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Utilization of Lightweight Materials Made from Coal Gasification Slags

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1.0 PROJECT OBJECTIVES, SCOPE AND DESCRIPTION OF TASKS

1.1 Introduction

The integrated-gasification combined-cycle (IGCC) process is an emerging technology that utilizes coal for power generation and production of chemical feedstocks. However, the process generates large amounts of solid waste, consisting of vitrified ash (slag) and some unconverted carbon. In previous projects, Praxis investigated the utilization of "as-generated" slags for a wide variety of applications in road construction, cement and concrete production, agricultural applications, and as a landfill material. From these studies, we found that it would be extremely difficult for "as-generated" slag to find large-scale acceptance in the marketplace even at no cost because the materials it could replace were abundantly available at very low cost. It was further determined that the unconverted carbon, or char, in the slag is detrimental to its utilization as sand or fine aggregate. It became apparent that a more promising approach would be to develop a variety of value-added products from slag that meet specific industry requirements. This approach was made feasible by the discovery that slag undergoes expansion and forms a lightweight material when subjected to controlled heating in a kiln at temperatures between 1400 and 1700°F. These results confirmed the potential for using expanded slag as a substitute for conventional lightweight aggregates (LWA). The technology to produce lightweight and ultra-lightweight aggregates (ULWA) from slag was subsequently developed by Praxis with funding from the Electric Power Research Institute (EPRI), Illinois Clean Coal Institute (ICCI), and internal resources.

The major objectives of the subject project are to demonstrate the technical and economic viability of commercial production of LWA and ULWA from slag and to test the suitability of these aggregates for various applications. The project goals are to be accomplished in two phases: Phase I, comprising the production of LWA and ULWA from slag at the large pilot scale, and Phase II, which involves commercial evaluation of these aggregates in a number of applications.

Primary funding for the project is provided by DOE's Federal Energy Technology Center (FETC) at Morgantown, with significant cost sharing by Electric Power Research Institute (EPRI) and Illinois Clean Coal Institute (ICCI).

1.2 Scope of Work

The Phase I scope consisted of collecting a 20-ton sample of slag (primary slag), processing it for char removal, and pyroprocessing it to produce expanded slag aggregates of various size gradations and unit weights, ranging from 12 to 50 lb/ft^3. In Phase II, the expanded slag aggregates are being tested for their suitability in manufacturing precast concrete products (e.g., masonry blocks and roof tiles) and insulating concrete, first at the laboratory scale and subsequently in commercial manufacturing plants. These products will be evaluated using ASTM and industry test methods. Technical data generated during production and testing of the products will be used to assess the overall technical viability of expanded slag production. Relevant cost data for physical and pyroprocessing of slag to produce expanded slag aggregates will be gathered for comparison with (i) the management and disposal costs for slag or similar wastes and (ii) production costs for conventional materials which the slag aggregates would replace. In addition, a market assessment will be made to evaluate the economic viability of these utilization technologies.

1.3 Phase I Task Description

A summary of the tasks performed in Phase I is given below:

Task 1.1 **Laboratory and Economic Analysis Plan Development**: Development of a detailed work plan for Phase I and an outline of the Phase II work.

Task 1.2 **Production of Lightweight Aggregates from Slag**: This task covered selection and procurement of project slag samples, slag preparation including screening and char removal, and slag expansion in a direct-fired kiln and fluid bed expander. The char recovered from the slag preparation operation was evaluated for use as a kiln fuel and gasifier feed. Environmental data for slag-based lightweight aggregate (SLA) production was collected.

Task 1.3 **Data Analysis of Slag Preparation and Expansion**: Analysis and interpretation of project data including development of material and energy balances for slag processing and product evaluation.

Task 1.4 **Economic Analysis of Expanded Slag Production**: Economic analysis of the utilization of expanded slag was conducted by determining production costs for slag-based LWAs and ULWAs. Expanded slag production costs were compared with the market value of similar products both with and without taking into account the avoided costs of disposal and with the costs of management of slag as a solid waste.

Task 1.5 **Topical and Other Reports:** Preparation of topical, financial status, and technical progress reports in accordance with the Statement of Work.

1.4 Phase II Task Description

A summary of the tasks to be performed in Phase II is given below.

Task 2.1 **Test Plan for Applications of Expanded Slags (Field Studies):** This task involves the development of selection criteria and a field test plan for applications of expanded slag. This plan will serve as a guide in the selection and implementation of field demonstrations for the most promising expanded slag utilization applications. Field applications will be selected on the basis of laboratory results, the marketability of the products, and the suitability of the project slags for producing them. The following applications are under consideration for testing:

- Lightweight concrete blocks made from 50 lb/ft^3 SLA
- Lightweight roof tiles made from 40 lb/ft^3 SLA
- Loose fill insulation made from 16 lb/ft^3 SLA
- Lightweight insulating concrete made from 16 lb/ft^3 SLA

Task 2.2 **Field Studies to Test Expanded Slag Utilization**: Under this task, field testing of the applications identified in Phase II, Task 2.1, will begin with test work to optimize the concrete mixes made from expanded slag.

Task 2.3 **Data Analysis of Commercial Utilization of Expanded Slags**: The objective of this task is to assimilate the data and test results collected during Phase II, Task 2.2, convert these findings to common engineering terms, and correlate these results with comparable information for conventional lightweight aggregates as reported in the literature. The data analysis will provide specific answers to the following issues:

- Performance of expanded slag vs. that of conventional materials
- Technical viability of lightweight and ultra-lightweight slags as aggregates.

Task 2.4 **Economic Analysis of Expanded Slag Utilization**: The objective of this task is to expand upon the preliminary economic assessment of expanded slag utilization conducted during Phase I. The economics will be studied based on the production costs for SLA in comparison with current market prices for conventional materials. During the Phase I preliminary evaluation, two production scenarios emerged:

- Production of SLA at the gasifier location (on-site production)
- Production of SLA at a lightweight aggregate facility (off-site production).

The impact of the avoided costs of slag disposal on the economics of SLA production will also be evaluated. Slag utilization data and product samples will be made available to commercial lightweight aggregate users for validation of estimated market prices. The impact of SLA market prices on the economics of SLA production will also be studied.

Task 2.5 **Final Report:** The data generated and collected during the project will be compiled in a final report to be submitted at the end of the project that will be a comprehensive description of the results achieved, consistent with the Reporting Requirements. Data from topical and field reports will be summarized. The report will include the original hypothesis of the project and present the investigative approaches used, complete with problems encountered or departures from the planned methodology, and an assessment of their impact on the project results.

1.5 Scope of This Document

This is the eighteenth quarterly report and summarizes the work undertaken during the performance period between 1 December 1998 and 28 February 1999. It is the eleventh quarterly report for Phase II. This document summarizes the Phase II accomplishments to date along with the major accomplishments from Phase I.

2.0 SUMMARY OF WORK DONE DURING THIS REPORTING PERIOD

2.1 Summary of Major Accomplishments

In the current reporting period, work focused on preparation of a test plan for pilot-scale testing of the use of slag as a kiln feed additive in the production of Portland cement. In addition, work continued on data consolidation, analysis of test results, and preparation of the final report.

2.2 Chronological Listing of Significant Events in This Quarter

The following significant events occurred during the current reporting period:

Date	Description
2/1/00	Final report writing was continued
3/31/00	Laboratory testing of using slag as cement feed additive was completed
3/31/00	Test plan completed for pilot testing of slag use in Portland cement kiln feed

3.0 TO DATE ACCOMPLISHMENTS

A summary of the work completed in Phase I is given below.

Date	Phase I Accomplishments Description
10/24/94	Draft Laboratory and Economic Analysis Plan (project work plan) submitted
11/18/94	Slag char removal completed on the advance sample and prepared slag laboratory expansion testing initiated
12/02/94	Final "Laboratory and Economic Analysis Plan" prepared and submitted
05/21/95	Primary slag sample (20 ton) received at Penn State for preparation
06/01/95	Pilot unit for char removal set up and processing work started
08/20/95	Primary slag sample processing for char removal completed
9/10/95	Laboratory expansion studies of slag and slag/clay blends started
10/15/95	1-ft and 3-ft diameter kilns commissioned for pilot testing
11/15/95	Pilot testing of Slag I and Slag II and pellets in 3-ft dia. direct-fired kiln completed
11/17/95	Pilot testing using fluidized bed expander completed
12/12/95	SLA product characterization initiated
1/20/96	Laboratories for testing of SLA products identified
2/16/96	Test plan for second batch of fluid bed expander testing at Fuller completed

A summary of the work completed in Phase II to date is given below.

Date	Phase II Accomplishments Description
4/30/96	Application for continuation of the project to Phase II submitted
5/31/96	Phase I Final Report (draft) submitted to METC
7/1/96	Phase I Topical Report (final version) submitted
7/14/96	Approval for continuation of the project to Phase II was received from METC
7/14/96	Structural concrete laboratory tests started
7/15/96	Lab testing of SLA for roof tile and insulating concrete applications completed
7/15/96	Evaluation of SLA for completed
7/30/96	Evaluation of SLA for loose fill insulation completed
10/10/96	Mix designs for block production selected
11/10/96	Laboratory evaluation of the Slag II completed
10/30/96	Structural concrete laboratory tests completed
11/10/96	Laboratory evaluation of Slag III for LWA production completed
1/10/97	Laboratory testing of SLA for structural concrete application completed
2/19/97	First batch of laboratory tests of selected block mixes completed
4/30/97	Processing of Slag III for char removal completed
5/20/97	Preparatory work for Slag III pyroprocessing completed
7/30/97	Preparation of Slag III for SLA production completed
8/20/97	Utilization of SLA as growing medium for potted plants completed successfully
10/2/97	Exploratory testing of expanded slag for nursery applications completed
10/2/97	Laboratory testing of expanded Slag I for block application completed
11/1/97	Testing of expanded slag in panel application completed
12/2/97	Second phase of testing of expanded slag for nursery applications started
1/20/98	Evaluation of expanded Slag III for block and roof tile applications initiated
2/15/98	2^{nd} round evaluation of expanded Slag III in panel application completed by end user
3/12/98	Testing of Slag IV included in the current project
3/23/98	Roof tile evaluation of expanded slag/clay 50/50 blends was reconfirmed
4/25/98	Commercial production and economic evaluation of expanded slag in panel application was initiated per user request
6/12/98	Production of a large batch of blocks using expanded slag successfully completed
7/12/98	Testing of Slag IV for char removal completed in the current period
7/20/98	Testing for optimization of the strength for roof tile application conducted
8/15/98	Laboratory testing initiated for concrete panels flexural strength development
9/30/98	Slag/char separation vendor testing and data analysis for Slag IV completed

10/15/98	Testing of Slag IV for lightweight aggregate production completed
10/24/98	Presentations on slag/char separation process to the two DOE-funded IGCC facilities completed
11/20/98	Applications-oriented testing of Slag IV completed
2/20/99	Disposal of surplus slag samples was started
5/1/99	Testing of slag as cement kiln feed was started
7/1/99	Laboratory evaluation of slag as cement kiln feed was continued
9/1/99	Data consolidation and analysis was started in support of the final report
10/1/99	Final report writing was started
3/31/00	Laboratory testing of using slag as cement feed additive was completed
3/31/00	Test plan completed for pilot testing of slag use in Portland cement kiln feed

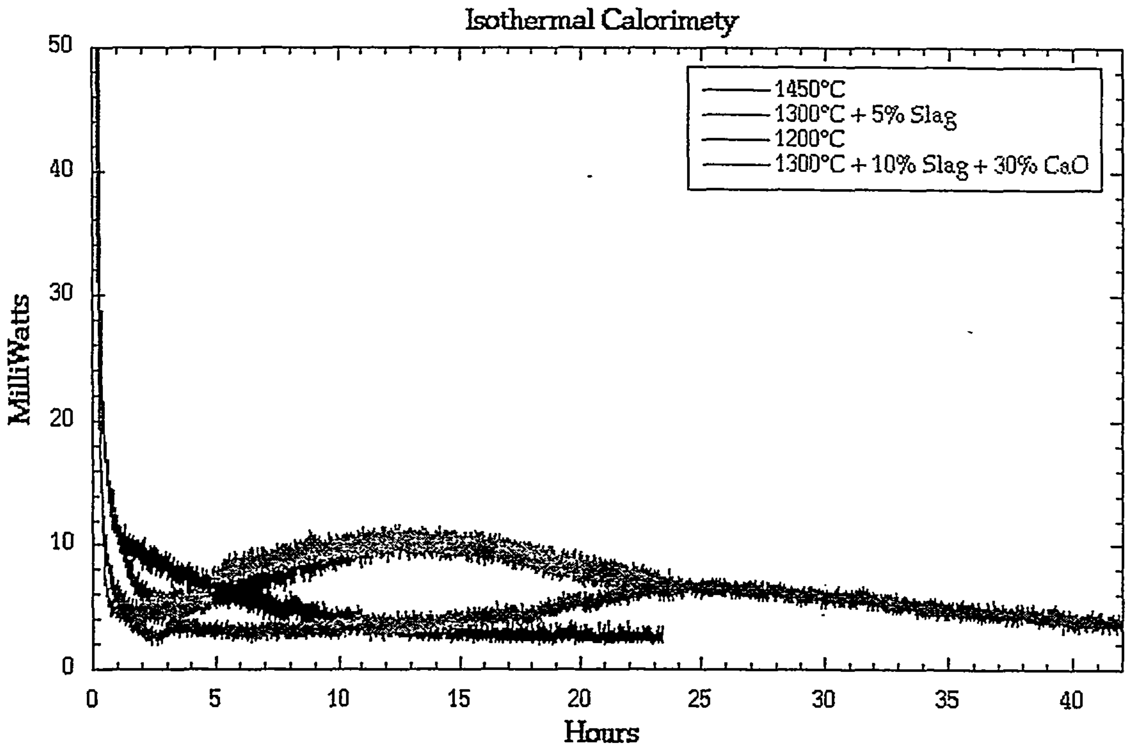
4.0 TECHNICAL PROGRESS REPORT

4.1 Slag as Raw Material Additive in Portland Cement (Laboratory Testing)

Within this last quarter, efforts were continued to maximize the amount of slag incorporation into the cement kiln feed while minimizing energy consumption, at a laboratory scale. With the addition of the specially prepared slag to the conventional cement raw feed, the overall chemistry of the kiln feed changes. Predictions can be made using ASTM C150 based on the chemistry of the various ingredients in the kiln feed. The addition of ground slag, which is almost entirely an aluminosilicate, the expected calcium silicates in the clinker decreases dramatically. In order to compensate for the addition of the slag, limestone was added to increase the amount of expected C_3S back to about 63%. A range of temperature and heating profile was studied in order to minimize the heat required for proper clinkering. Successful samples were subjected to x-ray diffraction analysis in order to examine the various crystalline phases that were formed in the sintering process. In addition, promising samples were crushed and studied using isothermal calorimetry to examine their hydraulic property and reactivity. The samples were clinkered in a high temperature molybdenum furnace at various temperatures using a platinum crucible. The temperature profiled used was:

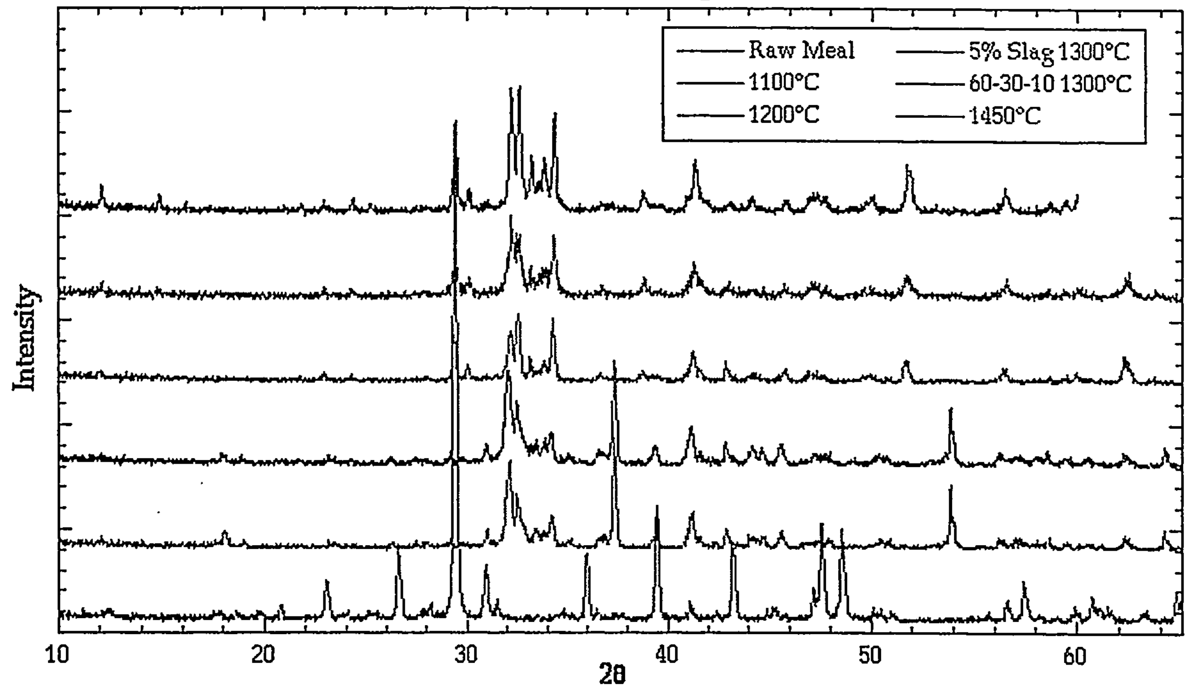
Ramp from Room Temperature to 900°C in 4 hours
Hold at 900°C and calcine for 4 hours
Ramp further from 900°C to the *clinkering temperature* in 2 hours
Sinter at *clinkering temperature* for 4 hours
Air quench *cooling phase from clinkering temperature* to the Room Temperature

The clinkering temperatures used for this study were 1100°C, 1200°C, 1300°C and 1450°C. In comparison, the normal clinkering temperature in a cement kiln is about 1450°C. Shown in the following figure is the isothermal calorimetry of the select samples.



Isothermal calorimetry of the samples was conducted at 25°C. First each sample clinker was ground and passed through 200 mesh. 3 grams of material and 3 grams of deionized water were allowed to equilibrate inside the calorimeter separately. The sample was then hydrated with the water (w/s=1) within the calorimeter after equilibrium has been reached. Data were collected every 10 seconds until the hydration reaction ceased. Hydration reaction liberates heat and can be detected in very minute amount. The heat liberated (or absorbed if the reaction is endothermic) is recorded with time. The resultant heat evolution curve is indicative of the reactivity of the sample or in the case cement clinker, the relative hydraulic activity. Tests were conducted only on the clinkered samples and not on the raw feed. As can be seen in this figure, typical of all hydration experiments, all the curves have an initial wetting peak at very early stages (within the first few minutes). At longer time, the sample that was clinkered at 1200°C showed no reaction peak whereas the 1450°C sample and the 1300°C (with 5% slag) showed a very pronounced reaction peak beginning at about 5 hours, lasting through almost 24 hours with the maximum at about 14 hours. Both the 1300°C curve and the 1450°C curve were very similar with the 1450°C curve having a slightly broader wetting curve. The 60-30-10 sample (60% raw feed, 30% limestone, and 10% ground slag) differs in that it has a much broader reaction peak centered at about 25 hours. The hydration reaction begins at about 10 hours after initial surface wetting and lasts through 40 hours. This is not unexpected given that the overall chemistry of the 60-30-10 mix results in a much higher calculated C_2S content using the Bode equation as given in ASTM C150. Test were done at lower clinkering temperature with 10% slag but the resultant samples did not sinter properly and therefore the results not shown here. X-ray diffraction pattern of these samples are shown in the following figure.

X-ray Diffraction of Slag-Clinker



The x-ray diffraction pattern of the raw meal shows mostly diffraction peaks from calcium carbonate. With clinkering temperature of 1100°C and 1200°C, the samples were not acceptable in that no sintering reaction occurred. The pronounced sharp peaks at 37.5° and 54° are due to lime, resulting from the liberation of CO_2 from the limestone with heating. These samples had no hydraulic property. Increasing the clinkering temperature to 1450°C, where most commercial cement kilns operate, the samples exhibited normal cement properties. The x-ray diffraction pattern of this sample show characteristic diffraction peaks from constituent cement phases. This x-ray pattern is very different from the poor samples clinkered at 1100°C and 1200°C. With the addition of 5% ground slag, the samples made at 1300°C had a similar diffraction pattern to the plain cement clinker. Similarly, the 60-30-10 sample (60% raw meal, 30% limestone, and 10% ground slag) has a x-ray pattern very similar to the plain cement clinker sample.

Summary of Laboratory Tests

X-ray diffraction and isothermal calorimetry were used to study the characteristics of the raw cement kiln feed and clinkers made at 1100°C, 1200°C, 1300°C and 1450°C. 5% and 10% ground slag were added to the raw feed and clinkered at 1300°C. Limestone was added to the sample containing 10% ground slag to adjust the bulk chemistry. Current data suggests that 5% and 10% addition of ground slag can produce a clinker at 1300°C with similar hydration characteristics as a clinker produced at 1450°C without added slag. However, with high dosage of ground slag, the clinker exhibited slower reactivity and this may be due to a higher C_2S content. This suggests a significant heat energy saving of 150°C, which would translate to lower heating cost or higher kiln capacity. In addition, the slag can be used successfully as an aluminosilicate source for cement clinker.

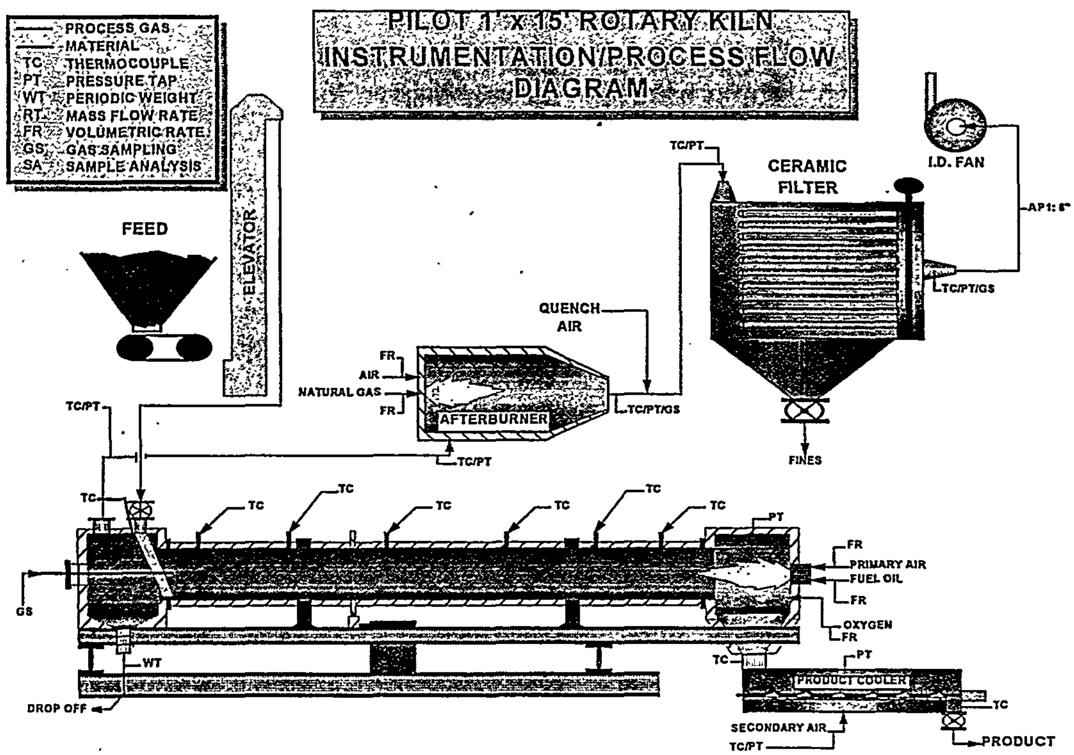
4.2 Slag as Raw Material Additive in Portland Cement Kiln Feed (Pilot Testing)

During the current reporting period, pilot-scale testing of the utilization of slag as a raw material additive in Portland Cement was added to the project scope. A test plan was developed, and arrangements were made to select the necessary equipment and prepare the materials required. The test work has been scheduled for May 2000. The activities planned are presented below.

The objectives of the pilot-scale test work are to:

1. Produce cement clinker from a raw meal containing gasifier slag and determine the effects of the slag addition on kiln operating parameters
2. Produce a sufficient quantity of slag cement to facilitate cement analysis and evaluation.

A pilot plant consisting of a 1' by 15' rotary kiln, capable of operating at a feed rate of 20-25 lb/hour, has been commissioned at the Fuller Company test site which has been used throughout this project. A schematic of the pilot plant is shown in Figure below.



Pilot Rotary Kiln

Pilot Testing Schedule

The following schedule has been established for the pilot test work:

Week 1/2: Finalize test outline.
Schedule rotary kiln test program.

Week 2: Prepare 250 lb of extrusions from each of the following feed samples:
Feed Sample #1: Type I/II Cement raw meal (59 C_3S)
Feed Sample #2: 54% cement raw meal + 5% slag + 36.5% limestone + 4.5% sand
Feed Sample #3: 10.5% cement raw meal + 10% slag + 71% limestone + 8.5% sand
Feed Sample #4: 97% cement raw meal + 3% slag (fluxing quality to boost the C_3S content and lower the heat content)

Week 3: Setup rotary kiln system.

Week 4: Perform rotary kiln test program.
Ship clinker samples to Penn State laboratory.
Begin test report.

Weeks 4-8: Complete and issue a complete report, summarizing test results.

Preparation of Feed Materials

The following methodology has been established for the preparation of feed materials for the test work:

1. Grind samples of gasifier slag (950931), limestone (960917) and sand (200016) to 80% passing 200 mesh.
2. Calculate the quantity of limestone that must be added to feed samples #2 and #3 to obtain a potential C_3S level of 59 and a burnability index of 3.
3. Blend ground materials with cement raw meal to produce 250-300 lb each of feed samples #2, #3 and #4.
4. Extrude each of the four feed samples to produce ½" diameter x 1" long extrusions. Produce 250-300 lb of feed samples #2, #3 and #4. Produce 500 lb of feed sample #1. Air dry pellets to reduce the free moisture level to <5%.
5. Place dry extrusions in sealed drums with appropriate labels.

Pilot Kiln Operating Parameters and Test Procedure

1.	Process Temperature:	1400°C+
2.	Kiln Speed:	0.55 rpm
3.	Material Retention Time:	2 hours
4.	Cooler/Kiln Total Air Input:	50-60 scfm
5.	Oxygen Injection:	As required to support combustion
6.	Kiln Exit $\%O_2$:	2-3%
7.	Kiln Fuel:	#2 Fuel Oil
8.	Feed Rate:	25 lb/hr

The following test procedure will be followed:

1. Start Datavue trend screens (3 trend files). Calibrate gas analyzers and place online.
2. Start all valves and ceramic filter pulse. Confirm valve operation.
3. Check and zero all manometers.
4. Start kiln drive and set shell speed to 0.55 rpm.
5. Set the cooler air input to 50 scfm and kiln burner atomizing airflow to 10 scfm, and then light the kiln burner. Slowly increase the firing zone temperature to 1400°C, and then hold this temperature for several hours to preheat the kiln lining. Add oxygen as required to support combustion. Adjust the I.D. fan to maintain a neutral pressure in the firing hood.
6. Charge the belt feeder with 25 lb of feed sample #1. Start feed to the kiln system and set the rate to 25 lb/hr.
7. Monitor the firing zone. If no sticking is observed, increase the hot zone temperature to 1450°C. Begin collecting clinker samples every 30 minutes from the cooler bypass port and submit to the chemical lab for free lime analysis. Adjust the hot zone temperature as required to obtain a clinker free lime level in the range of 1-1.5%.
8. Stabilize operating conditions and begin recording operating data every 60 minutes and enter into the process data spreadsheet. Print the Datavue screen every 60 minutes (screen print/open in Paint/save).

9a. Collect cooler bypass clinker samples every 30 minutes (or as requested). Cool sample and place in bag with label.

9b. Collect one composite feed sample per shift. Place sample in bag with label.

10a. Record net weight of each feed container (suggested net weight: 25 lb) and the time it was added on datasheet. Add feed to the feed system hopper when the inventory reaches a specified level.

10b. Record net weight of clinker cooler discharge container and time removed on datasheet every 60 minutes. Allow container to cool and bag with label.

10c. Measure drop off net weight on an hourly basis, record on datasheet and place in bag with label.

10d. Measure ceramic filter net weight on an hourly basis, record on datasheet and place in bag with label.

11. Observe rotary kiln operating conditions and note on log sheet.
12. Measure and record system shell temperatures using the infrared unit prior to the conclusion of each test phase.
13. Produce a minimum of 100 lb of clinker from Feed Sample #1.
14. Empty the feed system and charge with Feed Sample #2. Begin feeding at a rate of 25 lb/hr. Adjust the kiln hot zone temperature as required to satisfy the clinker free lime specification of 1-1.5%. Stabilize operation, record operating data and sample as outlined above.
15. Proceed to process Feed Sample #3, followed by Feed Sample #4.
16. Shut down system and inspect. Open clinker cooler, collect inventory and weigh. Prepare clinker samples for shipment to Penn State laboratory.
17. Grind the final products to meet specifications for Type I and Type II Portland cements.

Sample Analysis
The following analyses are planned for the product samples:

1. Selected composite feed samples will be analyzed for moisture, sizing, bulk density and complete chemistry.

2. Selected cooler bypass clinker samples will be analyzed for free lime.

3. Acceptable composite product samples of the clinker cooler discharge will be analyzed for sizing, bulk density, complete chemistry and free lime.

4. No analysis of fines is planned.

4.3 Preparation of Final Report

Work was continued on data analysis and preparation of the project final technical report.

5.0 PLAN FOR THE NEXT QUARTER

The following activities are planned for the next quarter:

- Complete pilot-scale testing of slag as a kiln feed additive in making Portland cement
- Continue data evaluation and preparation of the final technical report.