gti.

GASIFICATION AND GAS PROCESSING

# Cement-Lock<sup>®</sup> Technology for Decontaminating Dredged Estuarine Sediments

Enhancements and Process Improvements at the Cement-Lock Demonstration Facility

-

**Submitted To:** Brookhaven National Laboratory Upton, New York 11973

Prepared by: T. Robert Sheng Project Manager 847-768-0773 robert.sheng@gastechnology.org

Michael C. Mensinger Senior Chemical Engineer 847-768-0602 mike.mensinger@gastechnology.org

October 22, 2004

*Gas Technology Institute* 1700 S. Mount Prospect Rd. Des Plaines, Illinois 60018 www.gastechnology.org

### EXECUTIVE SUMMARY

The Gas Technology Institute (GTI) prepared this report on "Enhancements and Process Improvements at the Cement-Lock Demonstration Facility" as part of a project with Brookhaven National Laboratory (BNL, Upton, New York). This work was conducted under the project "Cement-Lock<sup>®</sup> Technology for Decontaminating Dredged Estuarine Sediments – Phase V.

The overall objective of this program is to conduct a large-scale test of the Cement-Lock Technology to confirm the attributes of the technology for treating contaminated sediment. A specific objective includes processing about 350 cubic yards of sediment dredged from the New York/New Jersey harbor (Stratus Petroleum site, Upper Newark Bay) to demonstrate that –

- 1. The organic contaminants contained in the sediment are destroyed by the Cement-Lock process with an overall destruction and removal efficiency of at least 99 percent,
- 2. The heavy metals present in the sediment are immobilized in the cement produced from the sediment as demonstrated by the Toxicity Characteristic Leaching Procedure (TCLP) and the Multiple Extraction Procedure (MEP), and
- 3. The cement produced from the sediment meets ASTM standards for blended as well as Portland cements.

The initial startup and operation of the Cement-Lock demo plant in December 2003 was successful in producing the first samples of Ecomelt<sup>®</sup> from Stratus Petroleum sediments. However, plant operation was terminated involuntarily when molten slag accumulated in the drop-out box, solidified, and eventually caused the Ecomelt drag conveyor to jam.

Several equipment-related problems and concerns contributed to this situation: Among them were inadequate mixing and conveying of feed materials, inadequate control of combustion air and natural gas to the rotary kiln, melt, and secondary combustion chamber burners, excessive air leakage into the system, inadequate air seal around the rotary kiln discharge, insufficient thermal energy input to the drop-out box, insufficient real-time information on the developing situation in the drop-out box, and inadequate atomization of flue gas quencher water.

In GTI's proposal to BNL (GTI Proposal No. 18656-18R2, dated March 10, 2004) a list of equipment enhancements and process improvements was proposed to address the above issues. The first deliverable under the current contract amendment was payable upon placement of orders for the retrofits and process enhancements described above. This deliverable, which consisted of a complete listing of the purchase orders placed (purchase orders and relevant invoices from equipment manufacturers and engineering company for engineering work) and costs incurred by GTI, was submitted to BNL on September 15, 2004. The second deliverable under the current contract amendment calls for a report documenting the completion of initial start-up of the Cement-Lock demo plant with retrofits and enhancements in place.

This report describes the implementation and status of those enhancements and process improvements at the Cement-Lock demonstration facility and describes the initial start-up of the Cement-Lock demo plant with those retrofits and enhancements in place. The report covers the work conducted during the time period from April through July 2004.

iv

# TABLE OF CONTENTS

.

~

.

<u>Section</u>		Page
	EXECUTIVE SUMMARY	iii
I	INTRODUCTION	1
	Cement-Lock Technology	1
	Cement-Lock Demonstration Project	4
II	IMPLEMENTATION OF PLANT EQUIPMENT RETROFITS AND ENHANCEMENTS	6
	Feed Solids Handling	7
	Ecomelt Generation and Drop-Out Box	8
	Gas Clean-Up	10
	Miscellaneous Repairs / Retrofits	11
III	INITIAL PLANT START-UP WITH RETROFITS AND ENHANCEMENTS IN PLACE	13
	Preparations for Plant Operations	13
	Initial Start-Up Test #1 (July 16, 2004)	15
	Start-Up Test #2 (July 21, 2004)	18
	Post-Test Evaluation	21
IV	SUMMARY	24
Appendix A.	Instrumentation Calibration Report (KGB Controls, Tyrone, GA)	A-1
		<i>1</i> <b>3</b> - 1
Appendix B.	Report on Burner Status by North American Manufacturing Co., Inc.	B-1

# LIST OF FIGURES

Figure No. Pag		Page
1	Process Flow Diagram for Cement-Lock Demonstration Plant	3
2	Time-Temperature History of the Initial Start-Up of the Cement-Lock Demo Plant with Retrofits and Enhancements (July 16-17, 2004)	19
3	Time-Temperature History of the Second Start-Up Test of the Cement-Lock Demo Plant with Retrofits and Enhancements (July 21-22, 2004)	22

.

.

.

### I. INTRODUCTION

The Gas Technology Institute (GTI, Des Plaines, IL – formerly the Institute of Gas Technology) developed the Cement-Lock<sup>®</sup> Technology in response to the need identified by the U.S. Environmental Protection Agency (Region 2) and the U.S. Army Corps of Engineers (New York District) under the federal Water Resource Development Act (WRDA). The overall objective of the WRDA Program was to bring fast-track sediment decontamination technologies to the commercial market to decontaminate sediment from the New York/New Jersey waterways and to find beneficial uses for these sediments. The Cement-Lock Technology is a patented technology. The intellectual property was jointly developed by GTI and Unitel Technologies (Mount Prospect, IL) and is held by Cement-Lock Group, L.L.C. (Des Plaines, IL).

The Cement-Lock Technology not only decontaminates the sediment but it also converts it into construction-grade cement, which has properties similar to those of ordinary Portland cement. Under the WRDA program, GTI conducted tests at the bench-scale as well as at continuously operating pilot-scale using Newtown Creek (New York) sediment to illustrate the concept of the technology. The results of these tests demonstrate three favorable characteristics:

- 1. All the organic contaminants present in the sediment were destroyed
- 2. The inorganic contaminants were immobilized in the cement matrix; and
- 3. The cement produced from these tests surpassed the strength requirements for Portland cement as required by ASTM (American Society for Testing and Materials) protocols.

#### **Cement-Lock Technology**

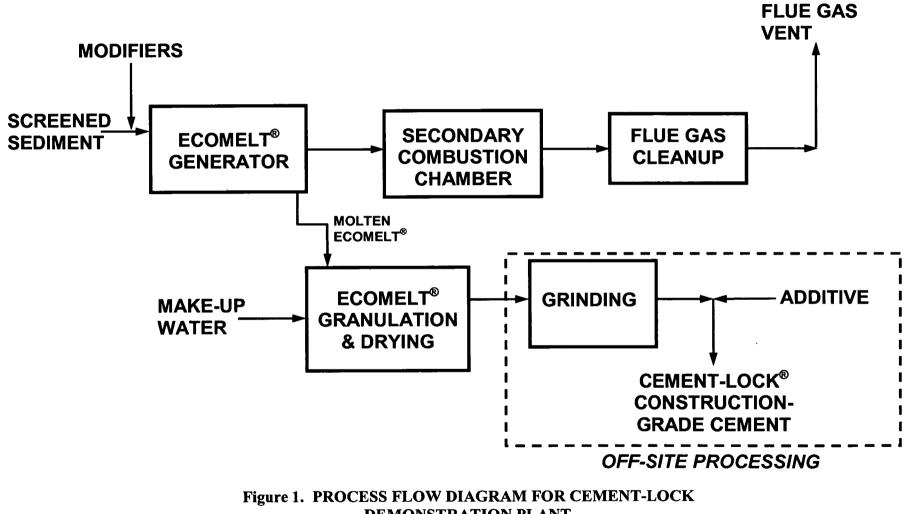
The Cement-Lock technology is a thermo-chemical remediation technology that converts contaminated materials into construction-grade cement, a marketable product for beneficial use. The Cement-Lock demonstration plant incorporates all of the major equipment components needed to demonstrate and characterize the process. The final steps in producing the Cement-Lock construction-grade cement, namely, grinding and

blending the Ecomelt with ordinary Portland cement or another lime (CaO) source will be done at an off-site facility.

The mixture of sediment and modifiers is conveyed to the rotary kiln melter (Ecomelt<sup>®</sup> Generator) by screw conveyor. The Ecomelt Generator itself is maintained at a temperature of 2500° to 2600°F by combustion of natural gas with air. This temperature is sufficient to yield a melt with a manageable viscosity and cause the minerals in the sediment and modifier mixture to react together. During processing, the sedimentmodifier blend is thermo-chemically transformed from the recognizable materials in the feed to a homogeneous melt. All nonvolatile heavy metals originally present in the sediment are incorporated into the melt matrix via an ionic replacement mechanism. The molten material moves through and exits the Ecomelt Generator by kiln rotation and gravity. It then falls into a plenum (the drop-out box) through high-pressure streams of water, which immediately quenches and granulates the melt. The quenched and granulated material is called Ecomelt. The Ecomelt is removed from the quench granulator by an inclined drag conveyor, which also partially dewaters it.

A portion of the quench water vaporizes during the quenching operation. Makeup water required is provided from a local supply of process water.

Flue gas from the Ecomelt Generator flows into the Secondary Combustion Chamber (SCC), which provides an additional 2 seconds of residence time at a minimum temperature of 2200°F to ensure complete destruction of any organic compounds that survive the severe thermal conditions in the Ecomelt Generator. Effluent gas from the SCC is rapidly cooled via direct water injection to prevent the formation or recombination of dioxin or furan precursors. Powdered lime is injected into the cooled gas to capture sulfur compounds and sodium and potassium chloride (NaCl and KCl) from seawater. The sulfur/salt/lime mixture is removed from the flue gas stream by a baghouse. The collected baghouse material is containerized and shipped off-site to an ordinary landfill. This material represents less than 2 percent of the raw sediment volume. Volatile heavy metals – such as mercury – are removed from the flue gas as it passes through a fixed bed of activated carbon pellets. The charge of activated carbon in



. .

• ,



the fixed bed is more than sufficient to capture the mercury from the approximately 350 cubic yards of sediment allocated for the pilot demonstration project. Cleaned flue gas is vented to the atmosphere at about 350°F via an I.D. (induced draft) fan. Figure 1 shows a simplified process flow diagram of the Cement-Lock demonstration facility in Bayonne, New Jersey.

After the demonstration, the activated carbon pellets will be analyzed for mercury content. If the carbon has reached its capacity, it will be removed and sent to an appropriate spent activated carbon reprocessor.

#### **Cement-Lock Demonstration Project**

The Cement-Lock Technology was selected by the New Jersey Dredging Project Facilitation Task Force (appointed by then New Jersey Governor Christine Todd Whitman) to participate in a two-phase sediment decontamination demonstration program. In Phase I, the technology vendor was to process 600 gallons (about 3 cubic yards) of sediment. If the results of Phase I were favorable, the vendor could be selected to participate in Phase II, in which from 30,000 to 150,000 cubic yards of contaminated sediment would be processed. The actual Phase I testing of the Cement-Lock technology will involve testing about 500 cubic yards of sediment dredged from the Stratus Petroleum site (Newark, NJ) in Upper Newark Bay.

A public-private partnership was created to carry out the demonstration project. Specifically, ENDESCO Clean Harbors, L.L.C. (ECH – a wholly owned subsidiary of GTI) licensed to transact business in New Jersey was formed as a separate entity with a mandate to demonstrate Cement-Lock Technology on harbor sediment. Parties to this partnership include the natural gas industry represented by the Gas Research Institute (GRI), Brookhaven National Laboratory (under the sponsorship of the EPA/ACOE WRDA project), a cement / concrete manufacturing company, the State of New Jersey Department of Transportation Office of Maritime Resources (NJ-DOT/OMR), and the Gas Technology Institute. Technical support in the areas of product development and product utilization is provided by Construction Technology Laboratories (the research arm of the Portland Cement Association) and the New Jersey Department of

Transportation. Anderson 2000 Inc. (A2K, Peachtree City, GA) manufactured the Cement-Lock demonstration plant equipment. A2K has also provided plant engineering, plant construction and start-up and commissioning support.

This report summarizes the work conducted by GTI for the second deliverable under the current contract amendment with BNL for completion of initial start-up of the Cement-Lock demo plant with retrofits and enhancements in place.

The work is supported under Contract No. 725043 with Brookhaven National Laboratory with funding provided, in part, through the Water Resources Development Act through Interagency Agreement No. DW89941761-01-1 between the U.S. EPA-Region 2 and the U.S. Department of Energy. Additional funding for the project has been provided by the Gas Research Institute (GRI, Des Plaines, IL).

## II. IMPLEMENTATION OF PLANT EQUIPMENT RETROFITS AND ENHANCEMENTS

The overall objective of this work is to implement equipment retrofits and enhancements to address problems encountered during the initial start-up of the Cement-Lock demonstration plant in December 2003. The problems encountered led to the involuntary shutdown of the Cement-Lock demo plant. The implementation of these mechanical or operational solutions will enhance the overall operability of the demo plant.

Several equipment-related problems contributed to the involuntary shutdown during initial start-up in December 2003. These problems are categorized into four major sections:

- 1. Feed Solids Handling
- 2. Ecomelt Generator and Drop-Out Box
- 3. Gas Cleanup, and
- 4. Miscellaneous Repairs / Retrofits

Among the problems identified were the inability to consistently and uniformly feed sediment into the system, inadequate air seal around the rotary kiln at the drop-out box, insufficient thermal energy input to the drop-out box, and insufficient real-time information on the situation developing in the drop-out box. Other problems related to sediment handling and feeding included sticking of sediment to and jamming of the sediment feed screw (C-101). Problems related to gas cleanup included inadequate atomization of the flue gas quencher water and excessive production of condensate from the flue gas quencher.

The proposed solutions to problems identified after the December 2003 campaign and the actions taken are listed below. Some of these items are self-explanatory. For others short descriptions are included. Please note that some of the proposed solutions were not instituted. After careful consideration, these were judged to be not cost-effective for the benefit that was originally conceived. Those proposed solutions are marked "not done" and are followed with a brief explanation.

#### **Feed Solids Handling**

٠	Install a tent to cover the raw sediment storage area	- Complete
•	Spread the raw sediment over the ground (under the tent) to reduce its moisture content by air drying	– Complete
•	Blend predetermined amounts of modifier solids with air-dried sediment	– Complete
٠	Install a removable cover on top of main sediment storage hopper (T-101)	– Complete
•	Install variable speed drive on the four conveyor motors of the main sediment storage hopper (T-101)	– Complete
•	Install forward/reverse switches on sediment feed conveyors (C-101 and C-112)	– Complete
•	Increase the speed of water-cooled screw/auger (C-151)	- Complete
•	Install a discharge chute on the main sediment feed conveyor (C-101)	– Complete
٠	Install removable covers on the sediment feed conveyor (C-101)	- Complete

These modifications will enable the sediment or sediment-modifier mixture to be fed more consistently and uniformly into the system via the existing screw conveyors.

Installing a tent over the sediment storage area significantly improved the feed preparation activities. The tent is 90 feet long and 60 feet wide. At its highest point it is 24 feet tall. The tent has 6 support poles located within the covered sediment storage area.

The removable cover on the main sediment hopper (T-101) consists of plastic tarps draped over a central horizontal pole and tied down with guy lines. When the hopper is being loaded, the guy lines are used to retract a portion of the tarp exposing the material in the hopper below.

Air drying of raw sediment and premixing of dried sediment with modifier solids will greatly improve the operability of the feed handling system. Adding forward/reverse motor switches to the conveyors will also improve the operability of the conveyors.

The fixed-speed drive of the water-cooled screw/auger C-151 was converted to a variable-speed drive. The original specification for screw/auger C-151 called for it to deliver 117 cubic feet (4<sup>1</sup>/<sub>3</sub> cubic yards) per hour of sediment and modifier mixture. This relates to a throughput capacity of 30,000 cubic yards per year of sediment, which is significantly higher than the target feed rate for demo plant testing. The variable-speed drive allows the conveyor to be set at a specified feed rate. If material begins to accumulate in the plenum above the C-151 screw/auger, the speed can be increased.

#### **Ecomelt Generation and Drop-Out Box**

٠	Repair air seal (overlapping leaf seals) assembly at kiln discharge	- Complete
٠	Seal weld gaps in seal frame around kiln discharge	– Complete
•	Repair and retune existing melt burners and air/fuel trains as needed	– Complete
•	Remove pusher plate/tile assembly and its cooling water supply and return system	– Complete
•	Install refractory lined wall to replace pusher plate/tile	– Complete
•	Install 5 melt burners at strategic locations around the kiln discharge	- Complete
٠	Install air and fuel gas trains and control panel for new melt burners	- Complete
•	Install combustion air blower system for new melt burners (including motor control center switch addition)	– Complete
٠	Install flue gas sampling nozzles at kiln discharge (for CO & $O_2$ measurement)	– Complete
٠	Install 2 sight ports at kiln discharge including safety blast gates and air purge	– Complete
٠	Install TV camera and monitor system at kiln discharge	- Not done
٠	Replace damaged thermocouples at kiln discharge	- Complete

- Install forward/reverse switch on granulator conveyor (C-203) Complete
- Verify settings of all existing and new burners (North American Manufacturing Co., Ltd.) – Complete
- Install operator access port (manway) at kiln discharge Complete

These modifications and retrofits will minimize the heat loss from the kiln discharge and provide additional thermal input so that the slag discharged from the kiln can be kept in a fluid molten state. The photograph (below left) shows the three new North American Manufacturing Co. Hot Spot burners installed at the drop-out box and connected to the natural gas and air supplies. The photograph (below right) shows the high-velocity Tempest burner installed on the north wall of the drop-out box (a second burner is installed on the south wall).



The air seal was improved by the simple addition of a wire strap wrapped circumferentially around the individual steel leaves connected with a spring (photograph above left). The spring keeps the wire taut and the leaves from flapping.

The new nozzle on the west side of the drop-out box enables the concentrations of oxygen  $(O_2)$  and carbon monoxide (CO) of the flue gases exiting the rotary kiln to be measured to facilitate the settings of natural gas and air inputs and to improve combustion efficiency and reduce excess air (which saves fuel).

The two new sight ports were installed on the west wall of the drop-out box. These will enable operators to visually note conditions where the slag exits the kiln and enters the drop-out box. After considerable discussion, it was decided that a camera would add only limited benefit to the operation of the rotary kiln melter system during this phase of testing. In addition to its initial acquisition cost, costs would be incurred to install and connect the camera to electrical and water utilities (it needs to be water cooled because of the local temperature) and lead the video feed to the monitor in the trailer. Additional manpower would be needed to maintain the camera and to monitor its output, which would detract from other staff duties. Another view port would be required for direct viewing if a camera were to be mounted on an existing view port. Taken together, these costs were considered too much for this phase of the project. For the extended operation of the Phase II effort, which will process up to 3,000 cubic yards of sediment, a camera could be useful.

The new operator access door (manway) was installed on the west wall of the drop-out box just below the level of the two new sight ports. The access is 27 inches wide and 24 inches tall.

#### **Gas Clean-Up**

•	Remove the double tipping valves at the bottom of the flue gas quencher	– Complete
•	Install condensate surge tank and recirculating pump on the exit of flue gas quencher (Z-301)	– Not done
•	Relocate spray nozzles in throat of flue gas quencher	- Complete
•	Install pressure indicators on air / water lines to spray nozzles of gas quencher	– Complete
•	Repipe air and water to the spray nozzles eliminating all of the solenoid valves and the by-pass air lines	– Complete
•	Install backup thermocouple at outlet of flue gas quencher	– Not done
•	Locate and repair air leakage in I.D. fan expansion joint	- Complete
•	Repair the Continuous Emissions Monitoring System	- Complete
•	Replace/repair ID fan expansion joint	– Not done

These modifications will enhance the performance of the flue gas quenching system, reduce pressure fluctuations, and reduce the amount of excess air leaking into the system.

The double tipping valves were removed. A simple flange was fabricated to cover the opening left by removing the double-tipping valves. Instead of installing a condensate surge tank with recirculating pump as originally proposed, we decided to route any condensate from the bottom of the flue gas quencher directly to the granulator recirculation tank (T-203). Both water sources are "contact" water, meaning that they have been in contact with process gases and, as such, cannot be disposed of without treatment. This was a logical and less expensive solution to this particular problem.

The four spray nozzles in the throat of the flue gas quencher generated a considerable amount of condensate during operation. To reduce or eliminate this unwanted situation, the spray nozzles were completely disconnected from the nozzle header and were then rewelded so that they extended about 3 inches farther into the spray plenum than before. This was sufficient to insure that the spray would not come into contact with the walls. Also, pressure gages were installed on each water header so that the water pressure as well as the air pressure could be monitored and adjusted according to the manufacturer's specifications for proper spray development. During subsequent operation, the quantity of condensate generated as well as the pressure and temperature fluctuations in the flue gas quencher were significantly reduced.

The damage to the I.D. fan expansion joint due to the temperature excursion that occurred October 2003 was determined to be superficial. No leakage was detected and not further action was taken.

#### **Miscellaneous Repairs / Retrofits**

٠	Recalibrate all flow transmitters	- Complete
٠	Repair water supply leaks at the hot box, trailers, safety shower, and conveyor C-151	– Complete
•	Repair Continuous Emission Monitoring System (CEMS)	– Complete

These modifications will ensure that the flow measurement devices are properly working and providing accurate information to the computer data acquisition and control system. A total of 18 instruments were recalibrated by KGB Controls (Tyrone, GA). These instruments are listed below.

FIT-203	Flow transmitter for water to granulator sprays (C-203)
FIT-502	Flow transmitter for natural gas to plant
FIT-511	Flow transmitter for natural gas to primary burner
FIT-570	Flow transmitter for natural gas to melt burners
FIT-164	Flow transmitter for air to melt burners
FIT-105	Flow transmitter for air to primary burner
TT-303	Temperature transmitter for baghouse outlet
TT-301	Temperature transmitter for flue gas quencher outlet
TT-206	Temperature transmitter for dryer outlet
TT-304	Temperature transmitter for activated carbon bed outlet
TT-305	Temperature transmitter for flue gas at stack outlet
TT-404	Temperature transmitter for water from C-151 (water-cooled screw)
TT-202B	Temperature transmitter for secondary combustion chamber (backup)
TT-202A	Temperature transmitter for secondary combustion chamber (primary)
TT-201B	Temperature transmitter for rotary kiln (backup)
TT-206A	Temperature transmitter for dryer combustion chamber
TT-203	Temperature transmitter for drop-out box (Z-203)
TT-201A	Temperature transmitter for rotary kiln (primary)

The report prepared by KGB for the calibration work is presented in Appendix A.

Information collected by the CEMS is crucial to the operation of the plant as the oxygen and carbon monoxide contents in the flue gas are limited by the NJ-DEP Air Quality Permit. While the CO monitor was being repaired by Rosemount Analytical, we rented another unit from Clean Air Rentals (Palatine, IL). The repaired CO monitor has been since returned and reinstalled in the CEMS cabinet. It is fully functional.

# III. INITIAL PLANT START-UP WITH RETROFITS AND ENHANCEMENTS IN PLACE

After the equipment retrofits and process enhancements were in place, GTI and its operating team provided by RPMS Consulting Engineers (RPMS, Monroe Township, NJ) prepared the system for its initial start-up.

During the initial start-up, GTI and RPMS conducted two tests in the Cement-Lock demo plant. The feedstock for both tests was air-dried sediment pre-blended with modifiers.

The objective of the first test was to operate the Cement-Lock demo plant with the newly installed equipment retrofits and process enhancements in place and begin processing the pre-blended sediment-modifier mixture. The test was not successful and was terminated involuntarily when slag accumulated in the drop-out box completely blocking the opening to the Ecomelt granulator.

The objective of the second test was to operate the Cement-Lock demo plant at conditions that would minimize slag accumulation including increasing the temperature of the drop-out box and adding additional modifiers and flux to the feed. The combined effects were to improve slag fluidity. This test was also involuntarily terminated when slag blocked the opening to the Ecomelt granulator.

This section of the report summarizes the work accomplished during the initial start-up of the Cement-Lock demo plant with equipment retrofits and process enhancements in place:

- Preparations for plant operations
- Initial Start-Up Test #1 (July 16, 2004)
- Start-Up Test #2 (July 22, 2004)
- Post Test Evaluation

### **Preparations for Plant Operations**

Prior to the first demo plant test, GTI conducted laboratory-scale tests to determine how much drying was required to improve the flowability and conveyability of sediment feed material. The results of these tests showed that an air-dried sediment and modifier mixture with a net moisture content of about 20 weight percent (or lower) could be conveyable. Based on these results, the operating crew prepared several large batches of air-dried sediment blended with specific quantities of modifier solids. The bobcat was used to scoop up a known volume of sediment (about 1000 pounds), and then the rototiller was used to blend the modifiers into the sediment. The individual modifiers were weighed using an electronic scale. The batches of air-dried sediment and modifiers were stored under the tent.

North American Manufacturing Co., Ltd. dispatched their service technician to the plant to tune the new burner system and check the operation of the other existing burners. Mr. Tamas Nemeth spent four days at the plant site (July 7-10, 2004) troubleshooting and tuning the burners. He found several items that needed to be rewired and control piping that needed to be installed. His report (included in Appendix B) indicated that the natural gas supply to the original melt burners was restricted by an undersized regulator. This meant that these burners could only be fired at about 240,000 Btu per hour instead of the design rate of 500,000 Btu/hr each. To overcome this limiting restriction, RPMS installed a manual bypass around the limiting regulator.

Trace Environmental Systems (TES) set up and confirmed the operation of the Continuous Emissions Monitoring System (CEMS). The original CO analyzer did not function properly and was removed and taken back to TES's shop for evaluation. In the meantime, GTI rented a CO analyzer from Clean Air Rentals (Palatine, IL). On July 13 TES's service technician returned to the plant to install and calibrate the rented CO monitor.

The emergency diesel generator was reconnected to the plant power system and successfully tested. It had been disconnected and removed after the December 2003 campaign.

The rotary feeder on the baghouse had rusted solid and could not be freed up. FMW removed the rotary feeder and replaced it with another 6-inch rotary feeder borrowed from the Ecomelt dryer baghouse that was not being used.

On July 13, we began calibrating the four augers in the main sediment hopper (T-101). The augers are driven by new variable-speed motors that can be adjusted to deliver the desired amount of material to the main sediment conveyor (C-101). C-101 is operated at a constant speed to deliver the feed material to the weigh conveyor (C-112) and then to the pug mill (M-131). Two loads of pre-blended feedstock were charged to the main sediment storage hopper (T-101) for the calibrations. During the calibration, it was observed that the four augers were not turning in the proper direction to propel the feed material toward the discharge end. This wiring problem was subsequently corrected by SM Electric.

The pre-blended feed material could be conveyed much more readily than the raw, wet sediment tested in December 2003. The calibration showed that at 33 percent (20 Hz), the nominal feed rate was 330 pounds per hour. At 100 percent (60 Hz), the nominal feed rate averaged about 1500 pounds per hour.

To keep rainwater out of the main sediment hopper (T-101), water-proof tarps were draped over the hopper over a central pipe in a tent-like structure. The tarps are held in place by guy ropes. When material is charged to the hopper, the tarps are pulled back.

The operators devised a high-pressure air lance to disrupt "rat-holing" and bridging in the T-101 sediment hopper. This was to facilitate consistent feeding from the hopper. The operator directed the high-pressure air stream at material in the hopper that had bridged.

Refractory curing was initiated at a low heat-up rate for the new refractory "lip" and new refractory around the new Hot Spot and Tempest burners. The curing rate was specified by Harbison-Walker Refractories Co. The primary burner on low fire was used for this operation.

#### Initial Start-Up Test #1 (July 16, 2004)

The objective of this test was to operate the Cement-Lock demo plant with the newly installed process enhancements and equipment retrofits in place and begin processing the pre-blended sediment-modifiers mixture.

Prior to test initiation, the temperatures in the rotary kiln (TIC-201) and secondary combustion chamber (SCC) (TIC-202) were measured at 2400° and 2250°F, respectively.

Temperatures of the refractory lining at various locations in the rotary kiln and drop-out box were also measured by hand-held pyrometer. These temperature readings showed that the fire bricks near the rotary kiln discharge were about 150°F higher than the reading indicated by TIC-201, which was measured by a thermocouple (TE-201) located on the ceiling of the drop-out box. The refractory "lip" in the drop-out box was about 100°F hotter than the TE-203 readings measured by a thermocouple located on the west wall of the drop-out box.

The flow of natural gas to the primary burner averaged 13,338 SCFH (or about 13.3 million Btu/hr). The flow of combustion air averaged 123,983 SCFH. The SCC burner was not fired during the test. As the temperature of the SCC was significantly higher than the minimum permitted level of 2100°F, it was not considered necessary.

The water flow to the granulator sprays was reduced from 55 to 40 gpm and the flow of water to the weir was reduced to 60 gpm. As a result of these flow reductions, the dropout box temperature (TIC-203) increased from 1780° to 1845°F.

The test was initiated at 10:00 am when the four augers in T-101 were started at 67 percent (40 Hz) or a nominal feed rate of about 1000 pounds per hour.

During the initial part of the test, the oxygen concentration in the flue gas exiting the system through the vent stack averaged 7 mole percent (dry basis) By the end of the test, the  $O_2$  concentration had been brought down to about 5.5 mole percent by adjusting excess air. The carbon monoxide concentration in the flue gas averaged 2.8 ppm during the initial part of the test and then increased to about 3.5 ppm for the remainder of the test.

As expected, it took several hours of feeding before molten slag completely coated the surface of the rotary kiln and Ecomelt<sup>®</sup> began to flow out of the Ecomelt granulator. The Ecomelt was black and granular with numerous -¼-inch spherical pieces. It resembled the Ecomelt generated during the pilot test at Hazen Research. Ecomelt was generated

during the test until the upset described below. Bulk samples of Ecomelt from this test are being stored on-site in 55-gallon drums.

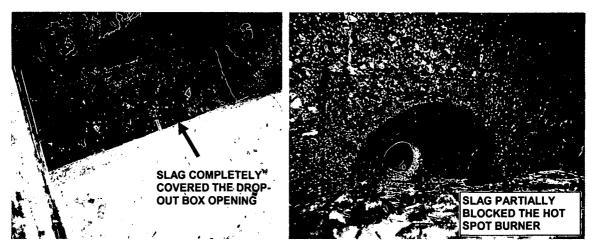
During the test, slag "rain" was observed to continuously fall from the ceiling of the kiln as it rotated. The presence of "rain" indicates that the viscosity of the slag is too high. Under the desired conditions, molten slag would form a rivulet flowing down the middle of the kiln floor. Slag "rain" was also observed during the December 2003 initial start-up test.

At about 2:30 pm, we increased the kiln temperature to 2450°F. Also, we took a feed rate measurement using the newly installed chute in C-101. The feed rate was determined to be about 1800 pounds per hour. The operator's air-lancing technique in the feed hopper was more efficient than expected. The auger speed rate was reduced to compensate.

Within about 15 minutes, the drop-out box temperature (TIC-203) began to increase. After about one hour (3:30 pm), the drop-out box temperature had reached 2200°F. Achieving a higher drop-out box temperature was one of the test objectives; however, it appeared to coincide with the accumulation of slag in the drop-out box.

At about 4:30 pm, we experienced a jam in the conveyor (C-205) that feeds the Ecomelt dryer as oversized slag material was being brought up by the Ecomelt granulator conveyor (C-203). As the conveyor jammed, the drive belt connecting its motor to the C-205 conveyor failed. Still hot slag material had to be extracted manually from the 6-inch diameter sample port on the C-205 granulator conveyor.

Feed was discontinued shortly thereafter (at 4:30 pm). Kiln rotation was reduced from 0.4 to 0.3 rpm to reduce the flow rate of slag to the drop-out box. The melt burners were fired continuously and the temperature of the drop-out box increased from 2200° to over 2600°F by 7:30 pm. The kiln temperature was maintained overnight at 2450°F in hope that the blockage would clear; however, it did not. On July 17, the test was terminated and the temperature in the rotary kiln was brought down per the prescribed rate of about 100°F per hour.

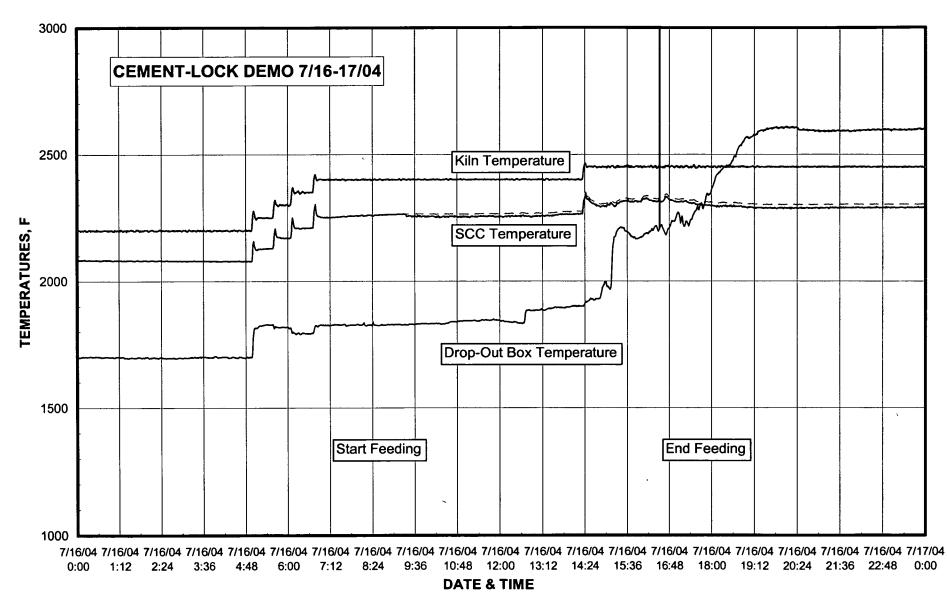


On July 20, the system had cooled sufficiently for personnel to enter and make a photographic record of the slag accumulation in the drop-out box. The photo (above left) shows that the top level of the slag was uniformly flat. It had completely covered over the granulator opening just below the new Hot Spot melt burners. The photo (above right) shows slag had begun to fill the nozzle of the Hot Spot burner under the new refractory "lip". As mentioned above, the drop-out box had been heated by the melt burners overnight at about 2600°F and the slag had flowed to a uniformly flat level. Thin layers of slag also flaked from the walls and kiln as shown in the photo.

Operating staff entered the drop-out box with a jackhammer and began chipping away at the slag. Within two hours, the slag had been completely broken up. Shards of slag were removed from the granulator through the sample port at the entrance to the C-205 conveyor. Later, to facilitate removal of any oversize slag in future tests, FMW installed an access/clean out port on the upper end of the C-203 conveyor. Figure 2 shows the time temperature history of the initial start-up of the Cement-Lock demo plant from July 16 to 17, 2004 with retrofits and enhancements in place.

#### Start-Up Test #2 (July 21, 2004)

The objective of this test was to operate the Cement-Lock demo plant at conditions that would minimize slag accumulation including increasing the temperature of the drop-out box and adding additional modifiers and flux to the feed. The combined effects were to improve slag fluidity.



•

٠

5

.

Figure 2. TIME-TEMPERATURE HISTORY OF THE INITIAL START-UP OF THE CEMENT-LOCK DEMO PLANT WITH RETROFITS AND ENHANCEMENTS (July 16-17, 2004)

Prior to test initiation, the temperatures in the rotary kiln (TIC-201) and secondary combustion chamber (SCC) (TIC-202) were measured at 2450° and 2285°F, respectively. The flow of natural gas to the primary burner averaged 14,513 SCFH (or about 14.5 million Btu/hr). The flow of combustion air averaged 142,446 SCFH. The SCC burner was also not fired during this test. As the temperature of the SCC was significantly higher than the minimum permitted level of 2100°F, it was not considered necessary.

The water flow to the granulator sprays was reduced from 40 to 8 gpm and the flow of water to the weir was reduced from 60 to 15 gpm. As a result of these flow reductions, the drop-out box temperature (TIC-203) increased from 1845° to 2210°F.

This test was initiated at 3:30 pm when the four augers in T-101 were started at 33 percent (20 Hz) or a nominal feed rate of about 500 pounds per hour. Additional modifier (limestone) and flux (fluorspar  $- CaF_2$ ) were added to the feed material to decrease its viscosity when molten.

During the test, the oxygen concentration in the flue gas exiting the system through the vent stack averaged 7 mole percent (dry basis). The carbon monoxide concentration in the flue gas averaged 3.0 ppm during the test.

After several hours of feeding, Ecomelt began to flow out of the Ecomelt granulator. Similar to the first test, the Ecomelt was black and granular with numerous minus <sup>1</sup>/<sub>4</sub>-inch spherical pieces. Ecomelt was generated during the test until the upset described below. Bulk samples of Ecomelt from this test are being stored on-site in 55-gallon drums.

Slag "rain" was also observed to fall from the ceiling of the kiln as it rotated during this test.

We increased the kiln temperature (TIC-201) to 2475°F at about 6:15 pm and then to 2500°F at about 7:00 pm. The objective of these operating changes was to decrease the slag viscosity in the kiln. Over the same time period, the drop-out box temperature (TIC-203) increased from about 2235° to about 2275°F.

At about 7:10 pm, we experienced a jam in the granulator conveyor (C-205) as oversized slag material was being brought up the granulator conveyor. The C-205 conveyor was halted. Hot slag had to be extracted manually from the port cut in the C-203 granulator conveyor plenum. Feed was discontinued shortly thereafter (about 7:15 pm).

At about 8:30 pm, we increased the kiln temperature to 2550°F in an attempt to disrupt the drop-out box blockage; however, there was no effect. By 9:15 pm, the drop-out box temperature had reached 2550°F and we brought the kiln temperature down to 2475°F. These conditions were held constant overnight.

The next morning (July 23), the temperature of the drop-out box had risen to 2620°F and the granulator water temperature had cooled to 180°F. In an effort to break the slag blockage, we momentarily opened the weir and spray water valves full in an attempt to fracture the blockage with cold water/steam impingement. There was no apparent effect of this action and the valves were closed. It was decided to terminate the test so that the slag in the drop-out box could be cleared.

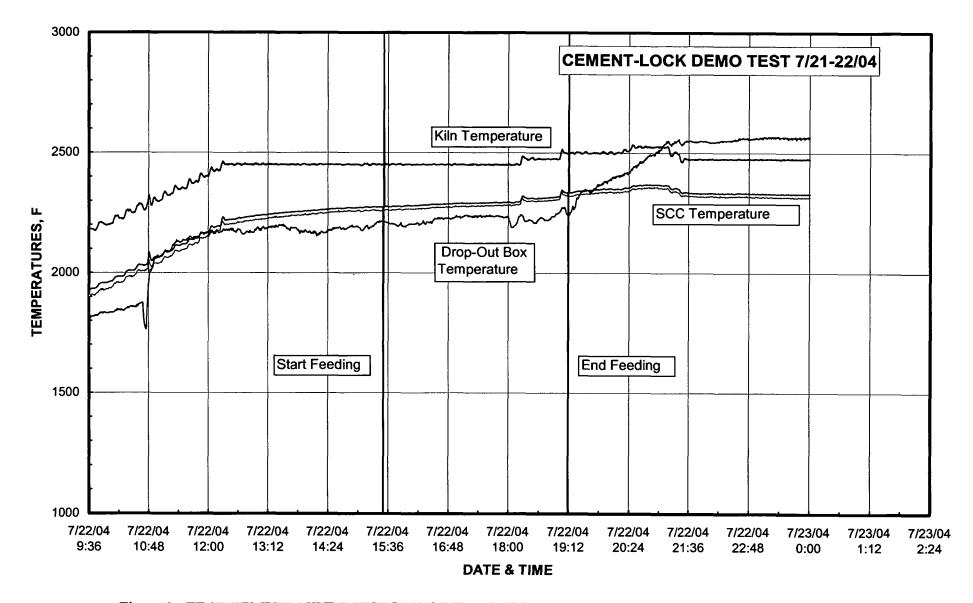
Figure 3 shows the time temperature history of the second start-up test of the Cement-Lock demo plant on July 21 to 22, 2004 with retrofits and enhancements in place.

#### **Post-Test Evaluation**

The system was cooled per the prescribed procedure. On July 26, the slag blocking the exit was removed using the jackhammer as before. Before the slag was broken, two adjacent 1-foot diameter holes were observed in the slag covering the drop-out box. These could have resulted from the water weir/spray action described above.

During an inspection of the rotary kiln refractory, we noticed that part of the cast refractory of the kiln "nose" at the discharge end of the kiln had cracked and spalled. The refractory contractor was brought back on-site to repair the damaged area and to touch up" two other problem areas in the kiln. That work was completed.

Even though process operating conditions were adjusted from the first test to minimize slag accumulation, the second was terminated for essentially the same reason – the dropout box was too cool to allow the molten slag to readily flow into the granulator. Merely



•

Figure 3. TIME-TEMPERATURE HISTORY OF THE SECOND START-UP TEST OF THE CEMENT-LOCK DEMO PLANT WITH RETROFITS AND ENHANCEMENTS (July 21-22, 2004)

changing the operating conditions (e.g., increasing the kiln temperature or adding more flux) would not provide a positive means to clear accumulating slag and ensure sustained operation. Therefore, we decided that before we conducted another test, a positive, mechanical means of breaking slag accumulating in the drop-out box was needed. Work planned for August 2004 focused on mechanical and other operating changes that could further improve plant operations.

Overall, the work performed above represents the initial start-up of the Cement-Lock demonstration plant with retrofits and enhancements in place. Also, as described above, several additional mechanical changes are necessary to ensure sustained operation of the Cement-Lock demo plant in subsequent testing.