreased rapidly in the first 4 hours of operation, changing from 340 gal/ft²-day to approximately 280 gal/ft²-day. The flux kept decreasing gradually during the 24-hour period. After Sr was added into the mixture, a rapid drop in permeate flux was observed in the first two hours followed by gradual decreasing of permeate flux thereafter. After temperature was raised to 110 °F, the permeate flux was increased first then decreased slowly. The permeate flux changed as a function of time,

temperature and metal concentration. At same temperature, the permeate flux decreased with time. The result shows that the Indulin AT sorbent cause continuous fouling on the membrane surface. The permeate flux increased as the temperature increased. The influence of temperature on permeate flux can be explained by the viscosity decrease at higher temperature. The addition of Sr salt caused rapid drop in permeate flux. Since the dosage of Sr was made in the form of $\text{Sr}(\text{NO}_3)_2$ which is an electrolyte, the ionic strength in the solution was increased. The greater ionic strength provided more ions to neutralize the charge of the electric double layer around the sorbent surface. As a result, more compact gel layer might have been formed, and permeate flux decreased.

The permeate and concentrate conductivity versus operational time are presented in Figure 17. The conductivity of the concentrate is always much higher than the conductivity of the permeate during the entire run. After Sr was added, the conductivity of both concentrate and permeate was increased. There is no significant change in conductivity as temperature increased. The permeate and concentrate pH values versus operational time are presented in Figure 18. The pH values of both permeate and concentrate decreased slightly with time. The permeate and concentrate turbidity versus operational time are presented in Figure 19. The permeate turbidity remained stable and concentrate turbidity increased slowly. The concentrate turbidity increasing may attribute to the continuous dissociation of sorbent as a result of high turbulence in the system.

The Sr concentration in permeate versus operational time is presented in Figure 20. The Sr concentration remained stable and was not affected by the temperature change. The average Sr concentration in permeate was 2.8 mg/L. Comparing with the original Sr concentration in the mixture, the result shows about 70% removal. COD results of permeate samples and concentrate filtrates are presented in Figure 21. The concentrate filtrate COD samples were collected by passing concentrate samples through 0.1 μm filter paper under vacuum. Both permeate and concentrate filtrate COD concentrations are stable and have approximately same average COD value of 105 mg/L. The result shows that the COD of permeate is only caused by dissolved organic which may come from disassociation of sorbent particles and presence of sorbent in the permeate is negligible.

Though this run was not completed as planned, results showed good removal of both sorbent and Sr. Also the adsorption of Sr by the sorbent seems to be unaffected by the change of temperature.

Sorbent Run 7: Indulin AT, PV-5k membrane

Because of the system failure happened in Run 6. This run was essentially a repeat of Run 6. Prior to the run, an 11.2 gallon (42.4 L) mixture of 1g/L sorbent in distilled water was prepared. The same PV-5k membranes from Sorbent Run 6 was used. The CWF for it was 230 gal/ft²-day.

The system was started at room temperature and the feed tank temperature was allowed to rise to 85°F and kept constant at this value using a heat exchanger. After 25.5 hours of operation, Sr stock solution was added to obtain Sr concentration of 10 mg/L in the mixture. AA samples of both permeate and concentrate were taken frequently for Sr measurement. After another 29.5 hours, the feed tank temperature was allowed to rise to 110°F and kept constant at this value.

Batch-down operation was performed after 50.5 hours. During the first batch-down, 21.2 liter of permeate was taken out of the system and concentration factor became 2. Then the system was operated in the recycle mode for 2 hours. During the second batch-down, 7.2 liter of permeate was taken out of the system and concentration factor became 3. The remaining volume was operated in the recycle mode for 2 hours. The system was then shut down and the same cleaning procedure was followed. A CWF of 134 gal/ft²-day was obtained.

The permeate flux versus operational time is presented in Figure 22. The permeate flux increased rapidly in the first 2 hours of operation, changing from 220 gal/ft²-day to approximately 260 gal/ft²-day. The flux decreased thereafter and kept decreasing gradually. After Sr was added into the mixture, the permeate flux dropped sharply. After temperature was raised to 110°F, the permeate flux was increased first then dropped slowly. During the batch-down mode, the permeate flux decreased continuously.

Feed and permeate temperatures versus operational time are presented in Figure 23. Feed temperature was well controlled at 85±2 °F and 110±2 °F. The permeate temperature was typically several degree higher than the feed temperature. The permeate and concentrate conductivity versus operational time is presented in Figure 24. The conductivity of the concentrate is always much higher than the conductivity of the permeate during the entire run. After Sr was added, The conductivity of both concentrate and permeate was increased. There is no significant change in conductivity as temperature increased. Both permeate and concentrate conductivity increased sharply during the batch-down mode. The permeate and concentrate pH values versus operational time are presented in Figure 25. The pH values of both permeate and concentrate decreased slightly before Sr was added, then increased slightly with time after Sr dosage. The permeate and concentrate turbidity versus operational time are presented in Figure 26. The permeate turbidity stayed stable and concentrate turbidity increased slowly with time. The concentrate turbidity increasing may attribute to the continuous dissociation of sorbent. During the batch-down operation, both permeate and concentrate turbidity were increased sharply in the first 2 hours and the permeate color became brownish yellow. Later both permeate and concentrate turbidity decreased slightly, although the permeate and concentrate color became darker.

The Sr concentration in permeate versus operational time is presented in Figure 27. The Sr concentration in the permeate decreased continuously from 3.0 mg/L to 1.2 mg/L and there was no sign that this decrease was caused by change of temperature. The average Sr concentration in permeate is approximately 2.0 mg/L. Comparing with the original Sr concentration in the mixture of 8.4 mg/L, the result shows about 75% removal. During the batch-down mode, the Sr concentration in the permeate increased sharply from 1.2 mg/L to approximately 3.2 mg/L. Assuming absorption equilibrium in the feed tank, the permeate Sr concentration can be regarded as the concentration of dissolved Sr. When the total metal concentration increase, the dissolved metal concentration at equilibrium will also increase. This may explain the increase of permeate Sr concentration in the batch down operation.

The permeate, concentrate, and concentrate filtrate COD concentrations versus operational time are presented in Figure 28. Both permeate and concentrate filtrate COD concentrations remained stable and had approximately the same average COD value of 110 mg/L. The concentrate COD

concentrations increased from 500 mg/L to 1200 mg/L during 50 hours operation. During the batch-down mode, The concentrate COD concentrations increased sharply from 1000 mg/L to 3000 mg/L, this can be explained by the increasing of sorbent concentration in the feed during batchdown operation.

Different from Run 6, metal concentration in this run kept decreasing during the recycling operation. But other results from recycling part of this run are similar to those from Run 6. Almost the same metal removal and the same permeate COD was obtained. In batchdown operation, metal removal of 62% was achieved while concentration factor increased to 3 and COD of permeate was not increased. The results show that the SpinTek system maintained excellent performance in batch down operation for treating this waste.

Table 1. Average Surfactant Concentration and Rejection for Surfactant Run 9

	Concentrate	Permeate	Rejection
Average Absorbance	1.209	0.099	-
Average Concentration (wt. %)	0.23	0.05	78.3%

Table 2. Average Naphthalene Concentration and Rejection for Surfactant Run 9

	Naphthalene Concentration (ppm)		Naphthalene Rejection (%)
	Permeate	Concentrate	
Dosage 1	0	34	100
Dosage 2	4	76	95
Dosage 3	26	190	86

Figure 2. Schematic of Experimental Apparatus.

Figure 3. Absorption Spectrum for Naphthalene

Figure 4. Spectrophotometry Calibration for Naphthalene Analysis

Figure 5. Concentration Versus Exposure Time for Solubilized Naphthalene

Figure 6. Spectrophotometry Calibration for Surfactant Analysis

Figure 7 Permeate and Concentrate Temperature Versus Operation Time for Surfactant Run 9

Figure 8. Permeate Flux Versus Operation Time for Surfactant Run 9

Figure 9. Permeate and Concentrate Naphthalene Concentration Versus Operation Time for Surfactant Run 9

Figure 10. Permeate and Concentrate Turbidity Versus Operation Time for Surfactant Run 9

Figure 11. GC Calibration for Naphthalene Analysis

Figure 12. Extraction Recovery of Naphthalene Versus Salt Dosage

Figure 13. Extraction Calibration for Naphthalene in 0.02% Surfactant Solution

Figure 14. Extraction Calibration for Naphthalene in 0.5% Surfactant Solution

Figure 15. COD Calibration for Surfactant Analysis

Figure 16. Permeate Flux Versus Operation Time for Sorbent Run 6

Figure 17. Permeate and Concentrate Conductivity Versus Operation Time for Sorbent Run 6

Figure 18. Permeate and Concentrate pH Versus Operational Time for Sorbent Run 6

Figure 19. Permeate and Concentrate Turbidity Versus Operational Time for Sorbent Run 6.

Figure 20. Permeate Sr Concentration Versus Operational Time for Sorbent Run 6.

Figure 21. Permeate and Filtrate Concentrate COD Versus Operation Time for Sorbent Run 6

Figure 22. Permeate Flux Versus Operation Time for Sorbent Run 7

Figure 23. Permeate and Concentrate Temperature Versus Operation Time for Sorbent Run 7

Figure 24. Permeate and Concentrate Conductivity Versus Operation Time for Sorbent Run 7

Figure 25. Permeate and Concentrate pH Versus Operation Time for Sorbent Run 7

Figure 26. Permeate and Concentrate Turbidity Versus Operation Time for Sorbent Run 7

Figure 27. Permeate Sr Concentration Versus Operation Time for Sorbent Run 7

Figure 28. Permeate and Concentrate COD Versus Operation Time for Sorbent Run 7

7.0 CONCLUSIONS

The SpinTek centrifugal ultrafiltration unit's experimental parameters (flux, temperature and turbidity) were studied for treating surfactant solutions with hydrophobic contaminant and sorbent suspension with heavy metal contaminant. Following conclusions were drawn according to the experiment results:

- Witcamide 5085 surfactant can significantly increase the solubility of naphthalene in water. In other words, micelles of this surfactant have a high capacity in solubilizing naphthalene.
- Micelles of Witcamide 5085 surfactant incorporated with naphthalene can be effectively retained by PV-5k membrane.
- In treating waste containing micelle-naphthalene complex, SpinTek systems yield a high permeate quality and flux.
- Analytical procedures were developed for the measurements of naphthalene and surfactant in micelle-naphthalene complex. The methods are easy to apply and provide satisfactory results.
- SpinTek system equipped with PV-5k membranes was able to separate Indulin AT effectively, yielding low turbidity and low COD permeate. Continuous decrease of permeate flux, however, indicates that the membrane was fouled gradually by the sorbent. The cleaning procedure was not able to restore the CWF.
- Sr removal of 70% to 75% was achieved with Indulin AT concentration of 1 g/L and initial Sr concentration of 10 mg/L.

8.0 WORK PLANNED FOR NEXT QUARTER

Witcamide 5085 surfactant/ naphthalene will be separated from the aqueous phase in the next quarter. The composition of the permeate will be quantitatively analyzed to determine the accurate separation efficiency. Other surfactant/ contaminant will also be treated. Selection of the surfactants and contaminant compounds will be based on their use at the proposed soil flushing demonstration at DOE's RMI site in Astabula, Ohio.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

There were no significant technical/administration difficulties.

FOCUS AREA 4.0

Cross Cutting Innovative Technologies

TASK 4.1

**USE OF CENTRIFUGAL MEMBRANE TECHNOLOGY WITH NOVEL
MEMBRANES
TO TREAT HAZARDOUS/RADIOACTIVE WASTES**

FETC Task No. 4.1

Quarterly Technical Progress Report
Reporting Period: 1/1/97 - 3/31/97

Work Performed Under Contract
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For:
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ABSTRACT

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs.

The centrifugal action continuously cleans the membrane surface decreasing the use of cleaning chemicals and maintenance and increasing the volume of waste treated per area of membrane. Because of the reactive nature of most DOE wastewaters, the use of conventional membrane materials may not be viable. Thus, specialty membranes that can separate hazardous contaminants from the permeate stream and are chemically and radiologically stable will also be tested.

The objective of this research is to investigate the feasibility of using centrifugal membrane technology combined with innovative membranes for the treatment of hazardous/radiological wastes. The following tasks will be completed during this research: 1) perform extensive literature review on membrane technology, 2) identify and acquire waste and membranes, 3) modify, acquire, and be trained in the use of SpinTek technology, and 4) test and evaluate SpinTek technology and specialty membranes. These experiments will provide baseline membrane performance data. The following variables will be monitored as a function of run time: 1) permeate flux, 2) permeate turbidity, 3) temperature and pressure, and 4) conductivity and pH of all liquids.

The result of this work will be a technology that can treat a variety of wastes such as contaminated groundwater, mixed-waste process water, and existing waste residual (*e.g.*, low-level waste). The process can be used for both inorganic (*e.g.* radionuclides and heavy metals) and organic contaminants. It is envisioned that the volume reduction factors for this technology will be high and a relatively a low amount of residual requiring additional treatment will be produced.

TABLE OF CONTENTS

Item	Page
Title Page	i
Disclaimer	ii
Abstract	iii
Table of Contents	iv
List of Tables	v
List of Figures/Illustrations	vi-vii
Executive Summary	viii
Introduction	1
Purpose	2
Background	3-7
Methodology	8
Work Performed During This Period	9
Results and Discussion	10-49
Conclusions	50
Work Planned for Next Quarter	51
Technical/Administrative Difficulties	52

LIST OF TABLES

Table #		Page
1	Average permeate back pressures at common rotational speeds.	6
2	Viscosity of water at 110 and 135 °F.	7
3	Characteristics of ceramic membrane.	15
4	CW 1 operational parameters.	16
5	Summary of turbidity, conductivity, and pH data for CW 1, CW 2, and CW 3.	17
6	Average virgin membrane permeate fluxes at 110 and 135 °F--Experiment CW 1.	18
7	Slopes of average permeate flux -v- corrected pressure regression lines through the origin at 110 and 135 °F--Experiment CW 1.	19
8	Summary of membrane resistance observed in Experiment CW 1.	20
9	Summary of operational conditions used in resistance model verification experiment.	21
10	Summary of experimentally observed average fluxes and predicted fluxes in resistance model verification experiment.	22
11	CW 2 operational parameters.	23
12	Average permeate fluxes at 110 and 135 °F--Experiment CW 2.	24
13	Summary of membrane resistance observed in Experiment CW 2.	25
14	CW 3 operational parameters.	26
15	Average permeate fluxes at 110 and 135 °F--Experiment CW 3.	27
16	Slopes of average permeate flux -v- corrected pressure regression lines through the origin at 110 and 135 °F--CW 3.	28

LIST OF FIGURES/ILLUSTRATIONS

Figure #		Page
1	Schematic of SpinTek Centrifugal Membrane Pressure Chamber.	29
2	Permeate and concentrate temperatures versus time--Experiment CW 1.	30
3	Permeate flux versus time--Experiment CW 1.	31
4	Permeate turbidity versus time--Experiment CW 1.	32
5	Permeate and concentrate pHs versus time--Experiment CW 1.	33
6	Permeate and concentrate conductivities versus time--Experiment CW 1.	34
7	Average permeate flux versus corrected transmembrane pressure--Experiment CW 1.	35
8	Permeate flux prediction using average permeate flux as a function of corrected transmembrane pressure model--Experiment CW 1.	36
9	Permeate and concentrate temperatures versus time--Experiment CW 2.	37
10	Permeate flux versus time--Experiment CW 2.	38
11	Permeate turbidity versus time--Experiment CW 2.	39
12	Permeate and concentrate pHs versus time--Experiment CW 2.	40
13	Permeate and concentrate conductivities versus time--Experiment CW 2.	41
14	Average permeate flux versus corrected transmembrane pressure--Experiment CW 2.	42
15	Permeate and concentrate temperatures versus time--Experiment CW 3.	43
16	Permeate flux versus time--Experiment CW 3.	44
17	Permeate turbidity versus time--Experiment CW 3.	45
18	Permeate and concentrate pHs versus time--Experiment CW 3.	46
19	Permeate and concentrate conductivities versus time--Experiment CW 3.	47

Figure #		Page
20	Average permeate flux versus corrected transmembrane pressure-- Experiment CW 3.	48
21	Average permeate flux versus corrected transmembrane pressure--Cleaning after Experiment CW 3.	49

EXECUTIVE SUMMARY

In the first quarter, the effect of permeate back pressure arising from the centrifugal force on the performance of the SpinTek centrifugal ultrafiltration system was investigated. A total of three experiments were conducted using a virgin ceramic membrane (0.11um pore size). A clean distilled water feed was used in experiment CW 1 and CW 2. In experiment CW 3, a feed of distilled water contaminated with a 10 wt. machine oil was used. A mathematical correction for the average permeate back pressure as a function of membrane rotation, supplied by the SpinTek Corporation, was tested. These experiments provided baseline membrane performance data. These experiments were conducted over a pressure range of 25 to 75 psig, a temperature range of 110 and 135 °F, and three rotational speeds (250, 1250, and 1750 rpm). Permeate flux and turbidity, and permeate/concentrate pH and conductivity were measured periodically. Additionally, these data were used to calculate the intrinsic resistance of the virgin ceramic membrane. This parameter will be used in the future to model permeate flux as a function of transmembrane pressure. A standardized cleaning procedure consisting of a surfactant and NaOH solution adjusted to pH 11.8 followed by an H₂SO₄ solution adjusted to pH 2.2. The primary technical difficulty during the first quarter was the mechanical condition of the feed pump(s).

1.0 INTRODUCTION

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface, decreasing the use of cleaning chemicals and maintenance, increasing the volume of waste treated per area of membrane, and increasing the contaminant/solids concentration in the residual (*i.e.* increases volume reduction factor). SpinTek's centrifugal membrane technology represents an improvement over conventional ultrafiltration technology and increases the number of waste streams that can be treated using membrane treatment.

2.0 PURPOSE

The purpose of this research is to investigate the effects of permeate back pressure due to membrane rotation on the permeate flux using clean distilled water and oil contaminated feed solutions. A mathematical model used to correct for the average permeate back pressure was tested and the resistance of the virgin ceramic membrane was determined. This parameter will be used in the future to model permeate flux as a function of transmembrane pressure. Additionally, these experiments will provide baseline membrane performance data.

The result of this work will be a further step toward optimizing this technology for the treatment of a variety of wastes such as waste from DOE decontamination/decommissioning efforts, biological wastes, and waste with a high solids content (e.g., low level tank waste), contaminated groundwater, mixed-waste process water, and existing waste residual. The process can be used for both inorganic (e.g. radionuclides and heavy metals) and organic contaminants. It is envisioned that the volume reduction factors for this technology will be high and a relatively a low amount of residual requiring additional treatment will be produced.

3.0 BACKGROUND

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The fluid stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate ("clean" liquid) is forced through the membrane and is collected in the hollow shaft and is discharged. The concentrate (liquid containing the contaminants) exits at the edge of the membrane packs. A schematic of the SpinTek system is presented in Figure 1.

With time, the membrane surface will become fouled resulting in a decrease in the permeate flow rate. To reduce fouling in conventional membrane systems, a large portion of the concentrate is recycled back to the membrane unit producing large liquid velocities near the membrane surface. The large velocities increase turbulence which reduces the thickness of the fouling layer. In centrifugal membrane systems, the rotation of the membrane disk induces the turbulence required to minimize the thickness of the fouling layer. In conventional membrane systems, maximum surface velocities of about 15 ft/s are possible while with centrifugal systems surface velocities of 60 ft/s are typical. As the concentrate thickens with treatment time, conventional systems are not able to maintain the high velocities because of the difficulty in pumping viscous material at large flow rates. Because the centrifugal membrane system does not rely on pumping to produce the required surface-velocities, extremely concentrated wastes can be treated.

Membrane systems are typically operated in either the semi-batch mode or the batch mode. In the semi-batch mode, fresh waste is added to the system at a rate equal to the permeate production rate. In the batch-mode, which normally occurs at the end of semi-batch operation, no fresh waste is added to the system and the concentrate remaining from semi-batch operation is concentrated further ("batched down"). During batch-down, large concentration factors are achieved (permeate is still being produced reducing the concentrate volume).

All membrane technologies are volume reduction technologies because contaminants are not degraded or destroyed. Thus, an important measure of the efficiency of membrane technology is the concentration factor (CF). The CF during semi-batch and batch-down operation are calculated using the following two equations:

Semi-Batch Operation

$$CF_{SB} = 1 + V_{perm}/V_{feed} \quad (1)$$

Batch-Down Operation

$$CF_{BD} = CF_{SB} \times [V_{feed}/(V_{feed} - V_{perm})] \quad (2)$$

where,

CF_{SB} = concentration factor during semi-batch operation.

V_{perm} = volume of permeate produced, gal.

V_{feed} = volume of the feed tank, gal.

CF_{BD} = concentration factor during batch-down operation.

Concentration factors are expressed as 1X, 2X, *etc.*, and increase with treatment time. CFs in excess of 100X are obtainable with the membrane technology. Another measure of treatment efficiency is the permeate flux (volume of permeate/membrane area per time). The permeate flux for a given membrane is dependent on the waste's viscosity, solids content, and temperature. These waste characteristics can also change during treatment.

Membranes that are used for ultrafiltration are characterized by the molecular weight of a molecule that is not allowed to pass through the membrane. For example, the membrane used in this quarter's research had a molecular weight cut-off (MWCO) of 100,000. In theory, compounds having a molecular weight greater than the MWCO would be retained by the membrane and compounds with molecular weights less than the MWCO would pass through the membrane and be contained in the permeate. It should be noted that the MWCO designation is somewhat misleading because a molecule having a molecular weight less than the membrane's MWCO may still be retained by the membrane because its unique geometry.

A back pressure results from permeate pooling at the outer perimeter of the "disk pack" as centrifugal force is exerted on the permeate by the rotating membrane. A mathematical correction for the average permeate back pressure has been developed by the SpinTek Corporation. This expression is given in (3). In this equation, the permeate back pressure is a direct function of membrane rotational speed, feed solution density, and the radius of gyration which is explicitly stated in (4). The average transmembrane pressure is the difference between the gauge pressure and this average permeate back pressure as indicated in (5). A summary of average permeate back pressures at common operating rotational speeds is presented in Table 1.

$$P_{back} = [\rho(I^* \omega)^2]/2 \quad (3)$$

$$I = [(R_i^2 + R_o^2)/2]^{1/2} \quad (4)$$

$$P_{tmp} = P_{gauge} - P_{back} \quad (5)$$

where,

P_{back} = average permeate back pressure

ρ = permeate mass density = 1 kg/L

I = radius of gyration

ω = angular velocity

R_i = inner disk radius = 0.975 in.

R_o = outer disk radius = 3.625 in.

P_{tmp} = transmembrane pressure

P_{gauge} = gauge pressure

The membrane resistance is an intrinsic material property of the membrane must be determined using a virgin membrane with a clean water feed solution. Darcy's Law, given in (6) is used to calculate the resistance of the membrane for the case of a virgin membrane with a clean water feed. This form of Darcy's Law is only applicable in laminar flow conditions. To estimate the flow regime through the membrane pores in these experiments the Reynold's number (Re) was calculated for the following set of typical conditions: flow = 1000 mL/min, temperature = 110 °F ($\mu = 0.62$ cP), density = 1.0 kg/L, and membrane surface porosity = 0.3%. In this case, $Re = 3.990 \times 10^{-2}$. Darcy's Law can be applied because the flow through the pores is well within laminar flow conditions characterized by $Re < 1800$. The slope of a regression line through the origin on a plot of permeate flux -v- transmembrane pressure is inversely proportional to the product of the absolute feed viscosity and the resistance of the membrane, R_m , as shown in (7). The transmembrane pressure, P , is corrected for permeate back pressure as outlined in (3) and (5). Since the R_m is a material property of the membrane, the change in the magnitude of the slope of the line fitted to the flux versus transmembrane pressure data should be proportional to the magnitude of the change in $1/\mu$ (in the absence of membrane fouling). The viscosities of water at 110 and 135 °F and the theoretical changes in $1/\mu$ with temperature are presented in Table 2.

$$J = P_{tmp}/\mu R_m \quad (6)$$

$$R_m = 1/\mu * \text{slope} \quad (7)$$

where,

J = permeate flux

R_m = membrane resistance

μ = absolute feed viscosity

Table 1--Average permeate back pressures at common rotational speeds.

Rotational Speed (rpm)	P _{back} (psi)
1750	11.1
1450	7.6
1250	5.7
1150	4.8
850	2.6
750	2.0
250	0.3

Table 2-- Viscosity of water at 110 and 135 °F.

Temp. (°F)	μ (cP)	$1/\mu$ (cP ⁻¹)	% change in $1/\mu$ from 110 to 135 °F	% change in $1/\mu$ from 135 to 110 °F
110	0.6178	1.619	27	21
135	0.4884	2.048		

4.0 METHODOLOGY

Centrifugal membrane technology was studied using a virgin ceramic membrane (0.11um pore size) with clean distilled water and oil contaminated feed solutions. A mathematical correction for the average permeate back pressure as a function of membrane rotation was studied and tested. In each of these experiments, the SpinTek system was operated in recycle mode. These experiments were conducted over a pressure range of 25 to 75 psig, at three rotational speeds (250, 1250, and 1750 rpm), at 110 and 135 °F. Permeate flux and turbidity, and permeate/concentrate pH and conductivity were measured periodically. Additionally, these data were used to calculate the intrinsic resistance of the virgin ceramic membrane. In the future, this parameter can be used to model permeate flux as a function of transmembrane pressure with contaminated feed solutions (oil-in-water emulsions, surfactant, etc.) These experiments provided baseline membrane performance data. A standardized cleaning procedure was applied to the new ceramic membrane and clean water flux measurements were made after each experiment to monitor the membrane's condition.

The following variables were monitored during testing:

1. Gauge pressure.
2. Membrane rotational speed.
3. Concentrate return flow.
4. Permeate flux.
5. Permeate turbidity.
6. Concentrate and permeate temperature.
7. Conductivity and pH of all liquids.

Details on how each run was conducted are provided in Chapter 6--Results and Discussion.

5.0 WORK PERFORMED DURING THIS PERIOD

The following work was conducted during the period 1/1/97 to 3/31/97: 1) continuation of literature search, 2) testing of permeate back pressure correction model, 3) calculation of ceramic membrane resistance, and 4) implementation of a standardized cleaning procedure for the ceramic membrane.

5.1 Literature Search

A comprehensive literature search on membrane technology was conducted in the first quarter of this research and over 50 articles and reports have been added to the membrane technology literature database. As new relevant documents come to light the material will be requested through West Virginia University's Interlibrary Loan program.

5.2 Waste and Membrane Identification and Acquisition

The identification of wastes and membranes that warrant further study will be an ongoing task for this research project. In this quarter a virgin ceramic membrane with an average pore size of 0.11 μm was acquired. Clean distilled water was used as the feed solution. Numerous researchers have investigated the performance of virgin tubular ultrafiltration membranes. Thus, the virgin ceramic membrane used in the SpinTek system can be compared with membranes used in conventional systems. As demonstrated in previous reports, it was decided that the majority of further research focus on the more promising ceramic membrane because of the poor performance of the PV-100K polymeric membrane.

5.3 Technology Evaluation

In the first quarter, the SpinTek technology with a ceramic membrane (0.11 μm pore size) was used to process clean distilled water and distilled water contaminated with 10 wt. machine oil. Results and discussion are presented in Section 6.0.

6.0 RESULTS AND DISCUSSION

In this chapter results from the treatment of clean distilled water and oil contaminated distilled water using a ceramic membrane with an average pore size of $0.11\text{ }\mu\text{m}$ will be presented and discussed. A schematic of the SpinTek centrifugal membrane pressure chamber is provided in Figure 1. Figures and Tables are included at the end of the text portion of Section 6.0

RUN CW 1 -- DISTILLED WATER -- VIRGIN MEMBRANE

Forty liters of distilled water were used as the feed solution in this experiment. The distilled water was obtained from the West Virginia University Department of Civil and Environmental Engineering's house supply.

A virgin ceramic membrane was obtained from the SpinTek Corporation. Characteristics of the ceramic membrane are presented in Table 3. Prior to installation, the virgin ceramic membrane was wetted in deionized water for four hours.

After installing the ceramic membrane in the centrifugal UF unit, the system was operated in recycle mode at each of the gauge pressure/rotation conditions outlined in Table 4 until the flux stabilized. The system was run for 11 h at 1750 rpm, 65 psig, $110\text{ }^{\circ}\text{F}$, prior to beginning the pressure/rotation experiments to allow for initial membrane conditioning. The return flow rate was maintained at 1.0 ± 0.2 gpm throughout the experiment. A large decrease in permeate flux was observed over the first 14 h of operation. Other researchers have observed a similar initial flux decline with a distilled water feed [Fane, A. and C. Fell. A Review of Fouling and Fouling Control in Ultrafiltration, *Desal.*, (1987) 62, 117-136]. The initial flux decline may be due to pore plugging by residual materials from the membrane manufacturing process which were not fully rinsed out when the membrane was washed and wetted prior to beginning the experiment. Additionally, a portion of this initial loss of permeate flux may be due to bacteria and trace colloids present in the distilled water [Elmaleh, S. and W. Naceur. Transport of Water Through An Inorganic Composite Membrane. *Journ. Membr. Sci.*, (1992) 66, 227-234].

After conditioning the membrane, the system was run at $110\text{ }^{\circ}\text{F}$ and each pressure/rotation combination was examined. The feed temperature was then increased to $135\text{ }^{\circ}\text{F}$ and the pressure/rotation conditions were repeated. Both temperatures were re-examined, beginning at $135\text{ }^{\circ}\text{F}$ and ending at $110\text{ }^{\circ}\text{F}$ (Set #2). Permeate and concentrate temperatures versus time are presented in Figure 2. Permeate flux, turbidity, pH and conductivity as well as concentrate pH and conductivity were measured regularly. A summary of turbidity, pH, and conductivity data for experiments CW 1, CW 2, and CW 3 is presented in Table 5. Permeate flux versus time is presented in Figure 3. The permeate turbidity versus time is presented in Figure 4. The average turbidity observed in this run was 0.29 NTU. Turbidity ranged from 0.12 to 1.03 NTU. Permeate and concentrate pHs versus time are presented in Figure 5. Permeate pH ranged from 5.78 to 6.51 and averaged 6.10. Concentrate pH ranged from 5.58 to 6.45 and averaged 6.05. Permeate and concentrate conductivities versus time are presented in Figure 6. Permeate conductivity ranged from 1.88 to 7.87 $\mu\text{S/cm}$ and averaged 3.90 $\mu\text{S/cm}$. Concentrate conductivity ranged from 2.75 to 8.54 $\mu\text{S/cm}$ and averaged 6.14 $\mu\text{S/cm}$.

Permeate fluxes were averaged at each rotation/pressure condition and plotted against corrected pressure, as shown in Figure 7. A summary of the average permeate flux at each pressure/rotation and temperature in Run CW 1 is presented in Table 6. A regression line was fitted to these data through the origin and a slope was calculated. These slope data are summarized in Table 7. The slope of the average permeate flux -v- transmembrane pressure line decreased when the feed temperature was increased from 110 to 135 °F, contrary to expected behavior. Theoretically, the flux should increase as the temperature is increased due to the decrease in viscosity. It is hypothesized that the pressure/rotation experiments were begun while the system was still experiencing a flux decline due to membrane conditioning. When the system was then run at 135 °F and decreased to 110 °F, the observed change in the magnitude of the slope (20%) corresponded to the theoretically anticipated change in $1/\mu$ (21%) as indicated in Table 7.

The resistance of the virgin ceramic membrane was determined to be $2.7 \times 10^{12} \text{ m}^{-1}$ using the method outlined in Section 3. This value is higher than typical ceramic membrane resistance values reported in the literature, which range from $1.5 - 6.0 \times 10^{11} \text{ m}^{-1}$ (depending on the average pore size and permeability of the membrane) [Nazzari, F. and M. Weisner. Microfiltration of Oil-In-Water Emulsions. *Wat. Environ. Res.*, (1986) 68, (7), 1187-1191]. However, based on the fact that the properties of membranes can vary widely on an individual basis, an R_m value of 2.7×10^{12} is reasonable. A summary of the slopes of the regression lines through the average permeate flux -v- transmembrane pressure data and the resistance of the membrane at 110 and 135 °F is presented in Table 8. R_m is a material property of the membrane and should be the same at any two temperatures as observed in this experiment (differences due to temperature are accounted for in the absolute viscosity term in (7)).

The resistance model developed in experiment CW 1 was tested using three corrected pressures (51, 31, and 21 psi). Each of the three corrected pressures was maintained using three different combinations of membrane rotation and gauge pressure as outlined in Table 9. These verification experiments were conducted at 110 ± 2 °F. The system was run at each of the pressure/rotation conditions until the permeate flux was stable. The flux data was then averaged and plotted on the average permeate flux -v- corrected transmembrane pressure plot presented in Figure 8. In this figure, the regression line obtained in experiment CW 1 is also presented for comparison. A summary of experimentally observed average flux data and fluxes predicted using the regression fit are presented in Table 10. In each case, there was good agreement (< 10% difference) between the average experimentally determined flux and the flux predicted using the resistance model developed in experiment CW 1.

MEMBRANE CLEANING PROCEDURE

After completing the experiment, the rotor was stopped. The lines were drained and the vessel was opened. A picture of the uncleaned top and bottom membranes was taken and physical observations were made. The membrane was reinstalled and the system was flushed with 50 gallons of hot tap water (membrane was not spinning). Five gallons of distilled water was adjusted to pH 11.8 using NaOH (from a 1M stock NaOH solution stored with a soda scrubber.)

Additionally, 30 mL *Dawn* detergent was added to this solution. The volume of 1M NaOH added was recorded. The pH and conductivity of the cleaning solution was measured and recorded. This cleaning solution was heated to 135°F and the membrane was cleaned for 30 minutes at 1750 rpm and 40 psig. Permeate flux was measured and recorded during cleaning. The system was drained and flushed with hot tap water (membrane was not spinning). The pH of the effluent water was monitored. The volume of hot tap water needed to return pH from return flow to pH of initial hot tap was recorded (determined to be 50 gallons). An additional 5 gallons of distilled water was pH adjusted 2.2 using a stock 1M H₂(SO)₄ solution. The volume of acid added was recorded. The pH and conductivity of the cleaning solution was measured and recorded. The cleaning solution was heated to 135°F and the membrane was cleaned for 30 minutes at 1750 rpm and 40 psig. The permeate flux was measured and recorded during cleaning. The system was flushed with hot tap water (membrane was not spinning). The pH of the effluent water was monitored. The volume of hot tap water needed to return pH from return flow to pH of initial hot tap was recorded (determined to be 50 gallons). The membrane vessel was opened and a picture of the cleaned membrane was taken (top and bottom). A clean water flux (CWF) was measured at 110°F, 1750 rpm, 25, 50, and 75 psig using a distilled water feed. The average permeate flux versus the corrected pressure was plotted and the slope of the line was determined.

RUN CW 2 -- DISTILLED WATER -- CLEANED MEMBRANE

The membrane was cleaned according to the standardized cleaning procedure detailed in Section 3. The system was then filled with 40 L of distilled water and run at 110 °F at each pressure/rotation combination outlined in Table 11. Each pressure/rotation combination was then repeated at 135 °F. The return flow rate was maintained at 1.0 ± 0.2 gpm throughout the experiment. Permeate and concentrate temperatures versus time are presented in Figure 9. Permeate flux, turbidity, pH and conductivity as well as concentrate pH and conductivity were measured regularly. A summary of turbidity, pH, and conductivity data for experiments CW 1, CW 2, and CW 3 is presented in Table 5. Permeate flux versus time is presented in Figure 10. The permeate turbidity versus time is presented in Figure 11. The average turbidity observed in this run was 0.23 NTU. Turbidity ranged from 0.12 to 0.33 NTU. Permeate and concentrate pHs versus time are presented in Figure 12. Permeate pH ranged from 5.12 to 5.97 and averaged 5.66. Concentrate pH ranged from 4.00 to 5.72 and averaged 4.91. Permeate and concentrate conductivities versus time are presented in Figure 13. Permeate conductivity ranged from 5.20 to 8.66 uS/cm and averaged 6.79 uS/cm. Concentrate conductivity ranged from 9.28 to 11.31 uS/cm and averaged 10.03 uS/cm.

Permeate fluxes were averaged at each rotation/pressure condition and plotted against the transmembrane pressure, as shown in Figure 14. A summary of the average permeate flux at each pressure/rotation and temperature in Run CW 2 is presented in Table 12. A regression line was fitted to these data through the origin and a slope was calculated. These slope data are summarized in Table 13. An initial decrease in permeate flux was observed in this experiment. However, the flux decrease was not as pronounced as in experiment CW 1. This is possibly due to the fact that many of the residual materials from the membrane manufacturing process had been effectively removed from the system in the course of the cleaning process. The resistance calculated after cleaning is presented in Table 13. The slope of the average permeate flux -v-

transmembrane pressure regression line increased when the feed temperature was increased from 110 to 135 °F, as anticipated. However, the magnitude of the percent change in slope between 135 and 110°F was 4%. This is considerably lower than the anticipated 20% change. Although the permeate flux appeared to be steady when the experiment was begun, the system may have been experiencing an initial flux decline similar to that observed in experiment CW 1. This initial flux decline observed in experiment CW 2 may also be due to changes in the membrane's surface properties from the chemical cleaning process.

RUN CW 3 -- DISTILLED WATER CONTAMINATED WITH 10 WT. MACHINE OIL

In experiment CW 3, the membrane resistance after fouling was examined. The system was then filled with 40 L of distilled water. This feed solution was then contaminated with 429 mg oil/L water. The system was then operated in recycle mode at 110°F overnight (for 12 h) to establish baseline operational characteristics. (It was easier to maintain 110 °F feed temperature when the system is left unattended overnight.) The feed temperature was then increased to 135 °F, and the flux was allowed to stabilize. The system was then operated at each pressure/rotation combination outlined in Table 14. After completing the experiment at 135 °F, the feed temperature was decreased to 110 °F and each condition was examined. Each pressure/rotation condition was then repeated at both temperatures (Set #2). The return flow rate was maintained at 1.0 ± 0.2 gpm. Permeate flux, turbidity, pH and conductivity as well as concentrate pH and conductivity were measured regularly. A summary of turbidity, pH, and conductivity data for experiments CW 1, CW 2, and CW 3 is presented in Table 5. Permeate and concentrate temperatures versus time are presented in Figure 15. Permeate flux versus time is presented in Figure 16. The permeate turbidity versus time is presented in Figure 17. The average turbidity observed in this run was 0.24 NTU. Turbidity ranged from 0.13 to 1.40 NTU. Permeate and concentrate pHs versus time are presented in Figure 18. Permeate pH ranged from 6.30 to 7.60 and averaged 6.61. Concentrate pH ranged from 6.20 to 7.10 and averaged 6.58. Permeate and concentrate conductivities versus time are presented in Figure 19. Permeate conductivity ranged from 1.63 to 7.28 uS/cm and averaged 3.84 uS/cm. Concentrate conductivity ranged from 1.17 to 10.80 uS/cm and averaged 7.56 uS/cm.

Permeate fluxes were averaged at each rotation/pressure condition and plotted against the transmembrane pressure, as shown in Figure 20. A summary of the average permeate flux at each pressure/rotation and temperature in Run CW 3 is presented in Table 15. A regression line was fitted to these data through the origin and a slope was calculated. These slope data are summarized in Table 16. An initial decrease in permeate flux was observed in this experiment as in experiments CW 1 and CW 2. However, in the average permeate flux versus corrected pressure plot, the permeate flux consistently decreased throughout the run. Additionally, a steady pronounced decrease in the average flux was observed at each gauge pressure and temperature condition. As the membrane rotational speed was decreased, the flux declined. This flux decline at lower rotations is consistent with previously observed flux response as a gel layer forms on the membrane surface. As the rotation is decreased, the thickness of the gel layer increases, resulting in a lower permeate flux. This gel layer formation is likely the reason the change in slopes with temperature did not correspond to theoretically predicted changes in viscosity as detailed in Table 16 and demonstrated in experiment CW 1.

After completing this experiment, the membrane was cleaned as detailed earlier and a clean water flux (CWF) was measured at 25, 50, and 75 psig. A plot of average permeate flux versus corrected pressure for this CWF experiment is presented in Figure 21. The slope of the regression line through these data was 19.720. This value is higher than the 16.137 observed at 110 °F in experiment CW 2 (after the membrane was cleaned). This may be due to changes in the membrane's surface characteristics after reagent cleaning. Additionally, it is possible that the membrane's pores are gradually being "opened" in the course of these initial cleanings.

Table 3--Characteristics of ceramic membrane.

Lot Number	CRM-011M-TA
Chemical composition	TiO ₂ /Al ₂ O ₃
Mean pore size	0.11 μm
Thickness	240 μm
Inner radius	0.975 in.
Outer radius	3.625 in.

Table 4—CW 1 operational parameters.

Gauge Pressure (psig)	Rotation (rpm)
65	1750
45	1750
25	1750
65	1250
45	1250
25	1250
65	250
45	250
25	250

Table 5--Summary of turbidity, conductivity, and pH data for CW 1, CW 2, and CW 3.

Run	Avg. Perm. Turb. (NTU) [range]	Avg. Perm. Cond. (uS/cm) [range]	Avg. Conc. Cond. (uS/cm) [range]	Avg. Perm pH [range]	Avg. Conc. pH [range]
CW 1	0.29 [0.12 - 1.03]	3.90 [1.88 - 7.87]	6.14 [2.75 - 8.54]	6.10 [5.78 - 6.51]	6.05 [5.58 - 6.45]
CW 2	0.23 [0.12 - 0.33]	6.79 [5.20 - 8.66]	10.03 [9.28 - 11.31]	5.66 [5.12 - 5.97]	4.91 [4.00 - 5.72]
CW 3	0.27 [0.13 - 1.40]	3.84 [1.63 - 7.28]	7.56 [1.17 - 10.80]	6.61 [6.30 - 7.60]	6.58 [6.20 - 7.10]

Table 6—Average virgin membrane permeate fluxes at 110 and 135 °F--Experiment CW 1.

press. (psig)	rot. (rpm)	CW 1--110°F	CW 1--135°F	CW 1--110°F	CW 1--135°F
		Set #1 avg. flux [range] (gal./ft ² -day)	Set #1 avg. flux [range] (gal./ft ² -day)	Set #2 avg. flux [range] (gal./ft ² -day)	Set #2 avg. flux [range] (gal./ft ² -day)
65	1750	869 [847 - 890]	753 [747 - 761]	459 [459-459]	580 [574 - 581]
45	1750	538 [538 - 538]	476 [470 - 481]	294 [294 - 294]	[-]
25	1750	172 [169 - 172]	165 [165 - 165]	83 [83 - 83]	127 [122 - 129]
65	1250	910 [883 - 948]	805 [797 - 818]	503 [503 - 503]	638 [632 - 646]
45	1250	589 [589 - 589]	546 [546 - 546]	416 [416 - 416]	[-]
25	1250	291 [287 - 294]	269 [266 - 273]	140 [140 - 140]	218 [215 - 219]
65	250	889 [883 - 898]	864 [858 - 876]	553 [553 - 556]	706 [703 - 718]
45	250	612 [603 - 617]	571 [567 - 581]	395 [395 - 395]	503 [503 - 503]
25	250	322 [316 - 327]	314 [309 - 316]	230 [230 - 230]	291 [291 - 291]

Table 7--Slopes of average permeate flux -v- corrected pressure regression lines through the origin at 110 and 135 °F--Experiment CW 1.

Temp. (°F)	Slope	observed % change in slope from 135 to 110 °F	theoretical % change in 1/μ from 135 to 110 °F
110--Set #1	14.751	9.2	21
135--Set #1	13.511		
110--Set #2	8.736	-20	21
135--Set #2	10.913		

Table 8--Summary of membrane resistance observed in Experiment CW 1.

Temp. (°F)	μ (cP)	Slope	R_m (ft ² -d-psi/gal-cP)	R_m (1/m)
110	0.6178	8.736	0.19	2.7×10^{12}
135	0.4884	10.913	0.19	2.7×10^{12}

Table 9--Summary of operational conditions used in resistance model verification experiment.

$P_{transmembrane}$	P_{gauge}	P_{back}	Rotation (rpm)
51	61	10	1650
	55	4	1050
	53	2	750
31	41	10	1650
	35	4	1050
	33	2	750
21	31	10	1650
	25	4	1050
	23	2	750

Table 10--Summary of experimentally observed average fluxes and predicted fluxes in resistance model verification experiment.

$P_{corrected}$ (psi)	Avg. flux (gal/ft ² -d) [range]	Predicted flux (gal/ft ² -d)	% difference between predicted and average
51	456 [433-492]	446	2
31	290 [272-314]	271	7
21	199 [189-210]	184	8

Table 11--CW 2 operational parameters.

Gauge Pressure (psig)	Rotation (rpm)
65	1750
45	1750
25	1750
65	1250
45	1250
25	1250
65	250
45	250
25	250

Table 12--Average permeate fluxes at 110 and 135 °F--Experiment CW 2.

press. (psig)	rot. (rpm)	CW 2--135°F avg. flux [range] (gal./ft ² -day)	CW 2--110°F avg. flux [range] (gal./ft ² -day)
65	1750	1042 [976 - 1120]	911 [908 - 912]
45	1750	623 [617 - 635]	596 [596 - 596]
25	1750	244 [237 - 248]	226 [223 - 226]
65	1250	942 [918 - 962]	1000 [998 - 1005]
45	1250	633 [632 - 635]	675 [675 - 675]
25	1250	330 [330 - 330]	357 [352 - 360]
65	250	933 [926 - 948]	1057 [1055 - 1059]
45	250	660 [660 - 660]	740 [739 - 723]
25	250	373 [373 - 373]	442 [442 - 442]

Table 13--Summary of membrane resistance observed in Experiment CW 2.

Temp. (°F)	μ (cP)	Slope	R_m (ft ² -d-psi/gal-cP)	R_m (1/m)
110	0.6178	16.137	0.10	1.6×10^{12}
135	0.4884	16.840	0.12	1.8×10^{12}

Table 14--CW 3 operational parameters.

Pressure (psig)	Rotation (rpm)
75	1750
50	1750
25	1750
75	1250
50	1250
25	1250
75	250
50	250
25	250

Table 15—Average permeate fluxes at 110 and 135 °F--Experiment CW 3.

press. (psig)	rot. (rpm)	CW 3--135°F	CW 3--110°F	CW 3--135°F	CW 3--110°F
		Set #1 avg. flux [range] (gal./ft ² -day)	Set #1 avg. flux [range] (gal./ft ² -day)	Set #2 avg. flux [range] (gal./ft ² -day)	Set #2 avg. flux [range] (gal./ft ² -day)
75	1750	1179 [1163 - 1195]	810 [797 - 833]	782 [775 - 797]	371 [370 - 373]
50	1750	742 [729 - 750]	474 [474 - 474]	488 [488 - 488]	237 [237 - 237]
25	1750	310 [287 - 316]	158 [158 - 158]	187 [187 - 187]	93 [93 - 93]
75	1250	1109 [1091 - 1149]	771 [757 - 800]	763 [761 - 775]	438 [438 - 438]
50	1250	718 [718 - 718]	481 [481 - 485]	505 [503 - 510]	273 [273 - 273]
25	1250	357 [345 - 359]	215 [215 - 215]	244 [244 - 244]	133 [133 - 133]
75	250	913 [883 - 991]	613 [567 - 721]	538 [495 - 617]	424 [424 - 424]
50	250	598 [289 - 603]	364 [355 - 370]	352 [352 - 352]	287 [287 - 287]
25	250	321 [316 - 323]	187 [187 - 187]	208 [208 - 208]	165 [165 - 165]

Table 16--Slopes of average permeate flux -v- corrected pressure regression lines through the origin at 110 and 135 °F--CW 3.

Temp. (°F)	Slope	observed % change in slope from 135 to 110°F--Set #1	theoretical % change in 1/μ from 135 to 110°F
135--Set #1	15.282	-33	21
110--Set #1	10.179		
135--Set #2	9.928	-41	21
110--Set #2	5.905		

Figure 1-- Schematic of SpinTek Centrifugal Membrane Pressure Chamber.

Figure 2-- Permeate and concentrate temperatures versus time--Experiment CW 1.

Figure 3-- Permeate flux versus time--Experiment CW 1.

Figure 4--Permeate turbidity versus time--Experiment CW 1.

Figure 5--Permeate and concentrate pHs versus time--Experiment CW 1.

Figure 6--Permeate and concentrate conductivities versus time--Experiment CW 1.

Figure 7--Average permeate flux versus corrected transmembrane pressure--Experiment CW 1.

Figure 8--Permeate flux prediction using average permeate flux as a function of corrected transmembrane pressure model--Experiment CW 1.

Figure 9--Permeate and concentrate temperatures versus time--Experiment CW 2.

Figure 10--Permeate flux versus time--Experiment CW 2.

Figure 11--Permeate turbidity versus time--Experiment CW 2.

Figure 12--Permeate and concentrate pHs versus time--Experiment CW 2.

Figure 13--Permeate and concentrate conductivities versus time--Experiment CW 2.

Figure 14--Average permeate flux versus corrected transmembrane pressure--Experiment CW 2.

Figure 15--Permeate and concentrate temperatures versus time--Experiment CW 3.

Figure 16--Permeate flux versus time--Experiment CW 3.

Figure 17--Permeate turbidity versus time--Experiment CW 3.

Figure 18--Permeate and concentrate pHs versus time--Experiment CW 3.

Figure 19--Permeate and concentrate conductivities versus time--Experiment CW 3.

Figure 20--Average permeate flux versus corrected transmembrane pressure--Experiment CW 3.

Figure 21--Average permeate flux versus corrected transmembrane pressure--Cleaning after Experiment CW 3.

7.0 CONCLUSIONS

The effects of permeate back pressure due to membrane rotation on the permeate flux using clean water and oil contaminated feed solutions were examined. A mathematical model used to correct for the average permeate back pressure was tested. It was demonstrated that this correction is an adequate initial model to predict the transmembrane pressure as a function of membrane rotation. Additionally, the resistance of the virgin ceramic membrane was determined to be $2.7 \times 10^{12} \text{ m}^{-1}$. This value was slightly higher than typical ceramic resistances reported by other researchers. However, due to the fact that the properties of membranes can vary widely on an individual basis, an R_m value of 2.7×10^{12} was reasonable. This parameter will be used in the future to model permeate flux as a function of transmembrane pressure. These experiments provided baseline membrane performance data. When the feed water was contaminated with 10 wt. machine oil, a steady pronounced decrease in the average flux was observed at each gauge pressure and temperature condition. This flux decline with decreasing rotation was consistent with previously observed flux response as a gel layer forms on the membrane surface.

8.0 WORK PLANNED FOR NEXT QUARTER

The following work will be conducted during the second quarter: 1) testing of the effects of membrane rotation on permeate flux and water quality when treating an oily wastewater and 2) the use of membrane resistance determined after each experiment as an indicator of cleaning effectiveness.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

The primary technical difficulty was the performance of the system pumps. Two primary mechanical difficulties were encountered: 1) worn mechanical seals and 2) a broken pump impeller. The impeller was repaired by a local machine shop and the mechanical seals were replaced with original equipment parts obtained from the pump manufacturer.

TASK 4.2

ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON

Report for Period
ending March 31, 1996

Work Performed Under FETC Task 4.2
as Part of the WVU-DOE Cooperative Agreement.
Contract No.: DE-FC21-92MC29467

For :
US Department of Energy
Federal Energy Technology Center
Morgantown, West Virginia

By:
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March 1996

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ABSTRACT

The use of electrochemistry is an effective means to remove heavy metal contaminants from aqueous effluent streams. This project deals with the development and testing of novel carbon materials for the electrochemical removal of radionuclides and heavy metal ions from aqueous wastes. Both electroplating and electrosorption are employed for this task. This is accomplished by using a "packed-bed" of the carbon material in a "flow-through" electrochemical remediation cell.

The concentration of heavy metal ions and radionuclides in waste streams is usually very low: typically 1 to 1000 ppm. These low concentrations lead to relatively low current densities and low conversion of electroactive species at electrodes with a small surface area. Therefore, the electrolytic cell must be designed to increase the degree of conversion of the electroactive species (metal ion) as it passes through the remediation cell. This increase in conversion can be obtained by two means: (1) the use of a 3-dimensional electrode (increase surface area and residence time) and (2) turbulence (rotated electrode or flowing stream). The desire is to create a very high ratio of electrode surface area to electrolyte volume within the cell, coupled with turbulent mass transport within the electrode material. In this type of electrode, it is possible to control conversion efficiency using the length of electrode material through which the contaminated waste must pass.

This purpose of this work is to develop and test carbonaceous materials with an extremely large ratio of surface area to volume. This will be accomplished by using a packed-bed of conductive carbon, either as carbon fibers or carbon foams. Activation of these materials can lead to even greater surface areas. The increased surface area can help minimize the length of electrode through which the solution must pass. This decreased distance leads to reduced resistance to solution flow.

The removal of cadmium, lead, copper, nickel, strontium and uranium from aqueous samples using a small-scale model of the electrolytic cell has been demonstrated. Removal efficiencies above 90% have been observed for the reductive removal of Cd, Pb, Cu and Ni. Removal of U via electrolytic precipitation was 99% effective. The removal of Sr utilized electrosorption and a removal efficiency of 68.0% was obtained using a coating of MnO_2 .

The continuous removal of lead was demonstrated over a 72 hour period. The removal of lead over this period was continually greater than 90%. This corresponds to an effluent concentration of less than 10 ppm for an inlet solution feed of 100 ppm.

A scaled-up version of the prototype was constructed and tested to handle even greater flow rates of contaminated wastes. After several modifications, a removal efficiency of 99% was achieved for lead. A "real" sample obtained from the EPA's cleanup process at Fort Benjamin Harrison was treated. 95% removal of lead from this sample was achieved for a single pass through the electrolytic cell. The final objective is to transfer this technology for "on-site" use in industrial applications.

A parametric study of removal efficiency versus cell variables of current, pH and flow rate was completed. Flow rate had the most significant effect on the removal of lead. The method worked well over the pH range of 3.5 to 7.0. At a pH below 3.5, the oxidation of the plated lead by the acid in solution becomes significant, thus decreasing the efficiency of the electrolytic cell. The scaled-up remediation cell was put to use treating 32.4 liters of a 100 ppm lead solution over a period of 54 hours. Removal efficiencies remained at 98% or greater for the study.

TABLE of CONTENTS

A. Prefatory Material

Cover	i
Disclaimer	ii
Abstract	iii
Table of Contents	iv
List of Tables	v
List of Figures	vi
Executive Summary	vii

B. Body of Report

Introduction	1
Purpose	1
Background	1
Experimental Methodology	3
Carbon Samples	4
Work Performed	6
Analytical Methods	6
Anodic Stripping Voltammetry	6
Polarography	6
Atomic Absorption	7
Experimental	7
Reagents	7
Surface Area Measurements	7
Construction of Small-Scale Remediation Cell	8
Evaluation Of Carbonaceous Material	8
Construction of the Scaled-Up Remediation Cell	9
Work Performed This Quarter	10
Experimental	10
Results and Discussion	10
Industrial Application	12
Radionuclides	13
Prototype Remediation Cell	14
Conclusion	15

C. Appendix

References	34

LIST of TABLES

Table I	Standard Reduction Potentials of the Metals	28
Table II	Properties of Carbon Materials (Surface Areas)	29
Table III	Removal Efficiencies for Cadmium and Lead	30
Table IV	Immediate Industrial Application (Nickel Removal)	31
Table V	Doe Application (Strontium Removal)	32
Table VI	Project Completion	33

LIST of FIGURES

Figure 1	Schematic of Electrochemical Remediation Cell	18
Figure 2	Schematic of Scaled-Up 3-inch Remediation Cell	19
Figure 3	Schematic of 1.5-inch Galvanic Remediation Cell	20
Figure 4	Long-Term Removal of Lead using Electroplating	21
Figure 5	Long-Term Removal of Lead using Electrosorption	22
Figure 6	Long-Term Removal of Uranium	23
Figure 7	Effect of Plating Current on Removal Efficiency	24
Figure 8	Effect of pH on Removal Efficiency	25
Figure 9	Effect of Flow Rate on Removal Efficiency	26
Figure 10	Long-Term Removal of Lead Using Scaled-Up Cell	27

EXECUTIVE SUMMARY

Novel types of carbon materials have been tested for their ability to remove radionuclides and heavy metal ions from aqueous samples via electroplating and electrosorption. Removal efficiencies above 90% have been obtained for the reductive removal of cadmium, lead and copper. Long-term removal of lead over using electroplating on carbon nanofibers, vapor grown carbon fibers and carbon foam decreased the concentration of a 100 ppm lead feed to less than 10 ppm. Even after 72 hours, the carbon nanofibers showed no sign of saturation. For the 72 hour period, a capacity of $1.025 \text{ g}_{\text{Pb}}/\text{g}_{\text{carbon fibers}}$ was realized. An even greater capacity is expected at saturation.

Removal via electrosorption using carbon nanofibers produced similar results, but with an early drop-off of efficiency and a much smaller capacity ($7.1 \times 10^4 \text{ g}_{\text{Pb}}/\text{g}_{\text{carbon fibers}}$ for a 57 hour period). This is due to the neutralization of the charge on the carbon surface caused by the electrosorption of the positively charged metal ions. It is envisioned that for aqueous streams, electrosorption will be applied to remove radionuclides and other metal ions that have reduction potentials more negative than that of water.

The recovery of metals was demonstrated using lead ions. The recovered effluent resulted in a 7-fold concentration of lead after reversal of the potential applied to the carbon material. This resulted in a decrease in the volume of lead waste by one-seventh.

The removal of nickel ions from solution directly onto the bare carbon materials was inefficient. By plating a small amount of copper onto the carbon surface prior to use, enhanced removal efficiencies were obtained. At pH 7.0, a removal efficiencies of 89% was achieved.

The electrosorption of strontium was also examined. A removal efficiency of 34.4% was achieved using "pelletized" carbon nanofibers. At pH 10.0 and using a coating of MnO_2 , removal efficiencies were improved to 68.0%.

The electrolytic removal of uranium was exceptional. For a inlet feed concentration of 100 ppm uranium, an effluent stream was obtained with uranium concentrations near or below 1 ppm. Long-term removal of uranium was accomplished using the smaller remediation cell. For a 42 hour period, excellent removal of uranium was achieved (99%). Drop-off in removal efficiency was observed beyond 42 hours due to clogging of the carbon material. Recovery and concentration of the uranium waste decreased the volume from 2 liters down to 95 mL and concentrated the uranium to 1543 ppm. This is equivalent to a capacity of $0.15 \text{ g}_{\text{uranium}}/\text{g}_{\text{carbon}}$.

A scaled-up version of the prototype was constructed and tested to handle even greater flow rates of contaminated wastes. After several modifications, a removal efficiency of 99% was achieved for lead. A "real" sample obtained from the EPA's cleanup process at Fort Benjamin Harrison was treated. 95% removal of lead was achieved for a single pass through the electrolytic cell.

A parametric study of removal efficiency versus cell variables of current, pH and flow rate was completed. Flow rate had the most significant effect on the removal of lead. The method worked well over the pH range of 3.5 to 7.0. At a pH below 3.5, the oxidation of the plated lead by the acid in solution becomes significant, thus decreasing the efficiency of the electrolytic cell. The scaled-up remediation cell was put to use treating 32.4 liters of a 100 ppm lead solution over a period of 54 hours. Removal efficiencies remained at 98% or greater for the study.

ELECTROCHEMICAL REMEDIATION OF RADIONUCLIDES AND HEAVY METAL IONS

INTRODUCTION

PURPOSE

The purpose of this research was to assess the feasibility of using novel carbonaceous devices to remove radionuclides and heavy metal contaminants from aqueous streams. This work addresses the fabrication, behavior and analytical utility of these novel carbonaceous materials. Electrochemical techniques were employed to meet this objective.

BACKGROUND

Currently, there exists a rather serious problem associated with several nuclear production and processing facilities in the US. Over the years, large quantities of low-level radioactive waste, some in aqueous solution, have been accumulating at these sites in storage. Moreover, at some sites, this material has already leaked out and contaminated the soil over wide surface areas. In order to attack this problem, the DOE has expressed its desire to investigate new remediation techniques by promoting extremely applied and focused research programs. The work centers on the development of new procedures to remove and concentrate heavy metals and radionuclides from these low-level wastes. Most of the waste is in the form of aqueous solution and the new technique must be capable of reducing the concentration of contaminants down to parts per million (ppm) and in some cases parts per billion (ppb). Since the quantity of these wastes is large, the remediation procedure must be efficient and capable of being scaled-up in a predictable manner.

Industrial contacts have voiced a desire to remove several heavy metals from various aqueous streams. Heavy metal contaminants discussed include nickel, cadmium, copper, lead and chromium. Based on the standard reduction potentials, all of these metals should be excellent candidates for the technique developed in this program.

A technique which can be used to remove metal ions from solution is the adsorption and/or electrochemical reduction of the metal ions. In this procedure, a conductive surface acts as an electrode. A negative potential is applied to the electrode surface. The metal ions in solution are transported to the electrode surface via convection. Upon reaching the electrode surface, the metal ions are

adsorbed. If the potential is sufficiently negative the ions will be reduced and plated onto the electrode surface. In either case the metal ions are removed from the aqueous waste. While such a procedure is effective at lowering the concentration of the metallic species on the small scale, application at the large scale has been hindered due to the relatively small surface area of the metallic electrodes. This work assesses the performance of novel carbonaceous materials with large surface areas to remove radionuclides and heavy metal ions and their ability to be scaled up to applicable size.

Electrochemical techniques pertain to measurements involving the flow of electrons during a reduction-oxidation (redox) reaction. These measurements involve current, potential, charge and/or time as the primary variables. Electrochemical studies typically incorporate the use of a three-electrode system. This system includes a working, reference and auxiliary electrode. The redox reaction occurs at the surface of the working electrode as a result of the applied potential. Therefore it is critical to choose a suitable material for the working electrode. To be effective, the material must meet certain requirements. The electrode material should exhibit good electrical conductivity, be chemically inert over the potential region of interest, show favorable electrochemical behavior with the analyte(s), have a low background current and have an easily reproducible surface (1). Carbon materials are well suited for use as working electrodes. Graphite (2,3), carbon paste (4), glassy carbon (5) and reticulated vitreous carbon (RVC) (6) are a few examples of carbonaceous materials that have been used as working electrodes. A detailed discussion of carbon electrodes has been compiled by Dryhurst and McAllister (7).

Carbonaceous materials have been used to produce flow-through porous electrodes. Electrode materials have included graphite chips (8) or granules (9), glassy carbon grains (10), crushed graphite (11), screens of graphite (12) or carbon (13) and RVC (14). These porous flow-through electrodes have been used to remove metal ions from aqueous solutions. This is of major importance to industrial processes and environmental applications. The large surface area of porous flow-through electrodes makes them excellent candidates for the removal of metal ions from solution. Removal efficiencies approaching 100% can be achieved (14).

Discussions with METC personnel have ascertained the identity of several transuranics and radionuclides of concern. These metals include uranium, plutonium, cesium and strontium. Due to their large negative standard reduction potentials and the instability of the solid metals in water, electrosorption will be assessed for their removal. Due to the hazards of working with radioactive species, non-radioactive isotopes or surrogates will be employed. Strontium and uranium can readily be measured using polarography.

Once the metals have been removed from solution and accumulated onto the electrode surface, they can be recovered by reversing the applied potential. To recover selectively the metals, careful control of the applied potential is crucial. By applying a potential sufficiently negative to electroplate only one metal, it can be removed from the stream. Subsequent remediation cells with increasingly more negative potentials can be used in series to remove other metals. Each metal can thus be removed and recovered selectively.

The present work addressed the use of various carbon materials, including carbon fibers and porous carbon foams, to produce porous flow-through electrodes for the removal of metal ions from aqueous media. The resulting effluent was monitored downstream to assess the decrease in analyte concentration and hence the electrode efficiency.

EXPERIMENTAL METHODOLOGY

A schematic diagram of the small-scale apparatus used to carry out these experiments is shown in Figure 1. It was designed in the WVU lab and was produced by the WVU machinist. The carbonaceous material to be tested is placed between a platinum mesh screen and a filter. The platinum mesh is used to establish electrical contact to the carbon material. The purpose of the filter is two-fold. The filter prevents the carbon from flowing out of the cell and acts to distribute the solution evenly throughout the carbon bed. A predetermined potential is applied to the carbon material using a potentiostat. The sample solution is pumped through the carbon material and the unwanted metal contaminants are removed.

For the cathodic removal of metal ions, a potential more negative than the standard reduction potential of the metal ions of interest is applied to the working electrode (see Table I). This applied potential forces the plating of the metals onto the electrode surface thus removing the contaminant metal ions from solution.

Many metals can not be reduced at the electrode surface due to their large negative standard reduction potentials. The problem with these species is that their reduction potentials are more negative than that required to electrolyze water to hydrogen gas (H_2) and hydroxide ions (OH^-). Hence for aqueous solutions, water will be electrolyzed before these metals are electroplated onto the carbon surface. In these cases, electrosorption is utilized. Electrosorption incorporates a negative potential which is more positive than the potential to reduce the metal ion. The positive charge of the metal ion is attracted to the negative charge on the electrode surface. The metal ions are loosely bound to the electrode surface due to electrostatic forces and are removed from the aqueous stream. Due to the neutralization of charge on the electrode surface,

lower removal efficiencies are expected. It is anticipated that most radionuclides will be removed by electrosorption since they generally have large negative standard reduction potentials (see Table I, for examples of Sr^{+2} and U^{+2}).

Experimental parameters require optimization to maximize the removal of these metal ions. These parameters include pH, electrolyte concentration, solution flow rate, applied potential and carbon bed thickness. Conversion efficiency, long-term stability and loading capacity must be assessed as well. Finally, the pressure drop and flow rate characteristics of the solution flowing through the carbon bed need to be evaluated.

CARBON SAMPLES

Several carbonaceous electrode materials have been obtained from a variety of sources for comparison. To date, the following samples have been obtained.

- Reticulated Vitreous Carbon (RVC) Foam (Duocel®, 3% density) from Energy Research & Generation, Inc. (ERG, Oakland, CA). 10, 20, 30, 45, 80 and 100 pores per inch (ppi).
- Vapor Grown Carbon Fibers (VGCF) (Pyrograf III, Lot ADNH, 104-107) from Applied Sciences, Inc. (ASI, Cedarville, OH).
- Vapor Grown Carbon Fibers (Pyrograf III, Lot AN-201, "as grown") from Applied Sciences, Inc.
- Vapor Grown Carbon Fibers (Pyrograf III, Lot AN-201, "oxidized") from Applied Sciences, Inc.
- Vapor Grown Carbon Fibers (Pyrograf III, Lot P-2, "pelletized", regular "AN" fiber) from Applied Sciences, Inc.
- "Coal-Derived" Carbon Foam (23 NMP, Sample # 001) from WVU (Morgantown, WV).
- "Coal-Derived" Carbon Foam (23 NMP, Sample # 001, crushed foam, 420-707 μm) from WVU.
- Carbon Nanofibers (#1094-A) from PSU (University Park, PA).
- Carbon Nanofibers (#1094-B) from PSU.
- Carbon Nanofibers (NF46, unsupported) from PSU.

- Graphite Fiber Mats from PSU.
- Carbon Nanofibers grown on Graphite Fiber Mats (#0295) from PSU.
- Graphite Felt (#0695-A) from PSU.
- Carbon Nanofibers grown on Graphite Felt (NF46, F100, CN37 and CF37 from PSU.

RETICULATED VITREOUS CARBON (15)

Reticulated Vitreous Carbon (RVC) is an open-pore material with a honeycomb (foam) structure (15). RVC is a form of carbon that combines some of the properties of glass with normal industrial carbon. RVC has a large void volume (97%) and a small surface area (0.09 m²/gram). It has a self-supporting rigid structure, a low resistance to solution flow and is resistant to temperatures up to 600 °F in air (16). The samples obtained had various pore sizes. Sizes included 10, 20, 30, 45, 80 and 100 ppi (pores per inch). The 100 ppi RVC foam was tested in our study.

VAPOR GROWN CARBON FIBER (17)

The VGCF manufactured by Applied Sciences, Inc. (ASI) is produced by a combination of catalytic and chemical vapor deposition processes which allow for control of the fiber dimensions, such as filament length and diameter. Lengths are available from 1 mm to tens of centimeters and diameters can vary from under 0.2 μm to over 100 μm. The resulting fiber has a lamellar morphology and is highly graphitic. Cross section and overall views of these samples were provided in the first year's quarterly reports.

"COAL-DERIVED" CARBON FOAM

These particular samples of carbon foam are made from coal-derived pitches produced at WVU by a novel solvent extraction process. By careful control of experimental parameters, an ultra-low ash foam precursor (pitch) is produced which upon careful heat treatment results in a low-density carbon foam structure. This "green" foam is then calcined at temperatures around 1000 °C and a highly conductive material results. The foam has an open-cell structure, enabling it to be used as an efficient carbon electrode.

CARBON NANOFIBERS

The carbon nanofibers were produced by our first year collaborators at The Pennsylvania State University (PSU). The nanofiber material is a microscopic version of a carbon fiber. It is produced by the metal catalyzed decomposition of certain hydrocarbons (18-19). Various degrees of graphitization are possible.

GRAPHITE FIBER MATS and GRAPHITE FELTS

Obtained commercially and supplied to WVU by Dr. Baker at The Pennsylvania State University.

All materials were treated with a dilute solution (10%) of nitric acid prior to use as the electrode material. If necessary to help activate and clean the surface, mild electrochemical or chemical methods were employed. A potential of +1.0 V was applied to the carbon surface for 10 minutes to remove contaminants and produce oxidized sites on the carbon surface. This was followed by applying a potential of -1.0 V for 10 minutes to condition the surface prior to use.

WORK PERFORMED

ANALYTICAL METHODS

Anodic Stripping Voltammetry:

The thin-layer flow cell was purchased from BAS (Model LC-44-01000). The working electrode was glassy carbon disk (0.3 cm diameter) over which the solution flows. Two 50 μm spacers were used to produce the flow channel and direct the sample solution across the carbon disk. Prior to use, the glassy carbon disk was polished using successive slurries of 1.0, 0.3 and 0.05 μm alumina until a mirror-like surface was obtained. The glassy carbon disk was then rinsed thoroughly with deionized water and assembled. All potentials were measured relative to the Ag/AgCl reference electrode (BAS, Model RE-4). Stripping voltammograms at the thin-layer flow cell were obtained using the BAS Model CV-27 Voltammograph in conjunction with a Hewlett Packard Model 7044B X-Y Recorder.

Polarography:

Polarographic data was obtained using a Static Mercury Drop Electrode (SMDE) (Model 303A, EG&G Instruments, Princeton Applied Research, Princeton, NJ) in conjunction with a potentiostat (Model 263A, EG&G) and

interface (Model 507, EG&G). This system has been received and has been assembled. Upon assembly and calibration, this instrument was utilized to measure the uranium ion concentrations in the effluent stream. (The 100 ppm solution being tested is below the detectable limits of Atomic Absorption. Polarography can be used to measure uranium concentrations down to 20 ppb). The SMDE can be used to measure many other metals in the ppb to ppm range.

Atomic Absorption:

Spectroscopic data were obtained using an Atomic Absorption Spectrophotometer (Model 2380, Perkin-Elmer, Norwalk, CT). Procedure and experimental conditions were outlined in the Perkin-Elmer "Analytical Methods of Atomic Absorption Spectrophotometry" Cookbook (20). All standards and samples were matrix-matched prior to analysis.

EXPERIMENTAL

Reagents:

Stock solutions of cadmium, lead, copper, uranium, strontium and nickel were prepared using atomic absorption standard solutions (Fisher Scientific, Fair Lawn, NJ) and deionized water. The deionized water used was further purified using a NANOpure™ ultrapure water purification system (Barnstead-Thermolyne, Dubuque, IA). These solutions were stored in polyethylene containers to avoid contamination. The 1.0×10^{-4} M mercury ion solution used for anodic stripping voltammetry was prepared from $\text{Hg}(\text{NO}_3)_2$ as needed. The mercury ion and sample solutions were prepared in 0.1 M KNO_3 (potassium nitrate) supporting electrolyte. The pH was adjusted using nitric acid. (All chemicals were certified ACS grade purchased from Fisher Scientific unless otherwise noted).

Surface Area Measurements:

The surface area of the samples has been determined and the data are shown in Table II. The surface areas were obtained at WVU using a Gemini III 2375 Surface Area Analyzer (Micrometrics Instrument Corporation, Norcross, GA). Standard operating procedures have been followed as outlined in the operator's manual.

A pre-weighed sample of the carbonaceous material was placed into a clean, dry sample tube. The sample was thoroughly de-gassed using nitrogen. The dewar was filled with liquid nitrogen and the system prepared for analysis.

The sample tube was connected to the analysis port and the balance tube was connected to the balance port. Care was taken to minimize exposure of the sample to air. The doors to the sample station were closed and the analysis initiated. Saturation pressure was set to 740.53 mm Hg and the evacuation rate to 599 mm Hg/min. The Gemini III 2375 performs automated analysis. It is capable of performing simultaneously BET multipoint and single point surface area, Langmuir surface area, t-method micropore and BJH adsorption/desorption distribution. The resulting data were recorded on an Epson plotter (Model P84FA).

Construction of the Small-Scale Remediation Cell:

The small-scale remediation cell (see Figure 1) was produced using a Lexan™ rod (5.0 cm diameter and 6.0 cm long). The flow channel (1.6 cm i.d.) was drilled through the Lexan™ body. A threaded Lexan™ plug was used to press the carbonaceous material between a platinum mesh and a piece of filter paper. The pressure on the carbon material can be easily adjusted by turning the threaded plug. The Ag/AgCl reference electrode (3M NaCl, Model RE-4, BioAnalytical Systems, Inc. (BAS) West Lafayette, IN) was introduced through a Lexan™ sleeve positioned 90 degrees to the flow channel. Leakage was prevented using an o-ring and compression fitting. A piece of coiled platinum wire inserted at the outlet of the cell was used as the auxiliary electrode. Contact to the working electrode was established via a platinum mesh.

Evaluation of Carbonaceous Materials:

The carbonaceous material to be studied was pretreated to clean, oxidize and assist in the wetting of the carbon surface. This was accomplished by placing the carbon material in a dilute solution (10%) of nitric acid overnight in a covered beaker. The material was then thoroughly washed using deionized water. The carbonaceous material was weighed and then positioned into the metal remediation cell using a clean glass rod.

The threaded plug is used to press the carbonaceous material between a platinum mesh and a piece of filter paper. The pressure on the carbon bed can be easily adjusted by turning the threaded plug. Rigid samples, such as RVC, were positioned using a snug fit. Electrical contact to the working electrode was established using the platinum mesh. The reference electrode was inserted into the sleeve and all three electrodes were connected to the potentiostat. The cell was connected to the pump using Tygon® tubing and a 0.1 M KNO₃ electrolyte solution was pumped through the flow cell. In order to help remove air bubbles and wet the electrode surface, a mild electrochemical pretreatment was used. The remediation cell was held at +1.00 V for 10 minutes followed by -1.00 V for 10 minutes.

The performance of the carbonaceous material under study was tested by placing a solution containing a known concentration of metal ions into the sample reservoir. The solution was then allowed to pass through the remediation cell at a known flow rate and applied potential. The potential of the remediation cell was controlled using a Power Module (Model PWR-3, BAS). The performance was assessed by measuring the metal ion concentration in the effluent and comparing it to the initial ion concentration. The percentage efficiency of the remediation cell is equal to the percentage of metal ions removed from the solution by the remediation cell. (For these studies, the effluent was collected in Nalgene® bottles for subsequent analysis via voltammetry and/or atomic absorption).

Construction of the Scaled-Up Remediation Cell:

Construction of the 3-inch prototype remediation cell has been completed. A schematic of the apparatus is shown in Figure 2. The assembled system includes a flowmeter to measure flow rates and a manometer to measure the pressure drop across the remediation cell. The remediation cell was produced by machining a 4-inch Lexan™ rod. The 3-inch diameter flow channel was drilled through the Lexan™ body. Via the threaded plug, the depth of the carbon electrode bed can be varied between 0.5 and 4.0 inches. Electrical contact was established in the same manner used in the smaller cell. The prototype has been assembled and parametric studies have begun. Several problems were encountered with this model (see Results and Discussion).

A redesigned version of the remediation cell (Figure 3) was designed and constructed to compensate for the problems associated with the above cell. This new galvanic cell utilizes a two electrode system. The anode reaction was separate from the cathode reaction using a porous ceramic cylinder (0.2 μm pores). The inner chamber contained the carbonaceous material (97.5 grams, WVU foam for this study) where the removal of metal ions takes place. The cathode material can be varied up to 8 inches in length. Upon passing through the cathodic chamber, the treated effluent was directed to the inlet of the anode chamber. No decrease in removal efficiency was noted and the need for the anolyte solution was eliminated. The electrode system was enclosed in a Plexiglas® tube. A solution flow rate of 10.0 mL/min was used for the sample and a flow rate of 60 mL/min was used for the anolyte solution. The pH of the lab prepared solutions was adjusted using nitric acid prior to testing.

Initial removal efficiencies for the scaled-up remediation cell were lower than expected. It was believed that the surface area of the platinum wire used as the anode was insufficient to carry the necessary current. The outer (anode) chamber of the electrolytic cell was filled with the same mass of carbonaceous material (97.5 grams, WVU foam for this study) to ensure an anode surface area

equivalent to that of the cathode. The use of the carbonaceous anode boosted charge transfer and thus removal efficiencies. Removal efficiencies above 90% were easily obtained for lead with this setup.

The above setup was used to perform a study of several parametric variables. These included plating current, solution pH and solution flow rate. Samples containing 100 ppm lead were passed through the cell as each parameter was varied. The concentration of lead in the effluent was measured and the effect of each parameter determined.

The sample obtained from the Fort Benjamin Harrison Site was pumped through the scaled-up remediation cell without any prior treatment. The Ft. Ben Harrison sample contained 279 ppm Pb and had a pH of 4.7. A constant plating current of 60 mA was employed to remove the lead.

WORK PERFORMED THIS QUARTER

EXPERIMENTAL

The presence of excess cations (i.e. Na^+ and K^+) in the solution from added electrolyte was examined to determine if they affect removal efficiencies. These spectator ions are present at concentrations up to 1000X greater than the target ion (i.e. Pb^{2+}). Successive experiments were carried out with increasing electrolyte concentrations (0.01, 0.05, 0.10 and 0.50 M NaNO_3 and KNO_3). The removal of lead ions from these samples was monitored, as well as any changes in solution conductivity.

The long-term application of the 1.5-inch remediation cell was tested over a period of 54 hours using a 100 ppm lead solution. 100 mL aliquot samples were collected at selected time intervals and the concentration of the lead ions was measured. The total volume treated was 33 liters of 100 ppm lead solution over a 54 hour time period.

RESULTS and DISCUSSIONS

Several carbonaceous materials were examined for their performance as the cathode material. Each sample was tested to determine the effect of adsorption (no applied potential) and electroplating. Their performance was evaluated on their ability to remove the metal ions from solution.

To confirm minimal contribution from the platinum mesh to the metal removal, the system was first tested in the absence of any carbonaceous material. The response of the original feed solution was compared to the

response of the effluent collected at -1.00 V. It was shown that the platinum mesh does not contribute significantly (<2%) to the removal of the metal ions. Therefore, the removal of the metal ions is due to their interaction with the carbonaceous material and is not significantly influenced by the platinum mesh.

In order to assess electrosorption, the carbon electrodes were tested for their ability to remove cadmium and lead at potentials more positive than that required to reduce the metal ions. The removal of cadmium was examined at various potentials above and below its theoretical reduction potential. Based on the initial cadmium feed concentration (100 ppb), the potential at which cadmium should be reduced is -0.58 V. Electrosorption removed 46% of the cadmium from the solution at -0.20 V and 78% at 0.40 V. At potentials sufficiently negative to reduce the metal ions, electroplating removes 89% at 0.60 V, 91% at 0.80 V and nearly all (99%) at -1.00 V.

The removal of lead was conducted under the same conditions. The theoretical reduction potential for lead is -0.54 V. Removal of lead via electrosorption is shown also to occur at potentials more positive than the reduction potential. The removal efficiencies were 88% at -0.20 V and 92% at -0.04 V.

Continuous removal of lead from an aqueous stream using electroplating and electrosorption was used to demonstrate the long-term effectiveness of carbon nanofibers. The lead solution was passed through the remediation cell and the ions were removed by applying the desired potential to the carbon nanofibers. A potential of -1.00 V was applied to the remediation cell to remove lead ions via electroplating. Over a 72 hour period, removal efficiencies of 89% or greater were achieved for an inlet concentration of 100 ppm lead. Calculation of the lead removed over this period of time demonstrates a removal capacity of $1.025 \text{ g}_{\text{Pb}}/\text{g}_{\text{carbon fibers}}$, but it should be stressed that even at 72 hours, the carbon showed no signs of saturation (see Figure 4).

Removal of lead ions via electrosorption was carried out using a potential of -0.40 V. For an inlet lead ion concentration of 100 ppb, good removal efficiencies (90%) were obtained for the first 12 hours (see Figure 5). After that time a noticeable decrease in efficiency is evident. The removal efficiency drops to less than 50% after 57 hours. This decrease in removal efficiency is expected due to the neutralization of charge on the electrode surface. Regeneration of the carbon nanofibers would be required to maintain high removal efficiencies. For the first 12 hours of removal a capacity of $1.9 \times 10^{-4} \text{ g}_{\text{Pb}}/\text{g}_{\text{carbon fibers}}$ is obtained. At 57 hours this capacity increases to $7.1 \times 10^{-4} \text{ g}_{\text{Pb}}/\text{g}_{\text{carbon fibers}}$. Electrosorption of lead using a 100 ppm lead feed was attempted. Due to the smaller capacity of the carbon nanofibers when using electrosorption, the efficiency should drop off very quickly. As expected, within the first two hours the removal efficiency dropped to less than 20%.

The final desire is to recover the metals once they have been removed from the aqueous stream. It is possible to recover the metal ions plated on the electrode simply by reversing the potential. The metal ion concentration in the resulting effluent is greater than that of the feed. Electrodeposition and the subsequent recovery from carbon nanofibers was demonstrated with a 100 ppm lead ion feed. The lead ions were removed from the aqueous stream using an applied potential of -1.00 V. Subsequent recovery of the lead at an applied potential of +1.00 V, produced an effluent stream more concentrated in lead. The lead concentration in the recovered effluent was 730 ppm, demonstrating a 7:1 concentration of the waste stream. Individual metal ions can be removed and recovered selectively by these means.

A summary of the electrodeposition removal efficiencies for the carbon samples tested to date are shown in Table III. These results demonstrate that carbonaceous materials are extremely effective for the removal of heavy metal ions from solution.

Industrial Application:

Contact has been established with Paul Brezovec at Concurrent Technologies Center (CTC), Johnstown, PA. It has been proposed that the electrochemical method developed in this research be applied to their nickel-plating line for the removal and recovery of nickel. The final rinse stage produces an aqueous waste contaminated with nickel. The objective is to purify the rinse water for reuse and to recover the nickel for recycling back into the plating process.

Contact has also been made with Dr. Dave Szlag of the Environmental Protection Agency (EPA) in Cincinnati. The desire is to remove lead from aqueous solution obtained by washing lead from the contaminated soil on military firing ranges.

A local plating industry, Swanson Plating Company (Morgantown, WV) has been identified as a source of "real" samples for testing the remediation cell. Identification of the metals and acquisition of samples is ongoing.

Removal of nickel at the experimental conditions used for lead and cadmium resulted in low removal efficiencies. Due to the unique chemistry of nickel, careful attention must be given to increase the removal. Table IV demonstrates the need for tailoring the removal process for nickel. By applying a more negative potential (i.e. -1.60 V) enhanced removal efficiencies are obtained (97.9%). But at this potential the water is reduced and current is wasted due to the production of hydrogen gas. By simply pre-plating a small layer of copper or mercury, enhanced removal efficiencies are obtained. The

removal efficiency is also dependent upon the pH of the contaminant solution. For a 100 ppm nickel solution at pH 7.0, a small layer of pre-plated copper and a remediation cell potential of -1.20 V, 89.1% of the nickel is removed.

Radionuclides:

Two metals have been identified that need to be removed from aqueous wastes generated at several nuclear production and processing facilities: strontium and uranium. Both of these metals are available in non-radioactive isotopes. Strontium can be easily measured by atomic absorption at all levels under study. Atomic absorption can not be used to determine uranium concentrations below 100 ppm. Therefore, polarography was used to facilitate the measurement of uranium. Polarography can determine uranium concentrations between 20 ppb and 500 ppm. The "static mercury drop electrode" used for polarography was received this quarter and has been assemble and tested.

No removal of strontium was observed using the untreated WWU foam. This was expected due to the relatively low surface area ($0.5 \text{ m}^2/\text{g}$) of the "non-activated" carbon foam. Activation of the foam surface could increase the surface area to values necessary for the electrosorption of strontium and such an activation apparatus is in preparation.

The improvement in removal efficiencies using the higher surface area carbon fibers is shown in Table V. Removal of strontium using carbon nanofibers resulted in 5.90 % of the strontium to be removed. Usage of the ASI carbon fibers improved this removal to 34.4%. This improvement is most likely due to differences in the surface properties of the two carbon fibers. Alternative removal schemes, similar to those used for nickel have been examined to improve the removal of strontium from the aqueous waste. A coating of MnO_2 on the carbon surface improved the removal efficiency in a pH 11.0 solution to 68.0%.

The removal of uranium was examined using ASI carbon nanofibers. No removal was observed for adsorption alone (no applied potential). Upon applying a potential of -1.00 V, almost immediate removal of uranium is seen. Long-term removal of uranium was examined over a 48 hour period. Within the first hour the uranium concentration drops well below 5 ppm and it was not until the carbon material began to clog that any appreciable amount of uranium passed through. Even at 47 hours, when the solution flow has noticeably slowed, the effluent concentration only rose to 16.3 ppm. This large removal is believed to be caused by electrochemically-induced precipitation of the uranium oxide. Results for the long-term removal of uranium are shown in Figure 9.

Prototype Remediation Cell:

Several problems became apparent with the 3-inch diameter prototype electrolytic cell. Due to the increased distance between the electrodes and the large amount of carbon material used, a very large potential drop was experienced. This large potential drop was sufficient to prohibit the potential on the carbon material from becoming adequately negative to reduce the metal ions. Also the diameter of the carbon bed was increased but the depth of the bed was not. Even though more carbon cathode was used, the residence time in the cathode material was not increased. An improvement would be to increase the diameter less while increasing the depth of the bed. Also it was believed that the surface area of the auxiliary electrode was insufficient to handle the current necessary to reduce the metal ions. Therefore, a redesigned electrolytic cell (Figure 3) was developed to address these problems. The new cell utilizes a two electrode system. The cell employed the carbon material as a cathode to remove the metal ions from solution. The material was also used as the anode and was isolated from the cathode using the porous ceramic tube. A flow rate of 10 mL/min was used in the initial tests with this new cell design. Removal efficiencies for lead of lab generated samples was 99%. This is comparable to those obtained using the small remediation cell with a 10-fold increase in throughput.

A sample was provide by Dr. Szlag of the EPA from the lead solution generated by the treatment of contaminated soil at the Fort Benjamin Harrison Site. The concentration of lead in the sample as received was determined to be 279 ppm. Following a single pass through the scaled-up version of the electrolytic cell and with no sample pretreatment, the concentration of lead was lowered to 14 ppm. This corresponds to a 95% removal of lead from the waste sample.

Examination of several key experimental parameters was performed. The effect of pH, flow rate and galvanic current was investigated. Figure 6 shows the dependence of the removal efficiency for lead on the current used to plate the metal ions from solution. At 30 mA and above, 99% of the lead ions are removed.

Another parameter that was addressed was solution pH. Waste streams generated by various processes can have varying pH values. In order for this treatment method to be useful, it must be applicable over a wide range of pH's. This will eliminate the need for the addition of chemicals to adjust the pH. In Figure 7 the results of this study are shown. The low removal at pH=2.6 is caused by the chemical oxidation of the plated lead by the acid solution. This reaction occurs simultaneously with the plating reaction. Therefore, minimal adjustment of the pH may be necessary for certain highly acidic waste streams. Lead hydroxide [$\text{Pb}(\text{OH})_2$ (s)] is precipitated at a pH of 8.2 and above for the 100

ppm lead solution. Therefore, the lead is not in the ionic state in solution and an alternative removal method is needed.

Results for the above parametric studies indicate percentage removal of lead based on the effluent concentration at 2 hours. For each parametric study, the effluent concentration was measured every 15 minutes over the three hour period of the study. Similar trends were clearly apparent throughout the experiments.

The lead ions must spend a sufficient amount of time (residence time) in the carbon bed in order to be reduced. Residence time is a function of flow rate and the void volume of the packed carbon bed. By increasing the flow rate, the ions spend less time in the carbon bed. It is necessary to determine the flow rate at which the maximum amount of solution can be treated without sacrificing removal efficiency. Figure 8 demonstrates the expected decrease in removal efficiency with increased flow rate. In order to remove 99% of the lead ions from the waste sample, a flow rate of 0.10 mL/min/g_{carbon foam} is needed. If a removal efficiency of only 80% is required, the flow rate can be increased to ~0.20 mL/min/g_{carbon foam}.

The concentration of added electrolyte had a small, but positive effect on the removal of lead ions. The electrolyte ions (Na^+ and K^+) did not compete with the lead ions for the electrode surface. The presence of these ions reduced solution resistance and even improved removal efficiencies for lead. The conductivity of the solutions containing 0.01M, 0.05M and 0.10M sodium nitrate, increased from 1.31 S, to 4.76 S and 8.95 S, respectively. This increase in conductivity resulted in the following lead removal efficiencies: 94.6%(0.01M), 98.6%(0.05M) and 99.6%(0.10M). These ions improved the electrical conductivity of the solution leading to decreased iR drop and lower parasitic power losses, which in turn was believed to improve the electron transfer rates.

The long-term removal of lead using the scaled-up 1.5-inch prototype was superb. The improvements to the cell design resulted in the ability to remove lead ions at a 98% efficiency for a period of 54 hours (see Figure 10). A total solution volume of 32.4 liters was treated over this time period.

CONCLUSION

All proposed objectives have been met (see Table VI). An extensive search of the literature has been conducted in the areas of heavy metal removal, waste water treatment and water remediation. More than 150 papers have been amassed in these areas with special interest in the areas of electrochemistry and electrowinning using carbonaceous material. Further effort has been exerted in order to find applications of this technology to radionuclides.

The performance of the commercially available RVC as a cathode was satisfactory. Improved removal efficiencies were obtained with the novel carbon materials utilized in this study. Loose carbon nanofibers with a relatively large surface area ($200 \text{ m}^2/\text{g}$) effectively removed over 90% of cadmium and lead from an aqueous sample. Vapor Grown Carbon Fibers supplied by ASI also worked well for the removal of cadmium and lead by electroplating. The "coal-derived" foam produced in the WVU laboratory was an excellent cathode material. The concentration of lead in the effluent dropped below detectable amounts after only 4 hours. This demonstrated removal efficiencies approaching 100%.

The current industrial application of this technology was for the removal of nickel from a rinse solution used in the plating industry. Upon selection of the proper experimental conditions, a removal efficiency of 89.1% was obtained. This was accomplished by adjusting the pH of the waste stream and by pre-plating a small amount of copper onto the carbon surface to help catalyze the reduction of nickel.

The removal of strontium was demonstrated using carbon nanofibers. Removal efficiencies up to 34.4% were obtained, but removal capacity was soon reached. Other removal schemes with similar metal reaction mechanisms to those used for nickel are being examined. The use of a coating of MnO_2 , resulted in a removal efficiency of 68.0%.

The removal of uranium was examined using ASI "oxidized" nanofibers. Preliminary work demonstrated high removal efficiencies due to the production of a precipitate on the electrode surface. Such large removal efficiencies are due to electrically-induced precipitation. The 48-hour-long study of uranium removal demonstrated excellent results until the remediation cell clogged. Prior to clogging, maximum removal efficiencies were obtained (99%). Reduction of the volume of uranium waste was demonstrated by decreasing the waste volume from 2000 mL to 95 mL, and concentrating the uranium from 100 ppm to 1543 ppm. This demonstrates a large capacity of $0.15 \text{ g}_{\text{uranium}}/\text{g}_{\text{carbon}}$. The use of the 1.5-inch remediation cell and larger quantities of carbon will allow for the treatment of larger volumes of low level radioactive wastes.

The utility of this technique has been demonstrated on actual samples obtained from the cleanup of lead from soil at Fort Benjamin Harrison. The aqueous waste was generated by washing the lead from the contaminated soil with acetic acid. The decontaminated soil was returned to the site as clean fill. The aqueous waste generated was contaminated with lead (279 ppm). This aqueous waste was purified using the electrolytic cell to remove 95% of the lead.

The experimental variables under which removal is carried out can widely affect the performance of the electrolytic cell. However, when the optimal operating conditions have been determined, maximum removal is achieved. By paying careful attention to the experimental parameters, this process can be modified to obtain maximum removal efficiencies and throughput while minimizing energy and chemical demand.

Figure 1. Schematic of Electrochemical Remediation Cell.

Figure 2. Schematic of Scaled-Up 3-inch Remediation Cell.
 (1-Flowmeter; 2-Cell; 3-Manometer; 4-Pump; 5- Auxiliary Electrode;
 6-Pt Contact to Working Electrode; 7-Reference Electrode.)

Figure 3. Schematic of 1.5-inch Galvanic Remediation Cell.

Electroplating

Capacity @ 72 hours > 1.025 grams Pb/ gram of carbon
(***NOT at Saturation***)

Flow Rate = 3.1 mL/min/(gram carbon)

Figure 4. Long-Term (72 hr) Removal of Lead Using Carbon Nanofibers. ($E_{\text{CELL}} = -1.0 \text{ V}$)

Figure 5. Long-Term (102 hr) Removal of Lead via Electrosorption.
(Conditions: same, $E_{\text{CELL}} = -0.40 \text{ V}$)

Electrolytic Removal of Uranium

Figure 6. Long-Term 48 hr) Removal of Uranium.
(Conditions: same, $E_{\text{CELL}} = -1.00 \text{ V}$)

Figure 7. Effect of Plating Current on Removal Efficiency.
(1.5-Inch scaled-up remediation cell. Conditions: pH=5.3, 0.10 mL/min/ g_{carbon})

Figure 8. Effect of pH on Removal Efficiency.
(Conditions: same as Figure 7; 30 mA, 0.10 mL/min/g_{carbon})

Figure 9. Effect of Flow Rate on Removal Efficiency.
(Conditions: same as Figure 7; 30 mA, pH=5.3)

Long-Term Removal of Lead

Figure 10. Long-Term Removal of Lead Using Scaled-Up Cell.
(Conditions: same as Figure 7; 30 mA, pH=5.3)

**TABLE I: STANDARD REDUCTION POTENTIALS OF THE METAL IONS
OF INTEREST**

Reduction Reaction	E, V (vs. Ag/AgCl)
ELECTROSORPTION	
$\text{Sr}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{Sr} (\text{s})$	-3.108
$\text{U}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{U} (\text{s})$	-2.022
$2\text{H}_2\text{O} (\text{l}) + 2\text{e}^{-} \rightarrow \text{H}_2(\text{g}) + 2\text{OH}^{-} (\text{aq})$	-1.050
ELECTROPLATING	
$\text{Cd}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{Cd} (\text{s})$	-0.624
$\text{Ni}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{Ni} (\text{s})$	-0.453
$\text{Pb}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{Pb} (\text{s})$	-0.348
$\text{Cu}^{+2} (\text{aq}) + 2\text{e}^{-} \rightarrow \text{Cu}(\text{s})$	+0.115

TABLE II: PROPERTIES of CARBON MATERIALS

Surface Areas

Material	Surface Area (m ² /gram)	
	BET	Langmuir
Vapor Grown Carbon Fibers (ASI)		
Regular "AN" Fiber	76.78 (73.6)*	122.66
Oxidized "AN" Fiber	25.78	-
Pelletized "AN" Fiber	20.25 (20.77)*	-
WWU "coal-derived" Foam (23 NMP)	0.52	-
Carbon Nanofibers		
1094-A	115.13 (227.8)*	186.78
1094-B	182.77 (192.3)*	286.03
NF-46	43.28	-
Graphite Fiber Mats	0.59	-
Graphite Mat with Fibers	99.92 (112.8)*	-
Graphite Felt	0.17	-
Graphite Felt with Fibers		
NF46	55.41	-
F100	54.54	-
CF37	13.96	-
CN37	35.31	-
VGCF	76.78 (73.6)*	122.66
RVC		
100 ppi	0.092	-
80 ppi	44.80	-
Activated Carbon	784.32	-

TABLE III: Removal Efficiencies for Cd and Pb

<u>Cathode Material</u>		<u>Metal Removed</u>	
	<u>Cathode Potential, V</u>	Cd	Pb
Platinum Mesh	0.00	0%	0%
	-1.00	0%	9%
RVC	0.00	16%	0%
	-1.00	50%	8%
Nanofibers (1094-A)	0.00	0%	0%
	-1.00	91%	95%
Nanofibers on Graphite Mats	0.00	0%	0%
	-1.00	90%	92%
Graphite Felt	0.00	13%	7%
	-1.00	22%	97%
Nanofibers on Graphite Felt	0.00	0%	0%
	-1.00	46%	99%
ASI Carbon Fibers			
"pelletized"	0.00	-	0%
	-1.00	-	98%
"oxidized"	0.00	-	0%
	-1.00	-	99%
WU "Coal-Derived" Foam			
"crushed"	0.00	-	0%
	-1.00	-	99%

TABLE IV: IMMEDIATE INDUSTRIAL APPLICATION

(REMOVAL OF NICKEL VIA ELECTROPLATING)

$$E^\circ = -1.050 \text{ V}$$

$$E^\circ = -0.453 \text{ V}$$

MATERIAL	SURFACE CONDITION	pH	E_{CELL}, Volts	TIME, hours	% REMOVED
WVU Foams	bare-crushed	3.5	-1.2	2	0.00
		7.0	-1.2	2	0.00
			-1.4	2	68.7
			-1.6	2	97.9
	w/ Cu	3.5	-1.2	2	49.0
				4	57.3
		7.0	-1.2	2	82.4
				4	89.1
	w/ Hg	3.5	-1.2	2	44.3
				4	47.6
				6	50.6

TABLE V: DOE APPLICATION
REMOVAL OF STRONTIUM VIA ELECTROSORPTION

$$E^{\circ} = -3.108\text{V}$$

$$E^{\circ} = -1.050 \text{ V}$$

MATERIAL	CONDITION	pH	E_{CELL}, Volts	TIME, hours	% REMOVED
Nanofibers	bare	3.5	-1.00	2	5.90
				4	5.60
			-1.20	2	5.70
				4	3.20
WVU Foams	bare	3.5	-1.20	2	0.00
		7.0	-1.20	2	0.00
ASI	bare	7.0	-1.20	0.5	34.4
Nanofibers					
"pelletized"		11	-1.20	0.5	52.8
	MnO ₂	11	-1.20	0.5	68.0%

TABLE VI: PROJECT COMPLETION

ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON

<u>Task #</u>	<u>ID</u>	<u>Task Name</u>	<u>Duration</u>	<u>% Complete</u>
1	138	Task 4.2 Environmental Pollution Control Devices	52w	
2	139	Industrial Contacts	52w	20%
3	140	Define Industrial Needs	52w	20%
4	141	Test Removal Efficiencies Using Small Cell	52w	20%
5	142	Optimize Experimental Parameters	52w	30%
6	143	Study Affect of Interfering Ions	16w	50%
7	144	Design and Construct Large Prototype	12w	100%
8	145	Apply Large-Scale Prototype	24w	20%
9	146	Test Carbon Materials	52w	0%
10	147	Develop Recommendations	4w	0%
11	148	Quarterly Report #1	1w	100%
12	149	Quarterly Report #2	0.2w	-
13	150	Quarterly Report #3	0.2w	-
14	151	Quarterly Report #4	0.2w	-
15	152	Final Report	4w	-
16	153			

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TASK 4.3

Quarterly Report January-March 1997

1997 WVU COOPERATIVE AGREEMENT

**CROSS CUTTING INNOVATIVE TECHNOLOGIES:
EFFICIENT SEPARATIONS Design of a Rotating Membrane Filtration
System for Remediation Technologies**

**Victor H. Mucino, Associate Professor
Todd Sidwell, Graduate Student
Michael Shellenberger, Graduate Student**

Department of Mechanical and Aerospace Engineering

Task ID 155. Parametric CAD Representation. This task consisted in capturing the geometric features of the SpinTek filtration system. In particular the disk-membrane and associated shaft components. These features were taken in a parametric form in order to conduct parametric variations and parametric sensitivity analyses in subsequent tasks. The features were captured and few details are yet to be provided by SpinTek people to complete the task. Task is 95% complete.

Task ID 156. Modeling. In this task, the geometric models captured through the parametric models, were taken to a finite element modeler, to yield mechanistic models to conduct stress analysis and vibration analysis of the rotating shaft. Stresses on the disk and in the interface between the disk and the membrane were of particular interest. Modes of vibration were obtained to characterize the dynamic behavior. Additional analyses are yet to be carried out to incorporate the effect of the fluid in the dynamic behavior. This task is 90% complete.

Task ID 157. System Response. The finite element models generated in the previous task were transferred to the solver (IDEAS-MASTER SERIES) to carry out the response analysis of the system. The stresses obtained were post-processed to yield stress contour plots. The vibration analysis provided the main modal characteristics, natural frequencies and modes of vibration, which were post-processed to illustrate the principal modes. This task is 90 % complete.

Task ID 158. Post-Processing Results. All the analyses conducted in the previous tasks have been carefully post-processed to produce the graphs and plots with the most relevant information. All of this information has been captured in the form of a homepage that has been set up for easy access. This task will be reviewed continuously to reflect the most recent results. This task is 90% complete.

Task ID 159. Validation of Results. The stresses results were verified against theory of elasticity solutions for the case of rotating disks. The results obtained in the models are consistent with those provided by the theoretical results. However further work will be needed in subsequent tasks. This task is 50 % complete.

Note: All the results have been documented on a homepage that can be accessed at
<http://spline.mae.wvu.edu/spin/spint2.htm>

1997 WVU COOPERATIVE AGREEMENT

CROSS CUTTING INNOVATIVE TECHNOLOGIES: EFFICIENT SEPARATIONS

Design of a Rotating Membrane Filtration System for Remediation Technologies

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**Victor H. Mucino, Associate Professor
Todd Sidwell, Graduate Student
Michael Shellenberger, Graduate Student
Department of Mechanical and Aerospace
Engineering**

Contents

- Problem Description
  - Approach
    - Vibrations
    - Stresses
  - Analysis
    - Vibrational Analysis
    - Stress Analysis
  - Conclusion
-

Project Description

This project involves the design analysis of a SpinTek^a Filtration System. This system uses a combination of rotating centrifugal forces and turbulence in the fluid to promote the filtration action through the rotating membrane. This device consists of one or more rotating disks mounted on a shaft, which are encased inside a pressurized vessel. As the shaft spins turbulence, is created inside the vessel which causes the water to enter the shaft through the membrane. The objective of this project is to optimize the design in order to enhance the flow rate capacity of the system.

To improve the current design, base models must first be created. These models will provide a base stress and vibrational analysis to which improved models can be compared. In this project the software IDEAS is used to create various Finite Element Models, to analyze the relevant stresses on the disks and the modal vibrations of the entire system.

[Return to Contents](#)

Vibrations Approach

Several approaches were considered to complete the vibrational analysis of the SpinTek device. One approach considered involved making mapped meshed finite elements of each part of the device and assembling them into one composite part. This idea however was abandoned do to its unnecessary complexity. Finally it was decided to use a combination of beam sections and thin shells to complete the analysis. This approach while simplifying the problem provided an acceptable analysis of the modes of vibration. To emulate the rotating disks, thin shells were created on round surfaces with a thickness equivalent to that of the actual disks. This can be seen in the following drawing.

To model the rim around the disks, a solid rectangular beam section was created. This section was placed between the nodes on the outer edge of the disk mesh. The beam section used is shown in the following picture.

The shaft of the device consists of several stepped tubular segments. To model this, it was decided to use a series of beam section elements which were then assembled together into one part. The various shaft diameters can be seen below.

After completing the models of the various parts, they were all assembled as one part to perform the vibrational analysis. Before the analysis could be performed, appropriate boundary conditions had to be established. First, the model was restrained at the bottom of the shaft and immediately below the second disk. Next, 8 dynamic degrees of freedom were placed on each disk and 2 were placed on the shaft. The model was now ready to be solved. Below is a picture of the assembled model with the boundary conditions applied.

[Return to Contents](#)

Stresses Approach

To perform the stress analysis of the device, it was decided to use a system of meshed surfaces and create an axisymmetric model. First, surfaces were created in the form of profiles of each piece. These surfaces were then assembled together into their appropriate locations as shown below.

The surfaces were then meshed and the nodes of adjacent parts were coupled together. In addition to these boundary conditions, one node on the axis of symmetry was fixed in all directions. All other nodes on the axis were allowed to translate in the X and Z directions. The only initial condition applied was an omega of 1800 RPM (188.5 rad/sec) about the Z axis. The meshed surfaces and boundary conditions are shown below.

[Return to Contents](#)

Vibrational Analysis

Using the IDEAS solver, several modes of vibration were found for the device based on the given boundary conditions. Modes one and two were found to occur at 14.6 cycles/sec and 14.8 cycles/sec respectively. These values are below the operating frequency of 30 cycles/sec, but they will be encountered during start up. The next mode occurred at 104.8 cycles/sec, well above the operating frequency of the device. The first three modes of vibration can be seen below.

* For a Quicktime movie animation of the mode of vibration click on its the picture.

[Return to Contents](#)

Stress Analysis

For the stress analysis, IDEAS solver was used to view tangential and von mises stresses as well as the displacement of the disks. The stresses obtained were all within the allowable limits for the material used.

RESULTS: 2- B.C. 1,LOAD 1,STRESS_2
 STRESS - Y MIN:-2.41E+03 MAX: 2.15E+04
 DEFORMATION: 1- B.C. 1,LOAD 1,DISPLACEMENT_1
 DISPLACEMENT - MAG MIN: 4.46E-07 MAX: 7.92E-04
 FRAME OF REF: PART

/u1/user1/mucino/spintek/vic.nf1.2

VALUE OPTION:ACTUAL
 SHELL SURFACE: TOP

2.15E+04
2.07E+04
1.99E+04
1.91E+04
1.83E+04
1.75E+04
1.67E+04
1.59E+04
1.51E+04
1.43E+04
1.35E+04
1.27E+04
1.19E+04
1.12E+04
1.04E+04
9.55E+03
8.76E+03
7.96E+03
7.16E+03
6.36E+03
5.57E+03
4.77E+03
3.97E+03
3.17E+03
2.37E+03
1.58E+03
7.78E+02
-2.00E+01
-8.18E+02
-1.62E+03
-2.41E+03

RESULTS: 1- B.C. 1,LOAD 1,DISPLACEMENT_1
 DISPLACEMENT - MAG MIN: 4.46E-07 MAX: 7.92E-04
 DEFORMATION: 1- B.C. 1,LOAD 1,DISPLACEMENT_1
 DISPLACEMENT - MAG MIN: 4.46E-07 MAX: 7.92E-04
 FRAME OF REF: PART

/u1/user1/nucino/spintek/vic.nf1.2

VALUE OPTION:ACTUAL

7.92E-04
 7.66E-04
 7.40E-04
 7.13E-04
 6.87E-04
 6.60E-04
 6.34E-04
 6.08E-04
 5.81E-04
 5.55E-04
 5.28E-04
 5.02E-04
 4.76E-04
 4.49E-04
 4.23E-04
 3.96E-04
 3.70E-04
 3.44E-04
 3.17E-04
 2.91E-04
 2.64E-04
 2.38E-04
 2.12E-04
 1.85E-04
 1.59E-04
 1.33E-04
 1.06E-04
 7.96E-05
 5.32E-05
 2.68E-05
 4.46E-07

Using Microsoft Excel, the radial and tangential stresses were plotted. This plot shows the stress distribution over the radius of the disk. The maximum stresses obtained will be used as critical design stress of the material of the device.

[Return to Contents](#)

Conclusion

The finite element models made of the SpinTek Filtration System were modeled using the IDEAS software package. Vibrational and stress analysis were conducted and the maximum relevant stresses and modes of vibration were computed. The stresses were within the allowable design limits of the material. Two modal vibrations were found to be within the operating range of the device. These models will serve as a good base line pending further analysis and optimizations.

[Return to Contents](#)

TASK 5.0

Outreach

TASK 5.1

Small Business Technical Based Support

1997 FETC Task No. 5.1

Quarterly Technical Progress Report

Reporting Period:

January - March 1997

Work Performed Under Contract

No. DE-FC21-92MC29467

For:

U.S. Department of Energy

Office of Fossil Energy

Federal Energy Technology Center

Morgantown, West Virginia

By:

E. E. Cook, Ph.D., P.E.

J. D. Quaranta, Ph.D., P.E.

Department of Civil and Environmental Engineering

West Virginia University

April 1997

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EXECUTIVE SUMMARY

The Small Business Support program was initiated to address problems that innovative site remediation technologies developed by the small businesses and funded by the US Department of Energy - Federal Energy Technology Center (FETC) encounter during commercialization. During this reporting period meeting and site visits with DOE's - Ashtabula Project Office, RMI Environmental Services, and INTERA Corporation were held to assist these organizations in support of their individual needs. Results of these meetings have resulted in furthering the exposure of WVU's developed technologies for direct application to DOE's needs.

INTRODUCTION

Subtask 5.1, "Small Business Technology Base Support" was initiated to address the technical problems or barriers to commercialization of innovative site remediation technologies. Through FETC, a very large percentage of the USDOE innovative remediation technologies being developed is done by small businesses. Small business owners are creative thinkers usually technically competent beyond their resource base. At times they find technical problems and their lack of highly technical personnel creates an unsurpassable barrier for the small business to get their innovative technologies developed and commercialized. By ferreting out these problems, this program will allow teaming of a faculty member with the small business to solve the problem allowing further development of the innovative technology to occur.

PURPOSE

The Small Business Support program was initiated to explicitly address the problems and barriers to commercialization of the innovative site remediation technologies developed by the small businesses funded by FETC.

BACKGROUND

Over the past four years the WVU Cooperative Agreement has funded numerous projects which has resulted in defining at least four areas of core competencies at WVU which may be called upon to provide assistance to the small businesses that FETC funds in the area of site remediation.

The core competencies include:

- Remediation processes such as surfactant flushing of soils and chemical and biological destruction of polyaromatic chlorinated hydrocarbons
- Confinement Barriers such as the Circulating (Desiccation) Air Barrier and Chemical Grouting Barriers
- Fluid Recirculating Systems such as the Drain Enhanced Soil Flushing using Prefabricated Vertical Drains (PVD's)
- Separation processes using novel membranes and novel forms of carbon

METHODOLOGY

By attending the kickoff meetings and other periodic meetings of the small businesses funded by FETC an understanding of the project is obtained. During the presentations, value added opportunities for collaboration between the small businesses and university researchers are carefully watched for. Discussions with program managers also provide opportunities for interaction. For the long range research opportunities, the initiative at USDOE HQ level bringing together elements from DOE, DOD, NASA, Commerce and others to coordinate interagency site remediation capabilities and needs will be very

helpful. Also, attendance at various meetings and conferences on site remediation provides insights into research needs.

From all of the previous sources, research opportunities are ferreted out and projects are developed with university faculty to carry them out.

WORK PERFORMED THIS PERIOD

During this reporting period association with RMI Environmental Services and the US DOE Ashtabula Project Office continued. Drs. Cook and Quaranta performed a site visit to RMI Ashtabula in February to observe first-hand demonstration of the RMI soil washing operation. Meetings were held with RMI engineers to continue discussions of where WVU research technologies may be inserted into the RMI remediation processes. Follow-up visits were made by representatives from RMI and DOE - Ashtabula to WVU and FETC in March. The purpose of this visit focused on observing operation of the SpinTek filtration apparatus and meeting with researchers to obtain information regarding this technology and the Prefabricated Vertical Drain operation.

Meeting were held with INTERA Corp. and WVU for responding to a Research Opportunity Announcement from FETC.

CONCLUSIONS/WORK PLANNED FOR NEXT PERIOD

During the next reporting period WVU will host the annual WVU Cooperative Agreement Program Review at FETC. During this meeting the 1997 program will be presented by each research Principal Investigator with their industry tie-in.