Specifications and Protocols for Acceptance Tests on Processing Additions in Cement Manufacturing

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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

NCHRP REPORT 607

Specifications and Protocols for Acceptance Tests on Processing Additions in Cement Manufacturing

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Subject Areas
Materials and Construction

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FOREWORD

By Amir N. Hanna
Staff Officer
Transportation Research Board

This report presents recommended changes to the cement specifications and test protocols contained in AASHTO Standard Specifications for Transportation Materials and Methods of Sampling and Testing (AASHTO M 85). These changes pertain to the amount of processing additions that can be incorporated in the cement and the tests required for evaluating acceptability of cements incorporating processing additions. The report also presents a recommended specification for evaluating processing additions that may be used in amounts exceeding those stipulated in the cement specification. These specifications will guide materials engineers and cement producers in evaluating cements and assuring that highway concrete is not deleteriously affected by the presence of such additions. The information contained in the report will be of immediate interest to state materials engineers and others involved in specifying and evaluating concrete mixtures for use in highway pavements and structures.

Processing additions, such as granulated blast furnace slag, limestone, and fly ash, are interground with clinker in the manufacture of some portland cements to improve the efficiency of the manufacturing process. These additions also may improve product quality, reduce carbon dioxide emissions and energy requirements during the cement manufacturing process, and provide other economic and environmental benefits. However, there has been considerable debate recently about the effects of such additions on cement and concrete properties and on the performance and durability of the highway pavements and structures in which these materials are used. In addition, current cement specifications do not address, in a consistent manner, the use of such additions in cement manufacturing. Also, limited research has dealt with the effects of incorporating processing additions in cement manufacturing on concrete properties and durability, and there are no clear conclusions concerning the effects of using such additions on the performance and durability of highway pavements and structures. Thus, research was needed to assess these effects, to develop recommendations to help improve cement specifications and test protocols with regard to the use of such processing additions, and to develop guidance on the use of these cements in highway concrete.

Under NCHRP Project 18-11, "Improved Specifications and Protocols for Acceptance Tests on Processing Additions in Cement Manufacturing," Construction Technology Laboratories, Inc., of Skokie, Illinois, worked with the objective of recommending potential improvements to specifications and test protocols to determine the acceptability of cements with processing additions. To accomplish this objective, the researchers reviewed the specifications and test methods currently used for evaluating portland cement and investigated their suitability for evaluating cements incorporating processing additions. The investigation included an extensive laboratory testing program that covered the types and ranges of processing additions currently used or expected to be used in the future in the United States; considered the chemical, physical, and mineralogical characteristics of cement; and evaluated the properties of a large number of paste, mortar, and concrete specimens incorporating different types and amounts of processing additions. The research recommended a Standard Specification for Mineral Processing Additions for Use in the Manufacturing of Hydraulic Cements (included as Attachment 1), to evaluate acceptability of processing additions when used in amounts exceeding those stipulated in the modified AASHTO M 85. Based on analysis of test results, the research recommended changes to AASHTO M 85, Standard Specification for Portland Cement (included as Attachment 2), that provides guidance on the testing and acceptance of portland cement incorporating maximum amounts of processing addition.

The recommended modification to AASHTO M 85 and the recommended specification for mineral processing additions will be particularly useful to highway agencies because their use will assure that highway concrete is not deleteriously affected by the presence of such additions. Their adoption as part of the AASHTO Standard Specifications for Transportation Materials and Methods of Sampling and Testing is, therefore, recommended.

Appendixes A through F contained in the research agency's final report provide detailed information on material characterization, experiment design, and data analysis. These appendixes are not published herein; but are available online at http://trb.org/news/blurb_detail.asp?id=8989. These appendixes are titled as follows:

Appendix A: Fly Ash and Slag Characterization;
Appendix B: Statistical Design and Material Combinations for Mortar Tests;
Appendix C: Microscopical Examination of Coarse Fractions of Cements without SCM's
Appendix D: Conduction Calorimetry Plots;
Appendix E: Analysis of Paste and Mortar Tests; and
Appendix F: Analysis of Concrete Data

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CHAPTER 1

Introduction

Inorganic processing additions (referred to as processing additions or PAs hereafter), such as granulated blast furnace slag and fly ash, are interground with clinker in the manufacture of some portland cements, primarily to improve the efficiency of manufacturing. These additions may also improve product quality, reduce carbon dioxide emissions and energy requirements during the cement manufacturing process, and provide other economic and environmental benefits. However, there has been considerable debate recently about the effects of such additions on cement and concrete properties and on the performance and durability of the highway pavements and structures in which these materials are used. In addition, current cement specifications do not address, in a consistent manner, the use of such additions in cement manufacturing.

Although a great deal of research has been performed on the effects of portland cement characteristics on concrete properties and durability, only limited research has dealt with the effects of incorporating inorganic additions in small amounts. There are no clear conclusions concerning the effects of using processing additions in the manufacturing process on the performance and durability of highway pavements and structures. This research is intended to assess these effects, to develop recommendations to help improve cement specifications and testing protocols with regard to the use of such processing additions, and to develop guidance on the use of these cements in highway concrete.

The objective of this research was to recommend potential improvements to specifications and test protocols to determine the acceptability of cements with processing additions. This research only dealt with inorganic processing additions (i.e., it did not deal with organic grinding aids commonly used in cement manufacture). The work does not aim to pass or fail any given material or class of materials, but rather provides users the tools to be able to evaluate a given material.

The purpose was to allow users of cements containing inorganic processing additions to be assured that the highway concrete is not deleteriously affected by the presence of such materials. The protocol also needs to provide guidance on how much processing addition can safely be included in any given cement, without being cost prohibitive for suppliers of the materials.

The specific objective of the work outlined in this report was to develop the required information for either modifying or revising, on a rational basis, the specification (AASHTO M 85) for portland cement with respect to processing additions. Some of the required information was found in the literature, while other information was developed in the laboratory. The objective was to recommend specifications that assure the users of a product that it delivers the performance and service life required, yet will allow cement producers to take advantage of the economic and environmental benefits of processing additions.

The revised specification includes both prescriptive and performance requirements for portland cement containing processing additions. A limit was rationally established below which processing additions can be added to the base cement with acceptance based simply on those criteria used to establish compliance of the base cement. Above this limit (up to a maximum defined by chemical requirements of ASTM C 150/AASHTO M 85 or 5.0% whichever is lower), performance and chemical requirements are necessary. These performance requirements were selected to assure the user and purchaser of acceptable performance of the cement in fresh and hardened concrete. This two-tiered approach allows a producer to use a processing addition up to a specific limit without the need to conduct a battery of additional tests other than showing compliance with the base cement specifications. If the cement producer exceeds this limit, then specific performance requirements must be met.

The following tasks were conducted.

Phase I

- Task 1: Information on the use of processing additions in cement production and the use of these cements in highway pavements and structures was collected and reviewed. This information included domestic and foreign literature, contacts with public and private agencies and industry organizations, and other sources. Also, a summary of current use, field performance, test methods, test data, and other information pertaining to use of these cements in all types of highway concrete was compiled.

- Task 2: Based on the information reviewed in Task 1, the types and ranges of processing additions currently used in the United States and those likely to be used in the future were identified. The types and ranges of processing additions that merit further evaluation in this project were discussed and recommended.
- Task 3: The work plan, executed in Task 5 was refined. The experimental investigation included testing cement samples and concrete specimens to evaluate the effects of incorporating processing additions on cement and concrete properties. The plan considered the types and ranges of processing additions recommended in Task 2. It also considered the relevant chemical, physical, and mineralogical characteristics of cement.
- Task 4: An interim report was prepared that documented the research performed in Phase I and included the updated work plan for Phase II.

Phase II

- Task 5: The plan approved in Task 4 was executed. Based on the results of this work, quantitative models were developed that relate the characteristics of cements manufactured with processing additions to system properties.
- Task 6: Based on the models developed in Task 5, potential improvements to AASHTO specifications and test protocols were recommended to determine the acceptability of cements with processing additions.
- Task 7: The applicability of the recommendations made in Task 6 was evaluated with respect to the effects of supplementary cementitious materials commonly used in the United States.
- Task 8: This report documents the entire research effort conducted, including an implementation plan for moving the results of this research into practice. Some verification tests were conducted on new sources of materials to assess whether the protocols were effective.

CHAPTER 2

Review of Literature and Survey

Summary of Literature

The known effects of additions are summarized below. Only a limited amount of literature has been published on this topic in recent years, possibly because it is considered old technology in Europe, and little work has been required in the United States to date.

The physical and chemical mechanisms behind some of these trends are discussed first, followed by a summary of the effects on the properties of fresh and hardened concrete.

Terminology

Different terms are used in the literature and specifications to address materials added to cement in relatively small amounts for any of a number of reasons. These terms are not necessarily interchangeable.

The European cement standard (EN 197) allows up to 5% minor additional constituent (MAC), which may or may not have any benefit on the manufacture or performance of the cement, although it is not permitted to impair the water requirement or durability, nor reduce corrosion resistance of the cement to reinforcing steel. The standard suggests that such materials are added to enhance cement performance, but no such requirements are set out. The standard also allows up to 1% additives to improve manufacturing or performance of the cement.

The U.S. approach is different. There is currently no direct comparison with the MACs used in Europe. Processing additions are permitted as long as they meet the requirements of ASTM C 465. This standard is intended to cover materials added to enhance manufacturing. The performance limits imposed, however, are in terms of the cement performance, with no requirements on the benefits to cement manufacture. The performance provisions in the US standards evolved primarily to address the use of organic grinding aids, which generally are used in amounts less than 1% by mass of clinker. No quantitative limit on ASTM 465 materials is imposed in ASTM C 150 or C 1157, although an upper limit of 1% is set in AASHTO M 85.

A recent revision to ASTM C 150 permits the inclusion of 5% limestone in portland cement with certain limitations. From a broad perspective, such an allowance may be considered to be a special case of the MAC approach. It is still permitted to use ASTM C 465 processing additions in addition to limestone. The selection of materials used in this project reflected this fact.

To broaden the amount of data available in the literature, the references and interviews described below have included those from other countries, meaning that the direct application of the materials added to cement may be different from that intended in this research project. This must be borne in mind when reviewing the information provided.

Materials to be used as inorganic additions to cement generally contain the same main constituents as portland cement: CaO, SiO_2, Al_2O_3, and Fe_2O_3. Based on European use, the materials most likely to be used are limestone, fly ash, bottom ash, slag, cement kiln feed, cement kiln dust, and calcined byproducts (Dhir, 1994, Rosani, 2003); of these, limestone, raw meal, and cement kiln dust are most frequently used (Moir, 1994). In North America, there has been a considerable amount of work conducted and reported on investigations into the effects of limestone on concrete performance when interground with cement in small quantities (Hawkins, 2003). This material is not considered a processing addition in this project, but some of the trends learnt from this work are still useful to consider. The term "processing addition" has been used in this document in terms of the ASTM C 219 definition.

In all of the following discussion, only inorganic materials are considered—organic grinding aids are specifically excluded.

Background

Processing additions are defined in ASTM C 219 as materials that are interground or blended into hydraulic cements to aid in their manufacture and handling. Inorganic materials are commonly included in grinding because they aid the process and help optimize the total grinding energy required to achieve a given fineness of cement. For example, slag and fly ash are harder than cement clinker, and because of their hardness, the particles are effective in cleaning the clinker particles from grinding media in ball mills, thus avoiding the cushioning effect of coated balls and improving grinding efficiency (Spellman, 1999). Such materials also increase the yield of a cement plant without the need for increasing kiln capacity or the energy required to heat the materials in the kiln. This is of significant benefit to society because the specific emission of CO_2 and other green house gases and the embodied energy in the final product are reduced.

Processing additions are materials added before, during, or after finish grinding of the cement. Figure 1 is a schematic of a cement grinding system. Clinker and gypsum are fed to the mill at a controlled rate. After passing through the mill, the ground material is conveyed to the separator where the portion of the material with the correct particle size is separated out as the final product, and the particles still too coarse are sent back to the mill for further grinding. The numbered boxes in the figure indicate the locations in the mill system where processing additions could be added. They could be part of the feed material for the mill [1] and be subjected to grinding. They could be added at the separator [2] and, if sufficiently fine, become incorporated in the final product, or could be part of the coarse fraction returned to the mill for further grinding. Finally processing additions could be added [3] as part of the finished product to improve handling characteristics.

The selection of the location for introducing a processing addition to the system often depends on the properties of the material, such as fineness and moisture content. A large proportion of the power consumed in the grinding process is transformed into heat, causing an increase in the temperature of the cement in the mill. This temperature rise has to be controlled to a maximum of about 110 to 120°C to avoid complete dehydration of the gypsum. This heat can be utilized for drying material containing some moisture. Coarse materials such as granulated blast furnace slag, bottom ash, or limestone (or moist material) would therefore be introduced with the clinker and gypsum. Fine and very fine materials not well suited for handling on a conveyor belt, could be introduced at the separator or with the final product.

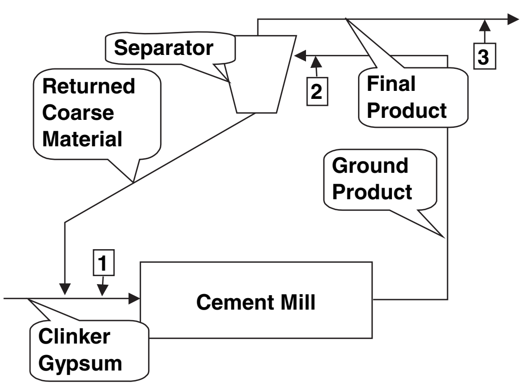


Figure 1. Schematic illustration of cement grinding circuit.
Note: Processing additions may be added to the material flow at Points 1, 2, or 3.

Usage and Specifications

Specifications

There is considerable variation in how cement additions are specified around the world. Different specifications for cement allow from less than 1% up to 5% maximum non-clinker material, with varying degrees of limitation on their composition or performance effects. The next few paragraphs summarize some of these specifications.

Processing additions are described in ASTM C 219 under the general term "addition," which is defined as "a material that is interground or blended in limited amounts into hydraulic cements during manufacture either as a 'processing addition' to aid in manufacturing and handling the cement or as a 'functional addition' to modify the use properties of the finished product."

Neither ASTM C 150, "Standard Specification for Portland Cement," ASTM C 595 "Standard Specification for Blended Hydraulic Cements," nor ASTM C 1157, "Standard Performance Specification for Hydraulic Cement," limit the amount of processing additions that can be used. However, all three specifications require that such additions conform to the requirements of ASTM C 465, "Standard Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements." There is nothing in the wording of ASTM C 150 or ASTM C 465 that prohibits the use, as processing additions, of any amount of inorganic materials such as limestone, fly ash, slag, or cement kiln dust. However, the amount used would be limited by the chemical requirements for loss on ignition, insoluble residue, and sulfate of ASTM C 150. ASTM C 150-04 permits the inclusion of up to 5.0% limestone, as long as the chemical and physical requirements of the standard are still met. The limits on loss on ignition and insoluble residue are again likely to limit the amount of limestone that can be included. This inclusion is not as a processing addition.

AASHTO M 85-01, "Standard Specification for Portland Cement," permits the use of up to 1% (by mass of portland cement clinker) processing additions at the option of the manufacturer, provided the material meets the requirements of ASTM C 465. Although slightly more than half of the state DOTs follow AASHTO, at least two of these states allow an increased level of processing additions to be used. The Texas and Illinois DOTs allow up to 3% and 4%, respectively, of either fly ash or slag to be used as a processing addition (limestone and cement kiln dust are not permitted). Information from the Illinois DOT indicates that one plant is using approximately 2.5% Class C fly ash and another plant uses approximately 3.0% slag as processing additions. The Illinois DOT specification limits the amount of fly ash and slag that may be used as a replacement for portland cement in concrete. The limits are 20% fly ash and 25% slag (by mass of total cementitious material) and these amounts would not include any fly ash or slag used as a processing addition in the manufacture of the portland cement.

Canadian Standards Association CSA A5-98, "Portland Cement," allows the use of processing additions ". . . provided that such materials, in the amounts used, have been shown to comply with ASTM Standard C 465." CSA A5 also permits up to 5% limestone to be added to both normal Type 10 and high-early-strength Type 30 cements. Such limestone must be of a suitable quality for manufacturing portland cement clinker. Processing additions meeting ASTM C 465 may also be used in the manufacture of blended cements to CSA A362-98, "Blended Hydraulic Cement."

The European standard (EN 197-1, 2000) allows use of additives, which are materials added to improve the manufacture or properties of cement, e.g. grinding aids, in amount limited to 1% by weight of cement. The European standard allows, for all cement types, up to 5% addition by weight of the cement "nucleus," excluding calcium sulfate and additives (i.e., the clinker) of minor additional constituents (Moir, 1994). The specification does state that these materials may not appreciably increase the water demand of the cement, impair the resistance of the cement or mortar to deterioration, or reduce the corrosion protection of the reinforcement (EN 197-1, 2000).

Moir (1994) notes that cements with minor additional constituents have performed satisfactorily in many European countries for many years (Table 1).

Minor additional constituents have been permitted up to 5% in the Sri Lankan standards since 1997. This standard is, in general, close to British Standards.

The cement standard used in South Africa (SABS 471, 1971) before the adoption of the current EN-based standard, allowed the use of up to 5% "inorganic mineral addition" without declaration of their amount or type. Cement producers could therefore change the amount or type of such material at will. The materials most commonly used as additives were fly ash (equivalent to Class F) and slag.

Bogue Calculation

The composition of cement can be determined by various analytical tests (AASHTO T 105). The results from these chemical tests can be used to calculate the *potential* amount of the major clinker compounds, C_3S, C_2S, C_3A, and C_4AF, by what is known as the Bogue calculation. The term "potential" is used because the equations are developed based on work on the relevant phase diagrams with the assumption that the clinker minerals are formed under ideal equilibrium conditions (Bogue, 1929). These assumptions are not always quite fulfilled; and the calculated content of the major clinker compounds may differ

Table 1. European countries where standards allow use of minor additional constituent (After Moir, 1994).

Country	Level %	Specified materials
Austria	≤ 5	
Belgium	≤ 5	
Denmark	≤ 5	
France	≤ 3	Limestone
Germany	≤ 5	Inorganic mineral material (slag, trass or partially burnt raw material used in the production of clinker)
Greece	≤ 3	Limestone, pozzolan, slag, etc.
Luxembourg	≤ 3	Inorganic material which may be pozzolan of limestone
Netherlands	≤ 5	
Norway	≤ 5	
Portugal	≤ 5	
Spain	1-5	
Sweden	≤ 5	
Switzerland	≤ 1	Fly ash
United Kingdom	≤ 5	Granulated blast furnace slag, natural pozzolans, fly ash, or filler.

from the actual amount observed in cement clinker by either microscopical examination or by X-ray diffractometry (Taylor, 1997; Lawrence, 1998). The potential compounds are defining characteristics in classification and specification limits of AASHTO M85.

Using the oxide composition of a cement for the Bogue calculation is meaningless when the system is a mixture of portland cement clinker and another material with a different mineralogy, so there is a question regarding the applicability of using this calculation for cements containing processing additions.

One method of addressing this concern would be to determine the clinker minerals in the cement directly, for instance, using quantitative x-ray diffraction methods (NIST, 2003). Another approach would be to modify the calculation to take into account the composition and quantity of the processing addition. This would require a test method for verifying the type and amount of addition in the cement as discussed below. A third approach is to rely simply on limits imposed on the oxide composition of the cement, rather than on the potential clinker compounds, although this is in principal no different from using calculated Bogue values.

Verification of Processing Addition Dosage

A concern for some users of a cement containing processing addition is verifying the amount and type of processing addition that has been used.

ASTM C 465 requires that the producer provide information about the type and amount of processing addition used, and a test method for verification of this information. In practice, such a determination is likely to be difficult because materials such as limestone, slag, and fly ash, do not have specific flags that would uniquely identify them chemically, and microscopic determination would not be sufficiently quantitative at the low doses involved.

The standards for cement have various limits on chemical composition that may help to address this concern. For instance, a limit is imposed on loss-on-ignition and insoluble residue; the loss-on-ignition limits the amount of limestone or cement kiln dust which can be added, while the insoluble limit may restrict use of lower purity limestone or cement kiln dust.

Effects on Cement Characteristics

Dilution of the Cement

A completely inert powder would be expected simply to act as a diluent with respect to the chemical reactions of the cement. For blended cements, any diluting effect would be observed as a decrease in early strengths. Müller-Pfeiffer et al. (2000) found that for cements with 10% slag or limestone, 2-day compressive strengths of mortars were close to the values that would be expected due to simple dilution of the clinker. A similar cement made with a fly ash had lower strength. For all cements, the 28-day strengths were above those expected from simple dilution. In practice, additional grinding will often be used to correct for the dilution, resulting in properties similar to those of the original cement at early ages. Hawkins (2003) investigated cements with 0, 3, 5, and 8% limestone interground with clinker to a constant residue on a 325-mesh. He found that the strengths were independent of limestone content. Livesey (1991) found that cement with 5% limestone behaved similarly to cement without limestone in concrete made to a constant slump. However, the limestone cements showed accelerated early hydraulic activity. Results from 5-year tests indicate that performance of cements with 5% limestone is, overall, indistinguishable from that of cements without addition (Matthews, 1994).

Particle Size Distribution, Grinding/Activation

Kenai et al., (2004) correlated the physical and mechanical properties of mortar and concrete with the level of replacement by limestone as well as the effect of fineness of both clinker and limestone. They tested several levels of limestone additions ranging from 0 to 35% and determined that use of a quality limestone (70% $CaCO_3$) with optimum levels and fineness of both limestone and clinker are the prerequisites to achieve best results for the short- as well as long-term performances.

Kumar et al. (2004) noted that the strength properties of cement largely depend on fine grinding of blended cement constituents. Using a slag-cement blend as the model, they used controlled grinding to mechanically activate the constituents, namely the clinker and the slag. In controlled particle size distribution cements, relatively low hydraulic activity of slag was compensated by increased reactivity of clinker due to increased fineness. It was also reported that for the same incremental increase in fineness, slag fineness gave better compressive strength than clinker. Tsivilis et al. (2000, ref 115) also concluded that the fineness of clinker and limestone was strongly connected with the limestone content and the fineness of the cement.

Bouzoubaa et al. (1999) reviewed the production of blended fly ash cements. They particularly discussed the production of blended fly ash cements by grinding portland cement clinker and fly ash together versus separate grinding followed by blending. Particular references were made to the optimum grinding processes and the effect of grinding fly ashes on their physical and chemical properties. Zhang and Zhang (2004) concluded that the dense packing of fine slag powder and cement improved the properties of slag cement. Slag powders ground to different particle size distribution and mixed with portland cement at different proportions were tested for performance to prove the point.

Babaian et al. (2003) employed mechano-chemical activation to develop cementitious materials from a combination of cement with cement kiln dust and fly ash. Various grinding regimes were used to activate the material. Vibratory grinding was found more effective than the ball mill grinding. Activation was confirmed through X-ray diffraction analysis and no correlation was found between activation and the mean particle size of the material. Properties tested including particle size distribution, initial time of set, heat of hydration, and compressive strength.

Rudert et al. (2005) also reported that the fineness affects the hydraulic reactivity of the ground granulated blast-furnace slag (GGBFS) considerably. The GGBFS was separated into different fineness classes by grinding and/or air separation. The particle size distributions were determined by laser analysis and the specific surface by means of BET nitrogen adsorption. The presence of GGBFS with a grain size >20 µm greatly decreased the reactivity, whereas the <5 µm fraction played an important role in hydration. To obtain a positive effect on strength increase of up to 28 days, GGBFS should be ground to at least smaller than 40 µm size particles.

Filler Effect

Inclusion of an inert very fine powder will significantly accelerate the hydration of alite and aluminates of the cement because the particles act as nucleation sites for the formation of the hydration products (Detwiler, 1988; Moir, 1994; Neville, 1997; Taylor, 1997). Another effect of finely divided additions is their action as fillers between the cement grains producing a denser paste and densifying the interfacial zone between the aggregate and cement paste (Taylor, 1997; Neville, 1997).

In a test series with additions of limestone of increasing fineness, Sprung and Siebel (1991) found a relatively greater increase in 2-day strengths compared with 28-day strengths, which they attributed to the filler effect of the limestone. Barker and Cory (1991) observed enhanced formation of calcium hydroxide at early ages in cements with addition of 5% and 25% limestone because the limestone provided nucleation sites for its growth. Improved strength development from additions of limestone, dolomite, and basalt with varying fineness was observed by Soroka and Setter (1977). Thus, inclusion of appropriate amounts of a very fine powder will enhance the performance of most cements, despite the dilution discussed above.

Pozzolanic Reaction

The net result of the reaction with a pozzolanic material and cement is the formation of calcium silicate hydrate by reaction between the silicate and aluminate of the material, and the calcium hydroxide from cement hydration, and water. Taylor (1997) explains the pozzolanic reaction as an attack on the silicate and aluminate of the pozzolanic material forming an intermediate amorphous product containing the alkalis from the cement pore solution. This will then (under the influence of the abundant supply of calcium ions) form calcium silicate hydrate and calcium aluminate hydrate.

A question applicable to pozzolanic processing additions is whether there would be sufficient calcium hydroxide left in the system for reaction with supplementary cementing materials added at the batch plant in the usual amounts. To illustrate the concept, if silica fume were to be used as a processing addition at a dose of 5% by mass of cement, it would have the *potential* to consume about half the calcium hydroxide available, leaving only half available for reaction with supplementary cementing materials. (Actual consumption may be less than this amount.) On the other hand, addition of small (1 to 5%) dosages of more likely materials such as slag or fly ash will not make significant changes to the amount of calcium hydroxide available for later reaction.

Latent Hydraulic Reactions

Slag is a latent hydraulic material, which means that it develops cementing properties when mixed with water and minimal amounts of an activator (Taylor, 1997). The activator can be calcium and alkali hydroxide and alkali from the hydrating cement. Production of cements with several main constituents, including slag, by inter-grinding or blending, is a well-established technology. Müller-Pfeiffer et al. (2000) show that the important parameters for cement performance are the hydraulic properties of the slag, its fineness in the blend, and the overall fineness of the cement. By carefully selecting these parameters, cement can be manufactured with performance comparable to cement with clinker and gypsum as the only constituents.

Effects on Fresh Concrete Properties

The properties of fresh concrete that are likely to be affected by the use of processing additions are discussed in the following paragraphs. Particular attention is paid to those properties that are of concern in placing, finishing, and maintaining concrete for highway structures. The test methods used to assess each of these properties are also discussed.

Change in Water Requirement

Water requirement is the amount of water needed to obtain a given workability for a given set of materials. In the field, workability is most commonly determined using the slump test. However, the variability of the slump test is likely to be greater than the small effects that may be expected at the dosages being considered in this work. The slump test is useful for the field operator to detect a distinct change in concrete, possibly due to changes in the materials from batch to batch. A more sensitive test is to determine the amount of water required to obtain a given flow in a mortar mixed to standard consistency. Small or indeterminate changes in mortar flow data due to the presence of processing additions is unlikely to translate to observable changes in concrete workability (Struble et al., 2001).

EN 197-1 requires that the minor additional constituents do not "significantly" modify water requirement. Slag and fly ash either do not change or marginally reduce water requirement at these dosages considered.

The amount of water required to wet the cement and the processing addition and to fill the space between particles will be strongly influenced by particle size distribution. The issue is complicated by the fact that processing additions are likely to have a different hardness from the cement with which they are being ground. This will affect the particle size distribution of the system. Softer processing additions, such as limestone, tend to be ground finer within a coarser clinker fraction while harder additions, such as slag, will be coarse within a finer clinker fraction. Sprung and Siebel (1991) noted that narrow particle size distributions generally lead to high water demands, while broader particle size distributions lead to lower water demands

Limestone, fly ash, and fine slag improve particle size distribution with respect to the rheological properties of pastes, mortars, and fresh concretes (Schmidt, 1992; Schmidt et al., 1993). In many cases, this will reduce the water demand, while water retention and workability of concrete may be improved, especially for interground additions (Schmidt et al., 1993). Based on work in Germany, Schmidt (1992) concluded that the water requirement for cement and concrete is sometimes significantly lower when using portland limestone cement or portland fly ash cements than when using plain portland cement.

The literature discusses the effect of the spherical shape of most fly ash particles on water requirement. Some, such as Yamakazi (1962), contend that the spheres act as ball bearings lubricating the mix. Others such as Bombled (1974) have reported that this effect is minimal and the reduction of water requirement of mixes containing fly ash is due to other mechanisms. It is possible that interground fly ash particles will lose their spherical shape and may therefore have a less beneficial effect on water requirement than if they were blended (Ravina, 1987). Effects such as fly ash-induced reduction of flocculation (Bombled, 1974) may not be affected by intergrinding.

Slump Loss

Slump loss is the rate at which slump is reduced with increasing time from mixing, either because of moisture loss, the loss of effectiveness of water-reducing admixtures, or early hydration. This is important to the concrete producer and the contractor because it is the slump at the time of placing, rather than at the time of mixing, that is of significance. The first two mechanisms are beyond the control of the materials supplier, although different cements may have varying rates of slump loss due to hydration effects. The differences will be primarily due to the reactions of the aluminate phases, which are, in turn, critically influenced by the sulfate system in the product. If a cement, whether or not it contains processing addition, is optimized for sulfate content, then there is likely to be only a limited effect on slump loss.

Air Entrainment

It is important that the amount of air-entraining admixture required to obtain a given air void system does not vary significantly because this property can only be determined after the concrete is mixed and cannot be rectified easily. Large variability would result in considerable wastage, or placement of potentially substandard concrete. AASHTO M 295 for fly ash allows up to 20% variation in admixture dosage as a uniformity requirement for fly ash. A similar requirement may be considered for cements containing processing additions.

Cements containing 3% interground slag require a marginally greater dosage of air entrainment admixture to achieve the same air content (Struble, 2001). For fly ash and slag used at higher amounts as supplementary cementitious materials, it has been found that the dosage might have to be increased 2 to 5 times, depending on content of residual carbon in the fly ash, the carbon activity, and the material fineness (Whiting and Nagi, 1998). Changes in cement alkali (from the clinker or processing addition) may influence air entrainment, particularly at low nominal alkali contents.

Heat Generation

The rate and amount of heat generated by hydration of a cement is of critical interest when constructing large concrete elements such as bridge piers because of the risk of cracking associated with temperature differentials between the concrete surface and interior. In pavements, the amount and rate of heat generated will influence the risk of cracking occurring. Large doses of pozzolanic materials are commonly used to reduce the heat of hydration of concrete.

No significant effects have been observed in studies of slag used at doses less than 5% (Struble, 2001). A review of the literature on limestone by Hawkins (2003) indicated that there were no simple overall trends related to use of limestone, except that increasing fineness tended to increase the rate and extent of temperature rise. Barker and Matthews (1989) reported a generally reduced peak rate of heat evolution with increasing quantities of limestone, with fineness having a strong influence. The effect was small at a 5% dosage.

Setting Time

Setting time is important in paving activities because it influences the interval of time before finishing and joint sawing activities can be carried out. Processing additions such as slag have been shown to cause small delays (less than 30 minutes) in setting time of cements (Struble, 2001). Batch intergrinding cement with 0, 3, 5.5, and 8% limestone to constant specific surface showed little effect on setting time, while grinding the cement to constant residue on a 325-mesh resulted in a reduction of setting time (Hawkins, 2003). Brookbanks (1989) concluded that cement containing 5% limestone had a marginally reduced setting time.

Effects on Hardened Concrete

The properties of hardened concrete likely to be affected by the presence of processing additions are discussed in the following paragraphs. Particular attention is paid to those properties of importance in constructing pavements and highway concrete structures.

Strength Development

The rate of strength development is important to contractors because there is an economic implication to the timing of finishing work, saw-cutting, removing molds, access to newly constructed surfaces, and opening to traffic. Particularly important milestones are the need for sufficient strength at 1 day to remove forms, 3 days to allow construction traffic onto new pavement, and 28 days to satisfy contractual requirements.

Strength is normally determined in compression using a cylinder test (AASHTO T 22) and for pavement concretes in flexure using a flexural beam test (AASHTO T 97). Flexural strength is required for pavements because of the need for a pavement to be able to resist flexural loads induced by traffic loading on slabs.

Mortar cube compression tests are more sensitive than concrete tests to changes in strength due to changing cement characteristics and are useful in assessing the effects of cement composition.

Work on concretes containing interground slag at 3% did not reveal measurable changes in concrete strength development (Struble, 2001). Hawkins' review of limestone work indicated that strength of concrete made with up to 6% limestone was not affected, even though the cements tested had not been optimized. Detwiler (1996) reported that a Type 10 cement containing 2.5% limestone gave slightly higher strengths than a similar Type 1 cement except when blended with fly ash. Livesey (1989) concluded that the strength class of Type I cements was not affected by the addition of up to 5% calcareous filler.

Shrinkage

Concrete shrinkage is a critical issue with respect to the risk of cracking, particularly at early ages. Increasing shrinkage will significantly increase the risk of cracking and may require changes to saw-cut spacing and practice.

Increasing the alkali content of a cement (Garci-Junger and Jennings, 2001) will increase the amount of shrinkage observed. This means that the inclusion of a highly alkaline processing addition such as cement kiln dust may increase shrinkage, depending on the dosages used. Adams and Race (1990) found increased drying shrinkage for Type I and Type II cements with 2 to 5% addition of limestone. Detwiler's data (1996) showed that 2.5% limestone did not affect shrinkage.

Alkali Silica Reactivity

Alkali-silica reaction (ASR) is reasonably well understood, and mitigation systems are available, many of which include the incorporation of supplementary cementing materials in the mix.

Hobbs (1983) reported that 5% dosages of fly ash, slag, or limestone did not increase the likelihood for deleterious expansion resulting from alkali silica reaction. However, small amounts (5–15%) of fly ash have been shown to increase expansion due to ASR under certain conditions. This effect is particularly noticeable with highly reactive opaline aggregates and with fly ashes of high alkali contents (Thomas et al., 1996). In addition, a processing addition with an alkali content, of 3% Na_2O_{eq}, if used at 5%, could increase the alkali content of a 0.60% low-alkali cement to 0.72%. However, if a cement with a specified alkali limit exceeds that limit due to the presence of the processing addition, then it is automatically rejected. Likewise, if a reactive aggregate is likely to be used, more active mitigation measures will be taken and the effect of the small amount of processing addition will be masked. An oxide analysis of the final cement composition should predict the potential effect of a processing addition on the risk of ASR in a mix.

Permeability

Permeability, a fundamental property of concrete, influences all forms of durability-related distress. All distress mechanisms in concrete are controlled by the access of moisture and/or gas. If the rate of water penetration through a concrete can be limited, the potential durability of that concrete will be significantly improved. A comparison of the permeability of samples made with different cements will provide a good indicator of their influence on potential durability. It is widely reported that appropriate use of supplementary cementing materials at dosages greater than those likely to be appropriate for process additions will reduce permeability in well-cured concrete. However, the small dosages associated with processing additions are not likely to influence permeability except when superfine, reactive materials such as silica fume are used. Hawkins' review (2003) indicated that with sufficient hydration, permeability of concrete containing limestone was improved or unaffected.

Chaniotakis et al. (2003) studied portland-limestone cements (PLCs) and concluded that the PLC concretes had comparable permeability characteristics with PCC; however, the limestone addition had an overall positive effect on the water permeability and the sorptivity of concrete. Six 100-mm-diameter concrete samples having varying limestone additions were tested using a modified commercial triaxial cell to determine N_2 gas absorption and water permeability.

Chloride Penetration

Chloride penetration is one of the more critical parameters for structures subjected to deicing. Chlorides do not directly affect concrete, but are catalysts in the corrosion of reinforcing steel. Relatively small amounts of chlorides will greatly accelerate corrosion of steel embedded in concrete (Taylor, 1999). Concretes exposed to such conditions are therefore required to resist the passage of chloride ions. This property may be improved with the inclusion of additional C_3A in processing additions because some chlorides that have penetrated the concrete will be chemically bound by C_3A and are thus not available for corrosion reactions. Otherwise, the rate of penetration will be largely controlled by the permeability as discussed above.

Freeze-Thaw and Salt Scaling

Both freeze-thaw and salt scaling deterioration are of interest in cold climates. The mechanisms are significantly controlled by the permeability of the concrete. Freeze-thaw damage is induced in concrete when water in the pores freezes and pressure builds up. Salt scaling damage occurs as a result of crystallization and expansion of salt solutions in the pores. The presence of processing additions is unlikely to influence these parameters except by influencing the permeability as discussed above. Specifications allow the use of air entrainers as functional additions in cement, but these are outside the scope of this study.

Detwiler (1996) reported that Type 10 cement containing 2.5% limestone and Type 1 cement performed comparably in ASTM C 666 and ASTM C 672 tests. Matthews (1989) concluded that freeze-thaw resistance of concrete was marginally reduced for concretes containing limestone, although the scatter was large relative to the observed variation.

Other Parameters

Creep is an important parameter in the design of prestressed concrete structures, but has been shown to be largely independent of cement composition and closely related to concrete strength.

Sulfate attack of concrete is related to the total C_3A in the cement with increasing C_3A resulting in increasing potential for deterioration. Class C fly ashes with high calcium content may add a small amount to the total C_3A in the cement; however the magnitude of this addition will be small. Soroka and Setter (1980) reported that time to cracking in sulfate exposure tests for mortars containing limestone (at dosages greater than 5%) was delayed. Increased fineness of the limestone increased the benefit imparted. Price reported (Price, 2004) that PLC concrete performs similarly to CEM I concrete of similar class, the only exception being resistance to aggressive sulfates.

Carbonation is the reaction of atmospheric carbon dioxide with calcium hydroxide hydration byproduct. This mechanism is relatively slow and extremely sensitive to the atmospheric relative humidity (Shima et al. 1989). The overall effect is to slightly densify the surface of the concrete, and more important, to lower the pH of the pore solution, markedly increasing the risk of corrosion of steel behind the carbonation front. Processing additions that consume the calcium hydroxide are therefore making the concrete theoretically prone to increased carbonation rates. The actual magnitude of this effect reported in the literature is varied for fly ash (Helmuth, 1987).

Moir and Kelham(1989) reported that incorporation of 5% limestone had no significant influence on carbonation of concrete.

The rate of carbonation is also controlled by the permeability of the concrete, which in general is improved by the presence of fly ash, thus mitigating the effect of the reduced calcium hydroxide content. The mechanism is of little interest in the United States, mainly because of the high humidity (and therefore low carbonation rates) experienced in the States that consume most concrete. Other damage mechanisms (e.g., freeze-thaw) tend to demand more attention than carbonation. The mechanism has not been considered as part of the matrix in this work because of the lack of importance attributed to it in highway concrete.

Interactions

Interaction with Alkalis

Increasing alkali content in the clinker will tend to accelerate reactions of pozzolanic processing additions and activate latent hydraulic constituents as discussed above. This may be considered beneficial, if it can be observed.

Interactions with Other Chemicals in Cement

Use of supplementary cementitious materials containing significant amounts of alkalis, C_3A, or sulfates may require modifications to the amount of SO_3 in the cement to maintain optimum setting and hydration characteristics (Roberts, 1995). Similar materials used at reduced rates as inorganic processing additions may influence optimum SO_3 requirement to a lesser extent. This is why a portion of our experimental program includes optimization of the sulfate content of each cement as described in the work plan. In the case of finely ground limestone (calcium carbonate) based processing additions, carboaluminates will be formed, potentially adding to early strength (Klemm and Adams, 1990; Neville, 1997; Taylor, 1997; Jackson, 1998). The limestone can also affect the SO_3 optimum of the cement (Camppiteli and Florindo, 1990).

Effect of Supplementary Cementing Materials

The most significant concern regarding supplementary cement materials is the availability of calcium hydroxide. Pozzolanic materials require that calcium hydroxide be available for reaction. If a significant amount of the calcium hydroxide is consumed by a processing addition, then less pozzolan can be effectively consumed in the final mixture. However, at the lower range of quantities of processing additions being considered, it is unlikely that a significant effect will be observed. At levels up to 10 percent, there may be a noticeable reduction in the amount of SCMs that can be consumed by the calcium hydroxide available.

Many state DOTs have set limits on the maximum amount of supplementary cementitious materials that may be included in a concrete. These limits are typically in the range of 15 to 20% for fly ash and 35 to 50% for slag. There is a concern that inclusion of a processing addition will cause a concrete supplier to unwittingly exceed these limits; for instance if 5% fly ash is included in a cement, and 20% more is added at the batch plant, then the limit of 20% will have been exceeded. There is also a concern that one pozzolanic material may be used as a processing addition, and another added at the batch plant, effectively resulting in a ternary mix with potentially unknown performance.

Effect of Chemical Admixtures

A processing addition may result in incompatible behavior with chemical admixtures. Based on the authors' experience, this is likely to occur only if the chemical balance of the cementitious system is close to instability before the small amounts of processing addition are used. Although several sources of admixture incompatibility are experienced in the field, the most likely problems will result from an imbalance between the sulfate demand of the C_3A and C_3S in the system with the sulfate availability (Roberts, 1995).

Survey of Processing Additions Use

Interviews with Cement Producers

The following questions were put to representatives of cement producers and cement associations in North America, Europe, Australia, and South Africa:

1. What type of processing additions are currently in use?
   - Limestone
   - Cement Kiln Dust
   - Fly ash (F or C)
   - Slag
   - Other (specify)
2. What levels are typically used?
3. Are there specifications (national or local) that limit the amount or type?
4. Is performance testing required or conducted to determine the effect of the addition?
5. When fly ash or slag are used—are the amounts included in calculations of the amount of ash or slag in the concrete mix?
6. Have there been any problems associated with the use of any particular type of processing addition?
7. Are there any materials not currently used as processing additions that may be used in the near future?

Eight cement producers in the United States, representing more than 50% of national production capacity, including the five largest companies and some smaller companies, were contacted. The requests were targeted at corporate, rather than plant, level to address a larger portion of the industry. Responses were received from six of those contacted: Lafarge, Holcim, Lehigh, Ashgrove, TXI, and Alamo. Two of these use processing additions in some of their plants. In one case, approximately 3% limestone is being used, in another 4.5% slag. In both cases, testing indicated that higher levels of these materials could be used in accordance with the criteria of ASTM C 465.

All of the producers indicated they had tested various mineral processing additions at some or all of their plants to determine what levels could be used in accordance with the criteria set forth in ASTM C 465. The results indicate that up to 5% fly ash, slag, or limestone could be used while complying with the performance requirements of ASTM C 465; however, higher levels would likely be prohibited by the chemical requirements of AASHTO M 85 (e.g., LOI and insoluble residue limits prevent higher amounts of limestone or fly ash being used).

Although there is an economic incentive to use mineral processing additions, there appears to be a number of reasons why some producers are reluctant to use them now. At least two producers cited the restrictions of AASHTO M 85 as the principal barrier to the wider use of processing additions. One producer argued that the intent of ASTM C 465 was that it be used to evaluate liquid/organic grinding aids and not mineral additions and that the appropriate specifications for cements containing inorganic additions other than clinker or gypsum were ASTM C 595 or ASTM C 1157. Another producer stated that it was not clear what was and what was not permissible under ASTM C 150 with respect to the limitations of ASTM C 465 and that the safest approach at this time was to avoid using mineral processing additions.

Most producers stated that they would seriously consider using limestone under what are now approved changes to ASTM C 150. Most of the producers believed that ASTM C 465 provides an acceptable protocol for assessing other mineral additions such as fly ash, slag, and cement kiln dust.

Two producers and cement associations were contacted in three different European countries, and responses were received from one producer and two associations. The following comments are based on the responses received.

Limestone, siliceous fly ash (<10% CaO), slag, and cement kiln dust are all used as minor additional constituents (MAC) in Europe and are counted as "cement" in calculations of water-to-cement ratio or cement contents. EN 197-1 does not permit the use of material as a MAC if it is already included as a main constituent of the cement. For instance, a cement classified as Portland-slag cement CEM IIB-S may contain between 21 to 35% GGBFS, but cannot contain slag as a MAC. However, the same cement could contain up to 5% fly ash as a MAC. Manufacturers in Europe indicated that the most common MAC was limestone.

In Europe, concerns have been expressed about using limestone as a MAC in cements that may be exposed to sulfates, because of the perception that increased availability of carbonate ions may increase the risk of thaumasite formation. A recent literature review published by the Portland Cement Association (Hooton and Thomas, 2002) concluded that the risk posed by low levels of limestone (i.e., <5%) was likely very small. In the United Kingdom, an expert group was formed to review the current state of the art and provide interim recommendations. The findings of this group were published in a report (Thaumasite Expert Group, 1999). In this report it is concluded that "On the basis of current information there is no evidence of increased risks of deterioration due to thaumasite expansion resulting from concrete mixes made with Portland cement incorporating limestone fillers, provided that the filler content is less than 5% by mass of cement."

Cement associations in South Africa or Australia were also contacted, but no responses were received.

Interviews with Users

Representatives of five State DOTs and the FHWA in the United States and two Provincial highway agencies in Canada were contacted by telephone or email to solicit their responses to the following list of questions (six responses were received):

1. What concerns do you have regarding the use of processing additions?
2. Are there any materials you feel should not be used as processing additions? (And, if so, why not?)
3. Should there be a limit on the quantity of inorganic material that can be used as a processing addition?
4. Are you aware of any problems in concrete that may have been attributed to the use of a processing addition?
5. In addition to the protocol set forth in ASTM C 465—what testing is required to evaluate a processing material?

Some users are skeptical about the motivation behind the use of certain processing additions. By definition, a processing addition should ". . . aid in the manufacture or handling . . ." of cement, but there is a perception that some additions merely reduce the clinker content of the cement and hence the cost of production.

Technical concerns about the performance of the finished cement appear to be largely limited to the effect of the processing addition on consistency and water demand, admixture compatibility, and concrete durability.

There is also a concern about non-disclosure of the type and amount of processing addition, and how this might affect concrete specifications that limit the amount of supplementary cementing materials, given that it may not be possible to account for all of the fly ash or slag in a mix if there is an unknown quantity of the material in the cement.

Users also mentioned the problem of calculating a Bogue composition from the chemical analysis of a cement that contains a significant quantity of inorganic material. (As an example of this problem, consider a Type I cement with 60% C_3S which falls outside of the maximum limit of 58% C_3S for a Type II cement according to AASHTO M 85-01. If 2 to 3% GGBFS were added to the cement and the Bogue composition recalculated using the chemical analysis of the finished cement, it is probable that the C_3S content would now be below the 58% limit.)

Finally, there is a concern that the language in ASTM C 465 permits "stacking" of the processing additions. Stacking is the practice of comparing a new material or dosage with another system already containing a processing addition. If this is repeated through several cycles, an unacceptable system may eventually be passed. Clause 4.3 requires that performance of cements containing the processing addition be compared with ". . . otherwise identical cements from the same source without the addition, or containing a processing addition which has been shown to comply with this specification using control cements without any additions . . .” In fact, the wording only permits the stacking of two different processing additions.

Agencies invoking AASHTO M 85 01 appear to be comfortable with a 1% maximum limit with the proviso that the addition also meets ASTM C 465. There is also a general feeling that the approach within ASTM C 465 is appropriate, but that additional tests and tighter acceptance limits may be required if the procedure were to be used to control the use of processing additions in the absence of a maximum limit. The additional tests should be selected to ensure that there is no adverse effect of the processing addition at the level used on either concrete durability or admixture compatibility. Furthermore, the effect of the processing addition on the water demand of the concrete should be evaluated, because the current protocol only determines the effect on cement consistency. ASTM C 465 does indirectly require the evaluation of the effect of the processing addition on the water demand of concrete as it requires cements with and without addition to be tested in concretes of equal slump by adjusting the water content. If the addition significantly increases the water demand, it is likely that the compressive strength requirement will not be met for the concrete containing the cement with the processing addition. It was also suggested that allowing the cement plus addition to increase the consistency of cement by up to 1.0%, change the setting time by up to 1 hour or 50%, or reduce the strength of concrete by 10% was not acceptable performance, and that “zero-tolerance” should be applied to the changes in these performance characteristics. (The limits in ASTM C 465 are close to zero tolerance if one considers the repeatability of the test methods used.)

In summary, most of the concerns raised by the users regarding an increase in the use of processing additions could be addressed by (1) including appropriate test methods and limits in ASTM C 465, (2) the producer reporting the type and amount of the addition, and (3) the producer reporting the Bogue composition or the chemical composition of the clinker component.

CHAPTER 3

Experimental Work

Research Approach

The objective of the research described in this report was to recommend potential improvements to specifications and test protocols to determine the acceptability of cements with processing additions.

The purpose was to assure highway engineers that the performance of concrete prepared with cements containing inorganic processing additions is not deleteriously affected by the presence of such materials. It was also necessary that the developed protocols would provide guidance on how much processing addition can safely be included in any given cement. Such protocols should not be cost prohibitive for suppliers of the materials.

The specific objective of the work outlined in this chapter was to develop the required information for either modifying or revising, on a rational basis, the current specification (AASHTO M 85 01) for portland cement with respect to processing additions. The objective was to recommend specifications that assure the users of a product that it delivers the performance and service life required, yet allow cement producers to take advantage of the environmental and economic benefits of processing additions.

Ideally, we would be able to analyze the chemistry of almost any inorganic material likely to be added to cement, and by reference to a model or database, predict the effect of such materials on concrete performance when added to cement. However, this is not feasible yet because of the complex interactions among the compounds in cements and cement additives. For instance, changing the alkali content of a given portland cement clinker will have a given set of direct effects, but linked to this alkali change will be a variation in the distribution of sulfate forms, with its own effects on the hydration behavior of the cementitious system. These effects are not simply associated with nor additive to each other, and while it is beyond current knowledge to be able to model them with sufficient reliability, the fundamental relationships are well enough understood that an experimental program can be devised to quantify the most prominent effects. The matter is further complicated by the wide range of supplementary cementing materials currently used in highway concrete and by the relatively small amounts of processing additions used in cement.

Because the number of combinations of potential cement clinkers, processing additions, and supplemental cementitious materials is essentially infinite, testing all combinations of materials to develop a database for evaluating potential changes either to specifications or to allowable combinations for field use would be impossible. A staged approach to reviewing processing additions is therefore necessary (Figure 2). The first stage of an evaluation of any material combination will be a review of the literature to establish known effects and trends. Experimental work on mortars can then be used to gain understanding of potential effects. Mortar tests are normally more sensitive to the effects of cement composition than concrete tests, and will act as an effective filter. Materials that "fail" the mortar tests can then be rejected. Materials that have no measurable or significant adverse influence on mortar properties can then be subjected to concrete performance tests.

The research team's approach was to test a matrix of clinkers, processing additions, processing addition dosage rates, supplemental cementing materials, and rates of addition of supplemental cementing materials. This experimental program was statistically designed to capture the boundary conditions that would be essential in developing rational specifications and test protocols. This thinking is reflected in Table 2. The matrix and materials selected are discussed in full in the next section.

Even with this matrix, it was not possible to test all possible combinations within the available budget. The work plan was based on completing mortar tests on a portion of the matrix and a selection of the most affected cements in concrete tests. The selection of mixtures to be tested was based on technical and statistical requirements, while remaining within budget

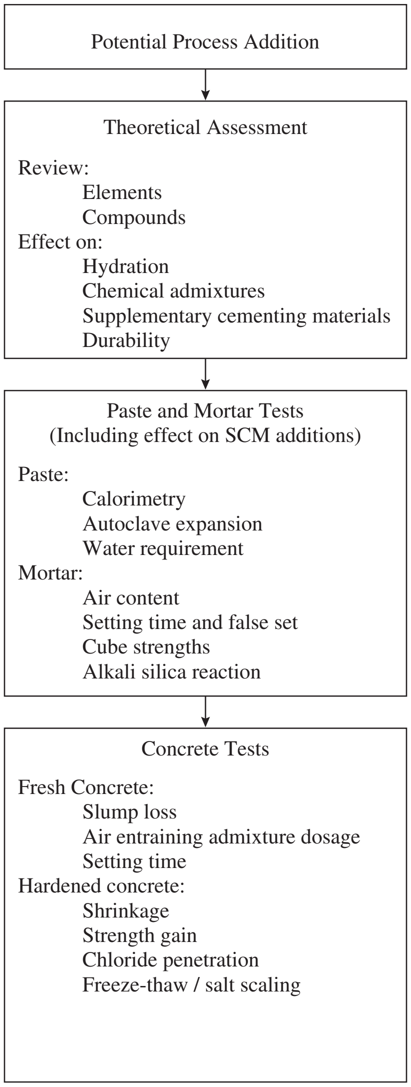


Figure 2. Flow diagram of experimental work.

constraints. The budget was based on a full suite of tests being conducted on 98 cements and 27 concretes. In the end, 109 cements and 28 concretes were tested.

Using the information derived from the tests conducted, the research team established a limiting content of processing addition that can be added to any clinker such that it would only be necessary to show compliance with the base cement specification parameters for approval of the processing addition. The research team members believe this is a viable approach, given that other worldwide specifications have adopted this methodology. For example, the European specification (EN 197-1, 2000) allows up to 5% percent minor additional constituents in any portland cement without any additional qualification other than meeting the baseline cement specification requirements. The research team members believe that an approach that brackets boundary conditions allowed the team to establish this value with an acceptable degree of confidence. Selection of the limit was based on a set of data focused on demonstrating that processing additions added below the threshold value do not degrade the performance of systems containing the resulting cement either in the fresh or hardened state.

Table 2. Outline of parameters investigated.

Materials	Number of Variables
Clinkers	4
Processing additions	3 + Control
Dosage rates of processing additions	3 + Control
Supplementary cementitious materials	3
Dosage rates of supplementary cementing materials	1

The other output of the experimental program is a rational test protocol for evaluating a particular processing addition at a specific dosage in excess of the selected limit up to an appropriate maximum. The research team developed recommendations for tests to which the portland cement containing the processing addition should be subjected, and a corresponding set of acceptance criteria. The test protocol recognized that the cement with the processing addition could be used in concrete containing supplementary cementing materials. This approach is based on that currently used in the ASTM C 465 protocol where the performance of a cement with a processing addition is compared with a control without a processing addition.

It is important that the test protocol developed in this project be validated, to avoid allowing an unacceptable material to be passed or an acceptable material to fail. In the interests of being conservative, at least until more data and experience are gathered, the intent will be to reject more potentially acceptable materials than accept potentially unacceptable materials. Validation is difficult because there is no test available to compare the proposed protocol against, except field experience. In addition, it is impossible to prove categorically that the protocol is reliable unless every possible material combination is tested. The potential set of unacceptable materials is very large, therefore, even a representative sample set will be large if it is to be rigorous, and testing such is outside the budget available. It is possible to run a number of random materials through the protocol, but the value of such a limited exercise may be questioned.

Ideally, a combination of materials that has been shown by field experience to be borderline/unacceptable should be subjected to the protocol. Such a combination could be tested under the protocol if such materials can be found with records of their field performance. In the long run, it is likely that the most rigorous validation of the protocol will be experience in the field over time.

Materials

The materials included in the test matrix are discussed first. The combinations of the selected materials tested are discussed in the section on Materials Combinations.

Clinkers

Three portland cement clinkers were obtained from commercial cement manufacturers.

One clinker (Clinker High) had high alkali (1.1%) and high C_3A (12%) contents, another (Clinker Low) had low alkali (0.44%) and low C_3A (1%) contents, and a third (Clinker Mid) had mid-range values (0.77% Na_2O_e, 8% C_3A). The mid-range clinker was also interground in the laboratory with limestone at 3.5% to form a fourth "clinker" (Clinker LS). This was done to address the performance of processing additions in cement with limestone additions. The limestone was obtained from the same source as the mid-range clinker.

The compositions of the three clinkers and the limestone are shown in Table 3.

Table 3. Clinker and limestone chemical composition.

Material	Clinker Low	Clinker Mid	Clinker High	Limestone (LS)
Analyte	**Wt. %**			
SiO_2	21.97	21.75	20.33	11.96
Al_2O_3	3.47	5.26	5.94	3.82
Fe_2O_3	4.61	3.65	2.22	1.50
CaO	65.02	65.00	64.20	43.48
MgO	2.5	1.15	2.23	0.86
SO_3	1.52	0.98	2.50	0.84
Na_2O	0.16	0.16	0.34	0.04
K_2O	0.44	0.93	1.15	0.72
TiO_2	0.31	0.20	0.31	0.14
P_2O_5	0.06	0.18	0.14	0.13
Mn_2O_3	0.11	0.24	0.21	0.14
SrO	0.05	0.20	0.08	0.13
Cr_2O_3	0.01	<0.01	<0.01	<0.01
ZnO	<0.01	0.02	<0.01	0.01
L.O.I.(950°C)	0.06	0.17	0.26	35.77
Total	100.30	99.88	99.90	99.51
Alkalis as Na_2O	0.44	0.77	1.10	0.51
Insoluble Residue	-	-	-	15.3
Calculated Compounds per AASHTO M 85.				
C_3S	63	56	57	-
C_2S	15	20	15	-
C_3A	1	8	12	-
C_4AF	14	11	7	-
Calculated Compounds				
Ca as $CaCO_3$	-	-	-	77.60
Mg as $MgCO_3$	-	-	-	1.79
$CaCO_3$+$MgCO_3$ as CO_2	-	-	-	35.05
L.O.I. / CO_2 Balance	-	-	-	1.02

Processing Additions

Cement Kiln Dust

The compounds in cement kiln dust that vary significantly include calcium oxide, sulfates, alkalis, and chloride. Chlorides are relatively simple to measure and limits exist in standards, therefore, chloride content was not used as a selection parameter. Given that elevated levels of calcium and sulfate are most likely to lead to problems, a cement kiln dust was sought with the highest measured calcium and sulfate contents. This material was found to be non-typical in that it was from a bypass-stream system, while cement kiln dust for use as a processing addition is most likely to be extracted from a kiln dust collector. On this basis, a cement kiln dust was obtained from a plant using a kiln dust collector. The oxide analysis of the material is shown in Table 4. The calcium content was approximately 40% and the sulfate content nearly 7%. XRD analysis indicated that the product comprised calcite ($CaCO_3$), quartz (SiO_2), dolomite ($CaMg(CO_3)_2$), sylvite (KCl), anhydrous calcium sulfate (CaSO4), and larnite (Ca_2SiO_4) in order of decreasing abundance.

Class C Fly Ash

Class C Fly Ash with a high C_3A content (>10%) was sought because this is the compound most likely to cause problems with stiffening and setting in the field. Such a material was obtained from a commercial supplier. The oxide analysis of the material is shown in Table 4.

Class F Fly Ash

Class F Fly Ash with a moderate to high Loss-On-Ignition content (LOI>4%) was obtained from a commercial supplier. The oxide analysis of the material is shown in Table 4. LOI was used as the selection parameter because this is the parameter most likely to cause problems with air entrainment in the field.

Slag

A Grade 100 slag was obtained from a commercial supplier. The oxide analysis of the material is shown in Table 4.

Sulfate System

The sulfate system used was a 50/50 mix of gypsum and plaster representing typical products made in cement manu-

Table 4. Cementitious materials chemical composition.

Material	Class C Fly Ash	Class F Fly Ash	Slag	Cement Kiln Dust	Gypsum
Analyte	Wt. %				
SiO_2	32.44	52.95	37.71	15.43	0.32
Al_2O_3	17.41	20.43	8.17	3.83	<0.01
Fe_2O_3	6.08	11.29	0.87	1.89	<0.01
CaO	26.8	4.24	38.9	40.67	32.52
MgO	7.9	0.99	10.9	2.58	0.35
SO_3	2.68	0.46	2.54	6.74	46.21
Na_2O	1.99	1.31	0.38	0.21	0.09
K_2O	0.36	1.87	0.34	4.52	<0.01
TiO_2	1.36	0.95	0.85	0.22	<0.01
P_2O_5	0.9	0.29	<0.01	0.14	0.02
Mn_2O_3	0.04	0.05	0.53	0.06	0.01
SrO	0.47	0.08	0.04	0.03	0.18
Cr_2O_3	0.02	0.03	0.01	<0.01	<0.01
ZnO	0.01	0.04	<0.01	0.06	<0.01
L.O.I.(950°C)	0.24	4.16	-1.04	22.16	20.78
Total	98.71	99.14	100.20	98.53	100.47
Alkalis as Na_2O	2.23	2.54	0.61	3.19	0.09
Insoluble Residue	25.8	84.8	0.4	14.2	0.3
C_3A	16.3	-	-	-	-

facturing. A fine gypsum was obtained from a commercial supplier. The chemical composition of the material is shown in Table 4. Plaster was prepared by heat treatment of a subsample of the gypsum, which was then blended with the gypsum to provide the required final blend. Each batch was tested using thermogravimetric analysis (TGA) to confirm that a sufficiently uniform material was produced between batches.

Supplementary Cementing Materials

The same fly ash and slag samples used as processing additions were used as supplementary cementing materials in the test program. Both of the fly ashes obtained were from commercial suppliers and are sold in the marketplace. The research team's experience is that high C_3A Class C ashes are available in the market for use in concrete. Although Class F with high LOI is meeting some market resistance, as demand for fly ash grows and availability of low-LOI material decreases, it is increasingly likely to be used for making concrete.

All of the supplementary cementing materials were used at the maximum dosages commonly allowed in specifications (25% for fly ash and 50% for slag). A control with no supplementary cementing material was used with each cement.

Aggregates

Aggregates were obtained from commercial sources. Fine aggregates for the mortar tests was as specified in the test methods discussed below. Aggregates for the concrete tests were calcareous crushed stone from Thornton, Illinois, and siliceous sand from McHenry, Illinois. The coarse aggregate was chosen as a local material that had a suitable water requirement that allowed the use of the selected cement and water contents in mixes with reasonable slump values.

Physical properties of the aggregates are given in Table 5.

Materials Combinations

The design of the experimental matrix was prepared on a statistical basis (factorial design) to obtain a maximum of information while remaining within budgetary constraints. A full matrix would have required preparation and testing of 184 cements. The statistician provided a matrix that limited the number of cements, while still allowing sufficient data to be obtained for subsequent analysis and interpretation. The need for repeating tests was considered, but was found to be superseded by the need to test as many of the points in the matrix as possible. No repeats were conducted unless data were observed to be anomalous.

Table 5. Aggregate properties.

Coarse Aggregate		Fine Aggregate	
Sieve No.	*Cum % Pass. Ind. Sieve*	*Sieve No.*	*Cum % Pass. Ind. Sieve*
1½	100.0	#8	93.2
1	98.0	#16	71.6
¾	79.0	#30	47.8
½	38.0	#50	14.6
3/8	18.0	#100	2.3
#4	4.0	#200	2.0
#200	2.0	Pan	0.0
		FM	2.70
Apparent Specific Gravity	2.8		2.8
Absorption, %	1.5		1.6
Dry Rodded Unit Weight, pcf	102.6		109.9

Table 6. Dosages of processing additions.

		Clinker Low	Clinker Mid	Clinker High	Clinker LS
Processing Addition	Dose Label	Mass %			
Class C Fly Ash	C	0.0	0.0	0.0	0.0
	S	1.4	1.4	1.2	-
	I	2.7	2.8	2.4	0.8
	U	4.0	4.1	3.7	2.1
Class F Fly Ash	C	0.0	0.0	0.0	0.0
	S	-	-	-	-
	I	1.4	1.4	1.3	-
	U	3.0	3.0	3.0	3.0
Slag	C	0.0	0.0	0.0	0.0
	S	2.5	2.5	2.5	2.5
	I	5.0	5.0	5.0	5.0
	U	7.5	7.5	7.5	7.5
Cement Kiln Dust	C	0.0	0.0	0.0	0.0
	S	2.5	2.5	2.3	0.8
	I	5.1	5.0	4.5	1.5
	U	7.5	7.5	6.9	4.0

Notes:

- Clinker Low, Mid, or High refers to the C_3A content of the clinker
- Dose labeling:
  - C – Control, no processing addition
  - S – Approximately half of dosage I
  - I – Maximum dosage to stay within prescriptive limits for LOI and Insoluble Residue
  - U – Dosage greater than prescribed to provide an outer bound. Dosage was based on exceeding the limiting parameter by 3 times the precision for the test method.
  - - – Dosage not used because calculated dosage was below 0.5%

Processing Additions Dosage

Selection of the dosages of processing addition in the test matrix was based on the current chemical limits of AASHTO M 85. The philosophy adopted was to add as much processing addition to each clinker as possible, while remaining within the specified limits for LOI and insoluble residue. Another dose, higher than this maximum, based on the precision of the analytical test methods, was also used in order to provide an outer bracket. The procedure for selecting dosages was as follows:

- A control with no processing addition was used for every clinker (Dose C).
- For each clinker/processing addition combination, a dosage (Dose I) was calculated based on the amount needed to reach either of the limits in AASHTO M 85 of 3% LOI or 0.75% insoluble residue.
- For each clinker/processing addition combination, another dosage (Dose U) was calculated based on the amount needed to reach either 3.3% LOI or 1.05% insoluble residue. These figures are based on adding three times the permissible variation for each test given in Table 1 of AASHTO T 105.
- Another dose (Dose S), if appropriate, was selected at a reasonable value of about half of that used in Dose I.

The amount of limestone in the fourth "clinker" was selected at 3.5% to stay within the AASHTO M 85 limits for LOI and insoluble residue and to allow sufficient "space" for a processing addition to be added.

Based on these parameters, the dosages of the different materials for each clinker are shown in Table 6. No dosage is shown for some of the combinations. This is because the calculated values are below 0.5% and are considered too low to be of use.

For the Class F Fly ash, the figures given under Dose Label "I" are those calculated for Label "U." This is because the values calculated for "I" were also very low and of questionable benefit to the program. The values given under label "U" express a higher value selected to provide an outside bound, even if the cements produced do not comply with the LOI and IR limits of current specifications.

The calculated values for the combinations containing slag were unreasonably high (in at least one case, 100%) because insoluble residue content of slag is very low and the LOI is negative. It is not uncommon for slag to gain weight with heating, thus exhibiting a negative LOI. The dosages for these combinations were therefore selected at 5% for the "I" set and 7.5% for the "U" set. These were similar to the maximum values determined using another material combination (cement kiln dust with Clinker "Low") and were not inconsistent with what may be considered as reasonable maxima without making these into "blended" cements.

Statistical Design of Processing Additions Combinations

The design of the experimental matrix was prepared on a statistical basis to obtain a maximum of information while remaining within budgetary constraints. The budget had been prepared on the basis of preparing and testing 99 "cements." A full matrix would have required preparation and testing of 160 "cements." During the process of research it was decided to increase the total matrix to 109 combinations.

The statistician was asked to provide a matrix that limited the number of cements, while still allowing sufficient data to be obtained for subsequent analysis and interpretation. The full statistician's report is given in Appendix B.

A fractional factorial design was used as the base design. The initial design was generated as if it were a full factorial of four levels of clinker, four levels of processing addition at three dosages each, and four levels of supplementary material. The full factorial would contain 192 combinations, 24 more combinations than in the original matrix.

From this design, a half fractional factorial using eight factors with two levels each was created. This produced a total of 128 (=256/2) combinations, of which those not in the original design were discarded leaving 84 combinations. There are no repeated combinations in this design. The combinations of factors are evenly spread over the levels of each factor.

Following analysis of the initial mortar and pastes tests, some additional combinations were tested in order to provide additional data points. The final matrix of tested combinations is given in Table 7 and Figure 3.

Selection of Cements for Concrete Tests

The matrix of systems chosen for testing in concrete mixtures was developed based on the following:

- Matrix should have a broad representation of materials without bias toward any given material.
- "U" dosages should not be used because they do not represent materials that comply with current specifications.

The matrix was also based on testing only systems without supplementary cementitious materials. This has the advantage that the matrix comprises a more statistically significant set of systems that can be tested for the remaining variables (clinker type, processing addition type, processing addition dosage). The effects of supplementary cementitious materials can be interpreted from the paste and mortar tests only. This is reasonable, based on experience that paste and mortar tests are normally more sensitive to changes in system composition than concrete tests, and the trends observed in the paste and mortar tests can be confirmed using the concrete tests conducted on systems made with supplementary cementitious materials.

Table 7. Cement combinations, from statistical design.

Combination number	Clinker	Processing Addition Type	Processing Addition Dosage	Supplementary Cementing Material
1	Low	None	C	None
2	Low	None	C	F Ash
3	Low	C Ash	S	None
4	Low	C Ash	S	Slag
5	Low	C Ash	I	C Ash
6	Low	C Ash	I	F Ash
7	Low	C Ash	U	C Ash
8	Low	C Ash	U	F Ash
9	Low	F Ash	I	None
10	Low	F Ash	I	Slag
11	Low	F Ash	U	None
12	Low	F Ash	U	Slag
13	Low	Slag	S	C Ash
14	Low	Slag	S	F Ash
15	Low	Slag	I	None
16	Low	Slag	I	Slag
17	Low	Slag	U	None
18	Low	Slag	U	F Ash
19	Low	Slag	U	Slag
20	Low	Cement Kiln Dust	S	None
21	Low	Cement Kiln Dust	S	Slag
22	Low	Cement Kiln Dust	I	C Ash
23	Low	Cement Kiln Dust	I	F Ash
24	Low	Cement Kiln Dust	U	C Ash
25	Low	Cement Kiln Dust	U	F Ash
26	Mid	None	C	C Ash
27	Mid	None	C	F Ash
28	Mid	None	C	Slag
29	Mid	C Ash	S	C Ash
30	Mid	C Ash	S	F Ash
31	Mid	C Ash	I	None
32	Mid	C Ash	I	Slag
33	Mid	C Ash	U	None
34	Mid	C Ash	U	Slag
35	Mid	F Ash	I	C Ash
36	Mid	F Ash	I	F Ash
37	Mid	F Ash	U	C Ash
38	Mid	F Ash	U	F Ash
39	Mid	Slag	S	None
40	Mid	Slag	S	Slag
41	Mid	Slag	I	C Ash
42	Mid	Slag	I	F Ash
43	Mid	Slag	U	None
44	Mid	Slag	U	C Ash
45	Mid	Slag	U	F Ash
46	Mid	Cement Kiln Dust	S	C Ash
47	Mid	Cement Kiln Dust	S	F Ash
48	Mid	Cement Kiln Dust	I	None

(continued on next page)

Table 7. (Continued).

Combination number	Clinker	Processing Addition Type	Processing Addition Dosage	Supplementary Cementing Material
49	Mid	Cement Kiln Dust	I	Slag
50	Mid	Cement Kiln Dust	U	None
51	Mid	Cement Kiln Dust	U	Slag
52	High	None	C	None
53	High	None	C	Slag
54	High	C Ash	S	C Ash
55	High	C Ash	S	F Ash
56	High	C Ash	I	None
57	High	C Ash	I	Slag
58	High	C Ash	U	None
59	High	C Ash	U	C Ash
60	High	C Ash	U	Slag
61	High	F Ash	I	C Ash
62	High	F Ash	I	F Ash
63	High	F Ash	U	C Ash
64	High	F Ash	U	F Ash
65	High	Slag	S	None
66	High	Slag	S	Slag
67	High	Slag	I	C Ash
68	High	Slag	I	F Ash
69	High	Slag	U	C Ash
70	High	Slag	U	F Ash
71	High	Cement Kiln Dust	S	C Ash
72	High	Cement Kiln Dust	S	F Ash
73	High	Cement Kiln Dust	S	Slag
74	High	Cement Kiln Dust	I	None
75	High	Cement Kiln Dust	I	Slag
76	High	Cement Kiln Dust	U	None
77	High	Cement Kiln Dust	U	Slag
78	LS	None	C	None
79	LS	None	C	C Ash
80	LS	C Ash	I	C Ash
81	LS	C Ash	I	F Ash
82	LS	C Ash	I	Slag
83	LS	C Ash	U	C Ash
84	LS	C Ash	U	F Ash
85	LS	F Ash	U	None
86	LS	F Ash	U	Slag
87	LS	Slag	S	C Ash
88	LS	Slag	S	F Ash
89	LS	Slag	I	None
90	LS	Slag	I	Slag
91	LS	Slag	U	None
92	LS	Slag	U	Slag
93	LS	Cement Kiln Dust	S	None
94	LS	Cement Kiln Dust	S	Slag

Table 7. (Continued).

Combination number	Clinker	Processing Addition Type	Processing Addition Dosage	Supplementary Cementing Material
95	LS	Cement Kiln Dust	I	C Ash
96	LS	Cement Kiln Dust	I	F Ash
97	LS	Cement Kiln Dust	I	Slag
98	LS	Cement Kiln Dust	U	C Ash
99	LS	Cement Kiln Dust	U	F Ash
100	Mid	None	C	None
101	LS	C Ash	S	None
102	Low	F Ash	S	C Ash
103	Mid	F Ash	S	Slag
104	High	F Ash	S	None
105	Low	F Ash	U	F Ash
106	Mid	F Ash	U	None
107	High	F Ash	U	Slag
108	LS	F Ash	U	C Ash
109	LS	F Ash	U	F Ash

Notes:

- Clinker Low, Mid, or High refers to the C_3A content of the clinker
- Dose labeling:
  - C – Control, no processing addition
  - S – Approximately half of dosage I
  - I – Maximum dosage to stay within prescriptive limits for LOI and Insoluble Residue
  - U – Dosage greater than prescribed to provide an outer bound. Dosage was based on exceeding the limiting parameter by three times the precision for the test method.

All 18 mixtures were tested using a "structural" mix design. Nine of the mixtures, those made with "Mid" and "High" clinkers were tested using a "paving" mix design. This selection was made based on the desire to compare two different clinkers in these tests.

The matrix of systems used in concrete tests is shown in Table 8 and Figure 4.

Preparation of Materials Combinations

As described above, some of the commercial gypsum ($CaSO_4.2H_2O$) was heat-treated to form plaster ($CaSO_4.\frac{1}{2}H_2O$) and blended with gypsum to produce a nominal 1:1 mixture of plaster and gypsum, to model a typical sulfate system in commercially available cements.

The cement clinkers were ground using a 40-lb mill, with a nominal sulfate system dose equivalent to 1% SO_3 and with processing additions at the planned levels, to a fineness of between 350 and 400 m^2/kg. The recorded Blaine fineness values of the cements are given in Table 9.

		PA	None	C Ash			F Ash			Slag			CKD			No. of Tests	
		PA Dose	C	S	I	U	S	I	U	S	I	U	S	I	U		
		SCM															
Clinker	Low	No SCM	1	3			X	9	11		15	17	20			6	27
		C Ash			5	7	X	102		13				22	24	7	
		F Ash	2		6	8	X		105	14		18		23	25	8	
		Slag		4			X	10	12		16	19	21			6	
	Mid	No SCM	100		31	33	X		106	39		43		48	50	8	28
		C Ash		26	29		X	35	37		41	44	46			7	
		F Ash	27	30			X	36	38		42	45	47			6	
		Slag	28		32	34	X	103		40				49	51	7	
	High	No SCM	52		56	58	X	104		65				74	76	7	28
		C Ash		54		59	X	61	63		67	69	71			7	
		F Ash		55			X	62	64		68	70	72			6	
		Slag	53		57	60	X		107	66			73	75	77	8	
	LS	No SCM	78	X	101		X	X	85		89	91	93			6	26
		C Ash	79	X	80	83	X	X	108	87				95	98	7	
		F Ash		X	81	84	X	X	109	88				96	99	7	
		Slag		X	82		X	X	86		90	92	94	97		6	
No. of Tests				6	12	9	0	12	18	8	8	10	9	9	8		
				27			30			26			26				

Numbers are the combination number used to identify each mixture. Blank cells were not tested. Cells labeled "X" were excluded from the matrix because PA dosages were too low to be significant.

Figure 3. Paste and mortar test matrix.

Table 8. Selection of cements for concrete testing.

Combination	Clinker type	Processing Addition type	Processing Addition Dosage, %
1	Low	None	0
3	Low	C Ash	1.4
9	Low	F Ash	1.4
15	Low	Slag	5
20	Low	Cement Kiln Dust	2.5
31	Mid	C Ash	2.8
39	Mid	Slag	2.5
48	Mid	Cement Kiln Dust	5
52	High	None	0
56	High	C Ash	2.4
65	High	Slag	2.5
74	High	Cement Kiln Dust	4.5
78	LS	None	0
89	LS	Slag	5
93	LS	Cement Kiln Dust	0.8
100	Mid	None	0
101	LS	C Ash	0.8
104	High	F Ash	1.3

All combinations tested in structural mixtures. Shaded rows are those combinations used in paving type mixtures as well.

The required amount of sulfate material to optimize each of the combinations of cements was determined in accordance with ASTM C 563. The sulfate contents of these cements are shown in Table 9. The particle size distribution of these cements was determined using a Malvern laser diffractometer. The data are provided in Table 10 and Figure 5.

The required supplementary cementitious materials were then blended with the clinkers and gypsum to form the final 109 cements tested.

The final cements with SO_3 levels higher than permitted by ASTM C 150 were tested in accordance with ASTM C 1038 to ensure that excessive expansions were not observed. All the systems tested had expansions below the specified maximum, as shown in Table 11.

Tests Conducted

The experimental work was conducted in a series of phases, which are discussed in turn.

Materials Combinations Characterizations

The cements with zero (Label C) or the highest (Label U) processing addition dosages and no supplementary cementing materials were examined microscopically to evaluate variations in particle shape in the fraction larger than 20 µm. The images are presented in Appendix C.

X-Ray Fluorescence (XRF) analyses were conducted on four of these cements and on the fractions of these cements greater than 20 µm. The data are shown in Table 12.

PA	None	C Ash			F Ash			Slag			Cement Kiln Dust		
PA Dose	C	S	I	U	S	I	U	S	I	U	S	I	U
Clinker													
Low	1	3		Y	X	9	Y		15	Y	20		Y
Mid	100		31	Y	X		Y	39		Y		48	Y
High	52		56	Y	X	104	Y	65		Y		74	Y
LS	78	X	101	Y	X	X	Y		89	Y	93		Y

Numbers are the combination number used to identify each mixture. Blank cells were not tested. Cells labeled "X" were excluded from the matrix because PA dosages were too low to be significant. Cells labeled "Y" were excluded from the matrix because these cements did not comply with current limits on chemical composition.

Figure 4. Concrete test matrix.

Tests on Cement Pastes

It is intended that in the protocol, paste and mortar tests are conducted as a screening exercise on the selected cements. In optimizing the sulfate content of all of the cements, it is likely that effects (such as false set) on fresh properties of concretes containing such materials will be minimized.

The following tests were conducted on pastes made from the 109 cements:

- Conduction calorimetry. In this test, the cement sample and the water (w/cm = 0.5) were temperature equilibrated at 23°C before mixing. They were then mixed in the calorimeter, and the heat evolution resulting from hydration was monitored as the sample was held at 23°C. The results are illustrated in Appendix D.
- Autoclave Expansion (AASHTO T 107). Results are given in Table 11.
- Water requirement was assessed by recording the water-cementitious materials ratio required to achieve a "normal consistency" (AASHTO T129) when making the Vicat specimens (AASHTO T 131). Results are given in Table 11.
- Setting time (AASHTO T 131). Results are given in Table 11.

Some tests were repeated because the data appeared to be outliers.

Tests on Mortars

All of the 109 cements were evaluated in the following mortar tests:

- Cube strength development (AASHTO T 106) at 1, 3, 7. and 28 days.
- Water requirement was assessed by recording the flow achieved with a fixed water content as required for preparing the cubes for strength testing by AASHTO T106.
- Air content (AASHTO T 137).
- Early stiffening (AASHTO T 185).
- Shrinkage (ASTM C 596).
- Alkali silica reactivity (ASTM C 227 for 28 days, using highly reactive Albuquerque aggregate).

Results are given in Table 11.

Some tests were repeated because the data appeared to be outliers.

Tests on Concretes

The selected 18 cements were used to prepare 27 concrete mixes. Eighteen mixes were based on the following parameters typical of 5000-psi structural concrete. A fixed water/cementitious ratio was used with no water reducing admixture.

- Cementitious materials content—658 lb/cu yd.
- Water/cementitious ratio—0.40.
- Maximum coarse aggregate size—1-inch.
- Air content—6 ± 1%.

Air content was controlled by using a single commercial vinsol-based air-entraining admixture. Slump was not controlled, but measured and reported.

Nine of the combinations were repeated in concrete mixtures based on a paving type mixture:

- Cementitious materials content—564 lb/cu yd.
- Water/cementitious ratio—0.42.
- Maximum coarse aggregate size—1-inch.
- Slump—1 to 2 inch.
- Air content—6 ± 1%.

The following tests were conducted on samples taken from all the concrete mixtures:

- Slump loss every half hour until zero slump (AASHTO T119).
- Rate of change of flow of mortar extracted from the concrete were measured on a flow table.

Table 9. Details of cements prepared before addition of supplementary cementing materials.

Combination Number	Clinker Type	Processing Addition Type	Processing Addition Dosage, %	Optimum sulfate, %	Blaine, m^2/kg
1-2	Low	None	0.0	2.37	392
3-4	Low	C Ash	1.4	2.77	379
5-6	Low	C Ash	2.7	3.02	379
7-8	Low	C Ash	4.0	2.87	374
9-10	Low	F Ash	1.4	2.88	376
11-12	Low	F Ash	3.0	3.05	386
13-14	Low	Slag	2.5	2.89	368
15-16	Low	Slag	5.0	2.83	397
17-19	Low	Slag	7.5	2.76	385
20-21	Low	Cement Kiln Dust	2.5	2.84	385
22-23	Low	Cement Kiln Dust	5.1	2.65	371
24-25	Low	Cement Kiln Dust	7.5	2.32	370
26-28	Mid	None	0.0	3.13	376
29-30	Mid	C Ash	1.4	3.21	377
31-32	Mid	C Ash	2.8	3.24	368
33-34	Mid	C Ash	4.1	3.65	380
35-36	Mid	F Ash	1.4	3.64	382
37-38	Mid	F Ash	3.0	3.00	389
39-40	Mid	Slag	2.5	2.78	379
41-42	Mid	Slag	5.0	2.84	367
43-45	Mid	Slag	7.5	3.05	403
46-47	Mid	Cement Kiln Dust	2.5	3.42	380
48-49	Mid	Cement Kiln Dust	5.0	3.07	378
50-51	Mid	Cement Kiln Dust	7.5	2.58	385
52-53	High	None	0.0	4.50	371
54-55	High	C Ash	1.2	3.81	367
56-57	High	C Ash	2.4	4.34	378
58-60	High	C Ash	3.7	4.44	398
61-62	High	F Ash	1.3	4.17	366
63-64	High	F Ash	3.0	3.82	366
65-66	High	Slag	2.5	4.42	401
67-68	High	Slag	5.0	4.52	368
69-70	High	Slag	7.5	4.20	364
71-73	High	Cement Kiln Dust	2.3	3.53	379
74-75	High	Cement Kiln Dust	4.5	3.78	367
76-77	High	Cement Kiln Dust	6.9	4.08	368
78-79	LS	None	0.0	4.18	377
80-82	LS	C Ash	0.8	3.32	388

(continued on next page)

Table 9. (Continued).

Combination number	Clinker type	Processing Addition type	Processing Addition Dosage, %	Optimum sulfate, %	Blaine, m^2/kg
83-84	LS	C Ash	2.1	2.88	378
85-86	LS	F Ash	3.0	3.82	375
87-88	LS	Slag	2.5	3.29	368
89-90	LS	Slag	5.0	3.54	375
91-92	LS	Slag	7.5	3.03	378
93-94	LS	Cement Kiln Dust	0.8	1.89	381
95-97	LS	Cement Kiln Dust	1.5	3.20	388
98-99	LS	Cement Kiln Dust	4.0	2.37	382
100	Mid	None	0.0	3.25	387
101	LS	C Ash	0.8	2.90	373
102	Low	F Ash	1.4	1.92	385
103	Mid	F Ash	1.4	3.18	375
104	High	F Ash	1.3	4.17	386
105	Low	F Ash	3.0	2.69	370
106	Mid	F Ash	3.0	2.90	387
107	High	F Ash	3.0	4.59	378
108-109	LS	F Ash	3.0	3.47	368

Table 10. PSDs of cements (prior to addition of SCMs).

Particle Size		Sample ID									
		Percentage Passing									
(μm)		1-2	3-4	5-6	7-8	9-10	11-12	13-14	15-16	17-19	20-21
0.1		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1.0		6.8	6.7	6.7	6.8	6.8	6.8	6.1	6.7	6.5	6.2
3.2		22.6	22.3	22.3	22.5	22.3	23.0	21.1	22.3	21.6	20.6
7.1		38.1	37.4	37.6	38.3	37.4	39.1	36.1	37.9	36.8	34.9
10.0		45.0	44.0	44.3	45.3	44.1	46.4	42.6	44.8	43.5	41.1
31.7		75.3	73.6	74.3	75.7	74.1	78.3	72.0	75.6	73.9	70.9
44.8		86.1	84.7	85.2	86.0	85.1	88.5	83.5	86.4	85.0	82.7
89.3		99.3	99.1	99.1	99.1	99.1	99.8	98.6	99.4	99.1	98.1
200.0		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	99.5
399.1		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	99.8
632.5		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Particle Size		Sample ID									
		Percentage Passing									
(μm)		22-23	24-25	26-28	29-30	31-32	33-34	35-36	37-38	39-40	41-42
0.1		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1.0		5.9	5.4	5.9	5.6	5.6	5.7	5.8	5.7	5.6	5.4
3.2		19.8	18.6	20.5	19.7	19.6	19.7	20.2	20.4	19.6	18.9
7.1		33.9	32.7	36.7	35.6	35.8	35.7	36.7	37.2	35.5	34.4
10.0		40.3	39.2	45.1	44.0	44.2	44.1	45.3	45.7	43.8	42.5
31.7		72.0	71.4	80.7	80.3	80.2	80.5	82.0	81.6	80.0	78.6
44.8		83.9	83.6	89.9	89.6	89.8	89.9	91.1	90.8	89.3	88.6
89.3		98.7	98.9	99.4	99.5	99.5	99.2	99.4	99.3	98.7	99.2
200.0		100.0	100.0	100.0	100.0	100.0	99.7	99.6	99.7	99.4	100.0
399.1		100.0	100.0	100.0	100.0	100.0	99.9	99.9	99.9	99.8	100.0
632.5		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Particle Size		Sample ID									
		Percentage Passing									
(μm)		43-45	46-47	48-49	50-51	52-53	54-55	56-57	58-60	61-62	63-64
0.1		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1.0		6.1	5.7	5.6	5.7	5.5	5.4	5.5	5.9	5.3	5.3
3.2		21.2	19.7	19.6	19.7	19.0	19.1	19.3	20.6	18.6	18.8
7.1		37.9	35.4	35.2	35.5	34.5	35.0	35.1	37.3	34.2	34.3
10.0		46.3	43.8	43.1	43.6	41.8	42.5	42.7	45.0	41.8	41.9
31.7		81.6	80.3	78.7	79.8	74.0	75.8	76.4	78.4	75.3	75.9
44.8		90.8	89.8	88.2	89.6	84.6	86.6	87.1	88.8	86.1	86.7
89.3		99.8	99.6	98.0	99.6	97.8	99.1	99.4	99.8	98.7	99.1
200.0		100.0	100.0	98.8	100.0	99.3	100.0	100.0	100.0	99.6	100.0
399.1		100.0	100.0	99.7	100.0	99.8	100.0	100.0	100.0	99.9	100.0
632.5		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Particle Size		Sample ID									
		Percentage Passing									
(μm)		65-66	67-68	69-70	71-73	74-75	76-77	78-79	80-82	83-84	85-86
0.1		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1.0		5.6	5.4	5.1	5.6	5.3	5.3	5.6	6.2	6.1	5.9
3.2		19.9	19.3	18.4	19.5	18.3	18.6	19.4	20.6	20.7	20.4
7.1		36.1	35.6	34.2	35.0	33.0	33.6	34.2	35.0	36.0	35.8
10.0		43.7	43.2	41.7	42.4	40.1	40.7	41.7	42.3	43.5	43.5
31.7		77.1	75.8	74.7	75.5	72.6	72.8	75.6	76.0	77.4	78.8
44.8		87.3	86.0	85.5	86.4	83.6	83.8	85.6	85.9	87.2	88.5
89.3		98.9	98.5	98.7	99.1	97.5	98.0	97.9	98.3	98.4	98.9
200.0		99.6	99.7	100.0	100.0	99.1	99.7	99.7	100.0	99.8	99.6
399.1		99.9	99.9	100.0	100.0	99.7	99.9	99.9	100.0	99.9	99.9
632.5		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Particle Size		Sample ID									
		Percentage Passing									
(μm)		87-88	89-90	91-92	93-94	95-97	98-99				
0.1		0.0	0.0	0.0	0.0	0.0	0.0				
1.0		5.7	5.8	5.8	6.0	6.1	5.8				
3.2		20.0	20.1	20.1	20.5	20.4	19.7				
7.1		34.8	35.1	35.3	35.4	35.2	34.5				
10.0		42.3	42.7	43.0	42.9	42.5	41.9				
31.7		77.1	77.8	78.1	77.1	76.0	76.7				
44.8		86.8	87.7	87.9	86.6	85.8	86.8				
89.3		97.9	98.5	98.9	97.6	97.3	98.2				
200.0		99.2	99.6	100.0	98.8	98.7	99.3				
399.1		99.7	99.9	100.0	99.6	99.5	99.8				
632.5		100.0	100.0	100.0	100.0	100.0	100.0				

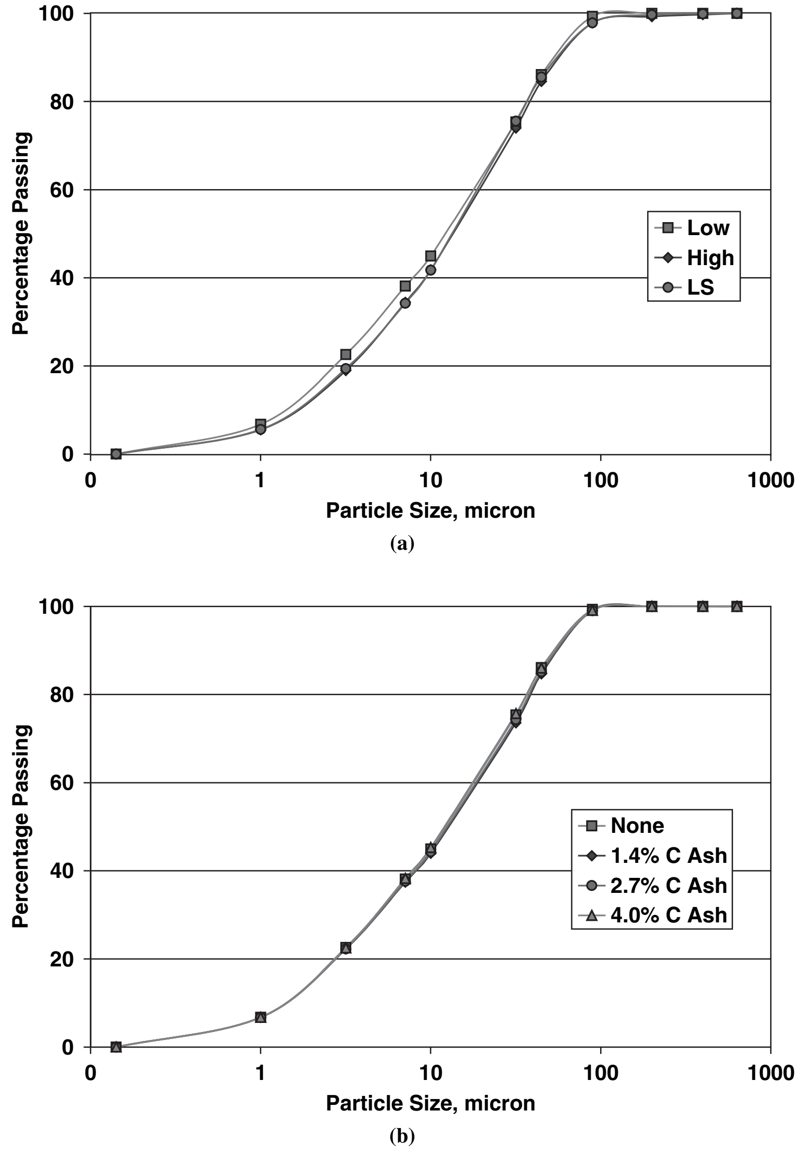
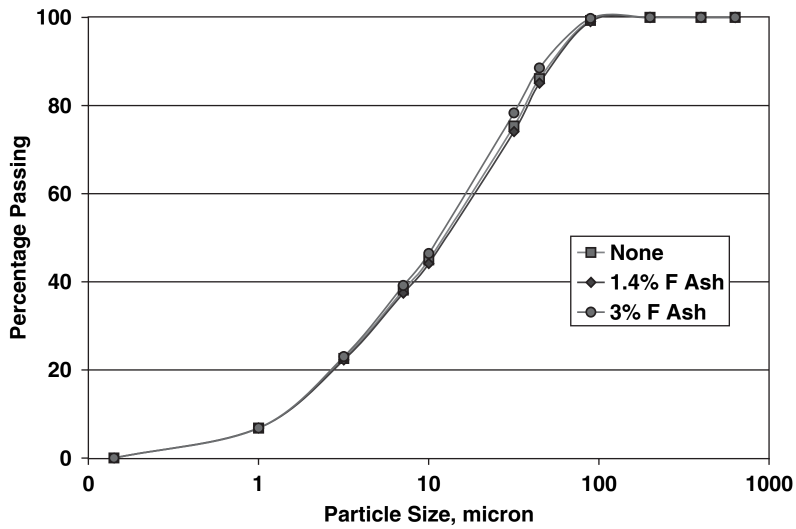


Figure 5. Particle size distributions of selected cements without supplementary cementing materials. (a) PSD curves for clinkers without processing additions. (b) PSD curves for low clinker with Class C Ash processing addition.



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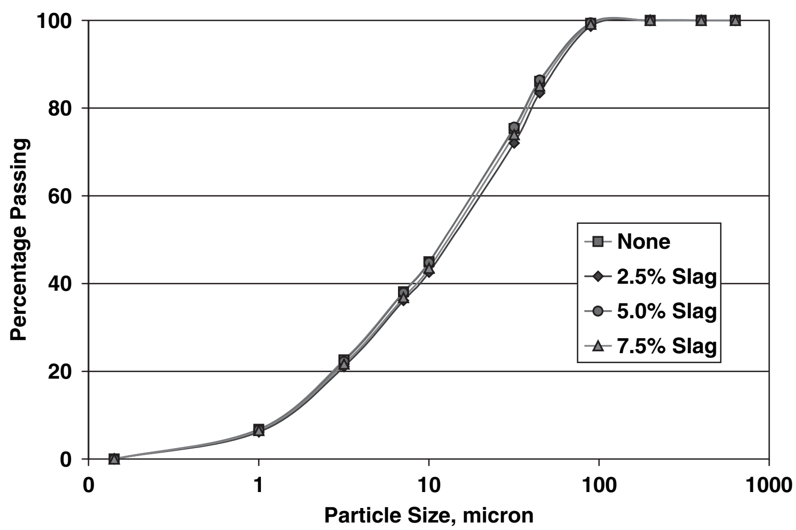

(d)

Figure 5. (Continued). Particle size distributions of selected cements without supplementary cementing materials. (c) PSD curves for low clinker with Class F Ash processing addition. (d) PSD curves for low clinker with slag processing addition.

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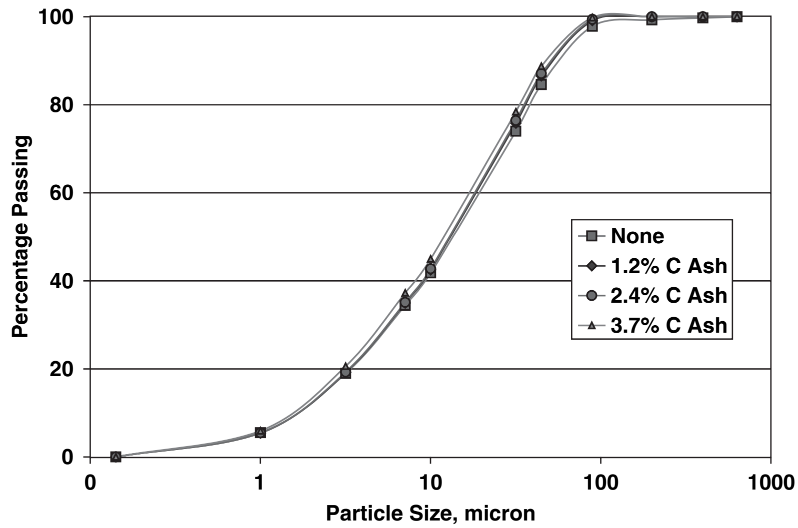

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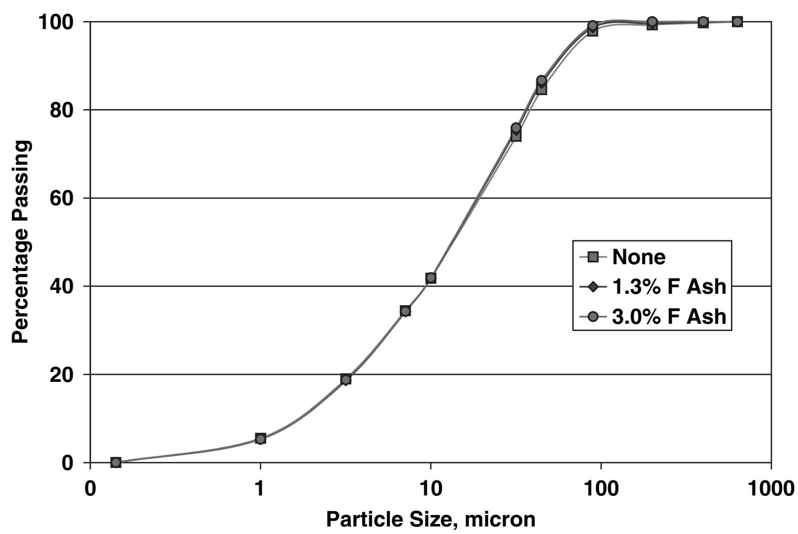
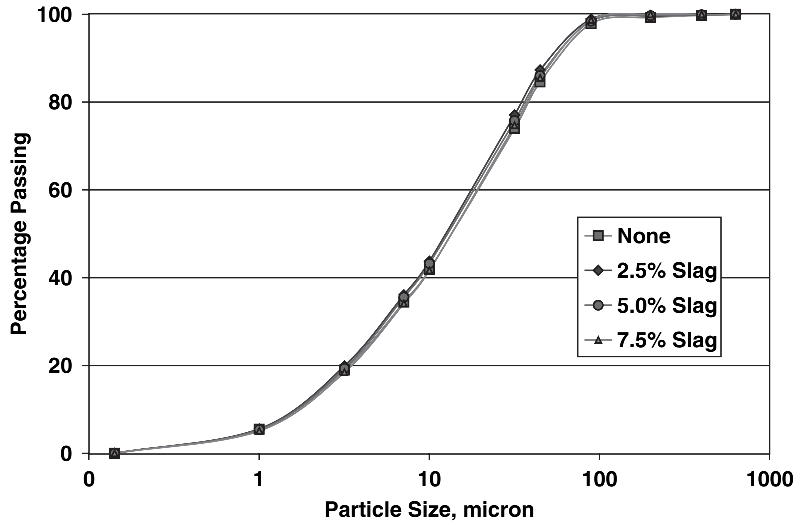


(f)

Figure 5. (Continued). Particle size distributions of selected cements without supplementary cementing materials. (e) PSD curves for high clinker with Class C Fly Ash processing addition. (f) PSD curves for high clinker with Class F Fly Ash processing addition.



(g)

Figure 5. (Continued). Particle size distributions of selected cements without supplementary cementing materials. (g) PSD curves for high clinker with slag processing addition.

- Setting time (AASHTO T 197).
- Shrinkage (AASHTO T 160), 3 bars.
- Hardened air content (ASTM C 457), 1 sample.
- Compressive strength development (AASHTO T 22) at 1, 3, 7, 28, 56, and 90 days, 3 cylinders per age; all cylinders moist cured until tested.
- Chloride penetration (AASHTO T 277) at 90 days, 2 cylinders.
- Freeze/thaw (AASHTO T 161, Method A) for 200 cycles (in order to fit the test into the time available), 3 prisms.
- Deicer Salt scaling (ASTM C 672 plus mass loss determination), 3 samples.

Flexural strength at 7 and 28 days was determined for the paving mixes in accordance with AASHTO T 97.

One mixture was repeated because the early strength data were lower than expected.

Results are shown in Table 13.

Verification Tests

Materials

In order to confirm the trends observed with a single set of each material, a series of additional tests were conducted using different commercially available materials:

- Two clinkers, one with a C_3A of 3% (Sample C3A3), and another with a C_3A content of 14% (Sample C3A14).
- One cement kiln dust from a long wet kiln (Sample cement kiln dust2).
- One GGBFS (Sample Slag2).
- Two Class C fly ashes with high calcium contents (CAsh2 and CAsh3).

Analyses of the materials are given in Table 14.

Eight combinations were prepared using these materials. The dosage of PA was based on the same approach as in the main tests, by limiting the dosage to ensure that standard limits on LOI and Insoluble Residue were not exceeded. Sulfate dosages were determined in accordance with ASTM C 563 at 1 day. Added sulfate was provided using a roughly half and half blend of gypsum and plaster prepared by partially dehydrating a laboratory grade gypsum. Samples were ground to a target fineness between 350 and 400 kg/m^2.

Details of the final cements are given in Table 15.

Samples that required sulfate dosages higher than permitted in the specification were tested in accordance with ASTM C 1038.

Tests

The following tests were conducted, based on the requirements of the proposed protocol:

- Fineness — AASHTO T 98
- Chemical analysis — AASHTO T 105
- Autoclave — AASHTO T 107
- Water requirement — AASHTO T 129/T 131
- Set time — AASHTO T 131
- Cube strength — AASHTO T 106 at 1, 3, 7, and 28 days
- Shrinkage — ASTM C 596

Table 11. Results of paste and mortar tests.
(Cells with bold borders indicate tests that were repeated)

Composition									Paste				Mortar								
Combination	Limits	Clinker type	Processing Addition type	Processing Addition Dosage, label	Processing Addition Dosage, %	SCM Type	% SCM	C1038, % expansion	Autoclave expansion, %	Vicat Time of Set, Initial, min	Calorimetry, silicate peak J/gh	Flow, mL/650*100	Strength, 1 day, psi	Strength, 3 day, psi	Strength, 7 day, psi	Strength, 28 day, psi	Flow, %	Shrinkage, %	AEA, mL	C359, Penetration, mm	ASR, Expansion, %
									1	1	0.5	0.5	0.5	1	1	0.5	0	1	0.5	1	1
All	Min							0.02	0.08	45			870	1740	2760	3943				50	
	Max									375											
Low	Min											0.222							0.60		0.010
	Max										16.85						140	-0.125			
Mid	Min											0.253							0.36		0.022
	Max										15.09						134	-0.145			
High	Min											0.262							0.42		0.059
	Max										19.39						126	-0.157			
LS	Min											0.253							0.42		0.025
	Max										14.83						132	-0.133			
1		Low	None	C	0	None	0	*	0.004	130	16.85	0.215	1643	3325	4227	5562	140	-0.100	0.50	50	0.007
2		Low	None	C	0	F Ash	25	*	-0.007	255	11.40	0.231	1236	2341	2859	4840	145	-0.086	1.00	50	0.000
3		Low	C Ash	S	1.4	None	0	*	-0.007	115	14.65	0.209	1750	3386	4231	5823	138	-0.100	0.35	4	0.001
4		Low	C Ash	S	1.4	Slag	50	*	0.000	160	9.12	0.249	730	1627	2731	6049	141	-0.098	0.50	10	0.002
5		Low	C Ash	I	2.7	C Ash	25	0.001	0.060	160	11.84	0.195	1206	2661	3451	6210	145+	-0.095	0.40	3	-0.004
6		Low	C Ash	I	2.7	F Ash	25	0.007	-0.032	190	11.49	0.231	1106	2417	3395	5246	145+	-0.086	1.00	1	-0.014
7		Low	C Ash	U	4	C Ash	25	*	0.064	110	11.14	0.200	1228	2655	3681	5936	145+	-0.085	0.30	50	0.002
8		Low	C Ash	U	4	F Ash	25	*	-0.016	155	11.75	0.231	1263	2398	3249	5340	145	-0.088	0.85	2	-0.013
9		Low	F Ash	I	1.4	None	0	*	-0.021	110	14.65	0.198	1973	3665	4777	6132	139	-0.099	0.35	40	-0.010
10		Low	F Ash	I	1.4	Slag	50	*	0.014	130	8.95	0.246	808	1617	2818	6552	142	-0.105	0.50	7	0.005
11		Low	F Ash	U	3	None	0		-0.019	110	14.91	0.215	1920	3858	4727	6433	142	-0.098	0.45	3	0.000
12		Low	F Ash	U	3	Slag	50	0.004	-0.003	230	9.82	0.249	714	1698	3247	6269	140	-0.076	0.50	50	0.001
13		Low	Slag	S	2.5	C Ash	25	*	0.063	90	12.54	0.198	1427	2564	3434	5543	145	-0.099	0.40	5	0.004
14		Low	Slag	S	2.5	F Ash	25	*	-0.026	195	11.67	0.237	1322	2642	3243	5035	145	-0.085	0.80	50	-0.017
15		Low	Slag	I	5	None	0	*	-0.001	100	15.46	0.215	1971	3570	4567	6088	145	-0.095	0.45	50	-0.007
16		Low	Slag	I	5	Slag	50	*	0.012	140	8.24	0.246	735	1472	2720	6386	140	-0.117	0.50	50	-0.002
17		Low	Slag	U	7.5	None	0	*	-0.003	110	14.56	0.209	1802	3281	4335	6202	134	-0.098	0.45	50	-0.002
18		Low	Slag	U	7.5	F Ash	25	*	-0.010	150	10.88	0.231	1138	2300	3003	5481	133	-0.087	0.85	50	-0.011
19		Low	Slag	U	7.5	Slag	50	*	0.012	140	8.42	0.249	717	1570	2576	6404	133	-0.108	0.50	50	0.000
20		Low	CKD	S	2.5	None	0	*	-0.008	115	15.35	0.222	1949	3566	4448	5539	131	-0.110	0.35	50	-0.005
21		Low	CKD	S	2.5	Slag	50	*	0.010	140	9.21	0.251	846	1872	3147	5966	135	-0.115	0.50	50	0.000
22		Low	CKD	I	5.1	C Ash	25	*	0.094	165	11.93	0.206	1291	2983	3925	5468	145+	-0.116	0.40	50	-0.002
23		Low	CKD	I	5.1	F Ash	25	*	0.007	155	11.67	0.238	1397	2717	3299	5043	145	-0.096	0.85	50	-0.007
24		Low	CKD	U	7.5	C Ash	25	*	0.106	190	12.72	0.212	1281	3043	3881	5253	145	-0.127	0.40	50	0.009
25		Low	CKD	U	7.5	F Ash	25	*	0.016	175	11.58	0.249	1321	2620	3488	4828	136	-0.110	0.85	50	-0.004
26		Mid	None	C	0	C Ash	25	0.008	0.083	100	13.16	0.225	1696	3273	4083	6025	143	-0.120	0.45	50	0.019
27		Mid	None	C	0	F Ash	25	0.004	-0.026	105	11.75	0.259	1883	2903	3353	5583	131	-0.104	0.85	50	-0.002
28		Mid	None	C	0	Slag	50	0.013	-0.007	120	10.09	0.265	880	1849	2741	6453	131	-0.081	0.50	50	0.002
29		Mid	C Ash	S	1.4	C Ash	25	0.006	0.084	135	13.42	0.231	1560	2888	3951	5326	145+	-0.126	0.45	50	0.018
30		Mid	C Ash	S	1.4	F Ash	25	0.003	-0.021	130	11.84	0.265	1910	3035	3880	5888	137	-0.099	0.80	50	-0.008
31		Mid	C Ash	I	2.8	None	0	0.005	0.004	95	14.30	0.252	2556	3809	4901	6885	132	-0.077	0.35	25	0.013
32		Mid	C Ash	I	2.8	Slag	50	0.014	0.005	115	8.77	0.265	921	1953	2728	6443	140	-0.079	0.50	40	0.006
33		Mid	C Ash	U	4.1	None	0	0.007	-0.004	95	15.79	0.246	2283	3806	4761	6363	134	-0.102	0.35	50	0.010
34		Mid	C Ash	U	4.1	Slag	50	0.014	0.004	130	7.10	0.265	883	1898	3023	6101	142	-0.113	0.50	50	-0.001
35		Mid	F Ash	I	1.4	C Ash	25	0.008	0.075	135	12.19	0.228	1798	3198	4096	6171	145+	-0.110	0.45	50	0.010
36		Mid	F Ash	I	1.4	F Ash	25	0.000	-0.031	120	11.23	0.257	1844	2786	3595	5668	143	-0.096	0.85	50	-0.008
37		Mid	F Ash	U	3	C Ash	25	0.003	0.093	140	13.25	0.234	1645	3206	4234	6228	145+	-0.109	0.45	50	0.017
38		Mid	F Ash	U	3	F Ash	25	0.007	-0.012	140	11.58	0.262	1803	3213	3796	5925	145+	-0.101	0.90	50	-0.011
39		Mid	Slag	S	2.5	None	0	0.007	0.013	85	16.58	0.245	2366	3708	5058	6668	134	-0.099	0.35	50	0.008
40		Mid	Slag	S	2.5	Slag	50	0.014	0.016	130	9.74	0.262	978	1855	3007	6476	139	-0.099	0.50	50	-0.004
41		Mid	Slag	I	5	C Ash	25	0.009	0.103	160	12.90	0.228	1485	2918	3538	5245	145+	-0.101	0.45	50	0.001
42		Mid	Slag	I	5	F Ash	25	0.009	-0.006	130	10.97	0.262	1679	2733	3840	5711	145	-0.096	0.90	50	-0.007
43		Mid	Slag	U	7.5	None	0	0.006	0.008	100	14.91	0.243	2569	3635	4926	6528	135	-0.099	0.35	50	0.002
44		Mid	Slag	U	7.5	C Ash	25	0.007	0.094	125	12.02	0.222	1436	2889	3832	5922	145+	-0.113	0.40	50	0.002
45		Mid	Slag	U	7.5	F Ash	25	0.005	-0.017	147	11.05	0.258	1603	2858	3566	5639	140	-0.099	0.95	50	-0.015
46		Mid	CKD	S	2.5	C Ash	25	0.010	0.079	155	13.69	0.235	1853	3601	4544	6303	145+	-0.104	0.40	50	0.005
47		Mid	CKD	S	2.5	F Ash	25	0.006	-0.025	135	14.04	0.258	1897	3110	4049	6095	145	-0.107	0.95	50	-0.012
48		Mid	CKD	I	5	None	0	0.010	0.009	120	18.69	0.252	2918	4676	5710	6773	131	-0.120	0.35	50	0.017
49		Mid	CKD	I	5	Slag	50	0.014	0.017	140	9.21	0.262	1149	2382	3985	6138	137	-0.087	0.50	50	0.006
50		Mid	CKD	U	7.5	None	0	0.010	0.026	125	17.81	0.255	2827	4034	5466	7021	133	-0.129	0.35	50	0.024
51		Mid	CKD	U	7.5	Slag	50	0.010	0.016	145	9.65	0.260	1013	2163	3842	6570	137	-0.118	0.50	50	0.010
52		High	None	C	0	None	0	0.011	0.113	80	19.39	0.255	2737	4489	5233	5966	126	-0.132	0.35	40	0.056
53		High	None	C	0	Slag	50	0.011	0.038	100	10.97	0.262	1286	2434	3967	6178	144	-0.122	0.50	50	0.002
54		High	C Ash	S	1.2	C Ash	25	0.007	0.248	110	17.90	0.228	2193	3195	4141	5495	145+	-0.134	0.40	50	0.035
55		High	C Ash	S	1.2	F Ash	25	0.006	0.067	95	16.23	0.255	2080	3144	3854	5333	145	-0.115	0.95	50	0.012
56		High	C Ash	I	2.4	None	0	0.007	0.122	75	21.23	0.252	2953	4169	5363	5743	134	-0.126	0.50	50	0.072
57		High	C Ash	I	2.4	Slag	50	0.012	0.029	95	11.75	0.262	1293	2585	4063	6330	143	-0.126	0.50	50	0.004
58		High	C Ash	U	3.7	None	0	0.007	0.103	70	19.30	0.255	3014	4602	5575	6598	139	-0.128	0.35	50	0.034
59		High	C Ash	U	3.7	C Ash	25	0.008	0.090	65	17.37	0.259	3070	4718	5370	6210	135	-0.132	0.40	50	0.110
60		High	C Ash	U	3.7	Slag	50	0.010	0.025	95	11.58	0.262	1274	2392	4012	6559	143	-0.120	0.50	50	0.000

Table 11. (Continued).

Composition									Paste				Mortar								
Combination	Limits	Clinker type	Processing Addition type	Processing Addition Dosage, label	Processing Addition Dosage, %	SCM Type	% SCM	C1038, % expansion	Autoclave expansion, %	Vicat Time of Set, Initial, min	Calorimetry, silicate peak J/gh	Flow, mL/650*100	Strength, 1 day, psi	Strength, 3 day, psi	Strength, 7 day, psi	Strength, 28 day, psi	Flow, %	Shrinkage, %	AEA, mL	C359, Penetration, mm	ASR, Expansion, %
									1	**1**	**0.5**	**0.5**	**0.5**	**1**	**1**	**0.5**	**0**	**1**	**0.5**	**1**	**1**
All	**Min**							**0.02**	**0.08**	**45**			**870**	**1740**	**2760**	**3943**				**50**	
	Max									**375**											
Low	**Min**											**0.222**							**0.60**		**0.010**
	Max										**16.85**						**140**	**-0.125**			
Mid	**Min**											**0.253**							**0.36**		**0.022**
	Max										**15.09**						**134**	**-0.145**			
High	**Min**											**0.262**							**0.42**		**0.059**
	Max										**19.39**						**126**	**-0.157**			
LS	**Min**											**0.253**							**0.42**		**0.025**
	Max										**14.83**						**132**	**-0.133**			
61		High	F Ash	I	1.3	C Ash	25	0.008	0.226	100	13.86	0.231	2274	3464	4436	5533	145+	-0.133	0.40	50	0.032
62		High	F Ash	I	1.3	F Ash	25	0.002	0.040	100	14.39	0.262	1993	3398	3964	5373	145	-0.111	0.95	50	-0.008
63		High	F Ash	U	3	C Ash	25	0.008	0.180	110	15.09	0.231	2136	3545	3895	4886	145+	-0.131	0.40	50	0.016
64		High	F Ash	U	3	F Ash	25	0.004	0.055	105	14.74	0.255	2057	3217	3926	5285	145	-0.084	0.95	50	-0.018
65		High	Slag	S	2.5	None	0	-0.001	0.102	80	20.36	0.248	2414	4231	5517	6438	135	-0.136	0.35	40	0.047
66		High	Slag	S	2.5	Slag	50	0.008	0.020	125	10.16	0.262	1169	2403	3874	5641	140	-0.116	0.50	50	-0.007
67		High	Slag	I	5	C Ash	25	0.008	0.193	110	13.77	0.228	2237	3288	4443	5807	145+	-0.146	0.40	50	0.035
68		High	Slag	I	5	F Ash	25	0.003	0.047	110	13.42	0.262	1676	3038	4062	5643	145	-0.118	0.95	50	0.003
69		High	Slag	U	7.5	C Ash	25	0.008	0.190	120	13.07	0.228	1953	3052	4101	5501	145+	-0.107	0.35	50	0.020
70		High	Slag	U	7.5	F Ash	25	-0.060	0.056	115	12.90	0.258	1669	3033	3837	5398	145+	-0.092	0.90	50	
71		High	CKD	S	2.3	C Ash	25	0.014	0.242	145	15.62	0.225	1933	3362	4316	5298	145+	-0.130	0.35	50	0.074
72		High	CKD	S	2.3	F Ash	25	0.010	0.094	115	15.27	0.258	2211	3313	3953	5609	145	-0.120	0.90	50	-0.010
73		High	CKD	S	2.3	Slag	50	0.012	0.045	110	11.14	0.260	1322	2530	3578	5182	125	-0.119	0.50	50	0.012
74		High	CKD	I	4.5	None	0	0.043	0.118	90	19.30	0.258	3241	4378	5095	6218	123	-0.139	0.35	38	0.189
75		High	CKD	I	4.5	Slag	50	0.009	0.047	120	9.74	0.262	1282	2677	3804	5614	145	-0.111	0.40	50	0.013
76		High	CKD	U	6.9	None	0	0.011	0.114	105	18.78	0.258	3041	4327	5343	5904	116	-0.154	0.35	32	0.120
77		High	CKD	U	6.9	Slag	50	0.007	0.050	135	10.00	0.262	1151	2573	3903	6106	144	-0.113	0.40	50	0.003
78		LS	None	C	0	None	0	0.002	-0.029	135	14.83	0.246	1826	3686	4828	6398	132	-0.108	0.35	50	0.000
79		LS	None	C	0	C Ash	25	0.003	0.059	200	10.61	0.228	1598	3266	4169	6701	145+	-0.101	0.35	50	0.009
80		LS	C Ash	I	0.8	C Ash	25	0.002	0.085	190	10.18	0.222	1687	3507	4869	6640	145+	-0.108	0.35	50	0.010
81		LS	C Ash	I	0.8	F Ash	25	0.000	0.000	160	11.58	0.249	1549	2948	4272	5848	145+	-0.095	0.80	50	-0.014
82		LS	C Ash	I	0.8	Slag	50	0.004	0.013	150	8.07	0.255	920	2146	3487	6142	140	-0.108	0.40	50	0.005
83		LS	C Ash	U	2.1	C Ash	25	0.004	0.106	170	12.19	0.222	1498	3308	4541	6488	145+	-0.112	0.35	50	0.017
84		LS	C Ash	U	2.1	F Ash	25	0.001	-0.009	150	11.75	0.252	1693	2833	3633	5963	145+	-0.102	0.90	50	-0.011
85		LS	F Ash	U	3	None	0	0.000	-0.031	120	14.30	0.249	2365	4281	5338	6808	135	-0.109	0.35	50	0.001
86		LS	F Ash	U	3	Slag	50	0.006	-0.011	150	7.98	0.265	905	2167	3702	6202	135	-0.107	0.40	50	0.006
87		LS	Slag	S	2.5	C Ash	25	0.005	0.081	175	10.26	0.225	1641	3385	4448	6153	145+	-0.104	0.35	50	0.009
88		LS	Slag	S	2.5	F Ash	25	0.003	-0.020	135	9.74	0.255	1551	2871	3730	5343	145+	-0.094	0.90	50	-0.006
89		LS	Slag	I	5	None	0	0.002	-0.004	120	12.90	0.249	1926	3901	4888	6083	128	-0.101	0.35	50	0.008
90		LS	Slag	I	5	Slag	50	0.005	0.009	150	8.16	0.262	878	1964	3505	6336	138	-0.108	0.40	42	0.009
91		LS	Slag	U	7.5	None	0	0.002	0.003	120	14.39	0.246	2098	3738	4912	6471	133	-0.103	0.35	50	0.012
92		LS	Slag	U	7.5	Slag	50	0.005	0.010	150	7.72	0.262	793	2093	3689	5823	138	-0.108	0.40	50	0.010
93		LS	CKD	S	0.8	None	0	*	0.043	115	14.83	0.246	1603	3366	4606	5963	132	-0.138	0.35	50	0.034
94		LS	CKD	S	0.8	Slag	50	*	0.018	140	8.42	0.262	705	2027	3555	6064	145+	-0.134	0.40	50	0.005
95		LS	CKD	I	1.5	C Ash	25	0.004	0.087	170	12.54	0.225	1648	3562	4718	6636	145+	-0.120	0.35	50	0.008
96		LS	CKD	I	1.5	F Ash	25	0.003	-0.016	140	11.23	0.249	1666	3010	3789	5919	145+	-0.105	0.92	50	-0.016
97		LS	CKD	I	1.5	Slag	50	0.006	0.007	140	8.60	0.258	888	2108	3548	6128	139	-0.115	0.45	50	0.004
98		LS	CKD	U	4	C Ash	25	*	0.125	170	12.02	0.231	1509	3586	4858	5928	145+	-0.139	0.35	50	0.002
99		LS	CKD	U	4	F Ash	25	*	0.012	150	11.05	0.262	1574	2872	3639	5438	143	-0.122	0.92	50	-0.014
100		Mid	None	C	0.0	None	0	0.003	-0.011	137	15.09	0.246	2863	4223	4969	7396	134	-0.104	0.30	50	0.0187
101		LS	C Ash	I	0.8	None	0	0.002	-0.048	195	13.86	0.245	2588	4160	5119	6285	133	-0.112	0.30	50	0.0250
102		Low	F Ash	I	1.4	C Ash	25	*	0.072	146	11.75	0.212	1326	2693	3892	6051	145+	-0.095	0.30	50	0.0077
103		Mid	F Ash	I	1.4	Slag	50	0.014	-0.005	198	9.47	0.265	1068	2173	3562	7132	132	-0.098	0.30	9	-0.0013
104		High	F Ash	I	1.3	None	0	0.007	0.057	146	16.06	0.258	2845	4431	5376	6627	128	-0.126	0.30	50	0.1163
105		Low	F Ash	U	3.0	F Ash	25	*	-0.027	315	10.35	0.231	1252	2481	3335	5323	143	-0.083	1.00	50	0.0003
106		Mid	F Ash	U	3.0	None	0	0.003	0.002	154	15.09	0.246	2763	4090	5142	7108	132	-0.101	0.30	50	0.0100
107		High	F Ash	U	3.0	Slag	50	0.006	0.003	213	8.95	0.274	1069	2613	3879	6258	136	-0.106	0.30	45	0.0067
108		LS	F Ash	U	3.0	C Ash	25	0.005	0.063	247	9.56	0.223	1800	3365	4785	6318	142	-0.099	0.30	50	-0.0077
109		LS	F Ash	U	3.0	F Ash	25	0.002	-0.057	220	9.47	0.260	1531	2781	3600	5496	138	-0.101	0.80	50	-0.0130

Table 12. XRF of coarse fraction of selected cement samples.

Sample	76			78			85			91		
	>20 micron	Full	Difference	>20 micron	Full	Difference	>20 micron	Full	Difference	>20 micron	Full	Difference
Analyte	Weight %			Weight %			Weight %			Weight %		
SiO_2	20.75	19.40	-1.35	21.96	21.11	-0.85	22.02	20.99	-1.04	21.77	21.63	-0.14
Al_2O_3	5.77	5.43	-0.34	5.07	4.75	-0.32	5.09	5.22	0.13	5.07	5.07	0.00
Fe_2O_3	2.22	2.13	-0.09	3.74	3.39	-0.35	3.86	3.63	-0.23	3.67	3.30	-0.37
CaO	64.52	61.55	-2.97	63.52	61.46	-2.06	62.80	60.68	-2.12	63.13	60.87	-2.26
MgO	2.15	2.15	-0.01	1.00	1.09	0.09	0.99	1.11	0.11	1.14	1.78	0.64
SO_3	2.16	4.44	2.28	1.86	4.11	2.25	2.27	3.98	1.71	2.30	3.27	0.96
Na_2O	0.29	0.38	0.09	0.14	0.18	0.04	0.16	0.23	0.06	0.17	0.20	0.03
K_2O	0.76	1.39	0.63	0.73	0.87	0.13	0.71	0.90	0.18	0.72	0.85	0.13
TiO_2	0.33	0.29	-0.04	0.20	0.18	-0.02	0.20	0.21	0.00	0.21	0.23	0.02
P_2O_5	0.13	0.13	0.00	0.18	0.16	-0.01	0.18	0.17	0.00	0.17	0.16	-0.02
Mn_2O_3	0.17	0.18	0.02	0.20	0.22	0.02	0.20	0.22	0.02	0.20	0.26	0.05
SrO	0.06	0.08	0.02	0.19	0.20	0.00	0.19	0.20	0.01	0.18	0.19	0.00
Cr_2O_3	0.01	0.01	0.00	0.01	0.01	0.00	0.01	0.01	0.00	0.01	0.01	0.00
ZnO	0.01	0.01	0.00	0.02	0.02	0.00	0.01	0.02	0.01	0.01	0.02	0.00
L.O.I. (950^oC)	0.82	2.73	1.91	0.94	2.64	1.70	1.19	2.70	1.51	1.26	2.28	1.02
Total	100.14	100.29	0.15	99.76	100.39	0.63	99.90	100.25	0.35	100.01	100.10	0.09

Table 13. Results of concrete tests.

Combination	Mix	Clinker type	Processing Addition type	Processing Addition Dosage, %	Slump, inch	AEA, mL	Air, %	Air/AEA	Hardened Air Content, %	Spacing Factor, per in	Unit weight, pcy	Set time, mins	Rate of slump loss, in/min	Rate of Flow loss, %/min	Shrinkage at 28 days, %	Shrinkage at 56 days, %	Compress. Strength at 1 day, psi	Compress. Strength at 3 day, psi	Compress. Strength at 7 day, psi	Compress. Strength at 28 day, psi	Compress. Strength at 56 day, psi	Compress. Strength at 90 day, psi	Chloride Penetration, coulomb	Freeze/thaw, RDM % 200 cycles	Salt Scaling, Rating	Salt Scaling, Mass loss, kg/m2	Ft 7	Ft 28
All									**4**	**0.008**													**4000**	**80**	**4**	**0.8**		
High												**318**	**0.019**	**0.548**	**-0.0327**	**-0.0407**	**1215**	**2106**	**2799**	**4833**	**5823**	**5940**						
Low												**330**	**0.032**	**0.497**	**-0.0507**	**-0.0564**	**1512**	**2349**	**3204**	**3942**	**4788**	**4653**						
LS												**312**	**0.022**	**0.418**	**-0.0397**	**-0.0451**	**1602**	**2205**	**2745**	**4374**	**5103**	**5706**						
Mid												**326**	**0.017**	**0.388**	**-0.0491**	**-0.0551**	**1431**	**2268**	**3114**	**4851**	**5445**	**5778**						
High												**310**	**0.028**	**0.419**	**-0.0401**	**-0.0687**	**1656**	**2592**	**3780**	**5121**	**5490**	**5913**					**594**	**729**
Mid												**313**	**0.013**	**0.484**	**-0.0437**	**-0.0477**	**1764**	**2943**	**4203**	**5418**	**6111**	**6489**					**630**	**828**
52	Structural	High	None	0.0	4.50	35	7.0	0.20	4.8	0.0065	143.4	228	0.017	0.498	-0.0253	-0.0333	1350	2340	3110	5370	6470	6600	4050	100	3.3	3.703		
104	Structural	High	F Ash	1.3	3.50	35	6.8	0.19	4.4	0.006	144.4	226	0.019	0.340	-0.0330	-0.0410	1590	1990	2980	5310	5940	6330	3990	99	4.8	7.987		
56	Structural	High	C Ash	2.4	3.50	35	7.0	0.20	4.9	0.0055	143.8	235	0.016	0.283	-0.0210	-0.0347	1400	2110	2640	4840	5480	5800	4720	100	3.0	2.744		
65	Structural	High	Slag	2.5	3.50	32	6.4	0.20	5.6	0.0073	144.8	244	0.019	0.429	-0.0270	-0.0510	1300	2230	2990	5240	6060	6480	4300	97	5.0	7.097		
74	Structural	High	CKD	4.5	4.50	32	6.9	0.22	5.0	0.0072	144.2	250	0.022	0.388	-0.0390	-0.0513	1730	2890	4310	6100	6400	6800	3460	98	4.5	1.988		
1	Structural	Low	None	0.0	7.50	55	7.0	0.13	7.2	0.0035	143.9	240	0.029	0.451	-0.0433	-0.0490	1680	2610	3560	4380	5320	5170	3000	99	0.5	0.050		
3	Structural	Low	C Ash	1.4	6.80	35	6.1	0.17	5.6	0.0054	145.4	270	0.024	0.326	-0.0493	-0.0543	1580	3140	4190	5890	6170	6170	3410	101	0.5	0.118		
9	Structural	Low	F Ash	1.4	3.80	35	5.4	0.15	4.3	0.0057	146.8	297	0.014	0.242	-0.0413	-0.0540	1340	1860	3550	5430	6110	6510	3330	99	0.3	0.037		
20	Structural	Low	CKD	2.5	4.00	32	5.5	0.17	4.8	0.0050	146.4	273	0.023	0.326	-0.0403	-0.0563	1490	2580	3150	4640	5350	6210	4180	100	1.0	0.056		
15	Structural	Low	Slag	5.0	7.75	40	5.6	0.14	4.6	0.0055	147.2	296	0.028	0.330	-0.0403	-0.0497	1100	2280	3310	4990	5800	6290	3050	99	0.5	0.074		
78 Orig	Structural	LS	None	0.0	3.50	35	6.9	0.20	-	-	143.2	293	0.010	0.215	-0.0327	-0.0407	960	1380	2460	4260	5390	6410	5120	95	5.0	6.800		
78	Structural	LS	None	0.0	2.50	32	6.4	0.20	5.0	0.0068	145.2	222	0.020	0.380	-0.0323	-0.0377	1780	2450	3050	4860	5670	6340	4740		1.0	0.400		
101	Structural	LS	C Ash	0.8	2.50	35	6.0	0.17	5.1	0.0051	146.0	264	0.012	0.210	-0.0263	-0.0350	1340	2370	4100	5430	5870	6630	3060	100	0.8	0.235		
93	Structural	LS	CKD	0.8	3.00	32	6.3	0.20	5.5	0.0049	144.4	256	0.019	0.249	-0.0477	-0.0517	2080	3630	4620	5790	6140	5830	2440	99	1.2	0.495		
89	Structural	LS	Slag	5.0	2.00	32	6.0	0.19	4.8	0.0073	145.8	264	0.011	0.288	-0.0363	-0.0510	1450	2160	3350	5220	6120	6610	3620		2.2	0.019		
100	Structural	Mid	None	0.0	3.50	35	7.0	0.20	5.7	0.0046	143.6	236	0.015	0.353	-0.0417	-0.0477	1590	2520	3460	5390	6050	6420	3380	101	2.0	3.115		
39	Structural	Mid	Slag	2.5	2.50	33	6.1	0.18	4.9	0.0051	145.2	239	0.011	0.352	-0.0280	-0.0483	2100	3710	5060	5920	6780	7250	2250	96	1.3	0.464		
31	Structural	Mid	C Ash	2.8	2.50	35	6.2	0.18	4.7	0.0061	145.6	260	0.012	0.226	-0.0390	-0.0417	1270	2880	3960	6380	6600	7960	3150	101	1.7	1.474		
48	Structural	Mid	CKD	5.0	2.50	32	6.1	0.19	4.8	0.0058	145.4	244	0.015	0.328	-0.0393	-0.0603	1760	2340	3110	4900	5710	6560	3140	98	1.3	0.576		
52	Paving	High	None	0.0	1.75	32	6.1	0.19	5.2	0.0066	145.8	220	0.025	0.381	-0.0327	-0.0613	1840	2880	4200	5690	6100	6570	3120	94	4.0	6.769	660	810
104	Paving	High	F Ash	1.3	1.25	30	5.3	0.18	3.6	0.0058	148.8	190	0.017	0.516	-0.0333	-0.0400	1990	3310	5020	6420	6730	7360	1800		2.0	1.666	740	960
56	Paving	High	C Ash	2.4	2.00	32	6.8	0.21	5.8	0.0052	146.0	218	0.019	0.395	-0.0440	-0.0500	1910	3190	4450	5400	6370	6830	3300	98	2.3	1.957	690	880
65	Paving	High	Slag	2.5	2.00	32	6.8	0.21	6.2	0.0058	145.2	204	0.029	0.328	-0.0233	-0.0423	1560	2250	2790	4600	5260	5650	4870	97	5.0	11.717	510	780
74	Paving	High	CKD	4.5	2.80	31	7.0	0.23	5.4	0.0062	145.8	233	0.024	0.391	-0.0480	-0.0557	1480	2640	3850	5390	5770	6020	3190	97	2.3	2.744	560	820
100	Paving	Mid	None	0.0	1.50	30	5.9	0.20	5.3	0.0055	146.6	223	0.012	0.440	-0.0363	-0.0403	1960	3270	4670	6020	6790	7210	2180		1.0	0.235	700	920
39	Paving	Mid	Slag	2.5	1.25	30	6.1	0.20	5.8	0.0058	147.4	250	0.008	0.033	-0.0293	-0.0430	1670	3330	4300	5610	6120	6780	2220		1.7	0.656	710	950
31	Paving	Mid	C Ash	2.8	1.75	40	7.1	0.18	6.0	0.0048	144.8	258	0.008	0.285	-0.0363	-0.0447	1470	2470	3600	5170	6040	6550	2420	100	3.8	2.199	600	770
48	Paving	Mid	CKD	5.0	1.50	30	5.6	0.19	5.3	0.0057	147.4	233	0.014	0.039	-0.0400	-0.0477	1640	2580	3190	5050	5960	6500	2580		0.3	0.025	500	810

Table 14. Analysis of verification test materials.

	C3A3	C3A14	Cement Kiln Dust2	Slag2	CAsh2	CAsh3
Analyte	**Weight %**					
SiO_2	21.64	20.28	15.29	31.71	39.71	39.41
Al_2O_3	3.40	6.53	3.72	9.53	19.87	19.88
Fe_2O_3	3.79	2.04	1.83	1.35	5.10	5.14
CaO	63.93	63.38	44.98	45.74	23.25	23.44
MgO	4.30	3.01	1.66	8.16	4.25	4.27
SO_3	0.67	1.83	6.63	2.90	1.16	1.17
Na_2O	0.13	0.24	0.23	0.19	1.60	1.64
K_2O	0.66	1.15	3.56	0.31	0.46	0.47
TiO_2	0.17	0.28	0.18	0.52	1.52	1.52
P_2O_5	0.06	0.22	0.14	0.09	0.97	0.97
Mn_2O_3	0.05	0.08	0.07	0.39	0.04	0.04
SrO	0.04	0.23	0.00	0.11	0.58	0.58
Cr_2O_3	0.02	0.00	0.00	0.01	0.02	0.02
ZnO	0.01	0.00	0.16	0.02	0.02	0.02
Cl	–	–	–	0.17	–	–
L.O.I. (950°C)	1.27	0.80	20.94	-1.13	0.30	0.33
Total	100.16	100.07	99.40	100.07	98.86	98.92
Insoluble Residue	0.19	0.60	11.71	1.14	32.94	31.59

Table 15. Analysis of verification test materials.

Mix ID	Clinker type	Processing Addition type	Processing Addition Dosage, %	Optimum sulfate, %
3-1	C3A3	None	0	2.87
3-2	C3A3	Cement Kiln Dust2	4.9	1.59
3-3	C3A14	None	0	4.04
3-4	C3A14	Cement Kiln Dust2	1.3	4.32
3-5	C3A3	Slag2	7.5	1.85
3-6	C3A14	Slag2	7.5	2.50
3-7	C3A3	Cash2	1.7	1.92
3-8	C3A14	Cash2	0.4	4.00
3-9	C3A3	Cash3	1.7	1.50
3-10	C3A14	Cash3	0.5	4.19

- Air content ASTM C 185
- Alkali silica reactivity ASTM C 227 for 28 days using reactive aggregate
- Compressive strength AASHTO T 22 at 1, 3, 7 and 28 days
- Flexural strength AASHTO T 97
- De-icer scaling ASTM C 672 with mass loss determination

Results

Results are shown in Table 16.

Table 16. Verification test data.

Combination number	Clinker	Processing Addition type	Processing Addition Dosage, %	Autoclave expansion, %	Water requirement	Vicat Time of Set, Initial, min	C 1038, %	Shrinkage	Air, mL
General limit				**0.8**					
Limit for C3A3 clinker				**0.14**	**22.8%**	**126-246**		**-0.148**	**0.36**
Limit for C3A14 clinker				**0.30**	**23.8%**	**67-187**		**-0.168**	**0.18**
C1	C3A3	None		0.04	21.8%	186	X	-0.123	0.30
C2	C3A3	Cement Kiln Dust2	4.9	0.04	22.5%	203	X	-0.108	0.24
C3	C3A14	None		0.20	22.8%	127	0.051	-0.143	0.15
C4	C3A14	Cement Kiln Dust2	1.3	0.20	22.6%	160	0.093	-0.144	0.15
C5	C3A3	Slag2	7.5	0.04	22.8%	371	X	-0.106	0.24
C6	C3A14	Slag2	7.5	0.13	24.5%	98	X	-0.119	0.18
C7	C3A3	CAsh2	1.7	0.03	22.6%	348	X	-0.105	0.22
C8	C3A14	CAsh2	0.4	0.21	24.3%	98	0.048	-0.153	0.18
C9	C3A3	CAsh3	1.7	0.05	22.2%	196	X	-0.101	0.15
C10	C3A14	CAsh3	0.5	0.23	22.6%	122	0.074	-0.152	0.15

Made with high C_3A clinker

Fail limits stated in the protocol

Combination number	Clinker	Processing Addition type	Processing Addition Dosage, %	ASR Expansion, %	Cube Compressive Strength at age, psi				
					1 day	3 days	7 days	28 days	Average
General limit									
Limit for C3A3 clinker									**2262**
Limit for C3A14 clinker									**4113**
C1	C3A3	None	0.0	7/17/07	893	2021	2558	4054	2381
C2	C3A3	Cement Kiln Dust2	4.9	7/17/07	1178	2973	3313	4789	3063
C3	C3A14	None	0.0	7/17/07	1766	3338	5450	6765	4330
C4	C3A14	Cement Kiln Dust2	1.3	7/17/07	1676	2758	4043	6318	3699
C5	C3A3	Slag2	7.5	7/17/07	992	2893	3873	4808	3141
C6	C3A14	Slag2	7.5	7/19/07	2833	4497	5658	6308	4824
C7	C3A3	CAsh2	1.7	7/19/07	1365	3191	3630	4819	3251
C8	C3A14	CAsh2	0.4	7/19/07	1928	3438	5193	6713	4318
C9	C3A3	CAsh3	1.7	7/19/07	1376	2993	3955	5353	3419
C10	C3A14	CAsh3	0.5	7/19/07	1836	3518	5025	7183	4391

Made with high C_3A clinker

Fail limits stated in the protocol

(continued on next page)

Table 16. (Continued).

Combination number	Clinker	Processing Addition type	Processing Addition Dosage, %	Cylinder Compressive Strength at age, psi				
				1 day	3 days	7 days	28 days	Average
General limit								
Limit for C3A3 clinker								**2788**
Limit for C3A14 clinker								**3978**
C1	C3A3	None		1450	2680	3290	4970	3098
C2	C3A3	Cement Kiln Dust2	4.9	1780	3300	4180	5400	3665
C3	C3A14	None		3020	4120	4650	5890	4420
C4	C3A14	Cement Kiln Dust2	1.3	2580	3360	4740	6850	4383
C5	C3A3	Slag2	7.5	1500	2960	4510	5260	3558
C6	C3A14	Slag2	7.5	3690	4460	5030	5980	4790
C7	C3A3	CAsh2	1.7	1860	3240	3950	5070	3530
C8	C3A14	CAsh2	0.4	2680	4350	5310	6450	4698
C9	C3A3	CAsh3	1.7	1620	3110	4180	4570	3370
C10	C3A14	CAsh3	0.5	2310	3820	4840	6070	4260

Made with high C_3A clinker

Fail limits stated in the protocol

Combination number	Clinker	Processing Addition type	Processing Addition Dosage, %	Flexural Strength at age, psi			Scaling, kg/m^2
				7	28	Average	
General limit							**0.8**
Limit for C3A3 clinker						**563**	
Limit for C3A14 clinker						**774**	
C1	C3A3	None		560	690	625	0.20
C2	C3A3	Cement Kiln Dust2	4.9	620	840	730	0.19
C3	C3A14	None		780	940	860	0.00
C4	C3A14	Cement Kiln Dust2	1.3	600	820	710	0.14
C5	C3A3	Slag2	7.5	680	780	730	0.44
C6	C3A14	Slag2	7.5	850	930	890	0.00
C7	C3A3	CAsh2	1.7	550	820	685	0.07
C8	C3A14	CAsh2	0.4	740	900	820	0.20
C9	C3A3	CAsh3	1.7	680	790	735	0.05
C10	C3A14	CAsh3	0.5	630	730	680	0.07

Made with high C_3A clinker

Fail limits stated in the protocol

CHAPTER 4

Discussion of Results

The effects of processing additions have been analyzed based on the work conducted, and a discussion is provided in the following sections. The questions addressed include the following:

- Is there a limit below which all processing additions may be considered acceptable?
- Are some processing additions more beneficial/deleterious than others?
- What tests were most sensitive to the presence of processing additions?
- What limits would be appropriate for such tests?

As discussed below, relatively few significant effects of processing additions were observed in the paste and mortar tests, with few clear or simple trends apparent. Seventy-two significant effects were observed out of a possible 740, both beneficial and detrimental. LS clinker appeared to be involved in affected systems slightly more than the other clinkers, C Ash and F Ash were more likely to be involved than slag or plain systems, and cement kiln dust was much more likely than the other processing additions. The paste and mortar parameters that appeared to be most sensitive to changing cementitious systems were shrinkage, ASR, and 28-day strength.

Likewise, in the concrete tests, few significant effects of processing additions were observed. Five significant effects were observed out of a possible 118, all of which were associated with the Mid or Low clinkers. The concrete parameter that appeared to be most sensitive to changing cementitious systems in the concrete tests was strength, with no clear trend as to the direction of the effect.

In all cases, the effects were small and potentially masked by variability in the test methods.

Materials Combination Characterization

Materials Analyses

The clinkers, processing additions, and supplementary cementing materials were analyzed by XRF. The results are shown in Tables 3 and 4. It was confirmed that all the supplementary cementitious materials complied with the chemical requirements of their respective AASHTO/ASTM standards (Appendix A).

A sample of Blend 59 containing cement, gypsum, and processing addition (and no SCM) was analyzed using XRF and compared with the theoretical composition. The data showed little significant difference between the two sets of analyses.

Optimum Sulfate Content

The amount of sulfate required to optimize each of the cements prepared before addition of supplementary cementitious materials was reviewed to see whether the presence of the processing additions had a measurable effect. The data are illustrated in Figure 6 in which the optimum total sulfate content has been plotted against processing addition dosage for each processing addition. Four plots have been produced—one for each clinker.

Several of the systems required sulfate contents greater than allowed in the current specification. All of these were tested in accordance with ASTM C 1038 and gave expansions below the permitted maximum.

The plotted data suggest that there is no consistent trend with respect to optimum sulfate based on the type of processing addition. Systems made with Low and Mid clinker and cement kiln dust appeared to indicate a small decreasing sulfate requirement with increasing cement kiln dust, while with the High clinker, the trend is reversed. There is no logical chemical

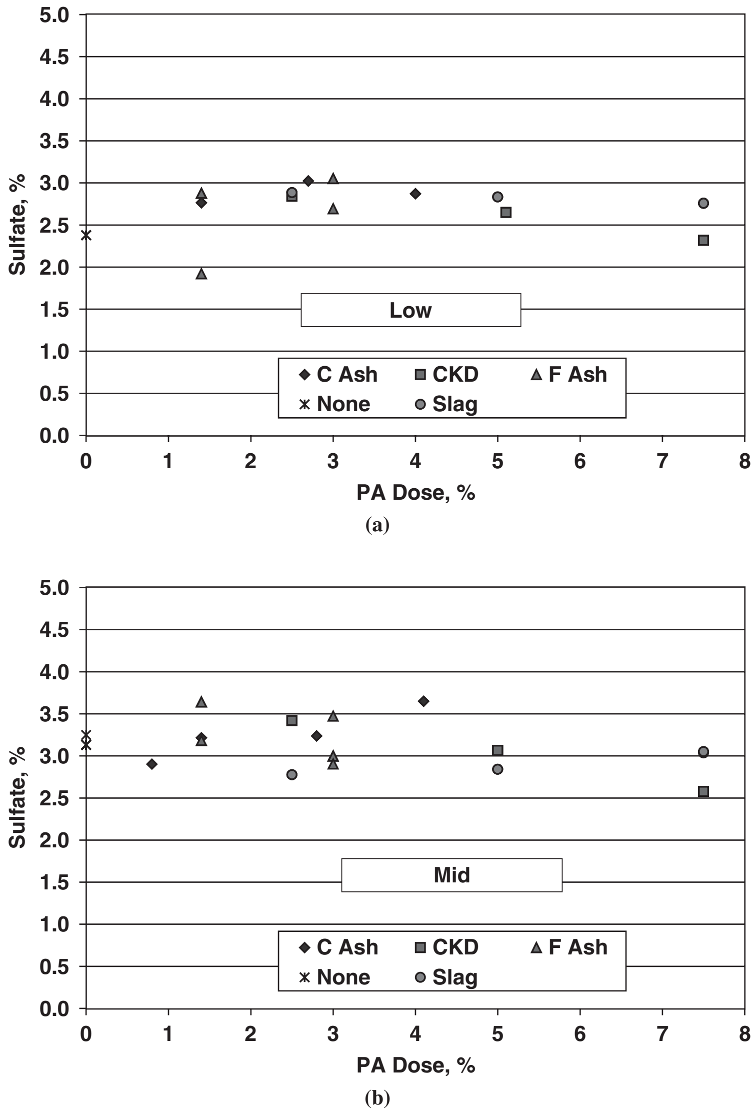


Figure 6. Sulfate dosages. (a) Sulfate dosage required for optimization for Low C_3A Clinker as a function of Processing Addition type and dosage. (b) Sulfate dosage required for optimization for Mid-range C_3A Clinker as a function of Processing Addition type and dosage.

explanation for this observation, and it is believed that the differences are largely noise from the test rather than real trends.

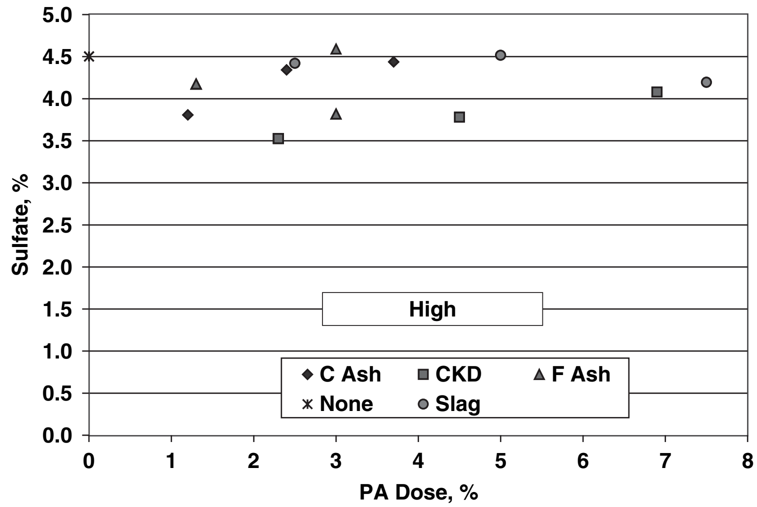
There is some variation in optimum sulfate with changing clinker type which is to be expected, as more gypsum is required with increasing C_3A content. There does not appear to be a significant difference between the mid-range C_3A cement and the same material containing limestone. Two data points in the system containing limestone appear to be outliers.

Particle Size Distribution

The particle size distributions of the cements prepared before addition of supplementary cementitious materials were also reviewed to see whether the presence of the processing additions had a measurable effect. Selected data are illustrated in Figure 5.

The particle size distribution for the samples in the Mid and LS clinkers is essentially the same, regardless of the amount or type of processing addition added to the clinker. Some effects were observed in the Low and High Clinkers containing fly ash as a processing addition. No effects were observed in the systems containing slag or cement kiln dust.

- *Class C Fly Ash.* The addition of this fly ash had no significant influence on PSD except for the High clinker, where the highest dosage of fly ash as a processing addition produced a slightly finer product in the 10 to 100 micron range.



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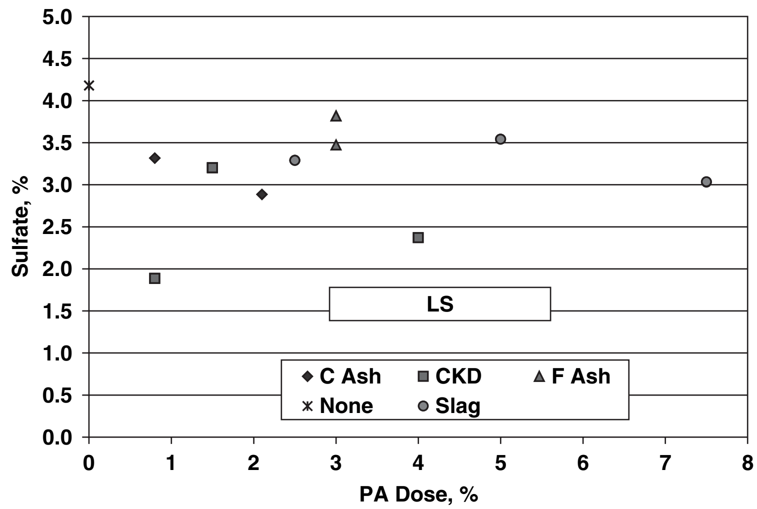

(d)

Figure 6. (Continued). Sulfate dosages. (c) Sulfate dosage required for optimization for High C_3A Clinker as a function of Processing Addition type and dosage. (d) Sulfate dosage required for optimization for Mid-range C_3A Clinker with limestone as a function of Processing Addition type and dosage.

- *Class F Fly Ash.* The addition of this fly ash had no significant influence on PSD except for the Low clinker, where the highest dosage of fly ash as a processing addition produced a slightly finer product in the 10 to 100 micron range.

Examination of Table 12 shows that there was some reduction in LOI in the samples that had the fine material removed. This is consistent with these samples made with LS Clinker, and it is likely that it is the limestone that is softer, and therefore ground finer that is likely to be removed with sieving. The reasons for the increase in calcium and silica contents are being investigated. No other significant trends were observed.

Microscopy

There were few effects related to the presence of processing additions visible in the images reviewed (Appendix C). The most notable observation is that in the systems containing fly ash, relatively large whole fly ash particles can be observed, indicating that they have not been crushed during laboratory grinding (e.g., Figure C11a).

Limestone is observed as small flakey particles in the LS Clinker (Figure C11b).

Generally, the ground particles were blocky and cubical (as opposed to flat and elongated), which is desirable as this provides a reduced surface area per unit volume, thus reducing water demand.

Calorimetry

Variations in the heat evolution were most strongly influenced by the supplementary cementing materials and by the type of clinker. The plots in Appendix D have been separated into sets by supplementary cementing material, then by Clinker. All tests were run on the same equipment by the same operator.

The final plot in Appendix D presents results from eight tests, all conducted on the same material (Combination 1) conducted to investigate the variability of the test method. Some tests were repeats from the same 100g sub-sample, while others were from different sub-samples. The plot illustrates the amount of variation that may occur within this test method. In general the maximum height of the second peak (silicate hydration) does not vary by more than 2.5 J/gh while the time of set does not vary by more than 3 hours. A large contributor to this variation is likely that water and powder were mixed by injection within the instrument. Newer equipment that allows more thorough external mixing has reportedly shown a markedly smaller variability.

The variation within sets for the silicate peaks in all of the plots is less than that of the repeatability plot. There is greater variation in some plots at later ages (12 hours and beyond), most notably in the sets of LS Clinker. There is a very tight spread in the sets comprising Low C_3A Clinker. This is consistent with the observations in optimum sulfate dosage noted above. The amount of variation in the systems containing no supplementary cementing material and Class F fly ash is small, and larger in those containing Class C fly ash and slag.

The magnitude of the silicate peak is greatest in the High C_3A cements and reduces with reducing C_3A content. The magnitude is greatest in the systems containing no supplementary cementing material, and lowest in that of slag, with the fly ash systems showing little difference between them and falling between the plain and slag data.

Most of the curves follow roughly the same shape, with the sulfate depletion hump generally masked in the main hydration peak. This is indicative of optimized systems. The exceptions to this are in the Mid C_3A and LS clinker systems containing Class C Fly ash as a processing addition, which all show a sulfate depletion hump occurring after the peak. Some Mid C_3A and LS clinkers containing slag as a supplementary cementing material also exhibit late sulfate depletion humps.

There is no definitive trend associated with processing addition type or dosage that correlates with these observations.

Paste and Mortar Tests

The data generated in Tasks 5.4 and 5.5 from paste and mortar tests and summarized in Table 11 were submitted to the statistician for analysis. The graphs and statistical analyses are provided in Appendix E.

The decision was made at the beginning of the project to conduct tests on as broad a range of materials as possible, without repetitions. This has meant that interpretation of the data has had to include reference to published precision statements for the various test methods to assess whether variations may be considered significant. Some repeat tests were conducted on test data that appeared to be outliers. Although every effort was made to minimize variability resulting from materials and sample fabrication and testing effects, some of the reported data points may still be outliers. Their inclusion in the analyses is considered conservative because they would indicate greater effects than may actually be occurring.

Appendix E has five sections wherein the data are presented in different ways:

- Appendix E1 provides the statistical analyses. The statistical analysis was an ANOVA with pair-wise comparisons of the least squares means to determine differences between combinations that do not contain processing additions with those that do, separated by clinker and/or supplementary material. Any pairing that showed an adjusted p-value using Dunnett's adjustment (reported in Appendix E1 as Adj p) (Example shown in Figure 7) that was less than 0.05 was considered to be significant.
- Appendix E2 provides a graphical overview of the effects of processing addition for each test. The plots do not differentiate between the types of processing addition used.

Differences of Least Squares Means

Proc Add	Proc Add	Estimate	Standard Error	DF	t Value	Pr > |t|	Adj P
C Ash	None	0.000162	0.003772	88	0.04	0.9658	1.0000
CKD	None	-0.01243	0.003781	88	-3.29	0.0014	0.0046
F Ash	None	0.003644	0.003864	88	0.94	0.3482	0.6639
Slag	None	0.001509	0.003761	88	0.40	0.6893	0.9702

Figure 7. Extract from Appendix E1 showing a Dunnett's Adjustment less than 0.05 in one data set, indicating a significant effect of cement kiln dust with respect to the control mix on the parameter being analyzed.

- Appendix E3 presents the same data as Appendix E2, but it identifies the individual types of processing addition.
- Appendix E4 shows regression plots through data that were considered meaningful to do so (as discussed below). These plots also indicated standard pass/fail limits for the given test methods as discussed below. (Figure 8)
- Appendix E5 presents the data from the mid and limestone clinkers in order to illustrate the effects of limestone on the performance of the systems.

The paste and mortar tests data reported in Table 11 were reviewed to identify those results of concern. Certain data were flagged (highlighted in Table 11) that were outside limits given in existing standards or outside limits selected as reasonable performance parameters. These limits were established as follows:

1. Mortar bar expansion, autoclave expansion, Vicat time of set, and 3- and 7-day cube compressive strength as established in AASHTO M 85 for Type I cement.
2. Shrinkage, paste flow, and air-entrainment as established in ASTM C 465.
3. 1-day strength of 50% of the 3-day strength and 28-day strength of 143% of the 7-day strength established in AASHTO M 85. These relative strengths are consistent with requirements for control cements in ASTM C 465 and industry norms for strength development.
4. Early stiffening, a penetration of greater than 50 at 11 minutes when tested in accordance with AASHTO T 185.
5. Comparative performance for all other parameters, equal to or better than the performance of the respective "control" clinker.

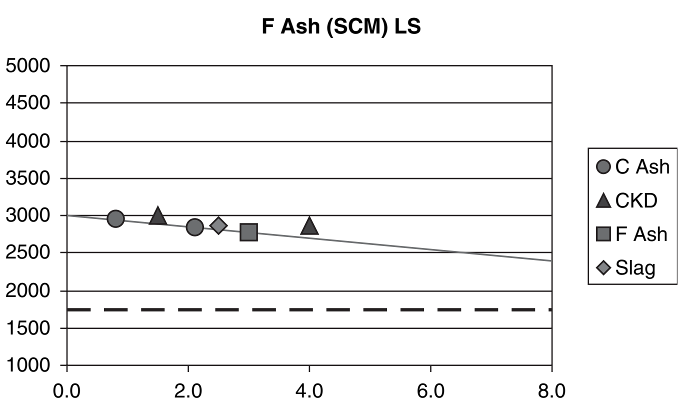

Figure 8. Example of a figure from Appendix E4 illustrating the effect of increasing PA dosage on a test parameter for a mixture made with LS Clinker containing 20% Class F fly ash. A line is plotted through the Class C fly ash as PS data points, and the dashed line shows the selected minimum pass fail limit for this test.

The data were also analyzed using the following process:

1. Test precisions were determined from the method statements. For instance, from AASHTO T 107, two tests by the same operator of similar samples may be expected to be within 0.07% of each other.
2. Using a linear extrapolation, the y-intercept (i.e. 0% PA dosage) for each test was determined for a given clinker/SCM combination, for all PA types within that combination.
3. The calculated m (slope) and B (y-intercept) for each test, SCM/clinker combination are reported in Tables 17 through 19.
4. The difference between performance at 0% and 5% PA dosage (for all PAs together) was calculated. Where this value exceeded the precision of the test method, and the r2 was greater than 70%, this was considered significant as shown in Tables 17 through 19.
5. For every combination of SCM and clinker, and where two or more dosages of a single PA were used, a linear regression was calculated (using the above y-intercept as an input) for each test. That is, a model correlating PA dosage with a change in performance was developed for each test and appropriate material combination.
6. The combinations in which the calculated models exhibited a difference in performance across 5% PA dosage that exceeded the precision of the test were identified. These were considered significant and plotted in Appendix E4. Those systems that gave a calculated modeled maximum variation less than the precision of the method were not considered significant.

Each of the following sections discusses:

1. The conclusions from the statistical analysis,
2. The observations from the data plots, and
3. Whether or not processing additions were influential in causing systems to exceed the limits described above.

Mortar Expansion: No trends due to changing processing addition dosage are observed. None of the samples tested failed.

Autoclave Expansion: The statistical analyses have identified systems containing cement kiln dust as a processing addition as affecting autoclave expansion for all clinkers considered together. Fly ash as a PA in High clinker with no SCM, and cement kiln dust in LS clinker with no SCM were found to be significant. The plots in Appendix E2 show an influence of clinker type with the High clinker exhibiting more autoclave expansion than the other clinkers. Some effect of the SCM is also observed with mixtures containing C ash as an SCM generally expanded more than the other SCMs. The plots in Appendix E4 indicate that increasing expansion is observed in the following:

<u>Clinker</u> LS, <u>SCM</u> C Ash, <u>PA</u> cement kiln dust and C Ash.

Table 17. Calculated model constants for effects of processing additions for tests on paste.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Autoclave	C Ash	High	-0.008	0.241	0.49	0.070	0.042	
		Low	0.007	0.048	0.71	0.070	0.037	
		LS	0.010	0.067	0.34	0.070	0.050	
		Mid	0.003	0.079	0.48	0.070	0.013	
	F Ash	High	-0.002	0.065	0.04	0.070	0.008	
		Low	0.004	-0.026	0.30	0.070	0.018	
		LS	0.003	-0.025	0.02	0.070	0.016	
		Mid	0.002	-0.026	0.38	0.070	0.011	
	None	High	0.004	0.091	0.48	0.070	0.020	
		Low	0.001	-0.009	0.02	0.070	0.003	
		LS	0.002	-0.017	0.04	0.070	0.011	
		Mid	0.003	-0.007	0.51	0.070	0.015	
	Slag	High	0.002	0.024	0.09	0.070	0.012	
		Low	0.001	0.004	0.12	0.070	0.005	
		LS	-0.001	0.009	0.02	0.070	0.003	
		Mid	0.003	-0.004	0.59	0.070	0.016	
Vicat Set Time	C Ash	High	0.8	113.1	0.02	30.0	4.0	
		Low	9.9	105.1	0.34	30.0	49.6	
		LS	-0.2	189.3	0.00	30.0	1.2	
		Mid	2.6	128.0	0.11	30.0	13.0	
	F Ash	High	2.4	98.7	0.50	30.0	11.8	
		Low	-13.4	253.0	0.36	30.0	67.1	
		LS	6.0	145.3	0.05	30.0	30.0	
		Mid	4.3	116.9	0.62	30.0	21.4	
	None	High	-0.2	93.0	0.11	30.0	1.2	
		Low	-2.3	119.8	0.42	30.0	11.6	
		LS	-4.4	146.6	0.17	30.0	21.8	
		Mid	-1.6	120.2	0.03	30.0	7.8	
	Slag	High	4.1	111.0	0.05	30.0	20.7	
		Low	-2.2	164.3	0.02	30.0	11.1	
		LS	1.1	143.3	0.32	30.0	5.4	
		Mid	-0.3	140.7	0.00	30.0	1.5	
Calorimetry	C Ash	High	-0.46	16.83	0.31	2.50	2.31	
		Low	0.10	11.59	0.16	2.50	0.52	
		LS	0.12	10.81	0.02	2.50	0.62	
		Mid	-0.12	13.32	0.26	2.50	0.62	
	F Ash	High	-0.43	15.94	0.74	2.50	2.14	
		Low	-0.01	11.38	0.00	2.50	0.04	
		LS	-0.38	11.69	0.20	2.50	1.91	
		Mid	-0.13	12.18	0.10	2.50	0.67	
	None	High	0.05	19.05	0.26	2.50	0.25	
		Low	-0.15	15.64	0.21	2.50	0.73	
		LS	-0.10	14.47	0.16	2.50	0.50	
		Mid	0.24	15.08	0.15	2.50	1.18	
	Slag	High	-0.17	11.08	0.12	2.50	0.87	
		Low	-0.15	9.48	0.39	2.50	0.75	
		LS	-0.08	8.42	0.51	2.50	0.42	
		Mid	-0.09	9.46	0.05	2.50	0.47	
Flow	C Ash	High	0.000	0.229	0.02	0.020	0.001	
		Low	0.001	0.199	0.13	0.020	0.006	
		LS	0.001	0.223	0.09	0.020	0.004	
		Mid	-0.001	0.231	0.13	0.020	0.003	
	F Ash	High	0.000	0.258	0.04	0.020	0.001	
		Low	0.001	0.229	0.27	0.020	0.007	
		LS	0.004	0.244	0.91	0.020	0.022	yes
		Mid	0.000	0.260	0.01	0.020	0.000	
	None	High	0.001	0.253	0.13	0.020	0.003	
		Low	0.000	0.212	0.00	0.020	0.001	
		LS	0.000	0.246	0.14	0.020	0.001	
		Mid	0.000	0.246	0.07	0.020	0.002	
	Slag	High	0.000	0.263	0.00	0.020	0.000	
		Low	0.000	0.248	0.00	0.020	0.000	
		LS	0.001	0.259	0.19	0.020	0.003	
		Mid	-0.001	0.265	0.55	0.020	0.003	

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope "m", y-intercept "b" and coefficient of determination "r2". If "r2" is greater than 0.7 then it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. "Delta" is the difference in result between 0 and 5% PA dosage based on calculated "m". If "delta" is greater than precision, the result is highlighted. If "r2" and "delta" are highlighted the effect is considered significant.

Table 18. Calculated model constants for effects of processing additions for tests on mortar.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Shrinkage	C Ash	High	0.003	-0.139	0.29	0.007	0.01	
		Low	-0.006	-0.081	0.62	0.007	0.03	
		LS	-0.005	-0.101	0.28	0.007	0.03	
		Mid	0.002	-0.116	0.19	0.007	0.01	
	F Ash	High	0.000	-0.110	0.00	0.007	0.00	
		Low	-0.002	-0.081	0.40	0.007	0.01	
		LS	-0.006	-0.089	0.48	0.007	0.03	
		Mid	0.000	-0.102	0.10	0.007	0.00	
	None	High	-0.003	-0.124	0.60	0.007	0.02	
		Low	0.001	-0.102	0.11	0.007	0.00	
		LS	0.003	-0.119	0.30	0.007	0.01	
		Mid	-0.003	-0.092	0.22	0.007	0.01	
	Slag	High	0.002	-0.121	0.22	0.007	0.01	
		Low	-0.002	-0.097	0.07	0.007	0.01	
		LS	0.002	-0.119	0.23	0.007	0.01	
		Mid	-0.004	-0.083	0.39	0.007	0.02	
AEA	C Ash	High	-0.005	0.400	0.23	0.050	0.03	
		Low	0.010	0.329	0.17	0.050	0.05	
		LS	-0.005	0.352	0.11	0.050	0.02	
		Mid	-0.005	0.451	0.30	0.050	0.03	
	F Ash	High	-0.005	0.950	0.23	0.050	0.03	
		Low	-0.019	0.977	0.34	0.050	0.09	
		LS	0.016	0.837	0.10	0.050	0.08	
		Mid	0.015	0.841	0.48	0.050	0.08	
	None	High	0.000	0.365	0.00	0.050	0.00	
		Low	0.006	0.398	0.05	0.050	0.03	
		LS	0.002	0.335	0.12	0.050	0.01	
		Mid	0.006	0.315	0.38	0.050	0.03	
	Slag	High	-0.017	0.504	0.20	0.050	0.09	
		Low	0.000	0.500	1.00	0.050	0.00	
		LS	-0.002	0.415	0.09	0.050	0.01	
		Mid	0.011	0.436	0.12	0.050	0.05	
Stiffening	C Ash	High	0.000	50.000	1.00	10.000	0.00	
		Low	4.836	15.966	0.20	10.000	24.18	
		LS	0.000	50.000	1.00	10.000	0.00	
		Mid	0.000	50.000	1.00	10.000	0.00	
	F Ash	High	0.000	50.000	1.00	10.000	0.00	
		Low	1.444	32.046	0.03	10.000	7.22	
		LS	0.000	50.000	1.00	10.000	0.00	
		Mid	0.000	50.000	1.00	10.000	0.00	
	None	High	-1.677	47.961	0.28	10.000	8.39	
		Low	2.244	28.617	0.07	10.000	11.22	
		LS	0.000	50.000	1.00	10.000	0.00	
		Mid	0.677	44.134	0.04	10.000	3.38	
	Slag	High	0.029	49.283	0.00	10.000	0.15	
		Low	6.058	15.164	0.45	10.000	30.29	
		LS	-0.421	49.971	0.12	10.000	2.10	
		Mid	2.308	35.033	0.14	10.000	11.54	
ASR	C Ash	High	-0.003	0.045	0.13	0.005	0.01	
		Low	0.000	0.001	0.02	0.005	0.00	
		LS	-0.003	0.013	0.25	0.005	0.01	
		Mid	-0.002	0.017	0.60	0.005	0.01	
	F Ash	High	0.000	-0.003	0.00	0.005	0.00	
		Low	0.000	-0.006	0.03	0.005	0.00	
		LS	0.001	-0.014	0.04	0.005	0.00	
		Mid	-0.001	-0.005	0.52	0.005	0.01	
	None	High	0.010	0.059	0.18	0.005	0.05	
		Low	-0.001	0.000	0.10	0.005	0.00	
		LS	-0.001	0.017	0.06	0.005	0.01	
		Mid	0.000	0.014	0.01	0.005	0.00	
	Slag	High	0.000	0.003	0.01	0.005	0.00	
		Low	-0.001	0.003	0.43	0.005	0.00	
		LS	0.001	0.004	0.92	0.005	0.00	
		Mid	0.001	-0.001	0.37	0.005	0.01	

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope “m”, y-intercept “b” and coefficient of determination “r2”. If “r2” is greater then 0.7 than it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. “Delta” is the difference in result between 0 and 5% PA dosage based on calculated “m”. If “delta” is greater than precision, the result is highlighted. If “r2” and “delta” are highlighted the effect is considered significant.

Table 19. Calculated model constraints for effects of processing additions for strength tests on mortar.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Strength 1	C Ash	High	-26.15	2209	0.19	165	130.8	
		Low	-9.05	1328	0.06	165	45.2	
		LS	-8.59	1643	0.01	165	43.0	
		Mid	-42.14	1764	0.47	165	210.7	
	F Ash	High	-76.59	2207	0.70	165	383.0	
		Low	3.26	1241	0.01	165	16.3	
		LS	-15.22	1629	0.06	165	76.1	
		Mid	-43.37	1932	0.87	165	216.9	yes
	None	High	63.65	2698	0.47	165	318.2	
		Low	14.54	1815	0.08	165	72.7	
		LS	10.68	2037	0.01	165	53.4	
		Mid	3.08	2631	0.00	165	15.4	
	Slag	High	-15.80	1281	0.12	165	79.0	
		Low	-11.04	797	0.23	165	55.2	
		LS	-2.60	856	0.01	165	13.0	
		Mid	14.90	935	0.14	165	74.5	
Strength 3	C Ash	High	-43.88	3466	0.36	259	219.4	
		Low	76.44	2471	0.73	259	382.2	yes
		LS	34.89	3356	0.14	259	174.5	
		Mid	-48.93	3284	0.22	259	244.7	
	F Ash	High	-45.64	3345	0.57	259	228.2	
		Low	11.00	2445	0.03	259	55.0	
		LS	-44.41	2989	0.37	259	222.0	
		Mid	-17.02	2999	0.06	259	85.1	
	None	High	-8.80	4402	0.22	259	44.0	
		Low	-11.17	3555	0.02	259	55.8	
		LS	11.21	3823	0.01	259	56.0	
		Mid	-17.63	4069	0.02	259	88.1	
	Slag	High	22.13	2456	0.18	259	110.6	
		Low	-25.28	1730	0.20	259	126.4	
		LS	-6.82	2105	0.06	259	34.1	
Strength 7	C Ash	High	-12.86	4265	0.02	329	64.3	
		Low	46.28	3532	0.20	329	231.4	
		LS	100.66	4427	0.28	329	503.3	
		Mid	-58.82	4214	0.22	329	294.1	
	F Ash	High	-3.36	3944	0.01	329	16.8	
		Low	28.45	3119	0.13	329	142.2	
		LS	-171.80	4175	0.59	329	859.0	
		Mid	11.05	3693	0.01	329	55.3	
	None	High	-1.37	5362	0.22	329	6.9	
		Low	5.69	4456	0.00	329	28.4	
		LS	10.60	4918	0.02	329	53.0	
		Mid	43.84	4939	0.13	329	219.2	
	Slag	High	-3.32	3895	0.00	329	16.6	
		Low	-51.75	3052	0.22	329	258.8	
		LS	17.95	3525	0.27	329	89.8	
		Mid	139.33	2806	0.44	329	696.7	
Strength 28	C Ash	High	29.83	5319	0.06	423	149.2	
		Low	-97.60	6010	0.50	423	488.0	
		LS	-196.04	6799	0.84	423	980.2	yes
		Mid	-33.82	5989	0.04	423	169.1	
	F Ash	High	13.52	5394	0.05	423	67.6	
		Low	22.10	5053	0.06	423	110.5	
		LS	-174.50	6072	0.52	423	872.5	
		Mid	-8.52	5812	0.01	423	42.6	
	None	High	-23.62	6285	0.04	423	118.1	
		Low	69.83	5761	0.28	423	349.2	
		LS	19.71	6278	0.04	423	98.6	
		Mid	-61.92	7093	0.23	423	309.6	
	Slag	High	9.57	5953	0.00	423	47.8	
		Low	31.34	6162	0.11	423	156.7	
		LS	-23.17	6188	0.13	423	115.8	
		Mid	-44.68	6622	0.11	423	223.4	

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope "m", y-intercept "b" and coefficient of determination "r2". If "r2" is greater than 0.7 then it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. "Delta" is the difference in result between 0 and 5% PA dosage based on calculated "m". If "delta" is greater than precision, the result is highlighted. If "r2" and "delta" are highlighted the effect is considered significant.

The plots in Appendix E4 indicate that decreasing expansion is observed in the following:

Clinker High, SCM C Ash, PA F Ash.

From Table 17, no trends were considered significant. Results exceeding pass/fail limits appear to be associated with clinker and SCM effects rather than with processing addition dosage.

Initial Set Time: No systems were found to show a statistically significant effect on initial set time. The plots in Appendix E2 show a small effect of clinker type with High clinkers generally setting sooner than the others. The LS clinker with C Ash as an SCM appeared to be generally slower than the other mixtures. From Appendix E3, it appears that systems containing slag as an SCM, Fly ash F as a PA resulted in some retardation. The plots in Appendix E4 indicate that increasing retardation is observed in the following:

Clinker Low, SCM C Ash, PA cement kiln dust
Clinker Mid, SCM F Ash, PA F Ash

However, reducing set times are observed in the following:

Clinker LS, SCM C Ash, PA C Ash
Clinker Low, SCM F Ash, PA C Ash, Slag and cement kiln dust
Clinker High, SCM None, PA C Ash
Clinker Mid, SCM None, PA C Ash

From Table 17, no trends were considered significant. Set times generally appeared to be within 30 minutes of each other for each family of Clinker, Processing addition, and Supplementary Cementing Material (Appendix E3). None of the tested samples failed the requirements of AASHTO M 80.

Calorimetry: No combination of materials was found to have a statistically significant effect on the amount of heat generated by the silicate reaction. The plots in Appendix E2 show a strong effect of SCM type with heat generated decreasing from None, through C Ash and F Ash to Slag. Clinker type is also apparent with High clinker type releasing slightly more heat than the others. No plots were considered significant for inclusion in Appendix E4. From Table 17, no trends were considered significant. The only combination of materials that exceeded the selected limits on heat generation associated with silicate reactions were related to supplementary cementing material rather than to any of the processing additions used in the program. There were no observable trends associated with PA dosage.

Flow: No combination of materials was found to have a statistically significant effect on paste flow. The plots in Appendix E2 show that systems containing Low clinker had lower water requirements than the other clinkers. C Ash as an SCM also reduced water requirement, while Slag slightly increased it. The plots in Appendix E4 indicate that increasing water requirement is observed in the following:

Clinker LS, SCM F Ash, PA cement kiln dust and C Ash

From Table 17, systems containing LS clinker and F Ash as an SCM exhibited increase in flow with increasing PA dosage for all PAs. Results exceeding pass/fail limits appear to be associated with clinker and SCM effects rather than with processing addition dosage.

Shrinkage: Systems containing cement kiln dust as a processing addition have a statistically significant effect on shrinkage for all clinkers together. The plots in Appendix E2 show that shrinkage is generally greater in systems made with High Clinker and lower when made with Low Clinker. The plots in Appendix E4 indicate that decreasing shrinkage is observed in the following:

Clinker High, SCM C Ash, PA F Ash
Clinker High, SCM F Ash, PA F Ash
Clinker High, SCM Slag, PA cement kiln dust
Clinker Mid, SCM C Ash, PA F Ash
Clinker LS, SCM None, PA Slag
Clinker LS, SCM Slag, PA Slag

However, increasing shrinkage is observed in the following:

Clinker High, SCM None, PA cement kiln dust
Clinker Low, SCM C Ash, PA cement kiln dust
Clinker Low, SCM F Ash, PA C Ash and cement kiln dust
Clinker LS, SCM C Ash, PA C Ash and cement kiln dust
Clinker LS, SCM F Ash, PA C Ash and cement kiln dust

From Table 18, no systems exhibited significant change in shrinkage with increasing PA dosage. All the systems that exceeded pass/fail limits contained cement kiln dust, including the following:

Clinker Low, SCM C Ash, PA cement kiln dust
Clinker LS, SCM C Ash, PA cement kiln dust

Air Entrainment: Systems containing Low Clinker, no SCM and cement kiln dust as PA were reported to have a statistically significant effect on the amount of air entrainer required to achieve a fixed air content. The plots in Appendix E2 show an increased AEA requirement for systems containing F Ash as an SCM. The plots in Appendix E4 indicate increasing AEA requirement in the following:

Clinker Low, SCM C Ash, PA cement kiln dust and C Ash
Clinker Mid, SCM F Ash, PA Slag and F Ash
Clinker Mid, SCM Slag, PA C Ash

However, decreasing AEA requirement is observed in the following:

Clinker High, SCM Slag, PA cement kiln dust
Clinker Low, SCM F Ash, PA cement kiln dust and C Ash

From Table 18, no systems exhibited significant change in AEA requirement with increasing PA dosage. Results exceeding selected limits appear to be primarily associated with SCM type.

Mortar Stiffening: Because the data were limited to an effective pass/fail set, statistics could not be used to flag the effects of processing addition on the risk of early stiffening. However, the plots suggest that increasing risk of early stiffening is indicated with Low clinker in all SCM types. From Table 18, no systems exhibited significant change in stiffening risk with increasing PA dosage. Results exceeding limits do not appear to be associated with processing additions, but rather the type of clinker and supplementary cementing material.

Alkali Silica Reaction: No combination of materials was found to have a statistically significant effect on alkali silica reactivity. The plots in Appendix E2 show that ASR expansion does not occur in systems containing F ash and Slag as an SCM. The greatest expansions are observed in systems with no SCM and made with High clinker. The plots in Appendix E4 show decreasing ASR related expansion is observed in the following:

Clinker High, SCM C Ash, PA F Ash and Slag
Clinker High, SCM F Ash, PA F Ash
Clinker Low, SCM F Ash, PA C Ash
Clinker Mid, SCM C Ash, PA Slag
Clinker Mid, SCM F Ash, PA F Ash and Slag
Clinker Mid, SCM None, PA Slag
Clinker LS, SCM C Ash, PA cement kiln dust

Increasing expansion is observed in the following:

Clinker Mid, SCM Slag, PA cement kiln dust
Clinker Mid, SCM None, PA cement kiln dust

From Table 18, no systems exhibited significant change in ASR with increasing PA dosage. However, comparison of figures on pages E3-34 to 36 in Appendix E shows that mixtures containing SCMs suppressed expansion, but some expansion did occur in some High C_3A systems without SCMs, including the control mixture. The control mixture (with no PA) expansion is significantly greater than the expansions of the control mixtures made with other clinkers. The aggregate was selected to be reactive; therefore, it is not surprising that expansion occurred in this system. Whether the additional expansion from the presence of the PAs results from testing variability or is a real effect is difficult to assess. XRF analysis of two samples from this set did not provide any additional information.

Strength—1 day: No combination of materials was found to have a statistically significant effect on 1-day strength. The plots in Appendix E2 show that 1-day strengths were affected by SCM type in that strengths were depressed with C Ash, F Ash, and slag in that order. There is a slight effect of the clinker type with highest strengths reported for High clinker and lowest for Low clinker. The plots in Appendix E4 show decreasing strength at 1 day in the following:

Clinker High, SCM F Ash, PA F Ash and PA Slag
Clinker Mid, SCM C Ash, PA Slag
Clinker Mid, SCM F Ash, PA Slag and F Ash
Clinker Mid, SCM None, PA C Ash
Clinker LS, SCM C Ash, PA cement kiln dust

Increasing strength at 1 day is observed in the following:

Clinker High, SCM None, PA C Ash

From Table 19, systems containing Mid clinker and F Ash as an SCM exhibited significant decreasing strength with increasing PA dosage for all PAs together. None of the test results fell outside the selected limits.

Strength—3 day: Systems containing Mid and LS Clinkers, no SCM and cement kiln dust as PA were reported to have a statistically significant effect on 3-day strength. The plots in Appendix E2 show that 3-day strengths were affected by SCM type in that strengths were depressed with C Ash, F Ash, and slag in that order. There is a slight effect of the clinker type with highest strengths reported for High clinker and lowest for Low clinker. The plots in Appendix E4 show decreasing strength at 3 days in the following:

Clinker High, SCM C Ash, PA Slag
Clinker Mid, SCM C Ash, PA Slag
Clinker Mid, SCM None, PA C Ash
Clinker LS, SCM F Ash, PA C Ash and cement kiln dust
Clinker Low, SCM None, PA Slag

Increasing strength at 3 days is observed in the following:

Clinker Low, SCM C Ash, PA cement kiln dust
Clinker Low, SCM None, PA F Ash
Clinker LS, SCM C Ash, PA cement kiln dust

From Table 19, systems containing Low clinker and C Ash as an SCM exhibited significant increasing strength with increasing PA dosage for all PAs together. None of the test results fell outside the selected limits.

Strength—7 day: Systems containing Mid Clinker, no SCM and cement kiln dust as PA, and LS Clinker, no SCM and F Ash as PA were reported to have a statistically significant effect on 7-day strength. The plots in Appendix E2 show that 7-day strengths were affected by SCM type in that strengths were depressed with C Ash, F Ash, and slag in that order. There is a slight effect of the clinker type with highest strengths reported for High clinker and lowest for Low clinker in some cases. The plots in Appendix E4 show decreasing strength at 7 days in the following:

Clinker Low, SCM Slag, PA Slag

Increasing strength at 7 days is observed in the following:

Clinker Mid, SCM Slag, PA cement kiln dust
Clinker LS, SCM C Ash, PA cement kiln dust

From Table 19, no systems exhibited significant change in 7-day strength with increasing PA dosage. All the systems close to or below the selected limit contained slag or C ash as an SCM including the following:

Clinker Low, SCM Slag, PA Slag

Strength—28 day: No systems were reported to have a statistically significant effect on 28-day strength. The plots in Appendix E2 show that 28-day strengths were affected by SCM type in that strengths were slightly depressed with Slag, C Ash, and F Ash in that order. In the system containing C Ash as an SCM, the LS Clinker gave the highest strengths. The plots in Appendix E4 show decreasing strength at 28 days in the following:

Clinker Low, SCM C Ash, PA cement kiln dust
Clinker Mid, SCM None, PA C Ash
Clinker Mid, SCM Slag, PA C Ash
Clinker LS, SCM C Ash, PA C Ash and cement kiln dust
Clinker LS, SCM F Ash, PA cement kiln dust

Increasing strength at 28 days is observed in the following:

Clinker High, SCM Slag, PA C Ash
Clinker Low, SCM None, PA F Ash

From Table 19, systems containing LS clinker and C Ash as an SCM exhibited significant decreasing strength with increasing PA dosage for all PAs together. None of the systems were below the selected limit.

Repeat Tests: The following paste and mortar tests, identified by the statistician as outliers, were repeated:

- Autoclave—Combinations 63 and 59;
- Set time—Combination 5;
- Calorimetry—Combination 59;
- Flow, Paste—Combinations 24 and 59;
- Shrinkage—Combination 76; and
- Strength 1, 7 and 28—Combination 59.

The results, compared with the original data are shown in Table 20. In all cases, when replacing the data with the results of the repeat tests, the effect was either insignificantly small or brought the data into the trends observed with the other test data.

Effects of Limestone

The following were observed when comparing datasets with and without limestone (Appendix E5):

- Autoclave expansions were slightly lower in systems containing limestone and no SCM.

Table 20. Comparison of original and repeat paste and mortar test results.

Test	Combination	Original Result	Repeat Result	Effect
Autoclave, %	59	0.115	0.090	Insignificant
Autoclave, %	63	0.000	0.180	Brings in line with other data
Set time, minutes	5	45	160	Brings in line with other data
Calorimetry, J/gh	59	19.92	17.37	Insignificant
Flow, Paste	24	0.23	0.21	Brings in line with other data
Flow, Paste	59	0.25	0.26	Insignificant
Shrinkage, %	76	0.208	0.154	Brings in line with other data
Strength, 1, psi	59	3198	3070	Insignificant
Strength, 7, psi	59	5298	5370	Insignificant
Strength, 28, psi	59	6711	6210	Brings in line with other data

- Setting times were about 20 to 40 minutes longer in systems containing limestone and C Ash and F Ash as a SCM.
- Heat generated was slightly lower in systems containing limestone and C Ash as an SCM, or no SCM.
- Water requirements were marginally lower in systems containing limestone and no SCMs.
- Shrinkage was higher in systems containing limestone and Slag as an SCM.
- AEA requirements were lower in systems containing limestone and C Ash and Slag as an SCM.
- The risk of stiffening was reduced in systems containing limestone.
- ASR expansions were not changed with the inclusion of limestone.
- 1-day strengths were slightly lower in systems containing limestone and no SCM or F Ash as an SCM.
- 3-day strengths were not changed with the inclusion of limestone.
- 7-day strengths were higher in systems containing limestone and C Ash as an SCM.
- 28-day strengths were higher in systems containing limestone and C Ash as an SCM.

These effects were generally small and notably not consistent for a given SCM and Clinker type.

Summary

Relatively few significant effects of processing additions were observed in the paste and mortar tests, with few clear or simple trends apparent.

Table 21 indicates whether a significant effect (based on statistical and graphical interpretations discussed above) was observed for any given test for a clinker, SCM, or PA combination. Seventy-two such effects were observed (out of a possible 740), without separating beneficial or detrimental effects. In an attempt to note whether these effects were biased toward any given material, the following totals were calculated:

- Clinker Type—LS = 23, Low = 19, Mid = 16, High = 14
- SCM Type—C Ash = 28, F Ash = 23, Slag = 12, None = 9
- PA Type—cement kiln dust = 25, C Ash = 19, Slag = 15, F Ash = 12, None = 1

These calculations indicate that LS clinker appeared involved in affected systems slightly more than the other clinkers, C Ash and F Ash were more likely to be involved than slag or plain systems, and cement kiln dust was much more likely to be involved than the other processing additions.

The parameters that appeared to be most sensitive to changing cementitious systems were shrinkage, ASR, and 28-day strength. For example:

- If using a clinker/SCM combination with high autoclave expansion, use of a PA may increase the risk of failing the test.
- C Ash may cause slight acceleration, while cement kiln dust could change setting time either way.
- Water requirement may be slightly increased with all PA types with some clinker SCM combinations.
- Cement kiln dust may increase shrinkage, while F Ash and Slag may decrease it.
- Cement kiln dust may reduce AEA requirement while F Ash may increase it.
- ASR expansion is generally likely to be reduced except for cement kiln dust which went both ways.
- Strengths may be changed either way.

In all cases the effects are small and not universally applicable for a range of clinkers and SCMs.

Based on the plots in Appendix E4, no significant effects resulting in a split between passing and failing were observed for any PA types at dosages below 1%. At higher dosages, occasional effects were observed for systems already at or close to the limit. Therefore, for systems using processing additions at dosages below 1%, no additional testing is required. Above 1%, systems should be tested to ensure compliance with a selected set of tests to ensure acceptable performance. The selection of tests is discussed below.

Concrete Tests

The data generated in Task 5.7 from concrete tests and summarized in Table 13 were submitted to the statistician for analysis. The graphs and statistical analyses are provided in Appendix F.

Appendix F has four sections wherein the data are presented in different ways.

- Appendix F1 provides the statistical analyses. The statistical analysis was an ANOVA with pair-wise comparisons of the least squares means to determine differences between combinations that do not contain processing additions with those that do, separated by clinker. Any pairing that showed an adjusted p-value using Dunnett's adjustment (reported in Appendix E1 as Adj p) that was less than 0.05 was considered significant.
- Appendix F2 provides a graphical overview of the effects of processing addition for each test.
- Appendix F3 provides the same data as Appendix F2, except the plots are separated out by clinker to allow effects of individual PA types to be observed.
- Appendix F4 presents that same data as Appendix F2, except only the data from the mid and limestone clinkers are

Table 21. Trends of combinations where test data are affected by processing addition.

Clinker type	SCM Type	Processing Addition type	Autoclave expansion, %	Vicat Time of Set, Initial, min	Flow, mL/650*100	Shrinkage, %	AEA, mL	ASR, Expansion, %	Strength, 1 day, psi	Strength, 3 day, psi	Strength, 7 day, psi	Strength, 28 day, psi
High	C Ash	C Ash										
High	C Ash	CKD										
High	C Ash	F Ash	X			X		X				
High	C Ash	Slag						X		X		
High	F Ash	C Ash										
High	F Ash	CKD							X			
High	F Ash	F Ash				X		X				
High	F Ash	Slag										
High	None	C Ash		X					X			
High	None	CKD				X						
High	None	F Ash										
High	None	None										
High	None	Slag										
High	Slag	C Ash										X
High	Slag	CKD				X	X					
High	Slag	F Ash										
High	Slag	None										
High	Slag	Slag										
Low	C Ash	C Ash										
Low	C Ash	CKD		X		X	X			X		X
Low	C Ash	F Ash										
Low	C Ash	Slag										
Low	F Ash	C Ash		X		X		X				
Low	F Ash	CKD		X		X	X					
Low	F Ash	F Ash										
Low	F Ash	None										
Low	F Ash	Slag		X								
Low	None	C Ash										
Low	None	CKD										
Low	None	F Ash								X		X
Low	None	None										
Low	None	Slag										
Low	Slag	C Ash										
Low	Slag	CKD										
Low	Slag	F Ash										
Low	Slag	Slag				X					X	
LS	C Ash	C Ash	X	X		X						X
LS	C Ash	CKD	X			X		X	X	X	X	X
LS	C Ash	F Ash										X
LS	C Ash	None										X
LS	C Ash	Slag										X
LS	F Ash	C Ash			X	X				X		
LS	F Ash	CKD			X	X						X
LS	F Ash	F Ash			X							
LS	F Ash	Slag			X							
LS	None	C Ash										
LS	None	CKD										
LS	None	F Ash										
LS	None	None										
LS	None	Slag				X						
LS	Slag	C Ash										
LS	Slag	CKD										
LS	Slag	F Ash										
LS	Slag	Slag										
Mid	C Ash	C Ash										
Mid	C Ash	CKD										
Mid	C Ash	F Ash				X						
Mid	C Ash	None										
Mid	C Ash	Slag						X	X	X		
Mid	F Ash	C Ash										
Mid	F Ash	CKD										
Mid	F Ash	F Ash		X				X				
Mid	F Ash	None										
Mid	F Ash	Slag					X	X	X			
Mid	None	C Ash		X					X	X		X
Mid	None	CKD						X				
Mid	None	F Ash										
Mid	None	None										
Mid	None	Slag						X				
Mid	Slag	C Ash					X					X
Mid	Slag	CKD						X			X	

included in order to illustrate the effects of limestone on the performance of the systems.

The concrete test data reported in Table 13 were reviewed to identify those results of concern. Certain data were flagged (highlighted in Table 13) that were outside limits given in existing standards or outside limits selected as reasonable performance parameters. These limits were established as follows:

1. Spacing factor maximum limit 0.008 in. as recommended by ACI 201.2R.
2. Set time is not more than 90 minutes longer than control (based on AASHTO M 194 and ASTM C 1602 recommendations).
3. Shrinkage is not more than 7.4% greater than control, based on the precision of test method.
4. Compressive strength is not less than 90% of control (based on ASTM C 465 requirements).
5. Chloride penetration is not greater than 4000 coulombs based on FHWA HPC Grade 1 (FHWA, 1997).
6. Freeze-thaw RDM is not less than 80% based on AASHTO M 194 requirements.
7. Salt scaling visual rating is not greater than 4 based on FHWA HPC Grade 1, and mass loss is not greater than 0.8 kg/m^2 based on some State DOT specifications.
8. Flexural strength is not less than 90% of control (similar to ASTM C 465 requirements for compressive strength).

The data were also analyzed using the following process:

1. Test precisions were determined from the method statements. For instance, from AASHTO T 197, two setting time tests by the same operator on similar samples may be expected to be within 23% of their average.
2. Using a linear extrapolation, the y-intercept (i.e., 0% PA dosage) for each test was determined for a given clinker/SCM combination, for all PA types within that combination.
3. The calculated m (slope) and b (y-intercept) for each test, SCM/clinker combination are reported in Tables 22 through 24.

Table 22. Calculated model constants for effects of processing additions for fresh concrete tests.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Slump	Paving	High	0.141	1.5	0.22	1.1	0.7	
		Mid	0.006	1.5	0.00	1.1	0.0	
	Structural	High	0.020	3.9	0.00	1.1	0.1	
		Low	0.150	5.7	0.02	1.1	0.8	
		LS	-0.136	2.7	0.57	1.1	0.7	
		Mid	-0.205	3.3	0.70	1.1	1.0	
AEA Efficiency	Paving	High	0.010	0.2	0.48	0.4	0.1	
		Mid	-0.002	0.2	0.17	0.4	0.0	
	Structural	High	0.004	0.2	0.61	0.4	0.0	
		Low	0.001	0.2	0.00	0.4	0.0	
		LS	-0.001	0.2	0.03	0.4	0.0	
		Mid	-0.002	0.2	0.20	0.4	0.0	
Set time	Paving	Mid	-2.298	211.6	0.04	56.0	11.5	
		Mid	2.395	234.8	0.10	56.0	12.0	
	Structural	High	5.536	224.8	0.80	56.0	27.7	yes
		Low	8.726	257.2	0.49	56.0	43.6	
		LS	4.945	243.3	0.31	56.0	24.7	
		Mid	1.955	239.7	0.14	56.0	9.8	
Slump loss	Paving	High	0.000	0.0	0.00	0.0	0.0	
		Mid	0.000	0.0	0.05	0.0	0.0	
	Structural	High	0.001	0.0	0.48	0.0	0.0	
		Low	0.001	0.0	0.03	0.0	0.0	
		LS	-0.001	0.0	0.51	0.0	0.0	
		Mid	0.000	0.0	0.00	0.0	0.0	
Flow loss	Paving	Low	-0.017	0.4	0.07	0.0	0.1	
		Mid	-0.078	0.4	0.64	0.0	0.4	
	Structural	High	-0.019	0.4	0.14	0.0	0.1	
		Low	-0.014	0.4	0.12	0.0	0.1	
		LS	-0.004	0.3	0.01	0.0	0.0	
		Mid	-0.007	0.3	0.06	0.0	0.0	

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope "m", y-intercept "b" and coefficient of determination "r2". If "r2" is greater than 0.7 then it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. "Delta" is the difference in result between 0 and 5% PA dosage based on calculated "m". If "delta" is greater than precision, the result is highlighted. If "r2" and "delta" are highlighted the effect is considered significant.

Table 23. Calculated model constants for effects of processing additions for hardened concrete tests.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Spacing	Paving	High	0.00	0.01	0.13	0.0	0.0	
		Mid	0.00	0.01	0.01	0.0	0.0	
	Structural	High	0.00	0.01	0.11	0.0	0.0	
		Low	0.01	-0.01	0.78	0.0	0.1	Yes
		LS	0.00	0.01	0.59	0.0	0.0	
		Mid	0.00	0.00	0.34	0.0	0.0	
Shrinkage 56	Paving	High	0.01	-0.06	0.44	0.0	0.0	
		Mid	0.00	-0.04	0.97	0.0	0.0	
	Structural	High	0.00	-0.03	0.56	0.0	0.0	
		Low	0.00	-0.05	0.00	0.0	0.0	
		LS	0.00	-0.04	0.37	0.0	0.0	
		Mid	0.00	-0.04	0.35	0.0	0.0	
C1202	Paving	High	524.60	2459	0.24	1357	2623	
		Mid	81.2	2141	0.80	1357	406	
	Structural	High	-97.5	4313	0.12	1357	488	
		Low	12.7	3368	0.00	1357	64	
		LS	-24.0	3505	0.03	1357	120	
		Mid	-43.7	3092	0.00	1357	218	
C672 Visual	Paving	High	0.0	3.32	0.00	1.0	0.0	
		Mid	-0.1	1.93	0.01	1.0	0.4	
	Structural	High	0.2	3.71	0.12	1.0	1.0	
		Low	0.0	0.51	0.03	1.0	0.1	
		LS	0.3	0.87	0.67	1.0	1.3	
		Mid	-0.1	1.93	0.91	1.0	0.7	
C672 Mass	Paving	High	0.46	4.81	0.01	0.2	2.3	
		Mid	-0.01	0.80	0.00	0.2	0.0	
	Structural	High	-0.60	6.00	0.14	0.2	3.0	
		Low	0.00	0.06	0.02	0.2	0.0	
		LS	-0.08	0.42	0.71	0.2	0.4	Yes
		Mid	-0.50	2.70	0.74	0.2	2.5	Yes

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope "m", y-intercept "b" and coefficient of determination "r2". If "r2" is greater than 0.7 then it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. "Delta" is the difference in result between 0 and 5% PA dosage based on calculated "m". If "delta" is greater than precision, the result is highlighted. If "r2" and "delta" are highlighted the effect is considered significant.

4. The difference between performance at 0% and 5% PA dosage (for all PAs together) was calculated. Where this value exceeded the precision of the test method, and the r2 was greater than 70%, this was considered significant as shown in Tables 22 through 24.

Each following section discusses

- The conclusions from the statistical analysis,
- The observations from the data plots, and
- Whether or not processing additions were influential in causing systems to exceed the limits described above

Slump: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show that the systems containing Low clinker had a large scatter, but no trend related to the PA dosage. Little variability was observed in the other clinkers. From Table 22, no systems exhibited significant change in slump with increasing PA dosage.

Air Void System: The air content of the individual mixtures was not considered because this is primarily controlled by the amount of air-entraining admixture added to each mix. Instead, the "efficiency" of the AEA was assessed by dividing the amount of air (in the fresh state) by the amount of AEA in the mix. The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 indicate some variability in the systems containing Low clinker but no clear trend in the structural mixtures. Increasing efficiency was observed in the paving mixtures made with High clinker with increasing PA dosage. In general, the air content determined using ASTM C 457 was lower than that determined in the fresh state. All of the systems contained a satisfactory air-void spacing factor. From Table 22, no systems exhibited significant change in AEA efficiency with increasing PA dosage. Appendix F2 indicates increasing spacing factor in structural mixes containing Low clinker with increasing PA dosage.

Initial Setting Time: The statistical analysis did not find significant effects from the presence of processing additions.

Table 24. Calculated model constants for effects of processing additions for concrete strength tests.

Test	SCM	Clinker	m	b	r2	Precision	Delta	Significant
Strength 1	Paving	High	-59.7	1917.5	0.1388	137.0	298.3	
		Mid	-68.8	1862.1	0.4792	137.0	343.9	
	Structural	High	62.5	1340.4	0.3313	137.0	312.3	
		Low	-107.4	1659.2	0.7839	137.0	537.0	Yes
		LS	-64.1	1768.3	0.0202	137.0	320.5	
		Mid	24.0	1618.1	0.1875	137.0	120.2	
Strength 3	Paving	High	-108.2	3075.2	0.0709	222.6	541.0	
		Mid	-147.8	3293.1	0.4513	222.6	739.1	
	Structural	High	135.1	2023.0	0.4147	222.6	675.3	
		Low	-60.1	2617.8	0.0568	222.6	300.4	
		LS	-123.9	2856.9	0.0142	222.6	619.4	
		Mid	-35.4	2953.6	0.1791	222.6	176.9	
Strength 7	Paving	High	-325.7	4619.8	0.1610	316.8	1628.4	
		Mid	-302.5	4719.0	0.8580	316.8	1512.6	Yes
	Structural	High	251.2	2668.5	0.4235	316.8	1255.8	
		Low	-94.9	3747.6	0.2005	316.8	474.7	
		LS	-79.4	3911.0	0.0269	316.8	397.0	
		Mid	-68.1	4072.9	0.0636	316.8	340.7	
Strength 28	Paving	High	-358.1	6082.5	0.3086	429.0	1790.3	
		Mid	-199.9	5977.3	0.8540	429.0	999.7	Yes
	Structural	High	143.7	5064.4	0.2737	429.0	718.7	
		Low	15.6	5033.8	0.0023	429.0	78.2	
		LS	-4.5	5332.4	0.0651	429.0	22.4	
		Mid	-80.0	5853.5	0.0007	429.0	399.9	
Strength 56	Paving	High	-177.5	6390.1	0.1097	471.6	887.5	
		Mid	-140.6	6627.1	0.6695	471.6	703.2	
	Structural	High	-11.3	6094.2	0.0022	471.6	56.6	
		Low	24.5	5699.5	0.0128	471.6	122.6	
		LS	60.9	5849.4	0.0622	471.6	304.7	
		Mid	-60.1	6439.8	0.3816	471.6	300.6	
Strength 90	Paving	High	-207.9	6924.8	0.1153	505.9	1039.7	
		Mid	-146.3	7136.6	0.8544	505.9	731.3	Yes
	Structural	High	40.5	6315.3	0.0317	505.9	202.5	
		Low	158.1	5744.3	0.3225	505.9	790.6	
		LS	70.9	6235.5	0.0206	505.9	354.6	
		Mid	49.6	6919.7	0.1859	505.9	248.2	
Flex 7	Paving	High	-33.5	701.9	0.1561	120.1	167.5	
		Mid	-40.4	731.6	0.7069	120.1	202.2	Yes
Flex 28	Paving	High	-1.6	860.0	0.0005	161.5	7.9	
		Mid	-24.4	920.5	0.2859	161.5	122.2	

Linear extrapolations of effect of PA dosage (for all PAs) on test results to fit data to a straight line, slope "m", y-intercept "b" and coefficient of determination "r2". If "r2" is greater than 0.7 then it is highlighted. Precision is based on acceptable range between tests as reported by the test methods. "Delta" is the difference in result between 0 and 5% PA dosage based on calculated "m". If "delta" is greater than precision, the result is highlighted. If "r2" and "delta" are highlighted the effect is considered significant.

The plots in Appendix F2 show increased set time in the Low clinker and shorter set time for the High clinker. Little significant variability is observed as a result of PA dosage. None of the systems failed the selected criteria. From Table 22, no systems exhibited significant change in setting time with increasing PA dosage.

Slump Loss and Flow Loss: The statistical analysis reported no significant effects from the presence of processing additions in flow loss in the structural mixtures. The plots in Appendix F2 do not show any clear trends except a slight increase in slump loss in systems containing Low clinker. From Table 22, no systems exhibited significant change in workability loss with increasing PA dosage.

Shrinkage: The statistical analysis indicated a significant effect of cement kiln dust as a processing addition in the structural mixes. The plots in Appendices F2 and F3 show the following trends with increasing PA dosage:

- Increasing shrinkage in paving mixes with both clinkers and
- Increasing shrinkage in structural mixes made with High and LS clinkers.

From Table 23, no systems exhibited significant change in shrinkage with increasing PA dosage.

The following systems exceeded the selected pass fail criteria as shown in Appendix F3:

- Structural mix, High Clinker, F Ash, Slag and cement kiln dust PA
- Structural mix, LS Clinker, cement kiln dust and Slag PA
- Structural mix, Low Clinker, cement kiln dust PA and C Ash

Chloride Penetration: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendices F2 and F3 do not show the clear trends in chloride penetration with increasing PA dosage, except that higher values are observed in systems made with High clinker. From Table 23, no systems exhibited significant change in chloride penetration with increasing PA dosage. Most of the systems exceeding the selected limit were made with the High clinker (including the control).

Freezing and Thawing: None of the systems have exhibited significant damage under test.

Salt Scaling: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendices F2 and F3 show little effect on scaling resistance from increasing PA dosage. However there is a clear trend that increasing damage is incurred with increasing C_3A (and alkali) content in the clinkers. This is likely because of osmotic effects associated with the increasing alkali contents of the systems made with increasing C_3A contents (Powers, 1965). From Table 23, systems made with LS and Mid clinker exhibited significant change in scaling resistance with increasing PA dosage. All of the systems containing High clinker exceeded the selected pass fail criteria.

1-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show the following trends with increasing PA dosage:

- Decreasing strength in paving mix, both clinkers;
- Decreasing strength in structural mix Low and LS clinkers; and
- Increasing strength in structural mix High clinker.

From Table 24, the structural mixes containing Low clinker exhibited significant change in strength with increasing PA dosage. Many of the systems containing high clinker exceeded the selected pass fail criteria (Appendix F3).

3-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show no clear trends with increasing PA dosage. From Table 24, no systems exhibited significant change in strength with increasing PA dosage. Many of the systems containing High clinker exceeded the selected pass/fail criteria (Appendix F3).

7-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show that the paving mixes containing Mid clinker showed decreasing strength with increasing PA dosage. From Table 24, the paving mixes containing Mid clinker exhibited significant change in strength with increasing PA dosage. Many of the systems containing High clinker exceeded the selected pass/fail criteria (Appendix F3).

28-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show that the paving mixes containing Mid clinker showed decreasing strength with increasing PA dosage. From Table 24, the paving mixes containing Mid clinker exhibited significant change in strength with increasing PA dosage. Few of the systems containing High clinker exceeded the selected pass/fail criteria, confined to the paving mixtures (Appendix F3).

56-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show no clear trends with increasing PA dosage. From Table 24, no systems exhibited significant change in strength with increasing PA dosage. Most of the systems exceeded the pass/fail criteria (Appendix F3). Few of the systems containing High clinker exceeded the selected pass/fail criteria, mainly in the paving mixtures.

90-Day Compressive Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show no clear trends with increasing PA dosage, except that strength decreases in paving mixers containing Mid clinker. From Table 24, the paving mixes containing Mid clinker exhibited significant change in strength with increasing PA dosage. Two of the systems containing High clinker exceeded the selected pass/fail criteria (Appendix F3).

7-Day Flexural Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show that the paving mixes containing Mid clinker showed decreasing strength with increasing PA dosage. From Table 24, no systems exhibited significant change in strength with increasing PA dosage. Some of the systems containing High clinker exceeded the selected pass/fail criteria (Appendix F3).

28-Day Flexural Strength: The statistical analysis did not find significant effects from the presence of processing additions. The plots in Appendix F2 show no clear trends with increasing PA dosage. From Table 24, the paving mixes containing Mid clinker exhibited significant change in strength with increasing PA dosage. Some of the systems containing Mid clinker exceeded the selected pass/fail criteria, all made with High Clinker (Appendix F3).

Repeat Tests

All of the tests were repeated on combination 78 because strength data were below the values expected. The results of the original data are shown in Table 13 as data set "78 orig." In all cases, the effect of replacing the data with the results of the repeat tests was insignificantly small to bring the data into the trends observed with the other test data.

Effects of Limestone

All of the data were reviewed to assess whether the addition of limestone to the Mid clinker affected system performance in concrete mixtures (Appendix F4). No significant trends were observed in the fresh, hardened, or strength properties of the mixtures.

Effects of Concrete Mix Design

All of the data were reviewed to assess whether the type of concrete mixture affected the trends discussed above. The following were observed:

- The effect of clinker type on slump loss was more marked in paving mixtures than in the structural mixtures.
- 3-Day, 28-day compressive strengths were lower in the structural mixtures than the paving mixtures

Summary

Few significant effects of processing additions were observed in the concrete tests, with limited clear or simple trends apparent. Tables 22 through 24 indicate whether a significant effect (based on statistical and graphical interpretations discussed above) was observed for any given test for a clinker, PA combination. Five such effects were observed (out of a possible 118), without separating beneficial or detrimental effects. All of these were associated with the Mid or Low clinkers. The parameter that appeared to be most sensitive to changing cementitious systems in the concrete tests was strength at relatively early ages, with no clear trend as to the direction of the effect. In all cases the effects are small and potentially masked by variability in the test methods.

Based on the plots in Appendix F2 and F3, no significant effects resulting in a split between passing and failing were observed for any PA types at dosages below 1%. Therefore, for systems using processing additions at dosages below 1%, no additional testing is likely required. Above 1%, systems should be tested to ensure compliance with a selected set of tests to ensure acceptable performance.

The research team considered whether a different lower limit, below which no testing was required, should be identified for each PA type. Table 25 shows the values below which no signal was detected based on plots in Appendix F.

There are fewer data points for the concrete tests, but they consistently suggest that more than 1% of any PA will result in observable changes in shrinkage, and early strength. If only the mortar and paste tests are considered (excluding Autoclave which can be adjusted for by the manufacturer) the different limits may be appropriate with F Ash showing no limit, C Ash—6%, Slag—4% and cement kiln dust 3%. Based on the need to be conservative and on the consideration that most cements will be used in concrete applications, it is recommended that a limit below which no testing is required is selected as 1%.

Several parameters govern the selection of an upper limit. At present ASTM C 465 does not limit the maximum amount of inorganic processing addition. One hard limit is imposed by the chemical requirements in the specification, i.e., LOI and insoluble residue. For some materials like Class F fly ash the maximum will be relatively low, while for slag it will be very high. The work conducted in this work did not go above 8%, therefore it would not be appropriate to recommend a limit above this value, although it is feasible that some materials will perform satisfactorily up to these levels. Practice elsewhere in the world is to allow up to 5% inorganic fillers. Selection of a limit is, therefore, somewhat arbitrary, but in order to assist harmonization with worldwide practice it would not be unreasonable to select an upper limit of 5%. Dosages above this may be considered supplementary cementitious materials and cements should be required to comply with AASHTO M 240.

The Selection of Tests to be Used in the Protocol

The following performance characteristics were observed to be influenced by the presence of processing additions in the paste and mortar tests:

- Autoclave expansion,
- Setting time,
- Water requirement,
- Shrinkage,
- AEA requirement,
- ASR expansion, and
- Strengths.

The following performance characteristics were observed to be influenced by the presence of processing additions in the concrete tests:

- Air void spacing factor,
- Salt scaling, and
- Strengths.

On this basis, these tests were recommended for inclusion in the protocol given in Chapter 2 with the exception of the following:

- ASR and salt scaling requirements have been omitted because these properties are primarily controlled by the other materials in the mixture and their proportions.

Table 25. Percentage of PA below which no effect was observed.

		% of PA below which no effect was observed															
	PA	**Class F Ash**				**Class C Ash**				**GGBFS**				**Cement Kiln Dust**			
	SCM	**N**	**F**	**C**	**S**	**N**	**F**	**C**	**S**	**N**	**F**	**C**	**S**	**N**	**F**	**C**	**S**
Paste Tests	C 1038																
	Autoclave							1								1	
	Set Time																
	Calorimetry																
	Flow						2								2		
Mortar Tests	Strength, 1																
	Strength, 3																
	Strength, 7												4				
	Strength, 28																
	Shrinkage						7	6						8	5	3	
	AEA																
	ASR													6			
	Conclusion					**6**				**4**				**3**			
Concrete Tests	AEA Effic																
	Set time																
	Slump loss	2								2				2			
	Flow loss																
	Shrinkage	1				1				1				2			
	C1202													2			
	Freeze thaw																
	Scaling																
	Strength 1	1				1				1				1			
	Strength 3	1				2				2				3			
	Strength 7					2				2				2			
	Strength 28					2				2				2			
	Strength 56	2				2				2				2			
	Strength 90					2											
	Flex 7					2				1				2			
	Flex 28					2								3			
	Conclusion	**1**				**1**				**1**				**1**			

Note: Blank cells indicate no effect was indentified for the PA dosages tested.

- Air void spacing in concrete has been omitted because that parameter is already required in the mortar tests.

Verification Tests

From the data collected to date, the following failures have been indicated:

- High C_3A clinker with 7.5% slag and with 0.4% CAsh2 exceeded maximum water requirement limits.
- Low C_3A clinker with 7.5% slag and the same clinker with 1.7% CAsh2 exceeded the maximum time of set. This is not surprising because slag may slow initial reactivity of cementitious systems.
- Four of five samples made with high C3A clinker exceeded C 1038 limits—including the control mix. This is likely indicating more about the methods used to optimize the mixtures than the performance of the PAs.
- High C_3A clinker with 1.3% cement kiln dust exhibited lower than acceptable strengths in mortar compression and concrete flexural tests.
- High C_3A clinker with 0.5% CAsh3 exhibited lower than acceptable concrete flexural strengths.

These data are consistent with the previous results in that the same tests are flagging potentially problematic systems. These data also indicate that the protocol is pointing out systems that may be unacceptable to the user or need to be fine-tuned by the manufacturer by adjusting system chemistry or fineness.

Evaluation of Recommendations Vis à Vis Concrete Proportioned with Supplementary Cementitious Materials

This section discusses how processing additions influence mixes containing supplementary cementing materials. This is to address the concern that if a concrete mixture is field batched to contain a maximum SCM content, but the cement already contains several percent of a processing addition (either the same material or another), then the mixture may perform in an undesirable manner and/or be out of specification. From the findings of this work the following conclusions are offered.

If a system containing an SCM is close to a specified limit for a performance requirement, largely due to the type of clinker or because of the effects of the SCM (e.g., high air-entraining admixture dosage requirement with the use of F Ash), then the probability increases that the system may not pass that test with increasing dosage of PA. Conversely, a system that is performing well (i.e., is well within a performance limit) before the addition of a processing addition is unlikely to fail such a parameter if a processing addition is added.

The effects of SCMs on concrete performance are well documented in the literature (e.g., Helmuth, 1987) and it appears that there may be a small incremental effect with the inclusion of PA, either beneficial or detrimental depending on the PA type and test under consideration. Effects from interactions between SCMs and PAs were not observed, neither beneficial nor detrimental.

The marked effects of SCM in paste and mortar tests were found to be as follows (Appendix E2):

- Autoclave expansion was increased with C Ash and showed lower scatter in the Slag mixture.
- Setting time showed increased scatter with the inclusion of C Ash.
- Heat of hydration dropped from control, C Ash, F Ash, to Slag in that order.
- Water requirement (flow) was improved with the use of C Ash.
- Shrinkage was reduced with the inclusion of F Ash and Slag.
- The AEA requirement to achieve a given air content was increased with the inclusion of F Ash.
- The presence of F Ash and Slag decreased ASR expansion.
- 1-, 3- and 7-day strengths reduced from control, C Ash, F Ash, and Slag in that order.
- 28-day strengths were slightly reduced in mixtures containing C Ash and F Ash.

All of these observations are consistent with previously reported trends due to the presence of supplementary cementitious materials in concrete.

At present, cement specifications do not provide for blending with additional materials at the concrete batch plant. This is not unreasonable, because it would be impossible for manufacturers to test their products with every available SCM on the market. The specifications should therefore be set up such that changes likely to be observed due to the use of supplementary cementitious materials in concrete are external to the cement specification.

Based on the observations that processing addition effects are small (particularly because dosages are much less than SCM dosages) and that current performance requirements appear to be providing sufficient protection, it is recommended that no provision for later addition of SCMs be made in the cement specifications, except to require that the amounts of processing additions be reported.

If, then, a specifier wishes to limit clinker content to a minimum (or maximum) amount for a specific performance requirement, then the cement supplier must be required to declare the type and amount of SCM, Limestone, and PA used to ensure that the concrete supplier can confirm that his cementitious system is in compliance with this requirement.

CHAPTER 5

Recommended Improvements to Specifications

Based on the testing conducted (Appendix E4), processing addition dosages of less than 1% by mass of cement were not observed to influence the performance of cementitious systems to a significant extent in any test. This was true for all the types of processing additions tested. Specifications should therefore allow the use of up to 1% processing addition without additional testing required beyond that already required in the cement specification.

The effects of PA dosages of 5 to 7.5% were largely insignificant, but there were some effects and trends observed, meaning that in order to be conservative, some testing is required if such PAs are to be used in a cement. A modified copy of AASHTO M 85 that reflects these changes is attached.

Some materials (e.g., cement kiln dust) appeared to be more likely to result in significant change than others. Although the experimental work was conducted using a limited range of materials most likely to be used, it is advisable that the protocol not be limited to a fixed list of materials that is clearly not exhaustive. All of the materials used as processing additions in this work were found to influence at least one performance criterion to a significant extent. Therefore, it is recommended that no distinction be made between the testing requirements for different processing additions.

If a dosage of PA greater than 1% is to be used, then the cementitious system should be tested to ensure that the performance of the system is satisfactory. ASTM C 465 sets out a number of tests as shown in Table 26. As noted in Chapter 3, there was some concern that the range of tests in ASTM C 465 did not cover some durability-related concerns, and this is supported by the test data (highlighted cells in Table 26). It is therefore suggested that a new protocol be developed based on those used in ASTM C 465, but including the tests shown in the last column in Table 26. Unnecessary tests (e.g., concrete strength testing beyond 28 days) should also be removed. No effect of processing additions was observed beyond 28 days in this work that was not already observed at younger ages. The primary reason for retaining strength testing in the protocol would be to ensure that mistakes in batching the test mixtures are detected. The quality of the air-void system, ASR resistance, and freeze-thaw resistance was not included in this protocol because the results of such testing are much more markedly affected by other parameters such as admixture type, amount of mixing, and aggregate grading. Any testing based on processing additions would largely be meaningless. Concrete specifications should require a given air-void system and durability, and concrete mixtures containing processing additions can be designed to achieve the required performance—as were the mixtures in this work.

A maximum amount of processing addition of 5.0% has been selected on the basis that this is consistent with global practice. Greater amounts may be considered as blended cements and can be covered under AASHTO M 240.

Pass-fail limits for the selected tests can be based on existing limits used in current standards, as shown in Table 26.

What should also be required is that the manufacturer should declare the amount and type of processing addition being used. Independent testing of this amount is desirable, and test methods will still have to be refined to accomplish this determination. The factors limiting precision of these techniques is that the elements and compounds in most processing additions are the same as those in the base cement. In addition, the dosages of processing additions are normally small, making data from a given analysis of a given element close to the detection limits of the test methods. Ideally, if the composition of the raw materials (i.e., clinker, sulfate system, limestone and processing addition) is known (or samples are available for analysis) then a least squares approach can be used to determine PA dosage. Some unique flags (e.g., insoluble residue and carbon content) can also be used to assess PA dosage if the composition of the raw materials is unknown, but the precision of the findings can vary.

Table 26. Current tests in ASTM C 465 and recommended protocol.

Tests on	ASTM C 465		Tests Conducted		Recommended Protocol Tests	
	Test	Method	Test	Method	Test	Limit
Raw materials	Chemistry	XRF	Chemistry	XRF/XRD/ AA	Chemistry	As per C 465
			Specific gravity	Helium pycnometer		
Cement	Fineness	C204	Particle size distribution	Laser	Fineness or particle size distribution	As per C 465
	Specific gravity	D891			Specific gravity	As per C 465
			Particle shape	Optical microscopy		
			Optimum SO_3	C 563		
			LOI, Insoluble residue	C 114		
Paste	Autoclave	C151	Autoclave	T 107	Autoclave	As per C 465
	Water requirement	C187	Water requirement.	T 129	Water requirement.	As per C 465
	Set time	C191	Set time	T 131	Set time	As per C 465
Mortar	Cube strength	C109	Cube strength	T 131	Cube strength	As per C 465
	Shrinkage	C596	Shrinkage	C 596	Shrinkage	As per C 465
	Air entraining admixture	C185	Air entraining admixture	T 137	Air entraining admixture	As per C 465
			Stiffening	T 185		
			Alkali silica reactivity	C 227		
Concrete	Compressive strength	C39	Compressive strength	T 22	Compressive strength	As per C 465 up to 28 days
	Flexural strength	C78	Flexural strength	T 97	Flexural strength	As per C 465 up to 28 days
			Slump loss	T 119		
			Set time	T 197		
			Air void system	C 457		
			Rapid chloride penetration	T 277		
			Freeze thaw	T 161		
			Deicer scaling	C 672 modified		

Highlighted cells indicate test parameters shown to be affected by inclusion of processing additions

The protocol should also require sufficient data be provided such that modifications to the phase calculations can be made to accommodate the PA. Guidance on how to make that accommodation is also needed.

A copy of the proposed protocol has been prepared in AASHTO format and is attached.

No changes have been made to the documents regarding organic processing additions because these were not covered under the scope of this project. Although some data were generated on the effects of limestone, changes to M 85 with respect to limestone have not been suggested.

CHAPTER 6

Conclusions and Recommended Research

Applicability to Highway Practice

The data obtained in this work have been used to develop recommended changes to current cement specifications and to recommend a test protocol that will allow producers to certify that cements containing processing additions are fit for the desired purpose.

Construction of highway pavements using slipforming is an activity that is sensitive to the performance of the concrete mixture, probably to a greater extent than any other structural system. The mixture is generally stiff, but it is essential that workability be retained during the time from mixing to final placing and consolidation. Loss of workability will result in poorly consolidated concrete, likely with a poor-quality surface. An additional complication is that entraining air is harder to achieve in low-slump mixtures, meaning that small changes in cementitious composition may have a significant influence on the air-void system, leading to potential loss of durability. Pavement slabs also have a large surface-to-volume ratio and the top surface is that which is directly exposed to loading and the environment, including deicing salt application. Durability is, therefore, a critical aspect of concrete pavement construction and loss of durability would be of great concern.

The test data have shown that the primary effects of processing additions are reflected primarily in paste and mortar tests that are not necessarily observed in concrete. This is consistent with previously reported observations. Both fresh and early hardened properties of pastes and mortars were in the list of properties observed to be influenced by the use of processing additions. However, concrete mixtures appear to be largely unaffected by processing additions.

Conclusions

In general, processing additions at low dosages do not have a large influence on the performance of cementitious systems. For dosages of all inorganic materials of less than 1%, no significant effects were observed in the tests conducted. Based on this, it is recommended that, for inorganic PA dosages less than 1%, no additional testing is required above that required of the AASHTO M 85 cement specification.

If the base cementitious system is close to the limits for a given parameter, then there is a possibility that inclusion of a PA may increase scatter in the test and/or increase the risk that the system may fail when tested. For this reason, it is recommended that, for dosages of PA greater than 1%, a suite of tests be conducted on the system in compliance with the requirements of a protocol attached. In order to address concerns, and as supported by the test data, some additional tests are recommended for inclusion in the protocol.

Laboratory tests were conducted on PA dosages up to and just above maximum limits imposed by existing cement specification limitations on loss-on-ignition and insoluble residue. These would appear to limit maximum dosages of the materials tested to between 3 and 8%, except for slag, which was not limited by this approach. A maximum dosage of any processing addition of 5.0% by mass of cement has been selected based on global practice.

The data indicated that LS clinker appeared to be involved in affected systems slightly more than the other clinkers; C Ash and F Ash were more likely to be involved than slag or plain systems; and cement kiln dust was much more likely involved than the other processing additions. Caution should therefore be applied with the use of cement kiln dust and similar materials as processing additions. The parameters that appeared to be most sensitive to changing cementitious systems were shrinkage, ASR, and 28-day strength.

No synergistic effects or interactions were observed with use of processing additions in systems containing supplementary cementitious systems.

A draft protocol has been prepared, along with a modified version of AASHTO M 85; these are attached.

Future Work and Recommended Research

A new protocol is provided as an attachment to this report in AASHTO format. Implementation of this protocol should take the form of adopting and referencing this protocol in cement specifications. Education modules should also be prepared and presented to cement manufacturers, test laboratories, and specifying authorities so that the implications and details of the protocol are understood by the relevant personnel.

Additional work that is required includes the need to

- Keep working on tests that can quantify the amount of processing addition in a given cement sample, particularly for blind samples in which the type of PA is unknown.
- Monitor the performance of systems constructed in the field, possibly built using "failing" materials, to assess the sensitivity of field concrete to the changes detected in the paste and mortar tests. This work should also review whether or not the selected limits in the protocol are appropriate.
- Develop statistical approaches to setting and applying limits to the various test methods. At present, compressive strength is specified based on statistical approaches, but no other test method is approached in this way. Work is needed to develop approaches to addressing any test result that narrowly fails (or passes) a given test result.

References

AASHTO M 85-01, "Standard Specification for Portland Cement"

AASHTO T 22-97, "Standard Specification for Compressive Strength of Cylindrical Concrete Specimens"

AASHTO T 97-97, "Flexural Strength of Concrete"

AASHTO T 106-01, "Standard Specification for Compressive Strength of Hydraulic Cement Mortar"

AASHTO T 107-00, "Standard Specification for Autoclave Expansion of Portland Cement"

AASHTO T 119-99, "Standard Specification for Slump of Hydraulic Cement Concrete"

AASHTO T 129-01, "Standard Specification for Normal Consistency of Portland Cement"

AASHTO T 131-01, "Standard Specification for Time of Setting of Hydraulic Cement by Vicat Needle"

AASHTO T 137-01, "Air Content of Hydraulic Cement Mortar"

AASHTO T 160-97, "Standard Specification for Length Change of Hardened Hydraulic Cement Mortar and Concrete"

AASHTO T 161-00, "Standard Specification for Resistance of Concrete to Rapid Freezing and Thawing"

AASHTO T 185-01, "Early Stiffening of Portland Cement "Mortar Method"

AASHTO T 197-00, "Standard Specification for Time of Setting of Concrete Mixtures by Penetration Resistance"

AASHTO T 277-96, "Standard Specification for Electrical Indication to Resist Chloride Ion Penetration"

ASTM C 33-02, "Specification for Concrete Aggregates"

ASTM C 114-00, "Test Methods for Chemical Analysis of Hydraulic Cement"

ASTM C150-02, "Specification for Portland Cement"

ASTM C 219-01, "Terminology Relating to Hydraulic Cement"

ASTM C 231-97, "Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method"

ASTM C 457-98, "Test Method for Microscopical Determination of Parameters of the Air Void System in Hardened Concrete"

ASTM C 465-99, "Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements"

ASTM C 563-96, "Test Method for Optimum SO_3 in Portland Cement"

ASTM C 595-02, "Specification for Blended Hydraulic Cements"

ASTM C 618-01, "Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete"

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ASTM C 672-98, "Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals"

ASTM C 1157-00, "Performance Specification for Hydraulic Cement"

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ATTACHMENTS

This section of the report includes two attachments:

1. Proposed Standard Specification for Mineral Processing Additions for Use in the Manufacture of Hydraulic Cements
2. Proposed Standard Specification for Portland Cement (i.e., Revised AASHTO Designation M85)

These proposed specifications are the recommendations of the NCHRP Project 18-11 staff at Construction Technology Laboratories, Inc. These specifications have not been approved by NCHRP or any AASHTO committee or formally accepted for the AASHTO specifications.

Proposed Standard Specification for

MINERAL PROCESSING ADDITIONS FOR USE IN THE MANUFACTURE OF HYDRAULIC CEMENTS

AASHTO Designation: M xxx

1. SCOPE

1.1 This specification pertains to the criteria and tests to be used for determining whether a single mineral processing addition, when used in the recommended amount at the option of the cement producer greater than 1%, up to and including 5.0% by mass in the manufacture of hydraulic cements, meets the requirements as prescribed by definition in specifications M 85 and M 240. Dosages at or below 1% do not require testing under this specification.

1.2 The following safety hazards caveat pertains only to the test methods described in this specification. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards

T 22 Compressive Strength of Cylindrical Concrete Specimens
T 97 Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)
T 98 Fineness of Portland Cement by the Turbidimeter
T 105 Chemical Analysis of Hydraulic Cement
T 106 Compressive Strength of Hydraulic Cement Mortar (Using 50-mm or 2-in. Cube Specimens)
T 107 Autoclave Expansion of Portland Cement
T 119 Slump of Hydraulic Cement Concrete
T 121 Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete
T 129 Normal Consistency of Hydraulic Cement
T 131 Time of Setting of Hydraulic Cement by Vicat Needle
T 137 Air Content of Hydraulic Cement Mortar
T 152 Air Content of Freshly Mixed Concrete by the Pressure Method
T 153 Fineness of Hydraulic Cement by Air Permeability Apparatus
T 177 Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading)

T 196 Air Content of Freshly Mixed Concrete by the Volumetric Method
T 231 Capping Cylindrical Concrete Specimens
M 85 Portland Cement
M 240 Blended Hydraulic Cement

2.2 ASTM Standards:
C 33 Concrete Aggregates
C 192 Making and Curing Concrete Test Specimens in the Laboratory
C 226 Air-Entraining Additions for Use in the Manufacture of Air-Entraining Hydraulic Cement
C 596 Drying Shrinkage of Mortar Containing Portland Cement
D 891 Specific Gravity, Apparent, of Liquid Industrial Chemicals
E 203 Water Using Volumetric Karl Fischer Titration

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3. MATERIALS

3.1 *Cements:*

3.1.1 The proposed addition shall be limited in use to a single plant, and the tests and test procedures shall be as specified and at least two pairs of cements shall be prepared from clinker representing each type under specific consideration.

3.1.2 The two companion cements to be made from any one clinker shall be ground to the same fineness within 7 m^2/kg when tested in accordance with T 98 or within 13 m^2/kg when tested in accordance with T 153. Each control cement shall comply with all requirements in the specification applicable to that type of cement, and shall not contain the proposed addition when tested by the method furnished by the producer or seller of the addition.

3.1.3 The percentage of each of the following shall be determined for each lot of cement tested: silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), ferric oxide (Fe_2O_3), calcium oxide (CaO), magnesium oxide (MgO), sulfur trioxide (SO_3), ignition loss, insoluble residue, sodium oxide (Na_2O), and potassium oxide (K_2O). There shall also be calculated the potential percentages of the following compounds: tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite. Determinations for the percentage of the addition shall be made, both on the control cements and on those with which the addition was interground, using the method proposed therefore by the sponsor.

3.2 *Aggregates*—The fine and coarse aggregates shall comply with M 6 or M 80 where appropriate; the coarse aggregate shall comply with the grading requirements for Size No. 57 or Size No. 67. A sufficient quantity from a single lot of coarse aggregate and from a single lot of fine aggregate shall be provided to complete all tests. To prevent the segregation of particle sizes in the fine aggregate, a single lot of sand sufficient for all tests shall either (1) be separated on the 4.75-mm (No. 4), 1.18-mm (No. 16), 300 µm

(No. 50), and 150 µm (No. 100) sieves and then be recombined in the required quantity for each batch; or (2) be blended while in a damp condition, and maintained in that condition for the duration of the tests. Under option (2), lots of appropriate size for single mortar and concrete batches shall be carefully split or quartered from the entire batch.

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4. GENERAL REQUIREMENTS

4.1 Processing additions shall conform to the respective requirements in this specification.

4.2 The source, character of the material, and means for the quantitative determination of the addition in the finished cement shall be furnished by the sponsor, manufacturer, or supplier of the addition, and the information shall form a part of the record of tests of the addition.

4.2.1 The specific gravity, run in accordance with 7.1.1 shall be within ±0.05 units of the value reported in 4.2.

4.3 Processing additions shall be evaluated by comparing cements containing the addition to otherwise identical cements from the same source without the addition, hereinafter designated the “control” cement.

4.4 The amount of the processing addition to be interground with the cement for evaluation purposes shall be determined by the sponsor of the addition.

4.4.1 The amount of the addition in the cement containing the addition and showing compliance with the requirements of this specification shall be determined quantitatively by means of the quantitative determination required by 4.2.

4.4.2 The amount of addition, so determined, shall be used to state the amount of addition that shows compliance with this specification.

4.4.3 When tests on cements containing the addition show compliance with the requirements of this specification, the addition in cement may be used in any amount up to the maximum amount showing compliance.

4.5 The cement produced for evaluation purposes with the processing addition shall comply with the appropriate Specifications M 85 or M 240, except that it contains the addition under test. The effect of the addition on the properties of the cement shall also be within the following limits:

4.5.1 The autoclave expansion of cement containing the addition, expressed as a percentage change in length, shall be not more than 0.10 greater than that of the corresponding control cement.

4.5.2 The percentage of water by mass of cement required for normal consistency of cement containing the addition shall not exceed that required by the corresponding control cement by more than 1.0. For those cements not limited to a fixed water requirement, the percentage of water by mass of cement required for standard consistency of the mortar used for strength determinations as described in 4.5.4 shall not be increased by more than 2.0 by the addition over that required for the control cement.

4.5.3 The time of setting of cement containing the addition shall not vary from the time of setting of the corresponding control cement by more than1h or 50%, whichever is the lesser.

4.5.4 The compressive strength of mortar cubes made with cement containing the addition, in accordance with ASTM C 109/C 109M, and tested at 1, 3, 7, and 28 days for all types, shall be compared with strengths obtained with the control cement at similar ages. The grand average of these individual strength percentages shall be not less than 95 % of the control cement values. It is required that cubes for companion cements be made and tested on the same days, with storage of specimens side by side in the same section of the moist cabinet during the 24-h curing period. Retesting of companion cements on the same, or a following, day is required in order to provide six, rather than three, test specimens for each cement and age of test.

4.5.5 The ultimate drying shrinkage (percent) of mortar made with cement containing the addition shall not be more than 0.025 greater than that of similar mortar made with the corresponding control cement when tested in accordance with ASTM C 596.

4.5.6 The amount of air-entraining addition required to produce 19 ± 3 % air in the mortar test made in accordance with T 137, with the cement containing the addition under test, shall be not greater than 120 % of the amount required to produce, within ± 1%, the air content obtained with the control cement. The air-entraining addition used shall meet the requirements of Specification ASTM C 226.

4.5.7 The compressive strength of the concrete made with cement containing the addition shall be compared with strengths obtained with the control cement at similar ages up to 28 days. The grand average of these individual strength percentages shall be not less than 90 % of the values for the control cement.

4.5.8 The flexural strength of concrete made with cement containing the addition shall be compared with strengths obtained with the control cement at similar ages up to 28 days. The grand average of these individual strength percentages shall be not less than 90 % of the values for the control cement.

4.6 Processing additions which provide maximum effects as grinding aids or pack set inhibitors may increase cement flowability to a point where mill retention time is reduced sufficiently to affect significantly the particle size distribution of the resulting cement and its physical-chemical properties. Since mill retention times are controllable by mechanical means in full-scale grinding mills, the true physical-chemical effects of the test additive may, in instances where full-scale tests have shown mill retention time reductions to have significant effects on the properties of the resulting cement, be determined for acceptance purposes by making supplementary laboratory or pilot-mill grinds.

In the event that the effects of the addition on the properties of cement are determined on the basis of laboratory or pilot mill grinds, this fact shall be entered in the report specified in Section 13, and the specific tests shall be indicated.

5. SAMPLING CEMENT

5.1 Samples of the plant-ground cement shall be taken from the product stream during grinding. Prior to the start of sampling a given lot of cement, the mill shall have run for 4 h or long enough to have reached equilibrium under the general conditions that are to govern during the sampling period.

NOTE 1 - Records should be kept as to the rate and continuity of feed of the addition, the form in which the addition is used, strength of solution, magnitude of circulating loads, mill discharge temperature, and feed rate of clinker and gypsum. Product fineness should be determined during the grinding immediately subsequent to sampling.

5.2 As the cement samples are taken, they shall be placed in sealable containers which shall be sealed immediately at the end of the sampling period. Prior to use, the samples of a given lot of cement shall be thoroughly blended to form a uniform, representative composite.

6. TEST METHODS

6.1 Determine the properties enumerated in this specification in accordance with the test methods prescribed in Sections 7 to 11.

7. TESTS ON CEMENT

7.1 Test cement in accordance with the following standards:

7.1.1 Chemical Analysis of Cement—T 105.

7.1.2 Compound Composition—M 85.

7.1.3 Fineness of Cement—t 98 or T 153.

7.1.4 Normal Consistency—T 129.

7.1.5 Time of Setting (Vicat)— T 131.

7.1.6 Autoclave Expansion—T 107.

7.1.7 Air Content of Mortar—T 137.

7.1.8 Compressive Strength of Mortar—T 106.

7.1.9 Drying Shrinkage of Mortar—ASTM C 596.

8. CONCRETE MIXTURES

8.1 Preparation and Weighing—Prepare all materials used in making the concrete mixtures and make all weighings as prescribed in ASTM C 192. Report the amount of mixing water used in each batch on the basis of saturated, surface-dry aggregates.

8.2 Proportions—Design one basic concrete mixture having an actual cement content of 307 ± kg/m3 (517 ± 6 5 lb/yd3), and use in all concrete tests herein specified. Adjust the water content of mixtures to provide concrete having a consistency equal to a 64 ± 13-mm (2 ½ ± 1/2 -in.) slump in each case.

Adjust the ratio of fine to coarse aggregate to the optimum for concrete to be consolidated by hand rodding. Recommended trial values for the percentage of fine aggregate in the total aggregate, by absolute volume, are as follows:

Coarse Aggregate, Maximum 25.0 mm (1 in.)	Concrete Without Entrained Air
Angular	45
Rounded	40

8.3 Mixing of Concrete—Mix the concrete in accordance with ASTM C 192 except as follows: Hand mixing will not be permitted. The rated capacity of the machine mixer shall not be more than twice the size of the batch used.

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9. TESTS ON FRESHLY MIXED CONCRETE

9.1 Test samples of the freshly mixed concrete for slump in accordance with T 119; unit weight in accordance with T 121; and air content in accordance with T 121, T 152, or T196.

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10 TEST SPECIMENS OF HARDENED CONCRETE

10.1 *Number of Specimens*—At least three specimens shall be made for each test condition. For each cement containing an addition and its companion control cement, make three rounds of concrete mixed on different days. One round of mixes on a given day shall include both the cement containing the addition and its companion control cement. From each round, make at least one test specimen for each test condition. If necessary, to obtain enough concrete for all test specimens to be made in any one round, it may be necessary to make more than one concrete batch for each round.

10.2 *Types of Specimens:*

10.2.1 *Compressive Strength*—Compressive strength test specimens shall be cylinders made and cured as prescribed in ASTM C 192. Cylinders shall be capped as prescribed in T 231.

10.2.2 *Flexural Strength*—Flexural strength test specimens shall be beams made and cured as prescribed in ASTM C 192.

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11. TESTS ON HARDENED CONCRETE

11.1 Test the specimens on hardened concrete, as specified in Section 11, in accordance with the following methods and at the specified ages:

11.1.1 *Compressive Strength*—Test specimens in accordance with T 22 at ages 3, 7, and 28 days, except also test Type III cement at 24 h.

11.1.2 *Flexural Strength*—Test specimens in accordance with T 177 or T 97, at ages 3, 7, and 28 days, except also test Type III cement at 24 h. By either method of test, turn the specimen on its side with respect to its position as molded and center it on the bearing blocks.

12. REPORT

12.1 The report covering the results of the evaluation of a material proposed for use as a processing addition in the manufacture of portland cement under this specification shall include the following information:

12.1.1 Trade name, source and character of the material, and the amount recommended for use, together with means for determination of the proposed addition in the finished cement, all as furnished by the sponsor, manufacturer, or seller of the addition,

12.1.2 If the proposed processing addition is a liquid, the specific gravity and percent water content by mass,

12.1.3 Detailed results of all analyses and tests prescribed by this specification, and the amount of the addition used, as well as other pertinent information required,

12.1.4 Comparison of test results to determine compliance with the requirements prescribed in 4.4,

12.1.5 Name and location of the laboratory or laboratories that made the tests covered by the report,

12.1.6 Include as an appendix to the report, letters of certification from the various cement manufacturers stating the name of the addition, the amount used, and the type of cement in which used, and

12.1.7 The highest amount of addition that has demonstrated compliance with the specification in a particular type of cement (see 4.4.3).

13. KEYWORDS

13.1 additions; hydraulic cements; processing

Proposed Standard Specification for

PORTLAND CEMENT

AASHTO Designation: M 85-xx
ASTM Designation: C 150-xx

1. SCOPE

1.1. This specification covers eight types of portland cement as follows (Note 1):

1.1.1. *Type I*—For use when the special properties specified for any other type are not required.

1.1.2. *Type IA*—Air-entraining cement for the same uses as Type I, where air-entrainment is desired.

1.1.3. *Type II*—For general use, more especially when moderate sulfate resistance or moderate heat of hydration is desired.

1.1.4. *Type IIA*—Air-entraining cement for the same uses as Type II, where air-entrainment is desired.

1.1.5. *Type III*—For use when high early strength is desired.

1.1.6. *Type IIIA*—Air-entraining cement for the same use as Type III, where air-entrainment is desired.

1.1.7. *Type IV*—For use when low heat of hydration is desired.

1.1.8. *Type V*—For use when high sulfate resistance is desired.

Note 1—Some cements are designated with a combined type classification, such as Type I/II, indicating that the cement meets the requirements of the indicated types and is being offered as suitable for use when either type is desired.

1.2. When both SI and inch-pound units are present, the SI units are the standard. The inch-pound units are approximations listed for information only.

1.3. The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:

- R 11, Indicating Which Places of Figures Are to Be Considered Significant in Specified Limiting Values
- T 98, Fineness of Portland Cement by the Turbidimeter
- T 105, Chemical Analysis of Hydraulic Cement
- T 106M/T 106, Compressive Strength of Hydraulic Cement Mortar (Using 50-mm or 2-in. Cube Specimens)

- T 107, Autoclave Expansion of Hydraulic Cement
- T 127, Sampling and Amount of Testing of Hydraulic Cement
- T 131, Time of Setting of Hydraulic Cement by Vicat Needle
- T 137, Air Content of Hydraulic Cement Mortar
- T 153, Fineness of Hydraulic Cement by Air Permeability Apparatus
- T 154, Time of Setting of Hydraulic Cement by Gillmore Needles
- T 186, Early Stiffening of Hydraulic Cement (Paste Method)
- M xxx, Mineral Processing Additions for Use in the Manufacture of Hydraulic Cements

2.2. *ASTM Standards*:

- C 33, Concrete Aggregates
- C 186, Heat of Hydration of Hydraulic Cement
- C 226, Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement
- C 452, Potential Expansion of Portland Cement Mortars Exposed to Sulfate
- C 465, Processing Additions for Use in the Manufacture of Hydraulic Cements
- C 563, Optimum SO_3 in Portland Cement
- C 1038, Expansion of Portland Cement Mortar Stored in Water

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. *portland cement*—a hydraulic cement produced by pulverizing clinker, consisting essentially of hydraulic calcium silicates, and usually containing one or more of the following:

- water,
- calcium sulfate,
- up to 5% limestone, and
- processing additions.

3.1.2. *air-entraining portland cement*—a portland cement containing an interground air-entraining addition.

3.1.3. *hydraulic cement*—a cement that sets and hardens by chemical interaction with water and is capable of doing so underwater.

4. ORDERING INFORMATION

4.1. Orders for material under this specification shall include the following:

4.1.1. This specification number and date;

4.1.2. Type or types allowable. If no type is specified, Type I shall be supplied;

4.1.3. Any optional chemical requirements from Table 2, if desired;

4.1.4. Any optional physical requirements from Table 4, if desired.

Note 2—Cement conforming to the requirements for all types are not carried in stock in some areas. In advance of specifying the use of other than Type I cement, determine whether the proposed type of cement is, or can be made, available.

5. INGREDIENTS

5.1. The cement covered by this specification shall contain no addition except as follows:

5.1.1. *Portland Cement Clinker.*

5.1.2. *Water or Calcium Sulfate, or Both*—The amounts shall be such that the limits shown in Table 1 for sulfur trioxide and loss-on-ignition shall not be exceeded.

Table 1—Standard Chemical Requirements[a]

Cement Type	Applicable Test Method	I and IA	II and IIA	III and IIIA	IV	V
Silicon dioxide (SiO_2), min, percent	T 105	—	20.0[b, c]	—	—	—
Aluminum oxide (Al_2O_3), max, percent	T 105	—	6.0	—	—	—
Ferric oxide (Fe_2O_3), max, percent	T 105	—	6.0[b, c]	—	6.5	—
Magnesium oxide (MgO), max, percent	T 105	6.0	6.0	6.0	6.0	6.0
Sulfur trioxide (SO_3),[d] max, percent	T 105					
When (C_3A)[f] is 8 percent or less		3.0	3.0	3.5	2.3	2.3
When (C_3A)[f] is more than 8 percent		3.5	[e]	4.5	[e]	[e]
Loss on ignition, max, percent	T 105	3.0	3.0	3.0	2.5	3.0
Insoluble residue, max, percent	T 105	0.75	0.75	0.75	0.75	0.75
Tricalcium silicate (C_3S)[f], max, percent	See Annex A.1	—		—	35[b]	—
Dicalcium silicate (C_2S)[f], min, percent	See Annex A.1	—	—	—	40[b]	—
Tricalcium aluminate (C_3A)[f], max, percent	See Annex A.1	—	8	15	7[b]	5[c]
Sum of $C_3S + 4.75C_3A$, max, percent[g]		—	100[h]			

[a] See Note 2.
[b] Does not apply when the heat of hydration limit in Table 4 is specified.
[c] Does not apply when the sulfate resistance limit in Table 4 is specified.
[d] There are cases where optimum SO_3 (using ASTM C 563) for a particular cement is close to or in excess of the limit in this specification. In such cases where properties of a cement can be improved by exceeding the SO_3 limits stated in this table, it is permissible to exceed the values in the table, provided it has been demonstrated by ASTM C 1038 that the cement with the increased SO_3 will not develop expansion in water exceeding 0.020 percent at 14 days. When the manufacturer supplies cement under this provision, he shall, upon request, supply supporting data to the purchaser.
[e] Not applicable.
[f] See Annex A.1 for calculation. Note that inclusion of mineral processing additions will require modifications to the Bogue calculations to prevent errors.
[g] See Note 4
[h] In addition, 7-day heat of hydration testing by ASTM C 186 shall be conducted at least once every six months. Such testing shall not be used for acceptance or rejection of the cement, but results shall be reported for informational purposes.

5.1.3. *Limestone*—The amount shall be not more than 5.0 percent by mass such that the chemical and physical requirements of this standard are met (See Note 3). The limestone, defined in ASTM C 51, shall be naturally occurring and consist of at least 70 percent by mass of one or more of the mineral forms of calcium carbonate.

Note 3—The standard permits up to 5 percent by mass of the final cement product to be naturally occurring, finely ground limestone, but does not require that limestone be added to the cement. Cement without ground limestone can be specified in the contract or order.

5.1.4. *Mineral processing additions* - They may be used in the manufacture of the cement up to a maximum of 5.0% by mass of cement. Only one mineral processing addition may be used at a time. If the total amount of processing additions used does not exceed 1 percent of the weight of portland cement clinker, no additional testing is required. For dosages greater than 1% and up to 5.0% such materials may be used provided they have been shown to meet the requirements of M xxx. If mineral processing additions are used, the manufacturer is required to declare the amount (or range) of processing addition used, expressed as a percentage of cement mass, along with the oxide composition of the processing addition.

5.1.5. *Organic Processing Additions*—They shall have been shown to meet the requirements of ASTM C 465 in the amounts used or greater and the total amount of organic processing additions used shall not exceed one percent of the weight of portland cement clinker.

5.1.6. *Air-entraining Addition (for Air-entraining Portland Cement Only)*—The interground addition shall conform to the requirements of ASTM C 226.

6. CHEMICAL COMPOSITION

6.1. Portland cement of each of the eight types shown in Section 1 shall conform to the respective standard chemical requirements prescribed in Table 1. In addition, optional chemical requirements are shown in Table 2.

Note 4—The limit on the sum, C_3S + 4.75 C_3A, in Table 1 provides control on the heat of hydration of the cement and is consistent with an ASTM C 186 7-day heat of hydration limit of 335 kJ/kg (80 cal/g).

Table 2—Optional Chemical Requirements[a]

Cement Type	Applicable Test Method	I and I A	II and II A	III and III A	IV	V	Remarks
Tricalcium aluminate (C_3A),[b] max, percent	See Annex A.1	—	—	8	—	—	for moderate sulfate resistance
Tricalcium aluminate (C_3A),[b] max, percent	See Annex A.1	—	—	5	—	—	for high sulfate resistance
Equivalent alkalies (Na_2O + 0.658K_2O), max, percent	T 105	0.60[c]	0.60[c]	0.60[c]	0.60[c]	0.60[c]	low-alkali cement

[a] These optional requirements apply only if specifically requested. Availability should be verified. See Note 2 in Section 4.

[b] See Annex A.1 for calculation.

[c] Specify this limit when the cement is to be used in concrete with aggregates that are potentially reactive and no other provisions have been made to protect the concrete from deleteriously reactive aggregates. Reference to ASTM C 33 for information of potential reactivity of aggregates.

7. PHYSICAL PROPERTIES

7.1. Portland cement of each of the eight types shown in Section 1 shall conform to the respective standard physical requirements prescribed in Table 3. In addition, optional physical requirements are shown in Table 4.

Table 3—Standard Physical Requirements

Cement Type[a]	Applicable Test Method	I	IA	II	IIA	III	IIIA	IV	V
Air content of mortar, volume, percent:[b]	T 137								
Max		12	22	12	22	12	22	12	12
Min		—	16	—	16	—	16	—	—
Fineness, specific surface, m^2/kg (alternative methods):[c]									
Turbidimeter test:	T 98								
Average value, min[d]		160	160	160	160	—	—	160	160
Min value, any one sample[e]		150	150	150	150	—	—	150	150
Average value, max[d]		—	—	240[f]	240[f]	—	—	240	—
Max value, any one sample[e]		—	—	245[f]	245[f]	—	—	245	—
Air permeability test:	T 153								
Average value, min[d]		280	280	280	280	—	—	280	280
Min value, any one sample[e]		260	260	260	260	—	—	260	260
Average value, max[d]		—	—	420[f]	420[f]	—	—	420	—
Max value, any one sample[e]		—	—	430[f]	430[f]	—	—	430	—
Autoclave expansion, max, percent	T 107	0.80	0.80	0.80	0.80	0.80	0.80	0.80	0.80
Strength, not less than the value shown for the ages indicated below:[f]									
Compressive strength, MPa (psi)	T 106M/106								
1 day		—	—	—	—	12 (1740)	10.0 (1450)	—	—
3 days		12.0 (1740)	10.0 (1450)	10.0 (1450) 7.0[h] (1020)[h]	8.0 (1160) 6.0[h] (870)[h]	24.0 (3480)	19.0 (2760)	—	8.0 (1160)
7 days		19.0 (2760)	16.0 (2320)	17.0 (2470) 12.0[h] (1740)[h]	14.0 (2030) 19.0[h] (1310)[h]	—	—	7.0 (1020)	15.0 (2180)
28 days		—	—	—	—	—	—	17.0 (2470)	21.0 (3050)
Time of setting (alternative methods):[i]									
Gillmore test:	T 154								
Initial set, min, not less than		60	60	60	60	60	60	60	60
Final set, min, not more than		600	600	600	600	600	600	600	600
Vicat test:[j]	T 131								
Time of setting, min, not less than		45	45	45	45	45	45	45	45
Time of setting, min, not more than		375	375	375	375	375	375	375	375

[a] See Note 2.

[b] Compliance with the requirements of this specification does not necessarily ensure that the desired air content will be obtained in concrete.

[c] Either of the two alternative fineness methods may be used at the option of the testing laboratory. However, when the sample fails to meet the requirements of the air-permeability test, the turbidimeter test shall be used, and the requirements in this table for the turbidimetric method shall govern.

[d] Average value shall be determined on the last consecutive five samples from a source.

[e] The value of any one sample shall be the result of a test or average of tests on any one sample.

[f] Maximum average and maximum single sample fineness limits do not apply if the sum of $C_3S + 4.75C_3A$ is less than or equal to 90.

[g] The strength at any specified test age shall be not less than that attained at any previous specified test age.

[h] When the optional heat of hydration in Table 4 is specified..

[i] The purchaser should specify the type of setting-time test required. In case he does not so specify, the requirements of the Vicat test only shall govern.

[j] The time of setting is that described as initial setting time in T 131.

Table 4—Optional Physical Requirements[a]

Cement Type	Applicable Test Method	I	IA	II	IIA	III	IIIA	IV	V
False set, final penetration, min, percent	T 186	50	50	50	50	50	50	50	50
Heat of hydration:	ASTM C 186								
7 days, max, kJ/kg (cal/g)		—	—	290 (70)[b]	290 (70)[b]	—	—	250 (60)[c]	—
28 days, max, kJ/kg (cal/g)		—	—	—	—	—	—	290 (70)[c]	—
Strength, not less than the values shown:									
Compressive strength, MPa (psi), 28 days	T106M/106	28.0 (4060)	22.0 (3190)	28.0 (4060) 22.0[b] (3190)[b]	22.0 (3190) 18.0[b] (2610)[b]	—	—	—	—
Sulfate resistance, 14 days max, percent expansion[d]	ASTM C 452	—	—	—	—	—	—	—	0.040

[a] These optional requirements apply only if specifically requested. Availability should be verified. See Note 2 in Section 4.

[b] The limit for the sum of the tricalcium silicate and 4.75 times the tricalcium aluminate in Table 1 shall not apply when this optional limit is requested. These strength requirements apply when the optional heat of hydration requirement is requested.

[c] When the heat of hydration limit is specified, it shall be used instead of the limits of C_3S, C_2S and C_3A listed in Table 1.

[d] When the sulfate resistance is specified, it shall be used instead of the limits of C_3A and $C_4AF + 2(C_3A)$ listed in Table 1.

[e] Cement meeting the high sulfate resistance limit for Type V are deemed to meet the moderate sulfate resistance required of Type II.

8. SAMPLING

8.1. When the purchaser desires that the cement be sampled and tested to verify compliance with this specification, sampling and testing should be performed in accordance with T 127.

8.2. Method T 127 is not designed for manufacturing quality control and is not required for manufacturer's certification.

9. TEST METHODS

Determine the applicable properties enumerated in this specification in accordance with the following methods:

9.1.1. *Air Content of Mortar*—T 137;

9.1.2. *Chemical Analysis*—T 105;

9.1.3. *Strength*—T 106M/T 106;

9.1.4. *False Set*—T 186;

9.1.5. *Fineness by Air Permeability*—T 153;

9.1.6. *Fineness by Turbidimeter*—T 98;

9.1.7. *Heat of Hydration*—ASTM C 186;

9.1.8. *Autoclave Expansion*—T 107;

9.1.9. *Time of Setting by Gillmore Needles*—T 154;

9.1.10. *Time of Setting by Vicat Needles*—T 131;

9.1.11. *Sulfate Expansion*—ASTM C 452;

9.1.12. *Calcium Sulfate (Expansion of) Mortar*—ASTM C 1038; and

9.1.13. *Optimum SO_3*—ASTM C 563.

10. INSPECTION

10.1. Inspection of the material shall be made as agreed upon between the purchaser and the seller as part of the purchase contract.

11. REJECTION

11.1. The cement may be rejected if it fails to meet any of the requirements of this specification.

11.2. At the option of the purchaser, retest, before using, cement remaining in bulk storage for more than six months or cement in bags in local storage in the custody of a vendor for more than three months after completion of tests and reject the cement if it fails to conform to any of the requirements of this specification. Cement so rejected shall be the responsibility of the owner of record at the time of resampling for retest.

11.3. Packages shall identify the mass contained as net weight. At the option of the purchaser, packages more than two percent below the mass marked thereon shall be rejected. If the average mass of packages in any shipment, as shown by determining the mass of 50 packages selected at random, is less than that marked on the packages, the entire shipment shall be rejected.

12. MANUFACTURER'S STATEMENT

12.1. At the request of the purchaser, the manufacturer shall state in writing the nature, amount, and identity of any air-entraining addition, and of any processing addition that may have been used, and also, if requested, shall supply test data showing compliance of such air-entraining addition with the provisions of ASTM C 226, and of any such processing addition with ASTM C 465.

12.2. When limestone is used, the manufacturer shall state in writing the amount thereof and, if requested by the purchaser, shall supply comparative test data on chemical and physical properties of the cement with and without the limestone (See Note 5). The comparative tests do not supersede the normal testing to confirm that the cement meets chemical and physical requirements of this standard. The amount of limestone in cement shall be determined in accordance with Annex A2.

Note 5—Comparative test data may be from qualification tests performed by the manufacturer during formulation of the cement with limestone.

13. PACKAGING AND PACKAGE MARKING

13.1. When the cement is delivered in packages, the words "portland cement," the type of cement, the name and brand of the manufacturer, and the mass of the cement contained therein shall be plainly marked on each package. When the cement is an air-entraining type, the words "air-entraining" shall be plainly marked on each package. Similar information shall be provided in the shipping documents accompanying the shipment of packaged or bulk cement. All packages shall be in good condition at the time of inspection.

Note 6—With the change to SI units, it is desirable to establish a standard SI package for portland cements. To that end, 42 kg (92.6 lb) provides a convenient, even-numbered mass reasonably similar to the traditional 94-lb (42.6 kg) package.

14. STORAGE

14.1. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weather-tight building that will protect the cement from dampness and minimize warehouse set.

15. MANUFACTURER'S CERTIFICATION

15.1. Upon request of the purchaser in the contract or order, a manufacturer's report shall be furnished at the time of shipment stating the results of tests made on samples of the material taken during production or transfer and certifying that the cement conforms to applicable requirements of this specification.

Note 7—Guidance on preparing the manufacturer's report is provided in Appendix X.1.

16. KEYWORDS

16.1. Hydraulic cement; portland cement; specification.

ANNEX

(Mandatory Information)

A1. CALCULATION OF POTENTIAL CEMENT PHASE COMPOSITION

A1.1. All values calculated as described in this annex shall be rounded according to R 11. When evaluating conformance to a specification, round values to the same number of places as the corresponding table entry before making comparisons. The expressing of chemical limitations by means of calculated assumed phases does not necessarily mean that the oxides are actually or entirely present as such phases.

A1.2. When expressing phases, C=CaO, S=SiO_2, A=Al_2O_3, F=Fe_2O_3. For example, C_3A=$3CaO{\cdot}Al_2O_3$. Titanium dioxide and phosphorus pentoxide (TiO_2 and P_2O_5) shall not be included with the Al_2O_3 content. See Note A1.

Note A1—When comparing oxide analyses and calculated phases from different sources or from different historic times, be aware that they may not have been reported on exactly the same basis. Chemical data obtained by Reference and Alternate Test Methods of T 105 (wet chemistry) may include titania and phosphorous as alumina unless proper correction has been made (see T 105), while data obtained by rapid instrumental methods usually do not. This can result in small differences in the calculated phases. Such differences are usually within the precision of the analytical methods, even when the methods are properly qualified under the requirements of T 105.

A1.3. When the ratio of percentages of aluminum oxide to ferric oxide is 0.64 or more, the percentages of tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite shall be calculated from the chemical analysis as follows:

Tricalcium silicate (C_3S) = $(4.071 \times \%\ CaO) - (7.600 \times \%\ SiO_2) - (6.718 \times \%\ Al_2O_3) - (1.430 \times \%\ Fe_2O_3) - (2.852 \times \%\ SO_3) - (5.188 \times \%CO_2)$ (*A1.1*)

Dicalcium silicate (C_2S) = $(2.867 \times \%\ SiO_2) - (0.7544 \times \%\ C_3S)$ (*A1.2*)

Tricalcium aluminate (C_3A) = $(2.650 \times \%\ Al_2O_3) - (1.692 \times \%\ Fe_2O_3)$ (*A1.3*)

Tetracalcium aluminoferrite (C_4AF) = $3.043 \times \%\ Fe_2O_3$ (*A1.4*)

A1.3.1. Unless limestone is used in the cement, the carbon dioxide content shall be considered to be equal to zero when calculating potential tricalcium silicate. In the absence of information on the limestone content of the cement sample, results shall note that no correction has been made for possible use of limestone. Cements containing limestone shall not be rejected on the basis of potential phase composition unless the values have been corrected for limestone content of the cement.

A1.4. When the alumina-ferric oxide ratio is less than 0.64, a calcium aluminoferrite solid solution (expressed as ss($C_4AF + C_2F$)) is formed. No tricalcium aluminate will be present in cements of this composition. Dicalcium silicate shall be calculated as in Equation A1.2. Contents of this solid solution and of tricalcium silicate shall be calculated by the following formulas:

ss($C_4AF + C_2F$) = $(2.100 \times \%\ Al_2O_3) + (1.702 \times \%\ Fe_2O_3)$ (*A1.5*)

Tricalcium silicate (C_3S) = $(4.071 \times \%\ CaO) - (7.600 \times \%\ SiO_2) - (4.479 \times \%\ Al_2O_3) - (2.859 \times \%\ Fe_2O_3) - (2.852 \times \%\ SO_3) - (5.188 \times \%CO_2)$ (*A1.6*)

A1.4.1. Unless limestone is used in the cement, the carbon dioxide content shall be considered to be equal to zero when calculating potential tricalcium silicate. In the absence of information on the limestone content of the cement sample, results shall note that no correction has been made for possible use of limestone. Cements containing limestone shall not be rejected on the basis of potential phase composition unless the values have been corrected for limestone content of the cement.

A1.5. If processing additions are included in the cement, the amounts of CaO, SiO_2, Al_2O_3, Fe_2O_3, SO_3 and CO_2 must be adjusted using the following equation before Equations A1.1 to 1.6 are applied.

$$\%\ \text{oxide in cement} = \frac{\%\ \text{oxide in sample} - (\%\ \text{oxide in PA} \times \%\ \text{PA in sample}/100) \times 100}{100 - \%\ \text{PA in sample}} \quad (A1.7)$$

A1.5.1. An example of the calculation is shown in the following table

	Sample	PA	Calculation	Base Cement
Percentage PA in sample		3.7		
Analyte	%	%		%
SiO_2	19.94	32.44	=(19.94-(32.44*3.7/100))/(100-3.7)*100	19.46
Al_2O_3	6.11	17.41	=(6.11-(17.41*3.7/100))/(100-3.7)*100	5.68
Fe_2O_3	2.27	6.08	=(2.27-(6.08*3.7/100))/(100-3.7)*100	2.12
CaO	61.48	26.8	=(61.48-(26.8*3.7/100))/(100-3.7)*100	62.82
SO_3	4.35	2.68	=(4.35-(2.68*3.7/100))/(100-3.7)*100	4.41
CO_2	0.00	0.00	=(0.00-(0.00*3.7/100))/(100-3.7)*100	0.00
C_3S				54
C_2S				15
C_3A				11
C_4AF				6

A2. LIMESTONE CONTENT OF PORTLAND CEMENT

A2.1. When limestone is used, the limestone content in portland cement shall be derived from the determination of CO_2 in the finished cement. Analysis of CO_2 shall be based on methods described in T 105. The percent limestone in the cement is calculated from the CO_2 analysis based on the CO_2 content of the limestone used. The limestone content of the cement is calculated as follows:

$$\frac{\%CO2 \text{ in the cement}}{\%CO2 \text{ in the limestone}} \times 100 = \% \text{ limestone in cement} \qquad (A2.1)$$

Note A2—For example, where the determined CO_2 content in the finished cement equals 1.5 percent and the CO_2 content of the limestone equals 43 percent ($CaCO_3$ in limestone equals 98 percent), then:

$$\frac{1.5}{43} \times 100 = 3.5\ \% \text{ limestone content in cement}$$

A2.2. This specification requires that the limestone to be used must contain a minimum of 70 percent $CaCO_3$. The manufacturer shall include the $CaCO_3$ content of the limestone on the manufacturer's report. Calculate the $CaCO_3$ content of the limestone as follows: % $CaCO_3$ = 2.274 × % CO_2.

Note A3—For verification of limestone content of cement, the purchaser must analyze for CO_2 content and make a correction for the content of $CaCO_3$ in the limestone in order for the data to be comparable to the manufacturer's report.

A2.3. Portland cements that do not contain limestone can contain baseline levels of CO_2 inherent in manufacture, for example, due to carbonation. This baseline CO_2 content is included as part of any calculated limestone content.

APPENDIX

(Nonmandatory Information)

X1. MANUFACTURER'S CERTIFICATION (MILL TEST REPORT)

X1.1. To provide uniformity for reporting the results of tests performed on cements under this specification, as required by Section 15 of M 85, Manufacturer's Certification, an example Mill Test Report is shown in Figure X1.1.

X1.2. The identity information given should unambiguously identify the cement production represented by the Mill Test Report and may vary, depending on the manufacturer's designation and purchaser's requirements.

X1.3. The Manufacturer's Certification statement may vary, depending on the manufacturer's procurement order, or legal requirements, but should certify that the cement shipped is represented by the certificate and that the cement conforms to applicable requirements of the specification at the time it was tested (or retested) or shipped.

X1.4. The sample Mill Test Report has been developed to reflect the chemical and physical requirements of this specification and recommends reporting all analyses and tests normally performed on cements meeting M 85. Purchaser reporting requirements should govern if different from normal reporting by the manufacturer or from those recommended here.

X1.5. Cements may be shipped prior to later-age test data being available. In such cases, the test value may be left blank. Alternatively, the manufacturer can generally provide estimates based on historical production data. The report should indicate if such estimates are provided.

X1.6. In reporting limits from the tables in M 85 on the Mill Test Report, only those limits specifically applicable should be listed. In some cases, M 85 table limits are superseded by other provisions.

ABC Portland Cement Company
Qualitytown, NJ

Plant Example Cement Type II Date March 9, 2002

Production Period March 2, 2002–March 8, 2002

STANDARD REQUIREMENTS

M 85, Tables 1 and 3

CHEMICAL

Item	Spec. Limit	Test Result
SiO_2 (%)	20.0 min	20.6
Al_2O_3 (%)	6.0 max	4.4
Fe_2O_3 (%)	6.0 max	3.3
CaO (%)	[a]	62.9
MgO (%)	6.0 max	2.2
SO_3 (%)	3.0 max	2.7
Loss on ignition (%)	3.0 max	2.7
Na_2O (%)	[a]	0.19
K_2O (%)	[a]	0.50
Insoluble residue (%)	0.75 max	0.27
CO_2 (%)	[a]	1.5
Limestone (%)	5.0 max	3.5
$CaCO_3$ in limestone (%)	70 min	98
Potential compounds (%)		
C_3S	[a]	50
C_2S	[a]	21
C_3A	8 max	6
C_4AF	[a]	10
$C_4AF + 2(C_3A)$	[a]	22
C_3S+4.75 C_3A, (%)	100 max	78.5

PHYSICAL

Item	Spec. Limit	Test Result
Air content of Mortar (volume %)	12 max	8
Fineness (m^2/kg) (Air permeability)	260 min 430 max	377
Average[b] fineness	280 min 420 max	385
Autoclave expansion (%)	0.80 max	0.04
Compressive strength (MPa)	Min:	
1 day	[a]	
3 days	7.0	23.4
7 days	12.0	29.8
28 days	[a]	
Time of setting (minutes) (Vicat)		
Initial	Not less than 45 Not more than 375	124
Heat of hydration (kJ/kg)		
7 days	[c]	300

[a] Not applicable.
[b] Average of last five consecutive samples.
[c] Test result represents most recent value and is provided for information only.

OPTIONAL REQUIREMENTS

M 85, Tables 2 and 4

CHEMICAL

Item	Spec. Limit	Test Result
Equivalent alkalis (%)	[d]	0.52

[d] Limit not specified by purchaser. Test result provided for information only.

PHYSICAL

Item	Spec. Limit	Test Result
False set (%)	50 min	82
Compressive strength (MPa)		
28 days	28.0 min	39.7

Signature: ______________________ Title: ______________________

Figure X1.1—Example Mill Test Report.

Abbreviations and acronyms used without definitions in TRB publications:

AAAE	American Association of Airport Executives
AASHO	American Association of State Highway Officials
AASHTO	American Association of State Highway and Transportation Officials
ACI–NA	Airports Council International–North America
ACRP	Airport Cooperative Research Program
ADA	Americans with Disabilities Act
APTA	American Public Transportation Association
ASCE	American Society of Civil Engineers
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATA	Air Transport Association
ATA	American Trucking Associations
CTAA	Community Transportation Association of America
CTBSSP	Commercial Truck and Bus Safety Synthesis Program
DHS	Department of Homeland Security
DOE	Department of Energy
EPA	Environmental Protection Agency
FAA	Federal Aviation Administration
FHWA	Federal Highway Administration
FMCSA	Federal Motor Carrier Safety Administration
FRA	Federal Railroad Administration
FTA	Federal Transit Administration
IEEE	Institute of Electrical and Electronics Engineers
ISTEA	Intermodal Surface Transportation Efficiency Act of 1991
ITE	Institute of Transportation Engineers
NASA	National Aeronautics and Space Administration
NASAO	National Association of State Aviation Officials
NCFRP	National Cooperative Freight Research Program
NCHRP	National Cooperative Highway Research Program
NHTSA	National Highway Traffic Safety Administration
NTSB	National Transportation Safety Board
SAE	Society of Automotive Engineers
SAFETEA-LU	Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (2005)
TCRP	Transit Cooperative Research Program
TEA-21	Transportation Equity Act for the 21st Century (1998)
TRB	Transportation Research Board
TSA	Transportation Security Administration
U.S.DOT	United States Department of Transportation