



# **Utilization of Lightweight Materials Made from Coal Gasification Slags**

**Cooperative Agreement No. DE-FC21-94MC30056**

**Final Technical Report**

**July 2001**

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## **Acknowledgments**

The authors would like to acknowledge the contributions of the late Sidney M. Cohen in his capacity as Director of Research and Development at Fuller Company. A pioneer in the development of lightweight aggregates from clay and shale materials, he became an early supporter of expanded slag, and devoted much personal time to developing economic, commercial-scale techniques for slag expansion. Without his interest and support the project would not have achieved its goals. He passed the torch to Michael Prokesch, who continued to make valuable contributions to the project at Fuller.

We also appreciate the tireless work and encouragement of Thomas Zimmerle at Silbrico in the effort to make ultra-lightweight aggregates from slag. We recognize the efforts of Phil and Skip Steck at Harvey Cement Products, who applied their years of experience in making blocks to expanded slag aggregates. They went the extra mile in helping the project by producing expanded slag blocks at the plant.

Praxis acknowledges the contributions of the organizations that participated in the project and provided cost sharing. In particular, we recognize the efforts of the lead contact persons at these organizations. They include Nandor Hertz and Michael Epstein at the Electric Power Research Institute (EPRI) and Dan Banerjee at the Illinois Clean Coal Institute (ICCI). We appreciate the constructive input from the project managers at DOE: Scott Renninger who helped keep the project focused on the main goals, and Leo E. Makovsky whose contributions to the final report significantly enhanced its clarity.

## **Abstract**

The objective of the project entitled "Utilization of Lightweight Materials Made from Coal Gasification Slags" was to demonstrate the technical and economic viability of manufacturing low-unit-weight products from coal gasification slags which can be used as substitutes for conventional lightweight and ultra-lightweight aggregates. In Phase I, the technology developed by Praxis to produce lightweight aggregates from slag (termed SLA) was applied to produce a large batch (10 tons) of expanded slag using pilot direct-fired rotary kilns and a fluidized bed calciner. The expanded products were characterized using basic characterization and application-oriented tests. Phase II involved the demonstration and evaluation of the use of expanded slag aggregates to produce a number of end-use applications including lightweight roof tiles, lightweight precast products (e.g., masonry blocks), structural concrete, insulating concrete, loose fill insulation, and as a substitute for expanded perlite and vermiculite in horticultural applications. Prototypes of these end-use applications were made and tested with the assistance of commercial manufacturers. Finally, the economics of expanded slag production was determined and compared with the alternative of slag disposal. Production of value-added products from SLA has a significant potential to enhance the overall gasification process economics, especially when the avoided costs of disposal are considered.

**Keywords**: slag, expanded slag, coal gasification slag, lightweight aggregates, gasification by-product utilization, waste utilization.

# TABLE OF CONTENTS

|   |           |
|---|-----------|
| <b>EXECUTIVE SUMMARY.....</b>   | <b>I</b>  |
| <b>1.0 INTRODUCTION, OBJECTIVES, SCOPE, AND METHODOLOGY.....</b>                        | <b>1</b>  |
| 1.1 Overview and Background Information.....  | 1         |
| <b>2.0 RESULTS AND DISCUSSION OF PHASE I WORK.....</b>                                  | <b>5</b>  |
| 2.1 Phase I Objectives.....   | 5         |
| 2.2 Phase I Project Methodology and Test Plan.....                                      | 6         |
| 2.2.1 Phase I Methodology.....  | 6         |
| 2.2.2 Phase I Test Plan (Task 1.1).....   | 7         |
| 2.3 Production of Lightweight Aggregates from Slag (Task 1.2).....                      | 9         |
| 2.3.1 Slag/Char Separation.....   | 9         |
| 2.3.2 Evaluation of Char as By-Product.....   | 11        |
| 2.3.3 Pilot Production of Lightweight Aggregates from Slag.....                         | 12        |
| 2.3.3 Data Analysis of Slag Preparation and Expansion.....                              | 14        |
| 2.3.4 Economic Analysis of Expanded Slag Production (Task 1.4).....                     | 18        |
| 2.3.5 Phase I Final and Other Reports (Task 1.5).....                                   | 19        |
| 2.4 Major Conclusions from Phase I Work.....  | 19        |
| <b>3.0 PHASE II OBJECTIVES AND SCOPE.....</b>   | <b>21</b> |
| 3.1 Phase II Objectives.....  | 21        |
| 3.2 Phase II Scope of Work.....   | 21        |
| Task 2.1 Test Plan for Applications of Expanded Slags (see also Appendix A).....        | 21        |
| Task 2.2 Field Studies to Test Expanded Slag Utilization.....                           | 22        |
| Task 2.3 Data Analysis of Commercial Utilization of Expanded Slags.....                 | 22        |
| Task 2.4 Economic Analysis of Expanded Slag Utilization.....                            | 22        |
| Task 2.5 Separation of Char from Slag for Tampa Electric Company IGCC Plant.....        | 22        |
| Task 2.6 Testing of Slag as Raw Material Additive in Portland Cement Kiln Feed.....     | 22        |
| Task 2.7 Utilization of Slag as Raw Material in Portland Cement (Pilot Testing).....    | 23        |
| Task 3.0 Final and Other Reports.....   | 23        |
| 3.3 Project Methodology.....  | 23        |
| <b>4.0 RESULTS AND DISCUSSION OF PHASE II WORK.....</b>                                 | <b>25</b> |
| 4.1 Test Plan for Applications Testing of Expanded Slags: Field Studies (Task 2.1)..... | 25        |
| 4.2 Field Studies to Test Expanded Slag Utilization (Task 2.2).....                     | 26        |
| 4.3 Data Analysis of Commercial Utilization of Expanded Slags (Task 2.3).....           | 26        |
| 4.3.1 Laboratory Evaluation of SLA for Roof Tile Application.....                       | 27        |
| 4.3.2. Lightweight Masonry Blocks (CMUs).....   | 34        |
| 4.3.3 Laboratory Evaluation of SLA for Structural Concrete Application.....             | 41        |
| 4.3.4 Laboratory Evaluation of SLA for Insulating Concrete Application.....             | 48        |
| 4.3.5 Laboratory Evaluation of SLA for Loose Fill Insulation Application.....           | 49        |
| 4.3.6 Laboratory Evaluation of SLA in Horticultural Applications.....                   | 50        |
| 4.3.7 Evaluation of SLA in Production of Cement Panels (Waterproof Boards).....         | 52        |
| 4.4 Economic Analysis of Expanded Slag Production and Utilization (Task 2.4).....       | 53        |
| 4.4.1 Costs of Production of LWA and ULWA Made from Expanded Slag.....                  | 55        |

|            |   |            |
|------------|---|------------|
| 4.4.2      | Market Assessment of Conventional LWA and SLA.....                                      | 58         |
| 4.4.3      | Solid Waste Management Costs .....  | 61         |
| 4.4.4      | Economic Evaluation of SLA Production.....  | 61         |
| <b>5.0</b> | <b>ADDITIONAL SLAG UTILIZATION WORK.....</b>  | <b>64</b>  |
| 5.1        | Separation of Char from Slag from Tampa Electric IGCC Plant (Task 2.5).....             | 64         |
| 5.1.1      | Laboratory Testing of TEC Slag for Char Removal.....                                    | 64         |
| 5.1.2      | Development of Conceptual Design for a Slag/Char Separation Facility .....              | 66         |
| 5.1.3      | TEC Clean Slag Production Economics .....   | 67         |
| 5.1.4      | Results of Applications-Oriented Testing of TEC Slag.....                               | 68         |
| 5.1.5      | Utilization of Slag/Char Separation Process at DOE-Funded IGCC Plants .....             | 69         |
| 5.1.6      | Conclusions .....   | 70         |
| 5.2        | Laboratory Studies of Slag as Raw Material in Portland Cement Kiln Feed (Task 2.6)..... | 70         |
| 5.2.1      | Summary of Laboratory Tests.....  | 80         |
| 5.3        | Pilot Studies on Slag as Raw Material in Portland Cement Kiln Feed (Task 2.7).....      | 80         |
| 5.4        | Pozzolonic Activity of Prepared Gasifier Slag.....                                      | 90         |
| <b>6.0</b> | <b>CONCLUSIONS AND RECOMMENDATIONS .....</b>  | <b>94</b>  |
| 6.1        | General Conclusions .....   | 94         |
| 6.2        | Specific Conclusions from Applications Testing.....                                     | 95         |
| 6.2.1      | Lightweight Aggregate Applications.....   | 95         |
| 6.2.2      | Ultra-lightweight Aggregate Applications.....   | 96         |
| 6.3        | Economics of SLA Production.....  | 97         |
| 6.4        | Recommendations for Future Work.....  | 98         |
|            | <b>LIST OF ACRONYMS AND ABBREVIATIONS .....</b>   | <b>99</b>  |
|            | <b>APPENDIX A. TEST PLAN FOR APPLICATIONS OF EXPANDED SLAGS (TASK 2.1).....</b>         | <b>A-1</b> |

## LIST OF TABLES

|   |    |
|---|----|
| Table 1. Estimated Product Requirements for Evaluation of Expanded Slag Applications .....  | 6  |
| Table 2. Laboratory-Scale Confirmatory Tests Planned.....                                   | 7  |
| Table 3. Pilot-Scale Production Runs Planned .....  | 8  |
| Table 4. Slag/Char Separation Results for Slag I.....                                       | 11 |
| Table 5. SLA Production Rotary Kiln Operating Conditions .....                              | 15 |
| Table 6. Direct-Fired Rotary Kiln Emissions .....   | 16 |
| Table 7. Analysis of SLA Crushed for Roof Tile Application and TCLP Results.....            | 17 |
| Table 8. Cement Levels to Test SLA I as Structural Aggregate.....                           | 27 |
| Table 9. Formulations and 7-Day Compressive Strength of Roof Tile Samples.....              | 28 |
| Table 10. 28-Day Compressive Strength Results for Roof Tile Application.....                | 30 |
| Table 11. Evaluation of SLA III as Aggregate in Roof Tile Application.....                  | 31 |
| Table 12. 28-Day Strength Results of SLA in Roof Tile Application.....                      | 32 |
| Table 13. Size Gradation of Aggregate Mix Used in Roof Tile Tests.....                      | 33 |
| Table 14. Evaluation of SLA III as Aggregate in Roof Tile Application.....                  | 34 |
| Table 15. Size Distribution of Aggregates Used for Concrete Block Application.....          | 35 |
| Table 16. LWA and Cement Concrete Requirements for CMU Applications .....                   | 36 |
| Table 17. Results of Batch Mix Tests Conducted for Masonry Blocks Using SLA .....           | 37 |
| Table 18. Results of Batch Mix Tests for Masonry Blocks Using SLA (contd.).....             | 38 |
| Table 19. Results of Batch Mix Tests for Masonry Blocks Using SLA III.....                  | 39 |
| Table 20. Additional Results of Batch Mix Tests for Masonry Blocks Using SLA.....           | 40 |
| Table 21. Mix Proportions for Production Batch of Lightweight Blocks.....                   | 40 |
| Table 22. Compressive Strength and Unit Weight Test Results for Lightweight Blocks .....    | 41 |
| Table 23. Summary of Test Results for Use of SLA in Lightweight Block Application.....      | 41 |
| Table 24. Cement Levels Used in Testing SLA as Structural Aggregate.....                    | 42 |
| Table 25. LWA and Concrete Unit Weight and Strength Requirements.....                       | 42 |
| Table 26. Samples of Materials Used for Structural Aggregate Testing .....                  | 43 |
| Table 27. Size Distribution of LWA Materials and Sand Used in Structural Concrete .....     | 43 |
| Table 28. Expanded SLA Products Tested and Cement Levels Used .....                         | 44 |
| Table 29. Use of SLA for Structural Lightweight Concrete Without Sand .....                 | 45 |
| Table 30. Evaluation of SLAs for Sand-Lightweight Concrete Application.....                 | 46 |
| Table 31. Resistance of Concrete to Rapid Freeze/Thaw (ASTM C 666, Procedure B).....        | 48 |
| Table 32. Apparent Thermal Conductivity and Thermal Resistance of SLA Test Specimens.....   | 49 |
| Table 33. Thermal Conductivity and Thermal Resistance of SLA for Loose Fill Insulation..... | 50 |
| Table 34. Evaluation of Expanded Slag in Supporting Plant Growth.....                       | 51 |
| Table 35. Mix Design and Modulus of Rupture Test Results for SLA in Panel Application.....  | 52 |
| Table 36. Design Criteria for Plant to Process Gasifier Slag.....                           | 55 |
| Table 37. Fuel Requirements for Various Case Studies .....                                  | 56 |
| Table 38. Comparative Costs of Kiln Production of LWA vs. SLA .....                         | 57 |
| Table 39. Comparative Costs of Producing ULWA vs. SLA .....                                 | 58 |
| Table 40. U.S. Production of Expanded Shales, Clays, and Volcanic LWAs .....                | 59 |
| Table 41. U.S. Production of Perlite and Vermiculite Ores.....                              | 60 |
| Table 42. Typical Regional Prices of Lightweight Aggregates.....                            | 60 |
| Table 43. Estimated Market Prices for LWAs, ULWAs, and SLA by Application.....              | 60 |
| Table 44. Economic Analysis Summary.....  | 63 |
| Table 45. Results of Slag/Char Separation for TEC Slag.....                                 | 65 |

|   |    |
|---|----|
| Table 46. Slag/Char Separation Results from Process Steps 1 & 2 for Various Slags .....             | 66 |
| Table 47. Slag/Char Separation Costs and Economics Summary .....                                    | 68 |
| Table 48. Utilization of Char-Free TEC Slag as Aggregate in Cement Concrete .....                   | 69 |
| Table 49. Utilization of TEC Slag as Feedstock for LWA Production.....                              | 69 |
| Table 50. Typical Composition of Cement Clinker.....  | 71 |
| Table 51. Typical Portland Cement Composition With Major Compounds .....                            | 72 |
| Table 52. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 0% Slag).....                  | 82 |
| Table 53. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 3% Slag).....                  | 83 |
| Table 54. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 5% Slag).....                  | 84 |
| Table 55. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 10% Slag).....                 | 85 |
| Table 56. Summary of Free Lime Tests Using Pre-Blended Cement Kiln Raw Meal.....                    | 85 |
| Table 57. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 0% Slag) .....            | 86 |
| Table 58. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 3% Slag) .....            | 87 |
| Table 59. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 5% Slag) .....            | 88 |
| Table 60. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 8.5% Slag) .....          | 89 |
| Table 61. Summary of Free Lime Test Using Raw Ingredients .....                                     | 89 |
| Table 62. Summary of Free Lime Test Using Raw Ingredients with Lower Silica Modulus .....           | 89 |
| Table 63. ASTM C 311 Results Using Specially Prepared Slag.....                                     | 90 |
| Table 64. ASTM C 593 Results Using Specially Prepared Slag.....                                     | 91 |
| Table 65. Initial Particle Size Distribution of Slag Prior to Bond Ball Mill Grindability Test..... | 91 |
| Table 66. Slag Bond Ball Mill Grindability Test Results.....  | 92 |
| Table 67. Sedigraph Particle Size Distribution of Ground Slag.....                                  | 92 |
| Table 68. ASTM C 311 Results Using -200 Mesh Ball Milled Slag.....                                  | 93 |
| Table 69. ASTM C 593 Results Using -200 Mesh Ball Milled Slag.....                                  | 93 |

## LIST OF FIGURES

|   |    |
|---|----|
| Figure 1. Slag Utilization Applications .....   | 3  |
| Figure 2. Schematic of Direct-Fired Rotary Kiln Pilot Plant.....                                | 13 |
| Figure 3. X-ray Diffraction Pattern of Raw Kiln Feed (5-65°, 2°/min).....                       | 73 |
| Figure 4. TGA Weight Loss Curve of Raw Kiln Feed (122-1922°F (50-1050°C), 10°C/min).....        | 73 |
| Figure 5. X-ray Diffraction Pattern of Clinker Formed at 2012°F (1100°C) (5-65°, 2°/min).....   | 75 |
| Figure 6. X-ray Diffraction Pattern of Clinker Formed at 2192°F (1200°C) (5-65°, 2°/min).....   | 75 |
| Figure 7. X-ray Diffraction Pattern of Clinker Formed at 2372°F (1300°C) (5-65°, 2°/min).....   | 76 |
| Figure 8. X-ray Diffraction Pattern of Clinker Formed at 2642°F (1450°C) (5-65°, 2°/min).....   | 76 |
| Figure 9. X-ray Diffraction Pattern of Raw Kiln Feed with 5% Ground Slag (5-65°, 2°/min).....   | 77 |
| Figure 10. X-ray Diffraction Pattern of Raw Kiln Feed with 10% Ground Slag (5-65°, 2°/min)..... | 78 |
| Figure 11. Isothermal Calorimetry of Clinkers Formed at Various Temperatures.....               | 79 |
| Figure 12. X-ray Diffraction Analysis of Clinkers Formed at Various Temperatures .....          | 80 |



## EXECUTIVE SUMMARY

Slag is a solid residue from the gasification of coal at integrated-gasification combined-cycle (IGCC) facilities, which is currently disposed of as a solid waste. The major objectives of the project, titled “Utilization of Lightweight Materials Made from Coal Gasification Slags,” were to demonstrate the technical and economic viability of commercial production of lightweight aggregates (LWA) and ultra-lightweight aggregates (ULWA) from slag and to test the suitability of these aggregates for various applications. LWAs are typically produced by thermal expansion or pyroprocessing of expansive shales and clays, while ULWAs are produced by pyroprocessing of perlite or vermiculite ores. While LWAs and ULWAs tend to be used in relatively low-volume applications, they command fairly high prices relative to normal-weight aggregates. It was envisaged that the utilization of a portion of the slag generated at a typical gasifier operation for the production of high-value materials such as LWAs would render slag utilization economically viable. This, in turn, would promote the utilization of the remaining slag in lower-value applications. This approach would lead to the utilization of all of the slag produced, and may even generate a revenue stream for IGCC facilities while eliminating disposal costs.

Primary funding for the project was provided by DOE's National Energy Technology Laboratory (NETL), with significant cost sharing by the Electric Power Research Institute (EPRI) and the Illinois Clean Coal Institute (ICCI). In addition, several industry participants, including Fuller Company, Harvey Cement Products, Inc., and Silbrico, Inc. provided significant in-kind cost sharing.

The project team consisted of Praxis Engineers, Inc. as the prime contractor, with significant participation from Fuller Company's R&D Division, as well as Harvey Cement Products, Silbrico Company, and Monier Lifetile, Inc.

The primary focus of the project was to demonstrate the production of LWAs and ULWAs from slag and test them as substitutes for conventional materials in a number of applications. The technologies for separating the residual char content of the slag—considered essential for its safe utilization—and for utilization of slag in both its as-generated and char-free forms, as well as production of LWAs and ULWAs from slag were all developed prior to this project. These technologies or process steps were applied to the slag samples collected for the project, using currently available commercial-scale equipment.

The project goals were accomplished in two phases. Phase I comprised the separation and recovery of char (unconverted carbon) from the char-free slag, and production of LWA and ULWA from slag at the large pilot scale. A 20-ton sample of slag (Slag I) was collected from one source. A second sample of slag (Slag II) was collected at the request of ICCI from an Illinois coal feedstock and used for confirmatory testing of both the char separation and pyroprocessing steps. A third slag, from the Wabash River Coal Gasification Repowering Project, was added to the project within the existing budget, based on interest in the technology by PSI Energy. At the request of DOE, a fourth sample, from the Tampa Electric Integrated Gasification Combined Cycle Project, was subsequently added to the program. Both the Wabash and Tampa projects are part of the U.S. Department of Energy Clean Coal Technology

Demonstration Program and use Destec<sup>1</sup> and Texaco gasifiers, respectively. Thus, the slag samples used in the project were generated from different gasification processes and originated from different bituminous coal feedstocks. The char separation and expanded slag production processes demonstrated during the project can therefore be generalized to apply to most of the coals or gasification processes in current application in the United States.

Since the char present in gasification slag constitutes a hindrance to its utilization, its removal is a critical step in the development of utilization applications for slag. Separation and recovery of char from slag using the process developed by Praxis Engineers, Inc. was demonstrated successfully under this program using a 400-lb/hour pilot plant.

This was followed by successful demonstration of the production of LWA and ULWA from slag (termed SLA) at the pilot scale, using technology previously developed by Praxis. Two sizes of rotary kilns and a fluidized bed expander were set up at the facilities of Fuller Company, a leading manufacturer of kiln equipment for the LWA industry, and used to pyroprocess the project slags to produce large quantities of expanded slag aggregates of various size gradations and with unit weights ranging from 18 to 50 lb/ft<sup>3</sup>. All of the project slag samples expanded at temperatures ~400°F lower than those required for pyroprocessing of expansive shales and clays. This represents significant savings in pyroprocessing fuel energy requirements. In all three expansion processes that were demonstrated, sufficient control of the product unit weight as a function of temperature was achieved to produce LWAs and ULWAs.

In Phase II, the SLA products were first tested at the laboratory scale for their suitability as replacements for LWAs in the manufacture of precast concrete products (e.g., masonry blocks and roof tiles), lightweight structural concrete, and concrete panels. They were also tested for their suitability as replacements for ULWAs in the manufacture of insulating concrete and loose fill insulation, and in horticultural applications. Subsequently some of these applications were tested at a larger scale with the involvement of commercial manufacturing plants, using ASTM and industry test methods to evaluate the products. The major findings for these applications are summarized below.

### **LWA Applications**

**Lightweight Blocks.** SLA aggregates were successfully tested for production of lightweight blocks or concrete masonry units (CMU). The target lightweight block product (8" CMU) weighs <27 lb on a dry basis. The ASTM and industry requirements for concrete used for this application include a compressive strength of 2,000 psi at a unit weight of <105lb/ft<sup>3</sup>, using a typical cement-to-aggregate ratio of <1:5. Following laboratory development work, 250 blocks were produced using a mix incorporating 40% SLA (by weight) and a cement-to-aggregate ratio of 1:6. In the production of SLA blocks, the entire automated production and post-production manufacturing process was used without modification. They were handled through the mechanized processing steps without any problems, and no special curing or handling was needed. The product met both industry and ASTM requirements.

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<sup>1</sup> Global Energy, Inc. now owns the Destec Gasification Process and markets the technology under the name E-GAS Technology™.

**Structural Concrete.** SLA was also successfully used to make structural concrete that met ASTM and industry requirements of compressive strengths of 2,500-4,000 psi for sand LWA concrete with unit weights in the 105-115 lb/ft<sup>3</sup> range, using a typical industry cement-to-aggregate ratio of 1:4. Concrete made from a combination of SLA and conventional clay-based LWA had an even higher strength of 5,500 psi.

**Concrete Panels.** Concrete panels (cement boards) are used for structural reinforcement and as water-resistant backing for ceramic tile installations. The target specification for concrete for this application was an industry requirement for a compressive strength of 2,500 psi with a panel weight of 3.2-3.6 lb/ft<sup>2</sup> using a cement-to-aggregate ratio of 1:2.5. Tests conducted at the facility of a panel manufacturer demonstrated that expanded slag in the 35-40 lb/ft<sup>3</sup> unit weight range met their requirements and may even perform better than conventional materials due to its lower unit weight.

### ***ULWA Applications***

SLA was also successfully tested as a total and partial substitute for several conventional ULWA applications, namely insulating concrete, loose fill insulation, and nursery/horticultural applications. Perlite-based ULWAs have unit weights in the <4-12 lb/ft<sup>3</sup> range. However, since the lowest unit weight we achieved for expanded slag was about 18 lb/ft<sup>3</sup>, we used SLA products ranging from 18 to 35 lb/ft<sup>3</sup> to evaluate its comparative performance in the ULWA applications. It was concluded that SLA meets some of the requirements for expanded perlite. However, optimal results were obtained when SLA was used as a partial substitute for perlite products.

**Insulating Concrete.** Insulating concrete is used as an insulating layer in built-up roofs and is typically manufactured using expanded perlite or shale. The application requires a 200-psi concrete. The typical thermal conductivity of perlite (Group I) is 0.45-1.5 Btu-in/hr-ft<sup>2</sup>-°F, and that of shale-based aggregates (Group II) is 1.5-3.0 Btu-in/hr-ft<sup>2</sup>-°F. The thermal conductivity of concrete made using 26 lb/ft<sup>3</sup> SLA was 0.984 Btu-in/hr-ft<sup>2</sup>-°F, which is superior to that of expanded shale but inferior to that of expanded perlite.

**Loose Fill Insulation.** Expanded perlite is used to fill cavities in blocks used for construction of building exterior walls to improve their insulation properties. The thermal resistance of 29 lb/ft<sup>3</sup> SLA was 1.46 hr-ft<sup>2</sup>-°F/Btu, which is lower than that of expanded perlite at 2.6-2.4 hr-ft<sup>2</sup>-°F/Btu. However, SLA has the advantage with respect to other industry requirements such as its free-flowing nature, low friability and hence low dustiness, and low moisture retention.

**Horticultural Applications.** SLA products with unit weights ranging between 18 and 35 lb/ft<sup>3</sup> were tested as partial substitutes for expanded perlite and vermiculite at a commercial nursery. The SLA proved successful as a partial substitute for perlite only but not for mixes calling for both perlite and vermiculite. The main problem with the SLA was its high drainage rate, which necessitated more frequent watering. However, its higher unit weight was seen as an advantage in providing greater stability to large potted plants and shrubs, and its higher strength made it suitable for mechanized field/nursery applications.

### ***Economics of SLA Production.***

An economic evaluation was conducted for a hypothetical single facility to first process raw slag for char recovery and then pyroprocess the char-free slag to produce lightweight and ultra-lightweights aggregates of various unit weights, as dictated by demand in local and adjoining markets. This single facility was assumed to be located at the gasifier site to eliminate double handling of slag. It was envisaged that the recovered char could be recycled to the gasifier. The costs of this facility were estimated by developing a process flowsheet based on pilot plant operations data generated during the project, along with process equipment-factored capital cost estimates. For the economic analysis, four scenarios were studied representing two sizes of IGCC facilities (200 MW and 400 MW), each using two process technologies for SLA production, the rotary kiln and fluidized bed calciner. The two scales studied are:

- A plant to process slag generated from a 200-MW gasifier facility, typically using 2,000 tons/day of bituminous coal containing 10% ash, and generating 220 tons/day of slag containing 10% char.
- A plant to process slag generated from a 400-MW gasifier facility, typically using 2,000 tons/day of bituminous coal containing 10% ash, and generating 440 tons/day of slag containing 10% char. This would approximate the feed capacity of a typical commercial LWA plant that currently uses conventional expansible clays.

The slag production economics were conducted using two parallel approaches:

- Comparison of the economics of SLA production vs. slag disposal
- Comparison of the economics of SLA production with the estimated market value of end products that can be made from it.

The market price of SLA was estimated taking into consideration the fact that it would likely command a lower price as a new, unproven material. The sale prices for slag lightweight aggregates were estimated at \$30/ton for block aggregates, \$35/ton for structural aggregates, and \$40/ton for roof tile and ultra-lightweight aggregate applications. Using a product mix based on the percentage of coarse and fine slag, the weighted average price of SLA was estimated at \$34.75/ton. This price was used to evaluate the economics of SLA production.

For purposes of this analysis, a value of \$15/ton was used as the cost of slag disposal, which is in the middle of the \$10-20/ton range indicated for fly ash. Since these avoided costs would provide substantial savings to the gasifier operation, this amount could potentially be made available to the SLA production facility as a tipping fee per ton of slag accepted.

For the rotary kiln processes, the SLA production costs were estimated at \$30.06/ton and \$24.40/ton for the 220 and 440 tons/day capacities (200-MW and 400-MW gasifiers) respectively. These costs are competitive with conventional LWA production costs, which were estimated at \$30.10/ton based on a survey of four operating plants. SLA production costs are considerably lower than the composited market price for such materials, estimated at \$34.74/ton. The payback period for the large rotary kiln system was estimated at less than four years.

The fluidized bed method of SLA production was found to be even more competitive because of lower capital and operating costs. Its production costs were \$26.48 and \$21.87 for the smaller and larger sizes respectively. The payback period for the large fluidized bed system was estimated at less than three years.

## 1.0 INTRODUCTION, OBJECTIVES, SCOPE, AND METHODOLOGY

### 1.1 Overview and Background Information

This document constitutes the final report for the project titled “Utilization of Lightweight Materials Made from Coal Gasification Slags.” The project was awarded to Praxis Engineers, Inc by the Department of Energy (DOE) under Cooperative Agreement No. DE-FC21-94MC30056, and was executed in two phases. Phase I consisted of production of expanded slag at the pilot scale, and Phase II consisted of testing and evaluation of the expanded slag as a replacement for conventional lightweight and ultra-lightweight aggregates. This document summarizes the findings of the Phase I work (reported in Topical Report No. 1) and provides detailed results of the Phase II work.

Primary funding for the project was provided by DOE's National Energy Technology Laboratory (NETL), with significant cost sharing by the Electric Power Research Institute (EPRI) and Illinois Clean Coal Institute (ICCI). Since the ICCI's mission is to promote the use of Illinois coals, one of the project slag samples was derived from an Illinois coal feedstock.

In addition, several industry participants provided significant in-kind cost sharing:

- Fuller Company, a major manufacturer of lightweight aggregate kiln equipment
- Pennsylvania State University, where some of the development work and char removal testing was conducted
- Harvey Cement Products, Inc., where we made blocks from SLA using their block manufacturing plant
- Silbrico Company, a manufacturer of perlite products and expansion equipment
- Monier Lifetile, Inc., a producer of lightweight concrete tiles
- Big River Industries, a manufacturer of lightweight aggregates
- Evergreen Nursery, where the horticultural application was tested
- Custom Building Products, a manufacturer of lightweight concrete panels.

Praxis Engineers, Inc., the prime contractor, also provided significant cost sharing.

The project team consisted of Praxis Engineers as the prime contractor, with significant participation from Fuller Company's R&D Division, as well as Harvey Cement Products, Silbrico, and Monier.

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 (termed slag) and some unconverted carbon. In previous projects, Praxis investigated and developed the utilization of “as-generated” slag 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. The reasons include the following:

- The unconverted carbon in the slag is detrimental to its utilization as sand or aggregate.
- The physical characteristics of slag (particle size, shape, density, appearance, etc.) are different from those of the materials it could replace, such as sand or fine aggregates.
- The materials it could replace are abundantly available at very low cost.
- There is a widespread reluctance to use new materials due to potential liability issues.

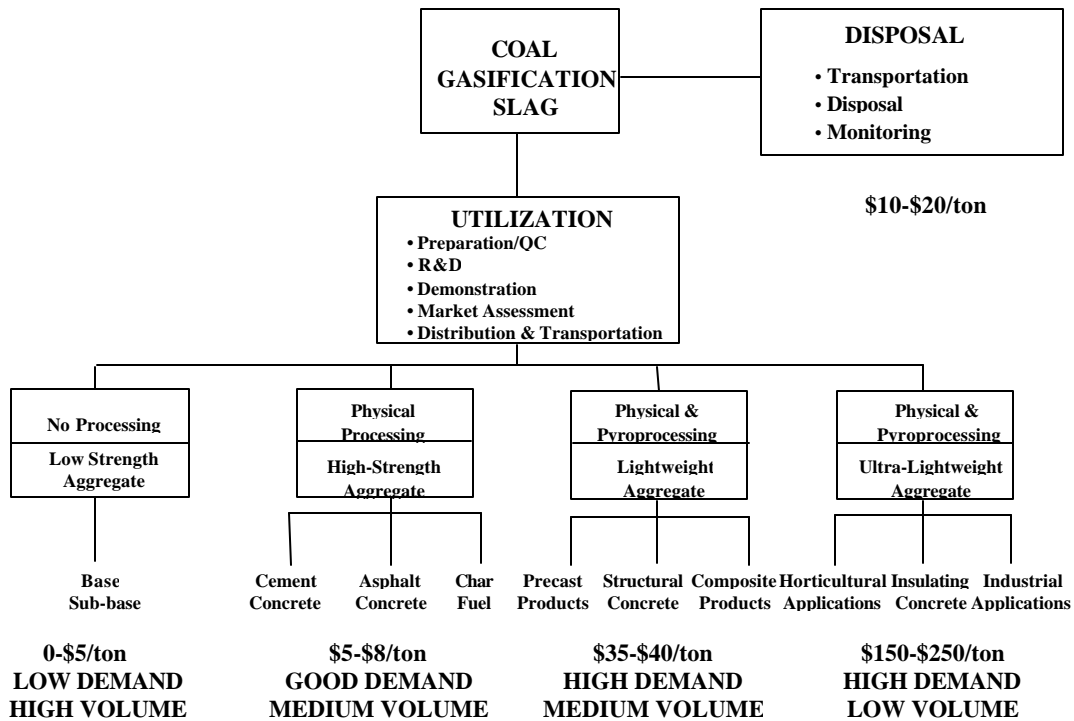
Total U.S. production of aggregates was 2.5 billion tons in 1995, consisting of 1.4 billion tons of crushed stone and 1.1 billion tons sand and gravel<sup>2</sup>. Conventional aggregate usage can be broken down into three broad categories: high-volume (low-value), medium-volume (medium-value), and low-volume (high-value) applications. For example, fill materials used for site preparation and as base and sub-base materials constitute the highest-volume application totaling to about 1.5 billion tons/year. These aggregates, which sell for up to \$5/ton, are considered low-value applications. Unprocessed (as-generated) slag would be in competition with this market segment. Medium-value aggregates used in the production of asphalt and cement concrete account for nearly 1.0 billion tons/year. As these are produced to more stringent specifications, they sell in the \$5-\$8/ton range depending on the region and are considered to have good demand. Slag which has been processed to remove its char component would be in competition with this market segment. In contrast, lightweight aggregates, produced from expansive shales or clays account for 4.22 millions tons/year, and are a comparatively low-volume application. However, with sale prices of about \$30/ton, this is a high-value application. Slag processed to form lightweight aggregates would target this market segment. SLA could complement existing expandable clay and shale operations by extending the life of the mineral reserves. As these deposits are limited, the products are in high demand and can compete over larger distances.

Figure 1 summarizes the concept of developing a variety of applications designed to achieve total utilization of slag. In previous development work performed by Praxis, it was determined that raw slag could be used in high-volume applications such as aggregate in road base and sub-base, with a value in the \$0-\$5/ton range. Conversely, several high-value applications such as lightweight and ultra-lightweight aggregates could be produced by pyroprocessing the slag.

Through a series of prior studies Praxis established that development of a balanced mix of low-, medium-, and high-value applications would ensure that all size fractions of the raw slag would be utilized. This would generate a steady revenue stream for IGCC facilities while gradually eliminating the need for slag disposal and the costs associated with disposal. In addition, development of a wide variety of applications would help compensate for seasonal variations in the demand for some of the applications.

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<sup>2</sup> "Aggregate Production to Continue Upward Trend in 1995." In *Rock Products*, December 1994, page 30.



**Figure 1. Slag Utilization Applications**

Based on these studies, it became apparent that to meet the goal of total utilization of slag it would be desirable to develop a wide variety of value-added products from slag, each designed to meet specific industry requirements. The low- and medium-value applications were demonstrated by evaluating slag, both in its as-generated form and after processing for char recovery, as a substitute for road base and sub-base aggregates and to produce cement and asphalt concretes. Following a significant amount of internal R&D at Praxis, we developed a high-value application for slag based on our determination that, upon controlled heating, slag undergoes expansion to form a lightweight material similar to those produced from expansive clays and shales. Subsequently, patents on the “Utilization of Slag from Coal Gasification Systems” were granted jointly to Vas Choudhry of Praxis Engineers, Seymour B. Alpert of EPRI, and Donald Meisel of Texaco, (European Patent No. 90121365.2, awarded 13 December 1990 and U.S. Patent No. 5,091,349, awarded 25 February 1992). The technology to produce lightweight and ultra-lightweight aggregates (ULWA) from slag was subsequently developed further and demonstrated in projects funded by the Electric Power Research Institute (EPRI), the Illinois Clean Coal Institute (ICCI), and internal resources.

The major objectives of the subject project were 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 a number of end-use applications. The project goals were accomplished in two phases. Phase I comprised the production of LWA and ULWA from slag at the large pilot scale. This involved the collection of a 20-ton sample of slag from one source, with subsequent collection of additional samples of other slags for confirmatory testing of the processing steps. A 400-lb/hour pilot plant was set up at Pennsylvania State University’s Materials Processing



Laboratory to process the slag samples for char removal. Phase I testing also covered preparation and testing of project slag samples at the laboratory scale for their expansion characteristics to produce LWA. Upon completing the development work, pilot plants consisting of two sizes of rotary kilns and a fluidized bed expander were set up at the facilities of Fuller Company, a leading manufacturer of kiln equipment for the LWA industry. These pilot plants were used to pyroprocess the project slag samples to produce large quantities of expanded slag aggregates of various size gradations and unit weights, ranging from 18 to 50 lb/ft<sup>3</sup>. Environmental (emissions) data for slag lightweight aggregate (SLA) production were collected to identify the type of pollution control equipment that would be required. In addition, the char recovered from the slag preparation operation was evaluated for use as a kiln fuel and as a recycled feed material mixed with the coal gasifier feed.

In Phase II, the expanded slag aggregates were tested at the laboratory scale for their suitability in the manufacture of precast concrete products (e.g., masonry blocks and roof tiles), lightweight structural concrete, concrete panels, and insulating concrete. Subsequently some of these applications were evaluated at a larger scale with the involvement of commercial manufacturing plants, using ASTM and industry test methods. Technical data generated during production and testing of the products were used to assess the overall technical viability of expanded slag production and utilization. The testing was followed by an economic evaluation of the production and utilization of the SLA-based products. This was based on cost information provided by commercial manufacturers of the target products, as well as data gathered on the potential market price the SLA-based products might command once their quality has been validated through manufacturer testing.

## 2.0 RESULTS AND DISCUSSION OF PHASE I WORK

### 2.1 Phase I Objectives

The objective of Phase I of the project was to demonstrate the technical and economic viability of producing lightweight aggregates (LWA) and ultra-lightweight aggregates (ULWA) from coal gasification slags. Conventional LWAs are typically produced by thermal processing of expansible clays and shales to achieve product unit weights in the 40-55 lb/ft<sup>3</sup> range. The resulting expanded aggregates are then used to produce end-use products such as lightweight structural concrete, lightweight blocks, and lightweight roof tiles. Conventional ULWAs are typically produced by pyroprocessing perlite or vermiculite ores to make expanded aggregates with unit weights in the 4-12 lb/ft<sup>3</sup> range. These aggregates are then used in various end-use applications, including insulating concrete, loose fill insulation, and in horticultural and other applications. During Phase I, we successfully demonstrated pilot-scale production of expanded slag lightweight aggregates (SLA) suitable for use as substitutes for conventional LWAs and ULWAs. Engineering data collected during pilot plant operation demonstrated the technical feasibility of producing LWA and subsequently utilizing all size fractions of the product in various end-use applications. Laboratory-scale tests were performed to demonstrate the use of the expanded slag aggregates in several applications including structural concrete, roof tiles, and loose fill insulation.

The technical objectives of Phase I were to:

- Demonstrate the technology for producing expanded slag aggregates (SLA) at the pilot scale (500 lb/hour), including collection of operational and emissions data.
- Produce a large batch (10 tons) of LWA and ULWA from slag for use in applications testing in Phase II.
- Perform a comparative evaluation of the quality of the expanded slag aggregates vis a vis conventional LWAs by conducting laboratory-scale tests in accordance with applicable ASTM or Perlite Institute procedures.
- Evaluate uses of the char recovered from the slag as recycled feed to the gasifier or for use as a fuel in the slag expansion process or—after blending with coal—the boiler.
- Perform environmental characterization of expanded slag products to confirm their safety prior to being used as substitutes for conventional LWAs and ULWAs.
- Conduct the preliminary economics of SLA production.

## 2.2 Phase I Project Methodology and Test Plan

### 2.2.1 Phase I Methodology

The project methodology is summarized below.

- Utilize the existing Praxis process for separation of char from slag, as the presence of char in as-produced slag has been found to be an impediment to its utilization in any form, including lightweight aggregate production.
- Build on previous developmental work performed by Praxis to identify potential applications for expanded slag aggregates, including EPRI-funded projects to produce LWAs from slag, ICCI-funded projects to produce ULWAs from slag, and internal studies.
- Obtain the participation of potential commercial users and producers of slag-based products throughout the course of the project to familiarize them with the capabilities of the new products and obtain their feedback.
- Seek the involvement of slag generators to keep them informed of the potential for utilizing slag as an alternative to disposal.
- Use conventional LWA production methods and equipment as much as possible to minimize the process development and commercialization timeframe and increase product acceptability to LWA and ULWA manufacturers and end-use industries.

The specific objective of Phase I was to produce about 10 tons of expanded slag materials for subsequent use in a number of end-use applications. The applications and estimated amounts of material required for each are listed in Table 1.

**Table 1. Estimated Product Requirements for Evaluation of Expanded Slag Applications**

| Item  | Application/Objective   | SLA Products   |
|-------|---|----------------|
| 1     | Lightweight blocks  | 4 tons         |
| 2     | Roof tiles  | 3 tons         |
| 3     | Construction applications<br>- insulating concrete<br>- loose fill insulation | ½ ton<br>½ ton |
| 4     | Horticultural applications  | ½ ton          |
| 5     | Industrial applications   | ½ ton          |
| 6     | Lightweight concrete  | 1 ton          |
| Total |   | 10 tons        |

### 2.2.2 Phase I Test Plan (Task 1.1)

At the outset of the project a detailed test plan (Task 1.1) was developed and used throughout Phase I as a guide for planning and implementation of the project goals. This consisted of laboratory-scale confirmatory tests, followed by pilot-scale production runs which were planned with the objective of utilizing all of the size fractions of slag to meet the varying size and unit weight requirements of the targeted commercial applications.

The laboratory confirmatory studies were conducted at Fuller Company and Silbrico Company facilities using various slag samples that had been processed for char removal, as identified in Table 2.

**Table 2. Laboratory-Scale Confirmatory Tests Planned**

| Slag/Size Fraction                                 | Product    | Unit Wt, lb/ft <sup>3</sup> | Test Batches |
|--|------------|-----------------------------|--------------|
| Slag I (used as the primary slag for bulk testing) |            |                             |              |
| 10 x 50M (advance sample)                          | Fine LWA   | 30-50                       | 8            |
| 1/4" x 10M   | Coarse LWA | 50                          | 2            |
| 10 x 50M   | Fine LWA   | 30-50                       | 6            |
| 10 x 50M   | Fine LWA   | <12                         | 6            |
| Minus 50M, pelletized                              | Coarse LWA | 50                          | 4            |
| Slag II (used for confirmatory testing)            |            |                             |              |
| 1/4" x 10M   | Coarse LWA | 50                          | 2            |
| 10 x 50M   | Fine LWA   | 30-50                       | 2            |
| 10 x 50M   | Fine LWA   | < 12                        | 4            |
| Minus 50M, pelletized                              | Coarse LWA | 50                          | 2            |

A list of production runs planned for pilot-scale operations is given in Table 3. The equipment used for these runs included a 3-ft diameter x 30-ft long kiln, a 1-ft diameter x 15-ft long kiln, and a 6-inch diameter fluidized bed calciner.

**Table 3. Pilot-Scale Production Runs Planned**

| <b>Production Run</b>     | <b>Slag/Size Fraction</b>      | <b>Equipment</b>     | <b>Product Target Unit Wt, lb/ft<sup>3</sup></b> |
|---------------------------|--------------------------------|----------------------|--|
| <b>Slag I</b>             |                                |                      |  |
| 1A                        | +10M                           | 3' x 30' rotary kiln | 50   |
| 1B                        | +10M                           | "                    | 40   |
| 1C                        | +10M                           | "                    | Minimum possible                                 |
| 2A                        | 10 x 50M                       | "                    | 50   |
| 2B                        | 10 x 50M                       | "                    | 40   |
| 2C                        | 10 x 50M                       | "                    | Minimum possible                                 |
| 2D                        | +10M & 10 x 50M (50:50 mix)    | "                    | 40   |
| 3A                        | Slag/clay (80:20 mix)          | 1' x 15' rotary kiln | 30   |
| 3B                        | Slag/clay (80:20 mix)          | "                    | 40   |
| 4A                        | Slag/clay (50:50 mix)          | "                    | Minimum possible                                 |
| 4B                        | Slag/clay (50:50 mix)          | "                    | 30   |
| 4C                        | Slag/clay (50:50 mix)          | "                    | 40   |
| <b>Clay (Control Run)</b> |                                |                      |  |
| 5A                        | 100%                           | "                    | 30   |
| 5B                        | 100%                           | "                    | 40   |
| 5C                        | 100%                           | "                    | Minimum possible                                 |
| <b>Slag II</b>            |                                |                      |  |
| 6A                        | +10M                           | "                    | 30   |
| 6B                        | +10M                           | "                    | 40   |
| 6C                        | +10M                           | "                    | Minimum possible                                 |
| <b>Slag I</b>             |                                |                      |  |
| 7A                        | ¼ x 10M & 10 x 50M (50:50 mix) | 6" fluidized bed     | 40*  |
| 7B                        | ¼ x 10M & 10 x 50M (50:50 mix) | "                    | 40   |
| 7C                        | ¼ x 10M & 10 x 50M (50:50 mix) | "                    | Minimum possible                                 |

\* Without fuel injection in the bed.

**Project Slag Samples.** The following slag samples were used to implement the test plan:

- Slag I: An advance sample of slag was collected to conduct laboratory testing of the various unit operations prior to collecting a bulk sample.
- Slag I: A bulk sample (~20 tons) of the same slag was collected and used for the entire test program. The source of Slag I cannot be disclosed as the supplier requested confidentiality.
- Slag II: A sample of another slag (1-ton) was used to conduct confirmatory testing to demonstrate the reproducibility of the Slag I results. Slag II, derived from an Illinois coal feedstock, was added at the request of the ICCI. It was obtained from the Tennessee Valley Authority (TVA) gasification facility at the National Fertilizer Development Corporation (NFDC) located in Muscle Shoals, AL.

- Samples of expansible clays used for commercial production of LWAs were collected for use as control samples and as binders for slag fines.

**Project Equipment.** Arrangements were made to use the following equipment for the project test work at various locations:

- Slag processing equipment for char removal at a rate of 100-400 lb/hour
- Screening equipment to screen prepared slag into three sizes at a rate of 50 lb/hour
- Extruder to pelletize 50M x 0 slag fines into 3/4" pellets at 50 lb/hour
- Granulator to crush the slag or pellets to the required particle size at 50 lb/hour
- Other laboratory equipment (crushers, etc.) used in a semi-batch mode
- Direct-fired rotary kiln (1-ft dia. x 15-ft) with baghouse and off-gas analysis to process slag at a rate of 50-100 lb/hour
- Direct-fired rotary kiln (3-ft dia. x 30-ft) with baghouse and off-gas analysis to process slag at a rate of 500-1000 lb/hour
- Fluidized bed calciner (6-inch diameter) with baghouse.

**Project Team.** The project team consisted of Praxis Engineers, Inc., Fuller Company, Pennsylvania State University, and Texaco's Montebello Research Laboratory. Praxis, the prime contractor, was responsible for overall program management, test planning, implementation, and data analysis. Fuller Company, an established pyroprocessing equipment and process development company, performed all work relating to testing and production of slag-based lightweight aggregates. Pennsylvania State University prepared the raw slag for pyroprocessing by screening it and removing its char component. Texaco's Montebello Research Laboratory evaluated the potential for recycling the recovered char to the gasifier and also assisted in estimating slag disposal costs. In addition, a number of potential users, such as roof tile and block manufacturers, as well as LWA and ULWA manufacturers, participated in the project.

### 2.3 Production of Lightweight Aggregates from Slag (Task 1.2)

Under Task 1.2, production of lightweight aggregates from slag was demonstrated using two slag samples generated from two different coal feedstocks. These samples are identified as Slag I and Slag II in this report. Prior to collecting the bulk sample of Slag I, a small advance sample was collected and processed for char removal, to reconfirm Praxis process for slag/char separation. The char-free Slag I was then tested for its expansion characteristics using a laboratory muffle furnace. After confirming all of the processing steps, we collected a 20-ton sample of Slag I. This sample was subjected to char separation and pyroprocessing to produce lightweight aggregates at the pilot scale. These aggregates were then used to make a number of end-use applications which were subjected to extensive applications-oriented testing. Separately, a 1-ton sample of Slag II, which was available from a previous project, was used for confirmatory purposes.

#### 2.3.1 Slag/Char Separation

Entrained-flow coal gasifiers generate a solid waste (slag) which is derived from the mineral matter in the coal. Typically, slag contains 15-25% carbon (termed char) which originates from

unconverted carbon in the coal. The carbon in the slag is a major hindrance to its commercial utilization in most applications. Prior to the subject project, Praxis Engineers developed a process to remove the char from slag generated from entrained-flow gasifiers, thus producing a carbon-free slag which can be used in a number of high-volume applications such as aggregate in cement concrete and road construction, and feed material for lightweight aggregate production. The recovered char may also be blended with coal and utilized as a fuel for power generation or recycled to the gasifier.

The Praxis proprietary slag/char separation process consists of the following processing steps:

1. *Praxis Single-Stage Slag/Char Separation:* A single slag/char separation step for processing raw slag from various gasifiers is capable of recovering a char-free slag product containing <1% carbon and a char product containing 30-50% ash. Both of these products are commercially usable.
2. *Praxis Two-Stage Slag/Char Separation:* For certain applications, a higher level of slag/char separation may be desired. This can be achieved by using a two-stage process consisting of the single-stage process mentioned above, followed by flotation processing of a portion of the stream for secondary separation and recovery of slag to further improve the char grade. However, in many cases the costs of flotation may not justify the use of the two-stage process.
3. *Dewatering of Char-Free Slag Product:* Because slag is a glass-like material, it is relatively easy to dewater using conventional mechanical dewatering equipment. In addition, further moisture reduction occurs by means of natural drainage when it is placed in storage piles or bins. This achieves product moisture levels corresponding to typical commercially available wet-screened sand or fine aggregates.
4. *Dewatering/Handling of Recovered Char Product:* Since the recovered char is assumed to be recycled to the gasification process for this site, it does not need to be dewatered. It is therefore retained in slurry form and mixed with the new coal feed to the wet grinding circuit. However, if it is to be sold or transported off site for utilization, it can be dewatered using conventional mechanical dewatering equipment.

Using the procedure finalized during laboratory testing with the advance sample, the bulk sample of Slag I was processed for char removal using the Praxis single-stage process. Table 4 presents the slag/char separation results for Slag I. The results are presented for the three size fractions (+10M, 10 x 50M, and 50M x 0) into which the slag would be screened prior to pyroprocessing to produce lightweight aggregates. The raw slag was also screened into the same size fractions for ready comparison. The weight recovery of the char-free slag (termed prepared slag) was 68.2%. The ash content of the recovered char was 100%, confirming that it was carbon-free. The char product, representing 31.8% of the raw slag feed, contained 50.3% carbon. In comparison, the advance sample contained 84.9% carbon and char-free slag recovery was 74.3%, with char accounting for 25.7% at 45.1% ash (or 54.9% carbon). The char can be further upgraded as discussed in the next section. These results compare well with those obtained using the advance

sample. Identical results were obtained for char separation tests using Slag II, thus confirming that the slag/char separation process is applicable to other slags.

**Table 4. Slag/Char Separation Results for Slag I**

| Size     | Raw Slag<br>(100%) |       | Prepared Slag<br>(68.2%) |       | Recovered Char<br>(31.8%) |      |
|----------|--------------------|-------|--------------------------|-------|---------------------------|------|
|          | Wt%                | Ash%  | Wt%                      | Ash%  | Wt%                       | Ash% |
| ¼" x 10M | 31.9               | 100.0 | 38.8                     | 101.1 | 0.1                       | 34.5 |
| 10 x 50M | 38.3               | 81.7  | 44.1                     | 100.9 | 36.4                      | 31.9 |
| 50M x 0  | 29.9               | 83.6  | 17.1                     | 101.3 | 63.5                      | 60.9 |
| Total    | 100.0              | 84.9  | 100.0                    | 101.0 | 100.0                     | 50.3 |

While the +50M fractions of Slag I were pyroprocessed in the form of discrete particles to make lightweight aggregates, the 50M x 0 char-free fines were mixed with a clay binder and pelletized (extruded) to produce coarse aggregates before pyroprocessing. This approach was designed to ensure that all of the slag could be utilized, and it was confirmed by laboratory-scale tests. Based on these results, tests at the pilot scale were planned accordingly for both slag samples.

### 2.3.2 Evaluation of Char as By-Product

The objective of this task was to determine the feasibility of recycling char to the gasifier and/or using it as a kiln fuel during slag expansion. Alternatively, the char could be fired in a utility boiler by blending it with power plant coal.

**Upgrading Char.** The objective of this subtask was to generate a char concentrate containing less than 20% ash (i.e., 80% carbon) from the char recovered from the slag. Since the char recovered from the physical separation operation has an ash content of approximately 40-50%, this step is designed to enhance the value of the char and make it more acceptable as a fuel in kiln processing or as a gasifier recycle stream. Samples of char generated from the slag/char separation step were processed for further ash rejection using froth flotation. Initial tests were conducted to establish flotation conditions and collector and frother requirements. Using these conditions, additional quantities of char were processed to produce a 50-lb char flotation concentrate containing 30% ash. A portion of the product was evaluated as a recycle stream to the gasifier and the remainder was evaluated as a fuel during slag expansion, as summarized below.

**Evaluation of Char as Fuel during Slag Expansion.** A char sample containing 50% ash (50% carbon) was subjected to thermogravimetric (TGA) analysis. This test helps identify heat release from the char as a function of temperature. It was observed that significant weight loss begins to occur at 600°C (1110°F), and a residual weight of 60% is recorded at about 750°C (1380°F). Since the char sample contains 50% ash, little carbon remains in the sample at this temperature. Therefore, heat release from the char occurs between 600 and 750°C (1100 and 1400°F), which is considerably below the expansion temperature of slag at ~850°C (1600°F). Therefore, theoretically, the char could be utilized during slag expansion. This premise was tested in the fluidized bed pilot plant, as described below.



Samples of as-recovered char containing 50% ash and upgraded char containing 30% ash were tested by Fuller as fuels during slag expansion in the fluidized bed expander. This system was considered more challenging than the rotary kiln, in which the char can be mixed with the kiln coal feed. In the fluidized bed expander, direct injection of the char as fuel did not produce any noticeable heat contribution to the process as the reaction kinetics of the relatively coarse particles were insufficient to oxidize the carbon in the short residence time offered by the process. Further testing using a pulverized char sample (>90% minus 200 mesh) was conducted but the results were inconclusive because we experienced problems with plugging of the char feed tube during the time allotted for these tests. Nevertheless, the system did operate for a short period of time prior to plugging. It is anticipated that further particle size reduction of the char would facilitate its use as a fuel in the fluidized bed.

**Evaluation of Char for Gasifier Recycle.** Two char samples, containing 50% ash and 30% ash respectively, were provided to Texaco for gasification evaluation at their Montebello Research Laboratory. Their preliminary conclusions were that blending char with the coal slurry feed in small concentrations (5-10%) would not pose any problems. However, they recommended testing the process steps at a larger scale to understand the feed rate impacts on the gasifier.

### **2.3.3 Pilot Production of Lightweight Aggregates from Slag**

The slag samples were processed to produce lightweight aggregates using patented technology<sup>3</sup>. Two commercially available equipment systems were employed: direct-fired rotary kilns (of two sizes) and a fluidized bed expander, developed by Fuller for calcining pelletized fine dust particles.

**Pilot Operation of Direct-Fired Rotary Kilns for Slag Expansion.** Two direct-fired rotary kilns (1-ft diameter x 15-ft and 3-ft diameter x 30-ft) were commissioned at the Fuller Company test site, which was used to conduct expansion tests and produce SLA at the pilot scale.

The smaller rotary kiln was capable of operating at a feed rate of 100 lb/hour, and the larger kiln can feed up to 1000 lb/hour. However, the feed system was set to deliver a slag feed rate of 50 lb/hour for the smaller kiln and 500 lb/hour for the larger kiln. Feed rates that are half the rated capacity of the pilot kilns were selected for the same quantity of slag feed because they provide sufficiently long operating times to generate the required operating data, including the temperature vs. density relationship. Fuel Oil No. 2 was selected as the kiln fuel for this program. However, coal could be used in a commercial kiln to lower energy costs. The kiln fuel oil burner was adjusted to obtain a starting hot zone temperature of 1500°F (816°C).

The smaller kiln was used for pyroprocessing of slag/clay pellets. Its vibratory feed system was set at a delivery rate of 50 lb/hour using 80/20 slag/clay pellets made from minus 50M fines using Slag I. Operating information was collected and recorded for each production run or phase, defined as the expansion of a single size fraction to produce a product of a specific unit weight.

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<sup>3</sup> Patents on the "Utilization of Slag from Coal Gasification Systems" were granted jointly to Vas Choudhry of Praxis Engineers, Inc., Seymour B. Alpert of the Electric Power Research Institute (EPRI), and Donald Meisel of Texaco: European Patent No. 90121365.2, awarded 13 December 1990; U.S. Patent No. 5,091,349, awarded 25 February 1992.

A schematic of the pilot plant consisting of the feed system, kiln, burners and ceramic filters is shown in Figure 2.

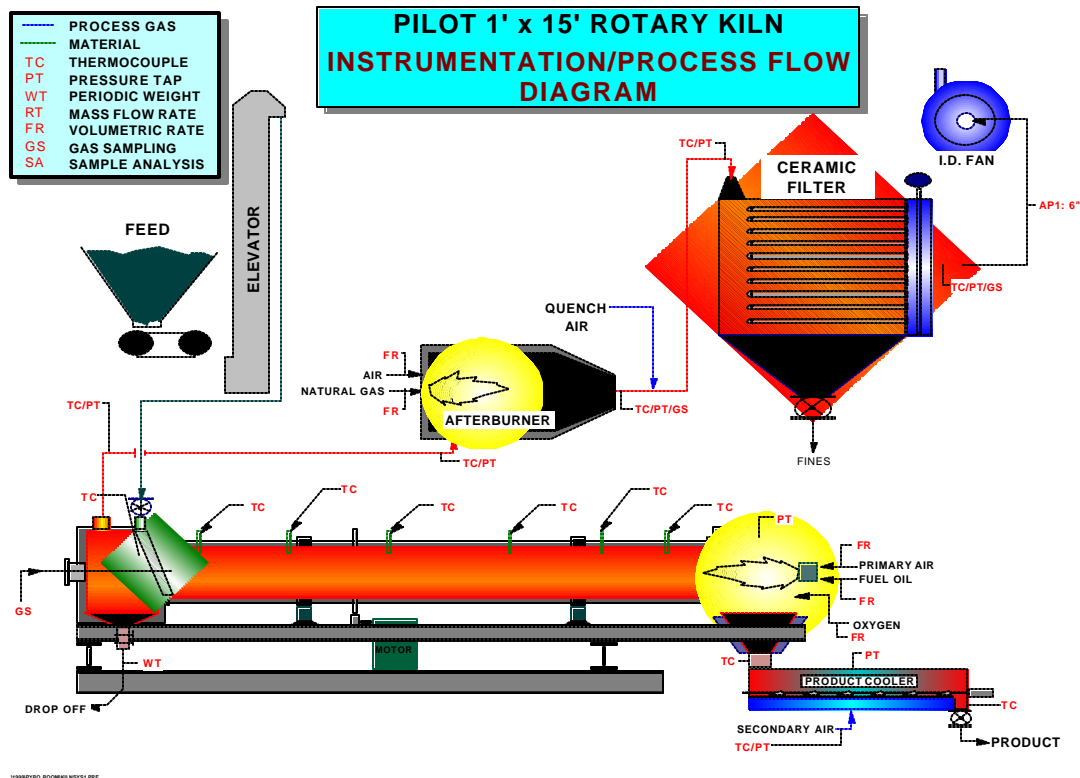


Figure 2. Schematic of Direct-Fired Rotary Kiln Pilot Plant

The following materials were used as feeds to the rotary kiln:

- 1/4" x 10M slag fraction to produce fine structural aggregates
- 10 x 50M slag fraction to produce roof tile aggregates
- Extruded 50M x 0 slag fines mixed with 20% by weight of a clay binder to produce coarse structural aggregates
- Extruded 50M x 0 slag fines using a 50/50 slag/clay blend.
- A control sample of extruded clay to produce conventional LWAs.

Pilot testing was started using the 1/4" x 10M size fraction of Slag I in the large kiln. After allowing the operating conditions to stabilize for 30-60 minutes, product samples were taken every 15 minutes for measurement of the unit weight. Since the unit weight of the product is a function of the kiln hot zone temperature, the latter was adjusted as necessary to obtain a product of the desired unit weight. Temperature changes were generally limited to 15-20°F increments and maintained for 60 minutes. This procedure was followed, increasing the temperature until the desired product unit weight was achieved. Steady-state conditions were maintained until all of the slag was processed. The product unit weight was measured every 30 minutes as part of operational quality control. All operating conditions were recorded every 30 minutes.

Operation of both kilns was successful and no problems were encountered when changing the feed materials and product unit weights as a function of temperature. Large quantities of lightweight products ranging in unit weight between 22 and 50 lb/ft<sup>3</sup> were made in the kilns from different size fractions of Slag I. The kiln system stack flow was monitored continuously for SO<sub>2</sub>, NO<sub>x</sub>, CO, CO<sub>2</sub>, and O<sub>2</sub> content, as discussed in Section 2.3.3.

**Pilot Operation of Fluidized Bed Expander for Slag Expansion.** The fluidized bed expander was developed by Fuller Company for calcining pelletized fine dust particles. In this system, high-temperature combustion products are bubbled through a bed of the feed material. Additional fuel (Fuel Oil No. 2) is injected directly into the fluidized bed immediately above the air distribution plate. The excellent mixing and thermal transfer characteristics of the fluidized bed provide a uniform thermal profile through the bed without formation of a high-intensity flame. The superficial fluidization velocity is maintained at a level not exceeding 110% of the minimum fluidization velocity to generate an actively fluidized bed while minimizing particle entrainment and particle attrition. This mechanism of combustion provides excellent bed temperature control, maximizes system capacity, and has the added benefit of reducing the formation of nitrous oxides. Also, this system has no rotating parts and its high level of insulation prevents energy losses. For these reasons, we believe the fluidized bed expander would be the preferred method for producing LWAs from slag.

A 6-inch fluidized bed expander was used to produce expanded slag in both discrete particle and pelletized form. Products with unit weights as low as 16 lb/ft<sup>3</sup> were produced from +50 mesh Slag I. Granulated Slag I/clay pellets and Slag II/clay pellets (generally 4 x 20 mesh) were processed to produce LWA products with a minimum unit weight of 30 lb/ft<sup>3</sup>.

### **2.3.3 Data Analysis of Slag Preparation and Expansion**

Under Task 1.3, material balances were developed around the kiln and fluidized bed operations. Expanded slag samples were collected, weighed in separate 55-gallon drums for each production run, and stored for later use. Operational material losses were between 3 and 8%, with the lower end of the range occurring for the smaller kiln. During operations, a sample of each production run was taken and analyzed for particle size distribution and unit weight.

**Rotary Kiln Operating Conditions.** Table 5 provides a summary of the production runs from the kiln operation. Similar operating conditions were used for the fluidized bed system.

**Table 5. SLA Production Rotary Kiln Operating Conditions**

| <b>Direct-Fired Rotary Kiln Pilot Plant (3-ft x 30-ft System)</b> |                       |      |                       |                 |      |                       |                 |                 |                     |       |      |
|---|-----------------------|------|-----------------------|-----------------|------|-----------------------|-----------------|-----------------|---------------------|-------|------|
| <b>Feed: Slag I</b>   | <b>¼" x 10M</b>       |      |                       | <b>10 x 50M</b> |      |                       | <b>¼" x 50M</b> | <b>Drop-off</b> |                     |       |      |
| Test phase  | 1A                    | 1B   | 1C                    | 2A              | 2B   | 2C                    | 2D              | 2E              |                     |       |      |
| Feed rate, lb/h   | 500                   | 670  | 525                   | 500             | 590  | 590                   | 610             | 610             |                     |       |      |
| Unit wt, lb/ft <sup>3</sup>                                       | 50                    | 40   | 34                    | 50              | 40   | 35                    | 38              | 38              |                     |       |      |
| No. 2 fuel oil, gal/h   | 13                    | 12.2 | 16.2                  | 12.8            | 13.6 | 15.4                  | 13.3            | 13.8            |                     |       |      |
| Hot zone temp., °F  | 1520                  | 1590 | 1925                  | 1585            | 1650 | 1800                  | 1650            | 1685            |                     |       |      |
| Gas analysis  |                       |      |                       |                 |      |                       |                 |                 |                     |       |      |
| Kiln exit, %O <sub>2</sub>  | 12.4                  | 12   | 8.6                   | 12.1            | 11.6 | 9.9                   | 11.9            | 11.6            |                     |       |      |
| Kiln exit, ppm CO   | 3                     | 2    | 2                     | 44              | 71   | 47                    | 27              | 62              |                     |       |      |
| BH inlet, %O <sub>2</sub>   | 17                    | 16.5 | 14.4                  | 17              | 16.4 | 15.7                  | 16.5            | 16.5            |                     |       |      |
| <b>Direct-Fired Rotary Kiln Pilot Plant (1-ft x 15-ft System)</b> |                       |      |                       |                 |      |                       |                 |                 |                     |       |      |
| <b>Feed Type</b>  | <b>80/20 Pellets*</b> |      | <b>50/50 Pellets*</b> |                 |      | <b>0/100 Pellets*</b> |                 |                 | <b>+10M Slag II</b> |       |      |
| Test phase  | 3A                    | 3B   | 4A                    | 4B              | 4C   | 5A                    | 5B              | 5C              | 6A                  | 6B    | 6C   |
| Feed rate, lb/h   | 73                    | 100  | 97                    | 97              | 100  | 98                    | 96              | 96              | 100                 | 100   | 100  |
| Unit wt, lb/ft <sup>3</sup>                                       | 30                    | 40   | 22                    | 30              | 40   | 30                    | 40              | 18              | 30                  | 30-50 | 22   |
| No. 2 fuel oil, gal/h   | 5.91                  | 5.43 | 5.49                  | 5.07            | 4.6  | 4.4                   | 4.2             | **              | 3.3                 | 3.1   | 4.6  |
| Hot zone temp., °F  | 2110                  | 2000 | 2050                  | 1980            | 1900 | 1900                  | 1770            | **              | 166                 | 1645  | 1820 |
| Gas analysis  |                       |      |                       |                 |      |                       |                 |                 |                     |       |      |
| Kiln exit, %O <sub>2</sub>  | 5.7                   | 6.9  | 5.9                   | 7.5             | 9.6  | 8.9                   | 9.7             | **              | 9.5                 | 10.5  | 9.8  |
| Kiln exit, ppm CO   | 74                    | 160  | 112                   | 267             | 10   | 708                   | 602             | **              | >1000               | 406   | 43   |
| Filter, inlet, %O <sub>2</sub>                                    | 12                    | 13.7 | 11.7                  | 12.4            | 13.4 | 13.5                  | 14.1            | **              | 15.6                | 15.8  | 13.3 |

\* Slag I

\*\* Equipment failure.

**Rotary Kiln Emissions.** The emission levels for all phases of the pilot kiln operations from the direct fired rotary kiln are given in Table 6. Emissions of NO<sub>x</sub>, CO<sub>2</sub> and SO<sub>2</sub> were not monitored for the fluidized bed system as emissions levels are expected to be similar to or lower than those in rotary kilns.

The SO<sub>2</sub> emissions are in the range of 13-30 ppm for the discrete particle pilot runs covered by Phases 1A, 1B, and 2A-2D. However, these emissions increased to 38-90 ppm when the extruded slag fines were processed. This reflects the higher sulfur content in the 50M x 0 fines compared to the +50-mesh slag. In addition, the higher temperature required for pellet expansion allowed volatilization of a higher percentage of the feed sulfur. The SO<sub>2</sub> emissions for Phase 3B were 140 ppm, which is unusually high compared to other phases. This is attributed to a fuel rate measurement error. A portion of the SO<sub>2</sub> emissions resulted from the sulfur content of the kiln fuel (Fuel Oil No. 2) which contained 0.25% sulfur. The use of natural gas as the fuel in a commercial plant would provide a means of reducing fuel-related emissions.

NO<sub>x</sub> emissions during the kiln operations were in the range of 25-40 ppm during the entire program and were generally a function of the temperature of operation.

**Table 6. Direct-Fired Rotary Kiln Emissions**

| Phase                     | Material      | Product Rate | Stack Flow | SO <sub>2</sub> |       | NO <sub>x</sub> |        | CO <sub>2</sub> |        |
|---------------------------|---------------|--------------|------------|-----------------|-------|-----------------|--------|-----------------|--------|
|                           |               |              |            | lb/h            | DSCFM | ppm             | lb/ton | ppm             | lb/ton |
| <b>Kiln Feed: Slag I</b>  |               |              |            |                 |       |                 |        |                 |        |
| 1A                        | +10M          | 496          | 1785       | 13              | 0.94  | 33              | 1.716  | --              | --     |
| 1B                        | +10M          | 650          | 1526       | 14              | 0.661 | 35              | 1.187  | 2.3             | 746    |
| 2A                        | 10 x 50M      | 475          | 1815       | 26              | 1.997 | 32              | 1.767  | 2.3             | 1215   |
| 2B                        | 10 x 50M      | 535          | 1811       | 30              | 2.041 | 35              | 1.712  | --              | --     |
| 2C                        | 1/4" x 50M    | 555          | 1829       | 30              | 1.987 | 35              | 1.666  | --              | --     |
| 2D                        | Drop off      | 533          | 1885       | 19              | 1.351 | 35              | 1.788  | --              | --     |
| 3A                        | 80/20 pellets | 62           | 618        | 90              | 18.03 | 40              | 5.761  | 2.8             | 3858   |
| 3B                        | 80/20 pellets | 80           | 702        | 140             | 24.69 | 36              | 4.564  | 2.7             | 3275   |
| 4A                        | 50/50 pellets | 82           | 588        | 60              | 8.648 | 35              | 3.626  | 3.2             | 3172   |
| 4B                        | 50/50 pellets | 82           | 586        | 75              | 10.77 | 35              | 3.614  | 2.9             | 2865   |
| 4C                        | 50/50 pellets | 85           | 546        | 55              | 7.101 | 32              | 2.97   | 2.5             | 2220   |
| 5A                        | 0/100 pellets | 74           | 537        | 40              | 5.834 | 32              | 3.355  | 2.5             | 2508   |
| 5B                        | 0/100 pellets | 72           | 527        | 38              | 5.591 | 32              | 3.384  | 2.4             | 2428   |
| <b>Kiln Feed: Slag II</b> |               |              |            |                 |       |                 |        |                 |        |
| 6A                        | +10M          | 88           | 518        | 10              | 1.183 | 25              | 2.126  | 1.8             | 1465   |
| 6B                        | +10M          | 88           | 505        | 13              | 1.5   | 22              | 1.824  | 1.8             | 1428   |
| 6C                        | +10M          | 88           | 578        | 15              | 1.98  | 33              | 3.132  | 2.3             | 2088   |

**Environmental Acceptability of Expanded Slag Products.** Major environmental issues with regard to utilization of a new aggregate are (i) leachability and (ii) adverse reactivity when blended with other aggregates. A sample of expanded slag was prepared to match the particle size distribution required for lightweight roof tile aggregates and subjected to elemental analysis and RCRA/TCLP testing in accordance with EPA SW-846. The results of the TCLP test are given in Table 7, along with the elemental analysis of the SLA sample used for the test. The SLA sample selected was the "worst case" scenario because extensive size reduction of expanded aggregates is needed to meet the size requirements, thus potentially making it the most leachable SLA product. The results indicated that TCLP leachate heavy metals concentrations, given in mg/l, were considerably lower than the RCRA maximum allowable concentrations.

**Table 7. Analysis of SLA Crushed for Roof Tile Application and TCLP Results**

| <b>Element</b>     | <b>SLA Sample<br/>mg/kg</b> | <b>TCLP Result<br/>mg/l</b> | <b>RCRA TCLP<br/>Max. Allowable Conc.<br/>mg/l</b> |
|--------------------|-----------------------------|-----------------------------|--|
| Antimony (Sb)      | <0.5                        | <0.03                       | 1  |
| Arsenic (As)       | <6.0                        | 0.018                       | 5  |
| Barium (Ba)        | 106                         | 0.5                         | 100  |
| Beryllium (Be)     | <0.7                        | <0.005*                     | 0.007  |
| Cadmium (Cd)       | <0.3                        | <0.02*                      | 1  |
| Chromium (Cr)      | 157                         | <0.03*                      | 5  |
| Lead (Pb)          | <6                          | <0.05*                      | 5  |
| Mercury (Hg)       | <0.007                      | <0.00007*                   | 0.2  |
| Nickel (Ni)        | 26                          | <0.03*                      | 70   |
| Selenium (Se)      | <0.5                        | <0.007*                     | 1  |
| Silver (Ag)        | 1                           | <0.02*                      | 5  |
| Thallium (Tl)      | <0.5                        | <0.005*                     | 7  |
| pH of TCLP Extract |                             | 3.12                        |  |

\* Concentration was below the detection limit.

**Laboratory-Scale Applications-Oriented Testing of SLA.** Laboratory-scale applications-oriented testing of SLA as a substitute for LWA and ULWA was conducted. The SLA aggregates were prepared to meet the particle size and unit weight requirements of selected applications by crushing and blending products of various sizes and unit weights. Procedures describing the mix designs to be used and applicable ASTM standards were developed and provided to selected test laboratories who used them to prepare test specimens, incorporating SLA as a substitute for conventional LWAs and ULWAs.

The applications tested are listed below:

- Structural concrete (three SLA products)
- Lightweight concrete masonry units (lightweight blocks)
- Insulating concrete
- Lightweight roof tile aggregate (three SLA products)
- Loose fill insulation
- Horticultural applications.

### **2.3.4 Economic Analysis of Expanded Slag Production (Task 1.4)**

An economic evaluation was conducted for a hypothetical single facility to first process raw slag for char recovery and then pyroprocess the char-free slag to produce lightweight and ultra-lightweights aggregates of various unit weights, as dictated by demand in local and adjoining markets. This single facility was assumed to be located at the gasifier site to eliminate double handling of slag. It was envisaged that the recovered char could be recycled to the gasifier. The costs of this facility were estimated by developing a process flowsheet based on pilot plant operations data generated during the project, along with process equipment-factored capital cost estimates. For the economic analysis, four scenarios were studied representing two sizes of IGCC facilities (200 MW and 400 MW), each using two process technologies for SLA production, the rotary kiln and fluidized bed calciner. The two scales studied are:

- A plant to process slag generated from a 200-MW gasifier facility, typically using 2,000 tons/day of bituminous coal containing 10% ash, and generating 220 tons/day of slag containing 10% char.
- A plant to process slag generated from a 400-MW gasifier facility, typically using 2,000 tons/day of bituminous coal containing 10% ash, and generating 440 tons/day of slag containing 10% char. This would approximate the feed capacity of a typical commercial LWA plant that currently uses conventional expansible clays.

The slag production economics were conducted using two parallel approaches:

- Comparison of the economics of SLA production vs. slag disposal
- Comparison of the economics of SLA production with the estimated market value of end products that can be made from it.

The market price of SLA was estimated taking into consideration the fact that it would likely command a lower price as a new, unproven material. The sale prices for slag lightweight aggregates were estimated at \$30/ton for block aggregates, \$35/ton for structural aggregates, and \$40/ton for roof tile and ultra-lightweight aggregate applications. Using a product mix based on the percentage of coarse and fine slag, the weighted average price of SLA was estimated at \$34.75/ton. This price was used to evaluate the economics of SLA production.

For purposes of this analysis, a value of \$15/ton was used as the cost of slag disposal, which is in the middle of the \$10-20/ton range indicated for fly ash. Since these avoided costs would provide substantial savings to the gasifier operation, this amount could potentially be made available to the SLA production facility as a tipping fee per ton of slag accepted.

For the rotary kiln processes, the SLA production costs were estimated at \$30.06/ton and \$24.40/ton for the 220 and 440 tons/day capacities (200-MW and 400-MW gasifiers) respectively. These costs are competitive with conventional LWA production costs, which were estimated at \$30.10/ton based on a survey of four operating plants. SLA production costs are considerably lower than the composited market price for such materials, estimated at \$34.74/ton. The payback period for the large rotary kiln system was estimated at less than four years.

The fluidized bed method of SLA production was found to be even more competitive because of lower capital and operating costs. Its production costs were \$26.48 and \$21.87 for the smaller and larger sizes respectively. The payback period for the large fluidized bed system was estimated at under three years.

### **2.3.5 Phase I Final and Other Reports (Task 1.5)**

Topical, financial status, and technical progress reports were prepared in accordance with the Statement of Work and delivered on schedule.

## **2.4 Major Conclusions from Phase I Work**

The primary objectives of the Phase I experimental work were to demonstrate the feasibility of producing lightweight and ultra-lightweight aggregates from a bulk sample of Slag I and to generate a sufficient quantity of expanded slag lightweight aggregates (SLA), ranging in unit weight from 18 to 50 lb/ft<sup>3</sup>, for subsequent applications-oriented testing in Phase II. The technology was also demonstrated for a second slag (Slag II) derived from an Illinois coal feedstock. The goal was to demonstrate the use of all size fractions of slag including the fines, which were mixed with a clay binder to make extruded pellets prior to expansion. Other goals included the collection of engineering data (energy consumption, material balances, and emissions) from pilot plant operations. The specific conclusions based on the work conducted in Phase I are given below.

**Slag Processing for Char Removal.** All the slag samples were successfully processed for char removal. For Slag I, a char-free slag product, termed prepared slag, containing 100% ash was recovered at yields ranging between 66 and 68%. A char product containing 45-54% ash was also recovered and was evaluated as a potential gasifier feed and kiln fuel. The char recovered from the first-stage separation was upgraded successfully to 30% ash (70% carbon) by a second step involving flotation, then evaluated as a gasifier feed material.

**SLA Production in a Direct-fired Rotary Kiln.** Slag expansion using a direct-fired rotary kiln was accomplished in two forms: (i) expansion of coarse (1/4" x 50M) slag in discrete particle form, and (ii) expansion of pellets made from extruded slag fines mixed with an expansive clay binder.

Expansion of the 1/4" x 50M size fraction of Slag I was demonstrated to be feasible in the rotary kiln as a single size interval. The slag could also be expanded in any other size interval within this range to meet the specific requirements of an end product.

Temperature vs. density studies were conducted and product unit weights could be varied in the 30-50 lb/ft<sup>3</sup> range by means of temperature control. It was feasible to further reduce the product unit weight to 20 lb/ft<sup>3</sup> but lower unit weights posed potential fusion problems.

The +10M Slag II sample was expanded to produce a product with a unit weight of 20-30 lb/ft<sup>3</sup> at a temperature of 1450-1500°F.



The expansion temperature was 400-500°F lower than that typically required for expansible clays and shale, which represents significant energy savings.

The objectives of expansion testing of the pelletized slag fines using an expansive clay binder were twofold: (i) to demonstrate the use of clay as a binder, and (ii) to demonstrate that the clay can be blended with slag fines for expansion. Both of these objectives were met. The following conclusions can be drawn from this work:

- Size enlargement of extruded pellets made from minus 50-mesh slag mixed with 20-50% by weight of a minus 20M expansive clay binder was successful. The resulting aggregates had a size of 3/8" which could be controlled as desired.
- The use of higher proportions of slag resulted in lower pellet moisture, which would have a major effect on overall process fuel consumption requirements, with greater use of slag lowering fuel costs.
- The expansion temperature for clay and slag when completely mixed together is lower than that required for clay alone. The firing temperature for the 80/20 and 50/50 slag/clay blends tested is approximately 1800-1900°F, which is higher than the expansion temperature of slag by itself but lower than that of clay. There was no indication of fusion with any of the extruded mixtures fired up to 2000°F.
- Expanded products with unit weights ranging between 27 and 33 lb/ft<sup>3</sup> were produced. The expansion temperature for these samples was nearly 200°F lower than that typically required for conventional expansible clay pellets, which represents considerable energy savings for slag expansion.
- Successful expansion of pelletized slag/clay blends in a 50:50 ratio indicates that these two materials can be blended to produce lightweight aggregates.

**Production of SLA Using Fluidized Bed Expander.** The fluidized bed expansion method was selected to demonstrate the production of lower-unit-weight products (approaching the unit weights of conventional ULWAs) because of its improved energy efficiency and better temperature control since feed particles do not come into contact with a flame. The objectives were to demonstrate the suitability of this expansion method and to test the acceptability of the recovered char as a fuel in the bed. These objectives were met, and the following specific conclusions were drawn:

- The various slag size fractions were expanded in discrete particle form in a pilot-scale fluidized bed expander to produce LWAs with unit weights ranging between 18 and 26 lb/ft<sup>3</sup>.
- Similar results were achieved for extruded granulated slag pellets made from minus 50M slag fines to make minus 10M expanded aggregates.

### 3.0 PHASE II OBJECTIVES AND SCOPE

#### 3.1 Phase II Objectives

The objectives of Phase II were to test the use of expanded slag products in a wide variety of applications with the goal of partially or fully substituting slag lightweight aggregates (SLA) for conventional lightweight materials. The high-value end product applications that were tested are listed below:

Lightweight aggregate applications:

- Lightweight roof tiles
- Lightweight masonry blocks (also known as concrete masonry units or CMUs)
- Structural concrete
- Concrete panels

Ultra-lightweight aggregate applications:

- Insulating concrete
- Loose fill insulation
- Nursery application as a substitute for perlite and/or vermiculite

Relevant cost data for physical and pyroprocessing of slag to produce expanded slag aggregates were gathered for comparison with (i) management and disposal costs for slag or similar wastes, and (ii) production costs for conventional lightweight aggregates which the slag aggregates would replace. In addition, a market assessment was made to evaluate the economic viability of these utilization technologies.

#### 3.2 Phase II Scope of Work

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

##### ***Task 2.1 Test Plan for Applications of Expanded Slags (see also Appendix A)***

This task involved the development of selection criteria and a field test plan for applications of expanded slag. The complete Phase II test plan is included with this report as Appendix A. This plan served as a guide in the selection and implementation of field demonstrations for the most promising expanded slag utilization applications. Field applications were 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 were considered for testing:

Lightweight aggregate applications:

- Lightweight roof tiles made from 40 lb/ft<sup>3</sup> SLA
- Lightweight masonry blocks made from 50 lb/ft<sup>3</sup> SLA
- Structural concrete made from 45-50 lb/ft<sup>3</sup> SLA
- Concrete panels made from 35-45 lb/ft<sup>3</sup> SLA

Ultra-lightweight aggregate applications:

- Insulating concrete made from 26 lb/ft<sup>3</sup> SLA
- Loose fill insulation made from 29 lb/ft<sup>3</sup> SLA
- Horticultural applications made from 15-18 lb/ft<sup>3</sup> SLA.

### ***Task 2.2 Field Studies to Test Expanded Slag Utilization***

Under this task, field testing of the applications identified in Task 2.1 began 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 was to assimilate the data and test results collected during Phase II, 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 was done to 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 was to expand upon the preliminary economic assessment of expanded slag utilization conducted during Phase I. The economics was 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 an existing lightweight aggregate facility (off-site production).

The impact of the avoided costs of slag disposal on the economics of SLA production were also evaluated. Slag utilization data and product samples were 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 were studied.

### ***Task 2.5 Separation of Char from Slag for Tampa Electric Company IGCC Plant***

The objective of this effort was to conduct laboratory-scale testing to process the slag from the Tampa Electric Company (TEC) IGCC facility for char removal, develop a conceptual design for a char removal facility, and present the results to the plant engineers.

### ***Task 2.6 Testing of Slag as Raw Material Additive in Portland Cement Kiln Feed***

The objective of this task was to conduct laboratory studies to test and evaluate the potential for using slag as a partial replacement in portland cement kiln feed. This included testing to evaluate the impact of various levels of slag addition on the clinker temperature.

### **Task 2.7      *Utilization of Slag as Raw Material in Portland Cement (Pilot Testing)***

The objective of this task was to conduct pilot-scale tests to confirm the laboratory-scale test results from Task 2.6. The kiln product was evaluated as cement after grinding.

### **Task 3.0      *Final and Other Reports***

Project reports were submitted on a quarterly basis, as required.

All project reports, including quarterly technical and financial status reports, were submitted in a timely fashion. Topical Report No. 1, which constituted a summary of the Phase I work, was submitted in May 1996.

The subject report represents the Final Technical Report for the project. It presents a compilation of the data generated and collected during the project, including a comprehensive description of the results achieved, consistent with the Reporting Requirements. The report includes the original hypothesis of the project and presents the investigative approaches used, complete with problems encountered or departures from the planned methodology, and an assessment of their impact on the project results.

## **3.3      Project Methodology**

The methodology followed in implementing the project was to:

- Build on the developmental work done by Praxis under previous EPRI-funded projects to produce LWA from slag, ICCI-funded projects to produce ULWAs from slag, and internal studies to identify potential applications for expanded slag aggregates.
- Obtain the participation of potential users and producers of SLA products throughout the project in order to familiarize them with the capabilities of SLA.
- Seek the involvement of slag generators in order to keep them informed of the potential for utilizing slag as an alternative to disposal.
- Use conventional LWA production methods and equipment in order to minimize the process development and commercialization time frame and increase product acceptability to LWA and ULWA manufacturers and end-use industries.
- Conduct laboratory-scale testing using industry and ASTM methods for each application to develop techniques for substituting expanded slag for the conventional materials.
- Work with end-users to ensure that the development approach would be acceptable to them.
- Work at the manufacturer's facility for small-scale testing, where possible. Review the results jointly to finalize processing techniques before conducting final large batch runs.

- Have the final products tested by outside laboratories, where possible.
- Conduct economic evaluation with the involvement of industrial partners.
- Periodically present project results to DOE, EPRI, ICCI, and potential end users, as well as to the IGCC facilities which provided the slag samples. DOE project personnel were invited to such meetings.

## 4.0 RESULTS AND DISCUSSION OF PHASE II WORK

### 4.1 Test Plan for Applications Testing of Expanded Slags: Field Studies (Task 2.1)

At the beginning of Phase II, a Test Plan was developed for conducting a systematic laboratory evaluation of target applications for the expanded slags produced in Phase I. As a first step, selection criteria for these applications were developed by Praxis with input from potential users of the aggregates and testing laboratories to select appropriate field demonstration tests. The criteria included the following:

- There should be a close match between the physical properties of the SLA and those of the target substitute materials
- Market penetration in the targeted applications should be cost driven
- The application should not involve undue perceived liability.

Applications that met these criteria were selected for testing of the suitability of replacing conventional lightweight aggregates (LWAs) with expanded slag aggregates. They are listed below:

- Lightweight roof tiles
- Concrete blocks
- Structural concrete
- Waterproof panels (concrete panels).

The following applications were selected for testing of the suitability of replacing conventional ultra-lightweight aggregates (ULWAs) with expanded slag aggregates:

- Insulating concrete
- Loose fill insulation
- Horticultural applications.

Upon selecting the applications for testing, a field test plan entitled “Test Plan for Applications of Expanded Slags (Task 2.1)” was developed. It is attached with this report as Appendix A. As the testing advanced, the test plan was modified where needed based on test results, the performance of the expanded slags, and suggestions by potential commercial users. In many cases potential users of the expanded slag were involved in the laboratory evaluation, and a major portion of the testing was done in their respective laboratories. This helped in making rapid adjustments to the methodology of using expanded slag aggregates in place of conventional aggregates.

## 4.2 Field Studies to Test Expanded Slag Utilization (Task 2.2)

Upon completing the laboratory evaluation, a number of applications were selected for field testing. The evaluation activities covered in this task were:

- Development and optimization of structural and non-structural lightweight concrete applications using SLA
- Evaluation of SLA for lightweight roof tile manufacturing
- Manufacture and testing of masonry blocks using SLA
- Investigating the demonstration of other applications
- Disposal of residual samples.

The following selection criteria were established to select demonstration applications:

- Close match between properties of SLA and those of the conventional materials
- Availability of user's personnel for consultations to adapt SLA for their application
- Availability of plant and willingness to manufacture finished products using SLA
- Low potential risks associated with the application.

Field studies were conducted for the following applications:

- Lightweight block production
- Concrete panels
- Horticultural applications.

Data evaluation and the results of both laboratory and field studies are reported together in the following section.

## 4.3 Data Analysis of Commercial Utilization of Expanded Slags (Task 2.3)

The work under this task involved two major activities:

- Performance of expanded slag in the conventional applications
- Technical viability of the SLA products corresponding to conventional LWAs and ULWAs.

The first activity or subtask involved laboratory studies to evaluate whether expanded slag products meet the basic requirements for a particular application (e.g., bulk density, compressive strength, drying shrinkage, etc. as applicable). This involved making slag-based mixes with minimal addition of conventional materials to enable us to study the properties of expanded slag. Specimens made for test purposes were 2" diameter x 4" long cylinders or 2" cubes. Preparation of these specimens required small batches of concrete to be made, which is cheaper and faster than preparation of larger batches. However, this entails some variability in results between one batch and another. Final testing was done using larger 4" wide x 4" high x 12" long specimens which provide more consistent results.

The second activity involved evaluation of more general issues such as whether the expanded slag meets the functional requirements of the application, whether it is amenable to the end user's manufacturing setup, and whether the end product can match the performance of the corresponding conventional products. The results of this evaluation are discussed for each application in this section.

#### **4.3.1 Laboratory Evaluation of SLA for Roof Tile Application**

The objective of this test program was to develop mix designs to produce sand and SLA-based cement concretes with compressive strengths in the 2500-4000 psi range and unit weights in the 115-105 lb/ft<sup>3</sup> range. The principal use of these high-strength cement-based materials would be as a replacement for 55 lb/ft<sup>3</sup> LWA to make lightweight roof tiles. Conventional concrete roof tiles are attractive for a number of reasons, including the fact that they are fire-safe and have a conservative life expectancy of 50 years. They can also be fabricated in large quantities and in various colors. However, traditional concrete is too heavy to be used in re-roofing applications without the addition of costly heavy bracing in the roof structure. Using LWA, it is possible to manufacture roof tiles with reduced unit weight, thereby eliminating the need for additional support or bracing and reducing construction costs.

We tested the use of SLA I as a replacement for LWA by running tests with varying proportions of cement. We determined that, with the use of chemical additives, it is possible to produce cement concretes with a range of compressive strengths and unit weights. The SLA I samples that were tested are identified in Table 8, along with a control sample of commercially available structural aggregate. As indicated in the table, 3/4" coarse SLA aggregates produced by expanding 50/50 extruded slag fines/clay pellets were tested at three strength levels (complete matrix) whereas the other samples were tested at only one level of cement.

**Table 8. Cement Levels to Test SLA I as Structural Aggregate**

| <b>SLA Products Tested</b>                         | <b>Tentative Cement Level, Sacks/Yard<sup>3</sup></b> |
|--|---|
| (i) 3/4" coarse SLA I (50/50 slag-clay pellets)    | 5½, 6½, and 7½  |
| (ii) 1/4" x 50M SLA I crushed as 3/8" combined LWA | One level of cement                                   |
| (iii) 3/4" expanded clay pellets (Control)         | One level of cement                                   |

Exploratory tests were conducted to establish appropriate sand, water, and cement requirements in order to achieve the target mechanical properties. The strength and unit weight of the resulting concrete specimens were measured and, based on these results, final mix designs were developed for the various expanded slags.

The test method for compressive strength specified in ASTM C-109 was used to test the specimens. The three aggregates consisted of 100% expanded slag, 50/50 expanded slag/clay, and a 100% clay control sample provided by a commercial roof tile manufacturer. The 100% slag aggregates and the 50/50 slag/clay aggregates were sized to match the size distribution of the clay aggregates used by the roof tile manufacturer. Typically, a roof tile mix uses a cement-to-aggregate ratio of 1:2.5, along with various additives such as accelerators and superplasticizers. In order to mimic products available in the market for purposes of comparison,



two different kinds of accelerators and a common commercial superplasticizer (Mighty 150) were evaluated. The accelerators tested were calcium chloride dihydrate ( $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) and sodium silicate. These were used to enable the products to set quickly and therefore increasing the manufacturing rate. Superplasticizers are used to reduce water requirements, thereby increasing strength.

During the testing, all of the aggregates were used in their saturated surface dry (SSD) condition as defined by ASTM. The moisture content of the three aggregates in their SSD condition was measured and recorded.

Because the specific mix formulations used by roof tile manufacturers are considered proprietary information and were therefore not available to us, we conducted many experiments with varying amounts of accelerator, superplasticizer, and water-to-cement ratios with the goal of obtaining the highest 7-day compressive strength without using excessive additives. Three 2" x 2" x 2" mortar cubes were cast and cured in a wet box (relative humidity of ~70%) for 2 hours and then steam-cured at ~60°C for 4 hours. The cubes were demolded and returned to the wet box for further curing to 7 days. The cubes were then weighed and broken in compression. A summary of the formulations and 7-day compressive strengths is presented in Table 9.

**Table 9. Formulations and 7-Day Compressive Strength of Roof Tile Samples**

| Aggregate Type  | $\text{CaCl}_2 \cdot \text{H}_2\text{O}$<br>Wt% of<br>cement | Super-<br>plasticizer<br>Wt% of cement | Mortar<br>Unit Wt<br>lb/ft <sup>3</sup> | Water/<br>Cement<br>Ratio | SSD<br>% | 7-day<br>Compressive<br>Strength, psi |
|-----------------|--|--|---|---------------------------|----------|---------------------------------------|
| SLA             | 2  | 5.5                                    | 90.5                                    | 0.26                      | 18       | 668                                   |
| SLA             | 2  | 5                                      | 92.3                                    | 0.29                      | 18       | 934                                   |
| SLA             | 2  | 2                                      | 92.6                                    | 0.32                      | 18.5     | 2303                                  |
| SLA             | 2  | 2                                      | 93.3                                    | 0.35                      | 18.5     | 2806                                  |
| SLA             | 2  | 2                                      | 96.8                                    | 0.38                      | 17.4     | 2028                                  |
| SLA             | 2  | 2                                      | 97.1                                    | 0.41                      | 17.4     | 1743                                  |
| SLA             | 2  | 1.5                                    | 98.3                                    | 0.38                      | 17.4     | 2650                                  |
| SLA*            | 2  | 1.5                                    | 101.7                                   | 0.38                      | 18       | 2432                                  |
| Control**       | 2  | 2                                      | 109.3                                   | 0.38                      | 17       | 2011                                  |
| Control**       | 2  | 2                                      | 108.7                                   | 0.41                      | 17       | 2802                                  |
| Control**       | 2  | 2                                      | 105.3                                   | 0.45                      | 17       | 3390                                  |
| Control**       | 2  | 2                                      | 105.2                                   | 0.50                      | 17       | 3106                                  |
| 50/50 slag/clay | 2  | 1.5                                    | 105.2                                   | 0.35                      | 26       | 2303                                  |
| 50/50 slag/clay | 2  | 1.5                                    | 101.9                                   | 0.38                      | 26       | 1917                                  |
| 50/50 slag/clay | 2  | 1.5                                    | 101.8                                   | 0.41                      | 26       | 1736                                  |

\* 1% sodium silicate was added for this test.

\*\* Control aggregate was produced at a commercial kiln and provided by a roof tile manufacturer.

The highest 7-day compressive strength for the expanded slag specimens was 2806 psi, which is 83% of the highest strength obtained for the expanded clay samples. Visual inspection of the crushed SLA-based cubes revealed that the cement/aggregate interface was sound and that

failure was chiefly due to aggregate breakage. This was confirmed by the specimens made from 50/50 slag/clay. The unit weight of the 100% expanded slag specimens ranged between 91 and 98 lb/ft<sup>3</sup>, that of the 50/50 specimens between 102 and 105 lb/ft<sup>3</sup>, and that of the 100% clay control specimens between 91 and 101 lb/ft<sup>3</sup>.

These experiments showed that the mechanical behavior of the samples is greatly affected by the water/cement ratio but not by the type of accelerators used. Typically, in cement systems, lowering the water/cement ratio improves strength if care is taken to keep the mix workable. However, in the case of the expanded slags, the water/cement ratio had to be kept relatively high (>0.35) in order to have the cement paste coat all the particles and keep the structure together.

Additional tests were conducted to measure the 28-day compressive strength of SLA in the roof tile application. In these tests, a total of six different aggregates were used: an aggregate supplied by a lightweight roof tile manufacturer, expanded slag, 50/50 slag/clay, 80/20 slag/clay, expanded clay from the pilot test program, and an expanded clay aggregate sample provided by a leading LWA manufacturer. The commercial samples and the expanded clay produced during the pilot run were used as controls for purposes of comparison. The aggregates were first immersed in deionized water and allowed to soak for 4-6 hours. After soaking, the standing water was decanted and the aggregates were allowed to dry in ambient air until they were in a saturated surface dry condition, as defined by ASTM. The moisture content of the aggregates was measured by weighing a sample of the aggregate before and after drying in a 105°C oven overnight.

The basic mix design included the use of 1.5 ml of Mighty 150 superplasticizer per 100 gm of cement, 2wt% CaCl<sub>2</sub>·2H<sub>2</sub>O (relative to the amount of cement used), and just enough water to create a mix with a slump of 0-1. The aggregate/cement ratio was kept constant at 2.5:1 by weight. A common type I cement was used. The amounts of superplasticizer and accelerator were fixed while the water/cement ratio (w/c) was varied, depending on the aggregate used, until the desired slump was achieved. All mixing was done according to ASTM C 305 for mortars. Based on the 7-day compressive strength results (shown in Table 9), two final mix designs for each aggregate were selected. Six 2" x 2" x 2" specimen cubes of each mix design were cast. During molding, care was taken to ensure that the mixtures were well compacted. After mixing, the molds were placed in a covered wet bucket at room temperature for four hours, after which the specimens had sufficient strength to be demolded. They were then placed in a stainless steel tray with holes in the bottom, which was in turn placed in an unsealed plastic bag. The whole assembly was placed in a steam bath with a steam temperature of 60°C. The plastic bag prevented any hot water from dripping onto the cubes and eroding the samples. Since the bag was not sealed, there was no possibility of hydrothermal reactions. The cubes were steamed for four hours, then removed from the steam bath and further cured at room temperature in a covered wet box. Three cubes of each mix design were tested at 7 days and 28 days for compressive strength to failure according to ASTM C 109. Prior to testing, each cube was weighed and the average weight was used to calculate the unit weight. The mix design, average unit weights, and average compressive strengths are given in Table 10.

**Table 10. 28-Day Compressive Strength Results for Roof Tile Application**

| Aggregate                                | Water/<br>Cement<br>Ratio | SSD, % | 28-Day Compressive<br>Strength, psi | Unit Weight<br>lb/ft <sup>3</sup> |
|--|---------------------------|--------|-------------------------------------|-----------------------------------|
| Commercial roof tile<br>sample (control) | 0.45                      | 16.8   | 4789                                | 102.2                             |
| 100/0 slag/clay                          | 0.35                      | 24.6   | 2823                                | 94.5                              |
| 50/50 slag/clay                          | 0.35                      | 19.6   | 2808                                | 97.4                              |
| 80/20 slag/clay                          | 0.38                      | 19.6   | 2940                                | 99.7                              |
| Expanded clay (control)                  | 0.65                      | 19.0   | 3066                                | 87.1                              |
| Big River clay (control)                 | 0.40                      | 15.1   | 7292                                | 115.6                             |

Superplasticizer (Mighty 150): 1.5 ml/100 g cement  
 Accelerator (CaCl<sub>2</sub>·2H<sub>2</sub>O): 2% by weight  
 Aggregate-to-cement ratio (by weight): 2.5  
 Water-to-cement ratio: to obtain 0-1 slump

As with the earlier experiments, the mechanical behavior of these samples was greatly affected by the water-to-cement ratio. The compressive strength of the 50/50 slag/clay sample was about 2808 psi, which is close to the value for the expanded clay sample at 3066 psi but lower than that of the 4789-psi control sample supplied by a lightweight roof tile company. However, the strength of the 50/50 sample should have been slightly higher than that for the test using 100% slag because of the higher unit weight of the former. This is an experimental error. The strength of the SLA-based concrete specimens is considerably lower than that of similar products made from conventional lightweight aggregates. However, its unit weight at 97.4 lb/ft<sup>3</sup> is also lower than that of the manufactured aggregate-based concrete at 102.2 lb/ft<sup>3</sup>. It is fair to assume that by producing a SLA-based concrete with a somewhat higher unit weight, the compressive strength would likewise increase.

**Laboratory Evaluation of Slag III SLA for Roof Tile Application.** The objective of this test program was to produce lightweight concrete suitable for the roof tile application from SLA III using a 50/50 ratio of Slag III and clay. As with the previous tests using Slag I, the compressive strength target requirement was in the 2500-4000 psi range, with corresponding unit weights in the 115-105 lb/ft<sup>3</sup> range. Although the compressive strength of the specimens in the previous tests using Slag I were 2800 psi and higher, we believed better particle size distribution of the aggregate in addition to refinement of the mix ratios would easily increase their compressive strengths.

The expanded slag aggregates used in the test program were made from a 50/50 blend of Slag III/clay which was crushed to match the size distribution of the LWA aggregates used by the roof tile manufacturer. The tests were performed using SLA III/clay in a saturated surface dry (SSD) condition as defined by ASTM. The moisture content of the aggregates was measured and recorded. Whereas both the accelerator and the superplasticizer were used in the earlier tests, in the current batch of tests (RT-1 and RT-2) only the superplasticizer was used.

Three 3” diameter x 6” long mortar cylinders were cast using the concrete mix formulation, cured in a wet box (relative humidity of ~70%) for 2 hours, and then steam-cured at ~60°C for 4 hours. The cylinders were demolded and returned to the wet box for further curing to 7 days. The cylinders were then weighed and broken in compression. A summary of the formulations and 7-day compressive strengths is presented in Table 11. These mixes were slightly different because the baghouse dust collected from the pilot kiln operation was added to the aggregates to compensate for the lack of fines in the SLA. It is envisaged that the dust would be added to the SLA in commercial SLA production. The highest 7-day compressive strength for the SLA III specimens was 5600 psi, which is considerably higher than the previous best results of 2800 psi for SLA I and exceeds the ASTM requirement of 4000 psi. One reason for these good results is that baghouse dust fines were added to the SLA III aggregate and used in the mix. The resultant mixture thus had a higher unit weight but, more importantly, had a much better particle size distribution. The unit weights of the specimens made from SLA III ranged between 113.6 and 114.6 lb/ft<sup>3</sup>.

**Table 11. Evaluation of SLA III as Aggregate in Roof Tile Application**

| LWA                       | SSD M% | CaCl <sub>2</sub> •H <sub>2</sub> O Wt% of cement | Plasticizer Wt% of Cement | Water/Cement Ratio | LWA/Cement Ratio | Mortar Unit Wt lb/ft <sup>3</sup> | Compressive Strength 7-day, psi |
|---------------------------|--------|---|---------------------------|--------------------|------------------|-----------------------------------|---------------------------------|
| 50/50 SLA III Test No RT1 | 18.1*  | -   | 2                         | 0.57*              | 2.5              | 113.6                             | 5598                            |
| 50/50 SLA III Test No RT2 | 18.1*  | -   | 2                         | 0.47*              | 2.5              | 114.6                             | 5603                            |
| SLA I**                   | 18.5   | 2   | 2                         | 0.35               | 2.5              | 93.3                              | 2806                            |
| Commercial LWA**          | 17.0   | 2   | 2                         | 0.45               | 2.5              | 105.3                             | 3390                            |
| Slag/Clay**               | 26.0   | 2   | 1.5                       | 0.35               | 2.5              | 105.2                             | 2303                            |

\*SLA was used in “as is” dry form using a higher cement-to-water ratio. However, the SSD moisture and water-to-cement ratios were calculated using another sample and reported for comparison.

\*\* Tests conducted with SLA I and commercial aggregate used by the roof tile manufacturer as reported in Table 9.

Superplasticizer (Mighty 150): 1.5 ml/100 g cement  
 Accelerator (CaCl<sub>2</sub>•2H<sub>2</sub>O): 2% by weight  
 Water-to-cement ratio: to obtain 0-1 slump

Some of the cylinders with RT1 and RT2 were allowed to cure further to obtain 28-day compressive strengths. The results are given in Table 12. These results were compared with those for tests conducted using the best conditions selected from the work done with SLA I.

**Table 12. 28-Day Strength Results of SLA in Roof Tile Application**

| <b>Aggregate</b>          | <b>W/C</b> | <b>SSD</b> | <b>Unit Weight<br/>lb/ft<sup>3</sup></b> | <b>28-Day<br/>Compressive<br/>Strength, psi</b> |
|---------------------------|------------|------------|--|---|
| SLA III-RT1               | 0.75       | 18.1*      | 113.6                                    | 5603  |
| SLA III-RT2               | 0.65       | 18.1*      | 114.6                                    | 6421  |
| Roof tile plant aggregate | 0.45       | 16.8       | 102.2                                    | 4789  |
| SLA I                     | 0.35       | 24.6       | 94.5                                     | 2823  |
| Slag/clay 50/50           | 0.35       | 19.6       | 97.4                                     | 2808  |
| Commercial aggregate      | 0.40       | 15.1       | 115.6                                    | 7292  |

\*SLA was used in “as is” dry form using a higher cement-to-water ratio. However, the SSD moisture and water-to-cement ratios were calculated using another sample and reported for comparison.

As may be seen in Table 12, the Slag III/clay mix resulted in a compressive strength of 5600-6400 psi, with corresponding unit weights of 114-115 lb/ft<sup>3</sup>. This is the first time that such a high strength has been obtained using an expanded slag aggregate. Unlike the tests using SLA I, these high-strength samples were made from higher-unit-weight SLA and contained no accelerator, which enables the manufacturers to demold their samples earlier but also may reduce ultimate strength in the longer term.

Based on these data, we proceeded to develop a commercial SLA mix design and verify the high compressive strengths by repeating some tests. Evaluation of the data and visual examination of the previous samples indicated that they were deficient in fines. Therefore, the original tests (RT-1 and RT-2) and the repeat tests RT-3 and RT-4 were performed using Slag III/clay along with large quantities of fines generated during the production of the same product. The roof tile aggregate mixes for tests RT-1 and RT-3 used 75% fines and those for tests RT-2 and RT-4 used 50% fines. The expanded fines were added to provide continuity in the gradation of the aggregate and cement. The particle size distribution of the expanded Slag III/clay blend is given in Table 13.

**Table 13. Size Gradation of Aggregate Mix Used in Roof Tile Tests**

| Size Fraction | Coarse Aggregate<br>Slag III/Clay | Fine Aggregate<br>Cyclone Fines | RT-3 Mix      | RT-4 Mix      |
|---------------|-----------------------------------|---------------------------------|---------------|---------------|
|               | <b>10 x 50M</b>                   |                                 | <b>25/75*</b> | <b>50/50*</b> |
|               | <b>Wt%</b>                        | <b>Wt%</b>                      |               |               |
| 4 x 8M        | 0.1                               | 53.0                            | 39.8          | 46.4          |
| 8 x 16M       | 1.3                               | 40.6                            | 30.8          | 35.7          |
| 16 x 30M      | 18.6                              |                                 | 9.5           | 7.9           |
| 30 x 50M      | 13.1                              | 0.0                             | 3.3           | 1.6           |
| 50 x 100M     | 9.4                               | 0.0                             | 2.4           | 1.2           |
| 100x 200M     | 10.9                              |                                 | 2.7           | 1.4           |
| 200M x 0      | 46.6                              | 0.0                             | 11.7          | 5.8           |
|               | 100.0                             | 100.0                           | 100.0         | 100.0         |

\*Proportion of expanded Slag III/clay vs. cyclone fines.

|   |   |
|---|---|
| Aggregate mix evaluated                             | 10 x 50M Slag III/clay 50/50 mixed with fines |
| Superplasticizer (Mighty 150):                      | 2.0 ml/100 g cement for all tests             |
| Accelerator (CaCl <sub>2</sub> ·2H <sub>2</sub> O): | 2% by weight where indicated                  |
| Aggregate-to-cement ratio (by weight):              | 2.5   |
| Water-to-cement ratio:                              | to obtain 0-1 slump                           |

The roof tile concrete mixes were prepared using the same procedure as was used for the previous batch of tests (RT-1 and RT-2). The results of these tests are shown in Table 14 along with previous results for comparison.

**Table 14. Evaluation of SLA III as Aggregate in Roof Tile Application**

| Test No.  | Aggregate/Fines |       | Water/<br>Cement<br>Ratio | Mortar<br>Unit Wt<br>(lb/ft <sup>3</sup> ) | Compressive<br>Strength (psi) |        |
|---|-----------------|-------|---------------------------|--|-------------------------------|--------|
|   | Coarse          | Fines |                           |  | 7-day                         | 28-day |
| <b>Slag III/Clay 50/50 aggregate blended with fines</b> |                 |       |                           |  |                               |        |
| RT-3  | 25              | 75    | 0.57                      | 104.4                                      | 5043                          | 5902   |
| RT-4  | 50              | 50    | 0.47                      | 107.2                                      | 6801                          | 6991   |
| RT-1  | 25              | 75    | 0.57                      | 113.6                                      | 5598                          | 5603   |
| RT-2  | 50              | 50    | 0.47                      | 114.6                                      | 5603                          | 6421   |
| <b>Other Aggregates</b>                                 |                 |       |                           |  |                               |        |
| SLA I   | 100             | -     | 0.35                      | 93.3                                       | 2806                          | 2823   |
| Roof tile plant<br>LWA (control)                        | 100             | -     | 0.45                      | 105.3                                      | 3390                          | 4789   |
| Slag I/Clay*  | 100             | -     | 0.35                      | 105.2                                      | 2303                          | 2808   |

\* Tests conducted with SLA I and commercial aggregate used by the roof tile manufacturer were done previously using a plasticizer (2% by weight of cement) and CaCl<sub>2</sub>•H<sub>2</sub>O.

Note: Tests RT-1, RT-2, RT-3 and RT-4 were done using a 50/50 mixture of expanded Slag III and clay with only plasticizer (2% by weight of cement).

As may be seen in Table 14, the Slag III/clay mixes (RT-3 and RT-4) resulted in compressive strengths of 5000-6800 psi in 7 days with corresponding unit weights of 114-115 lb/ft<sup>3</sup>. The 28-days strengths were even higher, ranging from 5600 psi to almost 7000 psi. These tests demonstrated that expanded slag/clay blends can be used to produce concrete suitable for the roof tile application.

#### 4.3.2. Lightweight Masonry Blocks (CMUs)

The objective of this subtask was to use commercial-scale concrete block manufacturing equipment and techniques to produce masonry blocks (also known as concrete masonry units or CMUs) from expanded slag lightweight aggregates (SLA). This work was done at the facilities of a major block manufacturer and distributor in the greater Chicago area. The LWA manufacturer was selected as their facility is located near the Wabash River IGCC plant that would be a potential permanent source for slag and hence SLA. A number of block mix designs were developed by Praxis using particle size distribution and unit weight information provided by the manufacturer. These mix designs were first tested in the laboratory in order to optimize them. We then prepared trial batches of concrete, followed by a full-scale run using their commercial batching plant and continuous block machine.

The objective in developing the mix designs was to substitute sufficient portions of SLA for conventional LWA in the mix while maintaining the proportions of other aggregates as per current commercial practice. Since each manufacturer has a choice of a variety of aggregates, mix designs were developed to test a number of blends. The particle size distribution of the

aggregates normally used by the plant is shown in Table 15. Also shown in the table is the size distribution of fine SLA (SLA/F) produced from 10 x 50M SLA I and coarse SLA (SLA/C) produced from coarse (1/4" x 10M) slag feed. The size gradation is critical as it determines the workability of the block mix and also directly affects the mechanical property of the blocks. Therefore, mixes should conform to the gradation range provided by the manufacturer as a guideline. Using these guidelines, five mix designs (SLA Mix Nos. 1-5) were selected for testing and submitted for review by the manufacturer.

**Table 15. Size Distribution of Aggregates Used for Concrete Block Application**

| Size Fraction               | LSS*, Wt% | Sand Slag**<br>Wt% | SLA/F<br>Wt% | SLA/C<br>Wt% | LWA (P)***<br>Wt% |
|-----------------------------|-----------|--------------------|--------------|--------------|-------------------|
| +3/8"                       | 0.0       | 0.0                | 0.0          | 3.5          | 0.0               |
| 3/8" x 4M                   | 0.2       | 9.3                | 0.5          | 8.7          | 1.3               |
| 4 x 8M                      | 18.2      | 26.2               | 9.2          | 61.5         | 26.5              |
| 8 x 16M                     | 30.7      | 20.1               | 54.8         | 24.1         | 25.6              |
| 16 x 30M                    | 19.4      | 14.8               | 23.0         | 1.1          | 17.0              |
| 30 x 50M                    | 13.0      | 11.8               | 10.8         | 1.1          | 11.9              |
| 50 x 100M                   | 9.2       | 8.7                | 1.7          | 0.0          | 7.3               |
| 100M x 0                    | 9.3       | 9.1                | 0.0          | 0.0          | 10.4              |
| Total                       | 100       | 100                | 100          | 100          | 100               |
| Unit wt, lb/ft <sup>3</sup> | 83.8      | 88.2               | 43.9         | 44.7         | 52.9              |

\* LSS: Limestone screenings used by the plant (by-product)

\*\* Blast-furnace slag fines used in place of concrete sand

\*\*\* LWA (P): Conventional LWA used by the plant.

ASTM C 331 specifies unit weight values for aggregates and unit weight and strength requirements for cement concrete used in manufacturing lightweight concrete masonry units. These requirements are summarized in Table 16.

As would be expected, the strength requirements for load-bearing blocks are higher than for nonload-bearing blocks. However, from the viewpoint of block production there is no real advantage to producing nonload-bearing blocks whereby small quantities of cement may be saved but a different product line is involved. The standard does not specify the cement concrete mix design for blocks, thus allowing a degree of flexibility in the choice of aggregates and cement-to-aggregate ratios.



**Table 16. LWA and Cement Concrete Requirements for CMU Applications**

| <b>Lightweight Aggregate Unit Weight Requirements for CMU (ASTM C 331)</b> |             |  |                                     |                                       |
|--|-------------|--|-------------------------------------|---------------------------------------|
|  |             | <b>Fine<br/>lb/ft<sup>3</sup></b>        | <b>Coarse<br/>lb/ft<sup>3</sup></b> | <b>Combined<br/>lb/ft<sup>3</sup></b> |
| Unit weight, max. values   |             | 70                                       | 55                                  | 65                                    |
| Industry preference  |             | NA                                       | NA                                  | 50                                    |
| <b>Lightweight Concrete Unit Weight and Strength Requirements for CMU</b>  |             |  |                                     |                                       |
|  |             |  | <b>28-Day Compressive Strength</b>  |                                       |
|  | <b>ASTM</b> | <b>Unit Weight<br/>lb/ft<sup>3</sup></b> | <b>Gross<br/>psi</b>                | <b>Net*<br/>psi</b>                   |
| Load-bearing   |             |  |                                     |                                       |
| - below grade  | C 90        | <105                                     | 1000                                | 2000                                  |
| - above grade  | C 90        | <85                                      | 700                                 | 1400                                  |
| Nonload-bearing  | C 129       | 105                                      | NA                                  | 600                                   |

\*Net compressive strength values calculated by assuming net cross-sectional area is 50% of gross area.

**Laboratory Testing of Concrete Block Formulations.** After two types of expanded slag materials were delivered at the concrete block facility, mix designs were prepared in the laboratory to test their compressive strength, rate of strength increase over 3-, 7-, and 28-day periods, and the unit weight of the concrete. Test mixes were formulated with the objective of manufacturing two types of blocks:

- Normal-weight blocks with a dry weight of approximately 33.5 lb
- Lightweight blocks with a dry weight of approximately 27 lb.

For both block mixes, the conventional lightweight aggregate LWA (P) used by the plant was replaced by slag lightweight aggregates of two types:

- Fine slag lightweight aggregate produced from 10 x 50M slag feed (SLA/F)
- Coarse slag lightweight aggregate produced from 1/4" x 10M slag feed (SLA/C).

The results of exploratory tests using the mix designs used for block production are shown in Table 17. As may be seen, the cement-to-aggregate ratio used was identical to that currently used at the plant. For lightweight blocks, the cement-to-aggregate ratio was 1:6.6, and for regular blocks it was 1:8.7. Water was added on an as-required basis depending on the overall workability of the aggregates and the cement paste in the mix.

**Table 17. Results of Batch Mix Tests Conducted for Masonry Blocks Using SLA**

| Test Batch   | Materials Used by Volume, ml |      |       |       |             |        |                                     | Compressive Strength, psi |       |        |
|--|------------------------------|------|-------|-------|-------------|--------|-------------------------------------|---------------------------|-------|--------|
|  | LSS                          | SS   | SLA/F | SLA/C | Total Aggr. | Cement | Concrete Unit Wt lb/ft <sup>3</sup> | 3-day                     | 7-day | 28-day |
| Unit wt, lb/ft <sup>3</sup>  | 83.8                         | 88.2 | 43.9  | 44.7  |             | 94.0   | NA                                  | NA                        | NA    | NA     |
| <b>Regular-weight block mixes (cement-to-aggregate ratio of 1:8.7 by volume)</b> |                              |      |       |       |             |        |                                     |                           |       |        |
| 21997-1  | 1650                         | 630  | 720   | -     | 3000        | 346    | 160.4                               | 1090                      | 1246  | 1636   |
| 21997-2  | 1650                         | 630  | -     | 720   | 3000        | 346    | 166.9                               | 1285                      | 1324  | 1519   |
| <b>Lightweight block mixes (cement-to-aggregate ratio of 1:6.6 by volume)</b>    |                              |      |       |       |             |        |                                     |                           |       |        |
| 21997-3  | 1290                         | -    | 1710  | -     | 3000        | 453    | 126.3                               | 1012                      | 1168  | 1402   |
| 21997-4  | 1290                         | -    | -     | 1710  | 3000        | 453    | 123.9                               | 934                       | 1012  | 1168   |
| 21997-5  | 645                          | -    | 855   | 0     | 1500        | 264    | 122.3                               | 1051                      | -     | 1519   |

Test specimens (2" diameter x 4" long cylinders) were made from the concrete and stored in a curing chamber used to cure commercial blocks. A total of nine specimens were made for each batch, which allowed for three cylinders per compression test. These tests were conducted after 3, 7, and 28 days of curing. For the last batch, only six specimens were made, which were tested after 3 and 28 days of curing.

The compression test results indicate that at the 1:8.7 and 1:6.6 cement-to-aggregate ratios, the 28-day strength was below the ASTM requirement of 2000 psi for load-bearing blocks. Adjusting the mix by adding a higher proportion of cement to the mix or increasing the slag sand or limestone sand content may increase these strength values.

The specimens made using fine expanded slag (SLA/F) proved to have higher compressive strength than those made from the coarser expanded slag (SLA/C). For example, a regular block mix using SLA/F (Test 21997-1) had a 28-day compressive strength of 1636 psi, while one made using SLA/C had a strength of 1519 psi (Test 21997-2). A similar trend was apparent in the case of lightweight blocks in which higher quantities of SLA were used.

The test results were discussed with the manufacturer and it was concluded that the lower strength values were obtained because the aggregate blend was not cohesive. Therefore, additional batches of tests were run using SLA I. During this phase of the testing, the cement-to-aggregate ratio used was identical to that currently used at the plant. As may be seen in Table 18, for the regular blocks (Mix 30S), the cement-to-aggregate ratio was 1:8.22, and for the lightweight blocks (Mix 19S) it was 1:5.97. However, a third test (Mix 19x1S) was prepared using a slightly higher cement-to-aggregate ratio of 1:5.64, with a lower quantity of the lighter-weight SLA. Water was added on an as-required basis depending on the overall workability of the aggregates and the cement paste in the mix. Test specimens (2" diameter x 3.5" long cylinders) were made from the concrete and stored in a commercial block-curing chamber. A total of three specimens were made for each batch. The compressive strength was measured after 28 days of curing, and the average values are reported in Table 18.

**Table 18. Results of Batch Mix Tests for Masonry Blocks Using SLA (contd.)**

| Test Batch   | Materials Used by Volume, ml |      |                  |       |             | Concrete |                                |                     |
|--|------------------------------|------|------------------|-------|-------------|----------|--------------------------------|---------------------|
|  | LS                           | SS   | SLA/F            | SLA/C | Total Aggr. | Cement   | Unit Weight lb/ft <sup>3</sup> | 28-day Strength psi |
| Unit wt, lb/ft <sup>3</sup>  | 83.8                         | 88.2 | 44.7             | 43.9  |             | 94.0     | -                              | -                   |
| <b>Regular-Weight Block Mix No. 30S (cement-to-aggregate ratio = 1:8.22 by volume)</b> |                              |      |                  |       |             |          |                                |                     |
| Aggregate Mix  | 55.0                         | 21.0 | 24 (SLA/F or /C) |       | 100         |          |                                |                     |
| 82797-1  | 1650                         | 630  | 720              | -     | 3000        | 346      | 110.1                          | 1558                |
| 82797-2  | 1650                         | 630  | -                | 720   | 3000        | 346      | 117.9                          | 1791                |
| <b>Lightweight Block Mix 19S (cement-to-aggregate ratio = 1:5.97 by volume)</b>        |                              |      |                  |       |             |          |                                |                     |
| Aggregate Mix  | 43.0                         |      | 57.0             |       | 100         |          |                                |                     |
| 82797-3  | 1290                         | -    | 1710             | -     | 3000        | 453      | 86.5                           | 1402                |
| 82797-4  | 1290                         | -    | -                | 1710  | 3000        | 453      | 78.6                           | 1090                |
| <b>Lightweight Block Mix No. 19x1S (cement-to-aggregate ratio = 1:5.64 by volume)</b>  |                              |      |                  |       |             |          |                                |                     |
| Aggregate Mix  | 60.0                         | -    | 40.0             |       | 100         |          |                                |                     |
| 82797-6  | 1800                         | -    | 1200             | 0     | 3000        | 494      | 102.2                          | 2180                |

The regular-weight block mix specimens using SLA/F (Test 82797-1) had a 28-day compressive strength of 1558 psi, while one made using SLA/C had a strength of 1791 psi (Test 82797-2). The unit weight of the concrete (110-118 lb/ft<sup>3</sup>) was considerably lower than the typical value of 150 lb/ft<sup>3</sup> for regular blocks.

Tests with lightweight block Mix 19S resulted in compressive strengths of 1090-1402 psi, which is consistent with the low unit weight of the concrete (78.6-86.5 lb/ft<sup>3</sup>).

The next batch of tests (Mix 19x1S) was conducted using a reduced proportion of SLA/F (40%) in order to increase the concrete unit weight and compressive strength. The 28-day strength for this mix (Test 82797-6) was 2180 psi, which is higher than the ASTM requirement of 1400 psi for above-grade blocks and 2000-psi for below-grade load-bearing blocks. The estimated weight of the block if made from Mix 19S was 20.4 lb, and that of the block made from Mix 19x1S was 22.3 lb. Both of these are below the preferred weight of 23 lb for lightweight blocks, which is excellent from the viewpoint of the industry. It was possible to further increase the strength of the SLA concrete by adding some fine sand to compensate for the lack of fines in the slag. However, this would increase the unit weight of the block.

**Laboratory Evaluation of Expanded Slag III for Testing of Masonry Blocks:** In order to verify the use of SLA in making lightweight blocks, Slag III was tested similarly. Test mixes were formulated based on the experience with the work done using Slag I.

The conventional lightweight aggregate was replaced by fine SLA III made from a 10 x 50M Slag III feed (SLA III/F). The cement-to-aggregate ratio used was identical to that currently used

at the block manufacturing plant. Test specimens (2" diameter, 3.5" tall cylinders) were made from the concrete and stored in a steam chamber. A total of three specimens were made for each batch. The compressive strength was measured after 7 days (1 specimen) and 28 days (2 specimens) of curing, and the average 28-day values are reported in Table 19.

As may be seen in Table 19, the unit weights of specimens made with SLA III varied between 87 and 118 lb/ft<sup>3</sup> with a maximum compressive strength of 1550 psi.

**Table 19. Results of Batch Mix Tests for Masonry Blocks Using SLA III**

| Test No.  | Materials Used by Volume, ml |          |           |             | Concrete |                                |                     |        |
|---|------------------------------|----------|-----------|-------------|----------|--------------------------------|---------------------|--------|
|   | Sand                         |          | SLA-III/F | Total Aggr. | Cement   | Unit Weight lb/ft <sup>3</sup> | Comp. Strength, psi |        |
|   | Washed                       | Unwashed |           |             |          |                                | 7-day               | 28-day |
| Unit wt, lb/ft <sup>3</sup>   | 106.6                        | 103.9    | 41.0      |             | 94.0     | -                              | -                   | -      |
| <b>Cement-to-aggregate ratio = 1:7.73 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB1   | 1978                         | --       | 1616      | 3594        | 465      | 114.6                          | 1225                | 1380   |
| <b>Cement-to-aggregate ratio = 1:8.66 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB2   | --                           | 2344     | 1683      | 4027        | 465      | 117.9                          | 1012                | 1029   |
| <b>Cement-to-aggregate ratio = 1:6.74 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB3   | --                           | 2344     | 1683      | 4027        | 598      | 108.7                          | 930                 | 1102   |
| <b>Cement-to-aggregate ratio = 1:7.46 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB4   | --                           | 2783     | 1683      | 4466        | 598      | 115.9                          | 1309                | 1575   |
| <b>Cement-to-aggregate ratio = 1:7.46 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB5   | --                           | 2930     | 1530      | 4460        | 598      | 114.2                          | 924                 | 1273   |
| <b>Cement-to-aggregate ratio = 1:9.59 by volume</b>                                 |                              |          |           |             |          |                                |                     |        |
| LB6   | --                           | 2930     | 1530      | 4460        | 465      | 108.2                          | 423                 | 689    |
| <b>Mix 30S (cement-to-aggregate ratio = 1:8.22 by volume) Regular block</b>         |                              |          |           |             |          |                                |                     |        |
| Aggr. Mix   | 55% (LSS)                    | 21% (SS) | 24%       | 100%        |          |                                | -                   | -      |
| 82797-1 SLA I/F   | 1650 LSS                     | 630 SS   | 720       | 3000        | 346      | 110.1                          | -                   | 1558   |
| <b>Lightweight Block Mix No. 19S (cement-to-aggregate ratio = 1:5.97 by volume)</b> |                              |          |           |             |          |                                |                     |        |
| 82797-3 SLA I/F   | 1290                         | -        | 1710      | 3000        | 453      | 86.8                           | -                   | 1402   |

These tests were also compared with results for SLA I (mix designs 30S and 19S). The regular-weight block mix using SLA I/F (Test 82797-1) had a 28-day compressive strength of 1558 psi, while one made using SLA I/C had a strength of 1402 psi (Test 82797-3). The unit weight of the concrete was considerably lower than the typical value required for regular blocks. Tests with

lightweight block Mix 19S resulted in compressive strengths of 1090-1402 psi, which is consistent with the low unit weight of the concrete (78.6-86.5 lb/ft<sup>3</sup>).

Additional tests were conducted in order to improve the compressive strength of the concrete. One test (LB-7) was performed to increase the strength to a target of 2000 psi by using a slightly higher quantity of cement (658 ml vs. 630 ml). The results are given in Table 20.

**Table 20. Additional Results of Batch Mix Tests for Masonry Blocks Using SLA**

| Test ID | Type I Cement ml | SLA III ml | Unwashed Sand, ml | Mighty 150, ml | Unit Wt. lb/ft <sup>3</sup> | 7-Day Strength psi | 28-Day Strength psi |
|---------|------------------|------------|-------------------|----------------|-----------------------------|--------------------|---------------------|
| LB-7    | 658              | 1616.4     | 2343.6            | 13.5           | 108                         | 1531               | 1998                |

As may be seen, the unit weight of the concrete specimens was 108 lb/ft<sup>3</sup> and the compressive strength was nearly 2000 psi. Analysis of the mix indicated that in spite of the addition of sand, it was deficient in minus 100-mesh fines. Based on this assessment, we decided to make a mix with additional fines in the form of a mineral filler. This filler, a fine aggregate dust, is another process by-product used by the plant.

**Commercial Production of Masonry Blocks.** Based on the experience acquired with SLA I and SLA III, the mix design for the production batch of blocks, given in Table 21, was finalized. The mix batch was calculated to make 250 8-inch blocks. The concrete mix batch size was 4,222 lb using 60 ft<sup>3</sup> of aggregate (40% SLA). Sufficient water was added to achieve the desired consistency. The ingredients were weighed and dumped in the plant feed hopper and the automated process for mixing and transportation was initiated using the batching plant and standard three-mold continuous block machine operated at a nominal rate of 250 blocks/hour.

The target specification for the 8-inch lightweight blocks was to achieve a concrete unit weight of <105 lb/ft<sup>3</sup>, which would result in a block dry weight of approximately 27 lb. The compressive strength target of the concrete would be 2,000 psi.

**Table 21. Mix Proportions for Production Batch of Lightweight Blocks**

|                                | LSS*  | SLA III 10 x 50M | Mineral Filler | Masonry Sand | Total | Type I Cement | Additive |
|--------------------------------|-------|------------------|----------------|--------------|-------|---------------|----------|
| Wt. %                          | 52    | 40               | 5              | 3            | 100   |               | -        |
| Wt. (lb)                       | 2226  | 1616             | 234            | 2344         | 3622  | 600           |          |
| Unit Wt. (lb/ft <sup>3</sup> ) | 71.36 | 42.6             | 78.0           | 77.1         | --    | 94            | 128      |
| Vol. (ft <sup>3</sup> )        | 31.2  | 24.0             | 3.0            | 1.8          | 60    | 6.38          | 48       |

\* LSS: Local, normal-weight aggregate used by the plant.

Compressive strength and unit weight test results conducted on randomly selected blocks are given in Table 22.

**Table 22. Compressive Strength and Unit Weight Test Results for Lightweight Blocks**

| <b>28-Day Compressive Strength, psi</b> |       |
|---|-------|
| Block 1                                 | 2041  |
| Block 2                                 | 2098  |
| Block 3                                 | 2190  |
| Average                                 | 2143  |
| <b>Unit Weight, lb/ft<sup>3</sup></b>   |       |
| Block 4                                 | 105.7 |
| Block 5                                 | 105.3 |
| Block 6                                 | 106.7 |
| Average                                 | 105.9 |

The overall test results for the utilization of SLA in making lightweight blocks are summarized in Table 23.

**Table 23. Summary of Test Results for Use of SLA in Lightweight Block Application**

|  | <b>Required Values</b> | <b>Tested Values</b> |
|--|------------------------|----------------------|
| Net area compressive strength, psi                   | 1900.00                | 2143                 |
| Gross area compressive strength, psi                 |                        | 1093                 |
| Density, lb/ft <sup>3</sup>                          | <105                   | 105.9                |
| Absorption, lb/ft <sup>3</sup>                       | 18.00                  | 11.4                 |
| Minimum face shell thickness, in.                    | 1.25                   | 1.25                 |
| Minimum web thickness, in.                           | 0.75                   | 1.00                 |
| Equivalent web thickness, in.                        | 2.25                   | 2.33                 |
| Equivalent thickness, in.                            |                        | 3.88                 |
| Net cross-sectional area, in <sup>2</sup>            |                        | 60.8                 |
| Net volume, ft <sup>3</sup>                          |                        | 0.26                 |
| Percent solids, %                                    |                        | 51.0                 |
| Calculated Fire Resistance Rating (NCMA-TEK 7-3), hr |                        | 1.75                 |

As may be seen, the compressive strength for lightweight blocks using SLA was over 2,000 psi using the quantity of cement that is typically used by the industry. Also, a concrete unit weight of 106 lb/ft<sup>3</sup> was obtained using 40% SLA, which is the LWA level currently used by the manufacturer, i.e., 100% replacement. The blocks met all other requirements. The test program was considered successful since all relevant requirements were met.

#### **4.3.3 Laboratory Evaluation of SLA for Structural Concrete Application**

The objective of this test program was to develop mix designs to produce sand and SLA-based cement concretes with compressive strengths of 2500-4000 psi and corresponding unit weights in the 115-105 lb/ft<sup>3</sup> range. These variations were accomplished mainly by changing the proportion of cement relative to the SLA. The SLA samples tested are identified in Table 24. In addition, a control sample of commercially available structural aggregates was also tested. As may be seen

in Table 1, the 3/4" coarse SLA was tested using three different cement levels to produce products with varying strengths (complete matrix) whereas the other samples were tested at only one strength level.

**Table 24. Cement Levels Used in Testing SLA as Structural Aggregate**

| Test No. | SLA Products Tested  | Cement Level, Sacks/Yard <sup>3</sup> |
|----------|--|---------------------------------------|
| 1        | 3/4" coarse SLA (50/50 slag-clay pellets)                    | 5½, 6½, and 7½                        |
| 2        | 1/4" x 50M SLA I crushed as 3/8" combined LWA                | One level of cement                   |
| 3        | 3/4" expanded clay pellets produced during the pilot program | One level of cement                   |

ASTM C 330 unit weight requirements for structural concrete aggregates are summarized in Table 25. Also provided in this table for purposes of reference are the unit weight and compressive strength requirements for cement concrete mixtures produced from 100% LWA or various mixtures of LWA and sand.

**Table 25. LWA and Concrete Unit Weight and Strength Requirements**

| Structural Lightweight Aggregate Unit Weight Requirements             |                             |                    |                    |
|---|-----------------------------|--------------------|--------------------|
|   | Fine                        | Coarse             | Combined           |
|   | lb/ft <sup>3</sup>          | lb/ft <sup>3</sup> | lb/ft <sup>3</sup> |
| Unit weight, maximum values   | 70                          | 55                 | 65                 |
| Lightweight Structural Concrete Unit Weight and Strength Requirements |                             |                    |                    |
| Concrete Unit Weight  | 28-Day Compressive Strength |                    |                    |
| lb/ft <sup>3</sup>  | lb/in <sup>2</sup>          |                    |                    |
| All Lightweight Aggregate   |                             |                    |                    |
| 110   | 4000                        |                    |                    |
| 105   | 3000                        |                    |                    |
| 100   | 2500                        |                    |                    |
| Sand-Lightweight Aggregate  |                             |                    |                    |
| 115   | 4000                        |                    |                    |
| 110   | 3000                        |                    |                    |
| 105   | 2500                        |                    |                    |

Table 26 lists the SLA-based aggregates and control aggregates that were tested. The table also provides the size gradation specified in ASTM C 330 in conformity with which the aggregates were prepared.

**Table 26. Samples of Materials Used for Structural Aggregate Testing**

| Sample ID | Aggregates and Production Methods  | Gradation to ASTM C 330 | Unit Weight lb/ft <sup>3</sup> |
|-----------|--|-------------------------|--------------------------------|
| 951132    | Pelletized slag/clay (50/50 SLA)   | 3/4" coarse             | 40.9                           |
| 951133    | Clay LWA produced in pilot plant from clay used as binder to produce 50/50 SLA pellets | 3/4" coarse             | 48.9                           |
| 950931    | SLA produced from 1/4" x 50M Slag I  | 3/8" combined           | 51.2                           |
| 960234    | Commercial LWA product (expanded clay)   | 5/8" coarse             | 34.0                           |
| 960235    | Commercial LWA product (expanded clay)   | 3/4" coarse             | 38.0                           |
| 960233    | Commercial LWA product (expanded clay)   | 3/8" combined           | 53.8                           |
|           | Concrete sand  | Fine size (4M x 0)      | 102.0                          |

Clay LWA and commercial LWA were used as control materials. The slag- and clay-based expanded aggregates produced in the pilot plant were crushed to meet ASTM C 330 size specifications prior to use. The samples of commercial LWAs were obtained from a commercial LWA plant and used without crushing in the control tests since they were prepared to the appropriate ASTM size gradation specification. The size distribution of these materials is given in Table 27. The data indicate that the size distributions of SLA and 50/50 SLA (both prepared as 3/4" coarse aggregates) are much coarser than commercial aggregates with the same designation although they fall within the allowable range.

**Table 27. Size Distribution of LWA Materials and Sand Used in Structural Concrete**

|                                       | Commercial LWA |             |               | 50/50 SLA/LWA | Clay LWA    | SLA           | Sand  |
|---------------------------------------|----------------|-------------|---------------|---------------|-------------|---------------|-------|
| ASTM C 330 Size                       | 5/8" coarse    | 3/4" coarse | 3/8" combined | 3/4" coarse   | 3/4" coarse | 3/8" combined |       |
| Unit weight lb/ft <sup>3</sup>        | 34.0           | 38.0        | 53.8          | 40.9          | 48.9        | 51.3          |       |
| <b>Size Distribution, Wt% passing</b> |                |             |               |               |             |               |       |
| 1"                                    | 100.0          | 100.0       | 100.0         | 100.0         | 100.0       |               |       |
| 3/4"                                  | 100.0          | 100.0       | 100.0         | 100.0         | 100.0       |               |       |
| 1/2"                                  | 81.3           | 95.9        | 100.0         | 52.1          | 33.8        |               |       |
| 3/8"                                  | 27.5           | 68.2        | 100.0         | 21.1          | 11.8        | 100.0         | 100.0 |
| 4 mesh                                | 12.6           | 6.1         | 97.0          | 8.4           | 4.4         | 91.9          | 99.0  |
| 8 mesh                                | 8.5            | 3.1         | 70.5          |               |             | 59.9          | 77.0  |
| 16 mesh                               | 6.0            | 2.5         | 41.3          |               |             | 50.3          | 55.0  |
| 30 mesh                               | 4.0            |             |               |               |             |               | 41.0  |
| 50 mesh                               | 3.3            | 1.7         | 14.2          |               |             | 20.5          |       |
| 100 mesh                              | 2.4            | --          | 8.9           |               |             | 15.0          | 7.0   |
| 200 mesh                              |                |             |               |               |             |               | 1.9   |

Table 28 lists the SLA and control tests that were carried out using different cement levels.



**Table 28. Expanded SLA Products Tested and Cement Levels Used**

| No.                                     | Lightweight Aggregate Products Tested   | Concrete Cement sacks/yard <sup>3</sup> |
|---|---|---|
| <b>Sand-Lightweight Aggregate Tests</b> |   |   |
| 1.                                      | 50/50 SLA as 3/4" coarse aggregates   | 5½, 6½, and 7½                          |
| 2.                                      | SLA (from 1/4" x 50M Slag I) as 3/8" combined aggregates                                    | Min. one level of cement                |
| 3.                                      | Clay LWA produced in the pilot plant from the clay used as a binder for producing 50/50 SLA | Min. one level of cement                |
| 4.                                      | Commercial LWA product (expanded clay)  | Min. one level of cement                |
| <b>All Lightweight Aggregate Tests</b>  |   |   |
| 5.                                      | SLA (from 1/4" x 50M slag) as 3/8" combined aggregates                                      | One level of cement                     |
| 6.                                      | Clay LWA produced in the pilot plant from clay used as a binder for producing 50/50 SLA     | One level of cement                     |

**Testing and Evaluation Procedure.** Cement concrete mixes were prepared from the slag aggregates listed above. The aggregate-to-cement ratios used were identified in exploratory tests, with the objective of achieving 28-day strengths of 2500, 3000, and 4000 psi, respectively. Approximately 12-15 test specimens were prepared for testing using the following procedure:

- Adjust the moisture content of the aggregates to saturated surface dry (SSD) conditions by saturating them in water overnight. Document the moisture content.
- Estimate the sand content required for the concrete mix to achieve a suitable gradation without exceeding the unit weight specification.
- Document the dosages of the air-entraining agent used.
- Prepare test specimens using the SLA sample with a pre-selected aggregate-to-cement ratio and slump. Measure the water added to document the cement-to-water ratio.
- Document the total weight and volumes of the ingredients used and calculate the sand-to-LWA ratio and water-to-cement ratio by weight. Measure the unit weight of the fresh concrete. Report the workability of the mix.
- Test a minimum of three specimens for compression following 1-day (early strength), 3-day, 7-day, and 28-day curing time periods. Save 3 cylinders for further testing.
- Prepare control test specimens using the commercial LWA sample with an identical aggregate-to-cement ratio and slump. Measure the water added to document the cement-to-water ratio. Measure the unit weight of the concrete and its compressive strength for 3-day, 7-day and 28-day curing time periods for purposes of comparison.

- Test specimens were saved in order to conduct the following tests at a later date if desired:
  - Freeze/Thaw, ASTM C 666
  - Drying Shrinkage, ASTM C 157
  - Staining, ASTM C 641

**Results of Laboratory Tests for SLA Concrete Mixes Made Without Sand.** Exploratory laboratory studies were conducted using SLA to make lightweight concrete without sand in order to evaluate its potential as a structural aggregate as per ASTM C 330. However, the resulting concrete was expected to be much lighter and hence lower in strength than that required by ASTM C 330. These tests were performed using three levels of cement. The results are summarized in Table 29. The following problems were experienced with these tests, with the exception of Test 4:

- The lack of fines made the concrete mix too coarse and hence unworkable.
- Due to the lack of fines the water separated from the mix.
- The product unit weights, in the range of 67-70 lb/ft<sup>3</sup>, were much lower than the target values of 100-110 lb/ft<sup>3</sup>.
- The 28-day compressive strength values were in the 843-1877 psi range, far lower than the target of 2500 psi.

Therefore, production of structural concrete by formulating lightweight concrete mixes using SLA without sand was rejected for further consideration. This constraint was necessary due to the fact that the SLA produced in the pilot program was devoid of fines as they were screened out and used to make pelletized SLA.

**Table 29. Use of SLA for Structural Lightweight Concrete Without Sand**

| Test No. | Aggregate Type, Application and Unit Weight |                    | Cement Sacks/yd <sup>3</sup> | W/C Ratio | Slump in. | Air % | Product Unit Weight lb/ft <sup>3</sup> | Compressive Strength psi* |        |
|----------|---|--------------------|------------------------------|-----------|-----------|-------|--|---------------------------|--------|
|          | Application per ASTM C330                   | lb/ft <sup>3</sup> |                              |           |           |       |  | 7-day                     | 28-day |
| 1        | SLA as 3/8" combined                        | 51.3               | 5½                           | 0.6       | 0         | 3.8   | 66.9                                   | 375                       | 843    |
| 1A       | SLA as 3/8" combined                        | 51.3               | 5½                           | 0.65      | 0.5       | 4.8   | 67.4                                   | 610                       | 963    |
| 2        | SLA as 3/8" combined                        | 51.3               | 6                            | 0.65      | 0.75      | 4.8   | 69.6                                   | 740                       | 1877   |
| 3        | SLA as 3/8" combined                        | 51.3               | 6½                           | 0.65      | 1.25      | 4.8   | 69.9                                   | 1180                      | 1840   |
| 4        | Commercial LWA (3/8" combined)              | 53.8               | 6                            | 0.65      | 1.5       | 3.9   | 70.0                                   | -                         | 2370   |

\*Average of three tests.

**Results of Laboratory Tests for Various LWA Concrete Mixes Using Sand.** The results of laboratory studies to make lightweight concrete mixes using SLA, LWA, and sand mixes are summarized in Table 30.

**SLA as 3/8" combined aggregate.** Tests to evaluate SLA as 3/8" combined aggregate (produced from 1/4" x 50M Slag I), using 6 sacks of cement/yd<sup>3</sup> of concrete, resulted in 7- and 28-day strengths for the SLA concrete of 1120 and 1750 psi respectively (Test 2199). The unit weight of the concrete was 107 lb/ft<sup>3</sup>. The 28-day compressive strength of the 3/8" combined commercial LWA (control sample) was 2400 psi at a unit weight of 115 lb/ft<sup>3</sup>. Neither of these aggregates met the ASTM C 330 requirement of a compressive strength of 2500 psi at 105 lb/ft<sup>3</sup>.

Since the SLA concrete 28-day strength failed to meet the ASTM strength requirement, additional tests were performed using 6.5 sacks of cement/yd<sup>3</sup> concrete, and the results are reported in Table 30. At the higher cement level, the 28-day strength was 2070 psi at 107 lb/ft<sup>3</sup> which is still considerably lower than the ASTM requirement. The 28-day strength results of the commercially produced LWA at 3440 psi and 112 lb/ft<sup>3</sup> (control test 2206) were much higher than those of the SLA concrete and met the ASTM requirement at the given unit weight level. It was concluded that the strength of the SLA concrete could be increased by further work to control the fines content.

**Table 30. Evaluation of SLAs for Sand-Lightweight Concrete Application**

| Test No.  | Aggregate Type, Application and Unit Weight |                    | Sand*/Aggregate | W/C Ratio | Slump in. | Air % | Unit Weight lb/ft <sup>3</sup> | Comp. Strength, psi |        |  |
|---|---|--------------------|-----------------|-----------|-----------|-------|--------------------------------|---------------------|--------|--|
|   | Type and Application per ASTM C330          | lb/ft <sup>3</sup> |                 |           |           |       |                                | 7-day**             | 28-day |  |
| <b>Tests Using 6.0 sacks of cement/yd<sup>3</sup></b> |   |                    |                 |           |           |       |                                |                     |        |  |
| 2205-A  | Commercial LWA (5/8" and 3/4" coarse)       | 36.0               | 48/52           | 0.65      | 4.0       | 3.5   | 119.6                          | -                   | -      |  |
| 2205  | Commercial LWA (3/4" coarse)                | 38.0               | 36/64           | 0.43      | 3.5       | 2.0   | 112.8                          | 2380                | 3400   |  |
| 2201  | Commercial LWA (3/8" combined)              | 53.8               | 45/55           | 0.65      | 3.5       | 4.0   | 114.8                          | 1500                | 2400   |  |
| 2199  | SLA as 3/8" combined                        | 51.3               | 43/57           | 0.65      | 4.0       | 3.0   | 106.7                          | 1120                | 1750   |  |
| 2208  | Commercial LWA as 5/8" coarse               | 34.0               | 41/59           | 0.46      | 2.5       | 2.0   | 113.8                          | 2420                | 3390   |  |
| 2207  | Commercial LWA as 5/8" coarse               | 34.0               | 39/61           | 0.46      | 2.0       | 2.0   | 108.6                          | 2220                | 3240   |  |
| 2209-A  | Clay LWA as 3/4" coarse                     | 48.9               | 54/46           | 0.52      | 7.0       | --    | 115.4                          | ***                 | ***    |  |
| 2209  | Clay LWA as 3/4" coarse                     | 48.9               | 49/51           | 0.45      | 3.0       | 1.0   | 114.8                          | 3430                | 4800   |  |
| 2211  | 50/50 SLA as 3/4" coarse                    | 40.9               | 44/56           | 0.48      | 2.5       | 2.0   | 112.3                          | 2910                | 4210   |  |
| <b>Tests Using 6.5 sacks of cement/yd<sup>3</sup></b> |   |                    |                 |           |           |       |                                |                     |        |  |
| 2200  | SLA as 3/8" combined                        | 51.3               | 43/57           | 0.65      | 4.0       | 3.8   | 107.0                          | 1350                | 2070   |  |
| 2206  | Commercial LWA as 5/8" coarse               | 34.0               | 37/63           | 0.43      | 2.0       | 2.0   | 111.8                          | 2730                | 3440   |  |
| 2210-A  | Clay LWA as 3/4" coarse aggregate           | 48.9               | 48/52           | 0.42      | 3.5       | 1.0   | 115.2                          | ***                 | ***    |  |
| 2210  | Clay LWA as 3/4" coarse aggregate           | 48.9               | 46/54           | 0.42      | 3.5       | 1.0   | 114.3                          | 4040                | 5100   |  |
| 2212  | 50/50 SLA as 3/4" coarse aggregate          | 40.9               | 45/55           | 0.44      | 3.25      | 2.0   | 114.7                          | 3480                | 4360   |  |

\*Sand unit weight was 102 lb/ft<sup>3</sup>, 99% passing 4M, 1.9% passing 200M (dry).

\*\*Average of three tests.

\*\*\*Test specimen exceeded 115 lb/ft<sup>3</sup>.

**50/50 SLA as 3/4" coarse aggregate:** Tests conducted with 50/50 slag/clay aggregates using 6.0 sacks of cement/yd<sup>3</sup> concrete resulted in 7-day and 28-day concrete strength measurements of 2910 and 4210 psi respectively. The 28-day value exceeds the ASTM requirement of 4000 psi at a unit weight of 115 lb/ft<sup>3</sup>. These results were far superior to those of tests done using SLA (3/8" combined) at the same cement level, as well as to control Test 2205 using commercially manufactured aggregates which had a 28-day strength value of 3400 psi.

The tests conducted at the higher (6.5 sacks/yd<sup>3</sup> concrete) cement level resulted in compressive strengths of 3480 and 4360 psi for the 7- and 28-day curing periods, respectively (Test 2212), at a unit weight below 115 lb/ft<sup>3</sup>. The control test strengths using clay LWA (Test 2210) were 4040 and 5100 psi, which are in a comparable range. The 28-day compressive strength of the concrete using commercial LWA at the 5/8" size designation was 3440 psi, which is lower than that resulting from use of the SLA/clay-based aggregate. These data indicate that blending expanded slag and clay results in a high-quality product. It was concluded that 50/50 SLA as 3/4" coarse aggregates with clay LWA would make an acceptable structural concrete using 6 sacks of cement/yd<sup>3</sup> concrete.

**Freeze/Thaw Testing of SLA Concrete.** In order to assess the durability of the structural concrete made from SLA, sand and cement ASTM C 66, Procedure B was performed on the specimens. The test subjected the samples to the stress of repeated freezing and thawing.

The mix designs were the same as previously used to produce sand and SLA-based cement concrete specimens:

- Mix 2211R prepared using 3/4" SLA made from 50/50 slag/clay blend
- Mix 2205R prepared using 5/8" and 3/4" LWA made from clay.

The 28-day compressive strength of the SLA concrete specimen (with 6.0 sacks of cement/yd<sup>3</sup> concrete) was 3000 psi at 114.7 lb/ft<sup>3</sup>, which is below the ASTM requirement of 4000 psi at a unit weight of 115 lb/ft<sup>3</sup>. However, some specimens using slightly different sand-to-aggregate ratios but the same cement content had compressive strength values of over 4000 psi at unit weights below 115 lb/ft<sup>3</sup>.

In order to determine the aggregate properties without an entraining agent, freeze/thaw tests were conducted for the two mixes without air entrainment. Test specimens were saturated for a period of four hours at 40°F prior to the start of the test. The results are shown in Table 31. They indicate that the specimens exhibited cracking as a result of freeze/thaw stresses after 64 cycles. At cycle 98, cracking was severe. Due to specimen deterioration, the fundamental transverse frequency could not be measured, thus precluding calculation of the relative dynamic modulus of elasticity. It is hypothesized that the cracking was due to the following reasons:

- The lower density of the SLA with a higher proportion of pores and higher moisture retention capacity
- The absence of an air-entraining agent in the concrete, which typically helps improve freeze/thaw performance.

**Table 31. Resistance of Concrete to Rapid Freeze/Thaw (ASTM C 666, Procedure B)**

| Specimen |   | Number of Freeze/Thaw Cycles |      |      |    |                           |      |      |     |
|----------|---|------------------------------|------|------|----|---------------------------|------|------|-----|
|          |   | 0                            | 32   | 64   | 98 | 0                         | 32   | 64   | 98  |
|          |   | Mix 2205-R <sup>(1)</sup>    |      |      |    | Mix 2211-R <sup>(2)</sup> |      |      |     |
| 1        | Relative dynamic modulus of elasticity, % | --                           | 100  | 76   | -- | --                        | (3)  | (3)  | (3) |
|          | Weight, gm, SSD                           | 5456                         | 5460 | 5491 | -- | 6063                      | 6072 | 6098 | --  |
|          | Weight change, gm                         | 0                            | +4   | +35  | -- | 0                         | +9   | +35  | --  |
| 2        | Relative dynamic modulus of elasticity, % | --                           | 98   | 67   | -- | --                        | (3)  | (3)  | (3) |
|          | Weight, gm, SSD                           | 5492                         | 5518 | 5547 | -- | 6053                      | 6069 | 6119 | --  |
|          | Weight change, gm                         | 0                            | +26  | +55  | -- | 0                         | +16  | +66  | --  |
| 3        | Relative dynamic modulus of elasticity, % | --                           | 96   | 64   | -- | --                        | (3)  | (3)  | (3) |
|          | Weight, gm, SSD                           | 5576                         | 5698 | 5729 | -- | 6043                      | 6060 | 6100 | --  |
|          | Weight change, gm                         | 0                            | +122 | +153 | -- | 0                         | +17  | +57  | --  |
| Average  | Relative dynamic modulus of elasticity, % | --                           | 98   | 69   | -- | --                        | (3)  | (3)  | (3) |
|          | Weight change, gm                         | 0                            | +51  | +81  | -- | 0                         | +14  | +53  | --  |

(1) Mix 2205R: Control mix using expanded clay LWA at 35.3 lb/ft<sup>3</sup>, which made concrete with a compressive strength of 3400 psi and unit weight of 112.8 lb/ft<sup>3</sup>.

(2) Mix 2211-R: 505/50 slag clay expanded to 39 lb/ft<sup>3</sup>, which made concrete with a unit weight of 114.8 lb/ft<sup>3</sup>.

(3) Due to specimen deterioration, the fundamental transverse frequency could not be measured, thus precluding calculation of the relative dynamic modulus of elasticity.

It was concluded that further testing and development was needed to overcome the freeze/thaw problem.

#### 4.3.4 Laboratory Evaluation of SLA for Insulating Concrete Application

Insulating concrete is applied in a layer on the flat roof surfaces of buildings to increase their insulating properties, particularly in warehouses located in climate zones that experience temperature extremes. Another application for insulating concrete is in making concrete panels used to improve the insulating properties of building walls. Insulating concrete is made using two materials: expanded perlite and expanded shale. ASTM C 332 provides specifications for LWA for insulating concrete applications. Three properties of importance are the unit weight, compressive strength, and thermal conductivity of the concrete. In testing the suitability of SLA for use in making insulating concrete, these three properties were measured and compared with those of conventional insulating concretes made from expanded perlite and expanded shale.

SLA with a unit weight of 26 lb/ft<sup>3</sup> produced using the fluidized bed expander was screened according to ASTM C 332. In order to evaluate SLA for use as an aggregate in insulating concrete, specimens were made in order to test their compressive strength and thermal properties. Using the mix proportions for perlite insulating concrete re-roofing material as a

guide, 2" cubes and a 12" wide x 12" tall x 1" thick slab were made using 3/8" x 0 expanded slag according to the following formula:

|                             |                          |
|-----------------------------|--------------------------|
| Type I cement:              | 800 g                    |
| 3/8" x 0 expanded slag:     | 4 times cement by volume |
| Water:                      | 640 g                    |
| Air-entraining agent:       | 8.3 g                    |
| 10 mm polypropylene fibers: | 8 g                      |

These samples were mixed according to ASTM C 109 and cured in a 98% relative humidity chamber at 25°C. After 7 days, the cubes were removed for testing of their compressive strength. The highest 7-day compressive strength achieved was 1750 psi, and the unit weight of the samples was approximately 51 lb/ft<sup>3</sup>.

The 12" wide x 12" tall x 1" thick slab was tested for its thermal conductivity. The typical thermal conductivity of perlite (Group I) is 0.45-1.5 Btu-in/hr-ft<sup>2</sup>-°F, and that of shale-based aggregates (Group II) is 1.5-3.0 Btu-in/hr-ft<sup>2</sup>-°F. As may be seen in Table 32, the thermal conductivity of the SLA concrete at 0.984 Btu-in/hr-ft<sup>2</sup>-°F is much lower (i.e., better) than that of Group II shale aggregates and falls within the Group I range. The expanded slag therefore, is an excellent insulator suitable for use in making insulating concrete panels.

**Table 32. Apparent Thermal Conductivity and Thermal Resistance of SLA Test Specimens**

| Aggregate                  | Concrete | Thermal Conductivity        |          | Thermal Resistance            |
|----------------------------|----------|-----------------------------|----------|-------------------------------|
|                            |          | Unit Wt, lb/ft <sup>3</sup> | W/m-K    | Btu-in/hr-ft <sup>2</sup> -°F |
| SLA, 26 lb/ft <sup>3</sup> | 45.1     | 0.142                       | 0.984    | 0.93                          |
| Perlite (Group I)          | 15-50    | 0.065-0.22                  | 0.45-1.5 | -                             |
| Shale (Group II)           | 50-90    | 0.22-0.43                   | 1.5-3.0  | -                             |

Note: The SLA tests were conducted at 25°C whereas the reference data are at 24°C.

#### **4.3.5 Laboratory Evaluation of SLA for Loose Fill Insulation Application**

Typically, the outer walls of commercial buildings are made with masonry blocks. The cavities or air gaps in the blocks are filled with expanded perlite to improve the building insulation. The cavity between the block wall and brick layer, if any, is also filled with expanded perlite. ASTM C 549 provides specifications for using expanded perlite as a loose fill insulating material. The key properties of interest are the degree of insulation provided, light weight, lack of dustiness, and lack of moisture absorption. Expanded slag produced using the fluidized bed expander was screened according to ASTM C 549 for use as loose fill insulation. The SLA sample had a unit weight of 29 lb/ft<sup>3</sup> and a thermal resistance of 1.46 hr-ft<sup>2</sup>-°F/Btu. This is higher than the value of 2.4 hr-ft<sup>2</sup>-°F/Btu for the 11 lb/ft<sup>3</sup> unit weight perlite, as shown in Table 33. However, expanded slag is much easier to work with due to its significantly lower degradation characteristics and may also be easier to apply.

**Table 33. Thermal Conductivity and Thermal Resistance of SLA for Loose Fill Insulation**

| Material Type | Unit Weight<br>lb/ft <sup>3</sup> | Temperature | Thermal Conductivity |                               | Thermal Resistance         |
|---------------|-----------------------------------|-------------|----------------------|-------------------------------|----------------------------|
|               |                                   | °C          | W/m-K                | Btu-in/hr-ft <sup>2</sup> -°F | hr-ft <sup>2</sup> -°F-Btu |
| Expanded slag | 29                                | 25          | 0.093                | 0.645                         | 1.46                       |
| Perlite       | 7.4-11                            | 24          | -                    | -                             | 2.6-2.4                    |

It was concluded that SLA does not offer the same degree of thermal resistance as does expanded perlite. Therefore, SLA may not be a good substitute for expanded perlite unless its unit weight can be lowered significantly.

#### **4.3.6 Laboratory Evaluation of SLA in Horticultural Applications**

Expanded perlite, vermiculite and peat moss are used by nurseries in potted plants, shrubs, and trees. These materials improve soil porosity which helps develop a strong root structure, and also improve water retention. Of these, the most expensive materials are expanded perlite and vermiculite. Expanded slag was therefore tested as a partial or complete substitute for these two materials in horticultural applications. It was anticipated that the higher density of SLA would have a favorable impact on the durability of potting mixes used in large plants and green field applications. This test work was done at a commercial nursery which was selected based on their expertise in this field, the unique techniques they employ to control the supply of nutrients, and their willingness to test a new material.

Three batches of SLA were used for this test work, along with a standard perlite mix and a soil mix. The topsize and unit weight of the SLA used for this work is given below:

- Sample A: 1/4" topsize and unit weight of 35-40 lb/ft<sup>3</sup>
- Sample B: 1/4" topsize and unit weight of 20-30 lb/ft<sup>3</sup>
- Sample C: 1/4" topsize and unit weight of <20 lb/ft<sup>3</sup>
- Control 1: Perlite/vermiculite mix
- Control 2: Typical soil mix

The evaluation consisted of observation of the growth rate, general health, and appearance of tomato plants grown in a solarium. For this purpose, a number of potting mixes measuring approximately 1 cubic yard were prepared using expanded slag as a partial or total substitute for perlite and vermiculite which are added to improve porosity and water retention.

As may be seen in Table 34, the initial weight of the three SLA samples (6.75-10.5 lb) used to fill the flats was considerably higher than that of the other materials. Also, the maximum moisture content under full saturated conditions (19-29%) was considerably lower than that of the other materials. The customary frequency of watering and fertilizer addition was followed. This approach allowed comparison of water and fertilizer retention capacity. Plant growth was measured over a period of 20 days between 8 July 1997 and 28 July 1997, and the general condition of the plants reported. For the three samples, the best results were obtained for Sample B, where only one plant wilted during the entire period. In the case of Sample A, five

plants wilted, while for Sample C, seven plants wilted. This trend does not seem to follow the moisture retention capability of the three samples of expanded slag. The reason for this is not fully understood at this time and requires further studies.

**Table 34. Evaluation of Expanded Slag in Supporting Plant Growth**

|   | SLA<br>Sample<br>A   | SLA<br>Sample<br>B | SLA<br>Sample<br>C | Vermiculite | Perlite | Soil<br>Mix |
|---|--|--------------------|--------------------|-------------|---------|-------------|
| Unit wt, lb/ft <sup>3</sup>               | 35-40  | 20-30              | <20                | -           | -       | -           |
| Dry wt, lb                                | 10.5   | 9.5                | 6.75               | 4.25        | 4.0     | 5.5         |
| Wet wt, lb                                | 13.0   | 12.25              | 9.0                | 13.0        | 9.25    | 11.5        |
| Water retention, lb                       | 2.5  | 2.75               | 2.75               | 8.75        | 5.25    |             |
| Max. moisture content, wt%                | 19.2   | 22.4               | 30.6               | 67.3        | 56.8    | 52.2        |
| Water retention, lb per 100 lb dry solids | 24   | 29                 | 41                 | 206         | 131     | 109         |
| 7/8/97                                    | Planted and watered all flats                                  |                    |                    |             |         |             |
| 7/14/97                                   | Fertilized all flats with CalMag 15-5-15                       |                    |                    |             |         |             |
| 7/15/97, height, inches                   | 1  | 1¼                 | 1¼                 | 1¾          | 1½      | 1¾          |
| No. of plants that died*                  | 5  | 0                  | 4                  | 0           | 0       | 0           |
| 7/16/97                                   | Fertilized (20-10-20) vermiculite, perlite, and soil mix flats |                    |                    |             |         |             |
| 7/21/97, height (inches)                  | 1½   | 1¾                 | 1¾                 | 3¾          | 2¾      | 3¾          |
| No. of plants that died*                  | 5  | 0                  | 4                  | 0           | 0       | 0           |
| 7/22/97                                   | Fertilized all flats with 20-10-20                             |                    |                    |             |         |             |
| 7/28/97, height (inches)                  | 3  | 2¾                 | 2½                 | 7           | 7       | 8           |
| No. of plants that died*                  | 5  | 1                  | 7                  | 0           | 0       | 0           |

\*Out of 18 plants per flat.

To identify the best application for SLA in horticultural application, we held a discussion with the nursery researcher. Based on the limited data generated it is believed that slag can be used in the following ways:

- SLA can be used as a partial substitute for conventional potting materials.
- The low moisture retention capacity of the SLA can be accommodated by using it along with materials that have a higher moisture retention capacity.
- The pores in the SLA provides an excellent root support structure, and the superior strength of the SLA indicates that it will have a longer useful life than conventional perlite and vermiculite-based materials.



- The higher unit weight of the SLA can add stability to larger potted plants, shrubs, and trees, such as those in containers over 3 gallons in capacity. This stability is currently achieved using bark chips.
- Due to its higher mechanical strength, SLA may be used in outdoor green fields where perlite degrades rapidly due to its weaker strength.

#### 4.3.7 Evaluation of SLA in Production of Cement Panels (Waterproof Boards)

Based on evaluation of the test results for the use of SLA in making insulating concrete, another similar application was identified: production of lightweight cement concrete panels used in the construction of bathrooms and other areas where walls are exposed to moisture. This is a relatively new but fast-growing application that requires aggregates with unit weights in the 35-45 lb/ft<sup>3</sup> range. These cement panels are used to ensure a waterproof, smooth and highly stable surface for the application of ceramic tiles. A Praxis engineer visited a panel manufacturer's laboratory to discuss material requirements and provide the results of earlier tests. The laboratory completed an initial evaluation of the SLA and found the material satisfactory. The test product showed excellent potential due to its low unit weight and excellent workability imparted by the unique slag particle shape. Samples of SLA I were sent to the manufacturer for laboratory evaluation. Exploratory tests indicated that SLA could be used as a substitute for conventional lightweight aggregates to make these panels. Two 55-gallon drum samples of 10 x 50M SLA III were then supplied to the manufacturer, along with cyclone fines. One of the drums contained 35-40 lb/ft<sup>3</sup> SLA, and the other contained 40-45 lb/ft<sup>3</sup> SLA. SLA III was used for these tests as there was an insufficient quantity of SLA I in the required size fraction and unit weight.

In testing the suitability of expanded slag for use as panels, a 1:4 ratio of cement to coarse slag was used, without additives. Fine slag was added to adjust the unit weight and to improve the particle size distribution of the mix. Specimen bars (1" x 1" x 12") were cast and cured in a moist environment. After 3 and 7 days, the bars were removed and tested for Modulus of Rupture per ASTM C-293 using a 3-point bend test. The mix design used and results are given in Table 35.

**Table 35. Mix Design and Modulus of Rupture Test Results for SLA in Panel Application**

| Mix Design                           | Quantity |
|--------------------------------------|----------|
| Type I cement, g                     | 150      |
| Coarse slag, g                       | 600      |
| Fine slag, g                         | 200      |
| Water, g                             | 200      |
| <b>Strength Evaluation</b>           |          |
| Unit weight, lb/ft <sup>3</sup>      | 78.27    |
| Modulus of Rupture (ASTM C 293), psi |          |
| 3-day                                | 171.0    |
| 7-day                                | 178.1    |

The 3-day flexural strength of 171 psi compares well with the minimum value of 100 psi for autoclave cellular concrete used for the production of wallboard.

The unit weight of the current sample equates to 3.26 lb per 12" x 12" x 1/2" piece, which is almost identical to the target weight of 3.2 lb. In order to improve upon the flexural strength of this material, the cement-to-aggregate ratio or the ratio of coarse to fine sand may be increased.

It was concluded that SLA may be used in making cement panels. As was the case with the roof tile application, the flexural strength of wall panels made from SLA would be improved by optimizing the particle size distribution, the use of chemical additives, and the addition of cement.

#### **4.4 Economic Analysis of Expanded Slag Production and Utilization (Task 2.4)**

The objective of this task was to develop cost estimates for commercial production of slag lightweight aggregates (SLA) and to study the process economics. Two approaches were used in this evaluation:

- The economics of SLA production was compared with slag disposal.
- The economics of SLA production was evaluated with respect to the estimated market value of end products.

The comparative evaluation with respect to the costs of disposal involved establishing the disposal costs of slag based on experience with similar wastes such as fly ash. The avoided costs of disposal were then compared with the costs of producing SLA. The premise was that the slag could be pyroprocessed and then given away as an alternative to disposal. However, since the costs of SLA production were considerably higher than the avoided costs of disposal, pyroprocessing could not be justified as an alternative to disposal.

The second evaluation involved establishing the economics of SLA production and utilization using the estimated market price of the expanded slag. For this assessment a market survey was conducted to establish the regional prices of conventional LWAs and ULWAs. These data were used to establish the prices that the slag products could command in each application. This evaluation took into consideration the fact that slag is a new material and would be expected to command lower prices even if it performed better than the conventional applications. The projected prices were then used to develop the economics as discussed in this section.

Estimation of the costs of expanded slag production involved developing a process flowsheet based on pilot plant operations data generated during the project, compilation of a list of process equipment needed for physical and pyroprocessing of slag to produce marketable lightweight aggregate products, and development of equipment-factored capital cost estimates. Two sizes of SLA production plants were considered in this study, as described below:

- A plant to process slag generated from a gasifier facility with a 200-MW equivalent capacity. Such a facility typically uses 2,000 tons/day of bituminous coal containing 10% ash. Depending upon the carbon conversion rate, this facility may generate 220 tons/day of slag containing 10% char. This approximates the size of a number of existing gasifiers in the United States.

- A plant to process slag generated from a gasifier facility with a 400-MW equivalent capacity. Such a facility would typically use 4,000 tons/day of bituminous coal containing 10% ash. Depending upon the carbon conversion rate, this facility may generate 440 tons/day of slag containing 10% char. This would approximate the feed capacity of a typical commercial LWA plant that currently uses conventional expandable clays.

The relative advantages and disadvantages of the following options were considered:

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

Several factors, such as the need to process slag for char removal at the gasifier site and the costs of transporting slag to an off-site production facility, play a significant role in evaluating these options. It was concluded that, by and large, the SLA production facility would be more efficient if it were integrated with the gasifier operation. However, this does not preclude the sale of slag to an off-site LWA production facility which may be interested in using it as an alternative feed material to shale.

Since the commercial SLA production plant is assumed to be located "across the fence" from a gasifier facility, water and power requirements are assumed to be available at market cost at the site. The SLA production facility is envisaged to consist of two sections:

- (i) Slag receiving and processing section for recovery of char, and
- (ii) Pyroprocessing section for SLA production and product storage.

Coarse (+10 mesh) slag will be used to produce SLA in discrete particle form, and the fines will be used to produce extruded pellets of the desired size using a clay binder prior to expansion using the same pyroprocessing equipment. The facility includes product crushing equipment and product storage and handling bins sized in accordance with modern LWA industry practices. Major process equipment costs were estimated based on the above assumptions and used to prepare equipment-factored capital cost estimates. The capital and operating costs thus generated have an accuracy of  $\pm 25\%$ , which is considered sufficient for conducting first-level economic assessments.

Slag expansion process energy requirements were estimated based on the pilot plant operations data generated during the project and scaled-up by Fuller Co. Labor and other costs were estimated based on industry experience.

The basic data were generated during Phase I and updated where needed in Phase II. A computer worksheet was developed and used to conduct economic evaluations of various alternative scenarios, as discussed in subsequent sections.

#### 4.4.1 Costs of Production of LWA and ULWA Made from Expanded Slag

As a first step, the costs of production of various expanded slag products were estimated for two different sizes of commercial-scale plants. The design criteria for a plant processing slag from a 200-MW capacity gasifier are given in Table 36. Similarly, material balances were generated for the 400-MW system and are provided in the same table.

**Table 36. Design Criteria for Plant to Process Gasifier Slag**

| Criteria                        | 200 MW                 | 400 MW                 |
|---------------------------------|------------------------|------------------------|
| Coal usage, t/d                 | 2000                   | 4000                   |
| Coal ash, %                     | 10                     | 10                     |
| Plant operation, days/year      | 365 @ 90% availability | 365 @ 90% availability |
| Slag generated, t/d             | 220<br>(72,270 t/y)    | 440<br>(144,540 t/y)   |
| Prepared slag quantity, t/d     | 161.3<br>(52,987 t/y)  | 322.6<br>(105,974 t/y) |
| Char primary concentration, t/d | 58.7                   | 117.4                  |
| Char concentration (fuel), t/d  | 17.6                   | 35.2                   |
| Reject slag (disposal), t/d     | 41.1                   | 82.2                   |

The reject slag, which has a high char content, is assumed to be a disposal stream for purposes of SLA production in this evaluation. However, this stream can be processed further to remove the char fraction from it. The remaining fine slag could then be used with the rest of the minus 10-mesh fines to prepare extruded pellets.

The following assumptions were made in projecting SLA production costs:

- Expanded slag products with unit weights ranging between 20 and 50 lb/ft<sup>3</sup> would be produced by controlling the expansion temperature. SLA product densities would be targeted to match the requirements of most LWA applications and selected ULWA applications.
- Fuel requirements for the two sizes of plants, as estimated by the kiln designer, are given in Table 37. The char recovered from the slag is assumed to provide 50% of the fuel requirements for pyroprocessing using a rotary kiln. In a fluidized bed system, the char would provide up to 80% of the fuel requirements.

**Table 37. Fuel Requirements for Various Case Studies**

| Case                    | Slag Feed (t/d) | Fuel                | Fuel Rate (Million Btu/ton of feed) | Btu from Char (%) |
|-------------------------|-----------------|---------------------|-------------------------------------|-------------------|
| A1: Small rotary kiln   | 220             | Coal & char         | 2.32                                | 50                |
| A2: Large rotary kiln   | 440             | Coal & char         | 1.80                                | 50                |
| B1: Small fluidized bed | 220             | Bunker C oil & char | 2.50                                | 80                |
| B2: Large fluidized bed | 440             | Bunker C oil & char | 2.50                                | 80                |

- Coal is the preferred fuel for a rotary kiln system because it is relatively cheap and readily available at the gasifier site. The cost of 11,500 Btu/ton of coal delivered to the site is assumed at \$30/ton or \$1.30/MBtu. Oil would be the preferred fuel for a fluidized bed expander system. The cost of Bunker C oil is assumed at \$2.80/MBtu.
- A plant useful life of 20 years is assumed, and straight-line depreciation is used in assessing plant capital expenses.
- Plant capital costs are allowed a contingency of 15%.
- The cost of borrowing capital is assumed at 8%, and interest expenses are applied to operating costs.

**SLA-Based Lightweight Aggregate Production Costs Using a Rotary Kiln.** Based on the assumptions given above, SLA production costs were compiled for two plant sizes for both the rotary kiln and fluidized bed pyroprocessing methods. Table 38 provides estimated costs for production of SLA at 220 t/d and 440 t/d feed capacity respectively using similar (rotary kiln) processing systems. The table also provides current conventional LWA production costs which were compiled based on a survey of four plants. The costs compiled for the slag processing plant assume that the necessary capital is borrowed at 8% interest.

As may be seen in the table, the cost advantages for the SLA production operation are provided by (i) lower overall energy requirements due to the lower temperature of expansion, (ii) the ability to use char as a fuel in the kiln to meet 50% or more of the total energy requirements, and (iii) the absence of shale mining costs. The disadvantages are: (i) high interest on capital expenses and (ii) the inability to take advantage of economies of scale in a 220-t/d operation. However, a larger plant servicing the output of a 400-MW capacity gasifier would be able to use a large rotary kiln of a comparable size to those used in most commercial LWA operations.

**Table 38. Comparative Costs of Kiln Production of LWA vs. SLA**

| Cost Item              | Current Method<br>Using Shale,<br>\$/ton <sup>(1)</sup> | SLA \$/ton <sup>(2)</sup> | SLA, \$/ton <sup>(3)</sup> |
|------------------------|---|---------------------------|----------------------------|
| System type            | Large rotary kiln                                       | Small rotary kiln<br>(A1) | Large rotary kiln (A2)     |
| Fuel                   | Fuel Oil  | Coal & char               | Coal & char                |
| Mining and preparation | 6.00  | -                         | -                          |
| Transport ore to plant | 0.50  | -                         | -                          |
| Processing Costs       |   |                           |                            |
| Clay binder            | -   | 1.45                      | 1.45                       |
| Labor (O&M)            | 6.23  | 7.50                      | 6.25                       |
| Fuel                   | 5.09  | 2.12                      | 1.64                       |
| Power                  | 1.37  | 1.35                      | 1.35                       |
| M&S                    | 1.85  | 1.94                      | 1.48                       |
| Other                  | 1.11  | 1.10                      | 1.10                       |
| Overhead               | 2.24  | -                         | -                          |
| Depreciation           | 5.71  | 5.62                      | 4.28                       |
| Interest on capital    | Unknown   | 8.99                      | 6.85                       |
| Total product costs    | 30.10   | 30.07                     | 24.40                      |

- (1) Fuller survey of four U.S. LWA plants; mining costs added later.
- (2) Praxis/Fuller estimate for 220-t/d raw slag system.
- (3) Praxis/Fuller estimate for 440-t/d raw slag system.

***SLA-Based Ultra-Lightweight Aggregate Production Costs Using a Fluidized Bed Expander.***

In Table 39, the estimated costs for producing ULWA from slag using the fluidized bed method are presented alongside those for conventional ULWA production. The fluidized bed expander manufactured by Fuller Co. was used as it provides improved control of the product unit weight. The estimated cost of production of conventional ULWA is based on information provided by a leading manufacturer of expanded perlite. The cost advantages for the SLA operation are similar to those described in the section above. The disadvantages include the inability (thus far) to produce a true ultra-lightweight product (that is, unit weight in the 4-12 lb/ft<sup>3</sup> range) from slag. However, we have produced expanded slag products with unit weights as low as 16-18 lb/ft<sup>3</sup>, and it is assumed that further reduction is achievable with more testing and development work.

In comparing the production costs of expanded slag with those of expanded perlite, a volumetric correction may need to be applied to the slag because it has a considerably higher unit weight in lb/ft<sup>3</sup>. As may be seen in the above table, the production costs of expanded slag would be lower than those of conventional perlite-based ULWAs. In addition, the equivalent SLA product has a higher strength which can provide many advantages.

**Table 39. Comparative Costs of Producing ULWA vs. SLA**

| Cost Item                            | Current Method<br>Using Perlite<br>\$/ton | SLA, \$/ton<br>16 lb/ft <sup>3</sup> | SLA, \$/ton<br>16 lb/ft <sup>3</sup> |
|--------------------------------------|---|--------------------------------------|--------------------------------------|
| Processing method                    | Vertical shaft furnace                    | Small fluidized bed<br>(B1)          | Large fluidized<br>bed (B2)          |
| Mining and preparation               | 40.00                                     | -                                    | -                                    |
| Shipping ore to plant                | 40.00                                     | -                                    | -                                    |
| <b>Processing Costs</b>              |   |                                      |                                      |
| Binder for fines                     | -   | 1.45                                 | 1.45                                 |
| Labor                                | 12.00                                     | 7.50                                 | 6.25                                 |
| Fuel                                 | 8.00                                      | 1.34                                 | 1.29                                 |
| Power                                | 4.50                                      | 1.35                                 | 1.35                                 |
| M&S                                  | 3.00                                      | 1.70                                 | 1.29                                 |
| Other, loading                       | 2.00                                      | 1.10                                 | 1.10                                 |
| Overhead                             | 10.00                                     | -                                    | -                                    |
| Depreciation                         | 4.75                                      | 4.63                                 | 3.52                                 |
| Interest                             | Unknown                                   | 7.41                                 | 5.63                                 |
| Total product costs                  | 124.25                                    | 26.48                                | 21.87                                |
| Costs after volumetric<br>correction | 124.25                                    | 52.96                                | 43.74                                |

The assessments in the tables above indicate that the production costs for SLA are generally comparable to or lower than those for conventional LWAs, depending upon economies of scale. SLA production costs would be considerably lower with larger-scale production.

With regard to ULWAs, production costs of comparable SLA products are estimated to be significantly lower than those for conventional materials provided that the higher product densities of slag-based ULWA are acceptable. Even if a 2-to-1 correction factor is applied for the higher density of expanded slag (which necessitates use of a larger quantity for the same volumetric fill), slag-based ULWA is far cheaper to produce than the conventional products.

#### **4.4.2 Market Assessment of Conventional LWA and SLA**

The objectives of this subtask were to obtain an initial assessment of the market value of various conventional LWA products targeted for substitution by slag and to estimate the market value of the corresponding products if they were made from expanded slag. The market value of LWA is used to estimate the projected value of the SLA taking into account its quality and performance relative to the conventional materials. As a first step, various trade associations and major users of LWA and ULWA were contacted to obtain price structures and marketing information. Contacts with these organizations allowed us to gauge accurately the current sale prices of various aggregates. The organizations contacted included:

- Expanded Shale, Clay and Slate Institute
- Perlite Institute
- National Concrete Masonry Association.

**Conventional Lightweight Aggregate Production, Costs, and Markets.** Development of a market assessment for SLA included identification of the current market for conventional LWAs and ULWAs and specific applications for which SLA would be an acceptable substitute. According to data from sources including the U.S. Bureau of Mines and from Mineral Commodity Summaries, U.S. Geological Survey, 2001, summarized in Table 40, production and consumption of naturally occurring and manufactured lightweight materials was 8.1 million tons in 1996, including expanded shale production of 4.2 million tons. These products are typically sold for \$20-\$45 per ton. This excludes fly ash, which has a unit weight of 70 lb/ft<sup>3</sup> and is also used as a medium lightweight material.

Consumption and production of these materials is greatly dependent on production and transportation costs. Therefore, if cheaper by-product materials could be used to produce these products, especially at lower energy requirements, consumption would be significantly increased.

**Table 40. U.S. Production of Expanded Shales, Clays, and Volcanic LWAs**

| <b>Mineral Material</b>                | <b>1990</b> | <b>1991</b> | <b>1996 (est.)</b> |
|--|-------------|-------------|--------------------|
| Shales and clays, million tons         | 4.18        | 3.96        | 4.22               |
| Pumice, pumicite, million tons         | 0.49        | 0.44        | 0.89               |
| Volcanic cinders, scoria, million tons | 3.20        | 3.20        | 3.0                |
| Total, million tons                    | 7.87        | 7.60        | 8.11               |

**Conventional Ultra-Lightweight Aggregate Production, Costs, and Markets.** Conventional ULWAs have unit weights in the range of 4-12 lb/ft<sup>3</sup> and are produced by thermal expansion of perlite and vermiculite ores at temperatures of 1600-2000°F. Their low unit weight and thermal conductivity (as low as 0.35 Btu-in/h-°F at a loose weight of 2.5 lb/ft<sup>3</sup>) make ULWAs ideal insulating materials for loose fill insulation and aggregates for the manufacture of insulating concrete and numerous other insulation applications. Other applications for expanded perlite include filtration media, industrial fillers, abrasive in cleaners and polishes, soil amendment for horticulture, carrier of chemicals for pesticides and fertilizers, and acoustic material. The current annual production of lightweight and ultra-lightweight materials is estimated at 10 million tons.

U.S. Department of the Interior production figures for expanded perlite and vermiculite are given in Table 41. The domestic consumption of perlite ore in 1996 was 848,000 tons, which takes into account the import of 138,000 tons and export of 42,000 tons. The increase in production in 1996 is related to higher demand due to a pickup in industrial and commercial construction activity. Production of vermiculite in 1996 was 193,000 tons. The market value of expanded perlite products was \$204 per ton. Expanded perlite typically retails at \$2.00/ft<sup>3</sup>, which corresponds to \$500/ton based on an average unit weight of 8 lb/ft<sup>3</sup> for the expanded products. The consumption of these materials is also highly sensitive to their costs of production. The availability of low-cost alternative feedstocks such as slag could increase the consumption of these materials.



**Table 41. U.S. Production of Perlite and Vermiculite Ores**

| Mineral                            | 1989    | 1990    | 1991    | 1996    |
|------------------------------------|---------|---------|---------|---------|
| Perlite, tons/year                 | 601,000 | 635,000 | 567,000 | 772,000 |
| Vermiculite, tons/year             | 275,000 | 230,000 | 185,000 | 193,000 |
| Total ULWA raw material, tons/year | 876,000 | 865,000 | 752,000 | 965,000 |

**Economics of Production of ULWA from Slag.** Slag has been demonstrated to produce an expanded aggregate, which may be used as a substitute for ultra-lightweight aggregates (ULWA) for some applications. The technical and economic advantages of producing ultra-lightweight aggregates from slag include the fact that, being a waste material, it is available at no or low cost. Prepared perlite ore, in contrast, sells at \$80 per ton, which includes the high costs of transportation from New Mexico to various production facilities. In contrast, no mining costs are involved for slag, energy requirements for expansion are lower than those for perlite expansion, and avoided disposal costs may be a major factor favoring its utilization.

**Assessment of Market Price of SLA.** In order to estimate sale prices of SLA for use in making various end products, we contacted manufacturers to conduct a market survey of the prices of structural LWAs by region and by application. The prices varied considerably by region, as indicated in Table 42.

**Table 42. Typical Regional Prices of Lightweight Aggregates**

| Location   | \$/yd <sup>3</sup> | \$/ton |
|------------|--------------------|--------|
| East coast | 17.00              | 25.00  |
| Midwest    | 20.00              | 30.00  |
| West coast | 30.00              | 44.00  |
| Average    | 22.33              | 33.00  |

Prices also varied for each application due to factors such as quality and product size preparation. Based on the above, typical prices quoted for major applications are given in Table 43. Based on our experience, we estimated the price that the SLA would command in each application. The estimated SLA prices, along with the slag fraction that would be used to produce it, are given in the same table.

**Table 43. Estimated Market Prices for LWAs, ULWAs, and SLA by Application**

| Application                         | LWA Price (\$/ton) | SLA Price (\$/ton) | SLA Product    |
|-------------------------------------|--------------------|--------------------|----------------|
| <b>Lightweight Aggregates</b>       |                    |                    |                |
| Block aggregate                     | 37.00              | 30.00              | 10 x 50M SLA   |
| Structural concrete                 | 45.00              | 35.00              | Extruded fines |
| Roof tiles                          | 50.00              | 40.00              | Extruded fines |
| SLA weighted average                | --                 | 34.75              | Entire product |
| <b>Ultra-lightweight Aggregates</b> |                    |                    |                |
| Expanded perlite                    | 150.00             | 40.00              | +10M SLA       |

Since a new, unproven product would command a lower price, the sale prices for SLA aggregates were established at \$30/ton for block aggregates, \$35/ton for structural aggregates, and \$40/ton for roof tile and ultra-lightweight aggregate applications. Using a product mix based on the percentage of coarse and fine slag, the weighted average price of SLA was estimated at \$34.75/ton. This price was used for purposes of economic evaluation of SLA production.

#### **4.4.3 Solid Waste Management Costs**

The objectives of this subtask were to compile solid waste management costs for slag from a gasifier on a \$/ton and \$/ft<sup>3</sup> basis. When disposal is avoided through utilization, these costs are used as credits in the economic evaluation of expanded slag. Solid waste management costs typically include the following:

- Site preparation
- Handling and transportation
- Storage and compaction
- Land reclamation
- Runoff, drainage, and seepage monitoring.

Solid waste management costs tend to be highly site-specific due to transportation and site-related costs. Therefore, they vary considerably depending on the distance over which the solid waste has to be transported for disposal. These costs also vary on a regional basis depending on the availability of land for solid waste disposal. Thus, disposal costs in the northeastern United States are the highest due to the limited availability of disposal sites. Our information is that typical utility waste disposal costs range between \$10 and \$20 per ton. For purposes of this analysis, a value of \$15/ton is used as the disposal cost. Since these avoided costs would provide substantial savings to the gasifier operation, this amount could potentially be made available to the slag aggregate production facility as a tipping fee per ton of slag accepted.

#### **4.4.4 Economic Evaluation of SLA Production**

An economic evaluation was conducted for a hypothetical integrated SLA production facility which would produce lightweight and ultra-lightweight products of various unit weights for local and adjoining markets. This facility was assumed to be located at the gasifier site and integrated with the gasifier slag handling operation in order to eliminate double handling of slag. The facility would process the raw slag for char recovery, then pyroprocess the char-free slag to produce lightweight and ultra-lightweights aggregates. The recovered char would be recycled to the gasifier. In order to estimate the costs of this facility, a process flowsheet was developed based on pilot plant operations data generated during the project, after which process equipment-factored capital cost estimates were developed. Two sizes of SLA production plants were considered in this study, using two commercially available pyroprocessing technologies. As a result, four case studies were developed to study the process economics, as described below.

- Case A1: Small rotary kiln plant for SLA production using the slag output from a 200-MW equivalent gasifier generating 220 t/d slag
- Case A2: Large rotary kiln plant for SLA production using the slag output from a 400-MW equivalent gasifier generating 440 t/d slag
- Case B1: Small fluidized bed plant for SLA production using the slag output from a 200-MW equivalent gasifier generating 220 t/d slag
- Case B2: Large fluidized bed plant for SLA production using the output from a 400-MW equivalent gasifier generating 440 t/d slag.

A computer worksheet was developed to compile the capital costs and conduct overall economic analyses for various case scenarios for the production of SLA products. The worksheet covered the following issues:

- Capital costs for slag handling, preparation, pyroprocessing, and contingencies
- Direct operating costs (operating and maintenance labor, maintenance materials, consumables, and other costs)
- Indirect costs (depreciation, interest on capital)
- Credit for avoided costs of disposal of slag
- Costs for producing SLA and the impact of avoided disposal costs on SLA production
- Economics (payback period and return on investment) based on market prices commanded by SLA products.

The economic advantages of SLA over conventional materials are that no mining costs are involved and pyroprocessing costs are almost identical. Since slag expands at a temperature ~400°F lower than shale, it requires 50% less energy during the thermal processing step. Adjustments were made for various items where additional costs are incurred for slag expansion. The preliminary economics of production of SLA vs. conventional LWA based on data generated during pilot kiln operation are summarized in Table 44. The data indicate that the production costs of SLA are essentially the same as those for conventional LWAs.

As may be seen in Table 44, SLA production costs for the small rotary kiln (Case A1) at \$30.06/ton are close to the production costs of conventional LWAs. Being a small operation, this case does not benefit from economies of scale and would not be profitable unless the avoided costs of slag disposal are taken into account. For Case A2, the projected production costs are \$24.40/ton, which is fairly competitive with production costs at typical conventional LWA plants.

Cases B1 and B2, based on the use of small and large fluidized bed systems respectively, would be considerably more competitive because of lower capital and operating costs. Therefore, such systems should be considered for commercial SLA production, especially for lower-capacity plants. The economics for the larger-sized plant (B2) are especially attractive if the avoided costs of slag disposal are taken into account, as indicated by the payback period of under three years.

**Table 44. Economic Analysis Summary**

|  | Case A1           | Case A2           | Case B1         | Case B2         |
|--|-------------------|-------------------|-----------------|-----------------|
| <b>Plant Operational Data</b>                  |                   |                   |                 |                 |
| Pyroprocessing System                          | Small rotary kiln | Large rotary kiln | Small fluid bed | Large fluid bed |
| System fuel                                    | Coal/char         | Coal/char         | Bunker C/char   | Bunker C/char   |
| Slag feed, t/d                                 | 220               | 440               | 220             | 440             |
| Pyroprocessing feed, t/d                       | 188               | 377               | 188             | 377             |
| Capital costs, \$                              | 6,960,375         | 10,600,000        | 5,735,625       | 8,700,000       |
| Total fuel rate Btu/t                          | 2.32              | 1.80              | 2.50            | 2.50            |
| Purchased fuel costs, \$/MBtu                  | 1.30              | 1.30              | 2.68            | 2.68            |
| Fuel component from char, %                    | 30                | 30                | 80              | 80              |
| Pyroprocessing throughput, t/y                 | 61,921            | 123,841           | 61,921          | 123,831         |
| Direct O&M costs, \$/t                         | 15.46             | 13.27             | 14.44           | 12.73           |
| Indirect costs (depreciation & interest), \$/t | 14.61             | 11.13             | 12.04           | 9.14            |
| Total SLA production costs, \$/t               | 30.07             | 24.40             | 26.48           | 21.87           |
| Avoided disposal credit, \$/t                  | -14.23            | -14.23            | -14.23          | -14.23          |
| Net SLA production cost, \$/t                  | 15.83             | 10.17             | 12.25           | 7.64            |
| <b>Economic Analysis</b>                       |                   |                   |                 |                 |
| SLA Production, t/y                            | 61,921            | 123,841           | 61,921          | 123,831         |
| Average sale price, \$/t                       | 34.74             | 34.74             | 34.74           | 34.74           |
| Total sales revenues, \$/yr                    | 2,151,106         | 4,302,213         | 2,151,106       | 4,302,213       |
| Projected gross margin, \$/t                   | 18.91             | 24.57             | 22.49           | 27.10           |
| Projected gross margin, \$/y                   | 1,170,801         | 3,043,151         | 1,392,780       | 3,356,469       |
| Payback period, years                          | 5.9               | 3.5               | 4.1             | 2.6             |
| Return on investment, %                        | 16.8              | 28.7              | 24.3            | 38.6            |

Note: All capital costs and prices are given in 1996 dollars.

## 5.0 ADDITIONAL SLAG UTILIZATION WORK

Several tasks were added to the project during its course. These included:

- Application of the Praxis slag/char separation process to the TEC slag (Task 2.5)
- Exploration of the feasibility of using slag a raw material in portland cement (Task 2.6)
- Pilot studies on the utilization of slag as a raw material in portland cement (Task 2.7)
- Investigation of the pozzolanic activity of slag.

The results of this work are discussed in the following sections.

### 5.1 Separation of Char from Slag from Tampa Electric IGCC Plant (Task 2.5)

The slag from entrained-flow coal gasifiers typically contains 15-25% carbon, termed char. Char originates from the unconverted carbon in the coal. The presence of carbon in the slag is a major hindrance to its utilization. Praxis has demonstrated that slag generated from entrained-flow gasifiers can be processed to remove its char content, thus producing a carbon-free slag which can be used in a number of high-volume applications such as aggregate in cement concrete and road construction and high-value applications such as feedstock for lightweight aggregate production. The recovered char may be blended with coal and utilized as a fuel for power generation or recycled to the gasifier.

DOE added a task to the existing contract to demonstrate the applicability of the char separation process developed by Praxis to the slag generated at Tampa Electric Company's (TEC) Polk Power Station IGCC facility. This 250-MWe facility, co-funded by DOE, was installed under Round III of the Clean Coal Technology Demonstration Program.

The major objectives of the study were to:

- Test the TEC slag for char removal (Subtask 2.5.1)
- Develop a conceptual design for a char separation facility (Subtask 2.5.2)
- Develop the economics of clean slag production at TEC (Subtask 2.5.3)

#### 5.1.1 Laboratory Testing of TEC Slag for Char Removal

An "as generated" slag sample weighing approximately ½ ton (two 55-gallon drums) was procured from the TEC IGCC plant to test its performance using Praxis' char separation process. The feed sample was analyzed at 68.4% ash (or 31.6% carbon). Please note that this char content is somewhat higher than normal as the facility was undergoing commissioning and testing when the sample was obtained. The slag generated during normal operations has a lower carbon content of ~20% as subsequently reported by plant personnel.

The Praxis slag/char separation process consists of five processing steps:

1. Screening and Recovery of Char-Free Slag
2. Gravity Separation of Slag and Char
3. Reprocessing of Char to Upgrade its Carbon Content (to achieve a user-specified char grade)
4. Dewatering of Slag Product. The recovered slag product can be dewatered using mechanical dewatering equipment and by reducing moisture through natural drainage in bins or storage piles. This would achieve product moisture levels corresponding to typical commercially available wet-screened aggregates.
5. Handling of Recovered Char Product. Since the recovered char is assumed to be recycled to the gasification process, it does not need to be dewatered but can be retained in slurry form and mixed with the new coal feed to the wet grinding circuit.

There is also an optional step (3A) to further reduce the residual char content in the clean slag.

The TEC slag sample was processed using the 400-lb/hour pilot plant set up to process the 20-ton slag sample for the main project. Two product fractions were produced:

- A char-free (clean) slag fraction
- A char fraction (primary char) containing ~35-40% ash (or 65-60% carbon).

Subsequently, the char fraction was reprocessed to upgrade its carbon content (Step 3). Table 45 presents the results for the three stages of processing. As may be seen, 14.8% of the material containing essentially no carbon was recovered in the screening step. This step, which reduced the amount of feed material to the gravity separation unit by only a small amount, is designed primarily to prevent any coarse material from entering the separation unit.

In the gravity separation step, 31.4% of the feed material, containing no carbon (i.e., 100% ash), was recovered. This, combined with the screen fraction, accounts for a total of 46.3% of the original sample recovered in the form of saleable carbon-free slag. The remaining 53.7% of the material was mass analyzed at 59.8% carbon (or 41.2% ash).

**Table 45. Results of Slag/Char Separation for TEC Slag**

| Step     | Process   | Process Feed |      | Char Product |      | Slag Product |      |
|----------|---|--------------|------|--------------|------|--------------|------|
|          |   | Wt%          | Ash% | Wt%          | Ash% | Wt%          | Ash% |
|          | TEC raw slag feed                                 | 100.0        | 68.4 |              |      |              |      |
| 1        | Slag/char separation using Praxis process         | 100.0        | 68.4 | 36.1         | 21.2 | 63.9         | 95.0 |
| 1,2,3    | Other slag combined results, whole slag basis     | 100.0        | 84.9 | 21.4         | 34.1 | 78.6         | 98.8 |
|          | Slag/char separation with secondary recovery step |              |      |              |      |              |      |
| 1,2,3,3A | Combined results, whole slag basis                | 100.0        | 68.4 | 38.4         | 21.5 | 61.6         | 97.6 |

**Reprocessing of Char to Upgrade its Carbon Content (Process Step 3):** This step is aimed at further upgrading the recovered char fraction to 70-80% carbon (or 20-30% ash). The process parameters for this step are slag-dependent and need to be established for each slag by laboratory

testing. In most cases, the slag recovered from this step would be blended with the main slag stream recovered from Process Steps 1 & 2. All of the recovered slag is then dewatered in Process Step 4. The results of the char upgrade step for the TEC slag are given in Table 45 above. This step recovered an additional 17.6% material containing 82% ash (or 18% carbon). This slag, when combined with the slag fractions recovered previously, accounts for an overall slag recovery of 63.9%, containing 95% ash (or 5% carbon). This product meets TEC's target specifications for the carbon content of the slag product.

**Further Reduction of Char Content (Process Step 3A):** In the event that the char content of the slag product from Process Step 3 needs to be lowered further to permit its use for certain high-value applications, it can be processed by flotation, as indicated in Process Step 3A in Table 45. If this step is used, nearly zero solid waste disposal can be achieved at the gasifier plant. Praxis tested this step and the results were very encouraging. However, this line of testing was not pursued as it was not in the scope of work or requested by TEC. This processing step involves additional costs and the decision to use it can only be made after full consideration of the potential applications for which the slag and char are being prepared.

**Dewatering (Process Step 4):** The clean slag will be dewatered using vibratory screens and stored in a bin or stockpile. Because slag is a glass-like material, dewatering is relatively easy and would continue when it is placed in storage piles. Dewatering of the char was not included as it is assumed that the char would be recycled to the gasifier in slurry form.

Table 46 provides the results of char separation tests conducted by Praxis on other slag samples for comparison with the TEC results. As may be seen, most slags had considerably higher (64-85%) recovery of clean slag compared to the TEC sample, which had 46.3% clean slag recovery. The low recovery for the TEC slag is attributed to the fact that the sample, which was obtained during the early stages of gasifier commissioning and start-up, contained an unusually high carbon content. The plant reported that the slag generated subsequently contains ~20% carbon. Therefore, in the subsequent analysis recovery of 70% char-free slag was assumed.

**Table 46. Slag/Char Separation Results from Process Steps 1 & 2 for Various Slags**

| Slag                | Feed |       | Clean Slag |       | Char |       |           |
|---------------------|------|-------|------------|-------|------|-------|-----------|
|                     | Wt%  | Ash % | Wt%        | Ash % | Wt%  | Ash % | Carbon* % |
| Slag Sample 1       | 100  | 91.2  | 85.7       | 100.0 | 14.3 | 38.3  | 61.7      |
| Slag Sample 2       | 100  | 84.9  | 70.0       | 99.4  | 30.0 | 51.1  | 48.9      |
| Slag Sample 3       | 100  | 85.8  | 74.3       | 100.0 | 25.7 | 45.1  | 54.9      |
| Slag Sample 4       | 100  | 83.7  | 73.1       | 99.3  | 26.9 | 41.0  | 59.0      |
| TEC (Steps 1, 2, 3) | 100  | 68.4  | 63.9       | 95.0  | 36.1 | 21.2  | 78.8      |

\*Carbon content (determined by loss on ignition) would be 100% minus % Ash

### 5.1.2 Development of Conceptual Design for a Slag/Char Separation Facility

Tampa Electric Company's 250-MWe Polk Station IGCC facility uses a coal slurry feed consisting of Illinois No. 6 and Pittsburgh No. 8 coals which contain 10% ash and 2.5-3.6% sulfur. The facility uses approximately 2300 tons of coal per day on a dry basis. Clean slag target specifications of no more than 5% char were set by the plant to meet the requirements of a

potential buyer. However, since no specifications were provided for the char product, we did not consider Step 3A, which involves flotation. Upon completion of this subtask, TEC was to make a determination whether or not to include Praxis' design for a char separation process in its slag handling facility.

As noted above, the slag sample collected for testing had a relatively high carbon content (31.6% carbon or 68.4% ash), which may be due to the fact that the facility was undergoing commissioning and testing when it was collected. Our assumption that the slag generated during normal operations would have a lower carbon content was borne out by subsequent reports from plant personnel. Therefore, in the design of the facility, it was assumed that the slag feed would contain a maximum of 17% unconverted carbon or char by weight. This would generate ~276 tons/day of slag under post-commissioning steady-state operations. Assuming that the slag/char separation facility would be operated for one shift per day at ~90% availability, its required throughput capacity would be 40 tons/hour.

The installed capital cost for a char separation facility using ground storage (Steps 1-3) was estimated at \$307,000. The direct operating costs for the facility were estimated at \$0.84/ton of slag feed, which includes operating and maintenance labor, and maintenance materials.

### **5.1.3 TEC Clean Slag Production Economics**

The economics of producing a char-free, saleable slag product which can be used as a substitute for conventional aggregates such as sand and gravel were developed and compared with slag disposal costs. Disposal costs for such materials are typically in the \$10-\$20/ton range, depending upon the site conditions and the availability of space for disposal at the site. The slag processing costs of about \$0.84/ton are considerably lower than the costs of disposal. The price of conventional aggregates in the vicinity of the gasifier were estimated in the \$5-\$8/ton range, although for purposes of the economic evaluation a conservative figure of \$5/ton was used. The facility would generate product slag at 70-80% recovery on a feed basis. This analysis is presented in Table 47.



**Table 47. Slag/Char Separation Costs and Economics Summary**

| <b>Item</b>   | <b>Design Basis</b> |
|---|---------------------|
| <b>Plant Design Data &amp; Assumptions</b>            |                     |
| <i>Coal used</i>                                      | 2,300 tons/day      |
| Ash in coal (10%)                                     | 230 tons/day        |
| Slag generated  | 276 tons/day        |
| Slag processing facility operating schedule           | 8 hours/day         |
| Slag processing average feed                          | 34.5 tons/hour      |
| <i>Facility design basis</i>                          | 40.0 tons/hour      |
| <i>Gasifier operation</i>                             | 300 days/year       |
| Raw slag feed generated/year                          | 82,800 tons/year    |
| Slag saleable product, 70-80% of feed                 | 70%                 |
| Saleable slag produced/year                           | 57,960 tons/year    |
| <b>Facility Capital &amp; Direct Operating Costs*</b> |                     |
| <i>Capital costs for the facility</i>                 | \$307,000           |
| Direct operating costs per year                       | \$69,648/year       |
| Operating costs per ton                               | \$0.84/ton          |
| <b>Economics</b>                                      |                     |
| <i>Slag sale price</i>                                | \$5/ton             |
| Revenues generated from slag sale                     | \$289,800/year      |
| Net revenues after deducting operating costs          | \$220,152/year      |
| Payback period  | 1.4 years           |

\*All figures are given in 1998 dollars.

As may be seen in the table, there is a net minimum margin of \$4.16/ton, and the facility would generate net revenues of \$220,000 per year, with a payback period of less than two years. It was therefore concluded that separation of the char from the slag to make a saleable slag product is economically viable.

#### **5.1.4 Results of Applications-Oriented Testing of TEC Slag**

In view of the interest at TEC in using this technology, tests were conducted to identify and confirm the potential for utilizing the char-free slag in four applications. The results are summarized below.

**Cement Concrete Aggregate:** Tests were conducted to determine whether a 2,000-psi concrete could be made from the char-free slag using a mix comprising 5-6 sacks of concrete per cubic yard as is typically used in the production of masonry products (blocks, bricks, stepping stones, etc.), concrete grout, mortar mix, and ready mix, etc. These tests were performed at the laboratory scale using a mix design that had been developed for previous testing. Two-inch cubes were made to obtain 7-day and 28-day compressive strength values. The test results are given in Table 48. The results indicate that the TEC slag can be used as an aggregate to produce precast products and for concrete mixes requiring compressive strength values in the 2,000-4,000 psi range.

**Table 48. Utilization of Char-Free TEC Slag as Aggregate in Cement Concrete**

| Weight % Aggregates |      |       | Cement,<br>Sacks/Yd <sup>3</sup> | Compressive Strength,<br>psi |        |
|---------------------|------|-------|----------------------------------|------------------------------|--------|
| Coarse Aggregate    | Slag | Total |                                  | 7-Day                        | 28-Day |
| 50                  | 50   | 100   | 6                                | 3,975                        | 4,287  |
| 25                  | 75   | 100   | 6                                | 3,170                        | 3,487  |
| 0                   | 100  | 100   | 6                                | 3,479                        | 3,895  |

**Road Construction and Maintenance.** The char-free TEC slag was compared with similar slags for its suitability for utilization as a road construction aggregate, and was also checked against the duty requirements for the above application. The particle size and other general properties of the TEC slag are similar to those of most other slags. Therefore, it is considered to be suitable for road construction and maintenance applications such as sub-base and base material and seal coat aggregate.

**Industrial Material (Roofing Granule, Industrial Filler, Abrasive Grit).** A sieve analysis comparison of the char-free TEC slag indicated that it can be used as an industrial material for a variety of applications.

**Lightweight Aggregate Production.** Muffle furnace tests were performed to determine the suitability of the char-free TEC slag for making lightweight aggregates, based on the procedure used for many other slags previously. The results, given in Table 49, were positive. However, additional work is needed for the production of ultra-lightweight aggregates, which have unit weights below 12 lb/ft<sup>3</sup>.

**Table 49. Utilization of TEC Slag as Feedstock for LWA Production**

| Test No.                    | Feed    | 1    | 2    | 3    | 4    | 5    |
|-----------------------------|---------|------|------|------|------|------|
| Temp. °F                    | Ambient | 1450 | 1500 | 1600 | 1700 | 1800 |
| Unit Wt, lb/ft <sup>3</sup> | 109.0   | 94.2 | 88.9 | 80.0 | 64.0 | 55.0 |

### 5.1.5 Utilization of Slag/Char Separation Process at DOE-Funded IGCC Plants

Under this subtask, Praxis tested a slag sample obtained from the Wabash River Repowering Project IGCC plant to study its expansion characteristics. This slag was added to the test program with the objectives of extending the project findings to another slag, exploring the potential for removal of the char from the slag, and producing a high-strength expanded product with a unit weight in the 50-55 lb/ft<sup>3</sup> range which would meet the requirements for structural concrete applications. The results of the tests were presented to a management team representing the Wabash River Repowering Project, consisting of Destec (Dynege), and PSI Energy, at the gasification site. It was concluded that while specific process operating parameters may vary depending on the individual slag, the technology developed by Praxis for slag/char separation

and the production of lightweight aggregates could be applied at the commercial scale to all the gasification slags tested.

### 5.1.6 Conclusions

A sample of the slag from the TEC IGCC plant, which is currently disposed of as a solid waste, was obtained for testing. The sample contained 31.3% carbon (68.7% ash). Based on our testing and analysis, we arrived at the following conclusions:

- Tests with the TEC slag confirmed the results obtained for other slags that a saleable char-free slag product can be generated at the commercial scale. Application of Praxis' slag/char separation process resulted in two fractions:
  - A 63.9% slag fraction containing 5% carbon (the maximum acceptable limit for carbon in slag set by TEC)
  - A 46.3% saleable (0% carbon or 100% ash) slag product.

Higher recovery is achievable with slag samples obtained more recently.

- Weight percent recovery of char-free slag is a function of the carbon content of the as-generated slag. For the TEC sample, 70-80% recovery of char-free slag is possible.
- The study confirmed that the char-free TEC slag could be used in numerous commercial applications including as an aggregate in cement concrete. Tests also confirmed that it could be used in high-value applications such as feedstock for production of lightweight aggregates which sell for \$40/ton.
- Commercial-scale separation of slag and char can be accomplished by retrofitting the slag/char process modules as needed at the TEC IGCC plant. The Praxis process is especially applicable at this site (both in terms of the individual steps and as a total package) due to the high carbon content of the slag at this site.
- Both capital costs of the Praxis slag/char separation process at \$207,000 and operating costs at \$0.84/ton are low.

## 5.2 Laboratory Studies of Slag as Raw Material in Portland Cement Kiln Feed (Task 2.6)

The objectives of using coal gasification slag as a raw material in making Portland cement are threefold: (i) as a partial replacement for conventional raw materials, (ii) to explore the potential for using slag as a fluxing agent to reduce the firing temperature thus lowering kiln energy requirements, and (iii) to decrease the kiln retention time thus increasing the kiln capacity. Initially, bench-scale tests were conducted at Pennsylvania State University using a conventional high-temperature furnace. Although a high-temperature furnace is not quite comparable to the rotary kiln normally used for making cement clinker, its use as an initial test for the clinkering capability of the gasifier slag is appropriate. In the formation of cement clinker, the most important process is the liquid state sintering of the raw materials under high temperature in a kiln (usually of the rotary type). Therefore, the ability of slag to form a clinker is very important.

Basic mix designs for testing were developed based on the formulation of a conventional Type I portland cement. Several different levels of slag were tested, with the maximum dictated by the overall chemistry of the mix. A baseline portland cement kiln feed material was procured from a cement plant in Pennsylvania with the help of Fuller Co. and shipped to Pennsylvania State University for laboratory tests. A slag sample used previously for other tasks on this project was used in the laboratory testing after grinding to -200 mesh. The temperature and retention time of the raw material were monitored. 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.

**Composition of Portland Cement and Cement Nomenclatures.** The terminology pertaining to common portland cement is used in these sections on the use of slag as a portland cement kiln feed material. Portland cement clinker is produced by burning a mixture of limestone and clay or shale in a high-temperature rotary kiln. The resultant clinker contains a mixture of distinct oxide compounds. It is customary to report chemical analyses as contents of oxides since the compounds have empirical formulae given by the addition of the oxide formula. Although such relationships are useful for calculation of quantities they tell us nothing about the structural nature of the compounds. The typical chemical composition of portland cement clinker is given in Table 50.

**Table 50. Typical Composition of Cement Clinker**

| Oxides                         | Proportion, % | Symbol Used for Oxide |
|--------------------------------|---------------|-----------------------|
| SiO <sub>2</sub>               | 21.7          | S                     |
| Al <sub>2</sub> O <sub>3</sub> | 5.3           | A                     |
| Fe <sub>2</sub> O <sub>3</sub> | 2.6           | F                     |
| CaO                            | 67.7          | C                     |
| MgO                            | 1.3           | -                     |
| K <sub>2</sub> O               | 0.5           | K                     |
| Na <sub>2</sub> O              | 0.2           | N                     |
| SO <sub>3</sub>                | 0.7           | S                     |
| Free lime                      | 1.5           | -                     |
| H <sub>2</sub> O               | -             | H                     |

The compounds, on the other hand, are usually a mixture of these oxides. Therefore, a special shorthand notation is used to simplify formulas. As in refractories technology, single letters replace the usual oxides formula, as shown in Table 50.

For example, the most abundant compound in portland cement clinker is tricalcium silicate (3CaO-SiO<sub>2</sub>), which in shorthand notation is: C<sub>3</sub>S. The major compounds typically present in portland cement clinker are shown in Table 51.

**Table 51. Typical Portland Cement Composition With Major Compounds**

| Compounds         | Wt.% |
|-------------------|------|
| C <sub>3</sub> S  | 65.4 |
| C <sub>2</sub> S  | 12.9 |
| C <sub>3</sub> A  | 9.6  |
| C <sub>4</sub> AF | 7.9  |

**Laboratory Studies.** Prior to usage, the pre-blended baseline portland cement kiln feed material was subjected to x-ray diffraction analysis. The result is shown in Figure 3. TGA thermal analysis over the range of 122-1922°F (50-1050°C) of this material is shown in Figure 4. The x-ray diffraction pattern of the raw feed shows a typical portland cement kiln feed with diffraction peaks dominated by calcium carbonate (limestone). 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 ~1382-1607°F (750-875°C) temperature range, which corresponds to the liberation of CO<sub>2</sub> 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 CO<sub>2</sub> 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 1652°F (900°C) in 4 hours
- 1652°F (900°C) calcine for 4 hours
- 1652°F (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 2012°F (1100°C), 2192°F (1200°C), 2372°F (1300°C), and 2642°F (1450°C). Normal clinkering temperature in a cement kiln is about 2642°F (1450°C).

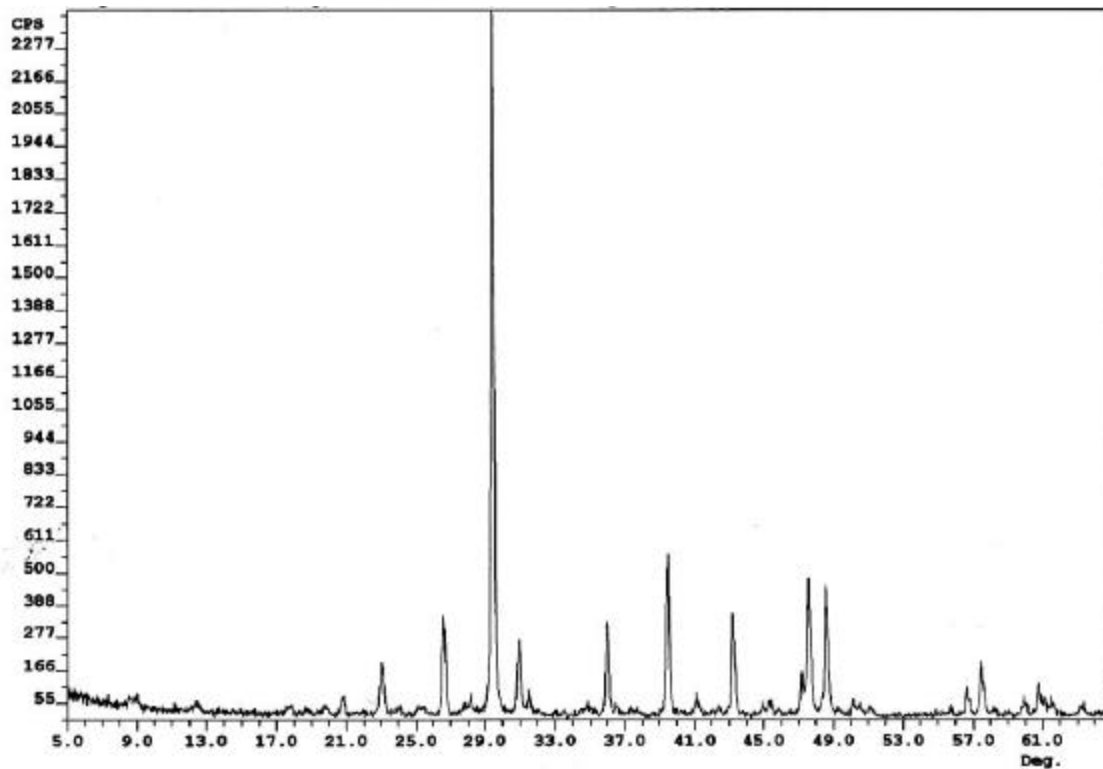


Figure 3. X-ray Diffraction Pattern of Raw Kiln Feed (5-65°, 2°/min)

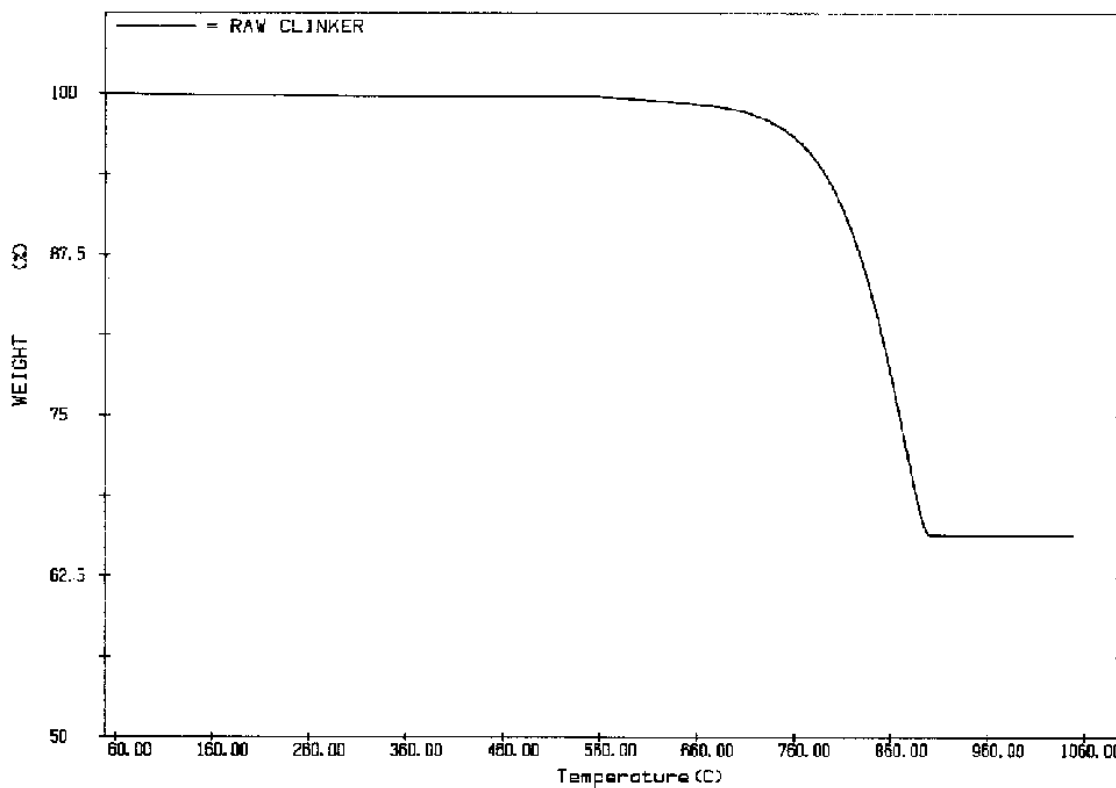


Figure 4. TGA Weight Loss Curve of Raw Kiln Feed (122-1922°F (50-1050°C), 10°C/min)

In order to study the hydraulic activity (i.e. reactivity with water) and 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. The hydration of cement produces various products one of which is calcium silicate hydrate. Calcium silicate hydrate is an amorphous material that is responsible for strength development of portland cement based products. 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 portland 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 can be used to ascertain the effectiveness of slag addition during the clinkering process.

Initially, a baseline was established using the pre-blended kiln feed. After clinkering the raw feed at various pre-selected temperatures, the product was crushed, ground, and sieved through a 325-mesh screen and x-ray diffraction patterns were collected at  $2^\circ/\text{min}$   $2\theta$  from  $5^\circ$  to  $65^\circ$ . The patterns are shown in Figures 4-7. The x-ray patterns confirm that the raw feed does not clinker well at  $2012^\circ\text{F}$  ( $1100^\circ\text{C}$ ) and  $2192^\circ\text{F}$  ( $1200^\circ\text{C}$ ). Above  $2372^\circ\text{F}$  ( $1300^\circ\text{C}$ ), the x-ray pattern showed successful clinkering of the raw feed into cement phases. The pattern at  $2642^\circ\text{F}$  ( $1450^\circ\text{C}$ ) contains peaks from calcium silicate oxide (International Committee on Diffraction Data #73-0599). The pattern from the  $2372^\circ\text{F}$  ( $1300^\circ\text{C}$ ) sample is essentially the same as that from the  $2642^\circ\text{F}$  ( $1450^\circ\text{C}$ ) sample except for the relative intensity of the peaks. As will be shown later by isothermal calorimetry, the hydration property of the  $2642^\circ\text{F}$  ( $1450^\circ\text{C}$ ) sample is typical of portland cement clinker. On the other hand, in addition to the calcium silicate phases, the  $2012^\circ\text{F}$  ( $1100^\circ\text{C}$ ) and  $2192^\circ\text{F}$  ( $1200^\circ\text{C}$ ) samples contained diffraction peaks from calcium oxides, the product from the release of carbon dioxide from the limestone in the raw material. The existence of calcium oxides proved that the limestone did not react completely with the other raw materials and consequently resulted in a poor cement clinker

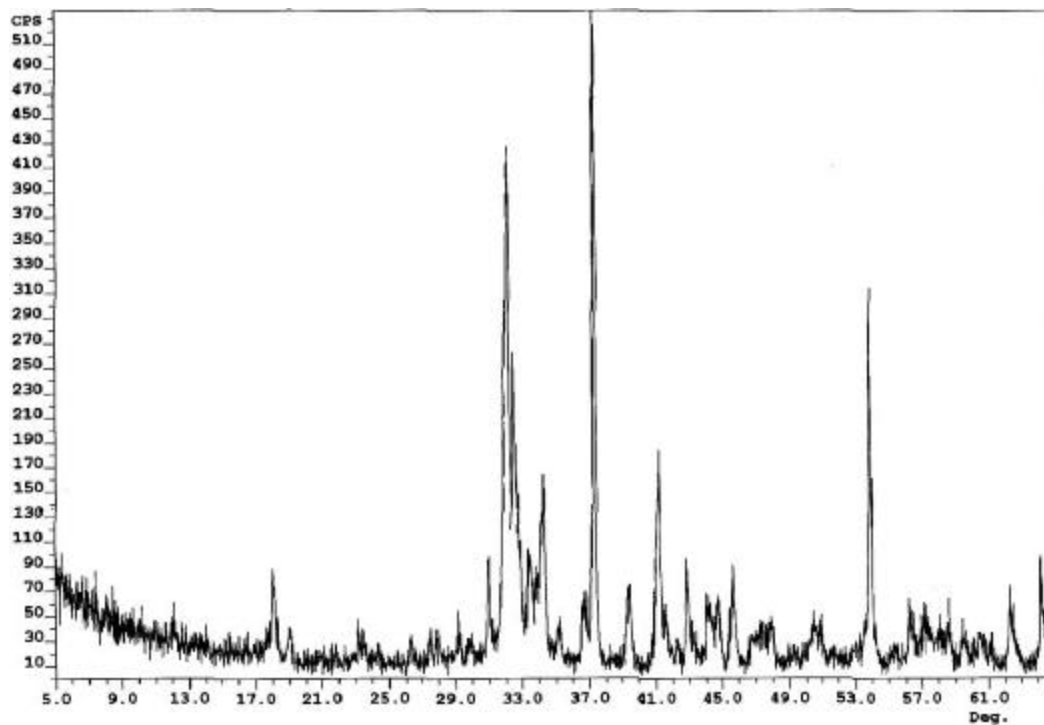


Figure 5. X-ray Diffraction Pattern of Clinker Formed at 2012°F (1100°C) (5-65°, 2°/min)

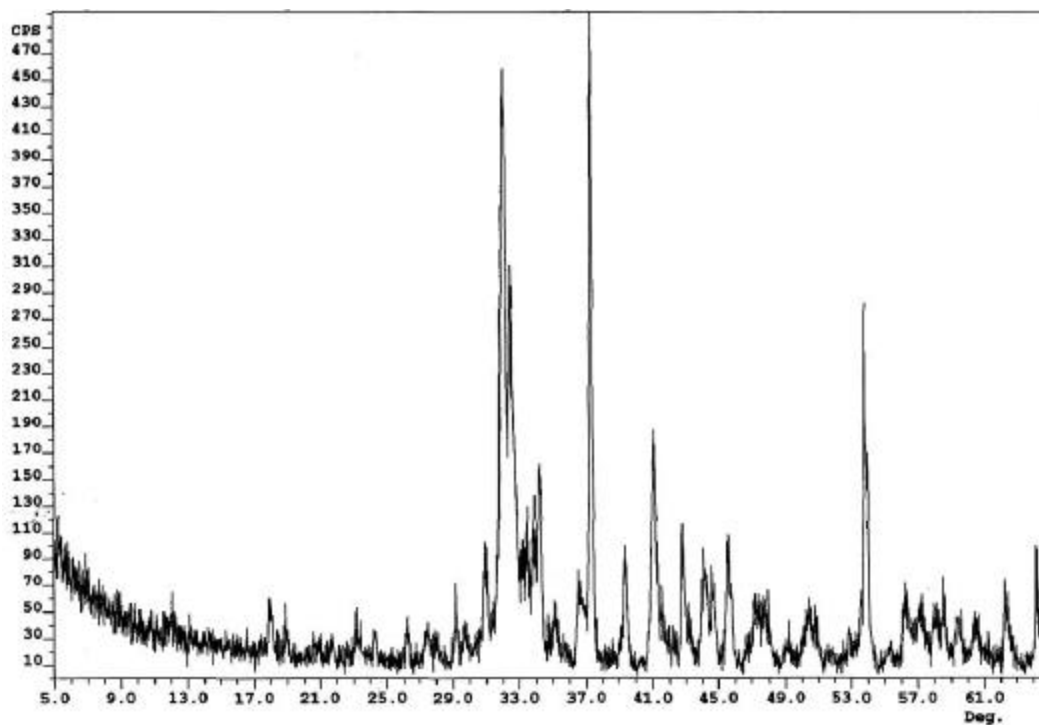
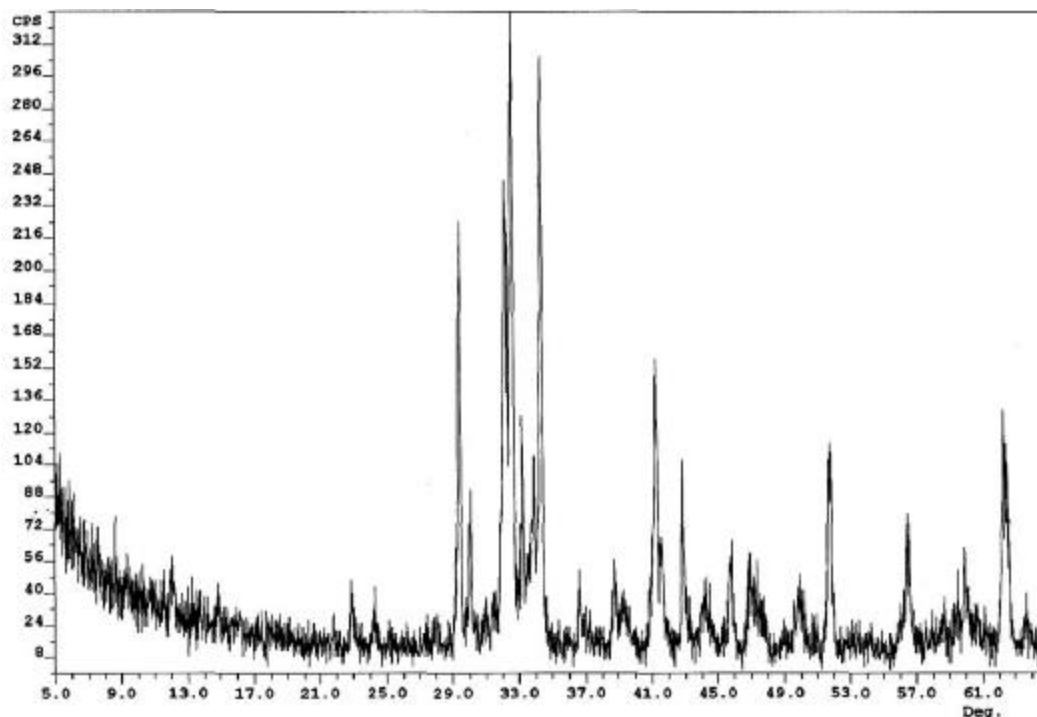
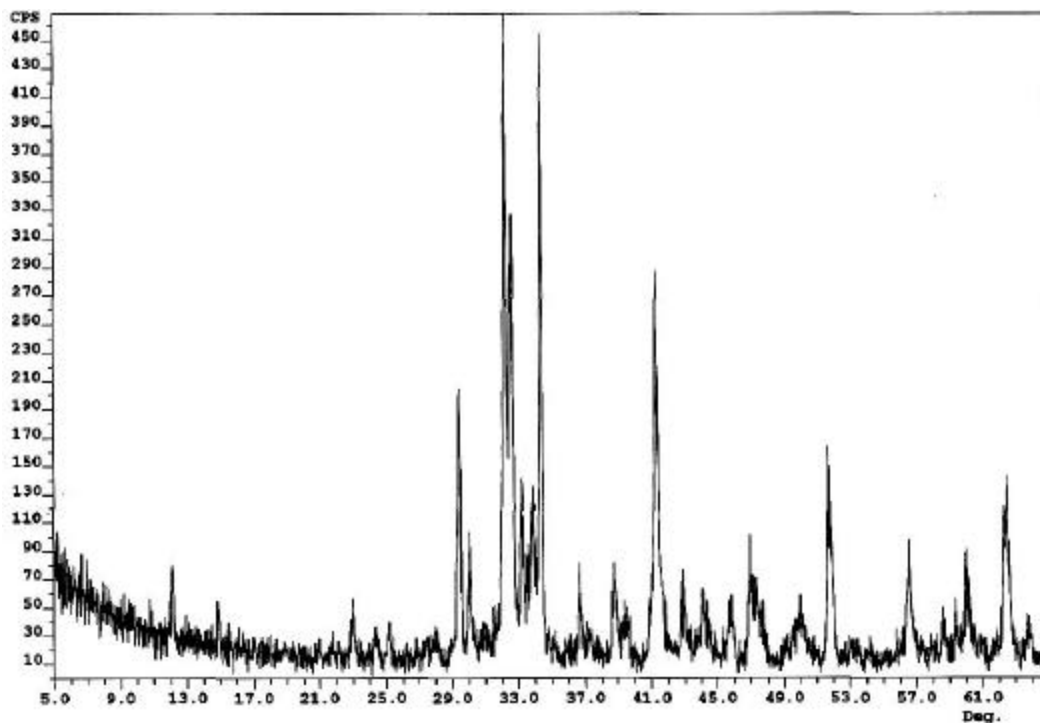


Figure 6. X-ray Diffraction Pattern of Clinker Formed at 2192°F (1200°C) (5-65°, 2°/min)





**Figure 7. X-ray Diffraction Pattern of Clinker Formed at 2372°F (1300°C) (5-65°, 2°/min)**



**Figure 8. X-ray Diffraction Pattern of Clinker Formed at 2642°F (1450°C) (5-65°, 2°/min)**

In order to study 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. The addition of 5% and 10% slag to the raw feed did not alter the x-ray diffraction pattern of the material prior to the clinkering process. 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 Figure 9 and Figure 10. However, 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 (see chemical analysis of the slag in Sec 2.7), 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%. With the replacement of 10% slag for the raw feed, 30% limestone was needed to correct for the overall chemistry. This sample, consisting of 60% raw feed, 30% limestone, and 10% slag, is designated 60-30-10.

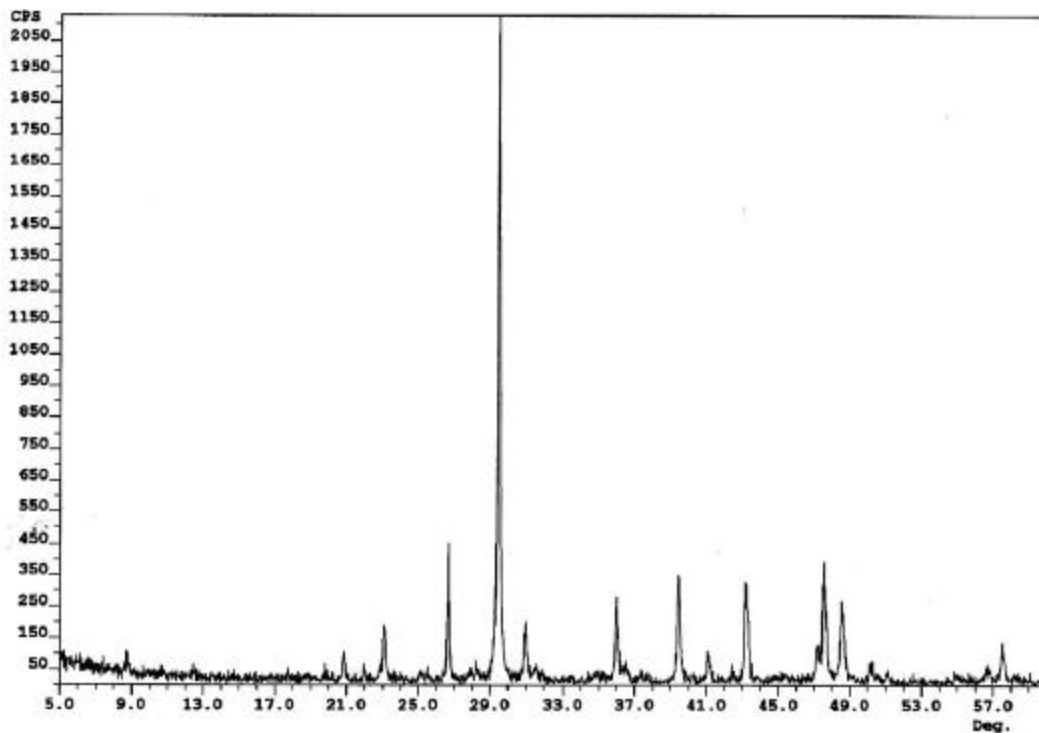
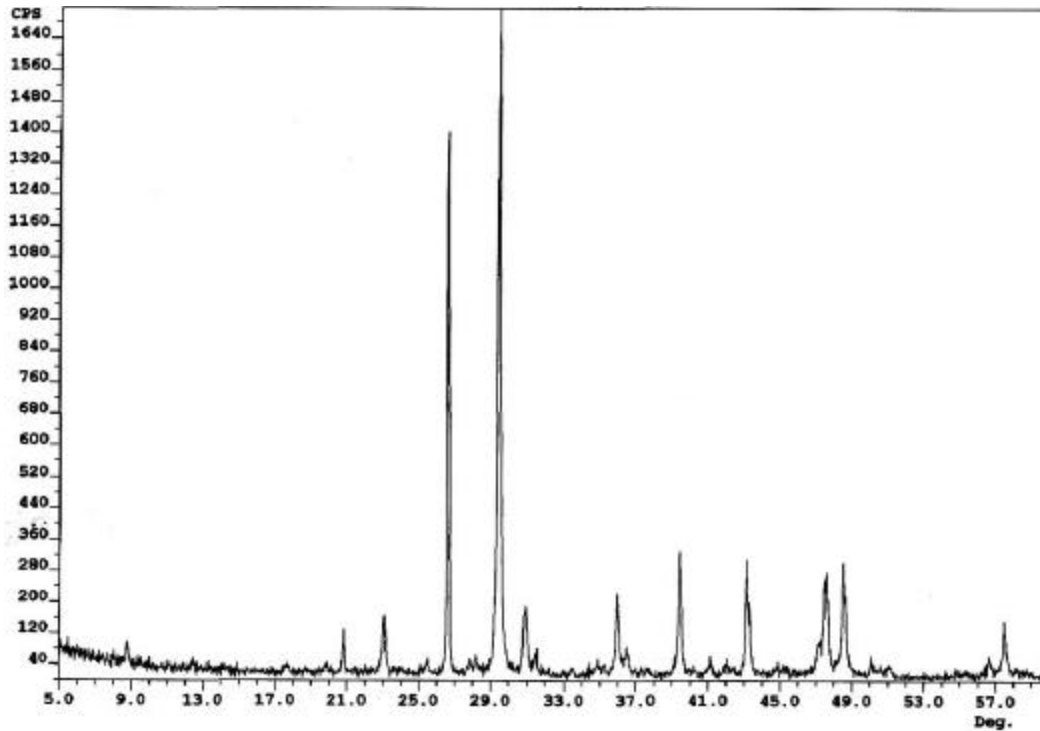


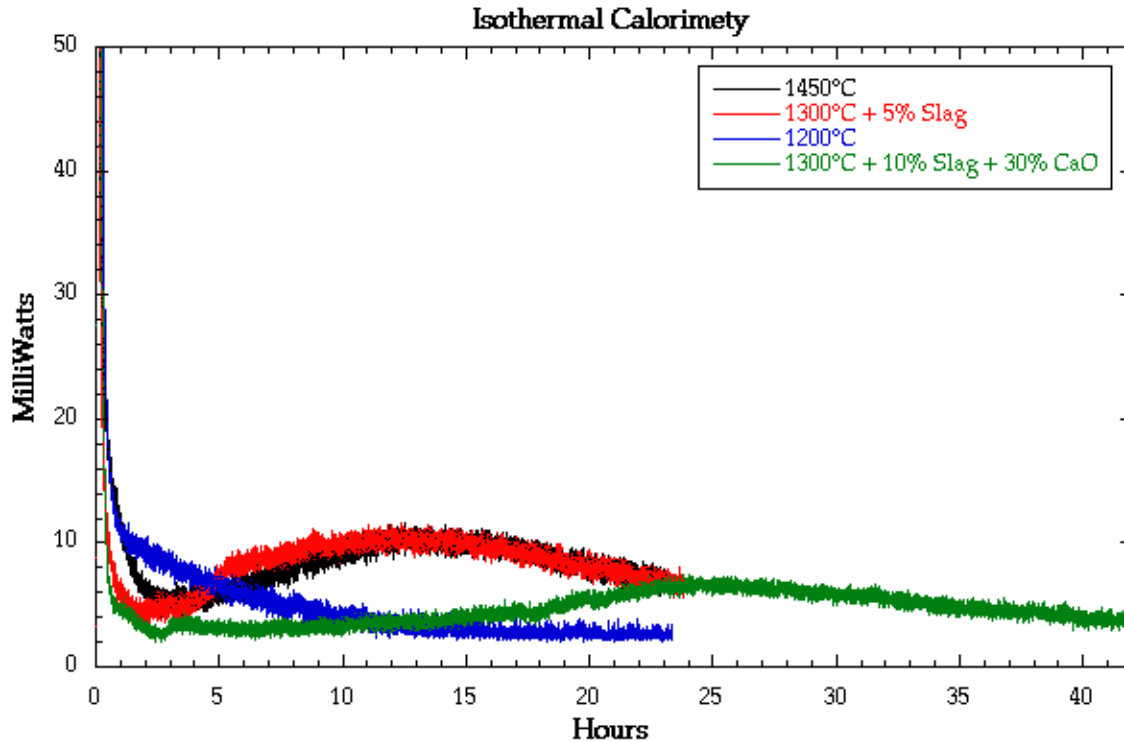
Figure 9. X-ray Diffraction Pattern of Raw Kiln Feed with 5% Ground Slag (5-65°, 2°/min)



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

In order to study the hydration characteristics of the products, isothermal calorimetry of the clinker product samples was conducted at 77°F (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. 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. As is typical for hydration experiments, all the heat evolution curves, shown in Figure 11, have an early initial wetting peak (in the first few minutes). At longer time, the sample that was clinkered at 2192°F (1200°C) showed no reaction peak whereas the 2642°F (1450°C) sample and the 2372°F (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 2372°F (1300°C) curve and the 2642°F (1450°C) curve were very similar with the 2642°F (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<sub>2</sub>S content using the Bode equation as given in ASTM C 150. 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. On the basis of these calorimetry curves, it was concluded that the hydration characteristics of the

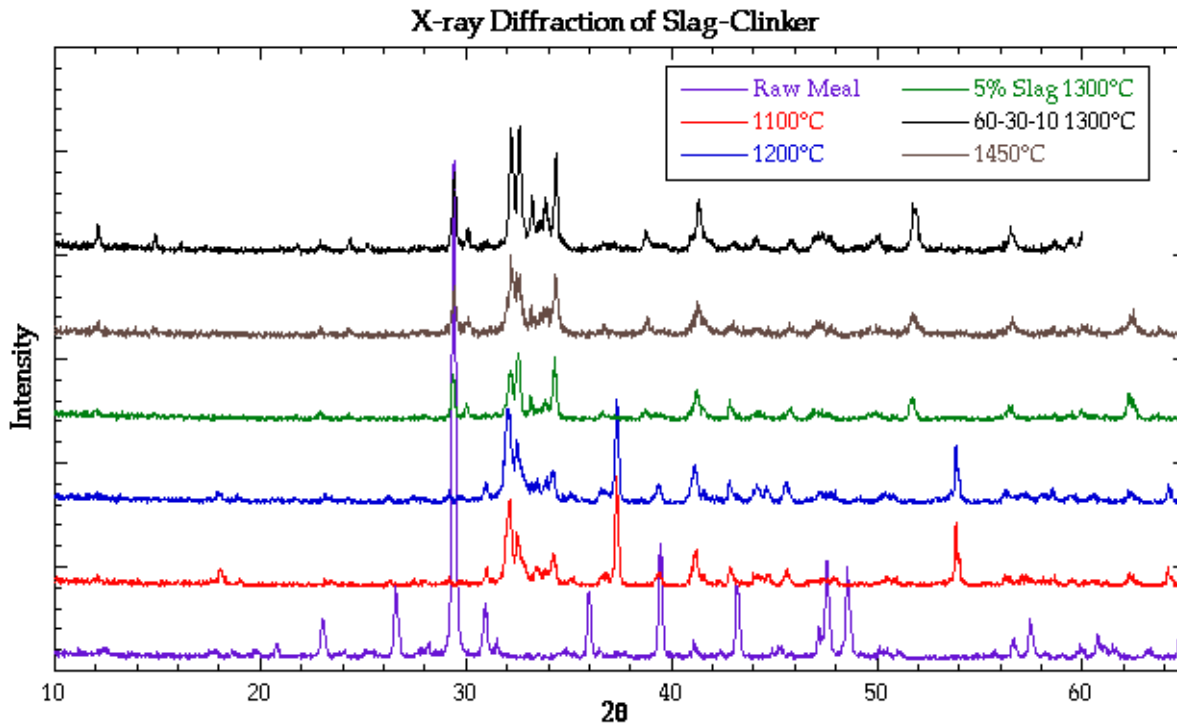
sample with 5% slag clinkered at 2372°F (1300°C) are very similar to those of the sample clinkered at 2642°F (1450°C). The delayed reaction curve from the 60-30-10 sample would represent a slower reacting cement that may have special applications where a longer working time would be beneficial.



**Figure 11. Isothermal Calorimetry of Clinkers Formed at Various Temperatures**

X-ray diffraction analysis was used as a check of the crystalline phases present in the clinker samples; the results are shown in Figure 12. The x-ray diffraction pattern of the starting material (raw meal), and clinkers formed at 2012°F (1100°C) and 2192°F (1200°C) were included for comparison. Mineralogically, the three samples clinkered at 2642°F (1450°C), 2372°F (1300°C) using 5% slag and at 2372°F (1300°C) using 10% slag and 60% limestone are the same. This suggests that using the laboratory heating curve shown above, the same mineral phases as the raw meal can be made with slag at a lower temperature. On the other hand, the samples made at 2012°F (1100°C) and 2192°F (1200°C) clearly showed diffraction peaks from calcium oxide. This shows that the clinkering reaction was not successful at these lower temperatures.

The data from these laboratory-scale tests suggested that replacing portland cement raw meal with 5% ground slag can produce a clinker at 2372°F (1300°C) with similar hydration characteristics to a clinker produced at 2642°F (1450°C) without added slag. In addition, with the addition of limestone to correct for overall chemistry, it is possible to produce a clinker at 2372°F (1300°C) with 10% slag replacing the raw meal with the added benefit of producing a specialty slower-reacting portland cement. This suggests that a significant heat energy savings of 302°F (150°C) is possible when incorporating slag into cement kiln feed, which would translate to lower heating costs or higher kiln capacity.



**Figure 12. X-ray Diffraction Analysis of Clinkers Formed at Various Temperatures**

### 5.2.1 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 2012°F (1100°C), 2192°F (1200°C), 2372°F (1300°C) and 2642°F (1450°C). Five % and 10% ground slag were added to the raw feed and clinkered at 2372°F (1300°C). Limestone was added to the sample containing 10% ground slag to adjust the bulk chemistry. Data suggested that 5% and 10% addition of ground slag can produce a clinker at 2372°F (1300°C) with similar hydration characteristics to a clinker produced at 2642°F (1450°C) without added slag. However, with high dosage of ground slag, the clinker exhibited slower reactivity which may be due to a higher C<sub>2</sub>S content. This suggests a significant heat energy saving of 302°F (150°C), which would translate to lower energy costs or higher kiln capacity. In addition to the energy savings, the slag can be used successfully as an aluminosilicate source for cement clinker.

### 5.3 Pilot Studies on Slag as Raw Material in Portland Cement Kiln Feed (Task 2.7)

Based on the promising results from the laboratory-scale testing at Pennsylvania State University, scale-up tests at the pilot scale was conducted at Fuller Co. to verify the lab test results using Fuller's commercial-scale equipment. A standard approach was used to evaluate the blended slag-raw meal mixes. Fuller's criteria for assessing the quality of cement clinker are based on measuring the percent free lime in the product after clinkering samples at 2600°F (1427°C). The free lime content of a portland cement clinker is an important property. It is well known in the cement industry that excess free lime will cause long-term durability problems in portland cement-based products. In order to validate the laboratory tests done at Pennsylvania

State University, large batches of samples were made using the same raw meal, slag and limestone. The proportioning was based on achieving a similar chemistry to the baseline raw meal, a commercially prepared cement kiln feed. The ratio of the raw meal and limestone were adjusted in order to achieve a similar burnability index while fixing the amount of slag addition. The burnability index is a calculated value based on the overall chemistry of the mixture. This index represents the relative ability of the mixture to undergo liquid-state sintering in the cement kiln. However, since the baseline raw meal is a ready-proportioned product, it was not possible to achieve an identical overall chemistry when blending it with slag and limestone. When designing and calculating the exact mix proportion for the test burns, it was found that silica sand was needed to adjust the burnability index of the mixes. Initial tests using the pre-blended raw meal showed the addition of slag increased the percent free lime in the product clinker. Tests were conducted using 3%, 5%, and 10% slag replacement for raw meal. Appropriate quantities of limestone and silica were used such that the chemistry of the mixes matches that of a conventional Type I cement. These results are shown in Table 52 to Table 55, and summarized in Table 56.

In the case of the sample containing 3% slag and no limestone, the percent free lime at 60 minutes decreased. This is not unexpected with the addition of slag alone since it is well known that the addition of aluminosilicates decreases the free lime content of cement clinker. This may be deduced from using the Bogue equations as documented in ASTM C 150. We can treat slag as an equivalent to aluminosilicate glass as evidenced by its chemical analysis and its glass-like property. In addition, we determined that replacing the raw meal with slag without also adding limestone to the 3% mix caused the overall chemistry to change. This sample was included in order to investigate the possibility of producing a high  $C_2S$  specialty cement. However, at increased levels of slag content (5% and 10%), the free lime percentage increased very significantly. This is not desirable as the excess free lime could cause durability problems in the long run and also affects the setting behavior of the cement.

**Table 52. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 0% Slag)**

| Material:                               | Raw          | Slag         | Limestone   | Sand        | Raw    | Slag | Limestone | Sand | Pellet       | Loss         | Volatile    | Volatile     |
|---|--------------|--------------|-------------|-------------|--------|------|-----------|------|--------------|--------------|-------------|--------------|
|   | Meal         | I            |             |             | Meal   | I    |           |      |              |              |             |              |
|   | Meal         | I            |             |             | 100.0% | 0.0% | 0.0%      | 0.0% | Feed         | Free         | Loss        | Clinker      |
| SiO <sub>2</sub>                        | 13.64        | 42.48        | 0.70        | 0.00        | 13.64  | 0.00 | 0.00      | 0.00 | 13.64        | 20.70        |             | 21.24        |
| Al <sub>2</sub> O <sub>3</sub> *        | 3.66         | 28.97        | 0.29        | 0.00        | 3.66   | 0.00 | 0.00      | 0.00 | 3.66         | 5.55         |             | 5.70         |
| Fe <sub>2</sub> O <sub>3</sub> **       | 2.22         | 21.21        | 0.35        | 0.00        | 2.22   | 0.00 | 0.00      | 0.00 | 2.22         | 3.37         |             | 3.46         |
| CaO                                     | 41.67        | 4.61         | 54.31       | 0.00        | 41.67  | 0.00 | 0.00      | 0.00 | 41.67        | 63.23        |             | 64.88        |
| MgO                                     | 2.59         | 1.11         | 0.70        | 0.00        | 2.59   | 0.00 | 0.00      | 0.00 | 2.59         | 3.93         |             | 4.03         |
| K <sub>2</sub> O                        | 0.81         | 1.82         | 0.09        | 0.00        | 0.81   | 0.00 | 0.00      | 0.00 | 0.81         | 1.23         | 1.11        | 0.12         |
| Na <sub>2</sub> O                       | 0.21         | 0.47         | 0.07        | 0.00        | 0.21   | 0.00 | 0.00      | 0.00 | 0.21         | 0.32         | 0.22        | 0.10         |
| SO <sub>3</sub>                         | 0.84         | 0.62         | 0.06        | 0.00        | 0.84   | 0.00 | 0.00      | 0.00 | 0.84         | 1.27         | 1.21        | 0.06         |
| Cl                                      | 0.00         | 0.00         | 0.00        | 0.00        | 0.00   | 0.00 | 0.00      | 0.00 | 0.00         | 0.00         | 0.00        | 0.00         |
| Loss(900°C)                             | 34.10        | -1.42        | 43.33       | 0.00        | 34.10  | 0.00 | 0.00      | 0.00 | 34.10        | 0.00         |             | 0.00         |
| <b>Total</b>                            | <b>99.74</b> | <b>99.87</b> | <b>99.9</b> | <b>0.00</b> |        |      |           |      | <b>99.74</b> | <b>99.61</b> | <b>2.54</b> | <b>99.59</b> |
| <b>Total Alkali as Na<sub>2</sub>O:</b> |              |              |             |             |        |      |           |      |              | <b>1.13</b>  |             | <b>0.18</b>  |

|  |          |  |
|--|----------|--|
| <b>Free Sulfur:</b>                      | -1.3E-06 | lbmol/lb feed                              |
|  | -0.00011 | lb/lb feed                                 |
|  | 0.00%    |  |
| % Sulfur Recovery:                       | 0.00%    |  |
| K <sub>2</sub> CO <sub>3</sub> Required: | 0        | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |

| Assumptions         |      |
|---------------------|------|
| Volatile Losses:    |      |
| K <sub>2</sub> O =  | 90%  |
| Na <sub>2</sub> O = | 70%  |
| SO <sub>3</sub> =   | 95%  |
| Cl =                | 100% |

| Calculated Cement Components |              |
|------------------------------|--------------|
| <b>C<sub>3</sub>S =</b>      | <b>59.42</b> |
| <b>C<sub>2</sub>S =</b>      | <b>16.26</b> |
| <b>C<sub>3</sub>A =</b>      | <b>9.26</b>  |
| <b>C<sub>4</sub>AF =</b>     | <b>10.51</b> |
| <b>LSF =</b>                 | <b>92.6</b>  |
| <b>HM =</b>                  | <b>2.1</b>   |
| <b>SR =</b>                  | <b>2.3</b>   |
| <b>AR =</b>                  | <b>1.6</b>   |
| <b>Burnability Index:</b>    | <b>3.01</b>  |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub>    \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>

**Table 53. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 3% Slag)**

| Material:                               | Raw   | Slag  | Limestone | Sand  | Raw   | Slag  | Limestone | Sand |      | Pellet | Loss        | Volatile | Volatile    |
|---|-------|-------|-----------|-------|-------|-------|-----------|------|------|--------|-------------|----------|-------------|
|   | Meal  | I     |           |       | Meal  | I     |           |      |      |        |             |          |             |
|   | Meal  | I     | Limestone | Sand  | 54.0% | 5.0%  | 36.5%     | 4.5% | 0.0% | Feed   | Free        | Loss     | Clinker     |
| SiO <sub>2</sub>                        | 13.64 | 42.48 | 0.70      | 98.04 | 7.37  | 2.12  | 0.26      | 4.41 | 0.00 | 14.16  | 21.51       |          | 21.88       |
| Al <sub>2</sub> O <sub>3</sub> *        | 3.66  | 28.97 | 0.29      | 0.06  | 1.98  | 1.45  | 0.11      | 0.00 | 0.00 | 3.53   | 5.37        |          | 5.46        |
| Fe <sub>2</sub> O <sub>3</sub> **       | 2.22  | 21.21 | 0.35      | 0.77  | 1.20  | 1.06  | 0.13      | 0.03 | 0.00 | 2.42   | 3.68        |          | 3.74        |
| CaO                                     | 41.67 | 4.61  | 54.31     | 0.13  | 22.50 | 0.23  | 19.82     | 0.01 | 0.00 | 42.56  | 64.66       |          | 65.77       |
| MgO                                     | 2.59  | 1.11  | 0.70      | 0.00  | 1.40  | 0.06  | 0.26      | 0.00 | 0.00 | 1.71   | 2.60        |          | 2.64        |
| K <sub>2</sub> O                        | 0.81  | 1.82  | 0.09      | 0.22  | 0.44  | 0.09  | 0.03      | 0.01 | 0.00 | 0.57   | 0.87        | 0.78     | 0.09        |
| Na <sub>2</sub> O                       | 0.21  | 0.47  | 0.07      | 0.08  | 0.11  | 0.02  | 0.03      | 0.00 | 0.00 | 0.17   | 0.25        | 0.18     | 0.08        |
| SO <sub>3</sub>                         | 0.84  | 0.62  | 0.06      | 0.02  | 0.45  | 0.03  | 0.02      | 0.00 | 0.00 | 0.51   | 0.77        | 0.73     | 0.04        |
| Cl                                      | 0.00  | 0.00  | 0.00      | 0.00  | 0.00  | 0.00  | 0.00      | 0.00 | 0.00 | 0.00   | 0.00        | 0.00     | 0.00        |
| Loss(900°C)                             | 34.10 | -1.42 | 43.33     | 0.31  | 18.41 | -0.07 | 15.82     | 0.01 | 0.00 | 34.17  | 0.00        |          | 0.00        |
| <b>Total</b>                            | 99.74 | 99.87 | 99.9      | 99.63 |       |       |           |      |      | 99.80  | 99.70       | 1.69     | 99.69       |
| <b>Total Alkali as Na<sub>2</sub>O:</b> |       |       |           |       |       |       |           |      |      |        | <b>0.82</b> |          | <b>0.13</b> |

|  |  |  |
|--|--|--|
| <b>Free Sulfur:</b>                      | -1.3E-05 lbmol/lb feed                       |  |
|  | -0.00104 lb/lb feed                          |  |
| % Sulfur Recovery:                       | 0.00%  |  |
| K <sub>2</sub> CO <sub>3</sub> Required: | 0 lb K <sub>2</sub> CO <sub>3</sub> /lb feed |  |

|                         |  |
|-------------------------|--|
| <b>Assumptions</b>      | <b>Calculated Cement Components</b>                      |
| Volitile Losses:        | Type I   |
| K <sub>2</sub> O = 90%  | <b>C<sub>3</sub>S = 59.37</b> <b>LSF = 91.9</b> (87-97)  |
| Na <sub>2</sub> O = 70% | <b>C<sub>2</sub>S = 18.11</b> <b>HM = 2.1</b> (1.7-2.3)  |
| SO <sub>3</sub> = 95%   | <b>C<sub>3</sub>A = 8.14</b> <b>SR = 2.4</b> (1.9-3.2)   |
| Cl = 100%               | <b>C<sub>4</sub>AF = 11.38</b> <b>AR = 1.5</b> (1.5-2.5) |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub>    \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>



**Table 54. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 5% Slag)**

| Material:                               | Raw   | Slag  | Limestone | Sand  | Raw   | Slag  | Limestone | Sand | Pellet | Loss  | Volatile    | Volatile |             |
|---|-------|-------|-----------|-------|-------|-------|-----------|------|--------|-------|-------------|----------|-------------|
|   | Meal  | I     |           |       | Meal  | I     |           |      |        |       |             |          | Less        |
|   |       |       |           |       | 54.0% | 5.0%  | 36.5%     | 4.5% | 0.0%   | Feed  | Free        | Loss     | Clinker     |
| SiO <sub>2</sub>                        | 13.64 | 42.48 | 0.70      | 98.04 | 7.37  | 2.12  | 0.26      | 4.41 | 0.00   | 14.16 | 21.51       |          | 21.88       |
| Al <sub>2</sub> O <sub>3</sub> *        | 3.66  | 28.97 | 0.29      | 0.06  | 1.98  | 1.45  | 0.11      | 0.00 | 0.00   | 3.53  | 5.37        |          | 5.46        |
| Fe <sub>2</sub> O <sub>3</sub> **       | 2.22  | 21.21 | 0.35      | 0.77  | 1.20  | 1.06  | 0.13      | 0.03 | 0.00   | 2.42  | 3.68        |          | 3.74        |
| CaO                                     | 41.67 | 4.61  | 54.31     | 0.13  | 22.50 | 0.23  | 19.82     | 0.01 | 0.00   | 42.56 | 64.66       |          | 65.77       |
| MgO                                     | 2.59  | 1.11  | 0.70      | 0.00  | 1.40  | 0.06  | 0.26      | 0.00 | 0.00   | 1.71  | 2.60        |          | 2.64        |
| K <sub>2</sub> O                        | 0.81  | 1.82  | 0.09      | 0.22  | 0.44  | 0.09  | 0.03      | 0.01 | 0.00   | 0.57  | 0.87        | 0.78     | 0.09        |
| Na <sub>2</sub> O                       | 0.21  | 0.47  | 0.07      | 0.08  | 0.11  | 0.02  | 0.03      | 0.00 | 0.00   | 0.17  | 0.25        | 0.18     | 0.08        |
| SO <sub>3</sub>                         | 0.84  | 0.62  | 0.06      | 0.02  | 0.45  | 0.03  | 0.02      | 0.00 | 0.00   | 0.51  | 0.77        | 0.73     | 0.04        |
| Cl                                      | 0.00  | 0.00  | 0.00      | 0.00  | 0.00  | 0.00  | 0.00      | 0.00 | 0.00   | 0.00  | 0.00        | 0.00     | 0.00        |
| Loss(900°C)                             | 34.10 | -1.42 | 43.33     | 0.31  | 18.41 | -0.07 | 15.82     | 0.01 | 0.00   | 34.17 | 0.00        |          | 0.00        |
| <b>Total</b>                            | 99.74 | 99.87 | 99.9      | 99.63 |       |       |           |      |        | 99.80 | 99.70       | 1.69     | 99.69       |
| <b>Total Alkali as Na<sub>2</sub>O:</b> |       |       |           |       |       |       |           |      |        |       | <b>0.82</b> |          | <b>0.13</b> |

|  |                    |  |
|--|--------------------|--|
| <b>Free Sulfur:</b>                      | -1.3E-05           | lbmol/lb feed                              |
|  | -0.00104           | lb/lb feed                                 |
|  | % Sulfur Recovery: | 0.00%                                      |
| K <sub>2</sub> CO <sub>3</sub> Required: | 0                  | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |

| Assumptions         |      |
|---------------------|------|
| Volatile Losses:    |      |
| K <sub>2</sub> O =  | 90%  |
| Na <sub>2</sub> O = | 70%  |
| SO <sub>3</sub> =   | 95%  |
| Cl =                | 100% |

| Calculated Cement Components   |                   | Type I    |
|--------------------------------|-------------------|-----------|
| <b>C<sub>3</sub>S = 59.37</b>  | <b>LSF = 91.9</b> | (87-97)   |
| <b>C<sub>2</sub>S = 18.11</b>  | <b>HM = 2.1</b>   | (1.7-2.3) |
| <b>C<sub>3</sub>A = 8.14</b>   | <b>SR = 2.4</b>   | (1.9-3.2) |
| <b>C<sub>4</sub>AF = 11.38</b> | <b>AR = 1.5</b>   | (1.5-2.5) |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub> \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>

**Table 55. Calculated Cement Clinker Chemistry (Pre-Blended Raw Meal, 10% Slag)**

| Material:                                    | Raw          | Slag         | Limestone                                  | Sand         | Raw                      | Slag  | Limestone | Sand | 0.0% | Pellet       | Loss                                | Volatile          | Volatile         |      |
|--|--------------|--------------|--|--------------|--------------------------|-------|-----------|------|------|--------------|-------------------------------------|-------------------|------------------|------|
|  | Meal         | I            |  |              | Meal                     | I     |           |      |      |              |                                     |                   |                  | Less |
|  |              |              |  |              | 10.5%                    | 10.0% | 71.0%     | 8.5% | 0.0% | Feed         | Free                                | Loss              | Clinker          |      |
| SiO <sub>2</sub>                             | 13.64        | 42.48        | 0.70                                       | 98.04        | 1.43                     | 4.25  | 0.50      | 8.33 | 0.00 | 14.51        | 22.06                               |                   | 22.26            |      |
| Al <sub>2</sub> O <sub>3</sub> *             | 3.66         | 28.97        | 0.29                                       | 0.06         | 0.38                     | 2.90  | 0.21      | 0.01 | 0.00 | 3.49         | 5.31                                |                   | 5.36             |      |
| Fe <sub>2</sub> O <sub>3</sub> **            | 2.22         | 21.21        | 0.35                                       | 0.77         | 0.23                     | 2.12  | 0.25      | 0.07 | 0.00 | 2.67         | 4.06                                |                   | 4.09             |      |
| CaO  | 41.67        | 4.61         | 54.31                                      | 0.13         | 4.38                     | 0.46  | 38.56     | 0.01 | 0.00 | 43.41        | 66.00                               |                   | 66.59            |      |
| MgO  | 2.59         | 1.11         | 0.70                                       | 0.00         | 0.27                     | 0.11  | 0.50      | 0.00 | 0.00 | 0.88         | 1.34                                |                   | 1.35             |      |
| K <sub>2</sub> O                             | 0.81         | 1.82         | 0.09                                       | 0.22         | 0.09                     | 0.18  | 0.06      | 0.02 | 0.00 | 0.35         | 0.53                                | 0.48              | 0.05             |      |
| Na <sub>2</sub> O                            | 0.21         | 0.47         | 0.07                                       | 0.08         | 0.02                     | 0.05  | 0.05      | 0.01 | 0.00 | 0.13         | 0.19                                | 0.13              | 0.06             |      |
| SO <sub>3</sub>                              | 0.84         | 0.62         | 0.06                                       | 0.02         | 0.09                     | 0.06  | 0.04      | 0.00 | 0.00 | 0.19         | 0.30                                | 0.28              | 0.01             |      |
| Cl   | 0.00         | 0.00         | 0.00                                       | 0.00         | 0.00                     | 0.00  | 0.00      | 0.00 | 0.00 | 0.00         | 0.00                                | 0.00              | 0.00             |      |
| Loss(900°C)                                  | 34.10        | -1.42        | 43.33                                      | 0.31         | 3.58                     | -0.14 | 30.76     | 0.03 | 0.00 | 34.23        | 0.00                                |                   | 0.00             |      |
| <b>Total</b>                                 | <b>99.74</b> | <b>99.87</b> | <b>99.9</b>                                | <b>99.63</b> |                          |       |           |      |      | <b>99.86</b> | <b>99.78</b>                        | <b>0.89</b>       | <b>99.78</b>     |      |
| <b>Total Alkali as Na<sub>2</sub>O:</b>      |              |              |  |              |                          |       |           |      |      |              | <b>0.54</b>                         |                   | <b>0.09</b>      |      |
| <b>Free Sulfur:</b>                          |              | -2E-05       | lbmol/lb feed                              |              |                          |       |           |      |      |              |                                     |                   |                  |      |
|  |              | -0.002       | lb/lb feed                                 |              |                          |       |           |      |      |              |                                     |                   |                  |      |
| <b>% Sulfur Recovery:</b>                    |              | 0.00%        |  |              | <b>Assumptions</b>       |       |           |      |      |              |                                     |                   |                  |      |
| <b>K<sub>2</sub>CO<sub>3</sub> Required:</b> |              | 0            | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |              | <b>Volatiles Losses:</b> |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              | K <sub>2</sub> O = 90%   |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              | Na <sub>2</sub> O = 70%  |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              | SO <sub>3</sub> = 95%    |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              | Cl = 100%                |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              |                          |       |           |      |      |              |                                     |                   |                  |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>Calculated Cement Components</b> |                   | <b>Type I</b>    |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>C<sub>3</sub>S = 59.99</b>       | <b>LSF = 91.7</b> | <b>(87-97)</b>   |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>C<sub>2</sub>S = 18.75</b>       | <b>HM = 2.1</b>   | <b>(1.7-2.3)</b> |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>C<sub>3</sub>A = 7.28</b>        | <b>SR = 2.4</b>   | <b>(1.9-3.2)</b> |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>C<sub>4</sub>AF = 12.44</b>      | <b>AR = 1.3</b>   | <b>(1.5-2.5)</b> |      |
|  |              |              |  |              |                          |       |           |      |      |              | <b>Burnability Index:</b>           |                   | <b>3.04</b>      |      |

**Table 56. Summary of Free Lime Tests Using Pre-Blended Cement Kiln Raw Meal**

| Test Temp = 2600°F (1427°C) | Raw Meal | 3% Slag | 5% Slag + Limestone | 10% Slag + Limestone |
|-----------------------------|----------|---------|---------------------|----------------------|
| % Free Lime at 60 minutes   | 0.35     | 0.15    | 0.54                | 0.85                 |
| Burnability Index           | 3.01     | 1.37    | 3.04                | 3.04                 |

In order to further quantify the effects of using slag as a partial replacement for raw meal in making portland cement clinker, Fuller Co. acquired a very hard-burning raw meal, consisting of limestone, sandstone, shale, and iron, this time in component form instead of as a pre-blended raw meal. A hard-burning mix is a cement mix for which it is very difficult to produce the liquid phase in the kiln. Hard-burning mixes usually require both higher temperatures and longer residence times during the clinking process.

We hypothesized that using a hard-burning mix would allow us to more easily detect whether the slag would perform as a fluxing agent, increasing the liquid phase in the kiln and therefore making the mixture easier to burn. Combining the various components in the appropriate ratios also made it much easier to adjust the bulk chemistry of the raw material when slag was used. Free lime tests were conducted at 0%, 3%, 5% and 8.5% levels of slag replacement (see Tables 49-52). The baseline mix (with no slag) was proportioned according to the manufacturer's specifications conforming to specifications of a Type I portland cement. Using these raw materials, we determined that the maximum amount of slag that can be added while retaining the same baseline initial chemistry was 8.5%.

**Table 57. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 0% Slag)**

| Material:                         | Lime-stone | Sand-stone | Shale | Iron   | Slag I | Lime-stone | Sand-stone | Shale | Iron  | Slag I | Pellet Feed | Loss Free                               | Volatile Loss | Volatile Less Clinker |  |             |
|-----------------------------------|------------|------------|-------|--------|--------|------------|------------|-------|-------|--------|-------------|---|---------------|-----------------------|--|-------------|
|                                   |            |            |       |        |        | 82.7%      | 6.3%       | 10.0% | 1.0%  | 0.0%   |             |   |               |                       |  |             |
| SiO <sub>2</sub>                  | 3.49       | 91.57      | 56.18 | 2.31   | 42.48  | 2.89       | 5.77       | 5.62  | 0.02  | 0.00   | 14.30       | 22.03                                   |               | 22.26                 |  |             |
| Al <sub>2</sub> O <sub>3</sub> *  | 1.25       | 3.15       | 24.12 | 0.56   | 28.97  | 1.03       | 0.20       | 2.41  | 0.01  | 0.00   | 3.65        | 5.63                                    |               | 5.68                  |  |             |
| Fe <sub>2</sub> O <sub>3</sub> ** | 0.53       | 0.84       | 7.34  | 100.29 | 21.21  | 0.44       | 0.05       | 0.73  | 1.00  | 0.00   | 2.23        | 3.43                                    |               | 3.47                  |  |             |
| CaO                               | 51.54      | 1.48       | 1.63  | 0.63   | 4.61   | 42.62      | 0.09       | 0.16  | 0.01  | 0.00   | 42.89       | 66.10                                   |               | 66.77                 |  |             |
| MgO                               | 0.93       | 0.02       | 0.71  | 0.00   | 1.11   | 0.77       | 0.00       | 0.07  | 0.00  | 0.00   | 0.84        | 1.30                                    |               | 1.31                  |  |             |
| K <sub>2</sub> O                  | 0.41       | 0.42       | 0.98  | 0.03   | 1.82   | 0.34       | 0.03       | 0.10  | 0.00  | 0.00   | 0.46        | 0.71                                    | 0.64          | 0.07                  |  |             |
| Na <sub>2</sub> O                 | 0.09       | 0.09       | 0.74  | 0.18   | 0.47   | 0.07       | 0.01       | 0.07  | 0.00  | 0.00   | 0.16        | 0.24                                    | 0.17          | 0.07                  |  |             |
| SO <sub>3</sub>                   | 0.04       | 0.05       | 0.08  | 0.07   | 0.62   | 0.03       | 0.00       | 0.01  | 0.00  | 0.00   | 0.04        | 0.07                                    | 0.07          | 0.00                  |  |             |
| Cl                                | 0.01       | 0.10       | 0.62  | 0.62   | 0.00   | 0.01       | 0.01       | 0.06  | 0.01  | 0.00   | 0.08        | 0.13                                    | 0.13          | 0.00                  |  |             |
| Loss(900°C)                       | 41.42      | 2.23       | 7.80  | -5.50  | -1.42  | 34.25      | 0.14       | 0.78  | -0.06 | 0.00   | 35.12       | 0.00                                    |               | 0.00                  |  |             |
| <b>Total</b>                      | 99.71      | 99.95      | 100.2 | 99.19  | 99.87  |            |            |       |       |        | 99.77       | 99.64                                   | 1.00          | 99.64                 |  |             |
|                                   |            |            |       |        |        |            |            |       |       |        |             | <b>Total Alkali as Na<sub>2</sub>O:</b> |               | <b>0.71</b>           |  | <b>0.12</b> |

|  |          |  |
|--|----------|--|
| Free Sulfur:                             | -4.5E-05 | lbmol/lb feed                              |
|  | -0.0036  | lb/lb feed                                 |
|  | 0.00%    |  |
| % Sulfur Recovery:                       | 0.00%    |  |
| K <sub>2</sub> CO <sub>3</sub> Required: | 0        | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |

| Assumptions             |
|-------------------------|
| Volitile Losses:        |
| K <sub>2</sub> O = 90%  |
| Na <sub>2</sub> O = 70% |
| SO <sub>3</sub> = 95%   |
| Cl = 100%               |

| Calculated Cement Components   |                    | Type I    |
|--------------------------------|--------------------|-----------|
| <b>C<sub>3</sub>S = 59.45</b>  | <b>LSF = 91.58</b> | (87-97)   |
| <b>C<sub>2</sub>S = 19.16</b>  | <b>HM = 2.13</b>   | (1.7-2.3) |
| <b>C<sub>3</sub>A = 9.20</b>   | <b>SR = 2.43</b>   | (1.9-3.2) |
| <b>C<sub>4</sub>AF = 10.55</b> | <b>AR = 1.64</b>   | (1.5-2.5) |
| <b>Burnability Index:</b>      |                    | 3.01      |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub>    \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>



**Table 59. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 5% Slag)**

| Material:                         | Lime-stone | Sand-stone | Shale | Iron   | Slag I | Lime-stone | Sand-stone | Shale | Iron  | Slag I | Pellet Feed | Loss Free                               | Volatile Loss | Volatile Less Clinker |
|-----------------------------------|------------|------------|-------|--------|--------|------------|------------|-------|-------|--------|-------------|---|---------------|-----------------------|
|                                   |            |            |       |        |        | 82.9%      | 7.7%       | 4.0%  | 0.4%  | 5.0%   |             |   |               |                       |
| SiO <sub>2</sub>                  | 3.49       | 91.57      | 56.18 | 2.31   | 42.48  | 2.89       | 7.05       | 2.25  | 0.01  | 2.12   | 14.32       | 21.95                                   |               | 22.17                 |
| Al <sub>2</sub> O <sub>3</sub> *  | 1.25       | 3.15       | 24.12 | 0.56   | 28.97  | 1.04       | 0.24       | 0.96  | 0.00  | 1.45   | 3.69        | 5.66                                    |               | 5.72                  |
| Fe <sub>2</sub> O <sub>3</sub> ** | 0.53       | 0.84       | 7.34  | 100.29 | 21.21  | 0.44       | 0.06       | 0.29  | 0.40  | 1.06   | 2.26        | 3.46                                    |               | 3.50                  |
| CaO                               | 51.54      | 1.48       | 1.63  | 0.63   | 4.61   | 42.73      | 0.11       | 0.07  | 0.00  | 0.23   | 43.14       | 66.09                                   |               | 66.76                 |
| MgO                               | 0.93       | 0.02       | 0.71  | 0.00   | 1.11   | 0.77       | 0.00       | 0.03  | 0.00  | 0.06   | 0.86        | 1.31                                    |               | 1.33                  |
| K <sub>2</sub> O                  | 0.41       | 0.42       | 0.98  | 0.03   | 1.82   | 0.34       | 0.03       | 0.04  | 0.00  | 0.09   | 0.50        | 0.77                                    | 0.69          | 0.08                  |
| Na <sub>2</sub> O                 | 0.09       | 0.09       | 0.74  | 0.18   | 0.47   | 0.07       | 0.01       | 0.03  | 0.00  | 0.02   | 0.14        | 0.21                                    | 0.15          | 0.06                  |
| SO <sub>3</sub>                   | 0.04       | 0.05       | 0.08  | 0.07   | 0.62   | 0.03       | 0.00       | 0.00  | 0.00  | 0.03   | 0.07        | 0.11                                    | 0.10          | 0.01                  |
| Cl                                | 0.01       | 0.10       | 0.62  | 0.62   | 0.00   | 0.01       | 0.01       | 0.02  | 0.00  | 0.00   | 0.04        | 0.07                                    | 0.07          | 0.00                  |
| Loss(900°C)                       | 41.42      | 2.23       | 7.80  | -5.50  | -1.42  | 34.34      | 0.17       | 0.31  | -0.02 | -0.07  | 34.73       | 0.00                                    |               | 0.00                  |
| <b>Total</b>                      | 99.71      | 99.95      | 100.2 | 99.19  | 99.87  |            |            |       |       |        | 99.75       | 99.62                                   | 1.01          | 99.62                 |
|                                   |            |            |       |        |        |            |            |       |       |        |             | <b>Total Alkali as Na<sub>2</sub>O:</b> | <b>0.71</b>   | <b>0.11</b>           |

|  |            |  |
|--|------------|--|
| <b>Free Sulfur:</b>                      | -4.871E-05 | lbmol/lb feed                              |
|  | -0.003897  | lb/lb feed                                 |
| % Sulfur Recovery:                       | 0.00%      |  |
| K <sub>2</sub> CO <sub>3</sub> Required: | -0         | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |

| Assumptions         |      |
|---------------------|------|
| Volitile Losses:    |      |
| K <sub>2</sub> O =  | 90%  |
| Na <sub>2</sub> O = | 70%  |
| SO <sub>3</sub> =   | 95%  |
| Cl =                | 100% |

| Calculated Cement Components   |                   |  | Type I    |
|--------------------------------|-------------------|--|-----------|
| <b>C<sub>3</sub>S = 59.82</b>  | <b>LSF = 91.8</b> |  | (87-97)   |
| <b>C<sub>2</sub>S = 18.63</b>  | <b>HM = 2.1</b>   |  | (1.7-2.3) |
| <b>C<sub>3</sub>A = 9.24</b>   | <b>SR = 2.4</b>   |  | (1.9-3.2) |
| <b>C<sub>4</sub>AF = 10.63</b> | <b>AR = 1.6</b>   |  | (1.5-2.5) |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub>    \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>

**Table 60. Calculated Cement Clinker Chemistry (Component Raw Ingredients, 8.5% Slag)**

| Material:                               | Lime-stone | Sand-stone | Shale | Iron   | Slag I | Lime-stone | Sand-stone | Shale | Iron | Slag I | Pellet Feed | Loss Free   | Volatile Loss | Volatile Less Clinker |
|---|------------|------------|-------|--------|--------|------------|------------|-------|------|--------|-------------|-------------|---------------|-----------------------|
|   |            |            |       |        |        | 83.0%      | 8.5%       | 0.0%  | 0.0% | 8.5%   |             |             |               |                       |
| SiO <sub>2</sub>                        | 3.49       | 91.57      | 56.18 | 2.31   | 42.48  | 2.90       | 7.78       | 0.00  | 0.00 | 3.61   | 14.29       | 21.80       |               | 22.02                 |
| Al <sub>2</sub> O <sub>3</sub> *        | 1.25       | 3.15       | 24.12 | 0.56   | 28.97  | 1.04       | 0.27       | 0.00  | 0.00 | 2.46   | 3.77        | 5.75        |               | 5.81                  |
| Fe <sub>2</sub> O <sub>3</sub> **       | 0.53       | 0.84       | 7.34  | 100.29 | 21.21  | 0.44       | 0.07       | 0.00  | 0.00 | 1.80   | 2.31        | 3.53        |               | 3.57                  |
| CaO                                     | 51.54      | 1.48       | 1.63  | 0.63   | 4.61   | 42.78      | 0.13       | 0.00  | 0.00 | 0.39   | 43.30       | 66.05       |               | 66.73                 |
| MgO                                     | 0.93       | 0.02       | 0.71  | 0.00   | 1.11   | 0.77       | 0.00       | 0.00  | 0.00 | 0.09   | 0.87        | 1.32        |               | 1.34                  |
| K <sub>2</sub> O                        | 0.41       | 0.42       | 0.98  | 0.03   | 1.82   | 0.34       | 0.04       | 0.00  | 0.00 | 0.15   | 0.53        | 0.81        | 0.73          | 0.08                  |
| Na <sub>2</sub> O                       | 0.09       | 0.09       | 0.74  | 0.18   | 0.47   | 0.07       | 0.01       | 0.00  | 0.00 | 0.04   | 0.12        | 0.19        | 0.13          | 0.06                  |
| SO <sub>3</sub>                         | 0.04       | 0.05       | 0.08  | 0.07   | 0.62   | 0.03       | 0.00       | 0.00  | 0.00 | 0.05   | 0.09        | 0.14        | 0.13          | 0.01                  |
| Cl                                      | 0.01       | 0.10       | 0.62  | 0.62   | 0.00   | 0.01       | 0.01       | 0.00  | 0.00 | 0.00   | 0.02        | 0.03        | 0.03          | 0.00                  |
| Loss(900°C)                             | 41.42      | 2.23       | 7.80  | -5.50  | -1.42  | 34.38      | 0.19       | 0.00  | 0.00 | -0.12  | 34.45       | 0.00        |               | 0.00                  |
| <b>Total</b>                            | 99.71      | 99.95      | 100.2 | 99.19  | 99.87  |            |            |       |      |        | 99.74       | 99.61       | 1.02          | 99.60                 |
| <b>Total Alkali as Na<sub>2</sub>O:</b> |            |            |       |        |        |            |            |       |      |        |             | <b>0.72</b> |               | <b>0.11</b>           |

|  |           |  |
|--|-----------|--|
| <b>Free Sulfur:</b>                      | -5.14E-05 | lbmol/lb feed                              |
|  | -0.004115 | lb/lb feed                                 |
| % Sulfur Recovery:                       | 0.00%     |  |
| K <sub>2</sub> CO <sub>3</sub> Required: | -0        | lb K <sub>2</sub> CO <sub>3</sub> /lb feed |

| Assumptions         |      |
|---------------------|------|
| Volatile Losses:    |      |
| K <sub>2</sub> O =  | 90%  |
| Na <sub>2</sub> O = | 70%  |
| SO <sub>3</sub> =   | 95%  |
| Cl =                | 100% |

| Calculated Cement Components   |                   | Type I    |
|--------------------------------|-------------------|-----------|
| <b>C<sub>3</sub>S = 60.07</b>  | <b>LSF = 92.0</b> | (87-97)   |
| <b>C<sub>2</sub>S = 18.03</b>  | <b>HM = 2.1</b>   | (1.7-2.3) |
| <b>C<sub>3</sub>A = 9.36</b>   | <b>SR = 2.3</b>   | (1.9-3.2) |
| <b>C<sub>4</sub>AF = 10.84</b> | <b>AR = 1.6</b>   | (1.5-2.5) |

\* Incl. P<sub>2</sub>O<sub>5</sub> & TiO<sub>2</sub>    \*\* Incl. Mn<sub>2</sub>O<sub>3</sub>

The free lime test results are summarized in Table 61. The hard-burning mix we used initially had a free lime content of 2.86% in 60 minutes of burn time at 2600°F (1427°C.) With the addition of 5% slag, the free lime content increased to 4.92%. This level, which was significantly higher than that of the initial baseline mix, was deemed unsatisfactory and further tests were abandoned.

**Table 61. Summary of Free Lime Test Using Raw Ingredients**

| Temp = 2600°F (1427°C)    | Initial Hard Burn Mix | 3% Slag | 5% Slag | 8.5% Slag |
|---------------------------|-----------------------|---------|---------|-----------|
| Burnability Index         | 3.01                  | 1.37    | 3.01    | 2.97      |
| % Free Lime at 60 minutes | 2.86                  | -       | 4.92    | -         |

We then contacted a number of cement manufacturers and they all said that they would never use such a hard-burning mix since the silica modulus is too high. As an added test, the silica modulus was therefore adjusted to a more common value once again using raw ingredients. The resultant percent free lime from tests burns are shown in Table 62.

**Table 62. Summary of Free Lime Test Using Raw Ingredients with Lower Silica Modulus**

| Temp = 2600°F (1427°C)    | Initial Hard Burn Mix with lower silica modulus | 3% Slag | 5% Slag | 8.5% Slag |
|---------------------------|---|---------|---------|-----------|
| % Free Lime at 60 minutes | 0.19  | 0.17    | 0.18    | 0.60      |

Using a blend with a more conventional silica modulus, the initial baseline test resulted in a free lime of 0.19% after 1 hour. With 3% or 5% slag replacement, although there was a very slight decrease of free lime to 0.17% and 0.18% respectively, the advantage is minimal. Finally, the mixture containing 8.5% slag had a free lime content of 0.60% after 1 hour, which is an unacceptably high value. We therefore concluded that, contrary to the initial laboratory-scale tests, slag did not perform well as a fluxing agent and consequently did not appreciably decrease the percent free lime of the cement clinker. Although using a small quantity of slag as a feed material is beneficial from the viewpoint of waste utilization, it neither proved to assist in producing a better quality cement clinker nor significantly reduced costs.

#### 5.4 Pozzolonic Activity of Prepared Gasifier Slag

The test work under this subtask focused on utilizing the property of gasification slag that is similar to that of an aluminosilicate glass. It is well known that certain types of fly ash can be used as a pozzolan in cement systems. Pozzolans have the unique property that, when combined with lime in a relatively high pH environment, they tend to form calcium silicate hydrate at a later time. As noted before, calcium silicate hydrate is the major binding phase of portland cement-based systems and is responsible for the strength development of these systems. With the addition of pozzolans, the resultant blended cement will usually have better mechanical properties over the long term and also, more importantly, better durability. The increase in durability is the result of the ability of pozzolans to tie up free lime and other undesirables such as alkalis that would otherwise cause durability problems with the cement. In terms of overall chemistry, slag is similar to Class F coal fly ash, which is well known to be a good pozzolan. Although mineralogically slag is completely different from Class F fly ash, their similarity in overall chemistry makes slag a viable candidate as a pozzolan. Therefore, tests were conducted to evaluate the pozzolonic activity of gasifier slag with the intention of using it as a blended material in cement. Two ASTM tests were used to examine the suitability of slag as a pozzolan. The tests are:

- ASTM C 311: Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland Cement Concrete
- ASTM C 593: Fly Ash and Other Pozzolans for Use with Lime.

As indicated by the ASTM tests, these are two different applications. Fine ground slag was prepared and tested according to the ASTM standards. The results for each test are shown in Table 63 and Table 64 respectively.

**Table 63. ASTM C 311 Results Using Specially Prepared Slag**

|                                   | <b>OPC</b> | <b>35% Slag Replacement</b> |
|-----------------------------------|------------|-----------------------------|
| 28-day compressive strength (psi) | 3,948      | 5,489                       |
| Pozzolonic activity               | 100%       | 139%                        |

As shown in Table 63, finely ground slag was indeed a very good pozzolan with a pozzolonic index of 139%. The compressive strength of the baseline sample using ordinary portland cement (OPC) was slightly less than 4,000 psi after 28 days of curing. With 35% replacement of OPC

by slag, the compressive strength increased dramatically to just under 5,500 psi after 28 days of curing.

Slag was also tested according to ASTM C 593 and the results are shown in Table 64.

**Table 64. ASTM C 593 Results Using Specially Prepared Slag**

|                                  | <b>Minimum Requirement</b> | <b>Test Sample</b> |
|----------------------------------|----------------------------|--------------------|
| 7-day compressive strength, psi  | 600                        | 1,104              |
| 28-day compressive strength, psi | 600                        | 1,326              |

The compressive strength of the slag samples as tested according to ASTM C 593 was excellent and far exceeded the ASTM requirement.

It is evident from these tests that finely ground gasifer slag is indeed a good pozzolan and may be used beneficially in portland cement blends.

In order to further quantify the pozzolanic effect of ground slag and its relations to particle size, additional tests were conducted both at Fuller Co. and Pennsylvania State University. A quantity of slag in storage at Fuller Co. was ground to -200 mesh and -325 mesh nominal, respectively, and the Bond Ball Mill Grindability Test was performed on these two samples. This is a standard ball mill test used to determine the relative power requirements for grinding various materials. Prior to testing, the slag was ground to -6 mesh. The initial particle size distribution is shown in Table 65.

**Table 65. Initial Particle Size Distribution of Slag Prior to Bond Ball Mill Grindability Test**

| <b>Size</b> | <b>Passing</b> |
|-------------|----------------|
| 6 M         | 100%           |
| 8 M         | 64.5%          |
| 12 M        | 21.9%          |
| 16 M        | 9.7%           |
| 20 M        | 6.1%           |
| 30 M        | 4.2%           |
| 40 M        | 3.0%           |
| 50 M        | 2.1%           |
| 70 M        | 1.4%           |
| 100 M       | 0.9%           |
| 140 M       | 0.6%           |
| 170 M       | 0.5%           |
| 200 M       | 0.4%           |
| 325 M       | 0.2%           |



The results of the grinding test are shown in Table 66.

**Table 66. Slag Bond Ball Mill Grindability Test Results**

|                           | <b>Blaine Surface Area</b> | <b>BWI*</b> |
|---------------------------|----------------------------|-------------|
| -200 M Grind              | 1592 m <sup>2</sup> /g     | 22.8 kw/mt  |
| -325 M Grind              | 2558 m <sup>2</sup> /g     | 27.9 kw/mt  |
| ** Typical cement clinker | ~3800 m <sup>2</sup> /g    | 13-15 kw/mt |

\*BWI: Bond Work Index, which represents the energy required for grinding.

\*\* Typical cement clinker shown here for comparison.

The BWI of the ground slag was higher than that of a typical cement clinker. The higher BWI characteristic of gasifier slag may be attributed to its crystal size. If the crystal size of the gasifier slag is larger than that of cement clinker despite other physical properties being similar, it will take considerably more power to grind the slag to a similar Blaine. Grinding aides were not used when determining the BWI. If the higher BWI of the slag is attributed to its crystal size, grinding aides may not provide much of a benefit. On the other hand, since the slag is slightly magnetic, if the magnetic properties of the material affect flow in the mill and reduce the grinding effectiveness of the mill charge, the addition of a grinding aide may help. Slags are generally harder to grind. A large percentage of cement producers use steel slag in their raw materials, and steel slag often has a BWI of >20.

The Sedigraph Particle Size Distribution of the ground slags is shown in Table 67.

**Table 67. Sedigraph Particle Size Distribution of Ground Slag**

| <b>Cumulative % Passing</b> |                 |                 |
|-----------------------------|-----------------|-----------------|
|                             | <b>200 mesh</b> | <b>325 mesh</b> |
| <b>Microns</b>              | <b>Grind</b>    | <b>Grind</b>    |
| 75                          | 100             | 100             |
| 53                          | 77.8            | 100             |
| 45                          | 66.3            | 100             |
| 32                          | 47.1            | 82.5            |
| 25                          | 36.1            | 66              |
| 20                          | 27.9            | 52.6            |
| 15                          | 20.5            | 39.2            |
| 10                          | 13              | 25.7            |
| 6                           | 6.4             | 14.2            |
| 5                           | 5.1             | 11.5            |
| 4                           | 3.6             | 9.2             |
| 3                           | 2.3             | 6.6             |
| 2                           | 1               | 4.6             |
| 1                           | 0               | 2.5             |

The -200 mesh material was subjected to ASTM C 311 and C 593 tests and the results are shown in Table 68 and Table 69.

**Table 68. ASTM C 311 Results Using -200 Mesh Ball Milled Slag**

|                                  | <b>OPC</b> | <b>35% Slag Replacement</b> |
|----------------------------------|------------|-----------------------------|
| 28-day compressive strength, psi | 3,948      | 3084                        |
| Pozzolanic activity              | 100%       | -8.64%                      |

**Table 69. ASTM C 593 Results Using -200 Mesh Ball Milled Slag**

|                                  | <b>Minimum Requirement</b> | <b>Test Sample</b> |
|----------------------------------|----------------------------|--------------------|
| 7-day compressive strength, psi  | 600                        | 987.5              |
| 28-day compressive strength, psi | 600                        | 813.3              |

As shown in Tables 54 and 55, gasifier slag can indeed be a very good pozzolan and deserves more in-depth studies. However, conventional ball milling may not be the best method for grinding this material. ASTM C 311 test results showed that -200 mesh ball-milled slag was not a very effective pozzolan with a pozzolanic activity of -8.64%. However, the -200 mesh ball-milled slag did pass the ASTM C 593 test, albeit not spectacularly. The processing (grinding) of gasifier slag into an effective pozzolan in a low-cost manner is not trivial and requires further experimentation and testing.

## 6.0 CONCLUSIONS AND RECOMMENDATIONS

### 6.1 General Conclusions

Separation and recovery of char from slag using the process developed by Praxis Engineers, Inc. was demonstrated successfully under this program. Since the char (unconverted carbon) present in gasification slag constitutes a hindrance to its utilization, its removal is a critical step in the development of utilization applications for slag. The results show that the Praxis process is effective in separating the char from the slag to yield a char-free slag product. The char-free slag can be sold as a substitute for several conventional aggregates used in high-volume applications.

Demonstration of the char separation process was followed by successful demonstration of the production of lightweight aggregates (LWA) and ultra-lightweight aggregates (ULWA) from slag at the pilot scale, using technology developed by Praxis. It was demonstrated that slag can also be blended with conventional expansive shale and clays to produce LWAs.

The slag samples used in the program were generated from two different gasification processes namely, those used by Destec and Texaco, each of which uses different bituminous coals. Thus the char separation and expanded slag production processes demonstrated during the project can be generalized to apply to most of the coals or gasification processes in current application in the United States. This includes the Tampa Electric and Wabash River Repowering IGCC projects co-funded by DOE.

All of the project slag samples tested expanded at temperatures ~400°F lower than those required for pyroprocessing of expansive shales and clays. This represents significant savings in pyroprocessing fuel energy requirements. In all of the types of expansion processes that were demonstrated, sufficient control of the product unit weight as a function of temperature was possible to produce LWAs and ULWAs. However, ULWAs produced using slag had unit weights of about 18 lb/ft<sup>3</sup>, which is considerably higher than those made from perlite which are typically in the 4-12 lb/ft<sup>3</sup> range. Nevertheless, it is important to note that the compressive strength of the slag-based ULWAs was also correspondingly higher. High strength, even though accompanied by high unit weight, was identified as a very useful property in several applications.

Extensive testing was performed to establish the potential for utilization of expanded slag aggregates in the 18-60 lb/ft<sup>3</sup> unit weight range in two distinct types of target applications:

1. **Conventional LWA Applications:** Expanded slags were successfully tested as total and partial substitutes for several conventional LWA applications, namely structural lightweight concrete, masonry blocks (CMUs), lightweight roof tiles and concrete panels (Wonderboard). It was concluded that expanded slag products meet the requirements for these lightweight aggregate applications. These applications use conventional LWAs in the 50-55 lb/ft<sup>3</sup> unit weight range which sell for ~\$50/ton. Since some of these applications could benefit from using aggregates with even lower unit weights, we used expanded slag in the 40-55 lb/ft<sup>3</sup> range to test these applications.

2. **Conventional ULWA Applications:** Expanded slags were successfully tested as total and partial substitutes for several conventional ULWA applications, namely insulating concrete, loose fill insulation, and nursery/horticultural applications. Perlite-based ULWAs in the <4-12 lb/ft<sup>3</sup> unit weight range sell for ~\$200/ton. Since the slag could only achieve a unit weight of 18 lb/ft<sup>3</sup> at the pilot scale, tests were conducted using this material to evaluate its comparative performance in these applications. It was concluded that this SLA meets some of the requirements for expanded perlite. However, optimal results were obtained when expanded slag was used as a partial substitute for perlite products.

## 6.2 Specific Conclusions from Applications Testing

Expanded slags were successfully tested as total and partial substitutes for several LWA and ULWA applications. The findings are summarized below.

### 6.2.1 Lightweight Aggregate Applications

LWA applications tested included lightweight roof tiles, lightweight blocks, structural concrete, and concrete panels. The findings for these applications are discussed below.

1. **Lightweight Roof Tiles.** Lightweight concrete roof tiles are being increasingly used on the West Coast and Florida because of their high fire rating, durability, and aesthetic appeal. The ASTM requirement for lightweight concrete is a compressive strength of 5,000 psi with a unit weight of <108 lb/ft<sup>3</sup> using a typical cement-to-aggregate ratio of 1:2.5. Two additives (a superplasticizer and an accelerator) are commonly used. Using an identical mix, the test results confirmed that ASTM and industry requirements were met for this application. In fact, a lower-unit-weight slag (<55 lb/ft<sup>3</sup>) may be used to produce slightly lighter tiles. Large-scale production and testing of SLA-based roof tiles could not be accomplished due to plant unavailability.
2. **Lightweight Blocks (CMUs).** Expanded slag aggregates were successfully tested for production of lightweight blocks. The target lightweight block product (8" CMU) weighs <27 lb on a dry basis. The ASTM and industry requirements for concrete used for this application include a compressive strength of 2,000 psi at a unit weight of <115lb/ft<sup>3</sup>, using a typical cement-to-aggregate ratio of <1:5. Commercial production of the lightweight blocks was accomplished successfully at a commercial manufacturing plant. We produced 250 blocks using the conventional block mix and the same materials (sand, cement, and fillers) currently used by the plant. SLA was used to replace the conventional LWA, and constituted 44% of the total aggregates by volume in the mix. In the production of slag LWA blocks, the entire automated production and post-production manufacturing process line was used without modification. The green CMUs were handled through the processing steps without any problems, and no special curing or handling requirements were needed. The product met both industry and ASTM requirements. SLA has an excellent potential to replace or complement the existing LWA for this application. The plant would use the SLA if its delivered price were lower than the price paid for LWA.

3. **Structural Concrete.** Structural concrete is used in the construction of high-rise buildings and bridges. Expanded slag was successfully used to make structural concrete that met ASTM and industry requirements of compressive strengths of 2,500-4,000 psi for sand LWA concrete with unit weights in the 105-115 lb/ft<sup>3</sup> range, using a typical industry cement-to-aggregate ratio of 1:4. SLA used as a partial replacement for clay-based aggregates produced concrete with an even higher strength of 5500 psi.
4. **Concrete Panels.** Concrete panels (Wonderboard) are used for structural reinforcement and as water-resistant backing for ceramic tile installations. The target specification for concrete for this application was the industry requirement of a compressive strength of 2,500 psi with a panel weight of 3.2-3.6 lb/ft<sup>2</sup> using a cement-to-aggregate ratio of 1:2.5. Tests conducted at the facility of a panel manufacturer demonstrated that expanded slag in the 35-40 lb/ft<sup>3</sup> unit weight range met their requirements and may even perform better than the conventional materials due to its lower unit weight. The panel manufacturer requested a quotation for supplying the expanded slag material with the intention of procuring a minimum of 20,000 tons/day of SLA products for one plant. They were also interested in participating in a joint venture to set up an SLA production facility to meet their increasing demand.

### 6.2.2 Ultra-lightweight Aggregate Applications

ULWA applications tested included insulating concrete, loose fill insulation, and nursery/horticultural applications. The findings for these applications are discussed below.

1. **Insulating Concrete.** Insulating concrete is used as an insulating top layer in built-up roofs and is typically manufactured using expanded perlite or shale. The application requires a 200-psi concrete. ASTM insulation requirement (thermal conductivity) for shale-based products with unit weights in the 50-90 lb/ft<sup>3</sup> range is 1.5-3.0 Btu-in/h-ft<sup>2</sup>-°F. For a perlite-based product (15-50 lb/ft<sup>3</sup> unit weight) the thermal conductivity required is 0.45-1.5 Btu-in/h-ft<sup>2</sup>-°F. In comparison, SLA concrete at a unit weight of 45.1 lb/ft<sup>3</sup> had a thermal conductivity of 0.98 Btu-in/h-ft<sup>2</sup>-°F. The thermal conductivity of SLA was better than that of the shale product but inferior to that of the perlite product. Both compressive strength and industry requirements were met or exceeded by the SLA product.
2. **Loose Fill Insulation.** Expanded perlite is used to fill the cavities in blocks used for construction of building exterior walls to improve their insulation properties. The property of interest defined by ASTM is the thermal resistance of the loose fill material. Thermal resistance requirements for perlite (7.4-11 lb/ft<sup>3</sup>) are 2.6-2.4 hr-ft<sup>2</sup>-°F/Btu. Tests using SLA with a unit weight of 29 lb/ft<sup>3</sup> showed a thermal resistance of 1.46 hr-ft<sup>2</sup>-°F/Btu, which is somewhat lower than the values for perlite ULWAs. However, SLA has the advantage with respect to other industry requirements such as its free-flowing nature, low friability and hence low dustiness, and low moisture retention.
3. **Horticultural Applications.** SLA with a unit weight of <20 lb/ft<sup>3</sup> was tested as a partial substitute for perlite and vermiculite at a commercial nursery. The control soil mix consisted of a combination of peat moss, bark, fiber, perlite, and vermiculite. Mixes were developed to test the use of SLA as a substitute for perlite only, as well as for both perlite and vermiculite. The SLA proved to be a successful substitute for perlite alone but not for mixes calling for a

mixture of perlite and vermiculite. The main problem with the SLA was its high drainage rate which necessitated more frequent watering. However, its higher unit weight was seen as an advantage in providing greater stability to larger potted plants and shrubs, and its higher strength was seen as an advantage in mechanized field/nursery applications.

### 6.3 Economics of SLA Production

The slag production economics were conducted using two parallel approaches:

- Comparison of the economics of SLA production vs. slag disposal
- Comparison of the economics of SLA production with the estimated market value of end products that can be made from it.

The market price of SLA was estimated taking into consideration the fact that it would likely command a lower price as a new, unproven material. The sale prices for SLA products were estimated at \$30/ton for block aggregates, \$35/ton for structural aggregates, and \$40/ton for roof tile and ultra-lightweight aggregate applications. Using a product mix based on the percentage of coarse and fine slag, the weighted average price of SLA was estimated at \$34.75/ton. This price was used to evaluate the economics of SLA production.

For purposes of this analysis, a value of \$15/ton is used as the cost of slag disposal, which is in the middle of the range of \$10-20/ton indicated for fly ash. Since these avoided costs would provide substantial savings to the gasifier operation, this amount could potentially be made available to the SLA production facility as a tipping fee per ton of slag accepted.

For the economic analysis, four scenarios were studied representing two sizes of IGCC facilities (200 MW and 400 MW) each using two methods of SLA production (rotary kiln and fluidized bed). For the rotary kiln processes, the SLA production costs were estimated at \$30.07/ton and \$24.40/ton for the 200 MW and 400 MW sizes respectively (220 and 440 tons/day capacity). Conventional LWA production costs were estimated at \$30.10/ton based on a survey of four operating plants.

The fluidized bed method of SLA production was found to be even more competitive because of lower capital and operating costs. Its production costs were \$26.48 and \$21.87 for the smaller and larger sizes respectively. Therefore, such systems should be considered for commercial SLA production, especially for lower-capacity plants. The economics for the larger-sized fluidized bed plant are especially attractive if the avoided costs of slag disposal are taken into account, as indicated by a payback period of under three years.

## **6.4 Recommendations for Future Work**

IGCC technology is likely to play a major role in meeting the demand for energy in the United States, as well as in generation of syngas for chemical production. This assumption is substantiated by the rising prices of natural gas. As IGCC technology becomes a significant player in power generation, the technology for total utilization of the solid waste generated by the process (slag) should also be advanced. The development of slag utilization applications under this project has gone a long way toward meeting this goal. However, additional work is needed to test the durability of various SLA-based products and production techniques. The commercial adoption of SLA-based products would be accelerated by their recognition as acceptable aggregates by ASTM and other industry standards for applications in which they have been proven to meet applicable requirements.

Recently, there has been considerable interest in blending the coal feed to gasifiers with low-cost energy sources such as sewage sludge or refuse-derived fuel. These feed components will alter the basic chemistry of the slag produced. Systematic evaluation of slags produced from blended feeds would help to quantify how changing slag chemistry affects its expansion properties.

## LIST OF ACRONYMS AND ABBREVIATIONS

|                   |  |
|-------------------|--|
| ASTM              | American Society for Testing and Materials                                   |
| BWI               | Bond Work Index, which represents the energy required for grinding           |
| C <sub>2</sub> S  | Bicalcium silicate   |
| C <sub>3</sub> S  | Tricalcium silicate  |
| C <sub>3</sub> A  | Tricalcium aluminate   |
| C <sub>4</sub> AF | Calcium ferro aluminate  |
| CMU               | Concrete masonry unit  |
| DOE               | U.S. Department of Energy  |
| EPRI              | Electric Power Research Institute  |
| ICCI              | Illinois Clean Coal Institute  |
| IGCC              | Integrated-gasification combined cycle                                       |
| LWA               | Lightweight aggregate  |
| NCMA              | National Concrete Masonry Association  |
| NETL              | National Energy Technology Laboratory  |
| OPC               | Ordinary portland cement   |
| Prepared slag     | Slag processed for removal of unconverted carbon (char) using Praxis process |
| RCRA              | Resource Conservation and Recovery Act                                       |
| Raw slag          | As-generated slag discharged from the gasifier                               |
| SLA               | Slag lightweight aggregate or expanded slag                                  |
| SSD               | Saturated surface dry  |
| TCLP              | Toxicity Characterization Leaching Procedure                                 |
| TEC               | Tampa Electric Company   |
| TGA               | Thermogravimetric analysis   |
| ULWA              | Ultra-lightweight aggregate  |



## **Appendix A. Test Plan for Applications of Expanded Slags (Task 2.1)**

### **1.0 Objectives**

In Phase I, Praxis demonstrated its process for recovery of unconverted carbon (char) from slag. We also produced expanded lightweight aggregates ranging between 15-50 lb/ft<sup>3</sup> unit weight from coal gasification slag at the pilot scale. In Phase II, the slag-based lightweight aggregates (SLA) will be characterized and tested for their suitability for various lightweight aggregate (LWA) and ultra-lightweight aggregate (ULWA) applications. This document outlines the testing procedure that will be followed during the evaluation process. This plan will also help potential users to identify the ways in which the expanded slag products differ from the conventional materials, and determine how best to modify the mix designs to utilize them while meeting the strength requirements specified by ASTM or industry standards.

SLA products will be characterized and tested for their suitability as substitutes for the following lightweight aggregate (LWA) applications:

- Structural concrete
- Concrete blocks
- Lightweight roof tiles
- Concrete panels.

SLA products will also be characterized and tested for their suitability as substitutes for the following ultra-lightweight aggregate (ULWA) applications:

- Insulating concrete
- Loose fill insulation
- Horticultural applications

The following other applications will also be tested:

- Pozzolanic material

ASTM Standard C 125 defines the term "coarse aggregate" as the aggregates that are predominantly retained on a 4-mesh (4.75-mm) sieve. The term "fine aggregate" applies to aggregates that almost entirely pass a 4-mesh sieve but are predominantly retained on a 200-mesh (0.074-mm) sieve. Another commonly used term is "combined aggregate," which represents a mixture of coarse and fine aggregates. Various potential applications for which specific expanded SLA products that may be evaluated are identified in Table A-1 and the test needs and procedures are outlined in various sections in this document.

**Table A-1: Application Tests and Associated Expanded Slag Samples to be Used**

|                           | Aggregates Targeted for Substitution  |                        | Expanded Slag Product Sample |                                  | Ref. Section |
|---------------------------|---|------------------------|------------------------------|----------------------------------|--------------|
| <b>LWA Applications</b>   |   |                        |                              |                                  |              |
|                           | Target Size/Specifications  | Unit Wt                | Expanded Slag                | Slag/Clay Pellets <sup>(1)</sup> |              |
| Structural concrete       | (i) 3/4" coarse LWA ASTM C330   | 50 lb/ft <sup>3</sup>  | -----                        | 50/50                            | 2.1.1        |
|                           | (ii) 3/8" combined LWA ASTM C330  | 50 lb/ft <sup>3</sup>  | 1/4" x 50M                   | -----                            | 2.1.2        |
|                           | (iii) 3/4" pilot plant LWA  | 50 lb/ft <sup>3</sup>  | -----                        | 0/100                            | 2.1.3        |
| CMU concrete              | Fine (-4M) LWA  | 50 lb/ft <sup>3</sup>  | 1/4" x 50M                   | 50/50                            |              |
| Roof tile concrete        | Two SLA samples of fine (-6M) LWA, size gradation given by manufacturer and two control samples | 40 lb/ft <sup>3</sup>  | Crushed<br>1/4" x 50M        | 50/50<br>(Crushed)               |              |
| <b>ULWA Applications</b>  |   |                        |                              |                                  |              |
| Insulating concrete       | ASTM C332 Group II (45-90 lb/ft <sup>3</sup> concrete)  |                        |                              | ---                              |              |
| Loose fill insulation     | ASTM C549   | <12 lb/ft <sup>3</sup> | 10 x 50M                     | ---                              |              |
| Horticulture              | Expanded perlite size range   | <12 lb/ft <sup>3</sup> | 10 x 50M                     | ---                              |              |
| <b>Other Applications</b> |   |                        |                              |                                  |              |
| Pozzolanic material       | ASTM C311   | 30 lb/ft <sup>3</sup>  | 10 x 50M                     | ---                              |              |

## 2.0. Testing of SLA for Structural Lightweight Concrete Applications

The objective of this test program will be to develop mix designs using expanded slag lightweight aggregates (SLA) to produce sand-lightweight aggregate-based cement concrete of 2500-4000 psi strength with corresponding unit weights ranging between 115-105 lb/ft<sup>3</sup> by varying the cement content in the mix. Samples of SLA to be tested are identified in Table A-2. In addition, a sample of a commercially available structural aggregate will be tested as a control sample during the testing of materials identified under items (i) and (ii) in the table. As may be seen in the table, testing of SLA as 3/4" coarse aggregates will involve demonstration of three levels of strength (complete matrix), whereas testing with the other samples will only involve one level of cement.

**Table A-2: SLA Products to be Tested and Tentative Cement Levels**

| <b>SLA Products to be Tested</b>                                   | <b>Tentative Level of Cement, Sacks/Yard<sup>3</sup></b> |
|--|--|
| (i) 3/4" expanded SLA (50/50 slag-clay pellets) as 3/4" coarse LWA | 5½, 6½ and 7½  |
| (ii) 1/4" x 50M expanded SLA crushed as 3/8" combined LWA          | One level of cement                                      |
| (iii) Expanded 3/4" clay pellets produced during the pilot program | One level of cement                                      |

Exploratory testing may be necessary to establish the appropriate sand, water or cement requirements and confirm the strength and unit weight of the resulting concrete prior to completing the final batch of tests.

The ASTM unit weight requirements for the structural concrete aggregates are summarized in Table A-3. Also provided in Table A-3 are the unit weight and compressive strength requirements for cement concrete mixtures produced from 100% LWA or various mixtures of LWA and sand for reference purposes.

**Table A-3: Lightweight Aggregate and Structural Concrete Unit Weight and Compressive Strength Requirements (ASTM C 330)**

| <b>Structural Lightweight Aggregate Unit Weight Requirements</b>             |   |   |   |
|--|---|---|---|
|  | <b>Fine Aggregate</b>                       | <b>Coarse Aggregate</b>                     | <b>Combined</b>                             |
|  | <b>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> | <b>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> | <b>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> |
| Unit weight, maximum values  | 70 (1120)                                   | 55 (880)                                    | 65 (1040)                                   |
| <b>Lightweight Structural Concrete Unit Weight and Strength Requirements</b> |   |   |   |
| <b>Concrete Unit Weight</b>  |   | <b>28-Day Compressive Strength</b>          |   |
| <b>lb/ft<sup>3</sup></b>   | <b>(kg/m<sup>3</sup>)</b>                   | <b>lb/in<sup>2</sup></b>                    | <b>MPa</b>                                  |
| <b>All Lightweight Aggregate</b>   |   |   |   |
| 110  | (1760)                                      | 4000  | (28)  |
| 105  | (1680)                                      | 3000  | (21)  |
| 100  | (1600)                                      | 2500  | (17)  |
| <b>Sand-Lightweight Aggregate</b>  |   |   |   |
| 115  | (1840)                                      | 4000  | (28)  |
| 110  | (1760)                                      | 3000  | (21)  |
| 105  | (1680)                                      | 2500  | (17)  |

**2.1 Procedure for Evaluation of Pelletized (50/50) SLA as 3/4" Coarse Aggregates**

The following procedure will be used for preparation of aggregate:

- Obtain a sample of commercially available 3/4" coarse structural LWA for use as a control. Measure its unit weight and conduct a sieve analysis. (This sample will be provided by Praxis.)
- Crush 25 lb 50/50 slag/clay blend extruded for pelletization, and expanded to 50 lb/ft<sup>3</sup> unit weight to pass 1" top size in order to meet the ASTM C 330 for 3/4" coarse aggregates. Compare the size distribution of this material with that of the commercial LWA sample. (This sample will be prepared and supplied by Fuller Co.)

The following procedure will be used for preparation of cement concrete:

- Saturate all the aggregates overnight to bring them to the saturated surface dry (SSD) condition and document their moisture content.
- Estimate the sand content for the concrete mix to achieve a suitable gradation in the mix.
- Prepare cement concrete mixes using the slag aggregates prepared above. Use an aggregate-to-cement ratio identified by exploratory test to achieve a 28-day strength of 2500, 3000, and 4000 psi respectively. Document the water required and calculate the

cement-to-water ratio. Also, report the final volume and weight of the ingredients used in the mix design and measure the unit weight of the concrete. Prepare sufficient test specimens for the following tests (estimated at 12-15).

- Conduct 3-day, 7-day, and 28-day compressive strength tests as per ASTM and use the average of 3 measurements.
- Save test specimens to conduct the following tests if decided at a later date:
  - Conduct Freeze Thaw as per C 666
  - Conduct Drying Shrinkage as per C157
  - Conduct Staining C 641.
- Prepare control test specimens using the commercial LWA sample with an identical aggregate-to-cement ratio and slump for the 3000 strength. Measure the water added to document the cement-to-water ratio. Measure the unit weight of the concrete and compressive strengths for 3-day, 7-day and 28-day curing time for comparison.

## 2.2 Procedure for Evaluation of SLA as 3/8" Combined Aggregates

Tests will be conducted using 1/4" x 50M SLA to evaluate it as 3/8" combined LWA which is 100% passing 1/2" topsize. The unit weight of the expanded slag is to be chosen such that the finished concrete is within 110 lb/ft<sup>3</sup>. A sample of commercially available 3/8" combined structural LWA will be used to prepare a control mix and to compare its gradation with the prepared 1/4" x 50M SLA. The sand content for the concrete mix will be estimated to achieve a suitable gradation for this application.

The following procedure will be used for preparation of aggregates:

- Procure a 3/8" combined commercially available LWA for use as a control. Conduct a sieve analysis for use as a guideline for preparing the expanded slag. (This sample will be provided by Praxis.)
- Prepare 10 lb of SLA to meet ASTM 330 3/8" combined aggregate size requirements using 50 lb/ft<sup>3</sup> expanded 1/4" x 50M slag (or by blending appropriate quantities of 1/4" x 10M and 10M x 50M SLA fractions). Crush this material to pass 1/2" topsize and screen out any excess fines. Run a sieve analysis of the final crushed expanded slag aggregates and compare these with the commercially available 3/8" combined aggregate. (This sample will be prepared and supplied by Fuller Co.)

The following procedure will be used for preparation of cement concrete:

- Saturate all the aggregates overnight to bring them to the saturated surface dry (SSD) conditions and document their moisture content
- Estimate the sand content for the concrete mix to achieve a suitable gradation in the mix.

- Prepare cement concrete mixes using the slag aggregates prepared above. Use an aggregate-to-cement ratio identified by exploratory testing to achieve a 28-day strength of 3000 psi. Document the water required and calculate the cement-to-water and aggregate-to-sand ratios. Also, report the final volume and weight of the ingredients used in the mix design and measure the unit weight of the concrete. Prepare sufficient test specimens for the tests listed below.
- Measure the unit weight of the concrete and conduct 3-day, 7-day, and 28-day compressive strength tests as per ASTM, using the average of 3 measurements. Save samples for freeze/thaw tests.
- Prepare control test specimens using the commercial LWA sample with an identical aggregate-to-cement ratio and slump. Measure the water added to document the cement-to-water ratio. Measure the unit weight of the concrete and compressive strength for 3-day, 7-day and 28-day curing time for comparison.

### **2.3 Procedure for Evaluation of Pelletized Clay as 3/4" Coarse Aggregates**

The purpose of this testing is to compare the performance of the control sample of expanded LWA produced from extruded clay pellets produced during pilot kiln operations.

The following procedure will be used for preparation of aggregates:

- Procure a sample of commercially available 3/4" coarse structural LWA for use as a control. Measure its unit weight and conduct a sieve analysis. (This sample will be supplied by Praxis.)
- Crush 10 lb of extruded clay pellets expanded to 50 lb/ft<sup>3</sup> unit weight to pass 1" top size in order to meet the ASTM C 330 for 3/4" coarse aggregates. Compare the size distribution of this material with that of the commercial LWA sample. (This sample will be prepared and supplied by Fuller Co.)

The following procedure will be used for preparation of cement concrete:

- Saturate all the aggregates overnight to bring them to saturated surface dry (SSD) conditions and document their moisture content
- Estimate the sand content for the concrete mix to achieve a suitable gradation in the mix.
- Prepare cement concrete mixes using the clay pellet aggregates prepared above. Use an aggregate-to-cement ratio to achieve a 28-day strength of 3000 psi. Target a slump identical to the other tests, document the water required, and calculate the cement-to-water and aggregate-to-cement ratios. Also, report the final volume and weight of the ingredients used in the mix design.

- Measure the unit weight of the concrete and conduct 3-day, 7-day, and 28-day compressive strength tests as per ASTM and using the average of three measurements.

## 2.4 Data and Reports

Provide laboratory data sheets with brief comments on the nature of the results.

## 3.0. ROOF TILE CONCRETE APPLICATION TESTING

The objective of this testing is to confirm if the (i) expanded slag, (ii) pelletized slag and (iii) pelletized clay aggregates can meet roof tile aggregate requirements. Lightweight concrete roof tiles are made with LWA and a high cement paste, which is molded continuously. The paste also contains a plasticizer and an accelerator. Each manufacturer use a proprietary mix design. Curing is done in a high-temperature, high-humidity atmosphere for the initial 4 hours. The remainder of the curing is done on shrink-wrapped pallets in the yard. Aggregates used for roof tile application are typically fine aggregates as per ASTM C 331 (85-100% passing 4 mesh). However, roof tile manufacturers also specify particle size distribution along with a unit weight of 40 lb/ft<sup>3</sup>. This application is very demanding due to the high compressive strength and flexural strength requirements. The typical 28-day compressive strength of the concrete is over 5,000 psi at unit weight of 108 lb/ft<sup>3</sup>.

### 3.1. Procedure for Evaluation of SLA 50/50 pellets (35-40 lb/ft<sup>3</sup>)

The first step is to prepare the manufacturer specified size gradation by stage crushing and screening. The target unit weight of the aggregates is 40 lb/ft<sup>3</sup>. The following procedure will be used for aggregate preparation:

- Procure a 50-lb sample of roof tile aggregate from a roof tile manufacturer for use as a control sample. Conduct moisture analysis and screen analysis, and measure the fractional unit weights.
- Crush 50:50 expanded slag/clay pellets with a unit weight of 35-40 lb/ft<sup>3</sup> to pass a minus-4M sieve in stages to reproduce the size distribution as per the range provided by the roof tile manufacturer. Conduct the following tests and compare the results with the manufacturer's specifications and with the conventional aggregate sample as appropriate:
  - Moisture absorption (target <15% by wt.)
  - Sieve analysis at 4, 8, 16, 30, 50, and 200M sieves
  - Unit weight by size fractions and for the whole sample.
- Crush the expanded all clay pellets of 35-40 lb/ft<sup>3</sup> unit weight to pass -4M sieve following the procedure given above. Conduct the following tests and compare the results with the manufacturer's specifications and with the conventional aggregate sample as appropriate:

- Moisture absorption (target <15% by wt.)
- Sieve analysis at 4, 8, 16, 30, 50, and 200M sieves
- Unit weight by size fractions and for the whole sample

The following procedure will be used for preparation of cement concrete:

- Saturate all the aggregates overnight to bring them to saturated surface dry (SSD) conditions and document their moisture content
- Prepare a batch of cement concrete sufficient to make test specimens using the mix design given below. Mix the ingredients thoroughly using a mechanical mixer or by hand, keeping track of the mixing time for preparing subsequent batches. Additives may be used based on the manufacturer's recommendation, as given below:
  - Cement-to-aggregate ratio by weight: 1:2.5 on dry basis
  - Cement type: Type II
  - Accelerator (HICO HB98): 3.5 ml/100 g of cement
  - Plasticizer (MBL Rheobuilt): 3.5 ml/100 g of cement
  - Slump: 0-1
- Prepare 15 2" specimens using the above mix design taking care to avoid the formation of air voids. The test specimens are to be cured in a chamber at 120°F and 95% RH for 4 hours. Subsequent curing will be done in a constant-temperature bath as per ASTM.
- Document the total weight and volume of ingredients used and calculate the water-to-cement ratio. Measure the unit weight of the fresh concrete. Report the workability of the mix.
- Test the specimens for compression following 1-day (early strength), 3-day, 7-day, and 28-day curing times. Save three cylinders for further testing.

### **3.2. Procedure for Evaluation of SLA (35-40 lb/ft<sup>3</sup>) as Roof Tile Aggregates**

The first step is to prepare the manufacturer's specified size gradation by stage crushing and screening. The target unit weight of the aggregates is 40 lb/ft<sup>3</sup>. The following procedure will be used for preparation of aggregates:

- Stage crush 10 x 50M SLA with a unit weight of 35-40 lb/ft<sup>3</sup> to pass a minus-4M sieve in stages to reproduce the size distribution as per the range provided by the roof tile manufacturer. Conduct the following tests and compare the results with the manufacturer's specifications and with the conventional aggregate sample as appropriate:
  - Moisture absorption (target <15% by wt.)
  - Sieve analysis at 4, 8, 16, 30, 50, and 200M sieves
  - Unit weight by size fractions and for the whole sample



The following procedure will be used for preparation of cement concrete:

- Adjust the moisture content of the aggregates to SSD conditions.
- Prepare a batch of cement concrete sufficient to make test specimens using the mix design given below. Additives may be used based on the manufacturer's recommendation. Mix the ingredients thoroughly using a mechanical mixer or by hand, keeping track of the mixing time for preparing subsequent batches.
  - Cement-to-aggregate ratio by weight: 1:2.5 on dry basis
  - Cement type: Type II
  - Accelerator (HICO HB98): 3.5 ml/100 g of cement
  - Plasticizer (MBL Rheobuilt): 3.5 ml/100 g of cement
  - Slump: 0-1
- Prepare 15 2" specimens using the above mix design taking care to avoid the formation of air voids. The test specimens are to be cured in a chamber at 120°F and 95% RH for 4 hours. Subsequent curing will be done in a constant-temperature bath as per ASTM.
- Document the total weight and volume of the ingredients used and calculate the water-to-cement ratio. Measure the unit weight of the fresh concrete. Report the workability of the mix.
- Test the specimens for compression following 1-day (early strength), 3-day, 7-day, and 28-day curing times. Save 3 cylinders for further testing.

### 3.3 Procedure for Testing Expanded Clay (35-40 lb/ft<sup>3</sup>)

The following aggregate preparation method will be used:

- Repeat the steps for size reduction given above for crushing 50/50 pellets
- Conduct the following tests and compare the results with the manufacturer's specifications and with the conventional aggregate sample as appropriate:
  - Moisture absorption (target <15% by wt.)
  - Sieve analysis at 4, 8, 16, 30, 50, and 200M sieves
  - Unit weight by size fractions and for the whole sample

The following procedure will be used for preparation of cement concrete:

- Prepare a concrete mix design using the procedure given above.
- Prepare 12 2" cylinders to conduct early (1-day), 3-day, 7-day and 28-day strength tests.

#### 4.0. LIGHTWEIGHT BLOCK AGGREGATE SPECIFICATIONS

ASTM C 331 specifies unit weight requirements for aggregates and unit weight and strength requirements for cement concrete used in manufacturing lightweight concrete masonry units. These requirements are summarized in Table A-4.

**Table A-4: LWA and Cement Concrete Requirements for CMU Applications**

| <b>Lightweight Aggregate Unit Weight Requirements for CMU (ASTM C 331)</b> |             |   |  |  |
|--|-------------|---|--|--|
|  |             | <b>Fine<br/>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b>        | <b>Coarse<br/>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> | <b>Combined<br/>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> |
| Unit weight, max. values   |             | 70 (1120)   | 55 (880)   | 65 (1040)  |
| Industry preference  |             | NA  | NA   | 50 (800)   |
| <b>Lightweight Concrete Unit Weight and Strength Requirements for CMU</b>  |             |   |  |  |
|  |             |   | <b>28-Day Compressive Strength</b>                     |  |
|  | <b>ASTM</b> | <b>Unit Weight<br/>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> | <b>Gross<br/>psi (MPa)</b>                             | <b>Net<br/>psi (MPa)</b>                                 |
| Load-bearing   |             |   |  |  |
| - below grade  | C 90        | <105 (1682)   | 1000 (14)  | 2000 (28)*   |
| - above grade  | C 90        | <85 (1362)  | 700 (10)   | 1400 (20)*   |
| Nonload-bearing  | C 129       | 105 (1682)  | NA   | 600 (8.4)  |

\*Net compressive strength values calculated by assuming net cross-sectional area is 50% of gross area.

As would be expected, the strength requirements for load-bearing blocks are higher than those for nonload-bearing units. The standard does not, however, specify the cement concrete mix design for blocks, thus allowing a degree of flexibility.

#### 5.0 CONCRETE PANELS

This work will be done using the laboratory of a leading manufacturer of cement concrete panels. They will use their internal procedure for evaluation of the SLA in this application.

## **6.0 INSULATING CONCRETE**

Perlite insulating concrete is made from perlite (a Group I aggregate), portland cement, water, and other additives including an air-entraining agent. Insulating concrete made with perlite has an oven-dry unit weight of 15-50 lb/ft<sup>3</sup>. Insulating concrete may also be made from expanded shale, blast-furnace slag, or fly ash (Group II aggregates), resulting in a concrete unit weight of 50-90 lb/ft<sup>3</sup>. The minimum compressive strength for perlite insulating concrete recommended by the Perlite Institute is 350 psi at a concrete unit weight of 36 lb/ft<sup>3</sup>, and 80 psi at a concrete unit weight of 22 lb/ft<sup>3</sup>.

### **6.1 Procedure for Preparation Of SLA For Use In Insulating Concrete**

ASTM C 332 provides specifications for insulating concrete aggregates. The detailed specifications for perlite and vermiculite aggregates (Group I) and shale-based aggregates (Group II) for insulating concrete are given in ASTM C 332 and summarized in Table A-5. The category selected for testing of SLA is Combined (Fine and Coarse) 3/8" x 0 Aggregates. The material used for these tests would be an appropriate mix of 1/4" x 10 mesh, 10 x 50 mesh expanded slag and necessary portions of pulverized expanded slag to replicate the ASTM C 332 (Table A-5) size requirements. A 70-lb batch of aggregate will be prepared for this purpose. Aggregate sample preparation will be done at Fuller and the preparation of test specimens and compression testing will be done at Pennsylvania State University. The thermal conductivity specimen will be shipped to Energy Materials Testing Laboratory in Biddeford, Maine.

**Table A-5: Aggregate Specifications for Insulating Concrete (ASTM C332)**

| <b>Aggregate Size and Unit Weight</b>                              |                          |                           |  |       |                |        |
|--|--------------------------|---------------------------|--|-------|----------------|--------|
|  | <b>Size</b>              |                           | <b>Minimum</b>   |       | <b>Maximum</b> |        |
|  | <b>Mesh/inch</b>         | <b>(mm)</b>               | <b>Unit Weight, lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b> |       |                |        |
| <b>Group I Aggregates (Perlite and Vermiculite)</b>                |                          |                           |  |       |                |        |
| Perlite  | -4 M                     | (4.75)                    | 7.5  | (120) | 12             | (196)  |
| Vermiculite (coarse)   | -3/8"                    | (9.5)                     | 5.5  | (88)  | 10             | (160)  |
| Vermiculite (fine)   | -8 M                     | (2.36)                    | 5.5  | (88)  | 10             | (160)  |
| <b>Group II Aggregates (Shale, Ash, and Blast Furnace Slag)</b>    |                          |                           |  |       |                |        |
| Fine   | 4 M x 0                  | (4.75 x 0)                | --   |       | 70             | (120)  |
| Coarse   | 1/2" x 4 M               | (12.5 x 4.75)             | --   |       | 55             | (880)  |
| Coarse   | 1/8" x 8 M               | (9.5 x 2.36)              | --   |       | 55             | (880)  |
| Coarse   | 4 x 8 M                  | (4.75 x 2.36)             | --   |       | 55             | (880)  |
| Combined   | 1/2" x 0                 | (12.5 x 0)                | --   |       | 65             | (1040) |
| Combined   | 3/8" x 0                 | (9.5 x 0)                 | --   |       | 65             | (1040) |
| <b>Unit Weight and Thermal Conductivity of Insulating Concrete</b> |                          |                           |  |       |                |        |
|  | <b>Unit Weight</b>       |                           | <b>Thermal Conductivity</b>                              |       |                |        |
|  | <b>lb/ft<sup>3</sup></b> | <b>(kg/m<sup>3</sup>)</b> | <b>Btu-in./h-ft<sup>2</sup>-° F</b>                      |       | <b>(W/m-K)</b> |        |
| Group I Aggregates (perlite-based)                                 | 15-50                    | (240-800)                 | 0.45-1.5   |       | (0.065-0.22)   |        |
| Group II Aggregates (shale-based)                                  | 50-90                    | (800-1440)                | 1.5-3.0  |       | (0.22-0.43)    |        |

## 6.2 Preparation Of SLA-Based Concrete For Use In Insulating Concrete

The Perlite Institute has provided typical mix proportions for perlite insulating concrete. The mix design calls for using appropriate quantities of polypropylene fiber (15 denier, 1/4" to 1/2" long) and an air-entraining agent. These tests are to be done using a cement-to-concrete ratio of 1:5 by volume. However, if the addition of fiber becomes a problem, a cement-to-aggregate ratio of 1:6 is to be used without fiber. This will allow direct comparison of the results with the Perlite Institute data. The following test specimens are to be prepared:

- 2" cubes for unit weight, and 7-day and 28-day compression testing
- 12 inch square and 1" thick specimens for thermal conductivity testing.

## **7.0 LOOSE FILL INSULATION MATERIALS**

Expanded perlite is used to enhance the insulating properties of walls by filling the voids in masonry blocks. Typically, minus 4-mesh expanded perlite, with a 2-11 lb/ft<sup>3</sup> unit weight, is surface-treated with silicone to improve its water repellency prior to use in this application. Other material characteristics that are important for this application include high thermal resistance and low combustibility.

### **7.1 Preparation Of SLA For Use In Loose Fill Insulation**

ASTM C 459 provides specifications for loose fill insulation aggregates. The detailed specifications for expanded perlite used for loose fill insulation are given in ASTM C 549, which are summarized in Table A-6. A 10-lb batch of aggregate will be prepared (by screening) from SLA of the lowest unit weight produced from the fluid bed expander. Aggregate sample preparation and its characterization testing (unit weight, water repellency testing) will be done at Fuller. The sample will be shipped to Pennsylvania State University for shipment Energy Materials Testing Laboratory in Biddeford, Maine, along with the insulating concrete specimens for thermal conductivity testing.

**Table A-6: Perlite-Based Loose Fill Insulation Specifications (ASTM C 549)**

| <b>Physical Specifications</b>  |  |                     |                     |
|---|--|---------------------|---------------------|
|   | <b>Type I-II</b>   |                     | <b>Type III-IV</b>  |
| Unit weight, lb/ft <sup>3</sup> (kg/m <sup>3</sup> )                            | 2-11 (32-176)  |                     | 2-11 (32-176)       |
| Maximum +4 mesh (4.75 mm)   | 5%   |                     | 5%                  |
| Water repellency (min. ml H <sub>2</sub> O repelled)                            | NA   |                     | 175                 |
| Moisture absorption (max. wt%/14 days)  | 1.0%   |                     | 1.0%                |
| Wickability (max. grams wicked in 5 minutes)                                    | NA   |                     | 1.0                 |
| Combustibility (E 136)  | None   |                     | NA                  |
| Dust suppression (max. wt of collected material, mg)                            | NA   |                     | 85                  |
| <b>Thermal Resistance for 1-in. (25.4 mm) Thickness at Various Temperatures</b> |  |                     |                     |
| <b>Unit weight</b>  | <b>Thermal resistance, h-ft<sup>2</sup>-°F/Btu (m<sup>2</sup>-k/W)</b> |                     |                     |
| <b>lb/ft<sup>3</sup> (kg/m<sup>3</sup>)</b>                                     | <b>At 4°C</b>  | <b>At 24°C</b>      | <b>At 41°C</b>      |
| 2.0-4.1 (32.0-65.5)   | 4.3-3.9 (0.78-0.69)  | 3.9-3.3 (0.65-0.58) | 3.7-3.2 (0.65-0.56) |
| 4.1-7.4 (65.6-118.4)  | 3.9-3.3 (0.69-0.58)  | 3.3-2.8 (0.58-0.49) | 3.2-2.7 (0.56-0.47) |
| 7.4-11.0 (118.4-176.0)  | 3.3-2.9 (0.58-0.51)  | 2.8-2.4 (0.49-0.42) | 2.7-2.4 (0.47-0.42) |

- Type I: Expanded products
- Type II: Surface treated for H<sub>2</sub>O repellency
- Type III: Surface treated for dust control
- Type IV: Surface treated for H<sub>2</sub>O repellency and dust control.

## 8.0 HORTICULTURAL APPLICATIONS

SLA will be tested as a partial replacement for expanded perlite and vermiculite in horticultural applications. The specialized nature of this work requires that it be done with the involvement of a nursery. Procedures followed in evaluating conventional materials will be used to evaluate the expanded slag.