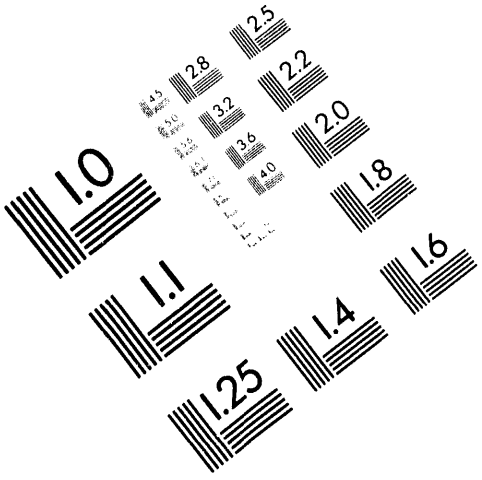

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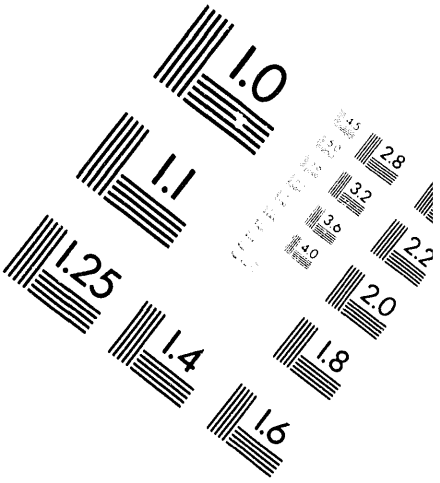

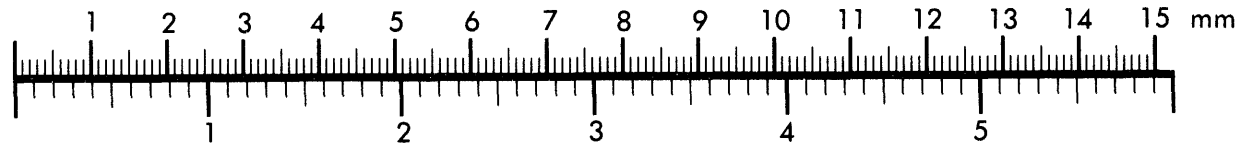

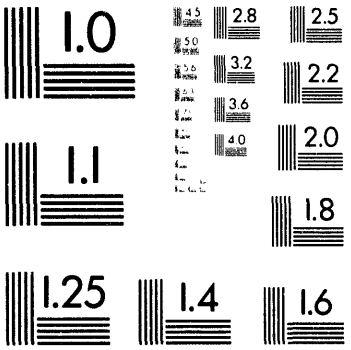

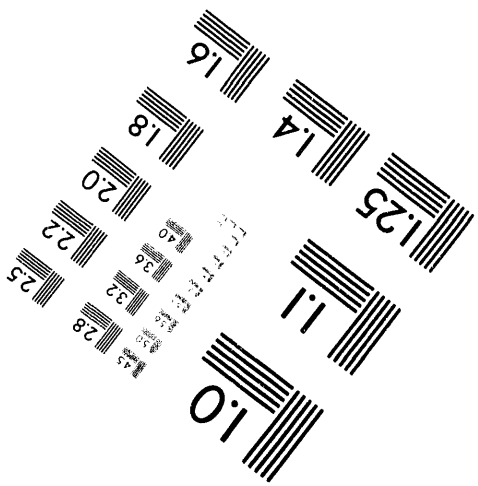

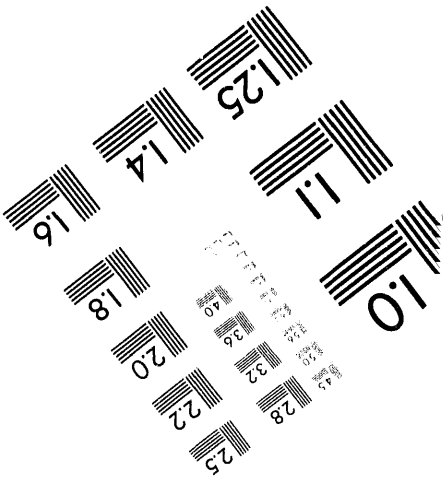

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DOE/PC/92521--T1&2

TECHNICAL REPORT
December 1, 1993 through February 28, 1994

Project Title: **LWA DEMONSTRATION APPLICATIONS USING ILLINOIS COAL GASIFICATION SLAG: PHASE II**

DOE Grant Number: DE-FC22-92PC92521 (Yr. 2)
ICCI Project Number: 93-1/4.4A-11M
Principal Investigator: Vas Choudhry, Praxis Engineers, Inc.
Other Investigators: Philip Steck, Harvey Cement Products, Inc.
Project Manager: Daniel Banerjee, Illinois Clean Coal Institute

ABSTRACT

The objectives of this program are to demonstrate the feasibility of producing ultra-lightweight aggregates (ULWA) from solid residues (slag) generated during the gasification of Illinois coals, and to test the products as substitutes for conventional aggregates produced by pyroprocessing of perlite ores. In Phase I of this project, Praxis developed a pilot-scale production technique and produced a large batch of expanded aggregates from an Illinois coal slag feed. The Phase II work focuses on characterization and applications-oriented testing of the expanded slag products as substitutes for conventional ULWAs. Target applications include uses such as loose fill insulation, insulating concrete, lightweight concrete precast products (blocks), waterproof wallboard, rooftiles, and filtration media. The expanded slag will be subjected to performance testing in conjunction with a commercial manufacturer of precast concrete products in order to obtain input from a potential user. The production of value-added products from slag will eliminate a solid waste and possibly enhance the overall economics of the gasification process, especially when the avoided costs of disposal are taken into consideration.

During this reporting period, major accomplishments were the selection of mix designs and test methods for preparation of specimens of expanded slag for testing in precast applications (Task 3) and construction aggregate applications (Task 4). In addition, characterization data (Task 1) were analyzed, and evaluation of the expanded slag products as substitutes for conventional ULWAs (Task 2) was completed. Potential applications that were identified are:

- Loose fill insulation
- Insulating concrete (roof, floor, and walls)
- Precast products (blocks and rooftiles)

Experimental work during the project is focused on these applications.

EXECUTIVE SUMMARY

Illinois coals are excellent feedstocks for coal gasification due to their high volatiles and Btu content and low cost. The advantages of Illinois coals in gasification can be further enhanced by utilizating the gasification solid residue (termed slag) as a by-product instead of disposing of it as a solid waste. Typical disposal costs for such solid wastes, including loading, transportation to a suitable disposal site, unloading, and subsequent monitoring, are currently estimated at $8-12 per ton. In addition to the high cost of disposal, public concern about environmental degradation makes utilization of solid wastes a high-priority issue.

Although Praxis has previously demonstrated a number of utilization applications for unprocessed slag such as use as aggregates in road construction and cement concrete production, these high-volume, low-value applications would not easily find acceptance in the marketplace where they would have to compete with cheap locally available conventional aggregates. Thus, it became apparent that value-added by-product applications need to be developed if slag is to be utilized in significant quantities.

The objectives of this project are to evaluate the lightweight and ultra-lightweight aggregates (ULWAs) produced from an Illinois coal slag in Phase I and to test their suitability as substitutes for conventional aggregates. Conventional ULWAs have unit weights between 3 and 12 lb/ft^3 and are commercially produced by subjecting naturally occurring expansible perlite ores to thermal processing. A number of ULWA applications have been targeted as potential applications for expanded slag, including the following:

- Loose fill insulation
- Insulating concrete
- Precast concrete products
- Filtration media
- Agricultural and horticultural applications

In Phase I of this project, a large batch of expanded aggregates with unit weights ranging between 12 and 45 lb/ft^3 was produced from an Illinois coal slag following extensive bench-scale testing. The work in Phase II is aimed at testing the utilization potential of the expanded slag aggregates and generating test data for various applications. Therefore, most of the testing involves potential users of expanded slag such as manufacturers of precast concrete products (blocks and rooftiles) and bulk users of expanded aggregates.

In the second quarter of Phase II, characterization of the expanded slag aggregates and their testing as substitutes for conventional ULWAs was begun. Although many of these tests are currently in progress, a preliminary evaluation of the

data indicates that expanded slag has potential for use as a loose fill insulation material. Other possible applications were deemed equally promising.

Compositional analysis of two of the expanded slag samples was performed using two different techniques in two laboratories. The results fall within the same range. In order to evaluate the environmental acceptability of the expanded slag, a sample was subjected to leachability testing using the EP Toxicity (TCLP) test. The tests indicated that the concentrations of RCRA metals in the TCLP extraction fluid were well below the EPA limits. The pH of the effluent with deionized water was in the alkali range.

An expanded slag sample was evaluated for its suitability as loose fill insulation, which is commonly used to fill voids in hollow concrete masonry units to improve the insulating property of walls and reduce heat loss. It was determined that this appears to be a viable application due to the easy flowing and nonfriable nature of the expanded slag and the fact that it is less dusty than perlite. Additional tests were conducted for this application, the results of which are not yet available.

Insulating concrete is made from expanded perlite (Group I) and expanded clay (Group II) aggregates to produce concretes with a wide range of unit weights. As a result, the concrete compressive strength varies widely due to the aggregate strength and the cement content of the mix. Testing of expanded slag for this application was conducted by preparing cement concrete mixes using expanded slag as the aggregate, using a 1:5 cement-to-aggregate ratio. Since only the +20-mesh slag was expanded in Phase I, the available expanded slag samples lack the requisite fines content. This can be made up by adding crushed expanded slag or natural sand. Therefore, two separate concrete mixes were made using sand and crushed expanded slag, respectively. Concrete test specimens were prepared from these mixes and are being cured. Compressive strength results will be available in the next reporting period.

At present, indications are that fine expanded slag or expanded perlite may be a better source of fines than crushed expanded slag. However, expanded perlite may be added in future tests. The results of these tests will also be used to finalize the insulating concrete mix designs.

Testing of expanded slag to make precast concrete blocks could not be undertaken due to the severe cold weather. The tests have been rescheduled for the next quarter. Evaluation of expanded slag in other applications is in progress.

OBJECTIVES

The Phase II work is aimed at testing and evaluation of the large batch of expanded slag encompassing a wide range of unit weights that was produced in Phase I. The major objectives of the Phase II work are to:

- Conduct extensive physical, chemical, and environmental characterization testing of expanded Illinois slag aggregates,
- Identify potential applications for expanded slag as a substitute for conventional ULWAs by comparing its physical and chemical properties with those of expanded perlite in various applications, with input from potential users,
- Demonstrate the use of the expanded slags in selected applications to generate performance data, and
- Conduct a detailed assessment of the technical and economic feasibility of using expanded slag in selected applications.

It is anticipated that the data generated will be applicable for implementation in DOE-funded coal gasification projects currently under way in the Illinois basin. The two leading projects are: (i) Wabash River Coal Gasification Repowering Project in Indiana, operated by Destec and PSI Energy, and (ii) IGCC Repowering Project at Lakeside Generating Station in Springfield, Illinois.

INTRODUCTION AND BACKGROUND

Based on previous work performed by Praxis, it was established that slags have inherent properties of expansion or bloating upon thermal treatment. Their bulk density can be controlled to achieve values between 12 and 55 lb/ft^3 by changing the process conditions, i.e., temperature and kiln retention time. This expansion characteristic is similar to that of naturally occurring expansible shales, clays, and perlite ores which are used to manufacture lightweight aggregates. All three commonly used expansion methods, i.e., direct-fired kiln, vertical shaft furnace, and indirect-fired kiln, were used to produce expanded slag aggregates.

The Phase I effort was directed at development of techniques for producing ULWA from Illinois coal slags using bench-scale equipment and a pilot vertical shaft furnace. The results of tests conducted in this phase confirm that a product with a

unit weight of 12-20 lb/ft^3 can be produced from Illinois slag in either a direct-fired vertical shaft furnace or an indirect-fired horizontal shaft furnace. However, the indirect-fired furnace allowed better process control during slag expansion. Using operating conditions determined during the pilot tests, a large batch of expanded Illinois slag was produced using an 18-in. diameter x 12-ft long indirect-fired horizontal furnace which is used by the manufacturer to generate scale-up data for the design of commercial furnaces.

In Phase II, expanded slag samples are being characterized for their environmental acceptability and evaluated for their suitability as substitutes for conventional ULWAs in a number of applications including loose fill insulation, insulating concrete, precast concrete products, and agricultural, horticultural, and industrial applications. The test data will establish their suitability for commercial use either directly or in blends with conventional aggregates.

EXPERIMENTAL PROCEDURES

Experimental procedures for evaluation of the expanded slag were selected based on ASTM and Perlite Institute standards and specifications, as indicated below.

Precast Concrete Applications

Expanded clay aggregates are used to produce precast concrete products such as lightweight blocks and rooftiles. Masonry blocks, a major application for LWAs, were selected for comprehensive application testing and development in this project. Cement concrete test specimens will be made from the expanded slag produced in Phase I, using appropriate mix designs, and tested in accordance with the following standards:

- Cement concrete unit weight and compressive strength (ASTM C 495)
- Flexural strength for rooftile application (ASTM C 293)
- Resistance to freeze/thaw (ASTM C 666)
- Dry shrinkage (ASTM C 157)
- Test for staining materials in lightweight aggregates (ASTM C 641)

Loose Fill Insulation

Expanded slag was tested for use as loose fill insulation in accordance with the general requirements for perlite given in ASTM C 549 by conducting the following tests:

- Density of loose fill insulation (ASTM C 520)
- Thermal transmission properties (ASTM C 177)
- Surface burning characteristics (ASTM E 84)
- Water repellency (PI 303-85)
- Resistance to compaction (PI 306-80)

Insulating Concrete

A number of insulating concrete mix designs were tested to develop a suitable mix using expanded slag. Subsequently, a specimen using the best mix design will be tested further for its insulating properties, which will involve the following tests:

- Unit weight of concrete (ASTM C 29)
- Compressive strength and density of insulation concrete (ASTM C 495)
- Thermal transmission properties of concrete (ASTM C 177)
- Fire rating (UL Design No. P920)

Other Applications

A number of other potential applications for expanded slag include filtration media and agricultural and horticultural applications. Test procedures for these applications will be finalized after reviewing their specifications and the results of the foregoing tests. This approach will provide sufficient test data for characterization of expanded slag and evaluation of potential applications. Characterization data will be similar to those typically provided for perlite products in Material Specifications Data Sheets (MSDS).

RESULTS AND DISCUSSION

Work on Phase II was started in September 1993 upon receipt of a letter of intent from ICCI. The technical work completed during this quarter is described by task.

Task 1: Characterization of Expanded Slag Aggregates

The unit weight and compaction resistance (PI 306-80) were measured for selected products, the results of which are presented in Figure 1. These data show some scatter due to the fact that the particle size interval of the slag feed samples was fairly wide. All of the expanded slag products were determined to have compaction resistance values 2-8 times higher than the 31.5 lb/ft^3 value measured for loose fill insulation grade (minus 4 mesh) expanded perlite. However, the compaction resistance-to-unit weight ratio for various slag samples is slightly higher than that of perlite.

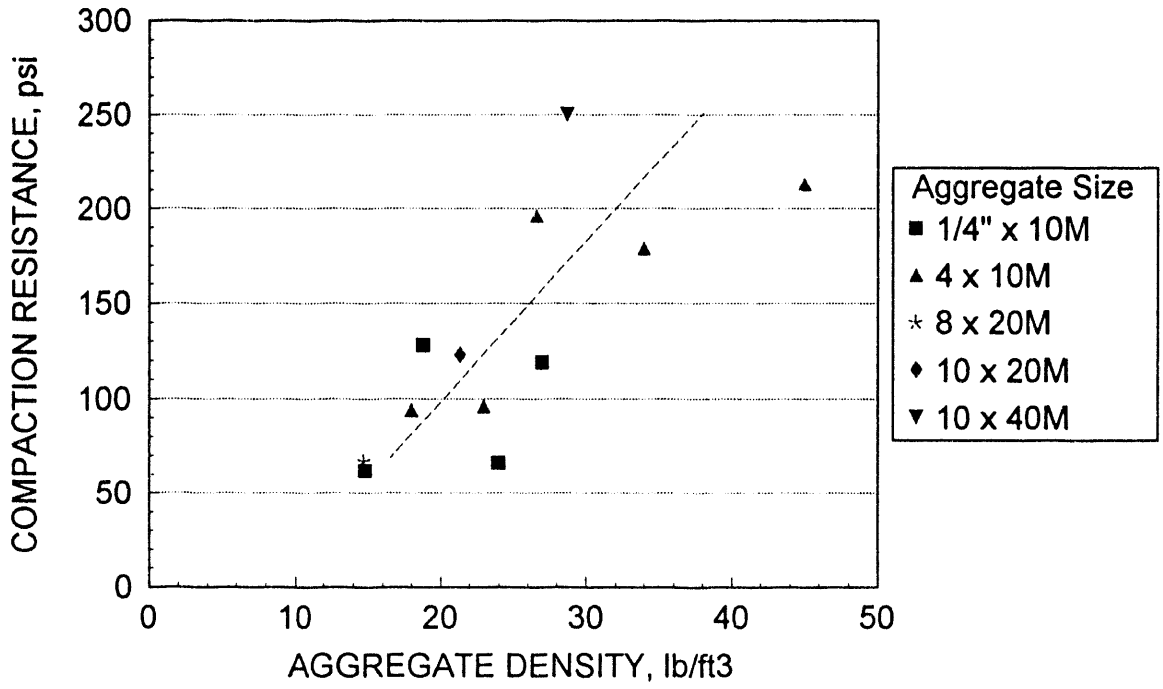

Figure 1. Compaction Resistance Test Results

Table 1 summarizes the results of physical characterization tests conducted on expanded slag samples, alongside data for expanded perlite at the same topsize. The true specific gravity of slag is slightly higher than that of perlite. The softening temperature of Illinois slag is in the upper range reported for perlite.

Table 1. Results of Physical Characterization of Expanded Slag

Parameter	ASTM	Expanded Slag	Expanded Perlite
Particle size		-4 mesh	-4 mesh
Hardgrove Grindability Index	D409	109	
Loss on ignition (char content)			
Apparent specific gravity		0.51	
True specific gravity		2.74	2.2-2.4
Friability			
Porosity		81	
Ash fusion temperature, °F (reducing)			
- initial deformation		2010	
- softening		2040	1600-2000
- hemispherical		2140	
- fluid		2220	2300-2450

Expanded slag from test 701-7.
Perlite information from Perlite Institute Data Sheet.

Table 2 presents the major and minor elements in the expanded slag reported as oxides, with equivalent values for perlite for purposes of comparison. The results eported are from two different laboratories for comparison of the variability which is very reasonable considering that the two analyses were conducted using two different samples. Lab 1 used XRF for all analyses except Mn which was done using DCP. Lab 2 used triple acid digestion per ASTM D 3683, followed by the Induced Coupled Plasma method per ASTM D 3682.

Table 2. Chemical Analysis of Expanded Slags and Expanded Perlite

Element, wt% or ppm	Slag Lab 1	Slag Lab 2	Perlite
Sample No.	701/11	701-7	Published Data
SiO_2	52.1 %	49.12	73.8
Al_2O_3	19.2	18.34	13.9
CaO	6.03	6.52	0.9
MgO	1.06	1.04	0.1
Na_2O	0.65	0.74	4.7
K_2O	2.12	1.87	4.3
Fe_2O_3	18.2	20.28	0.9
MnO	0.08	0.12	NA
Cr_2O_3	0.14	NA	NA
TiO_2	0.93	0.93	0.1
P_2O_5	0.23	0.23	NA
H_2O	0	0	0
LOI	<1.15	0	0
Total	99.74	99.7	98.7
Ba	540 ppm	NA	<1000 ppm
Nb	30 ppm	NA	
Rb	110 ppm	NA	
Sr	320 ppm	NA	
Y	30 ppm	NA	30 ppm
Zr	220 ppm	NA	

Table 3 gives the results of RCRA metals leachability using the EPA/TCLP test. The results indicate that the leachability of expanded slag is below the EP toxicity limits. Therefore, its utilization should pose no leachability problems.

Table 3. Leachate Analysis for RCRA Metals in TCLP Extract

No.	Parameter	Units	Expanded Slag	Expanded Perlite	RCRA Limit
1	pH with deionized H_2O		9.4	6.5-8.5	
2	pH with HCl		1.2	NA	
3	TCLP extract final pH		5.0		
4	Arsenic	mg/l	<0.05	<0.001	5.0
5	Barium	mg/l	0.41	<0.1	100
6	Cadmium	mg/l	<0.01	NA	1
7	Chromium	mg/l	<0.01	<0.0075	5
8	Lead	mg/l	<0.02	<0.001	5
9	Mercury	mg/l	<0.0002		0.2
10	Selenium	mg/l	<0.1		1
11	Silver	mg/l	<0.01		5

Note: Analyses for items 1-3 were done using procedure EPA 1311, item 9 was done per EPA 245.2, and all others were done using EPA 200.7.

Task 2: Preliminary Evaluation of Expanded Slag Products

In addition to holding discussions with industry experts, the work done on this task consisted of reviewing the following major items:

- Requirements for conventional materials
- General characteristics of expanded slag products
- Comparative economics of expanded slag aggregate production.

The following conclusions were drawn from the work done under this task:

- Expanded slag aggregates fall at the upper end of the ultra-lightweight classification in terms of unit weight but have considerably higher strength than expanded perlite.

- Expanded slag exhibits considerably lower dustiness than expanded perlite, which may make it a more

acceptable product for loose fill insulation applications. This is due to the slag expansion process which appears to trap gases as the outer surface of the slag particle begins to fuse. Perlite expansion is more rapid, leading to particle explosion which generates a higher amount of fine particles.

- Since the outer surfaces of the expanded slag are glassy, it may need to be crushed to open up its porous structure before it can be used in agricultural applications.

- Major costs for the production of expanded perlite are represented by the cost of the perlite ore ($40/ton) and its transportation to the Illinois area (also $40/ton). In comparison, slag feed materials are expected to be available at no cost and will be generated close to midwestern metropolitan areas where coal gasification power plants are likely to be located. However, the yield of the finished expanded products will only be nearly 50% from slag compared to perlite as the expanded unit weight of slag at 12 lb/ft^3 will is nearly double that of expanded perlite typically at 6 lb/ft^3.

Task 3: Testing of Slag in Precast Applications

Cement concrete mix designs to make specimens to test the suitability of expanded slag in precast concrete product applications are being selected in consultation with a leading block manufacturer (Harvey Cement Products, Inc.), located in the greater Chicago area. These mix designs were to be used to make sample products for testing during the second quarter of the project. However, due to severe weather conditions, which resulted in operational problems at the plant, this work could not be undertaken as scheduled.

Task 4: Testing of Expanded Slag in Construction Applications

Task 4.1 Loose Fill Insulation

Loose fill or masonry fill insulation is installed by filling the cavities in exterior and interior hollow masonry units to improve insulation and reduce heat loss. The requirements for this application were studied during this quarter and test work to evaluate slag was started. This application of slag was compared with expanded perlite, using the criteria given in Table 4. Expanded perlite is surface treated for water repellency and to reduce its dustiness prior to this

application. However, testing with slag was undertaken without any pretreatment.

Table 4. Expanded Slag as a Substitute for Loose Fill Insulation (ASTM C 549)

Parameter	Test Method	ASTM C 549*	Expanded Slag	Expanded Perlite
Particle size		95% minus 4 mesh		
Unit weight, lb/ft^3	ASTM C 520	2-11	>12.0	<6.0
Water repellency	PI 303-85	175	240	175
H_2O absorption, wt% in 14 days		1.0	in progress	
Thermal Conductivity**	C 177			
7.5 lb/ft^3		0.45	-	
12.0 lb/ft^3		1.5		
20.0 lb/ft^3		-		
30.0 lb/ft^3		-	1.34	
Surface burning characteristics	E 84			
Dustiness		85	NA	

* For untreated expanded product (perlite).
** For 7.4-11.0 lb/ft^3 unit weight material of 1" thickness at 20°C, in $Btu/h\text{-}ft^2\text{-}°F$.
NA: Not analyzed or not available.

The following tests are in progress:

- Moisture absorption on 12 lb/ft^3 sample
- Specific heat on two samples (12 and 20 lb/ft^3)
- Thermal conductivity on three samples (12, 20 and 30 lb/ft^3)
- Surface burning characteristics
- Free and combined moisture on 12 lb/ft^3 sample.

The performance of slag was compared with that of perlite for this application. Expanded slag exhibits considerably lower dustiness than expanded perlite, which will make it a more acceptable product for loose fill insulation applications. Also, its water repellency (Table 4) is considerably higher than that of perlite. However, since its unit weight is double that of perlite, double the amount of expanded slag

will be needed to fill the same volume. Our present assessment is that though the expanded slag will technically prove to be an acceptable material for this application, in order to achieve acceptance its delivered costs must be comparable to those of perlite on a volumetric basis.

Task 4.2 Lightweight Insulating Concrete and Precast Products

Expanded slag is being evaluated as insulating concrete in two steps: (i) laboratory evaluation and (ii) in a manufacturing environment. This approach requires fewer samples and is thus more economical. Laboratory tests were conducted with the expanded slag to evaluate its compressive strength and thermal conductivity. Table 5 presents the typical physical properties for perlite concrete which will be confirmed for slag. Three batches of cement concrete mixes containing a 1:5 cement-to-aggregate ratio were made using the following mix design:

- Slag and sand with low water content
- Slag and sand with high water content
- Slag and crushed slag with high water content

These mixes were cast into 2" cube specimens to test their compressive strength at 7-, 14-, and 28-day intervals. The results will provide preliminary data needed to develop mix designs for both precast concrete products and insulating concrete. One of the specimens will also be used to test the insulating properties of slag concrete. These tests will assist in establishing the suitable quantity of water needed to make insulating concretes using slag. The results will be available in the following quarter.

Table 5. Typical Physical Properties of Insulating Concrete

Mix Ratio, Cement/Aggregate	Unit Weight lb/ft^3 (dry)	Compressive strength, psi (min)	Thermal Conductivity
1:4	36	300	0.83
1:5	30.5	200	0.71
1:6	27	125	0.58
1:8	22	80	0.46

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions may be drawn based on the work conducted in this quarter:

- The lowest unit weight obtained for expanded slag (12 lb/ft^3) is at the upper end of the range typically measured for expanded perlite (5-12 lb/ft^3). However, the compaction resistance of the expanded slag is 2-8 times higher than that of expanded perlite, which could make lightweight slag products superior from a construction viewpoint.

- Expanded slag may be used as a substitute for expanded perlite in loose fill insulation applications. While its unit weight is twice that of perlite, it is considerably less dusty and has better water repellency.

- Testing of expanded slag in the loose fill insulation application successfully demonstrated its suitability. A large batch of insulating concrete will be made with slag to test its suitability in a commercial plant based on the results of the laboratory tests.

- Concrete mix designs will be developed using slag to demonstrate its use in use in precast products (lightweight blocks and rooftiles) in the next quarter.

Based on the technical evaluation and results of the tests done to date, the most promising initial applications for expanded slag appear to be in construction applications such as loose fill insulation, insulating concrete, and precast concrete products.

PROJECT MANAGEMENT REPORT
December 1,1993 through February 28, 1994

Project Title: **LWA DEMONSTRATION APPLICATIONS USING ILLINOIS COAL GASIFICATION SLAG: PHASE II**

DOE Grant Number:	DE-FC22-92PC92521 (Yr.2)
ICCI Project Number:	93-1/4.4A-11M
Principal Investigator:	Vas Choudhry, Praxis Engineers, Inc.
Other Investigators:	Philip Steck, Harvey Cement Products, Inc.
Project Manager:	Daniel Banerjee, Illinois Clean Coal Institute

COMMENTS

Work on the project proceeded at a slower pace than planned in tasks related to utilization of expanded slag to make a large batch of precast concrete products for the following reasons:

- Need for laboratory-scale testing to finalize mix designs prior to conducting the large-scale tests was identified;
- The plant where some of test work was to be done has had production problems due to severe winter weather.

However, the laboratory testing initiated earlier in this quarter is progressing well and we expect to make up the overall project progress in the upcoming quarter.

PROJECT EXPENDITURES

Projected expenditures and estimated actual expenditures for the first two quarters ending 28 February 1994 are given in Table 1 and plotted in Figure 1. As may be seen, the total cumulative expenditure for the first two quarters is slightly lower than the budgeted amount.

PROJECT SCHEDULE

The project schedule, presented in Figure 2, indicates a project start date of September 1, 1993, and a completion date of August 30, 1994. Due to the need to conduct preliminary testing in a lab to select mix designs prior to testing the precast concrete products applications, some adjustments have been made in the completion date of major milestones. Though work on the project has been slightly behind schedule in the first two quarters, it is expected that project progress will be back on schedule by the next quarter.

Table 1
PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

Quarter*	Type of Costs	Direct Labor	Fringe Benefits	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1993 to Nov. 30, 1993	Projected	3,549	710	0	780	0	3,726	6,224	14,989
	Estimated	6,396	1,279	0	0	0	0	9,940	17,615
Sept. 1, 1993 to Feb. 28 1993	Projected	8,280	1,656	0	1,820	0	8,694	14,522	34,972
	Estimated	11,092	2,218	0	511	400	0	17,305	31,526
Sept. 1, 1993 to Nov. 30, 1993	Projected	17,744	3,549	0	3,900	0	18,630	31,118	74,941
	Estimated								
Sept. 1, 1993 to Nov. 30, 1993	Projected	23,658	4,732	0	5,200	0	24,840	41,490	99,920
	Estimated								

* Cumulative by quarter

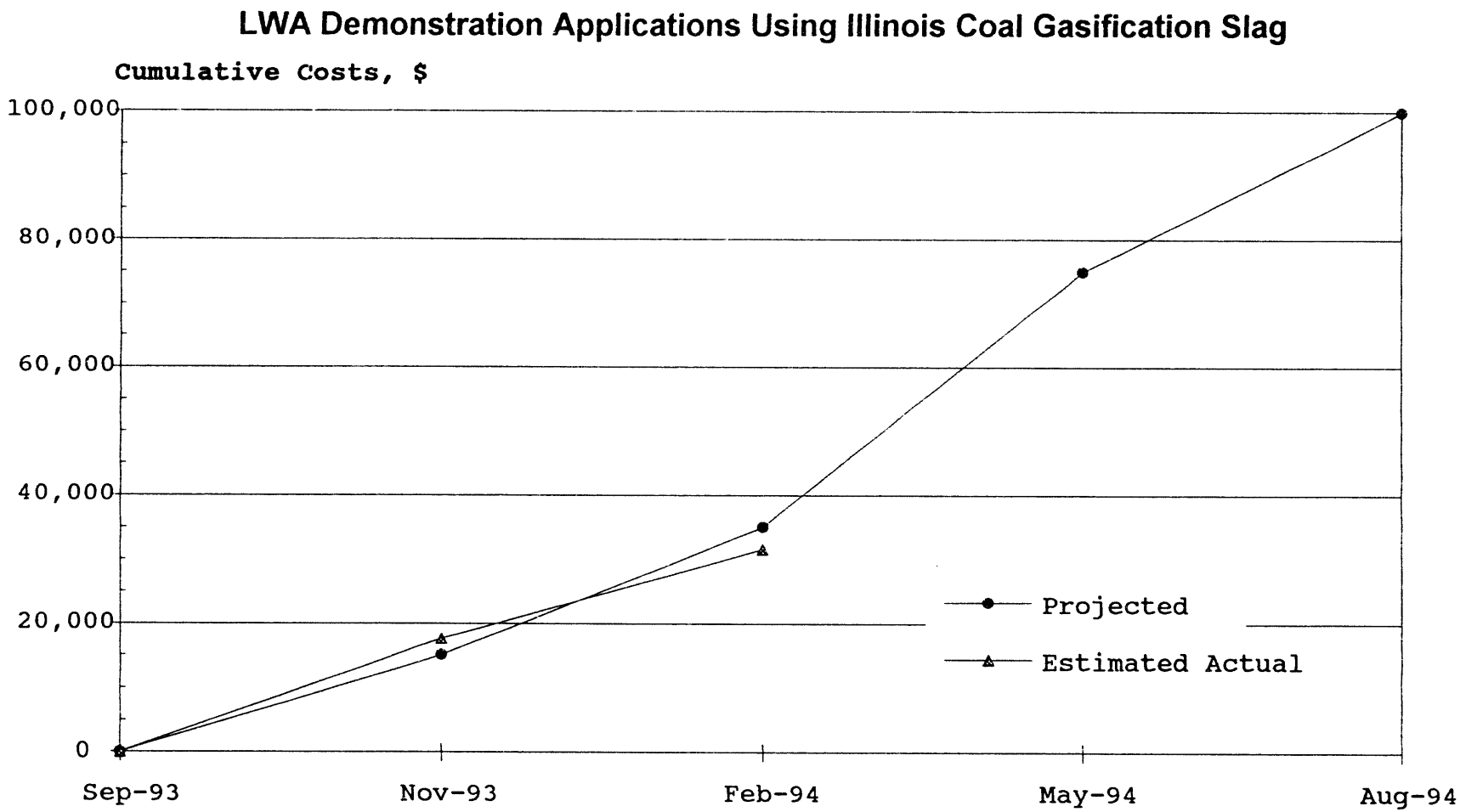

Figure 1
Costs by Quarter

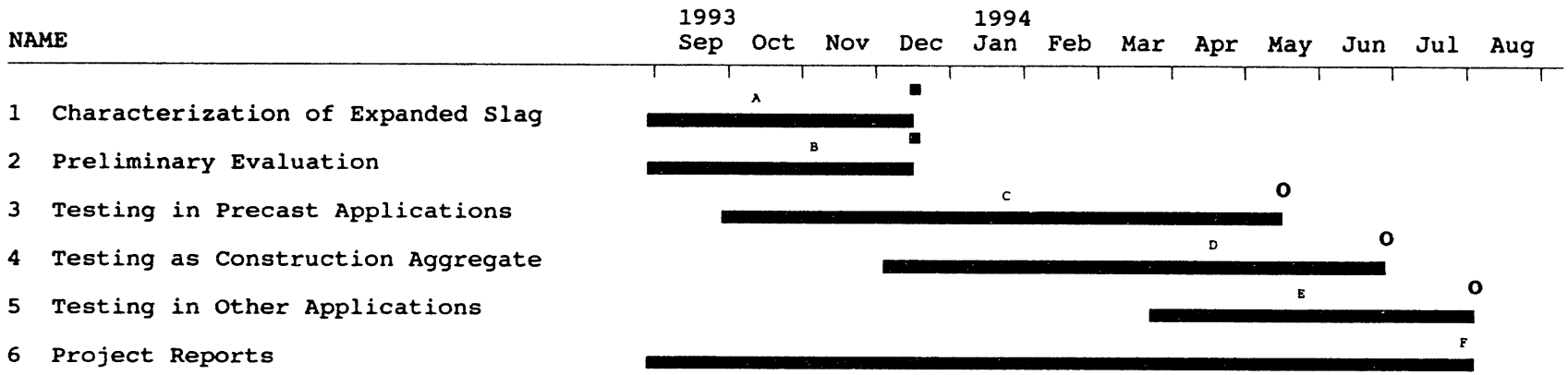

Figure 2. Schedule of Project Milestones

PROJECT MILESTONES

A. Characterization Completed
B. Applications for Expanded Slag Identified
C. Lightweight Blocks Made
D. Testing of Expanded Slag as Aggregate Completed
E. All Testing Completed
F. Final Report Submitted

■ Completed
O Revised completion date

DATE

FILMED

6/22/94

END