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TECHNICAL REPORT

September 1 through November 30, 1994

Project Title: **BRICK MANUFACTURE WITH FLY ASH FROM ILLINOIS COALS**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 3)
ICCI Project Number: 94-1/3.1A-10M
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ABSTRACT

This investigation seeks to utilize fly ash in fired-clay products such as building and patio bricks, ceramic blocks, field and sewer tile, and flower pots. This goal is accomplished by 1) one or more plant-scale, 5000-brick tests with fly ash mixed with brick clays at the 20% or higher level; 2) a laboratory-scale study to measure the firing reactions of a range of compositions of clay and fly ash mixtures; 3) a technical and economic study to evaluate the potential environmental and economic benefits of brick manufacture with fly ash. Bricks and feed materials will be tested for compliance with market specifications and for leachability of pollutants derived from fly ash. The laboratory study will combine ISGS databases, ICCI-supported characterization methods, and published information to improve predictions of the firing characteristics of Illinois fly ash and brick clay mixtures. Because identical methods are used to test clay firing and coal ash fusion, and because melting mechanisms are the same, improved coal ash fusion predictions are an expected result of this research. If successful, this project should convert an environmental problem (fly ash) into valuable products– bricks.

During this quarter we set up the manufacturing run at Colonial Brick Co., provided an expanded NEPA questionnaire for DOE, made preliminary arrangements for a larger brick manufacturing run at Marseilles Brick Co., revised our laboratory procedures for selective dissolution analysis, and began our characterization of brick clays that could be mixed with fly ash for fired-clay products.

U. S. DOE Patent Clearance is <u>NOT</u> required prior to the publication of this document.

EXECUTIVE SUMMARY

The proposed effort seeks methods for the efficient disposal or utilization of coal combustion wastes. This project precisely meets this purpose by examining the use of Illinois fly ash in the manufacture of bricks and similar fired-clay products. The project is composed of three parts: 1) one or more plant-level manufacturing runs, and 2) a set of laboratory-scale experiments designed to predict the firing properties of mixtures of a range of compositions of fly ashes with clays and shales that represent the range of compositions typical of mines in Illinois; 3) a technical and economic investigation of the potential environmental and economic benefits of brick manufacture with fly ash. The completion of these two program elements will provide strategies for maximizing the use of fly ash in bricks and related products.

The first task in this project is to obtain approximately 20 tons of fly ash from Illinois Power Co.'s Wood River Plant and ship it to Colonial Brick Company in Cayuga, Indiana, where in-plant tests of mixtures of Colonial's brick clays and I.P.'s fly ash will be made at the 20% or higher level. Colonial Brick will add progressively higher levels of fly ash until the bricks approach established specifications on water absorption. A single plant-scale run will probably be 5,000-10,000 bricks with fly ash, with pre- and post-fly ash baseline runs of about 10,000 bricks. A parallel part of the first task is to characterize the chemical and mineralogical content of the feed materials and to test the fired bricks by conventional procedures. A series of leaching tests also will be conducted on the feed fly ash, clays, and bricks. Procedures employed in previous ICCI studies will be used in the leaching tests of raw materials and bricks ([1]Dreher, Roy, and Steele, 1993). Chemical analyses of the feed materials will be by conventional methods. Methods developed in current investigations for ICCI will be used for mineralogical characterization ([2]Kruse et al., 1994; [3]Moore, Dreher, and Hughes, 1993).

In general, fly ash has a composition similar to raw materials used in brick manufacture. However, some fly ashes contain amounts of Ca^{2+} (from calcite) and Fe (from pyrite and marcasite) that would be considered too high by many manufacturers. Fairly high levels of each of these constituents can be accommodated, if special procedures are used. High levels of Ca^{2+} can be corrected for by adding water in the cool-down part of the firing cycle. This was the method by which bricks known as "Chicago Commons" were manufactured. Both the color and lower melting point caused by high levels of Fe are best adjusted for by increasing the quartz and/or kaolinite content of the clay/shale. If problems occur in the manufacturing run, we will resolve those in consultation with Colonial Brick Company.

The major goal of the first task is to make a realistic test under manufacturing conditions and detect and solve problems that might occur during scale-up at other sites. If we can make suitable arrangements, one or more plant-scale tests will be proposed for the second year of this investigation. Furthermore, if Colonial Brick is satisfied with the test run, they are willing to make arrangements for permanent use of fly ash in their plant.

The second task in this project is to attempt to improve the accuracy of methods that predict the firing characteristics of fired-clay raw materials. Part of the uncertainty about the exact level of fly ash that will be used at Colonial in the manufacturing test is the result of inadequate methods of prediction of firing behavior. Improving the prediction of the firing behavior of fly ash-clay mixtures requires a set of working and practical relationships (predictive tools) that takes into account the melting properties of each of the major minerals in the feed material. The preferred materials for these fired-clay products occur as underclays and roof shales associated with coals and contain variable amounts of three basic groups of minerals. These groups are 1) relatively low-melting-point illite, mixed-layered illite/smectite (I/S), and chlorite; 2) refractory kaolinite and mixed-layered kaolinite/expandables (K/E); and 3) somewhat refractory quartz. Common red-firing roof shales generally contain nearly ideal levels of groups 1 and 2, and adequate firing characteristics are obtained by blending clay-rich shale zones with sandier, quartz-rich zones. It is worth noting that fly ash will act as a sandier additive in Colonial's operation. If the manufacturer needs lighter color, greater strength, and/or increased refractoriness, a kaolinitic underclay (fireclay) can be blended with zones from the shale. The individual minerals within each of the three mineralogical groups are similar enough that the three groups can be used in a simplified model. Similarly, fly ash is made up of burned equivalents of these three mineral groups, and it should be possible to characterize the firing reactions of fly ash and fly ash-brick clay mixtures with similar simplifications.

The approach for task 2 will be to obtain from the ISGS reference collection clays that represent the range of compositional variation that is typical of fired-clay raw materials in Illinois. A set of Illinois fly ash samples will be selected to represent the range of composition of these materials. These samples will be fully characterized, and firing tests will be conducted to obtain data that describe the firing behavior of fly ashes, brick clays, and their mixtures.

Near the end of the project, existing information on brick clays and fly ash will be integrated with the results of this study to suggest solutions to problems that might inhibit use of fly ash in fired-clay products, to outline manufacturing and market strategies that might increase the use of fly ash in bricks, and to describe the environmental impact of using fly ash in these products. Methods to predict the firing characteristics of clays and fly ash, and of ash fusion of coals will be made available to interested parties. A technical and economic evaluation will be made of potential benefits of the use of fly ash in fired-clay products, both in economic and environmental terms. If the initial promise of this project is realized, we expect to be able to recycle large amounts of fly ash to valuable marketable fired-clay products such as bricks.

During the first quarter of this project, we set up the plant-scale manufacturing run at Colonial Brick Company. Based on recommendations from Illinois Power Company, fly ash will be obtained from their Wood River Plant, rather than the Vermillion Plant that we originally proposed. We have begun our characterization of brick clays available at Colonial Brick and we will soon begin constructing a data base of the compositions of brick clays and fly ashes that are available near existing brick companies. We have had initial discussions with Marseilles Brick Company about a plant-scale test for the second year of this project. Because they have problems with SO_2 emissions with their fireclays, we will include an evaluation of the potential of fly ash to absorb part of the SO_2 during firing of the brick.

We have recently reviewed and revised the protocols from the characterization studies for coal combustion wastes and minerals in coals that were part of ICCI's projects last year. We plan to complete the standard operating procedures for these analyses early in the next quarter. The PI has begun a series of ceramic tests for another project that may yield better measures of firing performance. Later in this first project year, these possible improvements will be discussed in detail.

The only difficulty in project startup has been the uncertainty about whether Colonial Brick Company required an Environmental Questionnaire, and, if they did, how it would be filled out and who would sign it. The PI has resubmitted the revised and longer questionnaire, as requested by Robert Dolence of DOE. In case we needed it, a blank signature page was sent to Mr. Swartz at Colonial Brick Company. Otherwise, all personnel are in place, and the project is preceding as planned.

[1]Dreher, G. B., W. R. Roy, and J. D. Steele. 1993. Geochemistry of FBC waste-coal slurry solid mixtures, Final Technical Report to the Illinois Clean Coal Institute, September 1, 1992 through August 31, 1993, 26 pp. and Appendix, 10 pp.

[2]Kruse, C.W., R.E. Hughes, D.M. Moore, R.D. Harvey, and J. Xu. 1994. Illinois Basin Coal Sample Program, Final Technical Report to the Illinois Coal Development Board, Center for Research on Sulfur in Coal, Carterville, IL;

[3]Moore, D. M., G. B. Dreher, and R. E. Hughes. 1993. New procedure for x-ray diffraction characterization of flue gas desulfurization (FGD) and fluidized bed combustion (FBC) by-products. Project funded by the Coal Combustion Residues Management Program, Carbondale, IL.

OBJECTIVES

The primary goal of the proposed investigation is to test the use of fly ash in fired-clay products such as bricks. This goal is achieved by three tasks, which have as their objectives:

1. the manufacture under normal plant-scale conditions of bricks with up to 20% or more of fly ash;
2. the measurement of the firing characteristics of the known compositional extremes of Illinois fly ashes and brick clays and shales. The derivation of practical correlations to predict the firing characteristics of any mixture of those materials. And finally, the optimization of mixtures of brick clays with typical fly ashes;
3. the integration of results of goals 1 and 2 with engineering and market assessments to evaluate the feasibility of large-scale utilization of fly ash in fired-clay products;
4. the creation of methods that more accurately predict the firing behavior of brick clays, fly ashes, and their mixtures, and that may improve predictions of ash fusion temperature of coals.

INTRODUCTION AND BACKGROUND

The large amounts of fly ash that are produced during the burning of Illinois coals represent a continuing disposal problem and a disincentive to increased consumption of those coals. If significant amounts of fly ash could be used in the manufacture of fired-clay products such as brick, this disposal problem would be eliminated and a valuable construction product would be created. Furthermore, the clay minerals in coals are fired during burning, and the energy for this firing is "saved" during brick manufacture. Manufacturers of fired-clay products also would reduce mining costs and clay use in direct proportion to the amount of fly ash used in their products. Because this project addresses the needs of industry at both the laboratory- and plant-scale levels, we believe the results can be more easily transferred to the private sector and that the time required for application of those results will be minimized.

Better methods of predicting the firing behavior of bricks and related products are a second important aspect of the proposed investigation. Although general principles guiding the selection of raw materials for fired-clay products have been known for many years (Grim, 1962; Burst and Hughes, 1994), the complexity of the firing reactions suggests the need for improved methods (Hughes, 1993). This need is emphasized by the proposed work at Colonial Brick Company. Because we lack adequate methods of prediction, we must resort to trial-and-error methods for our first plant-scale test. Completion of task 2 of this project will make it possible for us to analyze a ceramic producer's raw materials and locally available fly ashes, and suggest optimum levels of fly ash that could be employed. The large amount of background information at the ISGS and the sophistication of computer programs now available make possible a significant improvement in methods needed to evaluate all the compositions of fly ash, shale, and underclay that might be encountered.

Improved methods of predicting coal ash fusion temperature are a final important outcome that is expected from this study. These improvements should make it easier for consumers to use Illinois coals and should benefit our producers accordingly. If successful, the results obtained from this project should lead to an attractive solution, from an environmental and economic standpoint, to recycle fly ash to a high-value marketable product.

The ISGS has a long history of research related to coal and fired-clay products. The utilization of coal combustion wastes was the subject of recent studies by the Principle

Investigator (Hughes and DeMaris, 1992). Efforts to find better raw materials and improve the manufacture of fired-clay products have taken place over the last six decades at the Survey. Relevant parts of these efforts are summarized in Hughes and Bargh (1982), Hughes (1983), Hughes, DeMaris, and White (1983), and Hughes (1993). Slonaker (1977) showed that acceptable bricks were produced from feeds of 72% fly ash, 25% bottom ash, and 3% sodium silicate. A general discussion of the properties of fly ash that are important to its use in fired-clay products can be found in Kurgan, Balestrino, and Daley (1984). They report a fairly high alkalinity for fly ash from this region, and this could improve dispersion of the clay body during forming of bricks.

The development and use of leaching tests for the measurement of environmental impacts of coal combustion residues has been conducted by one of the Investigators (Dreher, Roy, and Steele, 1993). The PI and another Investigator have recently developed mineralogical characterization methods for the IBSP samples and coal combustion wastes, respectively (Kruse et al., 1994; Moore, Dreher, and Hughes, 1993). Mineralogical characterization methods for clays and shales are described in Hughes and Warren (1989) and Moore and Reynolds (1989). During the past three years, the PI also has carried out extensive research in ceramic clay products that are closely related to bricks and similar fired-clay products, and he has extensive experience in the clay processing industry. The capabilities of the ISGS in mineral process engineering and technical-economic studies are illustrated in several projects.

EXPERIMENTAL PROCEDURES

Subtask 1.1. Brick manufacturing runs. Illinois Power Company will provide a minimum of about 20 tons of dry fly ash from one of its power plants, preferably the Wood River station. Colonial Brick Company will plan a manufacturing run that will make about 5,000-20,000 bricks without fly ash and a similar number with a 15-30% fly ash-shale mixture. A single previous run with fly ash indicated that 20% fly ash additions resulted in bricks that were within standard specifications. A batch of bricks of each of the two compositions will be fired side-by-side in the kiln and tested. If the bricks remain well within standard specifications, a higher level of fly ash will be tested. If unexpected problems occur at 20% fly ash, a slightly lower level will be tested with the same experimental approach. This subtask will be completed during the second quarter of the first year of this investigation. Upon completion of the first runs, the standard properties of the bricks will be evaluated. If satisfactory results are obtained, additional tests will be planned for the second year of the study. Also, a major effort will be made to arrange additional manufacturing tests at other brick companies in Illinois.

Subtask 1.2. Standard specification tests. This subtask will be carried out by Colonial Brick and measures the degree of conformance of the manufactured bricks with standard market specifications. Samples with and without fly ash will be taken during firing to provide a measure of "clearing" or time required to completely oxidize the core of the bricks. Water absorption tests will be carried out on the fired products, and color will be described by comparing bricks with and without fly ash additions.

Subtask 1.3. Characterization. In this subtask we will characterize chemically and mineralogically each feed material and the resulting bricks. The characterization will include the quantitative determination of major, minor, and trace elemental constituents, and mineralogical components. The segregation of elements between different mineral phases in the feed materials will be estimated by using a step-wise dissolution procedure in conjunction with x-ray diffraction analysis (Moore, Dreher, and Hughes, 1993).

Subtask 1.4. Leaching tests. Leach testing procedures developed by Dreher et al. (1988, 1989) will be used to determine the extent to which environmentally toxic constituents might leach from bricks to the environment. Batch extraction and wet-dry leaching experiments, in which the substrate is exposed to deionized water for a given time period, will be conducted using raw fly ash, the mixture of clay and fly ash used in the brick-making process, and crushed and whole bricks prepared with up to five fly ash-clay mixtures. Except for the analyses conducted in Subtask 1.3, each solid will be analyzed chemically and mineralogically prior to extraction and leaching experiments.

In leaching whole bricks, five faces will be protected from leaching by application of an epoxy coating. One long face of each brick will be left uncoated to simulate exposure of one face to weathering.

Batch experiments will be conducted at solution-to-solid ratios of 4:1 for periods of 3, 10, 30, 90, and 180 days for each of the solids. Each batch extractions container will be agitated periodically during the extraction period to assure adequate contact between solution and solid. At the end of each extraction period, the solution and solid phases will be separated for chemical analysis. The solids will also be analyzed mineralogically. A total of up to twelve solids, that is, raw fly ash, clay, and up to five brick compositions (crushed and whole) will be tested in duplicate, for a total of up to 24 batch extractions.

Subtask 1.5. Integration. Upon completion of the manufacturing run and characterization described in subtasks 1.1 and 1.2, an evaluation will be made of the feasibility of manufacture of fired-clay products with fly ash additions. This evaluation will be used to modify possible plant-scale tests during year 2 and to focus detailed experiments planned for the research effort in Task 2.

Task 2. Predicting the Firing of Fly Ash and Brick Clay Mixtures.

Subtask 2.1. Background. This subtask seeks to assemble background information on the composition of Illinois fly ashes and on clays and shales used in fired-clay products manufacture. For fly ashes, data are needed on the range of chemical and mineralogical composition that is possible and on the plant location where these fly ashes are generated. For clays and shales, information must be collected together that describes where typical deposits occur and the relative content at each locality of the three basic raw materials used in fired-clay products in Illinois– 1) clays and shales with a red-firing or "shale-type" composition; 2) clays with refractory or "fireclay-type" compositions; and 3) sandier red-firing shales that are usually blended with shaley and refractory clays.

Subtask 2.2. Selecting samples. Based on the results of the background search, three fly ashes and three clays or shales will be selected to represent the range of composition encountered in Illinois. Fresh 50-pound samples of all six materials will be collected and stored at field moisture content.

Subtask 2.3. Characterizing samples. In this subtask, selected samples will be characterized chemically and mineralogically. The characterization will include the quantitative determination of major, minor, and trace elements and the mineralogical composition of the samples. The step-wise dissolution procedure in conjunction with x-ray diffraction analysis will also be conducted in order to estimate the segregation of elements among various mineralogical phases (Moore, Dreher, and Hughes, 1993).

Subtask 2.4. Firing tests. Determinations of the melting temperature or pyrometric cone equivalent (PCE) will be made for each of the six samples and for 30 mixtures and replicates that measure all possible combinations of the materials. Six replicate and eight standards PCE samples will be included to "calibrate" the method and measure errors. The color and integrity of each PCE sample will be described.

Subtask 2.5. Predicting firing. Using results from 2.4, factorial and regression computer software will be used to obtain equations that measure the effect of additions of each of the six materials on fired properties. The results of these computer runs also include an estimate of error, and this estimate can be used to confirm that sufficient samples were tested or that more experiments must be run.

Subtask 2.6. Programming. The equations generated in subtask 2.5 will be incorporated within one or more computer programs. These programs will be made available to interested parties.

Task 3. Integration, Evaluation, and Technology Transfer

Subtask 3.1. Integration and evaluation. This subtask will bring all the results together, evaluate them from an engineering- and market-oriented point of view, and estimate the overall feasibility of using significant amounts of fly ash in fired-clay products. This subtask will evaluate the technical, economic, and environmental aspects of recycling Illinois fly ash in bricks.

Subtask 3.2. Quarterly, interim final, and final reports. The results from all phases of this project will be brought together in a final report. This report will include a description of any computer programs that are generated.

Subtask 3.3. Technology transfer. The investigators will communicate the results of this study to interested parties in the public and private sector. This transfer of information will include presentations, publications, and visits or telephone calls with industrial representatives.

RESULTS AND DISCUSSION

During the first quarter of this project, we set up the plant-scale manufacturing run at Colonial Brick Company. Based on recommendations from Illinois Power Company, fly ash will be obtained from their Wood River Plant, rather than the Vermillion Plant that we originally proposed. We have begun our characterization of brick clays available at Colonial Brick and began constructing a data base of the compositions of brick clays and fly ashes that are available near existing brick companies. The only problems encountered in this work are related to the U. S. DOE Environmental Questionnaire that may or may not be required for this work. I refer to that problem in the Project Management Report.

We have had initial discussions with Marseilles Brick Company about a plant-scale test for the second year of this project. Because they have problems with SO_2 emissions with their fireclays, we will include an evaluation of the potential of fly ash to absorb part of the SO_2 during firing of the brick. This trial also could be designed to test some of the initial results of our optimization studies from Task 2 (to optimize mixtures of fly ashes and brick clays).

We recently reviewed and revised the protocols from the characterization studies for coal combustion wastes and minerals in coals that were part of ICCI's projects last year. We plan to complete the standard operating procedures for these analyses early in the next quarter. The PI has begun a series of ceramic tests for another project that may yield better measures of firing performance for the second year of this study. Later in this first project year, these possible improvements will be discussed in detail.

CONCLUSIONS AND RECOMMENDATIONS

It seems desirable at this time to revise the experimental protocols for dissolution analyses of fly ashes. These revisions promise to better assess the differences between fly ash samples from different sites.

Recent studies of the firing behavior of bricks and related materials suggest an expansion in the tests that are used to quantify the quality of fired-clay products. This expansion would add water adsorption and hardness to pyrometric cone equivalent (PCE) of the fired products.

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