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/ft³. 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/ft³ SLA
 - Lightweight roof tiles made from 40 lb/ft³ SLA
 - Loose fill insulation made from 16 lb/ft³ SLA
 - Lightweight insulating concrete made from 16 lb/ft³ 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 data consolidation and analysis of test results and preparation of the final report. Also, work was started on the testing of slag as raw material in cement production. The following was accomplished during this reporting period:

- Test planning and raw materials procurement for cement application of slag
- Analysis of the project test work results to consolidate the information to cover broad applications for slag lightweight aggregates that can be commercially developed.

- Work was initiated on preparation of the project final technical report
- Disposal of surplus samples of slag and products

2.2 Chronological Listing of Significant Events in This Quarter

The following significant events occurred during the current reporting period:

Date	Description
6/1/99	Data consolidation and analysis was started in support of the final report
6/1/99	Final report writing was continued
7/1/99	Laboratory evaluation of slag as cement kiln feed was continued

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
12/20/98	Data consolidation and analysis was started in support of the final report
1/26/99	Final report writing was started
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

4.0 TECHNICAL PROGRESS REPORT

4.1 Utilization of Slag as Raw Material Additive in Portland Cement Kiln Feed

The objectives of using coal gasification slag as a raw material additive in making portland cement are threefold: (i) to replace a portion of the conventional raw materials, (ii) to reduce the firing temperature, thus lowering kiln energy requirements, and (iii) possibly to lower the kiln retention time thereby increasing kiln capacity. Test method development, equipment (high-temperature furnace) setup, and procurement of raw materials were completed in the previous reporting period. Experimental work was begun in the current reporting period following equipment calibration and development of mix designs. Testing involves the development of mix designs for a portland cement kiln feed consisting of sources of calcium, silica, and alumina, and determination of the theoretical maximum quantity of slag that can be accommodated as an additive.

In order to ascertain the effects of slag addition, tests were performed to establish baseline data and to thoroughly characterize the cement clinker raw feed and clinkers made at different temperatures. These tests include x-ray diffraction analysis, isothermal calorimetry, and TGA thermal analysis.

A supply of raw feed was obtained from a company in Pennsylvania. Prior to usage, this raw feed was subjected to x-ray diffraction analysis. Its x-ray diffraction pattern is shown in Figure 1. TGA thermal analysis was also performed and its trace is shown in Figure 2 (50-1050°C). The x-ray diffraction pattern of the raw feed shows a typical kiln feed with peaks dominated by calcium carbonate. The TGA weight loss trace was used to examine the weight loss characteristics of the raw feed with temperature. As can be seen clearly in the thermal trace, the majority of the weight loss occurs in the ~750-875°C temperature range, which corresponds to the liberation of CO₂ from the calcium carbonate. This information was used to design a heating curve for the clinkering process in order to allow sufficient time for the CO₂ to be released. A batch of the raw feed was then clinkered in a high-temperature molybdenum furnace at various temperatures using a platinum crucible. The temperature profile used was:

- Ramp to 900°C in 4 hours
- 900°C calcine for 4 hours
- 900°C to *clinkering temperature* in 2 hours
- Sinter at *clinkering temperature* for 4 hours
- Air quench to ambient temperature

The clinkering temperatures used for this study were 1100°C, 1200°C, 1300°C, and 1450°C. Normal clinkering temperature in a cement kiln is about 1450°C.

In order to study the hydration characteristics of these clinkers, isothermal calorimetry, a traditional method of studying the hydration of cement and cement-related products, was used to study the rate of heat evolution of these samples. In an isothermal calorimeter, the heat evolved during the hydration of cement is recorded with time. The resultant curve has a characteristic

peak early on when water is injected into the chamber. This is due to the wetting of the various particles by the hydration medium, usually deionized water. Subsequently, when the sample undergoes a chemical reaction such as hydration, heat is either liberated (exothermic) or absorbed (endothermic). In the case of cement, the hydration is exothermic, and the result is shown as a reaction hump in the calorimetry curve. The area under the heat evolution curve and the timing of this reaction hump provide much information on the hydration characteristics of the sample. The suite of traces recorded for these clinkers will be used to ascertain the effectiveness of slag addition during the clinkering process.

After clinkering at 1100°C, 1200°C, 1300°C, and 1450°C, the product was crushed, ground, and sieved through a 325-mesh screen and x-ray diffraction patterns were collected at 2°/min from 5° to 65°. The patterns are shown in Figures 3-6. The x-ray patterns confirm that the raw feed does not clinker well at 1100°C and 1200°C. Above 1300°C, the x-ray pattern shows successful clinkering of the raw feed into cement phases. The pattern at 1450°C contains peaks from calcium silicate oxide (ICDD #73-0599) and other as yet undetermined phases. The pattern from the 1300°C sample is essentially the same as that from the 1450°C sample except for the relative intensity of the peaks. As will be shown later by isothermal calorimetry, the hydration property of the 1450°C sample is typical of portland cement clinker. Although the phase distribution of the crystalline phases in all the samples has not yet been completely analyzed, it is clear that the 1100°C and 1200°C have extra peaks that are not calcium silicates and, as will be shown later, their hydration property is very poor compared to the higher-temperature clinkers.

We have also begun studying the effect of adding ground slag to the feed. Prepared slag was added to the raw feed at 5% and 10% levels by weight and the resultant mixture clinkered at high temperature. The addition of 5% and 10% slag to the raw feed did not alter the x-ray diffraction pattern. This was not surprising since the slag is x-ray amorphous. The x-ray pattern of the raw feed with 5% and 10% slag addition prior to clinkering are shown in Figures 7 and 8.

Isothermal calorimetry of the clinker product samples was conducted at 25°C. Each sample clinker was first ground and passed through a 325-mesh sieve. Then 3 grams of material and 3 grams of deionized water (water:solids ratio of 1:1) were allowed to equilibrate inside the calorimeter separately. The sample was hydrated with the water within the calorimeter after equilibrium had been reached. Data were collected every 10 seconds up to 24 hours. Tests were conducted only on the clinkered samples and not on the raw feed since the unfired raw feed is expected to have no hydration property at all. In addition, we tested one sample clinkered at 1300°C with 5% added slag. As is typical for hydration experiments, all the heat evolution curves (not shown here) have an early initial wetting peak (in the first few minutes). At longer time, the sample that was clinkered at 1200°C showed no reaction peak whereas the 1300°C sample (with 5% slag) and the 1450°C sample showed a very pronounced reaction peak beginning at about 5 hours and lasting through almost 24 hours, with the maximum peak occurring at about 14 hours. The 1300°C and 1450°C curves were very similar, with the 1450°C curve having a slightly broader wetting curve. On the basis of these calorimetry curves, it was concluded that the hydration characteristics of the sample with 5% slag clinkered at 1300°C are very similar to those of the sample clinkered at 1450°C.

To summarize based on analysis conducted to date, the data suggest that 5% addition of ground slag can produce a clinker at 1300°C with similar hydration characteristics to a clinker produced at 1450°C without added slag. This suggests that a significant heat energy saving of 150°C is possible when incorporating slag into cement kiln feed, which would translate to lower heating costs or higher kiln capacity.

Figure 1: X-ray Diffraction Pattern of Raw Kiln Feed (5-65° 2θ, 2°/min)

Figure 2: TGA Weight Loss Curve of Raw Kiln Feed (50-1050°C, 10°C/min)

Figure 3: X-ray Diffraction Pattern of Clinker Formed at 1100°C (5-65° 2 θ , 2°/min)

Figure 4: X-ray Diffraction Pattern of Clinker Formed at 1200°C (5-65° 2 θ , 2°/min)

Figure 5: X-ray Diffraction Pattern of Clinker Formed at 1300°C (5-65° 2 θ , 2°/min)

Figure 6: X-ray Diffraction Pattern of Clinker Formed at 1450°C (5-65° 2 θ , 2°/min)

Figure 7: X-ray Diffraction Pattern of Raw Kiln Feed with 5% Added Ground Slag (5-65° 2 θ , 2°/min)

Figure 8: X-ray Diffraction Pattern of Raw Kiln Feed with 10% Added Ground Slag (5-65° 2 θ , 2°/min)

We are completing the phase analyses of all the x-ray diffraction patterns and the clinkering and calorimetry study of the 5% added slag samples at different temperatures. In the next reporting

period, we will study the effects of adding 10% slag to the cement clinker feed. Since the addition of 5% slag appears to reduce the temperature needed for clinkering, we will determine whether the addition of 10% slag further reduces energy requirements. If 10% slag proves to be too much in that it reduces the hydration property of the clinker, we will conduct additional tests to determine the maximum amount of slag that can be added while producing a good clinker. We will also examine the effect of residence time in the furnace. With the addition of slag to the raw feed, it is possible that the residence time may be reduced while still producing a good clinker. Further tests will be conducted to optimize residence time. After an optimized clinker is produced, a large batch will be made and tested along with a control clinker sample (without slag) for properties important to the cement industry; e.g., compressive strength.

4.2 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 the testing of slag as a cement kiln feed material
- Continue data evaluation and preparation of the final technical report.