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16. Abstract

Five fly ashes produced in Texas from sub-bituminous coals and lignites were analyzed in terms of variability and their effect on fresh properties of mortars. As these fly ashes contain significant quantities of CaO, a rapid and reliable method of determining the total CaO content was developed. Using the CaO Heat Evolution Test, the CaO content can be determined in a field laboratory in less than 10 minutes. The Texas fly ashes, although somewhat variable, exhibit excellent properties for use as a partial lime replacement in soil stabilization and as a partial portland cement replacement in concrete. Extremely strong correlations were found between many of the physical characteristics and chemical properties of the five fly ashes.

17. Key Words

Fly Ash, High Lime Fly Ash, Fly Ash Properties, Fly Ash Concrete, Fly Ash Mortar, Fly Ash Quality Control

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RESEARCH REPORT 240-1

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FOREWORD

The information contained herein was developed on Research Study 2-9-79-240 titled "Fly Ash Experimental Projects" in a cooperative research program with the Texas State Department of Highways and Public Transportation and the U.S. Department of Transportation, Federal Highway Administration.

This is the first report on this study.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the contributions of the Texas Department of Highways and Public Transportation, as exhibited by the Departmental personnel who collaborated on this cooperative research effort. In particular, Dr. Robert L. Long and Mr. Billy N. Banister of the Materials and Test Division provided direction, data, critiques, and support - all essential to the success of the research.

In addition, the five fly ash marketers in Texas made their fly ash available to us and shared data they had collected. Finally, thanks go to Mrs. Rosa Tabush and Miss Debbie Hamilton, who cheerfully typed the several drafts of the manuscript.

SUMMARY AND IMPLEMENTATION

The objective of this phase of the study was to analyze the variability of selected Texas fly ashes and develop laboratory procedures which will quickly ascertain those characteristics of fly ash important to their utilization in soil stabilization and concrete.

The scope of work involved the examination of five fly ashes produced in Texas through collection of a number of fly ash samples from each plant over a period of time (generally three months). The examination included physical and chemical characterization, development of laboratory procedures to quickly ascertain total CaO content and fineness of each fly ash, and determination of selected fresh properties of mortar made with fly ash-portland cement. The results of this examination were subjected to statistical correlation evaluation to ascertain the degree of correlation between physical and chemical characteristics measured. Major conclusions reached were:

1. The total CaO content of Texas fly ashes can be accurately and rapidly estimated <u>in the field</u> by the use of a simple test - termed the CaO Heat Evolution Test - which takes less than 10 minutes to perform.

2. The percentage retained on the No. 325 Sieve can be accurately and rapidly estimated by determining the percentage retained on the No. 200 Sieve.

3. There is a wide range of chemical compositions found in Texas fly ashes, both between different sources and - to a lesser extent - within a given source with time. This wide range makes it very important to be able to quickly estimate the total CaO content of a sample of fly ash.

4. There is a wide range in physical characteristics between different fly ashes and - to a lesser extent - within a given source with time.

5. All five fly ashes met the PAI and water requirement limits specified in ASTM C-618, indicating they all exhibit acceptable properties for use as a partial replacement for lime and for portland cement.

Based on the conclusions reached in this study the following recommendations are made concerning implementation of results.

1. Consideration should be given to specifying a <u>field</u> <u>acceptance</u> uniformity requirement for CaO content in the fly ash, based on the CaO Heat Evolution Test.

2. Consideration should be given to checking fineness of random shipments of purchased fly ash using the No. 200 Sieve.

3. Consideration should be given to "source qualifying" fly ash produced for the Texas highway market. This source qualification should be repeated on a <u>random</u> schedule from shipments of fly ash purchased for use in Texas highways.

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1. INTRODUCTION

1.1 Background

The continued high quality design, construction and maintenance of Texas highways has always been of primary importance to highway engineers and administrators. Rising construction costs coupled with the decreased buying power are spurring development of more cost effective construction methods and materials. One group of materials being given serious consideration in Texas are local fuel ashes.

Fuel ashes are the by-products of coal-burning power generation plants. They compose the residual matter remaining after a coal combustion process, and can be categorized as either a fly ash or a bottom ash. Fly ash constitutes the very fine particulate matter that escapes the combustion chamber with the flue gases (up to 90% of which will pass the No. 200 sieve). The fly ashes are extracted from the gases by various collection means and most are either stockpiled or stored in hoppers until disposal. Larger particles, on the other hand, fall to the bottom of the combustion chamber and are termed bottom ash (or slag). The main emphasis of this study has been on the fly ash.

The primary building blocks of fly ash are microscopic, generally spherical granules composed chiefly of silica, alumina, iron, and calcium oxides (Figure 1). These granules are formed when small particles of clay, pyrite and calcite from within the coal are exposed to temperatures in excess of 2700°F in the combustion chamber (1,2,3). While in the flame zone, the clay particles are transformed into glass-

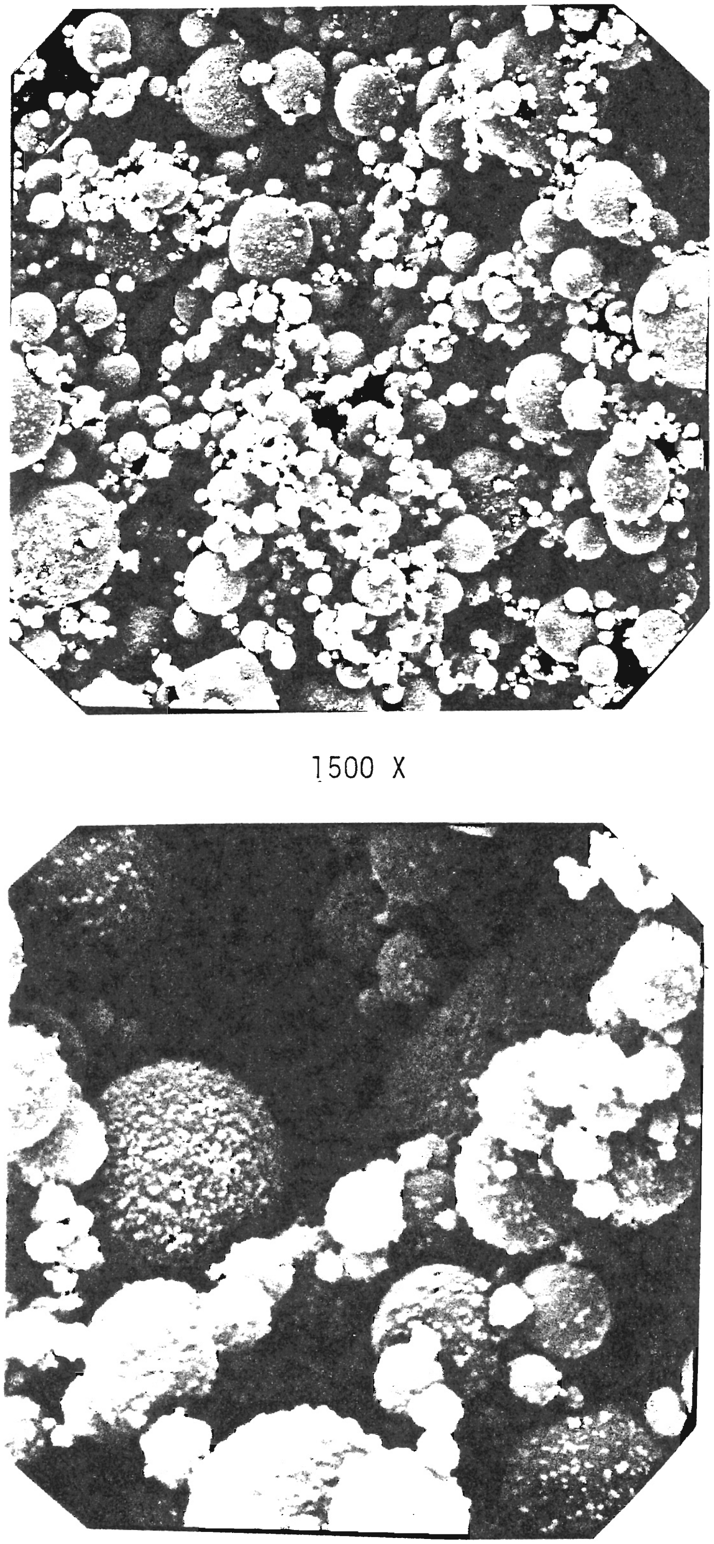

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FIGURE 1: Photomicrographs of Fly Ash From Sub-bituminous Coal.

like spheres of complex aluminates and silicates, the pyrite particles form iron oxides, and the calcite becomes calcium oxide. This transformation takes place in a molten state, and the tiny droplets formed are a mixture of the compounds mentioned above with smaller amounts of other minor compounds intermingled. The residual fly ash is - for the most part - a heterogeneous mixture of highly vitreous, spherical particles, crystaline matter, and unburned coal (1,2,3).

Fly ash is classified as a pozzolan, a siliceous/aluminous material which, in finely divided form and in the presence of moisture, will react with calcium hydroxide (or slaked lime) to form compounds possessing cementitious properties (4). The pozzolanic action takes place when the silica and alumina from fly ash react chemically with slaked lime that is present. It is important to note here that some types of fly ash, in addition to being pozzolanic as mentioned above, possess sufficient amounts of calcium silicates to exhibit cementitious properties similar to portland cement (5,6).

The utility industry produces the majority of the nation's fly ash as a by-product in electricity generation. As the coal used is a variable product, the ash produced is also highly variable in both chemical and physical properties. No two fly ash sources produce identical ashes, and furthermore, the variance in fly ash produced by the same plant can be quite noticeable. The factors which most influence individual ash properties are:

1. Coal source
2. Degree of coal pulverization
3. Boiler unit design
4. Loading and firing conditions

5. Ash collection and processing methods
6. Fly ash storage methods (1)

The above items are characteristics peculiar to each plant, and to varying degrees, they are factors in both the variability between plants and variability within a plant. By far the most influential factor of the ash produced is coal source. Faber and Styron note that, "The variable composition of coal is distinctive as it relates to the composition of the resulting fly ash produced through combustion. It is these variations that have and will continue to be of concern to the fly ash industry" (6). The variability of the coal introduced for combustion determines to a very large extent the predictability of the collected fly ash. Ashes with high variability are of low value for use in construction due to the unpredictable nature they can impart to the finished product in which they are used.

Apart from the variance that exists within each plant, there is an overall broad range in ash composition depending on the character of the coal source. In nature, coal exists in varying grades or amounts of burnable material per unit weight. The most common grades of coal used for power generation are bituminous, sub-bituminous, and lignite. Table 1 provides a breakdown of these classifications based on their energy potential.

Bituminous coal, sometimes referred to as eastern coal, is predominantly found in eastern and north central states and is usually obtained by deep mining operations. It is characteristically higher in carbon content; therefore, both the energy potential and burning

TABLE 1. GRADES OF COAL DELIVERED TO ELECTRIC UTILITIES, 1976 (7)

	Quantity (1000 Ton)	% of Total	Average Btu/Pound
Bituminous	364,227	80.3	11,400
Sub-bituminous	67,309	14.8	9,200
Lignite	22,211	4.9	6,500

efficiency are higher with bituminous than sub-bituminous or lignite coal. Sub-bituminous and lignite coals are somewhat 'dirtier' coals generally reaped from strip mines in western and southwestern regions of the country, hence their label as western coals. They usually possess higher quantities of noncombustible mineral matter per unit weight and so yield larger amounts of ash.

The American Society of Testing and Materials (ASTM) divides fly ash into two distinct classes based on coal source--class F from bituminous coal and class C from sub-bituminous and lignite origins. It is along these lines, bituminous and sub-bituminous/lignite, that a contrast exists between the chemical compositions of the resulting ashes. To what extent these differences affect concrete and soils has yet to be fully explored.

Table 2 lists the ranges in chemical composition for bituminous and lignite ashes, determined by three separate sources. Note that bituminous ashes have comparatively lower CaO (lime) contents and higher amounts of silicate, aluminate, and ferrite than do their class C counterparts. It is for this reason that class F ashes are often termed 'low-lime', and class C 'high-lime'. It is the high-lime fly ashes from sub-bituminous and lignite sources that possess both the pozzolanic and cementitious characteristics mentioned earlier. Figure 2 shows clearly the hydration reaction of sub-bituminous fly ash exposed to water for seven days. The products of the reaction appear to be similar to those of a portland cement paste, which indicates that the presence of CaO is very influential.

One point deserves special mention at this time. The CaO (lime)

TABLE 2. Ranges in Chemical Composition of Bituminous Ashes and Lignite (Weight Percentages)

Reference	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Alkalies	C
Bituminous								
Price (<u>7</u>)	34-52	13-31	6-25	1-12	0.5-3	0-2	0-2	1-12
DOE (<u>8</u>)	25-52	14-30	4-31	3-8	0.5-3	0.3-3	0.5-9	1-19
Faber (<u>6</u>)	40-55	25-35	5-24	0.5-4	0.5-5	0.5-5	0.7-4	0.5-12
Lignite								
Price (<u>7</u>)	15-52	8-25	2-9	11-36	2-11	0.7-27	0-7	1-12
DOE (<u>8</u>)	24-43	12-21	5-14	18-41	4-10	0.7-2.5	0.5-5	1-4
Faber (<u>6</u>)	20-40	10-30	3-10	10-32	0.5-8	1-8	1-8	0.5-2

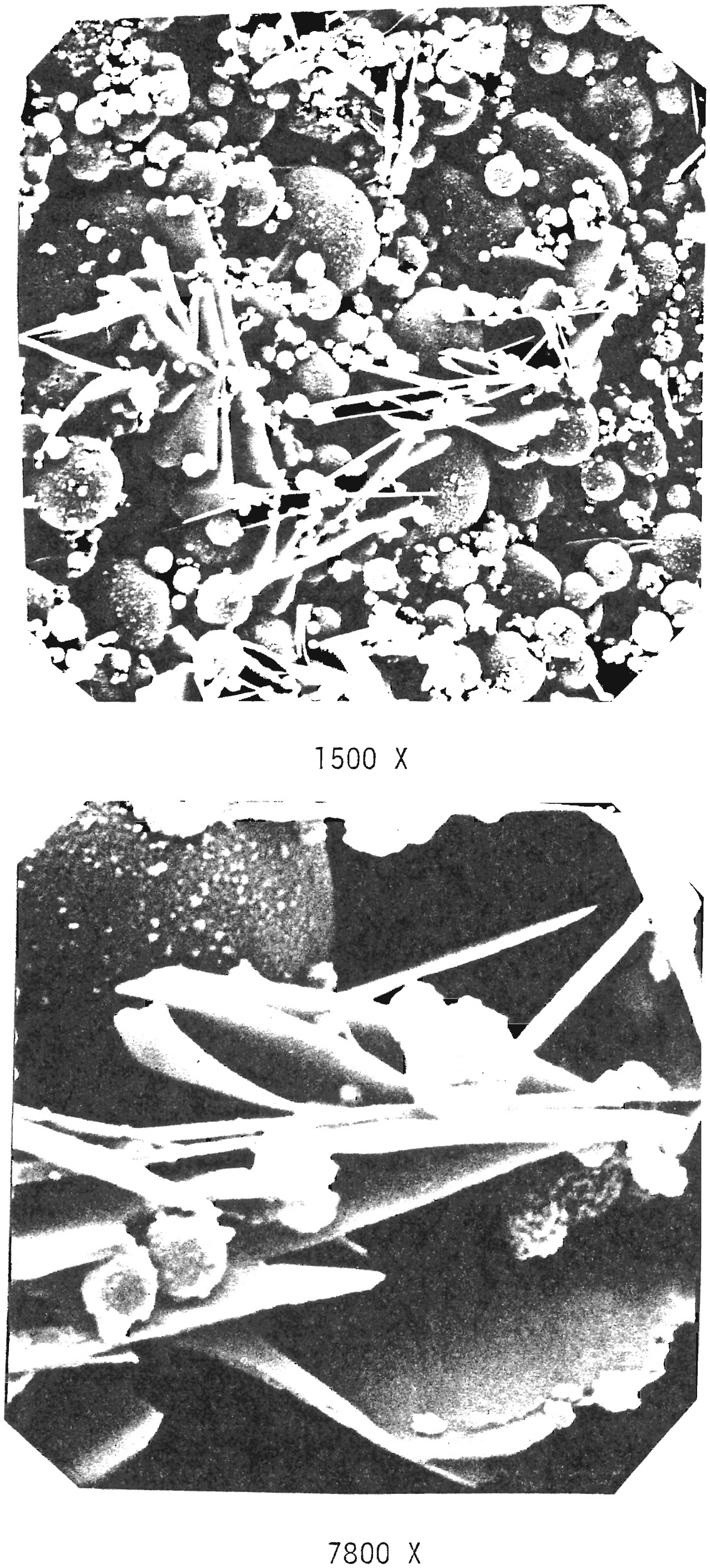

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FIGURE 2: Photomicrograph of Fly Ash From Sub-bituminous Coal Exposed to Moisture for Seven Days.

present in the fly ash is not in a free, or available, state. It is generally chemically combined with the silicates and aluminates in a manner similar to the CaO in portland cement. Thus this lime would not be readily available for use as a soil stabilizing agent (see section 1.3).

The rapidly increasing interest in Texas stems from the quantities of fly ash becoming available. In 1973, there were three coal powered units on line in Texas producing 1500 megawatts (MW) and 470,000 tons of fly ash per year. In 1979, there were 17 units generating 11,000 MW of electricity and producing over 3.2 million tons of fly ash per year. By 1983, approximately 30 units will be producing 17,000 MW of electricity and more than five million tons of fly ash per year (Figures 3-5). Thus, there is an increasing abundance of both bottom ash and fly ash, and disposal is rapidly becoming more of a problem.

Texas fly ash originates almost exclusively from western coals. In the past engineers were of the opinion that western fly ashes may be of lower quality than eastern fly ashes; however, that premise has been largely abandoned in light of successful experience. Due to the long history of eastern fly ash production, the vast majority of literature published concerning coal ash utilization has dealt almost exclusively with eastern, low-lime ashes. Only recently have class C ashes begun to gain acceptance and the need for detailed study emphasized. One of the more noticeable characteristics of western fly ashes is the large range of compositions that may be encountered. Recalling Table 2, it can be seen that class C ashes cover a broader spectrum of chemical make-ups. There are even drastic differences in the ranges of

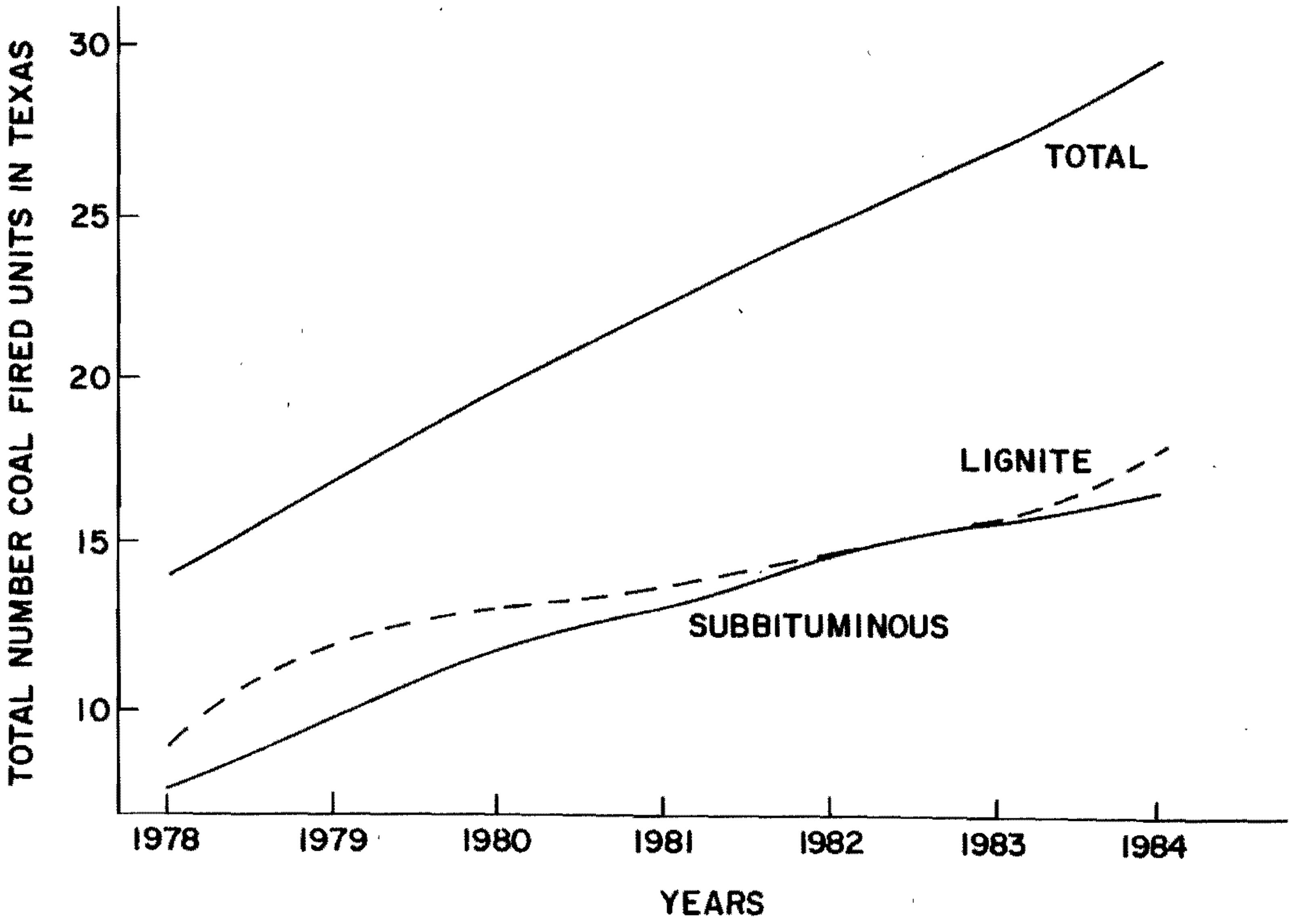

Figure 3. Number Coal Fired Units - Texas (9)

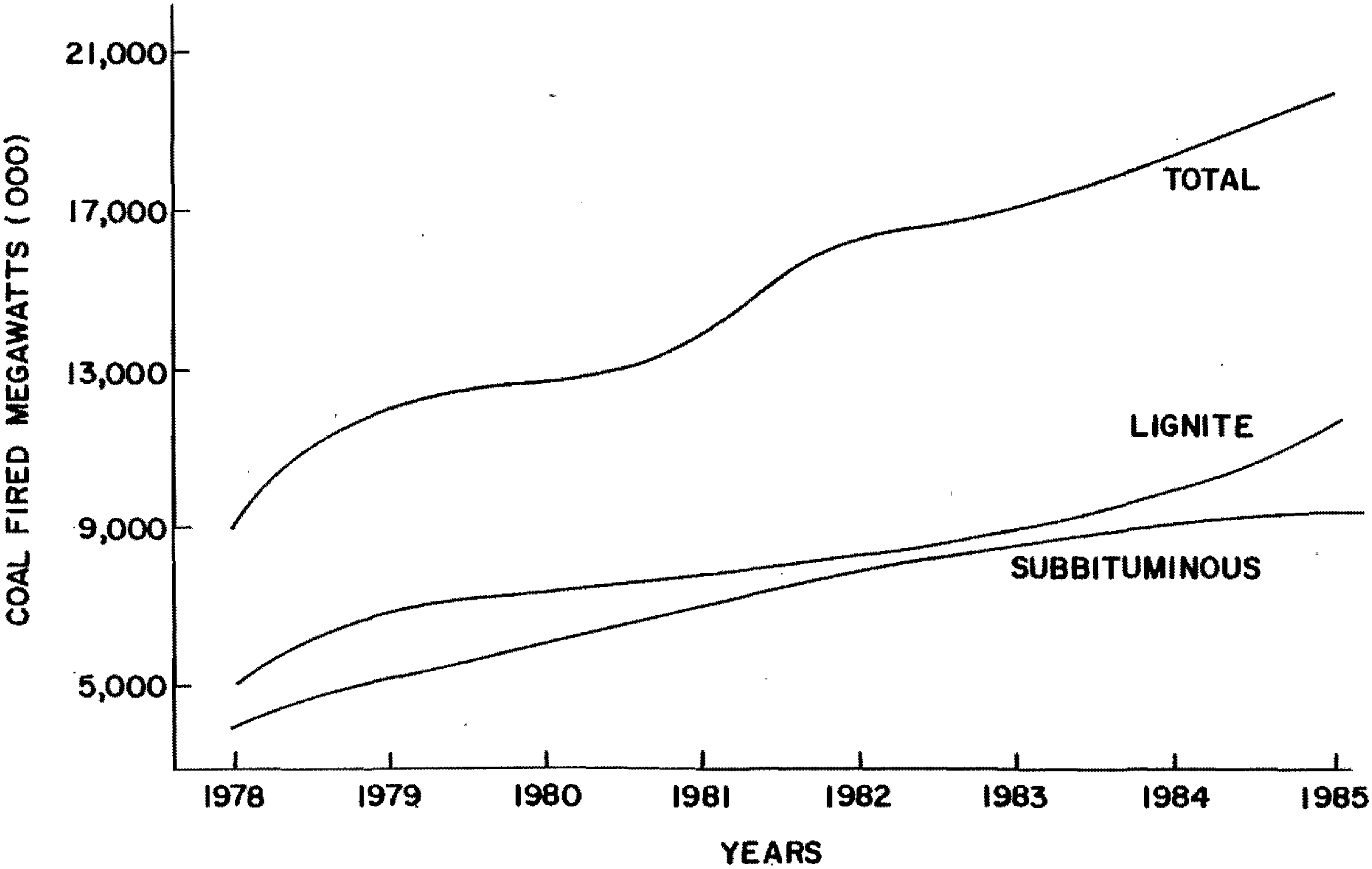

Figure 4. Coal Fired Megawatts - Texas (9)

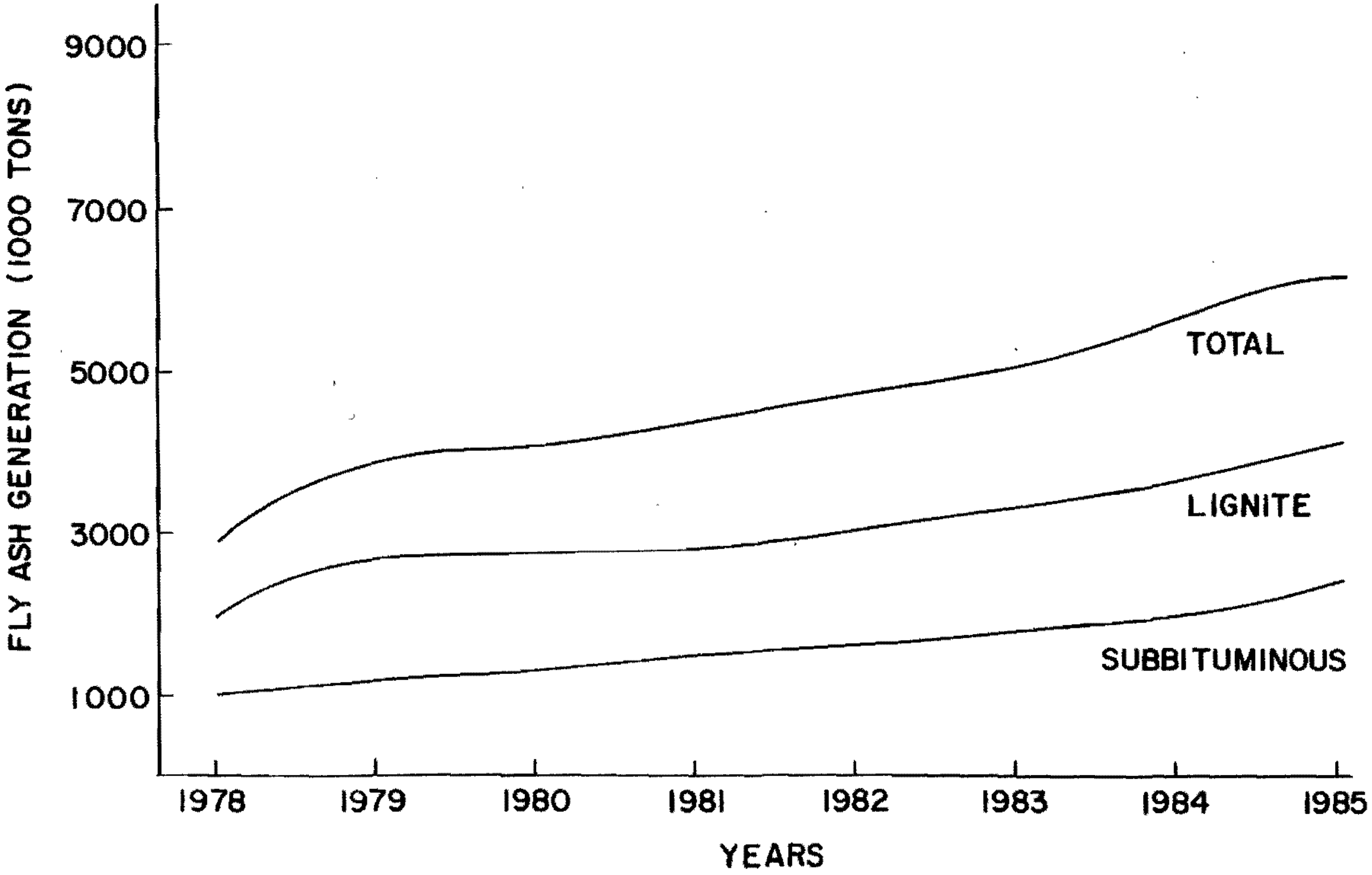

Figure 5. Fly Ash Generation - Texas (9)

composition reported by the three sources which, to some extent, indicates the newness and lack of research information that exists. Thus fly ash research is needed to analyze the variability of subbituminous and lignite ashes and to relate ash properties and variability to the behavior of end products such as concrete and stabilization that might utilize the fly ash.

1.2 Specification Development

ASTM currently has two specifications for both class F (bituminous), and class C (sub-bituminous and lignite) fly ashes. ASTM C-618 governs the use of fly ash as a mineral admixture in portland cement concrete (4) and ASTM C-593 governs the use of fly ash as a soil stabilizing agent with hydrated lime (10). Examination of these specifications reveal the items of concern deemed important by ASTM. They include fineness for both uses; and such items as loss on ignition, SO_3 content, alkali content, pozzolanic activity, uniformity in terms of specific gravity and fineness, and water content for use in concrete (ASTM C-618). The ASTM C-618 specification is by far the more stringent specification.

Magnesium Oxide (MgO), sulfur trioxide (SO_3), carbon (C), and various alkalies (Na_2O and K_2O) are all potential 'bad actors' in cement and must be limited to minimize their deleterious effects. MgO is a compound that hydrates similar to lime; however, its hydration occurs slower and can be accompanied by disruptive expansions in portland cement when present as periclase (11). Since SO_3 is added to portland cement in the form of gypsum to slow the fast-setting action of hydrating aluminates, the soluble portion of the SO_3 content of fly ash must be limited to avoid excessive delays in setting and perhaps lowering of resulting concrete strengths. The presence of alkalies also has an effect upon setting times of concretes. High alkali contents reduce the retarding action of lime and can contribute to flash setting without adequate SO_3 levels. Alkalies from within fly ash also have the potential to react with certain siliceous aggregates which can cause disruptive expansions in a finished product well after construction is complete. Interestingly, fly ash can be used as a silica flour to mitigate such alkali-aggregate reactions: however, if high quantities of alkali are present in the fly ash itself, the mitigating effect can be essentially nullified.

Unburned carbon particles, indicated by loss on ignition (LOI), contribute nothing to the hardened concrete structurally, and have been shown to demonstrate a strong affinity for air entraining agents (AEA). The effect of this AEA absorption is a significant reduction in the amount and character of the resultant air void system within the hardened concrete.

Two physical properties of fly ash that draw considerable attention are fineness and specific gravity. Fineness is controlled primarily by the efficiency of coal pulverizers. In general, finer ashes react quicker to produce slightly higher early strengths and slightly faster set times. These finer ashes are generally preferable in portland cement concrete because they tend to reduce the amount of water necessary for a given consistency (water requirement).

Some controversy exists over the significance of the specific gravity of fly ash. Several researchers have concluded this property to be of little practical use, while others claim strong correlations between specific gravity, fineness, and loss on ignition (12,13). The specific gravity is relevant to mix designs because variations significantly alter the volume proportions of a design.

Of special note is the ASTM variability limits for fineness and specific gravity which are included to provide tighter control of product uniformity.

The pozzolanic activity index (PAI) is an indication of how well the ash will react with portland cement. Review of published literature has revealed little information concerning the PAI of sub-bituminous and lignite ashes. Thus research is needed to determine the ash properties responsible for good pozzolanic activity.

A deficiency in specifications for class C fly ashes that appears to exist is the absence of a CaO content and uniformity requirement, in light of the cementitious activity associated earlier with this compound (Figure 2). The lack of such a specification probably stems from the difficulty in quickly determining CaO presence; nevertheless, the need for such a specification remains.

1.3 Use of Fly Ash in Stabilization

A promising use of fly ash in construction is in the stabilization of road subgrades and bases with fly ash (often in combination with lime or portland cement). The strength and durability of a lime-fly ash stabilized soil depends almost entirely on the quality of the resulting pozzolanic reaction and any cementitious action. Compaction, density, age, and the amounts of lime, silica, and alumina present determine to a very large degree the ultimate strength and durability delivered in a roadbed structure. The most critical physical factor influencing reactivity is particle size; however, high temperature, low percentage of carbon, and proper moisture contents will also increase the reaction speed and consequently realize a quicker strength gain.

Remember that the pozzolanic reaction involves the silicates and aluminates in the fly ash reacting with any hydrated lime [$Ca(OH)_2$] present. Since very little CaO is available in fly ash (most of it is bound up) the $Ca(OH)_2$ must be supplied from some other source - if pozzolanic activity is to be generated. Thus most designers specify some hydrated lime to be used with the fly ash. The lime provides the chemicals necessary for pozzolanic reactivity. If a clay is being stabilized, it will normally contain silicates and aluminates which can react with the hydrated lime. But lime is expensive and, hopefully, less lime can be used if fly ash is added to provide some of the silicates and aluminates. This pozzolanic reaction is slow, and in structures relying on it for strength, heavy loads must not be exerted during early life. Some states are now building lime-fly ash stabilized highways and allowing them to "cure" for several months before use. The nonreactivity of fly ash in cold weather is also a prime reason for delayed use after construction.

If, however, a load induced failure does occur, fly ash does have the unique capability of healing itself autogenously (1). This property is governed mainly by:

1) Age at failure
2) Degree of contact between cracked surfaces
3) Curing conditions (moisture, temperature, etc.)
4) Availability of the on going pozzolanic reactants.

Because of autogenous healing, lime-fly ash mixtures have been proven less susceptible to deterioration and fatigue than other materials not possessing this property. With sandy or coarse materials, the fine particles of fly ash and lime fill voids and "float" the coaser particles (greater than No. 4 sieve). With time and moisture a pozzolanic reaction occurs between the lime present and the silica/alumina particles of the fly ash. A strong matrix is formed which ultimately can develop unconfined compressive strengths as high as 3000 psi (more commonly between 500 and 1000 psi with 7 days cure at 100° F) (1). This pozzolanic reaction occurs with both class F and class C fly ashes. But with class C fly ashes, a cementitious hydration-like reaction has also been found to occur in a manner similar to portland cement. This reaction generally occurs very rapidly which necessitates quick manipulation and compaction if the full cementing action is going to be realized. Furthermore, this quick cementing action does not require hydrated lime and thus some class C fly ashes may be successfully used to stabilize granular materials without the addition of lime (much the same way you can construct a cement stabilized base with portland cement).

In clay soils, lime is required to break down plasicity and provide for pozzolanic reations. As the soil becomes more granular-like and workable, the pozzolanic reaction begins between the hydrated lime and the silicates and aluminates from both the clay and the fly ash. The degree to which lime-fly ash mixtures can improve a soil is a function of the mineralogy and fineness present.

In research by the Texas State Department of Highways and Public Transportation (SDHPT), seven soils and two marginal base materials were tested with respect to lime-fly ash ratios, and stabilization with lime-fly ash was concluded a usable contruction procedure (14). In 1959, a lime-fly ash-slag stabilized heavy haul road was constructed at Rockdale, Texas using lignite ash. The structure has delivered excellent performance over the past 20 years while being subjected to loads upwards of 70,000 pounds. The Texas study concluded that highways constructed with lime-fly ash stabilization have provided excellent performance, and materials stabilized with lime-fly ash possess greater strengths than the same materials stabilized with either lime or fly ash alone. This report outlined lab procedures for investigating strength characteristics of fly ash mixtures and recommended specifications for lime-fly ash treatment of materials in-place. It was finally suggested that all proposed highway construction and maintenance projects within economic haul distances of lignite based power plants be seriously considered for lime-fly ash stabilization.

A Federal Highway Administration (FHWA) investigation recently found lime-fly ash mixtures superior to their straight lime counterparts when designed to be economically competetive (1). In addition, they presented guidelines outlining layer thickness design and lime-fly ash stabilization conditions.

1.4. Use of Fly Ash as an Admixture in Portland Cement Concrete

The inclusion of class F fly ash in portland cement concrete has been shown to improve both fresh and hardened properties of the concrete at reduced cost. In general, the benefits obtained from class C fly ash are very similar to those witnessed with class F usage; however, due to the different compositions, some variations can be expected.

The first significant use of fly ash (which was class F) probably occurred in the 1940's when the Bureau of Reclamation started specifying fly ash as a partial replacement for portland cement in mass concrete to reduce the heat of hydration. Because the pozzolanic activity occurs at a much slower rate than cement hydration, the heat produced is dissipated over a longer period of time. In addition, the reduction in heat generation is largely responsible for the lower amounts of thermal cracking found in fly ash concrete.

The slower nature of the pozzolanic activity of class F fly ashes usually delays the initial and final sets, and ultimately lowers the rate of early strength gain when compared to a 100 percent portland cement concrete. However, the set times usually remain within specifications, and it is quite common for the sustained strength gaining characteristics of fly ash-portland cement concrete to produce higher long term strengths than comparable portland cement mixes (Figure 6) (15). Because of this phenomenon, class F fly ash has often been specified for high strength concrete. During construction of the Water Tower Place in Chicago, the world's tallest reinforced concrete building, high strength fly ash/portland cement concrete was specified, and the Material Service Corps noted that the high strengths obtained were <u>impossible</u> without the addition of fly ash (<u>16</u>).

Class C fly ash has also been noted for its contribution to high strengths in concrete. Gifford Hill and Company, Inc., a marketer of class C fly ash for use in concrete, noted that "fly ash is a must in

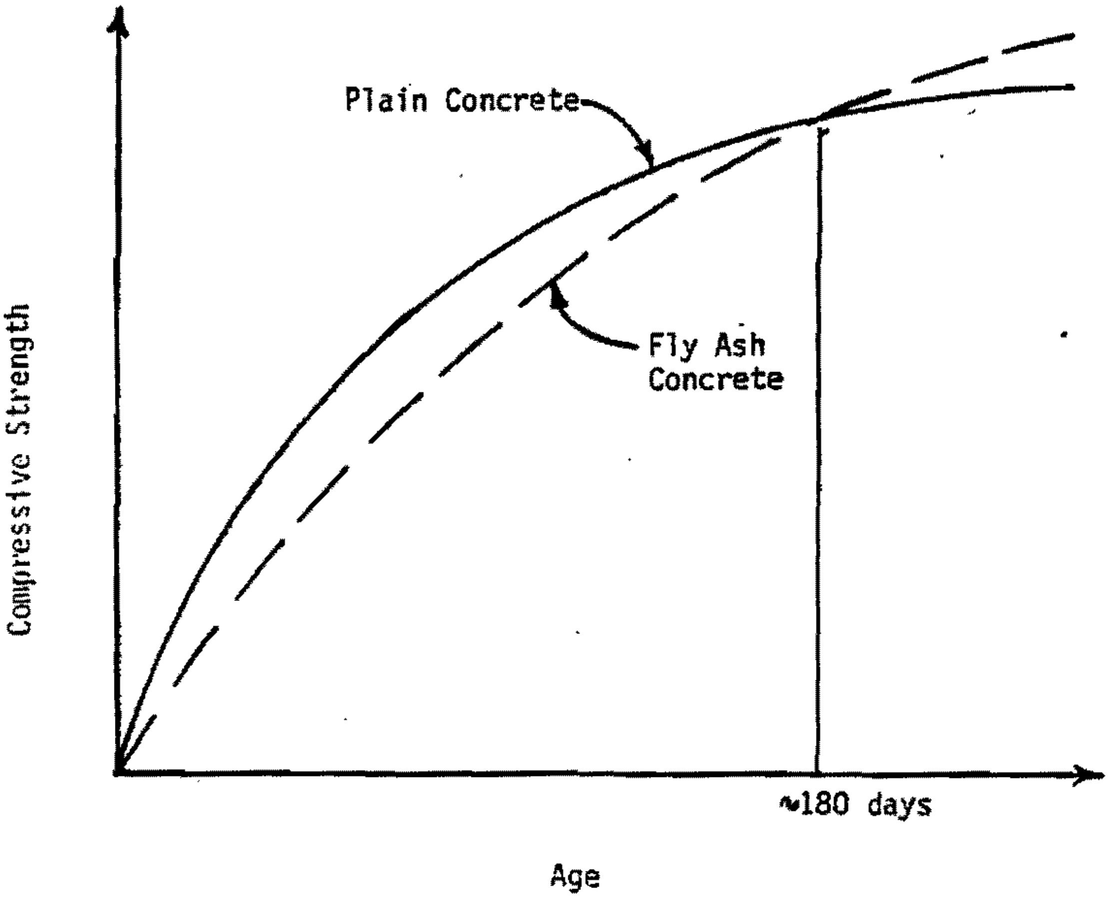

Figure 6. Comparison of strength development of concrete with and without fly ash (shematic) (from 15).

high strength concrete" (17). In addition, a North Dakota study has found that 15 percent weight replacement of portland cement with lignite fly ash yielded compressive strength results similar to 100 percent portland cement concrete.

Several advantages of both class C and class F fly ash/portland cement concrete are seen while the material is still plastic. The spherical particles of the ash serve to lubricate the mix. Better workability, placeability, finishability, and pumpability all result. The same lubricating effect often makes it possible to increase coarse aggregate contents and reduce water contents for mixes of equal slump. Larger amounts of coarse aggregate raise the cementing efficiency while reduction in water lowers water-cement ratios causing strength to increase.

Two interrelated benefits of fly ash-portland cement concrete are decreased permeability and decreased leaching. The pozzolanic activity previously mentioned is a reaction between the silica and alumina particles of the fly ash and the lime that, in this case, is freed by cement hydration. This reaction combines the potentially leacheable lime into insoluble calcium silicates and aluminates that are quite stable (the pozzolanic action), filling voids and reducing permeability (18). The results of this occurrence are improved resistance to chemical attack and, according to some researchers, reduced freeze-thaw damage (12).

Some controversy exists surrounding the effects of fly ash in concrete subject to freezing and thawing. Early research indicated that bituminous fly ash reduced freeze-thaw resistance; however, these efforts often failed to account for the slower strength gains and higher sensitivity to air entrainment found in fly ash concrete in which the fly ash had significant quantities of carbon (high LOI). Research sponsored by the Department of Energy has shown that fly ash mixes of equal strength and entrained air content demonstrated comparable freeze-thaw performance compared to 100% portland cement concrete (8). The North Dakota study concluded that the freeze thaw durability of portland cement concrete made with lignite ash was excellent.

Other advantages of fly ash portland cement concrete are (12):

1. Reduced segregation and bleeding
2. Reduced drying shrinkage
3. Improved molding and forming qualities
4. Reduced alkali reactivity (Provided the alkali content of the fly ash were kept low).

A final benefit of ash utilization in concrete is its economic impact. In the recent construction of the El Paso Tower in Houston, Texas, a class C fly ash was used as a partial cement replacement for the 7500 psi concrete. Not only did the concrete achieve strengths upwards of 8700 psi, but a quarter of a million dollars was saved due to the utilization of the fly ash (17).

Some of the disadvantages observed with fly ash-portland cement concrete have been mentioned already. Low early strengths can result from the slow strength-gaining characteristics of the pozzolanic reaction. Researchers have also found that carbon from fly ash absorbs air entraining agent. This phenomenon is more prevalent with ashes from the older, less efficient power plants. The absorption of AEA can adversely affect the air void system and subsequent freeze-thaw durability, especially when high carbon contents or highly variable carbon amounts exist in a given fly ash rendering control of AEA content impossible (12). Other compounds such as SO_3, MgO, and Na_2O also occur in class C or class F fly ashes and - if not controlled - deleterious reactions may occur. Finally, the initial expense incurred to provide a batch plant with fly ash handling capability is also a definite drawback (19).

1.5 Environmental and Energy Considerations

The advantages of fly ash utilization extend beyond the factors directly associated with construction. Fly ash is a waste product produced in large quantities (57.5 million tons in 1978) (20). Disposal of this waste has and will continue to be an enviromental, engineering and economic problem.

Recently, the Environmental Protection Agency (EPA) has become interested in fly ash with respect of the National Resource Conservation and Recovery Act (NRCRA). Basically, this act mandates the use of waste materials in government sponsored jobs where (21):

1. The product is technically equivalent.
2. The product price is reasonable.
3. Competition is maintained.
4. The product is available.

Fly ash fits these criteria extremely well, so well in fact, that the EPA has taken a special interest in fly ash as a possible model for implementation of the NRCRA. The implications of this attention will be increased pressure for ash utilization.

A second promotional factor is that fly ash/portland cement concrete saves energy. Several studies have examined the potential for energy conservation through the use of fly ash in concrete and have reached the conclusion that huge quantities of energy could be conserved if ash were utilized. These studies point out that fly ash/portland cement concrete is an accepted, if not necessary, construction material throughout the rest of the world. Twenty percent in Germany, 33 percent in Russia, 60 percent in France and the Netherlands, and every pound of cement in Hungary is blended with fly ash. American utilization of fly ash in cement production is less than 2 percent (7,8). If the United States would convert just 25 percent of their annual cement production to a 20 percent fly ash blended cement (ASTM C 595 covers blended cements), the equivalent in energy of over 5.1 million barrels of oil would be conserved every year (7).

In summation, engineering, environmental, energy, and economic pressures are all playing a role in the quickening development of this material. With these forces at work, it is becoming increasingly vital for research to keep pace and provide the knowledge necessary to insure efficient and cost effective utilization.

2. OBJECTIVE AND SCOPE

The objective of this phase of the study was to analyze the variability of selected Texas fly ashes and develop laboratory procedures which will quickly ascertain those characteristics of fly ash important to their utilization in soil stabilization and concrete.

The scope of work involved the examination of five fly ashes produced in Texas through collection of a number of fly ash samples from each plant over a period of time (generally three months). The examination included physical and chemical characterization, development of laboratory procedures to quickly ascertain total CaO content and fineness of each fly ash, and determination of selected fresh properties of mortar made with fly ash-portland cement. The results of this examination were subjected to statistical correlation evaluation to ascertain the degree of correlation between physical and chemical characteristics measured.

3. LABORATORY PROGRAM

3.1 General

Fly ash samples from each of five power plants in Texas were collected at random over a 90 day period (different periods for each plant). In addition a number of fly ash samples were taken from experimental test sites being constructed in various parts of the state (using the same five fly ashes). A total of 342 samples of fly ash were collected over a year and a half. Physical characterizations to include fineness (percent passing the No. 200 and No. 325 sieve) and specific gravity, were performed on all samples. Chemical oxide analyses were performed on selected samples. Concurrently, test procedures were developed to quickly ascertain the total CaO content and the CaO value was determined on all samples. To gain an insight on the behavior of mortar using Texas fly ashes, a number of mortar batches were prepared and selected properities determined. In the following sections the results of the laboratory program are presented.

3.2 Determination of Total CaO Content

As discussed in Section 1.1, one of the distinguishing characteristics of class C Fly ash is its relatively high CaO content (values from 10 to 41% have been reported - see Table 2). Since CaO content is an important compound, the question was posed "is there a quick, accurate, way to quantitatively determine the total CaO content in a laboratory?" Fortunately, research in Poland indicated that the heat evolved from a mixture of fly ash and hydrochloric acid could be correlated to CaO content (22). Using this idea the procedure was modified and refined to the point where a good correlation was obtained between the heat evolved from a mixture of 20g of fly ash and 75 ml of 15% HCl (see Appendix A for details of the procedure). Thirty-nine (39) laboratory determinations of CaO content of various fly ashes were regressed against the resulting temperature rise and the following linear equation was developed ($R^2 = 0.88$):

$$\text{CaO} = 0.395\ (\Delta T) + 3.234$$

Where: CaO = weight % of total CaO

ΔT = heat rise in °C

The data and linear line are shown in Figure 7. Neither starting temperature nor fly ash age affected the results! The main advantage to this test is that, with inexpensive equipment, the heat evolution can be determined in less than 10 minutes, yielding a reliable estimate of total CaO content (for Texas fly ashes with 10 to around 30% CaO contents). With such a test the variability of CaO content can be determined and a purchaser of fly ash can rapidly determine if the delivered fly ash is essentially the same product he ordered!

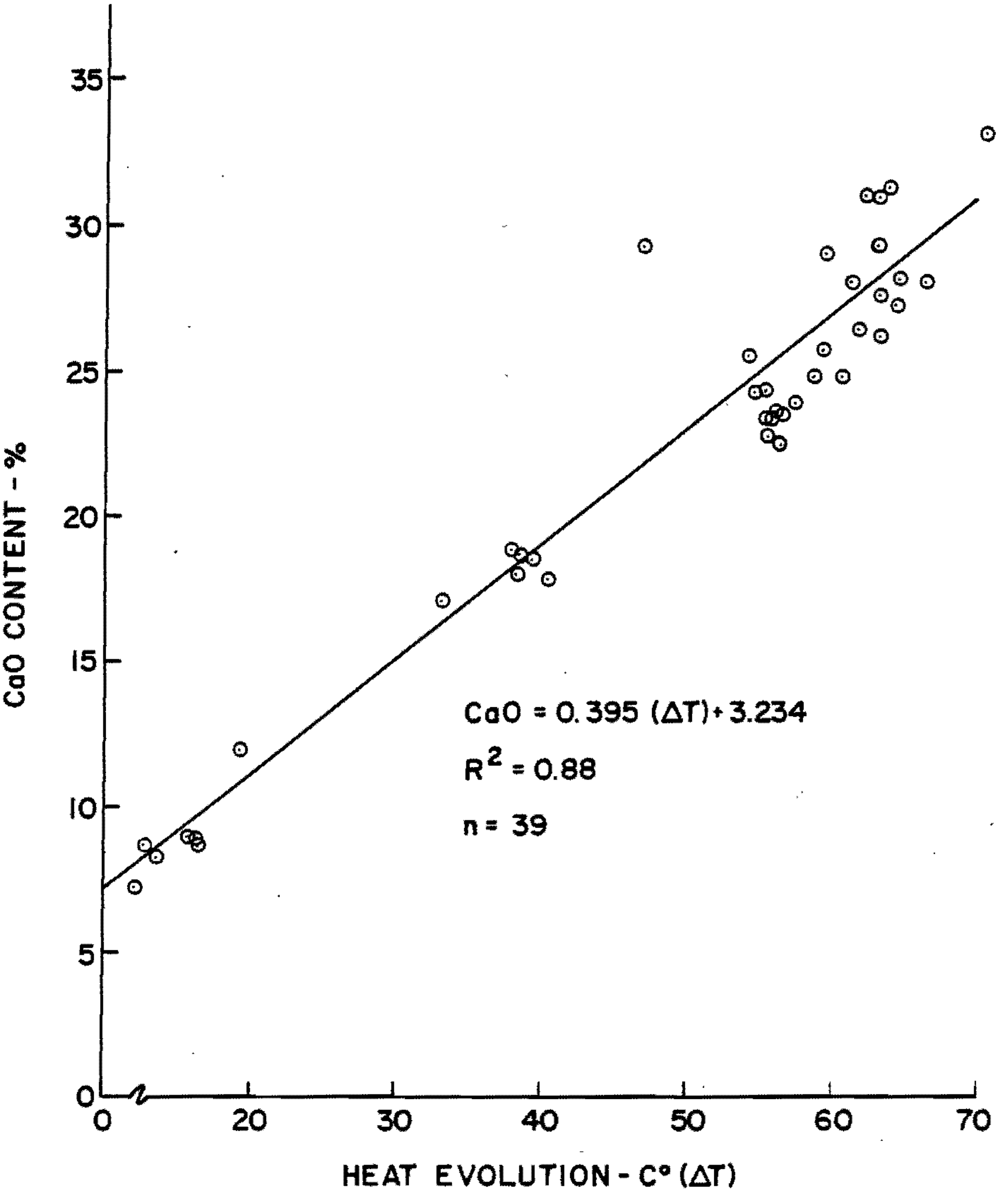

Figure 7. Calcium Oxide Content as indicated by the CaO Heat Evolution Test

To test the validity of this laboratory procedure a number of fly ash samples were evaluated by the Materials and Tests Division (File D-9), District 15, and District 18 of the Department. Excellent correlations between the various laboratories were achieved (Figure 8) indicating this test is repeatable and reliable. Thus this test procedure is recommended as a field test for rapid determination of total CaO content in Texas fly ashes.

3.3 Determination of Fineness

ASTM C 618 specifies a maximum of 34 percent retained on the No. 325 sieve. Also specified is a uniformity requirement which allows a maximum variation of 5 percentage points based on the average of the 10 preceding samples. These are excellent requirements and the test is easy to run in the laboratory. But, control of fineness in the field is another matter because the equipment necessary for this test is normally not present. Thus it was decided to see if the percent retained on the No. 200 sieve could be used as a field control test to quickly ascertain the fineness of various fly ashes. A 50 gram sample was used (see Appendix A for details). Results of this evaluation are shown in Figure 9 for all 342 test results from the five fly ashes. While each fly ash has its own characteristic relationship between the No. 200 and the No. 325 sieves, the combined equation of all values indicated a good correlation ($R^2 = 0.86$), indicating that the fineness can be reliably predicted by use of the No. 200 sieve. The resulting equation is:

$$\text{No. } 325 = 1.79\,(\text{No. } 200) + 3.03$$

$$R^2 = 0.86$$

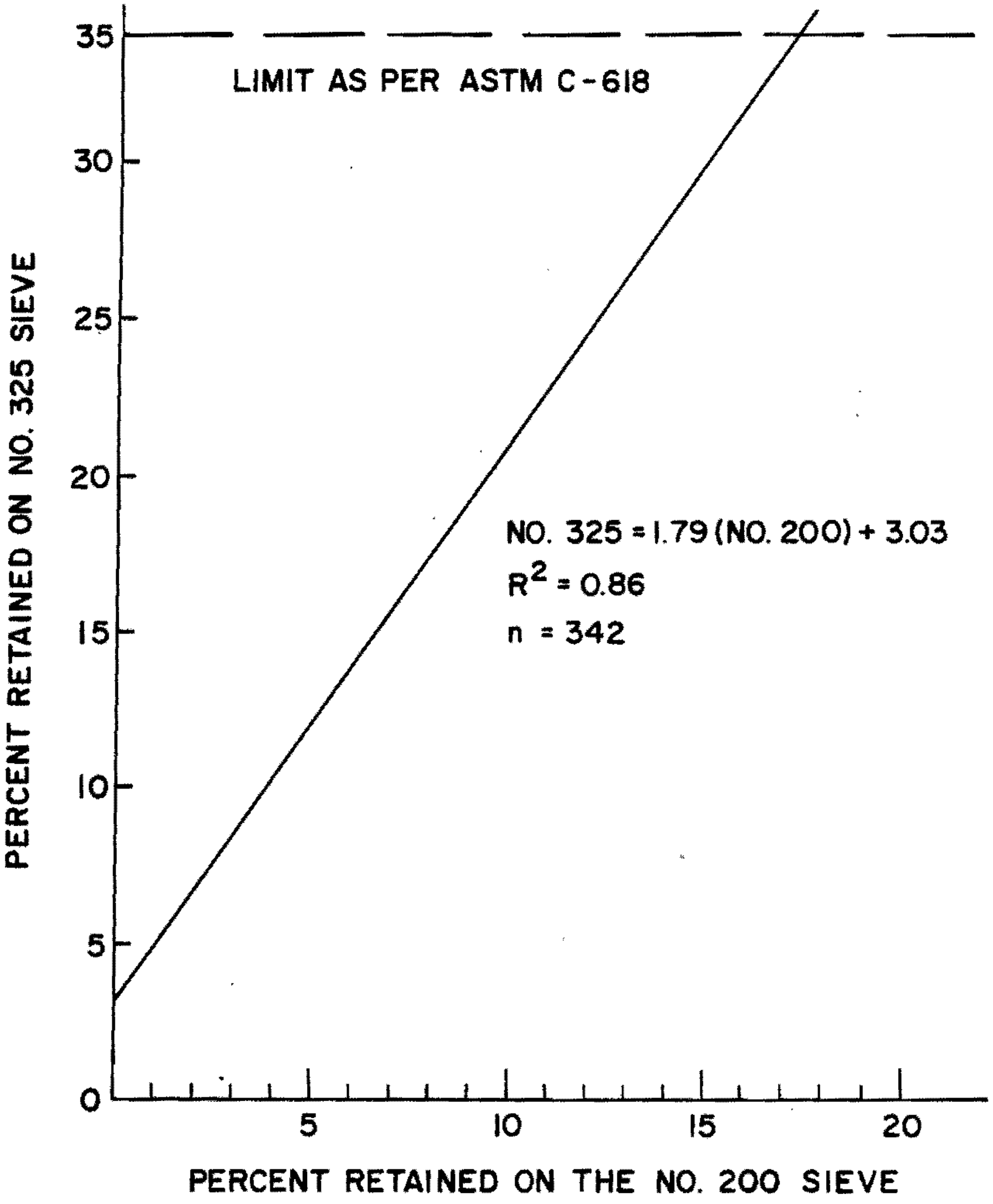

Figure 9. Least Square Regression of Fineness Quality Control

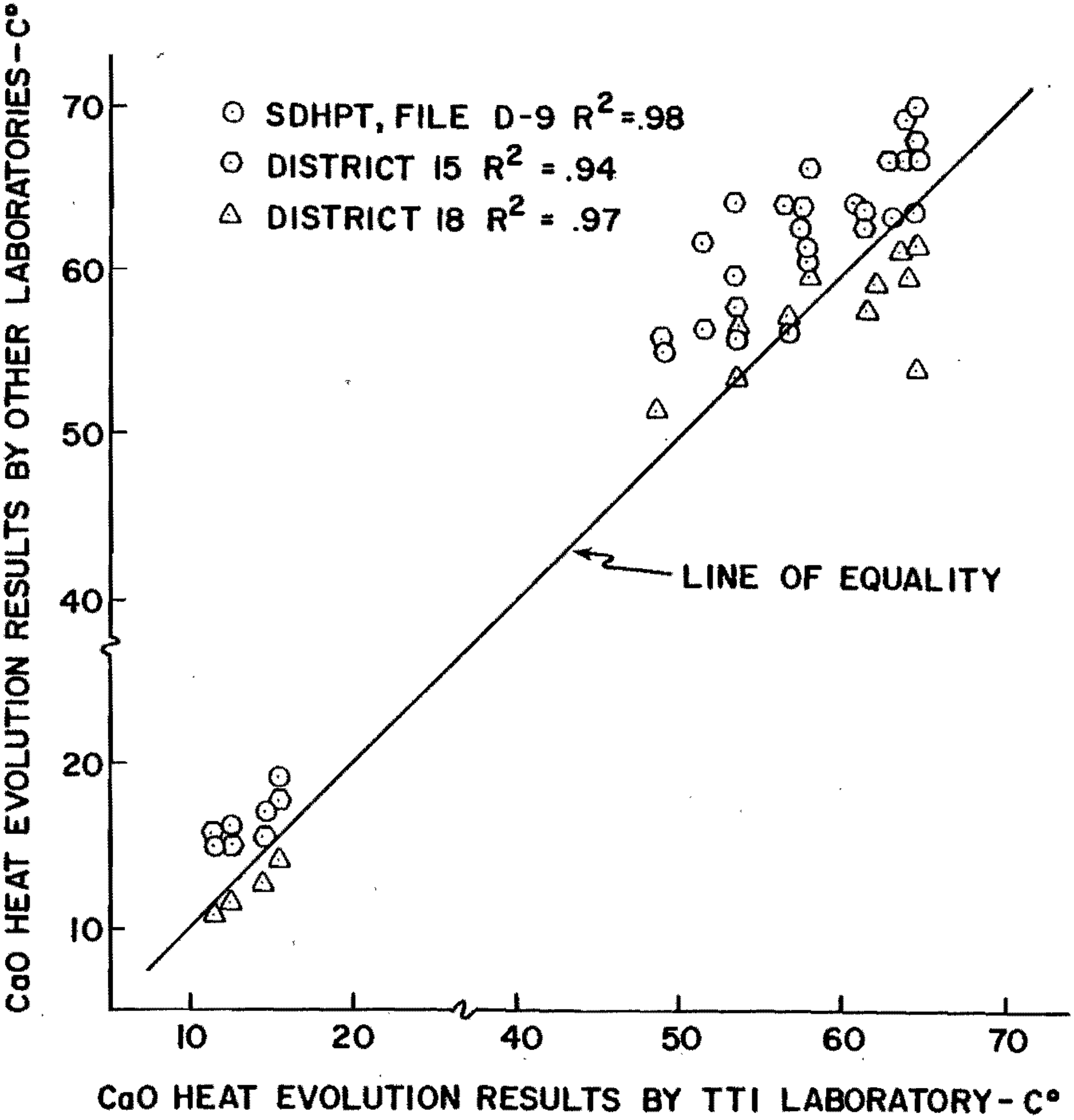

Figure 8. Interlaboratory Comparison of CaO Heat Evolution Results

3.4 Analysis of Fly Ash Compositions and Variability

To ascertain the overall chemical characteristics of the five Texas fly ashes selected for evaluation, a complete chemical oxide analysis was performed on 44 samples of fly ash (in accordance with ASTM C 311 (19)). Complete data are given in Appendix B, and the data are summarized for each fly ash in Table 3. The table gives the average and standard deviations for each oxide. Several items are worthy of note. One, there is a wide range in chemical compositions between the five fly ashes (for example the CaO contents vary from 10.6% to 29.1%), which means the performance of various fly ash sources can be significantly different. Two, the range of chemical compositions within a given fly ash source, although fairly small, may still cause variations in performance (note the standard deviations of the CaO content). Third, the LOI's are very, very low, indicating the absence of carbon in all the fly ashes. This is a distinct advantage when used in portland cement concrete. Fourth, three samples of fly ash (two from plant D and one from plant M) were significantly different than the other samples from these two plants (see Tables B-2 and B-7 in Appendix B). Notably, all three samples came from field construction sites where the fly ash source could not be verified. If the three samples are omitted, the mean values change slightly, but the standard deviation values change significantly (note numbers in parenthesis in the table). In the case of the fly ash from plant M, the suspect sample indicated a CaO content of 18.00%, or almost twice the average. This is cause for concern and adds to the need to be able to determine the CaO content quickly and easily. Fifth, the values of MgO for fly ash from plants

Table 3. Summary of chemical oxide analyses of selected Texas Fly Ashes.

Fly Ash	Statistic	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equivalent Na_2O	LOI
D	Average	39.6	22.7	4.9	25.5 (24.4)[a]	3.75	1.53	0.42	0.38
	Std. Dev.	2.94	1.67	0.76	3.51 (1.73)[a]	1.02	0.38	0.27	0.26
	n=16								
H	Average	35.2	21.9	6.4	27.5	4.55	2.32	0.73	0.40
	Std. Dev.	1.63	1.79	0.49	1.47	0.51	0.21	0.26	0.08
	n=7								
M	Average	60.6	22.3	3.3	10.6 (10.1)[b]	2.10	0.32	0.04	0.08
	Std. Dev.	2.30	3.61	0.14	4.54 (1.44)[b]	0.10	0.28	0.03	0.04
	n=8								
W	Average	33.9	22.8	5.6	29.1	3.86	3.09	1.17	0.40
	Std. Dev.	1.26	1.48	0.22	2.52	0.49	0.52	0.17	0.10
	n=7								
B	Average	49.9	17.2	7.3	18.2	3.46	1.45	0.19	0.42
	Std. Dev.	1.05	0.37	0.32	0.36	0.13	0.04	0.03	0.10
	n=6								

[a] Results if two samples are omitted. See Table B-2 in Appendix B.

[b] Results if one sample is omitted. See Table B-7 in Appendix B.

D, H and W are uncomfortably close to the 5.0% maximum allowed by ASTM C-618. This could pose performance problems as discussed in Chapter 1.

Since physical as well as chemical properties are important, fineness and specific gravity determinations were made on all 342 samples of fly ash from the five plants. In addition, the CaO content was determined on all samples using the CaO Heat Evolution Test (see Section 3.2). All data are given in Appendix B and summarized in Table 4, and there are several items of interest. First, concerning CaO content, the standard deviations are generally smaller than reported in Table 3. This is due, in part to the fact that 55 percent of the samples were collected directly from the various plants over a three-month period, with 45 percent of the samples collected from field construction projects during the following year or so. This is contrasted to the smaller number of samples on which complete chemical analysis was determined (Table 3). Relatively speaking, a much higher percentage of the samples used in Table 3 came from fly ash collected from field construction projects, accentuating any variability that might be present. Also the three suspect samples discussed earlier, even though included in Table 4, had less influence because of the much larger population of values being analyzed. A second observation from Table 4 is the relatively large variation in fineness (% retained on No. 325) that was found. More of this point will be made later! Third, although the variation in specific gravity appears to be low, in reality, the variations are significant as will

Table 4. Summary of variability testing.

Fly Ash	Statistic	% Retained No. 325 Sieve	% Retained No. 200 Sieve	Specific Gravity	CaO by Heat Evolution
D	Average	17.3	8.7	2.57	24.2
	Std. Dev.	3.32	2.40	0.05	1.78
	n=102				
H	Average	15.8	7.2	2.62	26.8
	Std. Dev.	1.79	1.42	0.03	1.36
	n=50				
M	Average	29.6	13.7	2.27	9.8
	Std. Dev.	4.87	2.96	0.04	1.56
	n=74				
W	Average	15.6	7.5	2.63	27.6
	Std. Dev.	3.70	2.18	0.05	0.88
	n=69				
B	Average	15.8	6.6	2.56	18.0
	Std. Dev.	2.26	1.06	0.03	1.33
	n=47				

be shown in the next paragraph.

Concerning the variation in fineness and specific gravity, a comparison in uniformity was made using ASTM C-618 as a standard. The results are given in Table 5. Strictly speaking uniformity could not be precisely determined because the samples were not taken in succession, as required by the specification. But, if the averages calculated represent the moving average of successive samples, then several samples exhibited excessive variability(in terms of fineness). The purpose of this evaluation was to examine the potential variability and the results indicate fly ash from a given source has the "potential" for being more variable than presently allowed by ASTM C-618.

One other point should be made here. The marketer of fly ash from Plant B states their fly ash is "processed", which means it has been run through an air classifier and from 15 to 30 percent of the coarser particles removed. The result is a relatively uniform product with only two samples failing to meet uniformity requirements. Fly ash from plant D is also supposed to now be "processed" in which they utilize a skimming technique to skim off the larger particles as they pass through a pipe. Comparison of both unprocessed fly ash (82 samples) and processed fly ash (20 samples) from Plant D indicate no significant differences in terms of fineness or specific gravity (see Tables B-1 and B-3 in Appendix B) and thus their results were combined for presentation in Tables 4 and 5.

In terms of overall variability the data in Table 5 suggest that fly ash from Plant H is the most uniform, while fly ash from Plant M is the least uniform (in terms of fineness and specific gravity).

Table 5. Summary of samples "potentially" failing to meet ASTM C-618 uniformity specifications for specific gravity and fineness.[a]

Fly Ash	No. of Samples	Number of Potential Uniformity Failures			Percent Failing
		No. 325 Sieve	Specific Gravity	Total	
D	102	7	0	7	7
H	50	1	0	1	2
M	74	14	1	15	20
W	69	16	0	16	23
B	47	2	0	2	4

---

[a] Assuming the overall average is representative of the average of the 10 preceding samples taken in succession.

3.5 Performance Variability of Mortars

The variability in performance resulting from the use of fly ash is an unresolved issue. It is quite possible for variability testing to indicate a variable ash yet the actual performance might be very uniform. For this reason, it was desirable to examine the variability of performance properties when fly ash was included as a construction material.

Several fresh properties of portland cement/fly ash mortars were investigated to determine the influence of fly ash on a construction product. The fresh properties of mortars were ideal for this purpose because 1) the variable nature induced by admixtures in portland cement mortar can more readily be attained than by examining soil and 2) the results from evaluating fresh properties were readily obtainable in the laboratory. The portland cement was Type I, conforming to the requirements of ASTM C-150 (4). The sand was graded Ottawa Silica Sand from Ottawa, Illinois.

The performance testing included the following tests:

1. Pozzolanic activity index
2. Water requirement
3. Time of initial set
4. Time of final set
5. Air content
6. Flow

The Pozzolanic activity index (PAI) and water requirement (WR) were determined for one representative sample from each power plant.

The purpose of this spot testing was to gain some insight into these properties for each of the five plants.

The water requirement for each of the samples was determined in accordance with ASTM C-311. The test for pozzolanic activity index also followed ASTM C-311. Table 6 displays the results of the Water Requirement testing and both 28-day and 7-day PAI testing for one representative fly ash sample from each power plant. All fly ashes exceeded the requirements established by ASTM C-618.

The flow, air content, time of initial set, and time of final set were all examined for various mortar compositions. One fly ash sample was selected from each of the five sources and used to make mortars with water-cement ratios of 0.4, 0.5, and 0.6 by weight. For each water-cement ratio, mortar specimens were prepared with 0, 10, 20, and 30 percent replacements of fly ash by weight from each of the five plants. Therefore, 15 test specimens and one control were prepared for each water-cement ratio, and 48 specimens generated In addition, two extra fly ash samples were selected from each of the five plants and used to prepare ten other mortar specimens, all with a 0.5 water-cement ratio, and 20 percent fly ash replacement. A total of 58 mortar specimens were thus prepared from which flow, air content, initial set and final set determinations were made. It was decided to investigate the effects of water-cement ratio and fly ash replacement percentage on the fresh properties; therefore, a wide range of mortar compositions was selected to insure adequate investigation of those two variables and to reveal any relationships

Table 6. Pozzolanic Activity and Water Requirement Results.

Fly Ash	Sample No.	Water Requirement (% Control)	Pozzolanic Activity Index 28 Day (% Control)
D	3-12	92	128
H	9	96	116
M	1-8	90	110
W	2-4	91	115
B	11-2	85	137
ASTM C-618 Specifications		max 105	min 75

that might be present.

The mortar specimens were mixed in a five gallon rotary mixer for four minutes after the cement and fly ash contacted the mixing water. The sand was slowly added between minute one and minute two in an effort to approximate ASTM C-305, "Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency" (9). Upon completion of mixing, the flow test was performed, the time of set cylinders prepared, and the air content measured, in that sequence. From the time of mixing, it took from between 16 to 18 minutes to complete these operations.

The flow was determined in accordance to ASTM C-109, "Compressive Strength of Hydraulic Cement Mortars" (4). The initial and final times of set were determined in accordance to ASTM C-403, "Time of Setting of Concrete Mixtures by Penetration Resistance", with two exceptions (4). ASTM C-403 called for the mortar tested to be sieved from a concrete sample, and this program used a straight mortar mixture. However, this irregularity is not anticipated to alter the effects of fly ash on setting times. Secondly, the specifications also called for three batches to be made for each test condition, but in this case, only one batch was made because of time constraints. Finally air content was measured in accordance with the American Association of State Highway and Transportation Officials (AASHTO) T-199, "Air Content of Freshly Mixed Concrete by the Chace Indicator" (23). The results of the performance testing are given in Appendix C.

To visualize the relative effect of the various fly ashes on these fresh properties, for each water-cement ratio and ash replacement combination, the entrained air, flow, and set times (both initial and final) for each fly ash were ranked from one to five (lowest value to the highest). The rankings were then averaged for each ash and listed in Table 7. From this table it is apparent that different fly ashes do behave differently when mixed with portland cement. The percentage replacement and water-cement ratio both influence mortar behavior as can be seen by the average values of relative ranking. Mortar using fly ash from plant B quite noticeably exhibited the highest flows, with the lowest flows exhibited for mortars using fly ash from plant W. As expected, both water-cement (w/c) ratio and ash replacement percentage strongly influenced flow. Figure 10 is typical of the influence of w/c ratio and fly ash percentage on flow. Of special interest is the fact that 0.4 w/c ratio concrete can be made to exhibit equivalent flow to 0.5 w/c ratio concrete by the replacement of 20 to 30% portland cement with fly ash. Undoubtedly, concrete applications involving pumping are taking advantage of this.

Air content, although variable, was not significantly different between the various fly ashes used, the w/c ratios employed or the percentages of the fly ash replacement investigated.

The most striking difference occurred in the setting behavior of the mortars. It was obvious during testing that the plant M ash was the quickest setting ash. At 10 percent replacement it was

Table 7. Average Relative Rankings of Flow, Set Time and Air Content for each fly ash.

Plant and Average Ranking[a] for		
Flow	Air Content	Set Time
W - 2.0	B - 1.8	M - 1.4
H - 2.4	H - 2.4	B - 2.3
M - 2.8	W - 2.8	D - 2.9
D - 3.1	D - 2.9	W - 3.7
B - 3.9	M - 4.2	H - 4.7

[a] Lowest to highest - one to five.

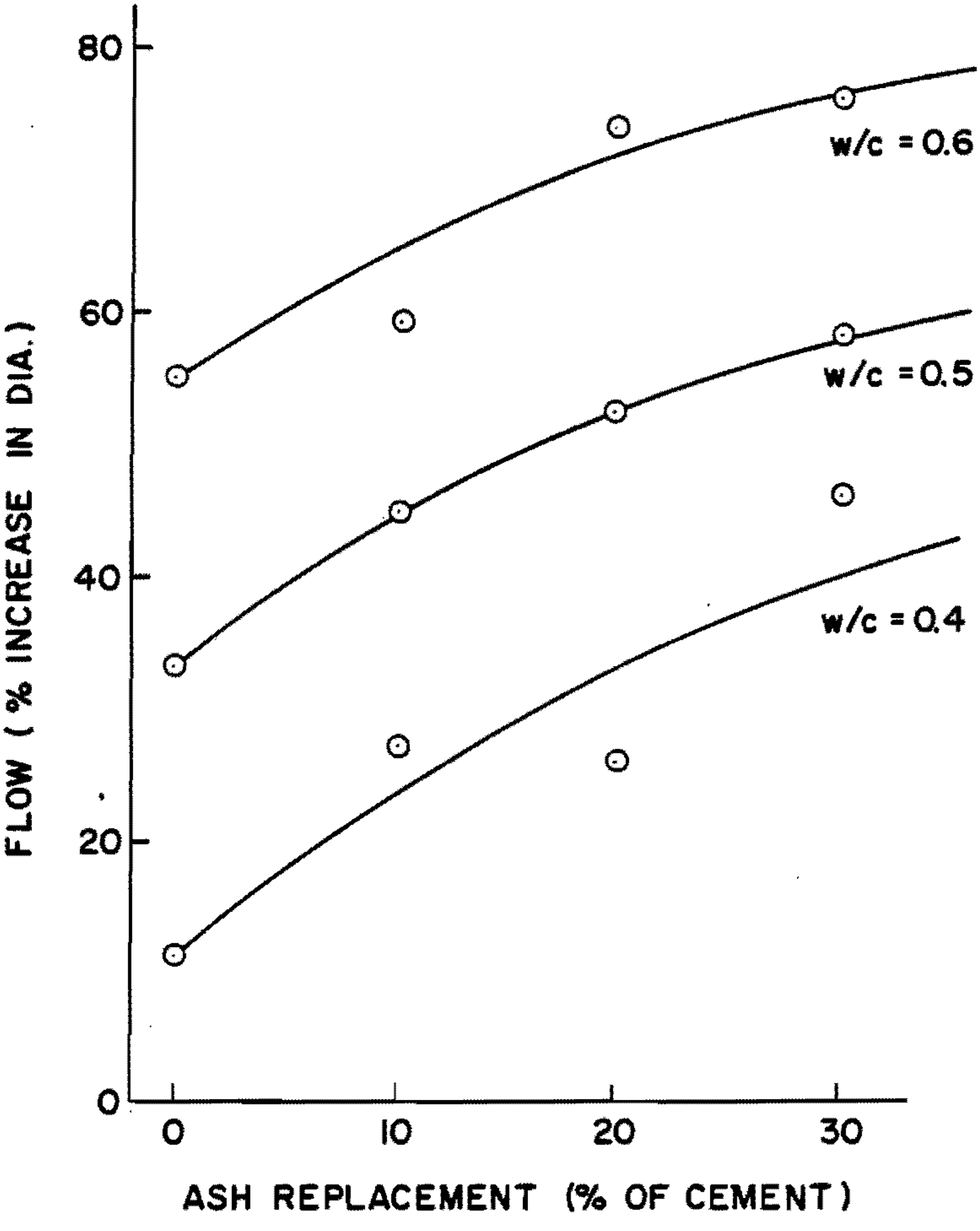

Figure 10. Flow results of plant W fly ash.

usually seen to set quicker than a 100 percent portland cement mixture. The samples made with plant H fly ash were consistently the last samples to set, retarding the final set as much as 4 1/2 hours at 30 percent replacement. The remaining three ashes usually set in the order of plant B, plant D, and plant W. Replacement of cement by fly ash almost invariably delayed initial and final set as replacement percentages were increased. Figure 11 illustrates the typical retarding action of fly ash as increasing quantities of cement were replaced. No other external factors such as water-cement ratio were observed to affect relative times of set.

All these results indicate different ashes behave differently in mortar. To analyze this behavior and see what interrelationship could be established, a complete statistical analysis of all the data (both physical and chemical) was made. The results of this analysis are presented in the next chapter.

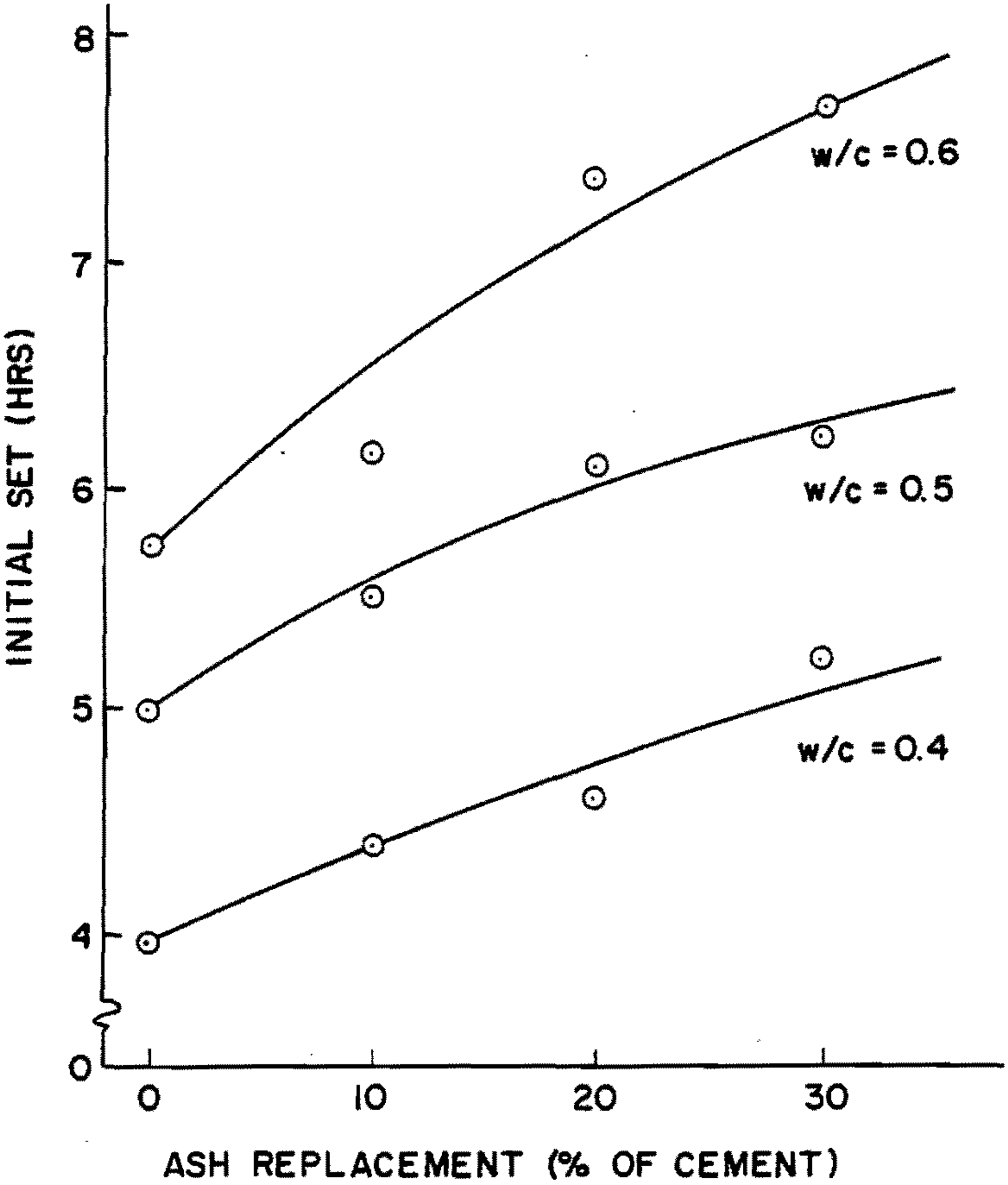

Figure 11. Initial set results of plant B fly ash.

4. STATISTICAL ANALYSES

4.1 Correlation of Fly Ash Properties

Very little is known about the interrelationships that exist between the properties of western fly ashes. For this reason, a portion of this study centered on analyzing the correlation between the physiochemical properties. Use of correlation analyses to investigate further or perhaps even estimate a given ash characteristic through knowledge of a second property could be a substantial contribution to ash utilization.

Analyses of the correlations between physical and chemical properties was done using Pearson, Spearman, and Kendall correlation methods (24, 25, 26). The results of the significant correlations for these analyses are presented in Appendix D. These three methods use slightly different assumptions to develop the degree of correlation between two parameters and calculate the probability of correlations occurring by chance, i.e., the probability that no correlation really exists. The degree of correlation is usually expressed as a level of confidence. For this study extremely strong correlations were identified when the probability of a chance correlation was < 0.001, strong correlations were identified when the probability of a chance correlation was > 0.001 and < 0.01, and good correlations were identified when the probability of a chance correlation was > 0.01 and < 0.05. While some slight differences in results did appear between the three methods they usually agreed very well. Those interrelationships exhibiting a very strong correlation by all three methods are given in Figure 12. As expected from the analysis presented in Chapter 3, excellent correlations were found between the fineness measurements (No. 200 and No.325 sieve with an R value of 0.93) as well as between the two methods of determining CaO content (chemical analysis and heat evolution with an R value

	% Ret. No. 200	Spec. Grav.	CaO by Heat Evol.	LOI	Moisture	SiO_2	Al_2O_3	Fe_2O_3	SUM*	CaO	MgO	SO_3	Equiv. Na_2O
% Ret. No. 325	●	●	●			●			●				
% Ret. No. 200													
Spec. Grav.			●			●			●	●		●	
CaO by Heat Evol.						●			●	●		●	
LOI													
Moisture													
SiO_2									●	●		●	●
Al_2O_3													
Fe_2O_3													
SUM*										●		●	●
CaO												●	●
MgO													
SO_3													●

*SUM = $SiO_2 + Al_2O_3 + Fe_2O_3$

● PROBABILITY OF CORRELATION OCCURRING BY CHANCE IS LESS THAN .001 FOR ALL THREE CORRELATION METHODS.

Figure 12. Extremely strong correlations indicated by Pearson, Spearman, and Kendall methods.

of 0.94). But a number of other excellent correlations were found as well.

The sieve analyses were tied very close to other ash characteristics. High negative or inverse correlations were found between the percent retained on a No. 325 sieve and both specific gravity and CaO content. This indicates that finer ashes, higher calcium contents, and heavier unit weights are all somehow interlinked. A very strong positive correlation existed between percent retained and the sum of silicates, aluminates, and ferrites (SUM), coarser ashes usually occurring in higher quantities of the SUM components. Further examination revealed that the correlations involving SUM elements seem to be almost exclusively governed by silicate contents; as the silicate quantities fluctuated, the SUM contents did likewise. The apparent reasons for such a tight relationship seem to be the small quantities of iron present, and an extremely non-variable nature exhibited by the aluminates.

Perhaps one of the more significant correlations surrounds the deleterious compounds (LOI, Na_2O equivalent, SO_3, and MgO). The analysis indicated that these compounds usually increased or decreased as a unit. The ashes which demonstrated higher fineness, higher specific gravities, higher calcium contents, and lower quantities of silicates, were accompanied by larger LOI, alkali, SO_3, and MgO values.

The very high negative correlation between SiO_2 and CaO appear to stem from the compositional nature of fly ash. The SiO_2 and CaO components were the only major constituents which varied significantly, all other components being relatively stable when compared to these two. The analysis indicated that as CaO was found to increase or decrease the difference was compensated for by SiO_2. Because of this strong inverse correlation, the SiO_2 always exhibited opposite correlations of the same magnitude as the CaO correlations.

Two deleterious substances were also strongly correlated with the calcium and silica contents. Both the alkali and SO_3 concentrations appeared to rise with increasing CaO, decreasing SiO_2 percentages. Larger amounts of SO_3 should not significantly affect unit weight.

By this time a pattern was seen to develop. The correlations that appeared to exist were more tightly interwoven than might be first anticipated. Figure 13 is intended to demonstrate the interconnected nature of ash properties as these properties range from low to high values. This simplified figure illustrates how an ash with, for example, a high calcium oxide content usually exhibited higher LOI, MgO, alkali, and SO_3 levels, larger specific gravities, smaller particle sizes, and lower silica contents. Continuing with CaO content as an example, when lower calcium oxide quantities were observed, lesser amounts of the deleterious substances were encountered also, accompanied by lower specific gravities. In addition, the particle size increased as did the silica content. This was the overall picture that the correlation analyses appeared to paint.

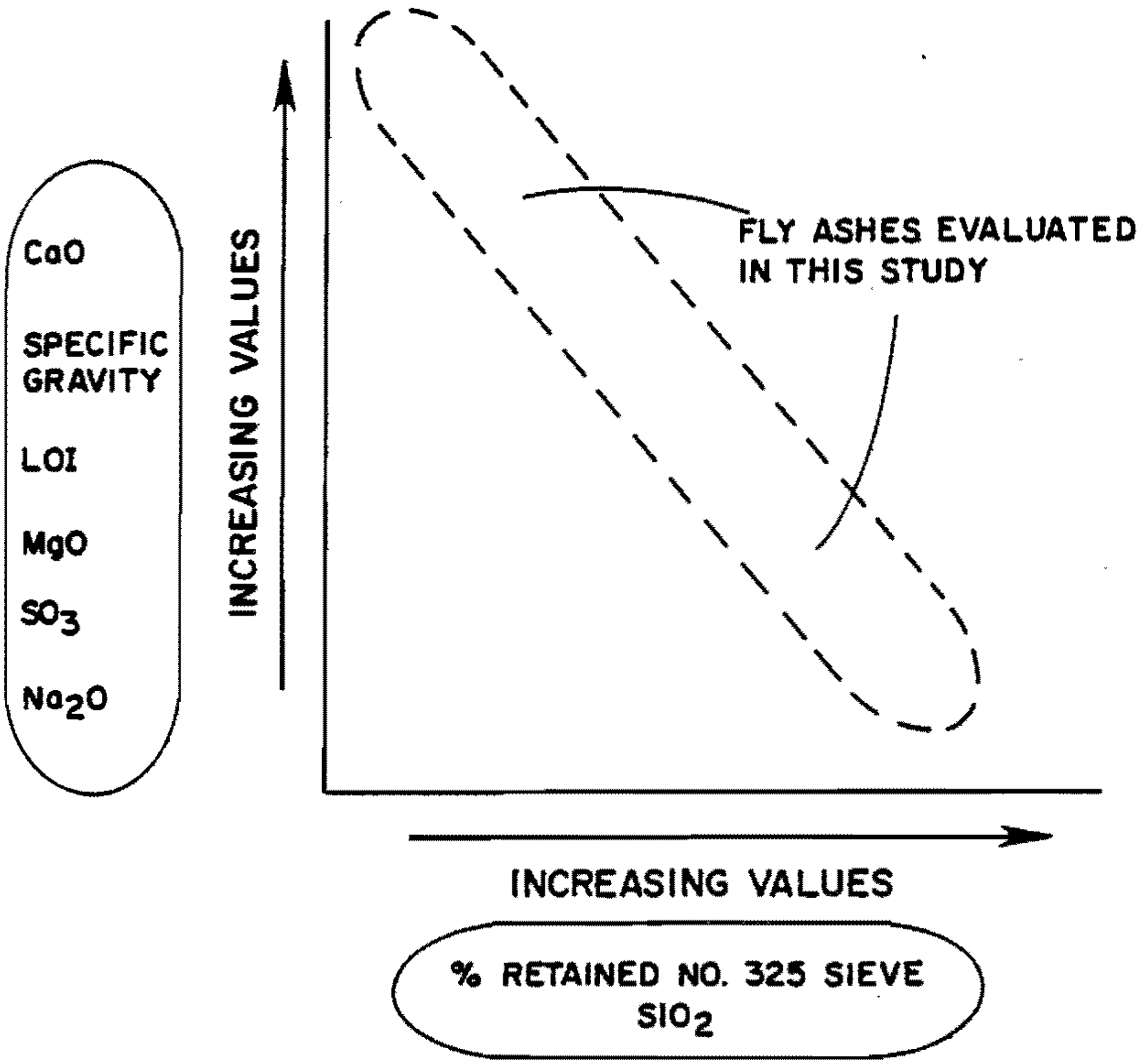

Figure 13. Compositional tendencies of ashes encountered.

4.2 Improved Predictions of Fly Ash Characteristics

As fly ash characteristics — especially fineness, specific gravity, and CaO content — are of major importance in this study, an additional statistical analysis was made to examine the interrelationships between these chacteristics with the goal of developing relationships which would improve the reliability of predicting important fly ash properties.

Regression analyses of the specific gravity and percent retained on a No. 325 sieve were performed using all 342 samples. Eighty-six percent of the variance in the No. 325 sieve results could be accounted for by the No. 200 sieve analysis (see Section 3.3). The accuracy of the model was improved to 92 percent when the CaO heat evolution results were included. The improved relationship is:

$$\text{No. } 325 = 1.40\,(\text{No.}200) - 0.12\,(\Delta T) + 11.86$$

Where: ΔT = Heat Evolution in °C

$R^2 = 0.92$

Both the No. 200 sieve analysis and the CaO heat evolution test contributed to the determination of the specific gravity. Eighty-six percent of the variance in specific gravity could be accounted for by the following equation:

$$\text{Specific Gravity} = 0.006\,(\Delta T) - 0.009\,(\text{No. } 200) + 2.33$$

$$R^2 = 0.86$$

Using these two equations, close approximations of fineness, CaO content, and specific gravity can be quickly obtained from the No. 200 sieve and

CaO heat evolution tests. These findings should significantly contribute to the expedient determination of fly ash uniformity in the field.

4.3 Statistical Analyses of Mortar and Fly Ash Characteristics

The laboratory results were processed through a multiple regression analysis to determine the physical and chemical properties of fly ash which appeared to govern the flow, air content, initial set, and final set of a fly ash/portland cement mortar. The 15 samples tested at water-cement ratios of 0.5 and 20 percent fly ash replacement were used because they reduced the complexity of the analysis by limiting the number of variables considered. The functional relationships that were determined are:

$$\text{Initial Set} = f(Al_2O_3,\ Fe_2O_3) \qquad R^2 = 0.87$$

$$\text{Final Set} = f\left(Al_2O_3,\ Fe_2O_3,\ \frac{1}{SiO_2}\right) \qquad R^2 = 0.87$$

$$\text{Air Content} = f\left(\%\ \text{retained No. 325},\ \frac{1}{LOI},\ \frac{1}{MgO},\ \frac{1}{SiO_2}\right) \qquad R^2 = 0.89$$

The specific forms of the statistical equations were not given because the data were too limited to establish the exact values in each equation. BUT, the high degree of correlations found do suggest that such relationships do exist!

The initial time of set was shown to be dependent on the presence of aluminate and ferrite, which together accounted for 87 percent of the variation in initial set time. Higher concentrations of these elements appeared to prolong initial set. This conclusion is not in keeping with the effects of high aluminate in portland cement, where flash setting is a result of high Al_2O_3 concentrations.

The most significant relationship found for final set also involved Fe_2O_3 and Al_2O_3. Again, as with initial set, increases in aluminate and ferrite percentages appeared to lengthen the time to final set. The analysis of final set times also indicated that an additional element, SiO_2, was significant. This implies that the effects of SiO_2 are not significant within the first hours of setting; however, with the passing of time, the presence of SiO_2 becomes more pronounced acting to accelerate the time to final set. These silicate compounds were linked earlier to reduced set times, and in light of these results, it is strongly suggested that the SiO_2 concentration is indeed related to the setting characteristics of a fly ash/cement mortar.

Note that the most significant model of air content indicated that four fly ash properties (LOI, fineness, SiO_2, MgO) accounted for 89 percent of the entrained air content variation. It stands to reason that if indeed the above compounds do affect air content, large variations of these compounds can cause entrained air to fluctuate.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the analysis of five fly ashes produced in Texas from sub-bituminous coal and lignite, the following conclusions were drawn:

1. The total CaO content of Texas fly ashes can be accurately and rapidly estimated in the field by the use of a simple test - termed the CaO Heat Evolution Test - which takes less than 10 minutes to perform. The total CaO content was found to be:

$$\text{CaO} = 0.395\ (\Delta T) + 3.234$$

2. An interlaboratory evaluation of the CaO Heat Evaluation Test indicates the test is repeatable and reliable.

3. The percentage retained on the No. 325 Sieve can be accurately and rapidly estimated by determining the percentage retained on the No. 200 Sieve. While each fly ash has its characteristic relationship between these two sieve sizes, a good correlation was found by combining all the data from the fly ashes. The combined equation is:

$$\text{No. } 325 = 1.79\ (\text{No. } 200) + 3.03$$

The reliability of determining the fineness (% retained on the No. 325) can be significantly improved if the value for CaO Heat Evolution is added to the equation. The resulting relationship that was developed is:

$$\text{No. } 325 = 1.40\ (\text{No. } 200) - 0.12\ (\Delta T) + 11.86$$

4. There is a wide range of chemical compositions found in Texas fly ashes, both between different sources and - to a lesser extent - within a given source with time. This wide range makes it very important to be able to quickly estimate the total CaO content of a sample of fly ash.

5. There is a wide range in physical characteristics between different fly ashes and - to a lesser extent - within a given source with time. In particular each fly ash tested exhibited sufficient variability in fineness to potentially exceed the uniformity requirements presently specified in ASTM C-618.

6. All five fly ashes met the PAI and water requirements limits specified in ASTM C-618, indicating they all exhibit acceptable properties for use as a partial replacement for lime and portland cement.

7. The wide range in both chemical and physical properties between fly ashes significantly affected the properties of fresh mortar made with varying percentages of fly ash. In particular, flow, setting times (both initial and final) and air contents were markedly different for different fly ashes.

8. Increasing the percent ash replacement of portland cement in mortar significantly increases the flow and significantly increases the initial set times of a given mortar mix. For example a 0.4 w/c ratio mortar can be made to flow like a 0.5 w/c ratio mortar by replacing 20 to 30 % of the portland cement with fly ash. This same replacement may increase setting times, although when coupled with a reduction in the w/c ratio, the increase in setting time may be reduced.

9. Extremely strong correlations were found between many of the physical characteristics and chemical properties of the five fly ashes. Of particular interest is the finding that increasing values of CaO, specific gravity, LOI, MgO, SO_3 and alkalies always were accompanied by decreasing values of fineness and SiO_2 content.

5.2 Recommendations

Based on the conclusions reached in this study the following recommendations are made.

1. Consideration should be given to specifying a field acceptance uniformity requirement for CaO content in the fly ash, based on the CaO Heat Evolution Test. This requirement should be reasonable, perhaps 5 percent points from the average of the 10 preceding samples. By having such a requirement the purchaser of the fly ash can be assured he is receiving the same fly ash he used in performing his preliminary laboratory designs.

2. Consideration should be given to checking fineness of random shipments of purchased fly ash using the No. 200 sieve. By so doing the variability in fineness can be more fully ascertained to see if additional specification requirements are necessary.

3. Consideration should be given to "source qualifying" fly ash produced for the Texas highway market. This source qualification should be repeated on a random schedule from shipments of fly ash purchased for use in Texas highways.

4. Specifications developed to insure quality of fly

ash being used in Texas highways should be periodically reviewed in the light of experience gained with the objective of <u>both</u> tightening and loosening parts of the specifications as warrented to provide the most cost effective utilization of fly ash.

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Appendix A. TEST PROCEDURES

A-1. CaO Heat Evolution Test

This test involves the isothermic reaction that occurs when hydrochloric acid is added to fly ash. The chemical reaction that occurs is:

$$CaO + 2HCl \rightarrow CaCl + H_2O + \underline{HEAT}$$

There is a linear relationship between the rise in temperature and the total Calcium Oxide present.

Materials and Equipment

The materials and equipment required to perform this test are:

1. U-Shaped Evacuated Thermos Bottle with a stopper that has a hole through the center to accommodate a thermometer
2. 100 ml graduated cylinder
3. Scales accurate to ± 0.2 grams
4. 15 percent hydrochloric acid solution
5. Two thermometers - one ranging from 0 to 100°C to measure the temperature rise and long enough to touch the bottom of the tube while being read in place, and the second covering a range in °C that will include the initial temperature of the fly ash.
6. A representative fly ash sample

Figure A-1 is a photograph of the materials and equipment necessary to perform this test.

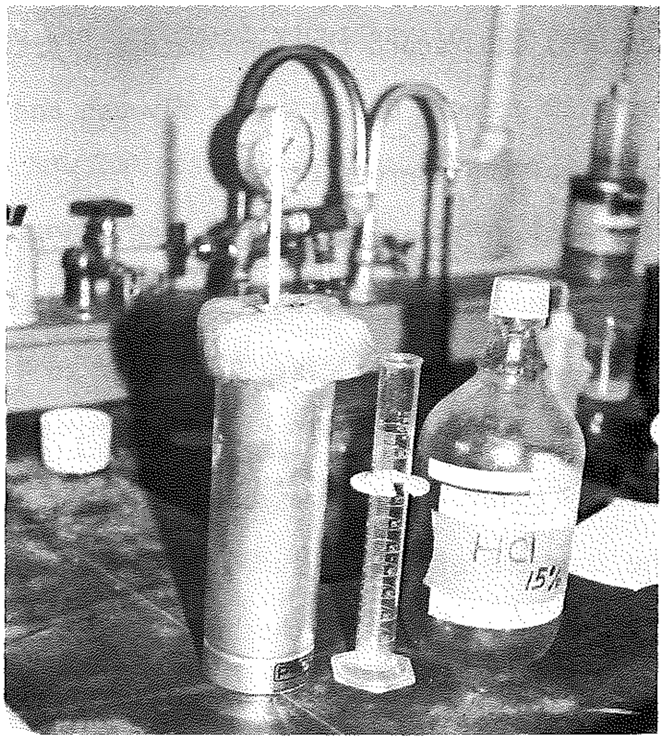

Figure A-1. Photograph of CaO heat evolution test materials and equipment.

Procedure

1. Allow the separated fly ash, acid and thermos bottle <u>each</u> to reach an equal and constant temperature (usually room temperature) and record the temperature (initial temperature).
2. Weigh 20 grams ± 0.2 grams of fly ash and place it in the bottom of the thermos bottle.
3. Add 75 ml of 15 percent HCl to the fly ash within the thermos bottle and stir to insure mixing. [Fifteen percent HCl is made by mixing 6 parts of distilled water to 4 parts of 12 molar HCl (37.5% pure).]
4. Quickly cover the thermos bottle with the stopper and insert the thermometer, being sure the tip of the thermometer is touching the bottom of the bottle.
5. Observe and record the thermometer readings until a drop in temperature is seen (usually occurs within 5 minutes).
6. Subtract the highest temperature observed from the original temperature found in Step 1. This will give the change in temperature in °C.
7. Either read and record the total CaO content from the appropriate graph (Figure 7), or calculate the total CaO content by use of the following formula:

$$\text{CaO} = 0.395\ (\Delta T) + 3.234$$

Where ΔT = Change in temperature in °C, found in Step 6.

CaO = total CaO content (record to nearest 0.1%)

A-2. No. 200 Sieve Analysis

Materials and Equipment

The materials and equipment necessary to the performance of this test are :

1. Standard No. 200 mesh sieve
2. Scale accurate to ± 0.5 grams
3. A source of gently flowing water
4. A source of heat to dry the fly ash
5. Small flexible bristled brush to clean retained particles from the sieve
6. Representative sample of fly ash.

Test Procedure

1. Weigh out 50 ± 0.5 grams of fly ash and place it on the No. 200 sieve.
2. Run a gently flowing stream of water across the sample accompanied by a slow wrist motion (being careful not to slosh any of the material over the sides of the sieve).
3. When all of the minus 200 sieve particles appear to have washed through, place the sieve where it can dry without being disturbed.
4. After drying, gently, but firmly brush the residue off the sieve and weigh it to determine the percent retained.

Appendix B. FLY ASH DATA

Table B-1. Results of variability testing for unprocessed fly ash from plant D.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution
1	18.8	9	2.54	22.8
2	21.0	12	2.59	23.2
3	20.2	11	2.54	23.8
4	20.2	11	2.58	24.0
5	22.3	13	2.66	22.7
6	23.4	14	2.58	25.1
7	23.2	14	2.61	24.1
8	22.6	13	2.60	
9	20.2	11	2.60	21.3
10	20.8	11	2.43	20.7
11	17.6	8	2.66	23.0
12	17.5	8	2.49	23.8
13	17.2	8	2.44	23.3
14	15.1	8	2.54	24.8
15	17.0	8	2.52	24.7
16	14.5	7	2.63	22.0
17	13.8	7	2.64	23.7
18	15.6	7	2.60	24.8
19	14.7	6	2.65	23.2
20	15.2	6	2.62	26.0
21	16.9	8	2.60	21.8
22	16.0	8	2.54	22.9
23	16.6	8	2.52	22.0

Table B-1. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
24	16.9	8	2.63	23.3
25	16.9	8	2.55	21.7
26	15.4	8	2.55	21.6
27	14.4	6	2.58	23.0
28	14.7	7	2.58	23.6
29	14.6	7	2.62	22.5
30	15.2	7	2.62	22.5
31	16.8	10	2.56	26.0
32	18.6	8	2.58	27.3
33	15.9	8	2.65	22.1
34	19.5	10	2.52	21.9
35	20.3	9	2.51	22.9
36	19.3	8	2.49	22.3
37	18.4	8	2.54	22.8
38	16.4	8	2.54	23.3
39	20.2	10	2.51	25.3
40	19.3	10	2.53	22.8
41	19.0	8	2.53	24.1
42	19.2	10	2.55	25.1
43	18.2	9	2.58	25.1
44	18.7	8	2.58	25.3
45	18.7	9	2.58	24.1

Table B-1. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
3- 1	14.0	7	2.48	25.4
3- 2	15.1	7	2.53	26.6
3- 3	16.1	9	2.59	26.5
3- 4	15.3	8	2.48	25.5
3- 5	15.2	9	2.57	25.8
3- 6	17.2	8	2.58	26.0
3- 7	16.6	9	2.59	24.9
3- 8	17.0	11	2.58	25.4
3- 9	15.9	8	2.59	26.6
3-10	12.5	6	2.64	27.8
3-11	14.5	8	2.62	26.6
3-12	15.7	10	2.63	26.7
3-13	14.9	8	2.61	25.0
3-14	15.1	8	2.60	26.3
3-15	12.2	6	2.64	28.2
4- 1	17.6	8	2.68	28.5
4- 2	19.3	9	2.59	23.2
4- 3	21.7	10	2.57	23.4
4- 4	19.2	8	2.56	23.3
4- 5	17.8	8	2.59	23.2
4- 6	13.2	5	2.59	25.3
4- 7	15.4	8	2.57	23.4
4- 8	17.4	8	2.54	22.3

Table B-1. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	Ca O by Heat Evolution Test
4- 9	17.2	8	2.55	24.1
4-10	23.4	10	2.53	21.8
4-11	16.9	8	2.58	23.0
4-12	18.9	10	2.56	22.4
4-13	16.9	9	2.55	22.5
4-14	15.8	8	2.59	23.4
4-15	17.0	8	2.50	22.9
4-16	18.0	9	2.60	23.3
5- 1	15.2	11	2.51	26.4
5- 2	12.6	7	2.51	26.4
5- 3	13.2	7	2.48	25.5
6- 3	13.1	6	2.65	27.2
6- 4	14.0	7	2.59	24.82
6- 5	28.8	15	2.63	25.7

Table B-2. Results of chemical oxide analysis of unprocessed fly ash from plant D.

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
6	41.37	24.50	4.82	23.29	3.35	1.12	0.49	0.55
20	41.47	24.49	4.81	23.82	3.31	0.99	0.51	0.14
32	38.78	23.73	5.26	24.87	4.32	1.78	0.73	0.29
39	40.69	25.40	5.14	22.70	3.96	1.12	0.66	0.27
3- 3	40.16	21.70	4.76	24.90	4.81	1.47	0.08	0.32
3- 7	40.92	22.60	4.86	24.30	4.81	1.48	0.12	0.02
3-12	40.00	21.40	4.85	25.70	4.31	1.52	0.05	0.08
3-13	40.14	21.00	4.89	24.30	4.69	1.42	0.13	0.12
4- 1	36.61	21.73	3.96	33.80	1.56	1.29	0.47	0.50
4- 6	42.17	24.76	4.49	23.20	2.07	2.28	0.76	0.34
4-10	38.54	22.20	4.14	29.31	2.67	1.58	0.84	0.42
5- 1	43.80	19.80	4.65	22.50	4.23	1.44	0.25	0.78
5- 2	39.60	23.10	4.98	23.50	4.70	1.42	0.21	0.18
5- 3	40.70	21.50	4.70	23.60	4.70	1.45	0.28	0.36

Table B-2. (Continued).

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
6- 3	38.42	24.70	4.58	25.76	3.06	1.67	0.78	0.88
6- 4 [a]	30.56	21.31	7.49	33.24	3.52	2.42	0.32	0.80

[a] These samples are significantly different than the remainder

Table B-3. Results of variability testing for processed fly ash from plant D.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
50	14.0	6	2.60	25.4
51	13.0	6	2.60	25.5
52	13.7	6	2.62	25.5
53	14.0	6	2.60	24.4
54	23.9	14	2.53	21.3
55	17.0	8	2.53	21.3
56	20.0	10	2.50	21.9
57	13.0	6	2.59	25.4
58	17.3	8	2.53	23.6
59	17.2	10	2.54	23.5
60	17.4	9	2.53	24.5
61	17.9	9	2.53	24.5
62	30.2	16	2.49	19.0
63	18.9	10	2.56	24.9
64	19.5	10	2.58	24.6
65	15.5	7	2.59	25.7
66	25.1	14	2.53	21.2
67	12.5	6	2.59	26.6
68	12.7	6	2.55	26.9
69	13.1	6	2.59	26.9

Table B-4. Results of variability testing for fly ash from plant H.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
1	13.8	6	2.60	28.4
2	13.7	7	2.60	26.5
3	15.2	7	2.56	26.0
4	14.0	7	2.62	29.0
5	14.0	6	2.63	27.6
6	14.0	6	2.60	28.3
7	14.5	7	2.60	26.3
8	14.0	6	2.53	27.6
9	14.9	6	2.56	27.7
12	15.7	8	2.56	25.0
16	18.1	9	2.58	25.4
17	18.1	8	2.59	24.7
18	18.0	9	2.56	25.0
19	16.8	8	2.59	
20	16.9	6	2.59	26.0
23	16.4	8	2.59	27.0
24	15.4	8	2.63	24.7
25	15.3	7	2.60	24.3
26	15.8	7	2.62	26.0
27	15.7	7	2.62	24.3
28	19.0	9	2.62	27.0
29	18.1	9	2.63	28.1
30	16.3	8	2.60	28.0

Table B-4 (Continued).

Sample No.	(% Retained)	(% Retained)	Specific Gravity	CaO by Heat Evolution Test
31	19.0	9	2.66	26.0
32	15.5	8	2.66	25.7
33	13.8	6	2.64	27.2
34	15.4	6	2.64	24.6
35	15.3	6	2.60	27.5
36	12.6	5	2.63	25.9
37	12.4	5	2.59	27.7
38	12.0	5	2.63	27.9
39	22.9	11	2.64	25.9
40	15.3	6	2.62	24.6
41	12.3	5	2.64	24.5
42	15.9	7	2.64	23.6
43	23.2	10	2.60	26.3
44	14.7	7	2.61	28.3
45	16.3	7	2.60	26.7
8-1A	15.0	6	2.66	27.8
8-1B	14.7	6	2.66	27.2
8-1C	16.0	7	2.66	27.8
8-1D	15.3	7	2.63	25.1
8-1E	16.0	7	2.65	27.3
8-2A	14.1	7	2.60	28.8
8-2B	14.4	6	2.65	26.9

Table B-4 (Continued).

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
8-2C	14.9	7	2.62	27.8
8-2D	14.5	7	2.65	28.4
8-2E	17.0	10	2.65	28.0
8-3A	14.6	6	2.65	26.9
8-3B	16.9	10	2.62	25.7

Table B-5. Results of chemical oxide analysis of fly ash from plant H.

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
1	32.43	23.32	5.72	29.39	4.52	2.76	1.20	0.42
9	36.09	23.86	6.20	26.40	3.95	2.27	0.85	0.26
17	36.95	22.85	6.77	25.76	4.06	2.06	0.79	0.36
19	37.00	22.79	6.55	25.96	4.13	2.22	0.78	0.36
8-1B	34.58	20.00	7.22	28.00	5.10	2.30	0.51	0.46
8-2A	34.50	21.50	6.03	28.10	5.00	2.32	0.50	0.46
8-3A	34.62	19.10	6.41	29.00	5.10	2.34	0.49	0.48

Table B-6. Results of variability testing for fly ash from plant M.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution
1	33.2	17	2.26	8.4
2	33.0	14	2.28	9.3
4	28.8	14	2.28	9.4
5	28.7	13	2.25	9.7
7	29.0	13	2.23	
14	26.0	12	2.30	9.6
15	25.6	12	2.26	9.3
17	27.6	13	2.24	10.0
18	28.4	13	2.31	9.5
19	28.1	13	2.28	9.5
20	29.2	14	2.22	9.8
21	29.9	13	2.31	9.4
22	33.8	16	2.26	9.1
23	29.2	14	2.26	10.0
24	30.3	14	2.21	9.5
25	30.6	14	2.21	9.7
26	27.0	12	2.29	10.9
27	30.6	14	2.29	10.0
28	25.8	12	2.30	9.6
29	33.6	16	2.26	8.3
30	29.8	13	2.34	9.7
31	29.5	14	2.29	10.0
32	28.3	14	2.26	9.2

Table B-6. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 325 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution
33	26.2	12	2.30	10.8
34	40.1	18	2.21	8.7
35	29.9	14	2.25	9.9
36	19.7	9	2.27	10.7
37	25.7	14	2.33	10.3
38	29.7	15	2.25	9.2
39	33.6	16	2.23	9.0
43	34.7	16	2.20	8.6
46	31.7	15	2.24	9.7
48	24.9	12	2.26	9.5
49	31.6	15	2.29	9.2
51	27.3	12	2.31	10.1
52	34.9	16	2.24	9.6
55	29.0	14	2.26	10.0
56	30.1	15	2.29	9.8
58	28.7	16	2.31	10.3
61	29.8	14	2.30	9.5
62	29.7	14	2.21	9.9
65	28.4	14	2.25	10.0
67	31.8	14	2.23	9.5
78	29.0	15	2.25	
1- 1	28.7	13	2.29	9.5
1- 2	31.6	12	2.29	10.9

Table B-6. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
1- 3	10.7	4	2.33	9.5
1- 4	24.6	5	2.30	9.4
1- 5	33.0	14	2.28	9.2
1- 6	19.6	8	2.35	11.6
1- 7	26.0	12	2.32	12.1
1- 8	20.1	9	2.34	11.0
1- 9	29.5	15	2.33	9.1
1-11	33.6	16	2.29	8.4
1-12	20.2	6	2.37	11.5
1-13	25.6	12	2.39	18.7
1-14	31.7	15	2.27	8.0
1-15	29.0	13	2.28	8.0
1-16	27.6	13	2.22	15.8
1-17	28.8	14	2.19	12.1
1-18	28.4	14	2.20	9.3
1-19	33.9	16	2.27	8.8
1-20	29.2	14	2.25	9.2
1-21	40.1	18	2.23	8.0
1-22	33.8	16	2.26	8.2
1-23	29.2	15	2.26	8.7
1-24	42.3	20	2.26	8.0
1-25	39.6	20	2.25	8.0
1-26	37.4	18	2.27	8.4

Table B-6. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
1-27	33.2	17	2.26	8.0
1-28	38.3	18	2.25	8.6
1-30	30.6	14	2.28	9.5
1-31	27.0	12	2.28	9.0
1-32	15.5	6	2.32	11.9

Table B-7. Results of chemical oxide analysis of fly ash from plant M.

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
29	60.13	25.47	3.29	8.73	2.10	0.18	0.02	0.11
30	60.65	24.77	3.47	8.91	2.06	0.05	0.03	0.07
34	60.32	25.65	3.33	8.40	1.99	0.14	0.03	0.14
52	60.28	25.34	3.34	8.77	2.08	0.05	0.03	0.04
1- 3	62.58	20.40	3.43	9.00	2.17	0.42	0.03	0-04
1- 8	61.56	18.90	3.41	12.00	2.27	0.52	0.05	0.12
1-13[a]	55.90	16.10	3.06	21.30	1.97	0.86	0.10	0.04
1-24	63.70	21.40	3.17	7.30	2.16	0.30	0.03	0.08

[a] This sample is significantly different than the remainder

Table B-8. Results of variability testing for fly ash from plant W.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained	Specific Gravity	CaO By Heat Evolution
1	10.5	5	2.55	27.5
2	18.0	8	2.63	28.4
3	19.0	11	2.67	27.0
4	21.4	11	2.65	27.3
5	19.9	10	2.65	28.2
6	9.8	4	2.59	28.5
7	7.9	4	2.57	28.2
8	9.2	4	2.60	28.6
9	9.3	3	2.60	29.5
10	8.7	4	2.59	27.9
11	8.4	4	2.59	28.5
12	9.5	5	2.61	28.3
13	9.2	4	2.60	27.9
14	9.5	4	2.60	28.5
15	10.1	4	2.61	28.5
16	10.7	5	2.56	28.6
17	17.8	8	2.61	26.6
18	17.4	8	2.57	26.6
19	17.0	8	2.57	27.4
20	14.2	6	2.59	28.1
21	16.6	7	2.53	28.0
22	17.3	8	2,57	27.7
23	18.5	9	2.59	27.8

Table B-8 (Continued).

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
24	18.7	10	2.56	27.0
25	17.1	8	2.56	27.5
26	16.6	8	2.57	28.0
27	16.2	8	2.67	26.2
28	19.5	10	2.56	27.5
29	19.6	9	2.63	28.6
30	19.4	9	2.56	27.7
31	18.6	9	2.53	28.2
32	20.8	10	2.55	27.5
33	17.5	9	2.61	28.1
34	17.6	8	2.60	28.3
35	18.9	10	2.57	28.4
36	17.9	9	2.61	28.6
37	17.5	9	2.63	28.1
38	19.9	10	2.60	28.4
39	19.1	10	2.57	28.3
40	17.7	9	2.65	29.2
41	17.6	9	2.63	28.1
42	18.9	10	2.63	28.9
43	17.2	9	2.63	28.5
44	18.3	9	2.63	28.6
45	18.0	9	2.63	

Table B-8. (Continued).

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
2- 1	15.0	8	2.70	31.2
2- 2	17.5	8	2.70	30.0
2- 3	17.0	8	2.70	29.8
2- 4	13.7	6	2.66	27.8
2- 5	14.2	6	2.67	28.9
2- 6	16.2	8	2.70	30.0
2- 7	17.0	8	2.70	29.4
2- 8	16.5	8	2.70	28.9
2- 9	17.3	9	2.69	28.0
2-10	16.9	9	2.69	27.6
2-11	15.5	8	2.69	29.0
2-12	18.0	9	2.69	28.2
2-13	17.9	10	2.70	28.0
2-14	15.6	9	2.68	30.0
2-15	16.2	8	2.70	28.6
2-16	16.5	8	2.70	30.6
2-17	9.2	4	2.70	27.6
2-18	8.4	4	2.69	29.0
2-19	8.4	3	2.68	28.8
2-20	16.8	8	2.69	28.0
2-21	15.6	6	2.70	29.2
2 22	14.0	6	2.70	30.8

Table B-8. (Continued).

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
2-23	14.0	5	2.67	28.2
2.24	15.2	6	2.69	28.8

Table B-9. Results of chemical oxide analysis of fly ash from plant W.

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
7	34.02	24.81	5.35	27.51	4.27	2.68	1.21	0.34
9	33.87	24.14	5.41	28.00	4.27	2.80	1.10	0.34
31	36.11	23.91	5.64	26.25	4.03	2.47	0.84	0.41
44	34.39	23.30	5.71	27.19	4.29	3.15	1.14	0.41
2- 1	31.87	20.90	5.71	33.00	3.44	4.07	1.35	0.30
2- 4	33.63	21.44	5.48	31.13	3.20	3.23	1.28	0.40
2 12	33.42	21.31	5.59	30.93	3.44	3.21	1.26	0.60

Table B-10. Results of variability testing for fly ash from plant B.

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO By Heat Evolution
2	16.6	6	2.59	18.7
3	16.0	6	2.60	18.4
6	16.9	7	2.60	19.3
7	15.8	6	2.62	18.5
8	18.2	6	2.52	16.1
9	17.4	7	2.51	15.6
10	17.9	7	2.54	15.3
11	17.8	8	2.54	15.1
15	17.9	8	2.54	18.4
18	17.6	7	2.54	18.8
21	18.3	8	2.58	18.0
22	18.4	7	2.54	16.8
23	18.3	6	2.56	17.4
25	14.2	5	2.55	18.0
27	13.2	5	2.56	19.0
28	11.7	5	2.56	19.2
29	13.6	6	2.56	18.7
30	10.2	5	2.57	19.6
31	13.1	5	2.59	20.4
32	12.9	6	2.60	19.3
33	16.7	7	2.55	17.8
34	16.0	7	2.54	16.7
35	13.0	6	2.58	20.5

Table B-10. (Continued)

Sample No.	No. 325 Sieve (% Retained)	No. 200 Sieve (% Retained)	Specific Gravity	CaO by Heat Evolution Test
36	17.9	8	2.53	16.7
37	16.1	7	2.54	17.4
38	15.8	7	2.54	17.7
39	16.6	8	2.60	17.2
40	18.5	8	2.51	16.8
41	18.2	8	2.54	17.8
42	18.9	8	2.53	18.5
43	19.2	8	2.50	18.9
44	13.6	6	2.56	20.4
45	10.6	4	2.60	21.2
46	14.4	6	2.53	17.4
47	14.9	6	2.52	18.0
48	13.1	5	2.54	19.2
49	19.1	8	2.51	19.0
50	14.7	7	2.53	17.3
51	16.8	7	2.53	17.7
52	15.4	6	2.55	19.9
53	15.4	6	2.55	18.6
54	14.3	6	2.54	17.0
55	14.9	6	2.54	17.0
56	13.7	6	2.54	17.0
57	15.1	6	2.55	16.7
11-1	16.0	7	2.53	18.2
11-2	15.4	8	2.53	16.3

Table B-11. Results of chemical oxide analysis of fly ash from plant B.

Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Equiv. Na_2O	LOI
2	48.98	17.40	7.58	18.50	3.56	1.38	0.18	0.34
7	49.26	16.80	7.66	18.60	3.53	1.48	0.16	0.50
15	49.96	17.40	6.92	18.00	3.23	1.45	0.20	0.52
28	49.56	17.60	7.26	17.90	3.53	1.45	0.19	0.28
11-1	51.64	16.80	7.06	17.80	3.47	1.47	0.23	0.44
11-2	52.04	16.40	7.05	17.10	3.40	1.41	0.23	0.50

Appendix C. MORTAR DATA

Table C-1. Results of flow test on mortar samples.

Fly Ash	Sample No.	Flow for 0-30% Wt. Replacements of Cement by Fly Ash (% Increase in Sample Base)			
		0	10	20	30
water-cement ratio = 0.4					
D	4-10	11	24	28	48
H	17	11	30	29	32
M	1-24	11	31	22	35
W	2-12	11	27	26	46
B	11-2	11	22	32	48
water-cement ratio = 0.5					
D	3-12	33	--	59	--
D	4-10	33	55	48	61
D	5-3	33	--	50	--
H	1	33	--	58	--
H	9	33	--	61	--
H	17	33	55	56	51
M	1-3	33	--	51	--
M	1-8	33	--	55	--
M	1-24	33	51	56	62
W	2-1	33	--	52	--
W	2-4	33	--	55	--
W	2-12	33	45	52	58
B	7	33	--	56	--
B	15	33	--	58	--
B	11-2	33	55	60	61
water-cement ratio = 0.6					
D	4-10	55	55	80	78
H	17	55	53	76	84
M	1-24	55	59	80	86
W	2-12	55	59	74	76
B	11-2	55	65	83	85

Table C-2. Test results for initial time of set of mortar samples.

Fly Ash	Sample No.	Initial Set Times for 0-30% Wt. Replacements of Cement by Fly Ash (Hrs.)			
		0	10	20	30
water-cement ratio = 0.4					
D	4-10	3.97	4.50	5.00	5.72
H	17	3.97	5.08	5.33	6.58
M	1-24	3.97	3.88	4.47	4.83
W	2-12	3.97	4.50	4.90	5.50
B	11-2	3.97	4.45	4.58	5.05
water-cement ratio = 0.5					
D	3-12	5.00	--	6.25	--
D	4-10	5.00	5.75	6.08	6.42
D	5-3	5.00	--	6.08	--
H	1	5.00	--	7.25	--
H	9	5.00	--	7.37	--
H	17	5.00	6.10	7.58	8.50
M	1-3	5.00	--	5.75	--
M	1-8	5.00	--	5.75	--
M	1-24	5.00	5.67	5.75	5.75
W	2-1	5.00	--	6.33	--
W	2-4	5.00	--	6.33	--
W	2-12	5.00	5.75	6.67	6.42
B	7	5.00	--	5.92	--
B	15	5.00	--	6.08	--
B	11-2	5.00	5.53	6.08	6.17
water-cement ratio = 0.6					
D	4-10	5.75	5.93	6.83	7.92
H	17	5.75	5.92	8.70	9.33
M	1-24	5.75	5.42	6.55	7.00
W	2-12	5.75	6.17	7.50	8.33
B	11-2	5.75	6.17	7.37	7.67

Table C-3. Test results for final time of set of mortar samples.

Fly Ash	Sample No.	Final Set Times for 0-30% Wt. Replacements of Cement by Fly Ash (Hrs.)			
		0	10	20	30
water-cement ratio = 0.4					
D	4-10	6.47	6.42	7.08	8.05
H	17	6.47	7.00	7.83	9.83
M	1-24	6.47	6.00	6.77	7.22
W	2-12	6.47	6.30	7.30	8.03
B	11-2	6.47	6.50	6.62	7.23
water-cement ratio = 0.5					
D	3-12	7.42	--	8.58	--
D	4-10	7.42	7.92	8.50	9.45
D	5-3	7.42	--	8.75	--
H	1	7.42	--	10.17	--
H	9	7.42	--	10.97	--
H	17	7.42	8.92	11.05	12.17
M	1-3	7.42	--	9.17	--
M	1-8	7.42	--	8.42	--
M	1-24	7.42	7.83	8.67	8.58
W	2-1	7.42	--	9.17	--
W	2-4	7.42	--	9.25	--
W	2-12	7.42	8.25	9.33	9.67
B	7	7.42	--	8.33	--
B	15	7.42	--	8.50	--
B	11-2	7.42	7.83	8.45	9.58
water-cement ratio = 0.6					
D	4-10	8.58	8.83	9.92	11.17
H	17	8.58	8.87	11.75	12.92
M	1-24	8.58	8.17	10.17	10.00
W	2-12	8.58	9.25	10.87	11.67
B	11-2	8.58	9.33	10.75	10.58

Table C-4. Test results for air content for 15 cubic feet of mortar per cubic yard of concrete.

Fly Ash	Sample No.	Air Content for 0-30% Wt. Replacements of Cement by Fly Ash (% by Volume) 0	10	20	30
water-cement ratio = 0.4					
D	4-10	4.1	3.9	4.2	3.9
H	17	4.1	3.8	3.9	4.1
M	1-24	4.1	4.3	4.3	4.2
W	2-12	4.1	3.9	4.0	3.7
B	11-2	4.1	3.7	4.1	3.8
water-cement ratio = 0.5					
D	3-12	4.2	-	4.3	-
D	4-10	4.2	4.3	4.5	4.0
D	5-3	4.2	-	3.8	-
H	1	4.2	-	4.1	-
H	9	4.2	-	4.2	-
H	17	4.2	4.3	4.2	4.2
M	1-3	4.2	-	4.0	-
M	1-8	4.2	-	4.0	-
M	1-24	4.2	4.7	4.3	4.3
W	2-1	4.2	-	4.3	-
W	2-4	4.2	-	4.5	-
W	2-12	4.2	4.2	4.2	4.2
B	7	4.2	-	3.8	-
B	15	4.2	-	4.0	-
B	11-2	4.2	4.0	3.8	3.5
water-cement ratio - 0.6					
D	4-10	4.7	4.2	3.9	3.9
H	17	4.7	4.4	3.9	3.8
M	1-24	4.7	4.7	3.9	3.8
W	2-12	4.7	4.3	4.1	4.1
B	11-2	4.7	4.0	3.8	3.5

	% Ret. No.200	Spec. Grav.	CaO by Heat Evol.	LOI	Moisture	SiO_2	Al_2O_3	Fe_2O_3	SUM*	CaO	MgO	SO_3	Equiv. Na_2O
% Ret. No.325	● .93	● N .80	● N .76	□ N .44		● .71		● N .59	● .75	● N .70	● N .62	● N .66	□ N .44
% Ret. No.200		● N .68	● N .59	/ N .33		● .57		● N .57	● .61	● N .56	N .46	● N .58	□ N .42
Spec. Grav.			● .89	● .56		● N .87		● .65	● N .88	● .88	● .58	● .78	● .59
CaO by Heat Evol.				● .50		● N .96		□ .46	● N .94	● .94	● .68	● .81	● .68
LOI					/ .37	● N .55		● .51	● N .53	● .55		● .49	□ .40
Moisture							□ N .47						
SiO_2								● N .53	● .96	● N .96	● N .67	● N .86	● N .74
Al_2O_3								□ .41					
Fe_2O_3									● N .55	□ .46	● .57	● .58	
SUM*										● N .97	● N .65	● N .85	● N .68
CaO											● .54	● .83	● .70
MgO												● .53	
SO_3													● .85

Probabilities of Correlations Occurring by Chance:

- ● $P < .001$
- □ $.001 \le P < .01$
- / $.01 \le P < .05$

*SUM = $SiO_2 + Al_2O_3 + Fe_2O_3$

N = Inverse Correlation

Figure D-1. Pearson correlation analysis of physical and chemical properties (44 samples).

	% Ret. No.200	Spec. Grav.	CaO by Heat Evol.	LOI	Moisture	SiO_2	Al_2O_3	Fe_2O_3	SUM*	CaO	MgO	SO_3	Equiv. Na_2O
% Ret. No. 325	● .81	● N .51	● N .55	/ N .36		● .54		□ N .47	● .53	□ N .48	● N .51	● N .57	□ N .45
% Ret. No.200		□ N .44	/ N .37			/ .37		● N .51	/ .36	/ N .34	/ N .30	□ N .48	● N .41
Spec. Grav.			● .70	□ .48		● N .71		● .50	● N .72	● .75	/ .35	● .68	● .37
CaO by Heat Evol.				□ .42		● N .87		/ .33	● N .84	● .83	● .51	● .77	● .78
LOI					/ .38	● N .51		● .51	● N .49	● .52		● .52	● .52
Moisture							□ N .48						
SiO_2								● N .51	● .95	● N .96	● N .50	● N .89	● N .83
Al_2O_3								/ N .39					
Fe_2O_3									□ N .49	□ .41	● .52	● .61	□ .45
SUM*										● N .96	● N .52	● N .84	● N .75
CaO											● .60	● .82	● .78
MgO												□ .49	
SO_3													● .83

Probabilities of Correlations Occurring by Chance:

- ● $P < .001$
- □ $.001 \leq P < .01$
- / $.01 \leq P < .05$

*SUM = $SiO_2 + Al_2O_3 + Fe_2O_3$

N = Inverse Correlation

Figure D-2. Spearman correlation analysis of physical and chemical properties (44 samples).

	% Ret. No.200	Spec. Grav.	CaO by Heat Evol.	LOI	Moisture	SiO_2	Al_2O_3	Fe_2O_3	SUM*	CaO	MgO	SO_3	Equiv. Na_2O
% Ret. No. 325	● .68	● N .37	● N .40	/ N .23		● .39		□ N .34	● .38	□ N .35	● N .36	● N .42	□ N .32
% Ret. No.200		● N .34	□ N .29			□ .29		● .40	/ .27	/ N .26		□ N .36	□ N .31
Spec. Grav.			● .54	□ .36		● N .55		□ .35	● N .57	● .59	/ .27	● .55	● .42
CaO by Heat Evol.				□ .29		● N .71		/ .24	● N .65	● .69	● .38	● .61	● .59
LOI					/ .28	● N .36		● .39	● N .36	● .39		● .36	□ .34
Moisture							□ N .37						
SiO_2								● N .38	● .81	● N .83	□ N .34	● N .72	● N .65
Al_2O_3								□ N .30					
Fe_2O_3									● N .37	□ .32	□ .35	● .40	□ .29
SUM*										● N .85	● N .39	● N .67	● N .58
CaO											□ .30	● .68	● .60
MgO												□ .34	
SO_3													● .64

Probabilities of Correlations Occurring by Chance :

● $P < .001$

□ $.001 \leq P < .01$

/ $.01 \leq P < .05$

*SUM = $SiO_2 + Al_2O_3 + Fe_2O_3$

N = Inverse Correlation

Figure D-3. Kendall correlation analysis of physical and chemical properties (44 samples).