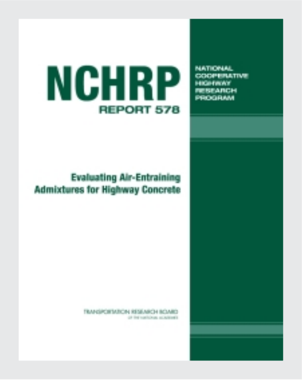

Evaluating Air-Entraining Admixtures for Highway Concrete

DETAILS

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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

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Evaluating Air-Entraining Admixtures for Highway Concrete

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TRANSPORTATION RESEARCH BOARD

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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

Systematic, well-designed research provides the most effective approach to the solution of many problems facing highway administrators and engineers. Often, highway problems are of local interest and can best be studied by highway departments individually or in cooperation with their state universities and others. However, the accelerating growth of highway transportation develops increasingly complex problems of wide interest to highway authorities. These problems are best studied through a coordinated program of cooperative research.

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The needs for highway research are many, and the National Cooperative Highway Research Program can make significant contributions to the solution of highway transportation problems of mutual concern to many responsible groups. The program, however, is intended to complement rather than to substitute for or duplicate other highway research programs.

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FOREWORD

By Amir N. Hanna
Staff Officer
Transportation Research Board

This report presents a recommended procedure for evaluating air-entraining admixtures used in highway concrete. The procedure involves the testing of non-air-entrained concrete and concrete containing the air-entraining admixture under simulated field conditions. Criteria are proposed for acceptance of admixtures for use in either highway pavements or structures. The recommended procedure and acceptance criteria will guide materials engineers in evaluating and selecting air-entraining admixtures that should contribute to appropriate freeze-thaw durability and thus to good performance and long service life. The content of the report will be of immediate interest to materials engineers, researchers, and others concerned with the design of concrete mixtures for use in highway pavements and structures.

Extensive laboratory testing and long-term field experience have demonstrated conclusively that concrete must be properly air entrained if it is to resist the action of freezing and thawing. For more than 50 years, neutralized Vinsol resin has been used effectively for air entrainment. However, other air-entraining admixtures have been introduced in recent years and their use has increased primarily because of the higher cost and limited supply of Vinsol resin. Current test methods, such as AASHTO T 157, *Air-Entraining Admixtures for Concrete,* set limits on the effects that any given air-entraining admixture may exert on certain properties of the fresh and hardened concrete mixture in comparison with a similar concrete mixture containing a standard-reference air-entraining admixture such as neutralized Vinsol resin. However, experience with bridge decks, pavements, and other highway structures that were constructed with concrete mixtures incorporating newer air-entraining admixtures indicated that current test methods do not accurately assess the impact of these newer admixtures on concrete properties and durability. Thus, research was needed to address the issues associated with the use of these air-entraining admixtures in highway concrete and to develop recommendations to help improve specifications for air-entraining admixtures.

Under NCHRP Project 18-10, "Procedures for Evaluating Air-Entraining Admixtures for Highway Concrete," Construction Technology Laboratories, Inc., of Skokie, Illinois, worked with the objective of developing procedures for evaluating and qualifying air-entraining admixtures for hydraulic cement concrete for highway applications. To accomplish this objective, the researchers conducted an extensive test program in which they examined the properties of concrete mixtures containing a variety of air-entraining admixtures and related these properties to freezing and thawing durability. Based on analysis of test results, the researchers developed a procedure for evaluating air-entraining admixtures that involves the testing of non-air-entrained concrete and concrete containing the air-entraining admixture under simulated field conditions. Admixture evaluation is made by comparing the results from these tests against proposed evaluation criteria. The procedure includes two protocols: one for evaluating admixtures intended for use in highway pavements and the other for evaluating admixtures intended for use in highway structures. Finally, validity of the recommended procedure was confirmed by laboratory tests conducted on concrete mixtures containing five air-entraining admixtures with known performance records.

The recommended test method and proposed acceptance criteria will provide a good indicator of the expected concrete properties and resistance to freezing and thawing and can, therefore, be used to evaluate air-entraining admixtures intended for use in highway pavements and structures. The test method will be particularly useful to highway agencies and is recommended for consideration and adoption by AASHTO as a standard test method.

Appendixes A through C contained in the research agency's final report are not published herein. These appendixes are accessible on the web as *NCHRP Web-Only Document 101* at http://www4.trb.org/trb/onlinepubs.nsf. These appendixes are titled as follows:

Appendix A: Literature Review and Survey;
Appendix B: Foam Drainage and Infrared Test Results; and
Appendix C: Detailed Test Results

CONTENTS

SUMMARY

Evaluating Air-Entraining Admixtures for Highway Concrete

The use of air entrainment has been an accepted practice in concrete technology for more than 60 years. Air is intentionally entrained in the concrete mixture to reduce the potential for damage from freezing and thawing. Until the early 1980s most air-entraining admixtures were based solely on the salts of wood resin (i.e., neutralized Vinsol resin). In recent years, a new generation of air-entraining admixtures was introduced and has been used to entrain air in concrete in many bridges and pavements. Air-entraining admixtures proposed for use in highway structures must conform to the requirements of AASHTO M 154 or ASTM C 260 test methods. The specifications were developed in the late 1970s to assure users that new admixtures would not have significant adverse effects on concrete properties and to verify their effectiveness in reducing freeze-thaw damage. These methods compare the properties of concrete prepared with the tested admixtures in a controlled laboratory environment with concrete prepared with standard Vinsol resin admixture. Field performance of some AASHTO-certified admixtures has been questionable; many highway structures where these admixtures were used have exhibited poor performance or unacceptable properties.

In this project, the current procedures available for evaluating air-entraining admixtures were reviewed and modified, and new procedures were developed to evaluate air-entraining admixtures. The main concept of the new procedures is to test the admixture under simulated field conditions and to compare the results with those of non-air-entrained concrete. Acceptance criteria were proposed for the two major highway applications (i.e., pavement and structural concrete) such that an admixture can be qualified for either or both applications.

As part of the research program, a statistically designed program was developed to identify significant factors that influence concrete air-entrainment and select those variables that should be included in the test procedures. It was recognized that all concrete materials, production procedures, construction practices, and field conditions influence to a degree the concrete air void system. Based on a survey of field performance of air-entraining admixtures and review of the available literature, several potential key factors were chosen for evaluation in this project. Cement alkali level, air-entraining admixture type, mixing time, aggregate shape, water-cement ratio, and concrete temperature were key factors selected because of their reported effect on the air-void system and strength. Based on preliminary laboratory screening involving infrared analysis and foam drainage tests of 42 air-entraining admixtures, 6 commercially available air-entraining admixtures, each having different chemical compositions and expected to vary in performance, were selected for the evaluation.

For each of the selected 6 admixtures, 16 different concrete mixes were prepared (96 mixes total). Fresh concrete properties were determined for each mix. Compressive, flexural strengths were determined at 28 days. In addition, beams prepared from each mix were used to measure the air-void parameters, including the spacing factor, specific surface, and total air content.

Findings of this study indicated that the type of air-entraining admixtures has a statistically significant effect on spacing factor. In addition, mixing duration, cement alkali level, and coarse aggregate shape were found to have a significant effect on the spacing factor. The spacing factor decreases as the mixing duration increases and increases when a rounded aggregate instead of a crushed aggregate is used.

The statistical analysis of the data obtained in the laboratory testing program indicated that compressive strength decreases with increasing temperature, water-cement ratio, and cement alkali, and by using round instead of crushed aggregate. The highest compressive strength (average of six air-entraining admixtures) was obtained for the mixes made with crushed coarse aggregate, having a low water-cement ratio, using low alkali cement, and mixed at standard laboratory temperatures.

The proposed evaluation procedures were validated by laboratory tests on mixtures containing five air-entraining admixtures, having different properties and performance records. Although these five admixtures were certified in accordance with AASHTO and ASTM standards, none of them conformed to all of the performance requirements of the proposed four testing protocols, indicating that changes in testing conditions (compared with standard laboratory conditions) better replicate the field performance of air-entrained concrete mixtures.

CHAPTER 1

Introduction

Project Background

Extensive laboratory testing and long-term field performance have demonstrated that concrete that is properly air-entrained can better resist the action of freezing and thawing. For more than 50 years, neutralized Vinsol resin has been used effectively for air entrainment. More recently, other air-entraining admixtures have been introduced and their use has increased primarily because of the higher cost and limited supply of Vinsol resin. AASHTO T 157, *Air-Entraining Admixtures for Concrete,* sets limits on the effects that any given air-entraining admixture under test may exert on certain properties of the fresh and hardened concrete mixture in comparison with a similar concrete mixture containing a standard reference air-entraining admixture such as neutralized Vinsol resin. However, many concrete mixtures incorporating these newer admixtures meeting AASHTO T 157 limits have exhibited unacceptable properties when used in bridge decks, pavements, and other highway structures. There has been considerable debate recently about the effects of these admixtures on concrete properties and durability.

Although a great deal of research has been performed to address various aspects of concrete resistance to freezing and thawing, there are no clear conclusions concerning the effect of newer air-entraining admixtures on concrete properties and durability. Further research was needed to address the issues associated with the use of these air-entraining admixtures in highway concrete and to develop recommendations to help improve specifications for air-entraining admixtures.

Objective and Scope of the Research

The objective of this research was to develop procedures for evaluating and qualifying air-entraining admixtures for use in hydraulic cement concrete for highway applications. To accomplish this objective, the following tasks were performed:

1. Information relative to the use of air-entraining admixtures in hydraulic cement concrete used in highway applications was collected and reviewed. This information was obtained from domestic and foreign literature, contacts with public and private agencies and industry organizations, and other sources. A summary of current use, field performance, test methods, test data, and other items pertaining to the use of air-entraining admixtures by state and other highway agencies was compiled.
2. Test procedures currently used in the United States and other countries for evaluating the effectiveness of air-entraining admixtures were identified based on the information gathered in Task 1.
3. A detailed experimental research plan, which encompasses laboratory tests, was developed for evaluating the relative importance of the various factors affecting air entrainment identified in Task 1, modifying the test procedures proposed in Task 2, and validating the modified procedures.
4. The plan developed in Task 3 was executed. The plan included testing of fresh and hardened concrete properties. Also, a plan for putting the results of this research into practice was suggested.
5. A set of test procedures for evaluating air-entraining admixtures was developed based on the results of the entire research effort. Protocols for these test procedures were prepared in an AASHTO format.

Research Approach

The research performed in this project included reviewing the current procedures and a survey of field performance of air-entraining admixtures (summarized in Chapter 2 and included in Appendix A), conducting laboratory tests to investigate factors affecting air-entrainment in concrete, and developing a procedure for evaluating air-entraining admixtures.

Factors Affecting Air-Entrainment

A statistically designed experimental program was developed to identify significant variables that influence concrete air-entrainment. Air-entraining admixture type, cement alkali, aggregate shape, mixing time, water-cement ratio, and mixing temperature were investigated. These factors were selected based on the literature review and survey of field performance of air-entraining admixtures. Forty-one commercially available air-entraining admixtures were analyzed and tested. Their physical properties and their compositions were identified and they were then subjected to preliminary (foam drainage) tests. Out of the 41, 6 admixtures were selected for further laboratory testing. The six chosen admixtures had distinctive compositions with significantly different foam drainage data and were expected to exhibit significantly diverse performance when used in concrete.

Statistical Analysis of Selected Factors

An analysis of variance and stepwise least-squares linear regression analysis were used to determine whether differences between fixed factors (experiment variables) and two-way interactions of fixed factors significantly affect the spacing factor. The spacing factor was chosen as an evaluation parameter because it is the most important air-void parameter affecting the freeze-thaw resistance of concrete. The effects of a change in significant variables on the air-void spacing factor were determined; the factors that cause increase in spacing factors were considered in developing the evaluation procedures.

The results of the stepwise least-squares linear regression were used to identify statistically significant main factors and two-way interactions affecting strength. Factors that lead to strength reduction were also considered in the conditions of the procedures for evaluating air-entraining admixtures.

Development and Validation of New Procedures

The procedures currently available for evaluating air-entraining admixtures were reviewed and modified. Modified procedures that involve tests under simulated field conditions for comparing properties of air-entrained concrete with those of non-air-entrained concrete were proposed. Acceptance criteria were proposed for major highway concrete applications (pavement and structures); the admixture can be evaluated for use in either or both applications.

The validation of the proposed procedures revealed that admixtures are sensitive to testing conditions. An admixture that satisfies the strength requirements when tested under laboratory conditions may not meet the same requirement when tested under simulated field conditions.

A plan was developed for validation of the proposed procedures through a field performance study. This plan is discussed in Chapter 4 of this report.

CHAPTER 2

Background and Literature Search

Introduction

In addition to air-entraining agents, all materials used in concrete (i.e., portland cement, supplementary cementing materials, water, chemical admixtures, and sand and aggregate) influence, to varying degrees, the air-void system (Whiting and Nagi, 1998). Production procedures, construction practices, and field conditions also affect the air-void system of concrete. The effects of these factors on the air-void system have been studied extensively. Recent research has focused on specific issues such as the compatibility of chemical admixtures with air-entraining admixtures and supplementary cementing materials.

Mechanism of Air Entrainment

Air bubbles are not formed by air-entraining agents (AEA), but stabilized by them. As the air-entraining agent molecules are inserted between adjacent water molecules at the water surface, the mutual attraction between the separated water molecules is reduced. Lowering the surface tension stabilizes the bubbles against mechanical deformation and rupture, making it easier for bubbles to be formed. Without the presence of an air-entraining agent, the smaller bubbles, which have higher internal pressure, coalesce to form larger bubbles that have a greater tendency to escape to the surface and burst.

Absorbed AEA molecules at the surface of the bubble form a film, with their polar heads in the water phase. If the molecule is charged, the bubble acquires this charge. The electrostatic repulsion keeps bubbles separated and prevents coalescence (Dodson, 1990).

The ends of the AEA molecules that protrude into the water are also attracted to cement grains. This allows for a coating of calcium salts (i.e., products of cement hydration) to form around each air bubble, making it more stable than bubbles formed in plain water.

Effects of Air Entrainment on Properties of Concrete

Fresh Concrete

The adherence of the entrained air bubbles to the cement particles reduces inter-particle friction between cement and aggregate grains. An increase in air content by 1/2 to 1 percentage point can increase the slump by about 1 inch (Whiting and Nagi, 1998), allowing a reduction in water needed to achieve the same slump. On the other hand, the attraction between bubbles and cement particles imparts a cohesion or "stickiness" to the concrete that makes it more difficult to place, consolidate, and finish, particularly at high air contents, and the compressibility of air can sometimes lead to problems in pumping.

Hardened Concrete

An increase in air content leads to reductions in compressive strength, elastic modulus, and flexural strength. For example, increase in air content by a percentage point leads to average reductions of 2 to 6 percent in compressive strength, 3 to 6 percent in elastic modulus (in compression), and 2 to 4 percent in flexural strength (Whiting and Nagi, 1998).

Compressive strength reductions of 20 percent or more have been reported for concrete having normal air contents compared with design strength. In some cases, when Vinsol resin was replaced by non-Vinsol admixtures, the 28-day compressive strengths decreased for comparable air contents; a failure mode of mostly shear at the interface between aggregate and paste with very few fractured aggregate particles was noticed. Microscopic examination indicated accumulations of air voids around the aggregate particles that reduced the bond strength between the aggregate and surrounding mortar.

The South Dakota DOT conducted a comprehensive investigation to define the causes of compressive strength reduction observed during the construction season of 1997 (Cross et al., 2000). The investigation concluded that incompatibility between low-alkali cement and synthetic admixtures caused the coalescence of air voids around the aggregate particles and that high summertime temperatures aggravated the problem. The thinner walled air bubbles formed by these admixtures apparently caused more flocculation than would be observed with Vinsol resin admixtures.

Factors Affecting Air Entrainment

Cement Alkali Level

For a given dosage of AEA, the air content increases as the alkali content of the cement is increased because alkalis allow more air-entraining agent to remain in solution during mixing, thereby maintaining lower surface tension longer and leading to greater volume of air entrained. Some studies have concluded the air bubble systems formed in a high-alkali environment are not stable while others suggest that they are stable, especially in the presence of chemical admixtures (Pigeon, 1992; Greening, 1967; Pistili, 1983).

Supplementary Cementitious Material

Use of supplementary cementitious materials can affect air entrainment. Carbon found in fly ash can attract and absorb surfactants used in air-entraining agents (Klieger and Perenchio, 1976; Ramachandran, 1995). Some fly ashes with a high loss on ignition (LOI) do not necessarily contain significant amounts of reactive carbon, possibly because of carbon phases that become encapsulated in glass spheres during cooling and are prevented from adsorbing the surfactants or when a portion of the LOI results from carbonate (Detwiler et al., 1996).

Ground granulated blast furnace slag (GGBFS) is normally used at high dosages, generally up to 50 percent by mass of cementitious materials. Because GGBFS is usually finer than cement, up to 100 percent more AEA may be needed when finely ground GGBFS is used at higher dosages.

Silica fume is normally used at dosages between 5 and 10 percent by mass of cement (3 to 6 percent in ternary mixes) and does not have a significant influence on the production and stability of the air-void system, but, because of its fineness, greater amounts of AEA are needed (Whiting et al., 1993).

Chemical Admixtures

Certain combinations of water reducers and air-entraining admixtures can be incompatible in such a way as to lead to unacceptable air-void systems. Plante et al. (1989) indicated that increasing the dosage of water reducers influences the air-void system. Doubling the recommended dosage of any of three water reducers adversely influenced the stability of the air-void system, but no consistent relationship between the admixture dosage and the resulting air content was found.

Accelerating admixtures have a minor effect on the air-void system. Stott et al. (1994) found that the air content and spacing factor of mixes containing both calcium chloride and non-chloride accelerating admixtures did not differ from those of the control mixes.

Attiogbe et al. (1992) indicated that properly air-entrained concrete containing superplasticizer could have adequate frost resistance, even if the spacing factors were relatively high. Concrete mixtures with spacing factors exceeding 0.008 in. (0.20 mm)—the maximum values recommended by the American Concrete Institute (ACI) Durability Committee—were found to have acceptable frost resistance.

Aggregates

Coarse aggregate surface texture and maximum size can influence the air content. Crushed stone aggregate will entrain less air than gravel aggregate (Dodson, 1990). Because the mortar volume generally decreases as the aggregate size increases and the entrained air is contained within the mortar fraction, the air content of concrete generally decreases. Brenfeld and Okundi (1999) showed that saturation of the aggregates influences the air-void system in concrete. When partially saturated aggregate was used in concrete, air bubbles, typically 100 microns in diameter, were formed at the surface of coarse aggregates leading to noticeable strength reduction. Air, trapped under pressure inside aggregate particles during water absorption, migrated out between concrete placing and setting to produce the voids.

Sand contributes to air entrainment by trapping air bubbles in the void spaces between sand grains. Sand in the middle-size fractions of No. 30 to 100 sieve is the most effective in entraining air, while fine sand (less than 100 mesh) has negligible effect because the effective screen size approaches the size of the largest bubbles.

Mixing Time

For a given mixer, the mixing time plays an important role in controlling the air content. An adequate air-void system can be achieved in as little as 60 seconds of mixing (Barbee, 1961). Variations in air content are much higher for a concrete produced at extremely short mixing times.

Retempering

The amount of added water and increase in air content will depend on the slump, air content, and concrete temperature prior to retempering. Nagi and Whiting (1994) evaluated the effect of retempering on the air-void system and showed that retempering at normal temperature did not influence the air-void parameters (e.g., spacing factor).

Concrete Temperature

Higher temperatures necessitate higher dosages of air-entraining admixtures in order to maintain a given air content (Whiting and Nagi, 1998). Powers (1965) indicated that a rise in temperature would lead to relatively rapid hydration of cement with less available water leading to fewer bubbles formed. Saucier et al. (1991) found that, although changes in temperature lead to changes in the total air content, the spacing factor of the hardened concrete is not significantly affected. At high placement temperatures, some new air-entraining admixtures can cause the air bubbles to accumulate at the surfaces of aggregate particles, thereby weakening the concrete (Cross et al., 2000).

Consolidation

Consolidation through vibration reduces the friction between the aggregate particles to remove pockets of entrapped air and make the concrete more flowable (Whiting and Nagi, 1998). The loss of air content because of vibration depends on the concrete slump, initial air content, vibration frequency, and time of vibration. Studies have shown that for a given vibration frequency and vibration time, the loss of air increases with increased slump (Higginson, 1952).

An increase in vibration frequency also has a significant effect on the air-void system. The spacing factor for concrete subjected to 20 seconds of vibration significantly increases as frequency is increased from 11,000 to 14,000 vpm (Stark, 1986). However, the spacing factor remains relatively unchanged for frequencies of 8000 vpm or less, even though the air content is decreased.

In some concrete pavement construction, scaling caused by cycles of freezing and thawing was observed in paths following the vibrator heads on the paving machine; it was attributed to excessive vibration (Stark, 1986). The Iowa DOT has observed an increase in premature deterioration in concrete pavements related to over-vibration. Tymkowicz and Steffes (1999) reported that cores taken from longitudinal cracks in Interstate 80 in Iowa contained 3 percent air in the top half and 6 percent in the bottom half; they attributed this condition to excessive vibration. They stated that excessive vibration can occur at lower paver speeds, even though the frequency was low (8000 vpm). Stutzman (1999) conducted a similar study on Iowa pavements and determined that the worst air-void systems of deteriorated pavements occurred in the visible vibrator trails.

Pumping

Pumping affects air entrainment because of free fall and pumping pressure. When handling concrete, the material is often dropped, with an accompanying loss in air content. Several researchers have been able to trace part or all of the observed loss of air associated with pumping to the impact of concrete as it falls freely down an unfilled pipeline, hitting pipe elbows and eventually splattering at the point of placement. This low frequency but variable intensity pulsing of the concrete is normally associated with the loss of coarser air bubbles and what can be a significant loss of total air content. Because large voids make only a small contribution to freeze-thaw protection, such losses are often inconsequential in terms of the in-place durability of the concrete (Hover, 1989; Pleau et al., 1995; Lessard et al., 1996; Hover and Phares, 1996).

Pumping can also alter the air bubble system by compressing and subsequently de-compressing the air bubbles. At sufficiently high pressure, the pump can compress the bubbles to the point of dissolution of the air into the mix water. As postulated by Meilenz et al. (1958) and summarized by Fagerlund (1990), smaller bubbles and lower surface tension will favor air dissolution. When the concrete is depressurized, the air comes out of solution at a rate determined by the rate of depressurization. Slow, reversible depressurization allows bubbles to reform into approximately their initial state. Rapid, uncontrolled depressurization allows the air to come out of solution explosively, escaping from the mixture or perhaps forming into large bubbles, immediately susceptible to removal on impact. Several researchers have demonstrated the use of various attachments and hose configurations to control depressurization to reduce air loss (Yingling et al., 1992; Macha et al., 1994; Pleau et al., 1995; Lessard et al., 1996; Hover and Phares, 1996).

Surface Finishing

Air loss caused by finishing has been seen at the surface and near-surface of the concrete. Use of a vibratory screed, for example, is merely surface-applied vibration, which would result in the types of effects already discussed in the consolidation section. Most vibrating screeds have a lower frequency than insertion vibrators, however, so normally only the coarser voids would be affected. Later finishing operations are applied at higher frequency and are more concentrated at the surface as the concrete hardens. The bursting of air bubbles can be observed at the trailing edge of finishing tools as they are passed over fresh concrete, and there is clearly a surface impact as the blades of a float- or toweling-machine strike the concrete surface. It is also possible that machine finishing results in a localized frictional heating of the surface that may contribute to air loss. Thus, finishing operations could lead to not only the loss of air, but also to the incorporation of bleed water into a thin surface layer, resulting in substantial localized increase in water content, water-to-cement ratio, porosity, and permeability, and a localized decrease in strength.

Identification and Evaluation of Test Procedures

The most common and widely used procedures for evaluating the effectiveness of air-entraining admixtures in the United States are AASHTO Specifications M 154 and T 157. The requirements of AASHTO M 154 can be divided into two types, uniformity requirements and performance requirements:

- Uniformity. These requirements have been established to assure the purchaser that the manufacturer is implementing adequate quality control of production of the admixtures. These tests include pH measurement to check the relative acidity of the solution, air content of mortar prepared with the tested admixtures (ASTM C 185), and residue after oven drying. The acceptable range of pH should differ by no more than 2 points. The air content should not vary by more than ±2 percentage points between successive lots, and residue should not vary by more than ±12 percent of midpoint of limits supplied by the manufacturer.
- Performance. The performance requirement calls for preparing concrete mixes using the tested air-entraining admixtures and comparing its mechanical properties with those of a reference concrete prepared using neutralized Vinsol resin, ASTM Type I cement, and well-graded aggregate under controlled laboratory conditions. The compressive and flexural strengths at ages of 3, 7, and 28 days and 6 and 12 months should not be less than 90 percent of that of the reference concrete. The maximum shrinkage should not exceed 120 percent of the reference concrete and the durability factor (freeze-thaw test, AASHTO T 161) should not be less than 80 percent.

The current uniformity tests are adequate to check the quality control of production of the admixtures. However, because of the increased number of air-entraining admixtures on the market and the lack of information on the source, type, or basic ingredients of such admixtures, chemical analysis can be a helpful quality control test. Infrared analysis, for example, can be used to identify chemical compounds in the admixtures. Chemists consider the data of such analyses as a "fingerprint" of an admixture. Infrared or other chemical analyses that identify harmful ingredients may be added to the efficacy tests.

With respect to performance requirements, the concept of comparing the tested air-entraining admixtures with Vinsol resin, which has good performance records, is a valid procedure. These tests have been widely used in the industry in the United States since their introduction to AASHTO and ASTM in 1978. It appears, however, that although most of the newly introduced admixtures have compared favorably in these tests with Vinsol resin, some of these admixtures have shown poor field performance such as low strength or poor durability. Testing these admixtures according to current protocols and under ideal laboratory conditions may not identify performance problems that are reported in the field, suggesting that field conditions and construction practices contribute to the problem, and that these conditions have not been taken into account in the test method. The coalescence of air-voids around aggregate particles, for instance, has been correlated with mixing time, hot weather, and cement composition (low alkali) and conditions of aggregates, but would not normally be evident in standard acceptance testing of the admixture. Although AASHTO T 157 allows the option of using the same materials that will be used in the field, such materials are still brought to the laboratory and tested under standardized conditions. For example, if the maximum size of coarse aggregate is greater than 1 in., the freshly mixed concrete is screened over a 1-in. sieve prior to fabricating the tested specimens.

Another shortcoming of standard admixture acceptance testing is the absence of an evaluation of the air-void system in hardened concrete (ASTM C 457). The results of the 8-to-12-week-long freeze-thaw test (AASHTO T 161) provide good indication of durability of the hardened concrete. However, microscopical analysis and determining the air-void parameters provide an insight to admixture performance and opportunity to avoid performance problems associated with air-void coalescence and clustering.

Most European countries, including Germany and Great Britain, follow the European Standard EN 934 "Admixtures for Concrete, Mortar and Grout." The Standard covers air-entraining admixtures and other chemical admixtures. Chemical admixtures are tested in a manner similar to ASTM C 494 procedures "Standard Specification for Chemical Admixtures for Concrete," with the exception of acceptance testing for air-entraining admixtures. The standard is based on comparing concrete made with the tested air-entraining admixture with similar non-air-entrained concrete. The specifications require that the air content of the reference concrete should be 2 percent or less and the air content of tested concrete should be between 4 and 6 percent. For acceptance, compressive strength of the air-entrained concrete should be 75 percent or more of that of reference concrete at the age of 28 days, and the spacing factor should be less than 0.20 mm (0.008 in.). As with the ASTM and AASHTO standards, concrete should be prepared under ideal laboratory conditions using well-graded aggregates and standard cements.

Selection and Procurement of Air-Entraining Admixtures

The purpose of this task was to select six air-entraining admixtures that exhibit different performance under given conditions to be used in the laboratory evaluation. The selection was based primarily on the chemical composition of the admixture. Admixtures were selected to represent the basic categories commonly used in the concrete industry. The foam drainage test was used to further narrow the selection process. In order to choose two admixtures from a class of admixtures, which have similar chemical-based materials (e.g., Vinsol), the foam drainage test results were used to decide which admixture should be included in the program. The criterion was to choose admixtures that have significantly diverse foam drainage test results.

Identification and Procurement of Admixtures

A list of air-entraining admixtures available in the United States was prepared. The list, presented in Appendix B, contains the manufacturer's name and address, recommended dosages, reported physical properties, and states approving the admixtures. Of these, 41 air-entraining admixtures were procured for testing in this research.

Testing of Admixtures

In addition to infrared analysis and foam drainage testing of all admixtures, physical properties of the selected admixtures were determined

Physical Properties

The pH of the sample solution, specific gravity, and solid content were measured. The pH was measured in accordance with ASTM E 70, *Standard Test Method for pH Aqueous Solution with the Glass Electrode*. The specific gravity was measured using equipment specified in ASTM E 100-03, *Standard Specifications for ASTM Hydrometers.* The solid content was measured by oven drying. Test results are shown with the infrared charts in Appendix B.

Infrared Analyses

Infrared spectroscopy was used primarily to identify organic compounds by interpreting an infrared spectrum. The Fourier Transform Infrared Spectrometer (FTIR) was used to measure the characteristic absorption of infrared light as a function of wavelength. Based on the infrared analyses, the 41 admixtures were categorized in five groups as follows:

- Alpha olefin sulfonate (6 admixtures);
- Benzene sulfonate (4 admixtures);
- Resin/rosin and fatty acids (13 admixtures);
- Vinsol resin (14 admixtures); and
- Combination (surfactants, urea, tall oil, and others) (4 admixtures).

The infrared charts shown in Appendix B (available as part of *NCHRP Web-Only Document 101*) list the admixture properties (i.e., pH, density, and solid contents) and identification of admixtures is based on the infrared spectrum (the text within the charts is information provided by manufacturers).

Foam Drainage Test

The foam drainage test was conducted to provide a preliminary indicator of the stability of the air-void system. A stable air-void system usually has a lower drainage rate and retains more water than an unstable system (Gutmann, 1980; Cross et al., 2000). The rate of drainage depends on the hydrodynamic characteristics of the foam, which include the size and shape of bubbles and the mobility of the liquid-air interface. The test procedures are based on the work conducted by South Dakota DOT (Cross et al., 2000).

In this test, 300 mL of water is mixed with 10 mL of air-entraining admixture in a blender. The resulting foam is poured into a 1000 mL graduated cylinder. The position of the foam/water line is recorded as a function of time. The amount of water drained as a function of time is fitted to kinetic equations and the value of V_0 is calculated as follows:

$$V_d = V_0 - 1/kt$$

Where V_d is the amount of liquid drained from the foam at time t. V_0 and 1/k are determined by plotting the amount of drained water versus 1/t. The slope of the line is $-1/k$ and V_0 is the intercept. The percent of water drained is also calculated using the following equation:

$$\%\ \text{Drained} = 100 \times (310 - V_0)/310$$

The value of $-1/k$ gives an indication of the rate of drainage. The higher the value of $-1/k$, the lower the rate of drainage. Admixtures, which have significant differences in values of $-1/k$ and the percentage of water drained from the foam (% Drained) are expected to have different air-void systems and different performance.

The foam drainage test of all admixtures was also performed in the presence of cement. In this test, 5 grams of ASTM Type I cement was premixed with the admixture and the water.

Table 1. Foam drainage data for water only tests (selected admixtures).

Admixture	Foam Drainage Statistics				Foam Height			
	V_0	-1/k	r^2	% drained	Initial	15 min	30 min	60 min
E	300.2	321.0	0.922	96.8	590	400	330	320
D	301.3	13.1	0.740	97.2	370	320	315	310
S	300.6	0.2	0.048	97.0	310.0	310.0	310.0	310.0
M	287.0	409.8	0.926	92.6	800.0	720.0	540.0	460.0
C	301.8	770.9	0.942	97.3	910.0	870.0	810.0	760.0
B	284.4	299.6	0.901	91.7	720.0	680.0	660.0	660.0

Results for both tests (with and without cement) are shown in Tables B-1 through B-10 (Appendix B, which is available as part of *NCHRP Web-Only Document 101*). Data were tabulated based on admixture classification. Results include the value of V_0, $-1/k$, coefficient of determination (correlation coefficient squared), and the calculated percentage of water drained. In addition the foam heights at 0, 15, 30, and 60 minutes are also presented.

Selection of Admixtures for Laboratory Test

Test results of the 41 admixtures were examined, and 6 of the tested admixtures (designated E, D, S, M, C, and B) were selected: two from the Vinsol group (E and D), two from the resin/rosin group (S and M), one from the benzene group (B) and one from the alpha olefin sulfonate group (C). The selected admixtures are approved by most State DOTs and are also commonly used in the concrete industry. Also, each of the selected admixtures is produced by a different company, which gives some assurance that different sources of raw materials were used in manufacturing the selected admixtures. Results of the foam drainage tests for the selected admixtures are listed in Tables 1 and 2.

The purpose of the selection procedure was to identify admixtures that exhibit largely different performance characteristics when tested with water only or with cement and water during the foam drainage evaluation. As shown in the test results, the admixtures with extreme differences in one test condition (e.g., test with water) may show closer values when tested with both water and cement. Large differences in drainage rates and total drainage percentage were taken into account, but changes in performance upon the addition of cement were also an important consideration.

From the Vinsol group, Admixtures E and D had significantly different drainage characteristics in both the water and cement with water drainage tests. When the admixtures were added to water alone, Admixture D had the lowest $-1/k$ value of 13.1 (fastest drainage rate among the Vinsol resins). Although Admixture E had an ultimately higher drainage than Admixture D by the end of the test with a $-1/k$ value of 321 it was the slowest among the Vinsol group. The test showed that Admixture E had an ultimately lower drainage at the end of the test than Admixture D. Admixture E had an inconsistent drainage rate and showed a “step function” whereas Admixture D showed a very fluid and consistent drainage.

From the resin/rosin group, Admixture S and Admixture M were selected. As with the Vinsol group, the presence of cement significantly affected the drainage characteristics of both admixtures. When the admixtures were tested with water, Admixture S had the fastest drainage among the resin/rosin group with a $-1/k$ value of 0.2, while Admixture M had the slowest drainage among the group with a $-1/k$ value of 410; the addition of cement greatly affected the performance of both admixtures.

Admixture S had a much slower drainage in the presence of cement and the resultant foam holds the most water of all the admixtures. Admixture M had the largest drop in drainage rate among all the admixtures, the $-1/k$ value dropped from 410 to 17.3 with the addition of cement. Less water was held in the foam and the water drained faster from the foam than when cement was not present.

Table 2. Foam drainage data for water plus cement tests (selected admixtures).

Admixture	Foam Drainage Statistics				Foam Height			
	V_0	-1/k	r^2	% drained	Initial	15 min	30 min	60 min
E	173.0	45.3	0.916	55.8	630	630	630	630
D	239.5	158.4	0.880	77.3	580	375	375	375
S	191.8	27.2	0.859	61.9	580.0	580.0	440.0	565.0
M	291.5	17.3	0.923	94.0	400.0	390.0	390.0	390.0
C	276.4	338.2	0.835	89.2	720.0	630.0	570.0	540.0
B	297.1	89.5	0.906	95.9	520.0	425.0	350.0	330.0

Admixture C was selected from the alpha olefin sulfonate admixtures and Admixture B was selected from the benzene sulfonate admixtures. Because fewer admixtures were classified under these two groups, only one admixture was selected from each. Admixture C has the most significant decrease in drainage rate (−1/k) within its group when cement was added. Admixture B also has the most significant decrease in −1/k value within its class when cement was added.

Experimental Program

Partial factorial design was selected to identify the significance of factors and combinations of interactions that influence air entrainment of concrete. The following variables were included in the research program:

- **Air-entraining admixture type**: Six types of air-entraining admixtures were used in the evaluation.
- **Cement alkali**: Two levels of alkali were used in the study, low alkali (0.45 percent as equivalent Na_2O) and high (1 percent as equivalent Na_2O). The average low-alkali cement produced in the United States is 0.45 percent; cements with alkali content as high as 1 percent are produced by some cement companies in the United States.
- **Aggregate shape/angularity**: Rounded and crushed aggregates were used in the study. The shape of aggregate particles factor is included in the program because of its influence on air-void system and influence on strength development, especially in cases where air void clustering around aggregate particles has occurred. The crushed aggregate was dolomitic carbonate from Illinois and the rounded aggregate was a siliceous (granitic) glacial gravel from Wisconsin.
- **Mixing time**: Short and long mixing times (2 minutes and 15 minutes, respectively) were evaluated. The short mixing time was selected to simulate the short mixing times used for concrete paving and the long mixing time was selected to simulate truck-mixed concrete and agitation. The average mixing time for concrete pavement is 90 seconds. Also, the minimum mixing time for central mixed concrete (AASHTO M 157) ranges from 1 to 3 minutes. AASHTO M 157 allows 70 to 100 revolutions for truck-mixed concrete at 6 to 18 rpm, which correspond to an average of 8 minutes of mixing time. In addition, most specifications allow for agitation of concrete at lower speed (2 to 6 rpm). A drum-type mixer was used in this program.
- **Water-to-cement ratio**: Low and high water-to-cement ratios (w/c) were evaluated. The different water-cement ratios were used to evaluate the influence of water content and workability on air entrainment. A w/c ratio of 0.39 was used without water-reducing admixture to achieve a slump of approximately 1 in., and w/c of 0.44 was used without water-reducing admixture to achieve a slump of 3 to 4 in. These w/c ratios represent the range of w/c ratios commonly used in highway constructions.
- **Mixing temperature**: Two temperatures (73° F and 90° F) were selected for the study. The higher concrete temperature represents the average maximum concrete temperature allowed by most State agencies. The lower temperature was chosen to provide data for comparison.

For each air-entraining admixture, the following variables were varied in the laboratory study:

1. Cement alkali (CA): —two levels (0.45 percent, 1 percent);
2. Aggregate shape/angularity (AS): —two levels (round, crushed);
3. Mixing time (MTI): —two levels (2 minutes, 15 minutes);
4. Water-to-cement ratio (w/c): —two levels (0.39, 0.44); and
5. Mixing temperature (MT): —two levels (73° F, 90° F).

For each of the six air-entraining admixtures, a full factorial design of the 5 variables would require a total of 32 (2^5) mixes. A half-fraction factorial design was used to reduce the number of mixes for each admixture to 16, as shown in Table 3 (shaded cells indicate the variable combinations tested); details of the 16 mixtures are shown in Table 4. The orthogonality of the experimental design as illustrated in Table 3 ensures that all main effects and two-factor interactions are additive and independent of one another.

For the six air-entraining admixtures evaluated, a total of 96 mixes were prepared. A typical mix containing 600 pounds of ASTM Type I/II cement per cubic yard was used. The fine aggregate was natural sand. The coarse aggregate size was #467 (nominal size 1-1/2 in. to No. 4) in accordance with AASHTO M 43. The fine aggregate constituted 40 percent of the total aggregate. The total aggregate content was adjusted based on the specific gravity of the aggregate. The air-entraining admixture dosage was adjusted to achieve a 6 ± 1 percent air content. For all mixes, a drum-type mixer was used. The sequence of charging of materials into the mixer was the same for all mixes. Aggregate and sand were first added followed by 40 percent of the mixing water and mixed for 15 seconds. The mixer was stopped for 10 minutes. The mixer was then started and air-entraining admixture was added, followed by cement and the remaining water. Mixing times were started after adding the cement and water.

All specimens were consolidated by external vibration using a table vibrator with a frequency of 7,000 vibrations per minute (117 Hz) and amplitude of 0.1 mm (0.004 in.). All concrete specimens were cured in a moist room maintained at 73° ± 3° F until the time of testing.

For each mix, the following properties of concrete were determined.

- Slump (AASHTO T 119);
- Unit weight (AASHTO T 121);

Table 3. Mix matrix for each air-entraining admixture.

				Mixing Temperature							
				73°F				90°F			
				Mixing Time				Mixing Time			
				Short		Long		Short		Long	
				Aggregate Shape/Angularity		Aggregate Shape/Angularity		Aggregate Shape/Angularity		Aggregate Shape/Angularity	
				Round	Crushed	Round	Crushed	Round	Crushed	Round	Crushed
Cement Alkalinity	High Alkali	Water-Cement Ratio	High		Mix 1	Mix 2		Mix 3			Mix 4
			Low	Mix 5			Mix 6		Mix 7	Mix 8	
	Low Alkali	Water-Cement Ratio	High	Mix 9			Mix 10		Mix 11	Mix 12	
			Low		Mix 13	Mix 14		Mix 15			Mix 16

Table 4. Mix variables for each concrete mixtures.

Mix No.	Mix Duration	Temperature	Aggregate Shape	Water-Cement Ratio	Cement Alkalinity
1	Short	73°F	Crushed	High	High
2	Long	73°F	Rounded	High	High
3	Short	90°F	Rounded	High	High
4	Long	90°F	Crushed	High	High
5	Short	73°F	Rounded	Low	High
6	Long	73°F	Crushed	Low	High
7	Short	90°F	Crushed	Low	High
8	Long	90°F	Rounded	Low	High
9	Short	73°F	Rounded	High	Low
10	Long	73°F	Crushed	High	Low
11	Short	90°F	Crushed	High	Low
12	Long	90°F	Rounded	High	Low
13	Short	73°F	Crushed	Low	Low
14	Long	73°F	Rounded	Low	Low
15	Short	90°F	Rounded	Low	Low
16	Long	90°F	Crushed	Low	Low

- Air Content (Pressure Method, AASHTO T 152);
- Air Content (Gravimetric Method, AASHTO T 121);
- Compressive Strength at 28 days (AASHTO T 22, three 6 × 12-in. cylinders);
- Flexural Strength at 28 days (AASHTO T 97, three 6 × 6 × 22-in. beams); and
- Air-Void Analysis (ASTM C 457, 6 × 6 × 22-in. beam sample).

The collected data were used to evaluate the statistical significance of each variable and each two-variable interaction affecting the air-void system. Factors that have a statistical influence on the total air content, spacing factor, and specific surface were identified. If a reduction in strength was observed, the air-void sample was examined to see whether the strength reduction was associated with excessive air voids clustered around the aggregate particles.

CHAPTER 3

Analysis of Test Results

Concrete Properties

For each air-entraining admixture, the dosages required to achieve an air content of 6±1 percent varied significantly for each variable. The mixes with crushed stone and low w/c ratio required the highest dosages, sometimes five times that used for high w/c ratio. Fresh concrete properties for all mixes are listed in Table 5.

Compressive Strength

For each mix, three 6-x-12 in. cylinders were prepared and tested for compressive strength at 28 days in accordance with AASHTO T 22. The average compressive strengths for each of the 96 mixes are shown in Figure 1. The figure presents the compressive strength of the 16 variables for each admixture. The compressive strength ranged from 3370 to 5900 psi with an average of 4350 psi; standard deviation was 541 psi. Detailed test results are shown in Appendix C.

Flexural Strength

For each mix, three 6-x-6-x-22-in. beams were tested for flexural strength at 28 days in accordance with AASHTO T 97. The average flexural strengths for the 96 mixes are shown in Figure 2. The flexural strength ranged from 560 to 930 psi, with an average of 720 psi; standard deviation was 64 psi. Variation in flexural strength was fairly consistent with compressive strength data. Figure 3 shows the average compressive and flexural strength for the different mixes. Detailed test results are shown in Appendix C.

Air-Void Analysis

Air-void analyses were conducted on beams made from the 96 mixes. Spacing factors, specific surface, and total air content were determined. Test results are presented in Table 5. Spacing factors varied between 0.002 and 0.007 in., and specific surface ranged from 800 to 1400 in.^{2}/in.^{3} The difference between fresh and hardened air content was within 2 percentage points for all mixes.

Strength Reduction Evaluation

Mixes which had a relatively low strength compared with the average strength of each mix were evaluated to determine whether the strength reduction was associated with excessive air voids clustered around the aggregate particles. Mixes C2 (Mix 2 with Admixture C) and M2 (Mix 2 with Admixture M), for example had compressive strengths of 3390 and 3750 psi, respectively; flexural strength of both mixes was 650 psi. Stereomicroscope images of both samples did not show excessive air bubbles around the aggregate particles (Figures 4 and 5). The air contents in the hardened concrete were 6.6 and 7.7 percent for Mix C2 and Mix M2, respectively. It seems that the relatively high air content contributed to the strength reduction.

Statistical Analysis

As indicated previously, the effects of the following independent variables on concrete air-void parameters and strength were considered in the laboratory study using a half fraction factorial design. The levels of cement alkali (CA), aggregate shape/angularity (AS), mixing time (MTI), water-to-cement ratio (w/c), and mixing temperature (MT) were included in the laboratory study.

Statistically significant single independent variables and two-factor interactions for individual admixtures were identified for the spacing factor and concrete strength. Significant effects and two-way interactions common to types of admixtures were used to develop or modify test procedures used to evaluate admixtures.

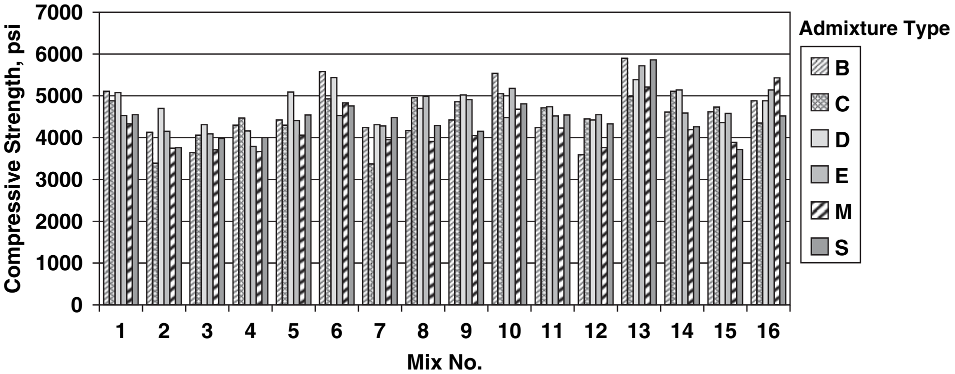


Figure 1. 28-day compressive strength.

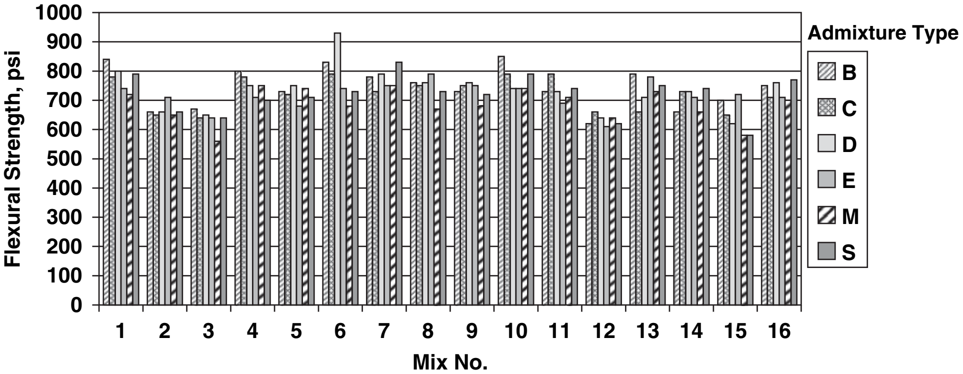


Figure 2. 28-day flexural strength test results.

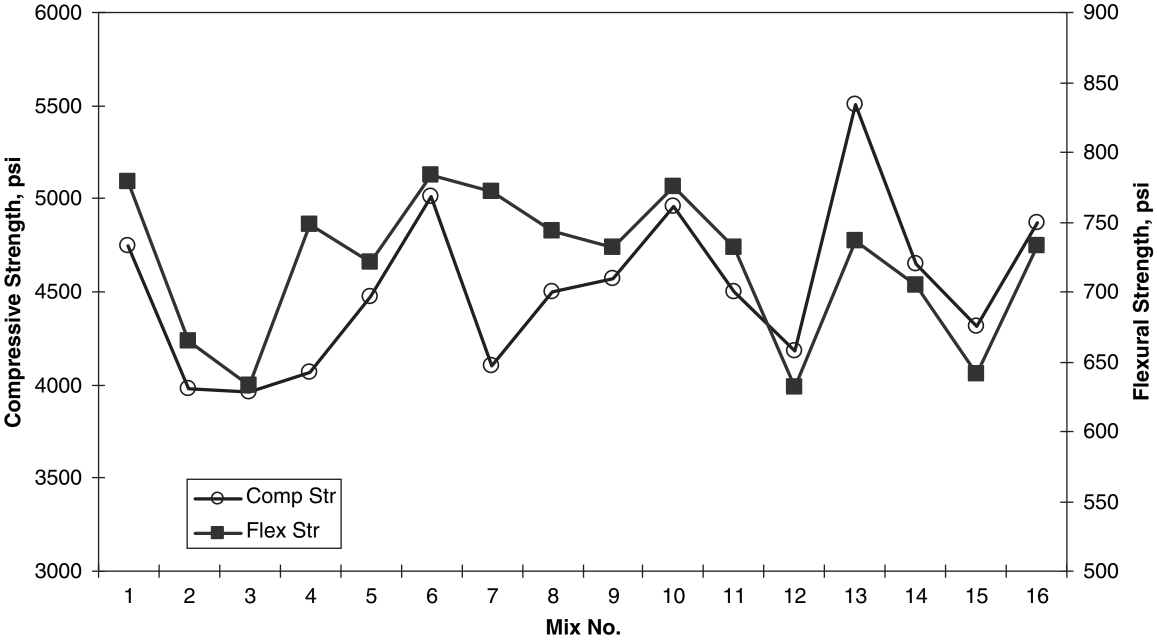


Figure 3. Average compressive and flexural strengths results.

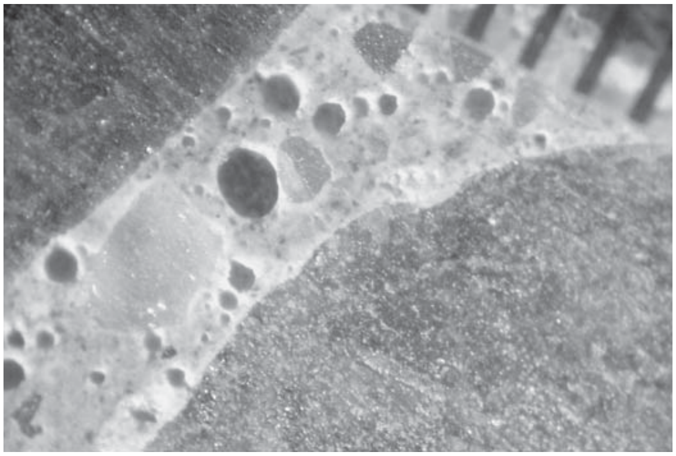


Image 1

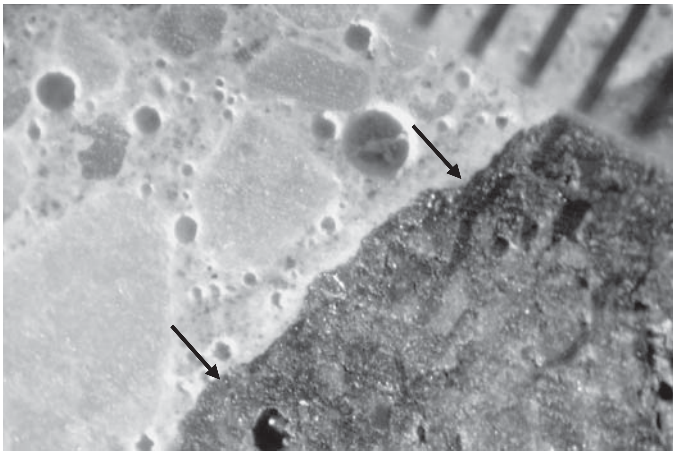


Image 2

Figure 4. Stereomicroscope images of the lapped cross-section of Sample C-2. Note the paste-aggregate interface (indicated by arrows) is relatively tight (no significant voids or separation gaps are observed around aggregate particles).

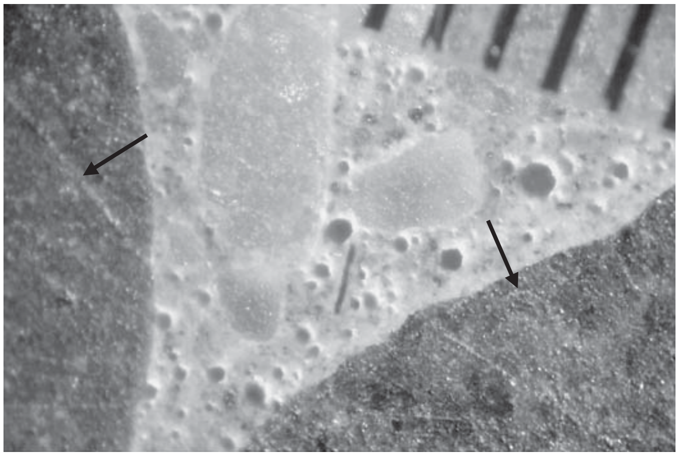


Image 1

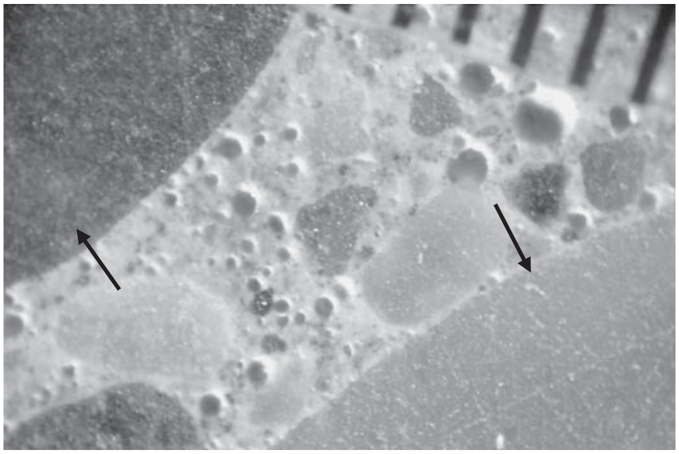


Image 2

Figure 5. Stereomicroscope images of the lapped cross-section of Sample M-2. Note the paste-aggregate interface (indicated by arrows) is relatively tight (no significant voids or separation gaps are observed around aggregate particles).

Factors Affecting Spacing Factor

An analysis of variance (ANOVA) was conducted for the hardened air content spacing factor. Spacing factor was chosen because it is the most important air-void parameter affecting freeze-thaw resistance of concrete. The analysis was used to determine whether differences between fixed factors (design of experiment variables) and two-way interactions of fixed factors significantly affect the spacing factor. Based on the results of the analysis, fixed factors and two-way interactions were rationally included or excluded from the linear regression analyses.

The first analysis was conducted on the dataset of all 96 concrete mixes (6 admixtures and 16 mixes per admixture). A 10 percent significance level (SL) was selected as the criterion for separation between significant and residual effects. Effects with a significance level of less than or equal to 10 percent were assumed to be significant. Conversely, effects with a significance level greater than 10 percent were assumed to be attributable to residual variation. An identification variable (random variable) to account for the six different air-entraining admixtures was included in the analysis for the 96 mixes. The analysis showed that the air-entraining admixture variable (ADMIX_ID) was significant (i.e., there are different significant variables among the different air-entraining admixtures). Separate ANOVA were next conducted for each admixture (16 mixes per admixture) to assess the relevance of the source of the air-entraining admixtures.

Results of the analysis are listed in Table 6. Significant main effects for the combined data set include mixing duration (DUR), coarse aggregate shape (SHAPE), and cement alkali level (ALK). The concrete temperature (TEMP) and water-cement ratio (WC) were not significant in any of the

Table 5. Fresh concrete properties and air-void parameters.

Mixture Designation	Ambient Temp, °F	Concrete Temp, °F	Slump, in.	Air, %	Unit Weight, lb/ft^3	Total Air, %	Specific Surface, (in^2/in.^{3})	Spacing Factor, in.
B1	73	75	3.50	6.8	144.5	8.1	663	0.005
B2	72	75	5.75	6.0	145.3	4.7	893	0.006
B3	90	92	4.20	6.0	144.7	4.9	1196	0.004
B4	90	88	3.00	6.3	145.7	6	930	0.004
B5	73	77	2.25	6.4	145.7	5.9	683	0.006
B6	75	78	0.75	6.2	147.3	5.3	1108	0.004
B7	90	87	0.75	6.0	147.3	3.4	1397	0.004
B8	90	91	2.00	6.1	145.5	6.5	1037	0.004
B9	73	75	6.00	6.0	144.1	6	718	0.006
B10	73	75	3.25	5.7	146.1	5.6	1288	0.004
B11	90	88	1.75	6.0	146	5.5	1306	0.003
B12	90	92	3.75	6.4	141.9	4.7	1316	0.004
B13	74	78	0.50	5.1	148.5	6.8	882	0.005
B14	73	76	1.50	6.9	145.1	7.2	1157	0.003
B15	90	92	1.00	5.5	147.1	5.6	905	0.005
B16	90	92	1.00	6.8	146.5	4.9	1514	0.003
C1	72	76	2.75	6.8	144.9	5.5	846	0.005
C2	73	75	5.25	6.8	143.7	6.6	638	0.007
C3	90	90	6.50	6.5	143.1	6.9	728	0.006
C4	90	88	2.50	6.1	146.5	5.7	1092	0.004
C5	73	76	2.25	7.0	144.9	9.3	716	0.004
C6	75	78	1.00	6.0	144.9	7.5	1310	0.003
C7	90	88	1.00	7.1	145.3	6.8	1437	0.003
C8	90	92	2.00	5.2	147.5	6.2	588	0.007
C9	72	75	4.75	6.2	144.5	5.1	734	0.006
C10	73	75	3.50	6.4	145.7	6	1051	0.004
C11	90	88	2.00	6.1	145.7	4.9	1022	0.005
C12	90	89	3.50	5.5	145.3	3.3	1420	0.004
C13	74	77	1.00	6.9	145.3	5.5	1108	0.004
C14	73	77	1.50	5.9	146.9	5.4	1080	0.004
C15	90	92	1.25	5.3	146.7	3.8	816	0.007
C16	91	89	1.00	6.7	145.7	4.8	1227	0.005
D1	73	76	3.00	6.4	144.9	7.8	700	0.005
D2	73	76	5.25	5.4	145.3	4.8	758	0.006
D3	90	91	5.50	6.5	143.9	6	660	0.007
D4	90	89	2.75	6.4	145.7	5.6	1057	0.005
D5	73	76	1.50	5.1	147.7	5.4	684	0.007
D6	75	78	1.00	6.0	147.3	4.3	865	0.006
D7	90	88	1.00	6.6	146.1	5.1	1181	0.004
D8	90	92	2.00	5.0	147.9	4.4	632	0.008
D9	73	75	5.50	5.9	144.5	6.7	652	0.005
D10	73	76	3.25	6.9	144.5	7	1082	0.004
D11	91	88	2.00	5.5	146.9	3.6	1131	0.005
D12	90	89	2.00	6.4	144.3	6.4	829	0.005
D13	74	77	0.75	5.2	148.9	5.1	812	0.006
D14	75	78	1.25	6.6	145.3	4.8	1128	0.005
D15	90	89	1.10	5.6	146.7	3.7	866	0.006
D16	91	90	1.00	6.9	145.7	4.9	2126	0.002
E1	72	75	3.75	7.0	144.9	6.3	781	0.004
E2	73	76	5.25	6.1	145.5	7.6	1004	0.003
E3	90	92	5.50	6.6	143.1	5.3	784	0.006
E4	91	89	2.00	6.0	146.5	4.1	1144	0.005
E5	73	76	2.00	6.8	144.9	5.9	767	0.006
E6	73	77	130	7.1	143.4	6.9	1103	0.004
E7	91	89	0.75	5.9	147.3	4.2	1445	0.004
E8	90	92	1.80	5.4	147.1	3.6	811	0.007
E9	73	76	5.25	6.8	144.5	6.4	690	0.006
E10	73	75	3.00	6.8	145.3	4.4	1382	0.004
E11	92	90	2.75	6.8	145.3	5.8	1074	0.004
E12	90	92	3.40	5.4	145.5	5.6	920	0.005
E13	73	76	1.25	7.3	145.3	4.1	737	0.007
E14	73	75	2.00	6.9	145.3	5	1492	0.003
E15	90	90	1.50	5.3	146.7	5.8	939	0.005
E16	91	86	1.00	6.1	146.9	5.6	1342	0.004

Table 5. (Continued).

Mixture Designation	Ambient Temp, ºF	Concrete Temp, ºF	Slump, in.	Air, %	Unit Weight, lb/ft^3	Total Air, %	Specific Surface, (in^2/in.^{3})	Spacing Factor, in.
M1	72	74	3.25	6.7	145.7	6.5	1111	0.004
M2	74	77	5.00	6.9	143.7	7.7	813	0.004
M3	90	92	3.80	6.2	143.1	7	823	0.005
M4	90	90	2.25	6.3	146.1	5.9	1541	0.003
M5	73	76	2.00	5.9	146.1	4.2	937	0.006
M6	73	77	1.00	7.0	145.7	5.8	1326	0.004
M7	90	90	0.75	6.3	147.3	4	1479	0.004
M8	90	92	1.60	7.2	144.7	4	1297	0.004
M9	73	76	5.00	6.2	144.5	6.6	1029	0.004
M10	72	76	3.00	6.7	145.3	5.2	1809	0.003
M11	90	88	2.00	6.2	145.7	3.9	1170	0.005
M12	90	90	4.20	6.4	143.9	5.4	1042	0.004
M13	75	78	0.75	5.1	148.5	5.3	1394	0.003
M14	73	76	1.50	6.8	145.3	7.5	1623	0.002
M15	90	89	1.25	6.0	145.9	3.8	1287	0.004
M16	90	92	0.50	6.8	147.3	4.1	1750	0.003
S1	72	75	3.25	6.4	145.7	5.1	1055	0.005
S2	73	75	5.50	7.1	143.3	7.2	686	0.006
S3	90	92	4.00	5.5	145.1	4.7	740	0.007
S4	90	88	2.00	6.0	146.1	5	1112	0.005
S5	73	76	1.75	5.5	148.5	4.5	882	0.006
S6	74	78	1.00	6.8	146.5	7.6	1300	0.003
S7	89	87	0.75	5.8	148.1	4.2	1357	0.004
S8	90	91	2.00	6.8	145.5	4.6	1106	0.005
S9	73	75	4.00	5.6	145.9	5.6	868	0.005
S10	73	76	2.75	6.4	145.3	3.6	1504	0.004
S11	91	88	1.75	6.3	146.5	5.3	1507	0.003
S12	90	90	4.40	6.7	143.1	6.6	1044	0.004
S13	72	75	1.00	5.0	149.7	4.6	1036	0.005
S14	73	75	1.50	6.0	146.5	4.2	1547	0.003
S15	90	92	1.30	5.0	146.7	5.5	662	0.007
S16	90	88	0.75	6.5	146.9	5	1175	0.004

Table 6. Analysis of variance for spacing factor.

Independent Variable	Significance Level, percent						
	Admix. B	Admix. C	Admix. D	Admix. E	Admix. M	Admix. S	Combined
ADMIX_ID	N/A	N/A	N/A	N/A	N/A	N/A	1
DUR	21	50	50	9	41	50	0
TEMP	16	30	71	21	80	84	91
SHAPE	21	11	21	13	63	43	0
WC	50	30	71	21	80	84	59
ALK	30	100	24	50	50	59	1
DURxTEMP	30	100	100	6	100	84	7
DURxSHAPE	50	100	71	21	80	59	24
DURxWC	21	30	10	50	100	59	34
DURxALK	30	16	30	13	100	84	1
TEMPxSHAPE	100	50	30	9	100	71	3
TEMPxWC	30	13	37	21	80	71	45
TEMPxALK	50	50	71	9	50	100	91
SHAPExWC	****	****	****	****	63	71	75
SHAPExALK	100	21	50	13	100	100	2
WCxALK	100	21	71	21	****	****	75

NOTES:

1. The variable ADMIX_ID is a dummy random variable that was used to identify if significant variables were different between admixtures (1 for Admixture B, 2 for Admixture C, etc.).
2. Significance levels identified by **** indicate that the SHAPExWC or WCxALK variables were eliminated from the individual admixtures analysis to minimize problems with a near-linear variance matrix among independent variables.

Table 7. Mean spacing factors for significant variables.

Independent Variable	Mean Spacing Factor
Admix. B	0.0044
Admix. C	0.0050
Admix. D	0.0055
Admix. E.	0.0048
Admix. M	0.0039
Admix. S	0.0046
Dur. 2 min.	0.0051
Dur. 15 min.	0.0043
Shape rounded	0.0052
Shape crushed	0.0042
Alkali 0.45	0.0045
Alkali 1.0	0.0049

individual or combined data set analyses. Significant two-way interaction effects include duration-temperature (DURxTEMP), duration-water-cement ratio (DURxWC), duration-alkali (DURxALK), temperature-shape (TEMPx-SHAPE), temperature-alkali (TEMPxALK), and shape-alkali (SHAPExALK).

The variable ADMIX_ID was significant for the combined data set analysis indicating that the significance of variables varied between the different admixtures. Significant variables identified between the different admixtures in Table 6 included DURxTEMP (significant for admixture E), DURxWC (significant for admixture D), TEMPxSHAPE (significant for admixture E), and TEMPxALK (significant for admixture E).

The mean spacing factors computed between significant main variables identified in the combined data set analysis are listed in Table 7. On average, the air-void spacing factor was relatively higher for Admixture D and relatively lower for Admixture M. The average spacing factor increased by approximately 19 percent when the mixing time was decreased from 15 to 2 minutes. The spacing factor increased approximately 24 percent when rounded aggregates were substituted with crushed aggregates. The average spacing factor increased by approximately 9 percent alkali level when the alkali level was increased from 0.45 to 1.0.

Based on the ANOVA analyses, the greatest increase in average spacing factor occurs when the mixing time is decreased from 15 to 2 minutes or the aggregate shape is changed from crushed to round.

A stepwise least-squares linear regression on the combined data set was conducted to predict air-void spacing factor from significant main effects and two-way interactions identified in the analysis. To reduce multicollinearity effects among independent variables identified in the analysis, independent variables were transformed by subtracting the mean value. Multicollinearity can lead to incorrect conclusions when using linear regression models. When independent variables are highly correlated (e.g., some of the independent variable can predict other independent variables) and taken as a group, they appear to be highly significant in predicting the dependent variable. If collinearity exists, when the group of independent variables is examined individually, are insignificant. Transformed independent variables are identified by the letter "D" at the end of the variable name (deviation from mean).

Significant variables affecting the air-void spacing factor identified in the stepwise linear regression include mixing time duration (DURD), coarse aggregate shape (SHAPED), cement alkali level (ALKD), duration-alkali (DURDxALKD), mix temperature-shape (TEMPDxSHAPED), and shape-alkali (SHAPEDxALKD). The dummy variable used to identify the different air entrainers was not significant. Results of the linear regression are summarized in Table 8. The linear regression identified six significant variables but resulted in a somewhat low coefficient of determination (R^2 of 0.361), indicating that the magnitude of air-void spacing factor cannot be adequately predicted from the six significant independent variables. The value of R^2 ranges between zero (no variability in spacing factor explained by the independent variables) to 1.0 (all variability in spacing factor explained by the independent variables). The analysis indicates that the air-void spacing factor is relatively stable for a range of mix design and mixing variables (e.g., no variable or combination of variables statistically influences the spacing factor).

The relative effects of a change in significant variables on the air-void spacing factors can be determined by examining the sign of the coefficients listed in Table 8. The air-void spacing decreases with increases in mixing time duration (negative coefficient for independent variable DURD), decreases as the aggregate shape is changed from rounded (SHAPE = 0) to

Table 8. Spacing factor analysis.

Independent Variable	Coefficient	Signif. Level, %
DURD	-5.609E-05	0.1
SHAPED	-1.021E-03	0.0
ALKD	1.023E-03	0.9
DURDxALKD	1.573E-04	0.9
TEMPDxSHAPED	-5.147E-05	4.0
SHAPEDxALKD	-1.742E-03	2.5
Constant	4.677E-03	0.0
R-squared (adj.) = 0.361		

NOTES:

1. DURD = mixing time duration minus mean (DURD = DUR minus 8.5)
2. SHAPED = coarse aggregate shape minus mean (SHAPED = SHAPE minus 0.5 where SHAPE=0 for rounded, 1 for crushed)
3. ALKD = cement alkali minus mean (ALKD = ALK minus 0.725)
4. DURDxALKD = DUR deviation times ALK deviation from mean
5. TEMPDxSHAPED = TEMP minus mean times SHAPED (TEMPD = TEMP minus 81.5)
6. SHAPEDxALKD = SHAPE deviation times ALK deviation from mean

crushed (negative coefficient for independent variable SHAPED), and increases with cement alkali level (positive coefficient for independent variable ALKD). As expected, this trend in spacing factor with changes in significant independent variables is the same as that listed in Table 7 for the ANOVA analysis. The air-void spacing factor increases when the combined mixing time duration and alkali level increases (positive coefficient for independent variable DURDxALKD), decreases when the combined concrete temperature and aggregate shape increases (from rounded to crushed), and decreases when the combined cement alkali and aggregate shape increases (from rounded to crushed).

Factors Affecting Strength

Statistically significant main factors and two-way interactions affecting strength were identified by conducting an analysis of variance (ANOVA) for the reported compressive strength and flexural strength data. The analysis was used to determine whether differences between fixed factors (design of experiment variables) and two-way interactions of fixed factors significantly affect compressive and flexural strength. An analysis was conducted on the dataset of all 96 concrete mixes (6 admixtures and 16 mixes per admixture) and included a dummy variable to identify if independent mix design variables were significant between different admixtures. As with the spacing factor analysis, a 10-percent significance level (SL) was selected as the criterion for separation between significant and residual effects. Results of the analysis are listed in Table 9.

Table 9. Analysis of variance for strength.

Independent Variable	Significance Level, percent	
	Compressive Strength	Flexural Strength
ADMIX_ID	0	0
DUR	>10	>10
TEMP	0	0
SHAPE	0	0
WC	0	3
ALK	0	1
DURxTEMP	1	7
DURxSHAPE	>10	>10
DURxWC	3	2
DURxALK	>10	>10
TEMPxSHAPE	1	>10
TEMPxWC	>10	2
TEMPxALK	>10	1
SHAPExWC	>10	1
SHAPExALK	5	>10
WCxALK	>10	0

NOTE:

The variable ADMIX_ID is a dummy random variable that was used to identify if significant variables were different between admixtures.

The analysis showed that the air-entraining admixture variable (ADMIX_ID) was significant (i.e., there are different significant variables among the different air-entraining admixtures) for both compressive and flexural strength (SL less than 10 percent). Significant main effects for the combined compressive strength data set include concrete temperature (TEMP), coarse aggregate shape (SHAPE), water-cement ratio (WC), and cement alkali level (ALK). The mixing duration (DUR) was not significant. Significant two-way interaction effects include duration-temperature (DURxTEMP), duration-water-cement ratio (DURxWC), temperature-shape (TEMPx-SHAPE), and shape-alkali (SHAPExALK).

Significant main effects for the combined flexural strength data set include concrete temperature (TEMP), coarse aggregate shape (SHAPE), water-cement ratio (WC), and cement alkali level (ALK). The mixing duration (DUR) was not significant. Significant two-way interaction effects include duration-temperature (DURxTEMP), duration-water-cement ratio (DURxWC), temperature-water-cement ratio (TEMPx-WC), temperature-alkali (TEMPxALK), coarse aggregate shape-water cement ratio (SHAPExWC), and water-cement ratio-alkali (WCxALK).

Significant variables common to both compressive and flexural strength include the air-entraining admixture variable (ADMIX_ID), concrete temperature (TEMP), coarse aggregate shape (SHAPE), water-cement ratio (WC), cement alkali level (ALK), duration-temperature (DURx-TEMP), and duration-water-cement ratio (DURxWC). Variables that were not significant for both compressive and flexural strength include mixing duration (DUR), duration-coarse aggregate shape (DURxSHAPE), and duration-alkali (DURxALK).

The mean compressive and flexural strengths computed between significant main variables identified in the combined data set analysis are listed in Table 10. On average, the compressive strength was relatively greater for Admixture D

Table 10. Mean strength for significant variables.

Independent Variable	Compressive Strength, psi	Flexural Strength, psi
Admix. B	4590	745
Admix. C	4540	725
Admix. D	4760	735
Admix. E	4620	715
Admix. M	4230	685
Admix. S	4410	720
Temp. 73°F	4740	735
Temp. 90°F	4310	705
Shape rounded	4330	685
Shape crushed	4720	755
Water-cement 0.39	4680	730
Water-cement 0.44	4370	710
Alkali 0.45	4690	710
Alkali 1.0	4360	730

and relatively lower for Admixture M. As the concrete temperature increased from 73° F to 90° F, the average compressive strength decreased by approximately 9 percent. The strength increased by approximately 9 percent when rounded aggregates were substituted with crushed aggregates. The average compressive strength decreased by approximately 7 percent when the water-cement ratio increased from 0.39 to 0.44. When the alkali level was increased from 0.45 to 1.0 the compressive strength decreased by approximately 7 percent.

On average, the flexural strength was relatively lower for Admixture M when compared to the other air-entraining admixtures. As the concrete temperature increased from 73° F to 90° F, the average flexural strength decreased by approximately 4 percent. The strength increased by approximately 10 percent when rounded aggregates were substituted with crushed aggregates. The average flexural strength slightly decreased (by approximately 3 percent) when the water-cement ratio increased from 0.39 to 0.44. When the alkali level was increased from 0.45 to 1.0 the flexural strength slightly increased (by approximately 3 percent) as opposed to the 7-percent decrease in average compressive strength.

A stepwise least-squares linear regression on the combined data set was conducted to predict strength from significant main effects and two-way interactions. To reduce multicollinearity, independent variables were transformed by subtracting the mean (two levels set in the design of experiment). Results of this linear regression analysis are summarized in Table 11. Significant variables affecting compressive strength include concrete temperature (three of the six air-entraining admixtures), aggregate shape (two of the six air-entraining admixtures), cement alkali level (two of the six air-entraining admixtures), and water cement ratio (one of the six air-entraining admixtures.

Concrete temperature, aggregate shape, and cement alkali level main factors were also significant for the linear regression conducted on the combined data set. Compressive strength decreased with increasing temperature, using rounded instead of crushed aggregate, and increasing the cement alkali level. The dummy variable used for the different admixtures was also significant indicating that compressive strength was a function of the different air-entraining admixtures.

Several interaction variables identified where not consistently significant among the different air-entraining admixtures (no more than three different air-entraining admixtures). Significant interaction variables included combinations of mixing time duration, concrete temperature, aggregate shape, and water to cement ratio. The concrete unit weight was significant in predicting compressive strength for two of the six different air-entraining admixtures and the combined data set.

Results of regression analysis of flexural strength are summarized in Table 12. Flexural strength was primarily affected by coarse aggregate shape (five of the six air-entraining admixtures and combined data set). As listed in Table 10, the average flexural strength for mixes made with crushed coarse aggregate was 755 psi and for mixes made with rounded aggregate was 685 psi.

Mix temperature and alkali level were also identified as significant factors in the combined data set. Flexural strength decreased with increasing temperature, using rounded aggregate instead of crushed aggregate, and decreasing the cement alkali level. Significant interaction variables were identified only with Admixture C and the data set combining all data for the six different air-entraining admixtures. The dummy variable used for the different admixtures was significant indicating that compressive strength was a function of the different air-entraining admixtures.

Significant main factors affecting both compressive and flexural strength for the combined data set (all six different air-entraining admixtures) were ADMIX_ID (dummy variable for the different air entrainers), concrete temperature (decreasing strength with increasing temperature), coarse aggregate shape (increasing strength using crushed aggregates instead of rounded aggregates), and cement alkali level. The change in strength with alkali levels was different between compressive and flexural strength. Compressive strength decreased and flexural strength increased with increasing cement alkali level.

Results of the linear regression are consistent with the ANOVA data summarized in Table 10 where the average strength decreased with increasing concrete temperature, the average strength increased when using crushed aggregates instead of rounded aggregates, the compressive strength decreased with increasing cement alkali levels, and the flexural strength increased with increasing cement alkali levels. The water-cement ratio was not significant in the linear regression analysis but significant in the ANOVA analysis.

Additional analyses were conducted to evaluate significant variables using reduced data sets. The first analyses conducted were linear regression analyses using strengths determined for just the lower concrete temperature and using strengths determined for just the higher concrete temperature. The second set of analyses conducted were linear regression analyses using strengths determined for just rounded coarse aggregates and using strengths determined for just crushed coarse aggregates. The last set of analyses conducted were linear regression analyses using strengths determined for just low alkali cement and using strengths determined for just high alkali cement. Results of the subset linear regression analyses for compressive and flexural strength are listed in Tables 13 and 14 below.

The significant main factor variables common to the compressive strength analysis at low and high temperatures were cement alkali level and unit weight. The compressive strength decreased as the alkali level increased or the unit weight

Table 11. Stepwise linear regression for compressive strength.

	Admixture B		Admixture C		Admixture D		Admixture E		Admixture M		Admixture S		All Data	
Independent Variable	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %
ADMIX_ID	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	-6.876E+01	0.0
TEMPD	-5.027E+01	0.1	****	****	-3.074E+01	0.0	****	****	****	****	-2.993E+01	3.0	-2.639E+01	0.0
SHAPED	****	****	****	****	2.800E+02	0.0	****	****	****	****	4.688E+02	4.3	2.167E+02	0.2
WCD	****	****	****	****	-7.650E+03	0.0	****	****	****	****	****	****	****	****
ALKD	****	****	-1.218E+03	0.0	****	****	-9.934E+02	2.9	****	****	****	****	-5.471E+02	0.0
DURDxTEMPD	****	****	3.348E+00	0.5	2.014E+00	0.0	****	****	****	****	****	****	1.238E+00	3.2
DURDxWCD	****	****	-9.000E+02	1.8	-1.023E+03	0.0	****	****	-1.559E+03	2.9	****	****	-7.023E+02	0.1
TEMPDxSHAPED	****	****	-4.088E+01	0.7	****	****	****	****	****	****	****	****	-3.398E+01	0.0
TEMPDxWCD	****	****	****	****	4.294E+02	0.1	****	****	****	****	****	****	****	****
SHAPEDxWCD	****	****	1.840E+04	0.1	-4.400E+03	1.2	****	****	****	****	****	****	****	****
UW	2.597E+02	0.3	****	****	****	****	****	****	2.926E+02	0.2	****	****	1.822E+02	0.0
Constant	-3.321E+04	0.7	4.533E+03	0.0	4.719E+03	0.0	4.649E+03	0.0	-3.832E+04	0.4	4.439E+03	0.0	-2.179E+04	0.0
R-squared (adj.)	0.666		0.854		0.975		0.246		0.456		0.374		0.678	

NOTES:

1. Dummy variable ADMIX_ID set to 1 for Admixture B, 2 for Admixture C, 3 for Admixture D, etc.
2. DURD = mixing time duration minus mean
   (DURD = DUR minus 8.5)
3. TEMPD = mix temperature minus mean
   (TEMPD = TEMP minus 81.5)
4. SHAPED = coarse aggregate shape minus mean
   (SHAPED = SHAPE minus 0.5 where SHAPE=0 for rounded, 1 for crushed)
5. WCD = water cement ratio minus mean
   (WCD = WC minus 0.405)
6. ALKD = cement alkali minus mean
   (ALKD = ALK minus 0.725)

Table 12. Stepwise linear regression for flexural strength.

	Admixture B		Admixture C		Admixture D		Admixture E		Admixture M		Admixture S		All Data	
Independent Variable	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %
ADMIX_ID	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	-8.472	0.0
TEMPD	****	****	****	****	****	****	****	****	****	****	****	****	-2.040E+00	0.0
SHAPED	1.025E+02	0.0	6.188E+01	0.1	7.813E+01	2.9	****	****	7.250E+01	0.4	9.250E+01	0.5	5.928E+01	0.0
WCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
ALKD	****	****	****	****	****	****	****	****	****	****	****	****	4.133E+01	0.4
DURDxWCD	****	****	-1.096E+02	2.3	****	****	****	****	****	****	****	****	-7.560E+01	0.2
TEMPDxWCD	****	****	****	****	****	****	****	****	****	****	****	****	-4.199E+01	2.3
TEMPDxALKD	****	****	****	****	****	****	****	****	****	****	****	****	3.556E+00	3.4
SHAPEDxWCD	****	****	2.075E+03	0.3	****	****	****	****	****	****	****	****	****	****
WCDxALKD	****	****	-3.682E+03	0.3	****	****	****	****	****	****	****	****	-2.326E+03	****
UW	****	****	****	****	****	****	****	****	****	****	****	****	1.454E+01	0.0
Constant	7.431E+02	0.0	7.234E+02	0.0	7.372E+02	0.0	****	****	6.863E+02	0.0	7.163E+02	0.0	-1.369E+03	0.2
R-squared (adj.)	0.576		0.777		0.248		****		0.413		0.407		0.647	

NOTES:

1. Dummy variable ADMIX_ID set to 1 for Admixture B, 2 for Admixture C, 3 for Admixture D, etc.
2. DURD = mixing time duration minus mean
   (DURD = DUR minus 8.5)
3. TEMPD = mix temperature minus mean
   (TEMPD = TEMP minus 81.5)
4. SHAPED = coarse aggregate shape minus mean
   (SHAPED = SHAPE minus 0.5 where SHAPE=0 for rounded, 1 for crushed)
5. WCD = water cement ratio minus mean
   (WCD = WC minus 0.405)
6. ALKD = cement alkali minus mean
   (ALKD = ALK minus 0.725)

Table 13. Stepwise linear regression for subsets of compressive strength.

	Low Temperature		High Temperature		Rounded Aggregates		Crushed Aggregates		Low Alkali		High Alkali		All Data	
Independent Variable	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefifcient	Signif. Level, %
ADMIX_ID	-1.055E+02	0.0	****	****	-6.821E+01	2.6	-7.234E+01	0.7	-7.124E+01	1.3	-6.636E+01	0.8	-6.876E+01	0.0
DURD	****	****	1.550E+01	2.4	****	****	****	****	****	****	****	****	****	****
TEMPD	****	****	****	****	****	****	-4.397E+01	0.0	-2.603E+01	0.0	-2.771E+01	0.0	-2.639E+01	0.0
SHAPED	5.067E+02	0.1	****	****	****	****	****	****	3.417E+02	0.2	****	****	2.167E+02	0.2
WCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
ALKD	-5.344E+02	0.2	-5.607E+02	0.1	****	****	-7.160E+02	0.0	****	****	****	****	-5.471E+02	0.0
DURDxTEMPD	****	****	****	****	****	****	****	****	1.930E+00	2.9	****	****	1.238E+00	3.2
DURDxWCD	****	****	-1.014E+03	0.0	-6.716E+03	3.7	-7.160E+02	0.7	****	****	****	****	-7.023E+02	0.1
TEMPDxSHAPED	****	****	****	****	****	****	****	****	****	****	-6.396E+01	0.0	-3.398E+01	0.0
TEMPDxWCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
SHAPEDxWCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
UW	1.806E+02	0.0	1.701E+02	0.0	-2.019E+03	0.0	2.106E+02	0.0	1.677E+02	0.0	2.242E+02	0.0	1.822E+02	0.0
Constant	-2.120E+04	0.0	-2.049E+04		1.705E+02	0.0	-2.581E+04	0.0	-1.893E+04	0.1	-2.807E+04	0.0	-2.179E+04	0.0
R-squared (adj.)	0.693		0.537		0.372		0.724		0.624		0.687		0.678	

NOTES:

1. Dummy variable ADMIX_ID set to 1 for Admixture B, 2 for Admixture C, 3 for Admixture D, etc.
2. DURD = mixing time duration minus mean
   (DURD = DUR minus 8.5)
3. TEMPD = mix temperature minus mean
   (TEMPD = TEMP minus 81.5)
4. SHAPED = coarse aggregate shape minus mean
   (SHAPED = SHAPE minus 0.5 where SHAPE=0 for rounded, 1 for crushed)
5. WCD = water cement ratio minus mean
   (WCD = WC minus 0.405)
6. ALKD = cement alkali minus mean
   (ALKD = ALK minus 0.725)

Table 14. Stepwise linear regression for subsets of flexural strength.

	Low Temperature		High Temperature		Rounded Aggregates		Crushed Aggregates		Low Alkali		High Alkali		All Data	
Independent Variable	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %	Coefficient	Signif. Level, %
ADMIX_ID	****	****	8.461E+00	0.5	-6.564E+00	1.9	-8.750E+00	3.4	****	****	-1.069E+01	0.4	-8.472	0.0
DURD	****	****	1.662E+00	3.3	****	****	****	****	****	****	****	****	****	****
TEMPD	****	****	****	****	-2.465E+00	0.0	****	****	-3.088E+00	0.0	****	****	-2.040E+00	0.0
SHAPED	6.250E+01	0.0	6.101E+01	0.0	****	****	****	****	6.667E+01	0.0	6.198E+01	0.0	5.928E+01	0.0
WCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
ALKD	****	****	7.153E+01	0.0	****	****	****	****	****	****	****	****	4.133E+01	0.4
DURDxTEMPD	4.872E+00	2.3	-4.518E+00	0.4	****	****	****	****	****	****	****	****	****	****
DURDxWCD	****	****	****	****	-1.285E+02	0.0	****	****	-8.974E+01	0.4	****	****	-7.560E+01	0.2
TEMPDxWCD	****	****	****	****	****	****	****	****	****	****	****	****	-4.199E+01	2.3
TEMPDxALKD	****	****	****	****	****	****	****	****	****	****	****	****	3.566E+00	3.4
SHAPExWCD	****	****	****	****	****	****	****	****	****	****	****	****	****	****
WCDxALKD	****	****	****	****	-3.125E+03	0.0	****	****	****	****	****	****	-2.326E+03	0.0
UW	****	****	1.943E+01	0.0	1.721E+01	0.0	****	****	****	****	2.296E+01	0.0	1.454E+01	0.0
Constant	7.371E+02	0.0	-2.098E+03	0.1	-1.792E+03	0.0	7.879E+02	3.4	7.108E+02	0.0	-2.575E+03	0.0	-1.369E+03	0.2
R-squared (adj.)	0.348		0.734		0.663		0.074		0.557		0.610		0.647	

Notes:

1. Dummy variable ADMIX_ID set to 1 for Admixture B, 2 for Admixture C, 3 for Admixture D, etc.
2. DURD = mixing time duration minus mean
   (DURD = DUR minus 8.5)
3. TEMPD = mix temperature minus mean
   (TEMPD = TEMP minus 81.5)
4. SHAPED = coarse aggregate shape minus mean
   (SHAPED = SHAPE minus 0.5 where SHAPE=0 for rounded, 1 for crushed)
5. WCD = water cement ratio minus mean
   (WCD = WC minus 0.405)
6. ALKD = cement alkali minus mean
   (ALKD = ALK minus 0.725)

decreased. The significant variables common to the flexural strength analysis at low and high temperatures were the coarse aggregate shape and the two-way interaction of duration and coarse aggregate shape. The flexural strength increased when using crushed aggregates instead of rounded aggregates. The flexural strength using coarse aggregates decreases when the mixing time duration increases.

The significant main factor variables common to the compressive strength analysis for rounded and coarse aggregates were the dummy variable for admixtures, the concrete unit weight, and the two-way interaction of duration and water-cement ratio. The compressive strength increases as with unit weight. When both the mixing time duration and water-cement increase, the compressive strength decreases. The only significant variable common to the flexural strength analysis for rounded and coarse aggregates was the dummy variable for admixtures.

The significant main factor variables common to the compressive strength analysis at low and high cement alkali levels were the dummy variable for admixtures, concrete temperature, and unit weight. The compressive strength decreased with increasing temperature and decreasing unit weight. The only significant variable common to the flexural strength analysis at low and high cement alkali levels was the coarse aggregate shape. The flexural strength increased when using crushed aggregates instead of rounded aggregates.

Effects of Air-Entraining Admixture Type on Strength

As shown in Figures 6 and 7, compressive and flexural strengths of concrete made with non-Vinsol resin air-entraining admixtures were generally lower than for concrete made with the Vinsol resin admixture. Compressive strength was lower for 55 of the 80 mixes (16 different mixes with 5 non-Vinsol resin air-entraining admixtures) by an average of 217 psi and flexural strengths were lower for 53 of the 80 mixes by an average of 21 psi.

Effects of non-Vinsol resin air-entraining admixtures on concrete compressive and flexural strength were analyzed. Admixture D was considered as the standard Vinsol admixture in the analysis. Strengths differences for each of the 16 different mixes were first computed by subtracting the average compressive or flexural strength using Admixture D (Vinsol resin) from the average strength using the other tested admixtures (refereed to as non-Vinsol resin) (Admixtures B, C, E, M, and S).

An analysis of variance (ANOVA) was conducted for the strength differences of all 96 concrete mixes (6 admixtures and 16 mixes per admixture). A 10 percent significance level (SL) was selected as the criterion for separation between significant and residual effects. Effects with a significance level of less than or equal to 10 percent were assumed to be significant. Conversely, effects with a significance level greater than 10 percent were assumed attributed to residual variation. An identification variable (random variable) to account for the six different air-entraining admixtures was included in the analysis for the 96 mixes.

Table 15. Analysis of variance for strength differences.

Independent Variable	Significance Level, percent	
	Compressive Strength	Flexural Strength
ADMIX_ID	3	0
DUR	>10	7
TEMP	>10	8
SHAPE	10	>10
WC	>10	0
ALK	0	0
DURxTEMP	>10	>10
DURxSHAPE	>10	4
DURxWC	3	0
DURxALK	>10	>10
TEMPxSHAPE	0	>10
TEMPxWC	1	2
TEMPxALK	>10	1
SHAPExWC	>10	0
SHAPExALK	>10	0
WCxALK	>10	1

NOTES:

1. The strength difference represents the strength for a given admixture minus the strength for the same mix design using the vinsol resin admixture.
2. The variable ADMIX_ID is a dummy random variable that was used to identify if significant variables were different between admixtures.

Results of the analysis are listed in Table 15. Factors affecting the average compressive strength change include the dummy variable for the different admixtures, coarse aggregate shape, cement alkali level, and the two-way interactions of mixing duration and water-cement ratio, concrete temperature and

Table 16. Change in mean strength differences for significant variables.

Independent Variable	Compressive Strength, psi	Flexural Strength, psi
Admix. B	-88	-6
Admix. C	-186	-14
Admix. E	-69	-23
Admix. M	-462	-51
Admix. S	-279	-21
Duration 2 mins.	N/A	-12
Duration 15 mins.	N/A	-29
Temp. 73°F	N/A	-28
Temp. 90°F	N/A	-13
Shape rounded	-287	N/A
Shape crushed	-147	N/A
Water-cement 0.39	N/A	-5
Water-cement 0.44	N/A	-2
Alkali 0.45	-7	-2
Alkali 1.0	-427	-39

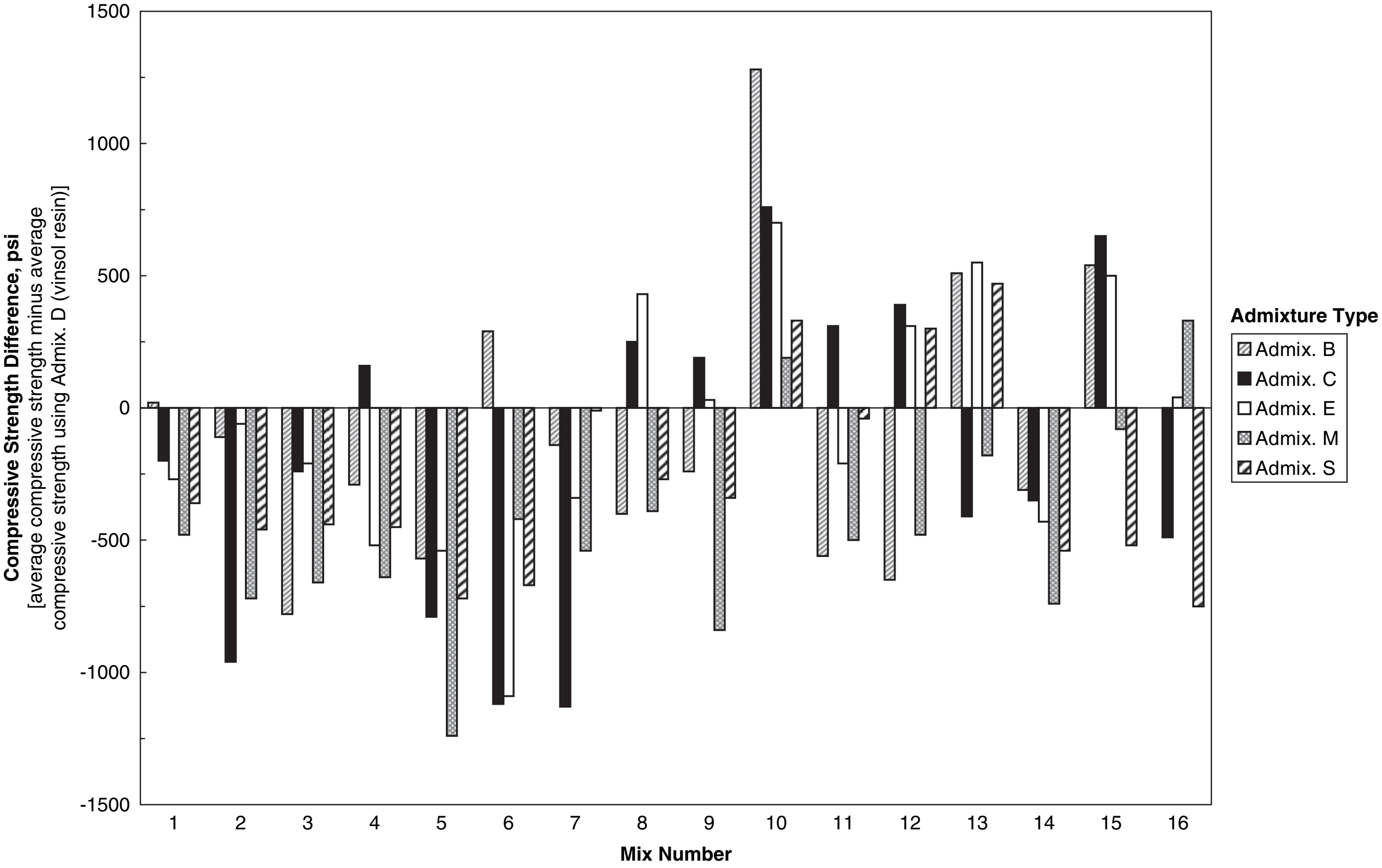


Figure 6. Compressive strength difference.

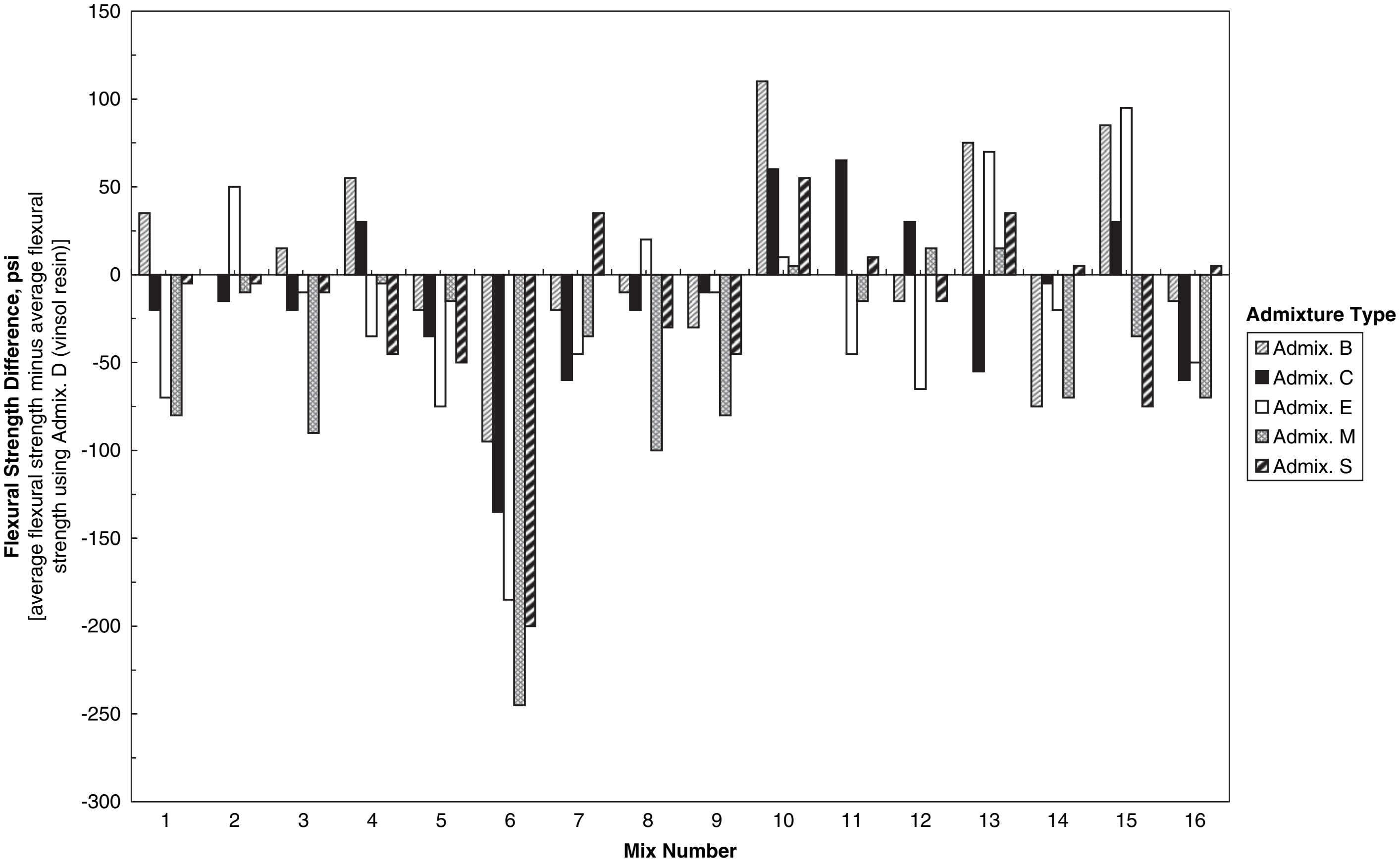


Figure 7. Flexural strength difference.

coarse aggregate shape, and concrete temperature and water-cement ratio.

Factors affecting the average flexural strength change include the dummy variable for the different admixtures, mixing time duration, concrete temperature, water-cement ratio, cement alkali level, and the two-way interactions of mixing duration and coarse aggregate shape, mixing duration and water-cement ratio, concrete temperature and water-cement ratio, concrete temperature and cement alkali level, coarse aggregate shape and water cement ratio, coarse aggregate shape and cement alkali level, and water cement ratio and cement alkali level.

The mean compressive and flexural strength differences (relative to Admixture D) computed between significant main variables identified in the combined data set analysis are listed in Table 16. On average, the compressive strength decrease was relatively greater for Admixtures M and S and relatively lower for Admixtures B and E. The average compressive strength difference was greater for round coarse aggregates (287 psi lower) and higher alkali cement (427 psi lower at the 1 percent alkali content).

On average, the flexural strength decrease was relatively greater for Admixture M (51 psi) and relatively lower for Admixture B (6 psi). The average flexural strength decrease was greater for the longer mixing duration (29 psi for 15 minutes mixing time), cooler concrete temperatures (28 psi at 73° F mixing temperature), lower water-cement ratio (5 psi at a water-cement ratio of 0.45), and higher alkali cement (39 psi at the 1.0 percent alkali content).

CHAPTER 4

Development of Air-Entraining Admixture Evaluation Process

Introduction

The current AASHTO M 154 and T 157 specifications and test methods deal with uniformity and performance requirements for air-entraining admixtures. The procedures developed in this study primarily focus on modifying the performance requirements. The AASHTO uniformity requirements were slightly modified.

Performance Requirements

The procedures are based on preparing concrete mixtures using the air-entraining admixtures in the laboratory under simulated controlled field conditions, and, comparing the physical and mechanical properties of these mixtures with those of reference, non-air-entrained concrete mixtures, prepared using materials from the same source having similar properties under the same conditions. This approach provides a more realistic evaluation of the effects of the air-entraining admixture on physical properties than that achieved by comparing concrete properties with those of mixtures prepared using a reference air-entraining admixture such as Vinsol resin.

The air content of the reference concrete is proposed to be 2 percent or less, and the air content of the concrete prepared with the tested air-entraining admixtures is proposed to be 6 ± 1.0 percent. Because it is sometimes difficult to control the air content of non-air-entrained concrete, the difference in air content between the two concretes (i.e., air-entrained and non-air-entrained) of 4 ± 0.5 percentage points may be used as an alternative requirement. In this case, air content of the reference concrete should be specified to not exceed 3 percent.

The proposed approach includes test procedures (test protocols) for two highway applications: Protocol A for Pavement Concrete and Protocol B for Structural Concrete. Also, different mixtures are proposed for concrete made without supplementary cementitious materials (A-1 and B-1 for pavement and structural concretes, respectively) and for concrete made with fly ass (A-2 and B-2 for pavement and structural concrete, respectively). This approach allows a specific air-entraining admixture to be certified for either one or both applications (i.e., structural and pavement). The testing conditions, such as mixing temperature, cement alkali content, and mixing time, prescribed in the procedure were chosen based on the findings of the laboratory test program conducted in this project.

Testing Protocols

Protocol A—Pavement Concrete

This protocol requires that concrete be mixed for 2 minutes; a drum-type mixer is preferred. It is also required that rounded aggregate with a maximum size of 1.0 in. be used; both coarse and fine aggregate should conform to the requirements of AASHTO M 6 and M 80. The temperature of materials, including water, used in making the concrete should be 90 ± 5° F (this can be achieved by storing the materials, including the water, overnight in a hot room or oven).

A-1—Plain Concrete Mixture requires the following:

- Cement content of 564 pcy,
- w/c of 0.44,
- Low-alkali cement (maximum of 0.60 percent as equivalent Na_2O), and
- Sand-aggregate ratio to achieve 1 to 3 in. slump.

A-2—Fly Ash Concrete Mixture requires the following:

- Cement content of 450 pcy,
- 110 pcy of fly ash (Class F or Class C) with loss-on-ignition (LOI) of 3 to 5 percent,
- Low-alkali cement (maximum of 0.60 percent as equivalent Na_2O),

- w/c of 0.40, and
- Sand-aggregate ratio to achieve 1 to 3 in. slump.

Protocol B—Structural Concrete

This protocol requires that concrete be mixed for 5 minutes; drum-type or pan-type mixers can be used. It also requires that rounded aggregate with a maximum particle size of 1 in. be used; both coarse and fine aggregates should conform to the requirements of AASHTO M 6 and M 80. The temperature of concrete materials prior to mixing should be 90 ± 5° F (this can be achieved by storing the concrete materials, including the water, overnight in a hot room or oven).

B-1—Plain Concrete (Bridge Deck) requires the following:

- Cement content of 605 pcy,
- w/c of 0.39,
- Low-alkali cement (maximum of 0.60 percent as equivalent Na_2O),
- Use of a high-range water reducer (HRWR); dosages to achieve slump of 4 to 7 in.

B-2—Fly Ash Concrete (Bridge Deck, Super- or Substructure) requires the following:

- Cement content of 515 pcy,
- 140 pcy of fly ash (Class F or C) with a loss-on-ignition (LOI) 3 to 5 percent,
- Low-alkali cement (maximum of 0.60 percent as equivalent Na_2O),
- w/c ratio of 0.42, and
- Use of a Type A water reducer; dosages to achieve slump of 3 to 6 in.

Each test protocol requires measurement of the properties of the fresh and hardened concrete. The fresh concrete properties to be measured are slump (AASHTO T 119), air content using the pressure method (AASHTO T 152), and time of setting (AASHTO T 197).

For hardened concrete, the protocol requires preparing twelve 4-by-8-in. cylinders for compressive (AASHTO T 22) and split tensile (AASHTO T 198) tests at ages of 7 and 28 days; six 3-by-3-by-11 1/4 in. beams for flexural strength (AASHTO T 97), three beams for air-void parameter measurement (ASTM C 457), three beams for freeze-thaw testing (AASHTO T 161), and three 12-by-12-by-3-in. slabs for deicer scaling (ASTM C 672) (if needed). All prepared specimens are to be consolidated by vibration and then subjected to moist curing for 3 days and air cured until testing.

The proposed performance requirements for the concrete mixtures made with the air-entraining admixtures are as follows:

- Compressive, flexural, and split tensile strengths at the ages of 7 and 28 days not less than 75 percent of those of the reference concrete.
- Average spacing factor (three specimens) within the range of 0.002 to 0.008 in. (0.05 to 0.2 mm), with no single test result greater than 0.010 in. (0.23 mm).

In any instances where the strength of the concrete is more than 75 percent of the reference, but the spacing factor is out of the required range, freeze-thaw and deicer scaling tests should be conducted on the air-entrained concrete mixture in accordance with AASHTO T 161 and ASTM C 672. Proposed performance requirements are as follows:

- The durability factor should be more than 80 percent.
- The condition of surface after 50 cycles in the deicer-scaling test should be rated 0 to 1 (no to very slight scaling).
- Both initial and final time of setting (AASHTO T 197) should not deviate from the reference concrete by more than ±75 minutes.

The 75-percent strength reduction criterion was selected to recognize an anticipated loss of strength of between 2 and 6 percent per unit addition of air (Bloem, 1950; Walker and Bloem, 1955; Whiting and Nagi, 1998, Pinto and Hover, 2001). Therefore, for a difference in air content of 4 to 5 percent between the reference and tested concretes, a maximum reduction of strength of approximately 25 to 30 percent is expected. A reduction of strength in excess of 30 percent will be most likely due to the air-void system generated by the tested air-entraining admixture. Thus, a reduction of 25 percent was selected as a primary acceptance criterion.

Although the proposed procedures may require additional effort compared with the current procedures for evaluating air-entraining admixtures (e.g., AASHTO M 154), they will better indicate concrete performance under field conditions. The proposed procedures are presented in Attachment A.

Validation of the Proposed Procedures

The proposed procedures were validated by testing five different air-entraining admixtures selected on the basis of their properties and performance records. Each admixture was tested following the four testing protocols; test results were compared with similar reference concrete mix (non-air-entrained). The acceptance requirements were examined, and the validity of the results was evaluated.

In addition, for two of the admixtures, the strength criteria (air-entrained versus non-air-entrained concrete) were examined using the standard concrete mix design and conditions listed in AASHTO T 157. This process verified whether

Table 17. Foam drainage test results for water only tests.

	Foam Drainage Statistics				Foam Height			
Admixture	V_0	-1/k	r^2	% drained	Initial	15 min	30 min	60 min
ES	238.0	314.6	0.886	76.8	740	710	670	620
EN	304.1	4.4	0.382	98.1	310	310	310	310
EA	240.8	241.2	0.758	77.7	870	825	800	780
ED	276.0	32.0	0.988	89.0	440	390	380	340
EV	280.2	267.7	0.950	90.4	620	560	530	520

Table 18. Foam drainage test results for water plus cement tests.

	Foam Drainage Statistics				Foam Height			
Admixture	V_0	-1/k	r^2	% drained	Initial	15 min	30 min	60 min
ES	262.3	254.8	0.949	84.6	520	480	400	310
EN	234.6	144.0	0.916	75.7	550	550	540	530
EA	235.7	49.2	0.427	76.0	620	580	430	400
ED	235.1	59.9	0.830	75.8	470	460	455	455
EV	122.2	150.3	0.549	39.4	725	720	720	720

the strength reduction was associated with field-simulated conditions in the proposed protocols.

Properties of the Selected Admixtures

The following five air-entraining admixtures were selected for use in validating the proposed test procedure:

- Admixture ES (Synthetic, Sodium Olfin Sulfonate);
- Admixture EN (Vinsol);
- Admixture EA (Synthetic, Alpha Olfin Sulfonate);
- Admixture ED (Resin/Rosin and Fatty Acid); and
- Admixture EV (Vinsol).

Admixtures ES, EN, and EA are commercially available but not widely used in the field. Admixture ED is commonly used, but it was reported by some highway agencies to have poor field performance. Admixture EV is a Vinsol resin-based admixture accepted and used by most highway agencies.

The foam drainage test results of the five admixtures are listed in Tables 17 and 18. The values of −1/k, which is an indication of the rate of drainage, are significantly different for the five admixtures in both water and water plus cement tests, suggesting potentially different air-void systems and different performance. Infrared spectra of the five admixtures are shown in Appendix B.

Test Results

Trial mixes were conducted to define the dosages needed for each admixture and to optimize the mixing procedures and practicality of conducting the evaluation (e.g., storing the materials at 90° F). The alkali content of the ASTM Type I cement was 0.24 percent (as equivalent Na_2O). The LOI of the ASTM Class Fly Ash used was 4.2 percent. For Protocol A, the sequence of charging of materials into the mixer was started by adding the aggregate and sand, followed by adding 40 percent of the mixing water, and mixing for 15 seconds. Air-entraining admixture was then added, followed by the cementitious materials and remaining water. Mixing time was considered to start after adding the remaining water. Similar batching and mixing sequences were used for Protocol B, except that chemical admixtures (water reducer or HRWR) were added after charging the cementitious materials.

After concrete mixing was complete, tests for slump (AASHTO T 119), air content (AASHTO T 152), and unit weight (AASHTO T 121) were performed on the fresh concrete; results are listed in Table 19. Concrete was accepted for specimen preparation if the measured properties met the proposed requirements. For hardened concrete tests, specimens were prepared following AASHTO T 126 procedures. Exposed surfaces of specimens were covered by polyethylene sheeting and wet burlap to prevent evaporation of water from the concrete during the first 18 to 24 hours after casting. The specimens were then demolded and placed in a moist room maintained at 73.4 ± 3° F. For each testing age, specimens were prepared from three different batches.

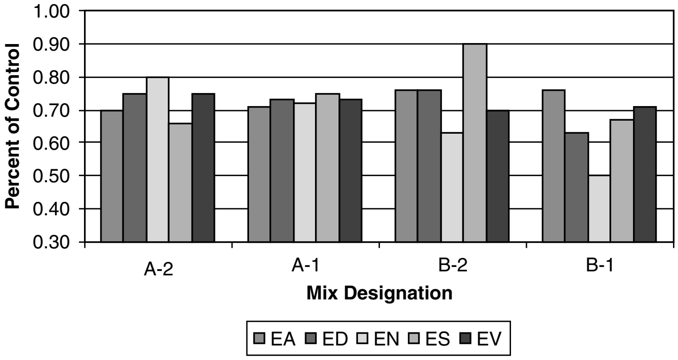
Compressive, flexural, and split tensile strength test results at the age of 7 and 28 days and hardened concrete air content and air-void spacing factors for each mix are listed in Table 20. Figures 8 through 10 show the variation in strength expressed as percent of control for each of the four protocols for the five admixtures.

Table 19. Mix design and fresh concrete properties of protocol mixes.

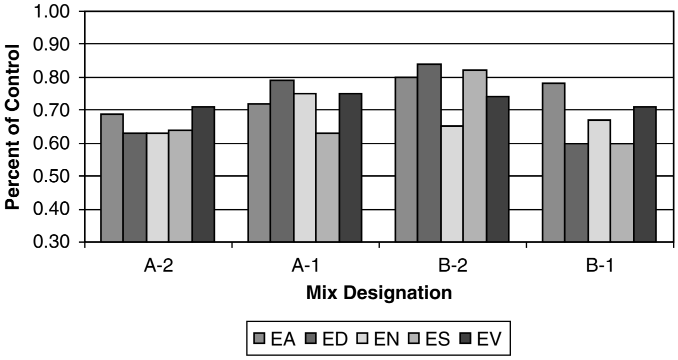
Mixture Designation	Description	AEA	Cement Constant (pcy)	Fly ash Constant (pcy)	Admixture	W/C	Air (%)	Units Weight (pcf)	Slump (in.)
APCON	Protocol A Plain	-	564	-	-	0.44	2.5	151	1
AFCON	Protocol A Fly ash	-	450	110	-	0.40	1.8	150	1.25
BPCON	Protocol B Plain	-	605	-	HRWR	0.39	2.4	153	4
BFCON	Protocol B Fly ash	-	515	140	WR	0.42	1.4	152	5.5
APEA	Protocol A Plain	Admixture EA	564	-	-	0.44	6.1	144	3
AFEA	Protocol A Fly ash	Admixture EA	450	110	-	0.40	6	144	1
BPEA	Protocol B Plain	Admixture EA	605	-	HRWR	0.39	5.8	147	4.25
BFEA	Protocol B Fly ash	Admixture EA	515	140	WR	0.42	6	142	3
APES	Protocol A Plain	Admixture ES	564	-	-	0.44	5.9	145	1.5
AFES	Protocol A Fly ash	Admixture ES	450	110	-	0.40	6.1	142	1.3
BPES	Protocol B Plain	Admixture ES	605	-	HRWR	0.39	6.2	144	4.25
BFES	Protocol B Fly ash	Admixture ES	515	140	WR	0.42	5.8	142	4.25
APEN	Protocol A Plain	Admixture EN	564	-	-	0.44	6	144	3
AFEN	Protocol A Fly ash	Admixture EN	450	110	-	0.40	5.4	146	2.75
BPEN	Protocol B Plain	Admixture EN	605	-	HRWR	0.39	5.6	145	5.25
BFEN	Protocol B Fly ash	Admixture EN	515	140	WR	0.42	6	142	5.25
APED	Protocol A Plain	Admixture ED	564	-	-	0.44	5.8	147	2.5
AFED	Protocol A Fly ash	Admixture ED	450	110	-	0.40	6	145	1.75
BPED	Protocol B Plain	Admixture ED	605	-	HRWR	0.39	5.9	146	4
BFED	Protocol B Fly ash	Admixture ED	515	140	WR	0.42	5.1	145	4.5
APEV	Protocol A Plain	Admixture EV	564	-	-	0.44	5.5	146	3
AFEV	Protocol A Fly ash	Admixture EV	450	110	-	0.40	6.3	144	3.25
BPEV	Protocol B Plain	Admixture EV	605	-	HRWR	0.39	6.4	147	6
BFEV	Protocol B Fly ash	Admixture EV	515	140	WR	0.42	5.8	145	5.5

Table 20. Protocol mixes strength test results.

Mixture Designation	Comp.		Flex.		Split		Air Cont. (%)	Hardened Air Cont. (%)	Spacing Factor (in.)
	7 day	28 day	7 day	28 day	7 day	28 day			
APCON	4850	5950	590	730	450	620	2.5	2.3	0.0186
AFCON	4120	6150	540	750	400	640	1.8	1.3	0.0159
BPCON	6110	8370	740	850	540	790	2.4	1.7	0.0240
BFCON	4850	6040	690	820	500	590	1.4	2.5	0.0108
APEA	3450	4290	580	630	420	490	6.1	5.1	0.0047
% of Control	*0.71*	*0.72*	*0.98*	*0.86*	*0.93*	*0.79*			
AFEA	2900	4270	460	600	360	470	6.0	6.4	0.0056
% of Control	*0.70*	*0.69*	*0.85*	*0.80*	*0.90*	*0.73*			
BPEA	4650	6570	600	690	530	640	5.8	5.4	0.0038
% of Control	*0.76*	*0.78*	*0.81*	*0.81*	*0.98*	*0.81*			
BFEA	3710	4820	520	610	350	400	6.0	6.7	0.0021
% of Control	*0.76*	*0.80*	*0.75*	*0.74*	*0.70*	*0.68*			
APES	3620	3750	510	690	370	400	5.9	6.2	0.0036
% of Control	*0.75*	*0.63*	*0.86*	*0.95*	*0.82*	*0.65*			
AFES	2730	3950	470	600	320	470	6.1	5.1	0.0034
% of Control	*0.66*	*0.64*	*0.87*	*0.80*	*0.80*	*0.73*			
BPES	4110	5050	660	670	420	550	6.2	6.2	0.0042
% of Control	*0.67*	*0.60*	*0.89*	*0.79*	*0.78*	*0.70*			
BFES	4380	4950	440	560	480	500	5.8	5.2	0.0034
% of Control	*0.90*	*0.82*	*0.64*	*0.68*	*0.96*	*0.85*			
APEN	3500	4440	470	540	420	510	6.0	5.5	0.0031
% of Control	0.72	0.75	0.80	0.74	0.93	0.82			
AFEN	3290	3880	440	510	360	430	5.5	4.8	0.0041
% of Control	*0.80*	*0.63*	*0.81*	*0.68*	*0.90*	*0.67*			
BPEN	3050	5630	550	710	380	600	5.6	6	0.0034
% of Control	*0.50*	*0.67*	*0.74*	*0.84*	*0.70*	*0.76*			
BFEN	3060	3930	510	610	320	410	6.0	6.3	0.0042
% of Control	*0.63*	*0.65*	*0.74*	*0.74*	*0.64*	*0.69*			
APCON	4850	5950	590	730	450	620	2.5	2.3	0.0186
AFCON	4120	6150	540	750	400	640	1.8	1.3	0.0159
BPCON	6110	8370	740	850	540	790	2.4	1.7	0.0240
BFCON	4850	6040	690	820	500	590	1.4	2.5	0.0108
APED	3530	4700	580	650	390	540	5.8	4.3	0.0027
% of Control	*0.73*	*0.79*	*0.98*	*0.89*	*0.87*	*0.87*			
AFED	3080	3860	540	630	320	480	6.0	5.3	0.0024
% of Control	*0.75*	*0.63*	*1.00*	*0.84*	*0.80*	*0.75*			
BPED	3870	5030	550	660	440	506	5.9	5.1	0.0035
% of Control	*0.63*	*0.60*	*0.74*	*0.78*	*0.81*	*0.64*			
BFED	3710	5060	580	678	360	504	5.1	5.3	0.0031
% of Control	*0.76*	*0.84*	*0.84*	*0.83*	*0.72*	*0.85*			
APEV	3530	4440	590	650	350	440	5.5	5.5	0.0043
% of Control	*0.73*	*0.75*	*1.00*	*0.89*	*0.78*	*0.71*			
AFEV	3080	4380	500	530	330	460	6.3	6.0	0.0041
% of Control	*0.75*	*0.71*	*0.93*	*0.71*	*0.83*	*0.72*			
BPEV	4350	5910	550	730	490	580	6.4	6.1	0.0051
% of Control	*0.71*	*0.71*	*0.74*	*0.86*	*0.91*	*0.73*			
BFEV	3410	4440	540	660	370	455	5.8	4.4	0.0052
% of Control	*0.70*	*0.74*	*0.78*	*0.80*	*0.74*	*0.77*			

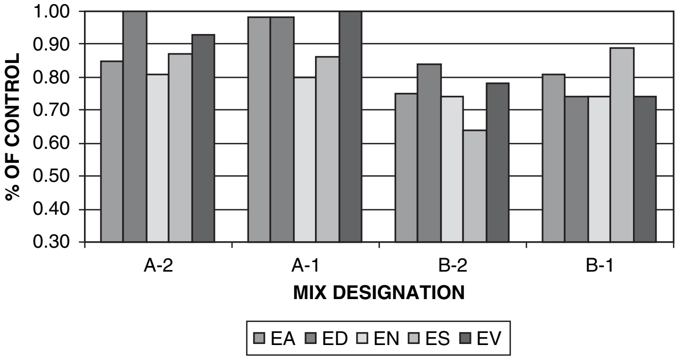


(a) 7-Day Compressive Strength

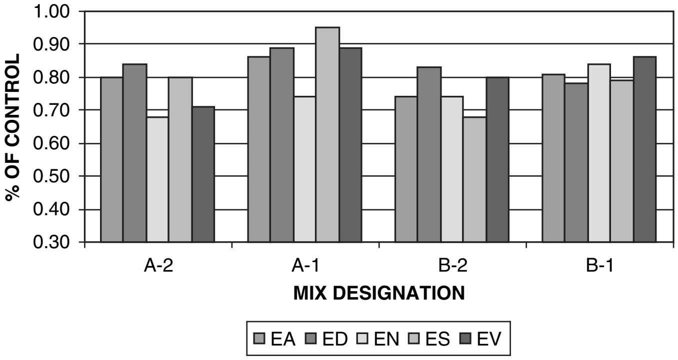


(b) 28-day Compressive Strength

Figure 8. Percent of Compressive Strength at 7 and 28 Days.

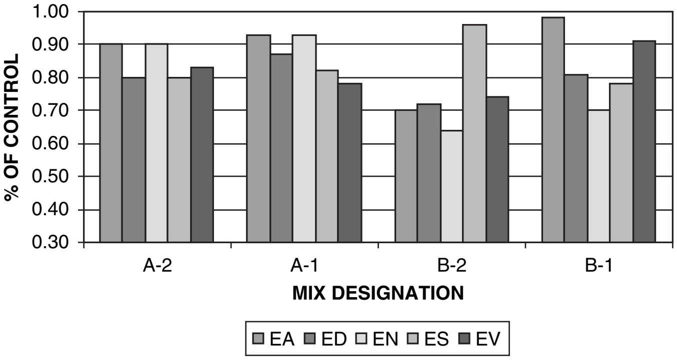


(a) 7-Day Flexural Strength

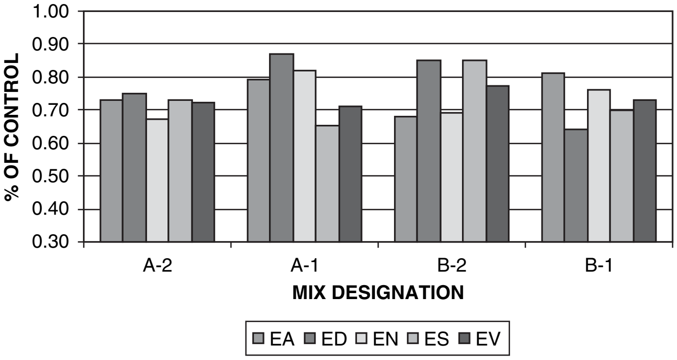


(b) 28-day Flexural Strength

Figure 9. Percent of Flexural Strength at 7 and 28 Days.



(a) 7-Day Splitting Tensile Strength



(b) 28-day Splitting Tensile Strength

Figure 10. Percent of Splitting Tensile Strength at 7 and 28 Days.

The difference in strength values between concrete prepared with the admixtures and reference concrete (control) ranged from 50 to 100 percent. Although none of the five admixtures completely satisfied the strength requirements (i.e., 75 percent of the control) for any of the four protocols, strength data of Admixtures EV and EA at both 7 and 28 days were relatively high (i.e., more than 70 percent of reference concretes) and the spacing factor for the four protocol mixes were within the limits (i.e., 0.002-0.008 in.). However, compressive strength test results of the other three admixtures (Admixtures ED, EN, and ES) were generally low (i.e., less than 70 percent of the reference), indicating that the strength reduction may be associated with the specific admixtures, not the reduction in air content. The difference in time of set between tested admixture concrete and that of reference concrete was less than 90 minutes for all tested admixtures.

The admixtures selected for the validation process were manufacturer-certified following ASTM C 260 and AASHTO M 154 procedures, stipulating that compressive and flexural strengths of concrete entrained with these admixtures and prepared under standard laboratory conditions were at least 90 percent of concrete entrained with standard Vinsol resin tested under the same conditions. However, the reduction in compressive strength obtained for the proposed test conditions suggests that poorer performance is likely to occur under field conditions. These data also indicate that the proposed test conditions are more rigorous than those proposed in the current standard.

To confirm such effect, two of the tested admixtures (ED and EV) were tested under standard laboratory conditions in accordance with AASHTO T 157, except that a non-air-entrained concrete was used for reference (instead of concrete entrained with Vinsol resin). As listed in Table 21, the compressive, flexural, and split tensile strength values at both ages (7 and 28 days) were greater than 75 percent of the control (majority of the data were 80 percent or higher of the reference mix strength values). This indicates that the reduction in strength is attributed to the testing conditions and their closer simulation to field exposures.

Based on the results of these tests, the performance requirements for evaluating air-entraining admixtures for highway concrete were kept the same as proposed earlier in this chapter. However, the reliability of these criteria for concrete pavement and bridge applications could be further evaluated in the field, and the proposed acceptance limits of spacing factors (0.002 to 0.008 in.) could also be examined.

Table 21. Physical properties of the standard mixes according to AASHTO T 157.

	Comp.		Flex.		Split		Air Cont.(%)
	7 day	28 day	7 day	28 day	7 day	28 day	
Control	**4350**	**5960**	**690**	**820**	**470**	**480**	**2.0**
(.75)*(control)	3263	4470	518	615	353	360	
Admixture ED	**3710**	**4990**	**550**	**660**	**410**	**450**	**5.3**
% of Control	*0.85*	*0.84*	*0.80*	*0.80*	*0.87*	*0.94*	
Admixture EV	**3540**	**4590**	**590**	**770**	**420**	**450**	**5.5**
% of Control	*0.81*	*0.77*	*0.86*	*0.94*	*0.89*	*0.94*	

CHAPTER 5

Summary of Findings and Suggested Research

Summary of Findings

Based on the results of the research in this project, the following findings are made:

1. Concrete materials, concrete production procedures, construction practices, and field conditions affect, to varying degrees, the air-void system of concrete.
2. The type of air-entraining admixture has a statistically significant effect on the concrete air-void system (spacing factor). Also, cement alkali level and coarse aggregate shape were found to have a significant effect on the spacing factor.
3. The air-void spacing factor decreases with increases in mixing duration, decreases as crushed aggregate are used instead of rounded aggregate, and increases with increased cement alkali level. The two-factor interaction analysis indicated that the spacing factor increases when the combined mixing duration and alkali level increases.
4. Compressive strength decreases with increasing temperature, using rounded aggregate instead of crushed aggregate, increasing w/c, and increasing cement alkali level.
5. The highest compressive strength (average of six different air-entraining admixtures) was measured for mixes made with crushed coarse aggregate, low water-cement ratio, and low-alkali cement and mixed at low temperature.
6. Compressive and flexural strengths with non-Vinsol resin air-entraining admixtures were generally lower than those for mixtures with Vinsol resin admixtures.

In addition to these findings, procedures are proposed to evaluate and qualify air-entraining admixtures for use in concrete for highway applications, using the laboratory test methods that are expected to yield comparable field performance. Also, performance (acceptance) requirements are proposed for paving concrete and structural concrete, such that an air-entraining admixture can be qualified for either or both applications.

Suggested Research

Suitability of the proposed procedures was confirmed based on a limited laboratory evaluation. Further evaluation using concrete mixtures prepared for use in highway pavements or bridge decks is recommended.

References

Attiogbe, E.K., et al. (1992) "Air-Void System Parameters and Freeze-Thaw Durability of Concrete Containing Superplasticizers," *Concrete International*, American Concrete Institute, July, pp. 57–61.

Barbee, J.F. (1961) "Effect of Mixing Time and Overloading on Concrete Produced by Stationary Mixers," *Highway Research Board Bulletin 295*, HRB, Washington, DC, pp. 26–37.

Buenfeld, N.R. and Okundi, E. (1999) "Release of Air from Unsaturated Aggregate During Setting of Concrete," *Construction and Building Materials*, Vol. 13, pp. 143–147.

Cross, W., et al. (2000) "Investigation of Low Compressive Strengths of Concrete Paving, Precast, and Structural Concrete," *Study SD 1998-03*, South Dakota DOT, Office of Research, August, 78 pp.

Detwiler, R. J. et al. (1996) "Supplementary Cementing Materials for Use In Blended Cements," *Research and Development Bulletin RD112T*, PCA, Skokie, IL.

Dodson, V. (1990) Chapter 6, "Air-Entraining Admixtures," *Concrete Admixtures*, Van Nostrand Reinhold, New York, NY, pp. 129–158.

Fagerlund, G., 1990 "Air-Pore Instability And Its Effect On Concrete Properties," *Nordic Concrete Research*, No. 9, Nordic Concrete Federation, Oslo, pp. 34–52.

Greening, N.R. (1967) "Some Causes for Variations in Required Amount of Air Entraining in Portland Cement Mortar," *PCA Journal*, Vol. 9, No. 2, pp. 22–36.

Gutmann, P.F. (1980) "Bubble Characteristics as They Pertain to Compressive Strength and Freeze-Thaw Durability," Bonding in Cementitious Composites, Materials Research Society Symposium Proceedings, Vol. 114, Materials Research Society, Pittsburgh, PA.

Higginson, E.C. (1952) "Some Effects of Vibration and Handling on Concrete Containing Entrained Air," *Journal of the American Concrete Institute*, Vol. 24, No. 1, pp. 1–12.

Hover, K.C. (1989) "Some Recent Problems with Air-Entrained Concrete," *Cement, Concrete, and Aggregates*, ASTM, Vol. 11, No. 1, pp. 67–72.

Hover, K.C., and Phares, R. (1996) "Impact of Concrete Placing Method on Air Content, Air Void System Parameters, and Freeze-Thaw Durability," *Transportation Research Record 1532*, National Academy Press, Washington, DC, pp. 1–8.

Klieger, P. and Perenchio, W.F. (1976) "Further Laboratory Studies of Portland-Pozzolan Cements." *Research and Development Bulletin RD041.01T*, PCA, Skokie, IL.

Lessard, M., et al. (1996) "Effect of Pumping on Air Characteristics of Conventional Concrete," *Transportation Research Record 1532*, National Academy Press, Washington, DC, pp. 9–14.

Macha, R.K., et al. (1994) Examination Of Air Entrainment Stability Factors of Pumped Concrete, *Research Report 1254-3f*, Texas Transportation Institute, Cooperative Research Program, 109 pp. plus 5 appendices.

Mielenz, R.C., et al. (1958) "Origin, Evolution, and Effects of the Air Void System in Concrete. Part 1—Entrained Air in Unhardened Concrete and Part 4—The Air Void System and Job Concrete," Proceedings, ACI, Vol. 55, pp. 95–121.

Nagi, M.A., and Whiting, D.A. (1994) "Achieving and Verifying Air Content in Concrete," Research and Development Information, RP324, PCA R&D Serial No. 1975, PCA, Skokie, IL, 40 pp. plus 4 appendices.

Pigeon, M., et al. (1992) "Influence of Soluble Alkalies on the Production and Stability of the Air-Void System in Superplasticized and Nonsuperplasticized Concrete," *ACI Materials Journal*, Vol. 89, No. 1, pp. 24–31.

Pistilli, M.F. (1983) "Air Void Parameters Developed by Air-Entraining Admixtures as Influenced by Soluble Alkalies from Fly Ash and Portland Cement," *ACI Journal*, Proceedings, Vol. 80, No. 3, pp. 217–222.

Plante, P., et al. (1989) "The Influence of Water-Reducers on the Production and Stability of the Air Void System in Concrete," *Cement and Concrete Research*, Vol. 19, pp. 621–633.

Pleau, R., et al. (1995) "Influence of Pumping on Characteristics of Air-Void System of High-Performance Concrete," *Transportation Research Record 1478*, National Academy Press, Washington, DC, pp. 30–36.

Powers, T.C. (1965) "Topics in Concrete Technology. 3-Mixtures Containing Intentionally Entrained Air," J. PCA Research and Development Laboratories, Vol. 7, No. 1, pp. 23–41.

Ramachandran, V.S. (1995) *Concrete Admixtures Handbook: Properties, Science and Technology*. Ottawa, Canada: Noyes Publications.

Saucier, F., et al. (1991) "Air-Void Stability, Part V: Temperature, General Analysis and Performance Index," *ACI Materials Journal*, Vol. 88, No. 1, pp. 25–36.

Stark, D. (1986) "Effect of Vibration on the Air-Void System and Freeze-Thaw Durability of Concrete," *Research and Development Bulletin RD092.01T*, PCA.

Stott, D. (1994) "Loss of Freeze-Thaw Durability of Concrete Containing Accelerating Admixtures," *Canadian Journal of Civil Engineering*, Vol. 21, No. 4, pp. 605–613.

Stutzman, P.E. (1999) "Deterioration of Iowa Highway Pavement," *NISTIR 6399*, National Institute of Standards and Technology, 72 pp.

Tymkowicz, S. and Steffes, R. (1999) "Vibration Study for Consolidation of Portland Cement Concrete," *Report No. MLR-95-4*, Iowa Department of Transportation, 94 pp.

Whiting, D.A. and Nagi, M.A. (1998) "Manual on Control of Air Content in Concrete," *EB116*, PCA, Skokie, IL.

Whiting, D.A., et al. (1993) "Synthesis of Current and Projected Concrete Highway Technology," *SHRP C-345*, National Research Council, Washington, DC.

Yingling, J., et al. (1992) "Loss of Air Content in Pumped Concrete," *Concrete International*, Vol. 14, No. 10, pp 57–61.

ATTACHMENT A

Recommended Method for Air-Entraining Admixtures for Highway Concrete and Standard Specification for Air-Entraining Admixtures for Highway Concrete.

The proposed methods and specification are the recommendation of NCHRP Project 18-10 Staff at Construction Technology Laboratories Inc. The method has not been approved by NCHRP or any AASHTO Committees or formally accepted for the AASHTO Specifications.

PROPOSED STANDARD METHOD FOR AIR-ENTRAINING ADMIXTURE FOR HIGHWAY CONCRETE

1. SCOPE

1.1 This method covers the testing of materials proposed for use as air-entraining admixtures in the field.

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards:*

- M 6, Fine Aggregate for Portland Cement Concrete
- M 80, Coarse Aggregate for Portland Cement Concrete
- M 85, Portland Cement
- M 154, Air-Entraining Admixtures for Concrete
- T 2, Sampling of Aggregates
- T 22, Compressive Strength of Cylindrical Concrete Specimens
- T 27, Sieve Analysis of Fine and Coarse Aggregates
- T 97, Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)
- T 105, Chemical Analysis of Hydraulic Cement
- T 119, Slump of Hydraulic Cement Concrete
- T 126, Making and Curing Concrete Test Specimens in the Laboratory
- T 137, Air Content of Hydraulic Cement Mortar
- T 141, Sampling Freshly-Mixed Concrete
- T 152, Air Content of Freshly-Mixed Concrete by the Pressure Method
- T 158, Bleeding of Concrete
- T 160, Length Change of Hardened-Hydraulic Cement Mortar and Concrete
- T 161, Resistance of Concrete to Rapid Freezing and Thawing
- T 196, Air Content of Freshly-Mixed Concrete by the Volumetric Method
- T 197, Time of Setting of Concrete Mixtures by Penetration Resistance
- T 200, pH of Aqueous Solutions with the Glass Electrode

2.2. *ASTM Standards*:

- C 33, Standard Specification for Concrete Aggregates
- C 136, Test Method for Sieve Analysis of Fine and Coarse Aggregates
- C 670, Preparing Precision and Bias Statements for Test Methods for Construction Materials
- C 672, Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
- D 75, Practice for Sampling Aggregates
- D 1193, Specification for Reagent Water
- D 1429, Test Methods for Specific Gravity of Water and Brine
- E 100, Specification for ASTM Hydrometers

2.3. *ACI Standard:*

- ACI 211.1-91, Recommended Practice for Selecting Proportions for Normal, Heavy-Weight and Mass Concrete

3. SIGNIFICANCE AND USE

3.1. This test method is used to develop data for comparison with the requirements of the proposed specifications.

4. MATERIALS

4.1. *Cement*-The cement used in any series of tests shall be Type I or Type II cement conforming to M 85. The maximum alkali content of the cement shall be 0.60% as equivalent Na_2O.

4.2. Fly ash- ASTM Class F or C (ASTM C 618) with a maximum LOI of 3 to 4%.

4.2.1. *Aggregates*-The fine and coarse aggregates used in any series of tests shall come from single well-graded, sound materials that conform to the requirements of M 6 and M 80.

4.2.2. *Coarse Aggregate Grading*-The coarse aggregate shall meet the requirements for Size 57 of ASTM Specification C 33.

The coarse aggregate used for each set of reference concrete and comparable test admixture treated concrete shall be essentially the same.

5. CONCRETE MIX PROPORTIONS

5.1. Mix proportions vary based on the testing protocols. There are two application-based protocols (pavement and structural concretes). Each protocol consists of two different mix designs.

5.2 Protocol A Pavement Concrete

5.2.1. Mix A-1. Plain Concrete: The cement content shall be 565 lb/yd^3 and water-cement ratio of 0.44. Sand-aggregate ratio shall be adjusted to achiever 1 to 3 in. slump.

5.2.1.1. Mix A-2. Fly Ash Concrete: The cement content shall be 450 lb/yd^3 and fly ash content shall be 110 lb/yd^3. Water-cementitious ratio shall be 0.40. Sand-aggregate shall be adjusted to achieve 1 to 3 in. slump

5.3 Protocol B Structural Concrete

5.3.1 Mix B-1. Plain Concrete: The cement content shall be 605 lb/yd^3 and water-cement ratio of 0.39. High-range water reducer (HRWR) shall be used. HRWR dosages shall be adjusted to achieve slump of 4 to 7 in.

5.3.2 Mix B-2. Fly Ash Concrete: The cement content shall be 515 lb/yd^3 and fly ash content shall be 140 lb/yd^3. Water-cementitious ratio shall be 0.42. ASTM Type A water-reducer shall be used. The dosage of the water reducer shall be adjusted to achieve slump of 3 to 6 in.

5.4 Reference Concrete- The reference is non-air-entrained concrete. For each of the four protocol mixes, reference concrete shall be prepared using the materials with same mix design of the specific mix (two reference mixes for each protocol). For each of the reference concrete mix, Slump requirements shall be similar to that of the identical air-entrained mix. (e.g., for mix B-2, dosage of water-reducer in the reference concrete shall be adjusted to achieve slump of 3 to 6 in.)

5.5 The temperature of all concrete materials including the water shall 90 ± 5° F. This can be achieved by storing all materials in hot room or oven overnight prior to mixing.

5.6 The air content for each of the mixes shall be 6 ± 1.0%. Air-entraining admixture dosage shall be adjusted to achieve an air content of 6 ± 1.0%. The air content of the reference concrete shall be 2% or less.

Note: If the air content of the reference is more than 2%, the air content of the tested admixture concrete shall be adjusted to achieve a difference air content between the two concretes (air-entrained and non air-entrained) of 4 ± 0.5 percentage points.

5.7. *Conditions*-Concrete mixtures shall be prepared with the air-entraining admixture under test. The admixtures shall be added in the amounts necessary to produce the air content selected in accordance with Section 5.6 within a tolerance of 1.0 percent of the volume of concrete.

6. MIXING

6.1. For Protocol A, a drum mixer is preferred with mixing time of 2 minutes. For Protocol B, a pan-type mixer or drum mixer is used with 5 minutes of continuous mixing. For both protocols, sequence of charging of materials into the mixer starts by adding the aggregate and sand followed by 40% of the mixing water and mixed for 15 seconds. Air-entraining admixture is then added followed by the cementitious materials and remaining water. Air-entraining admixture is premixed with water or added in the mixer separately. Mix timing is started after adding the cement and water.

7. TESTS AND PROPERTIES OF FRESHLY MIXED CONCRETE

7.1. Test samples of freshly mixed concrete from at least three separate batches for each condition of concrete in accordance with the following methods. The minimum number of tests shall be as prescribed in Table 1.

7.1.1. *Slump*-T 119.

7.1.2. *Air Content*-T 152. When lightweight aggregates are used under the provisions of Section 4.4, use T 196.

7.1.3. *Unit Weight*-T 121

7.1.4. Time *of Setting-T* 197, except the temperature at which the time of setting specimens are stored during the test period shall be 23.0 ± 1.7°C (73.4 ± 3°F).

Table 1-Types and Minimum Number of Specimens and Tests

Test	Number of Types of Specimen	Number of Test Ages	Minimum Number of Specimens
Slump	1	1	*
Air content	1	1	*
Time of setting	1	1	3
Unit weight	1	1	3
Compressive strength	1	2	6
Flexural strength	1	2	6
Split tensile strength	1	2	6
Air Void Analysis	1	1	3
Freezing and thawing**	1	1	3
Deicer scaling test**	1	1	3

* Determined on each batch of concrete
** If required

8. PREPARATION OF TEST SPECIMENS

8.1. Specimens for tests of hardened concrete, representing each protocol test and age of test shall be made from at least three separate batches. The minimum number of specimens shall be as prescribed in Table 1. On a given day at least one specimen shall be made for each test and age of test from each condition of concrete.

8.2. *Manifestly Faulty Specimens*-Each group of specimens representing a given test or a given age of test, including tests of freshly mixed concrete, shall be examined visually before or during the test, or both, whichever is appropriate. Discard any specimens found to be manifestly faulty by such examination without testing. Visually examine all specimens representing a given test at a given age after testing, and should any specimen be found to be manifestly faulty, the test results thereof shall be disregarded. Should more than one specimen representing a given test at a given age be found manifestly faulty, either before or after testing, the entire test shall be disregarded and repeated. The test result reported shall be the average of the individual test results of the specimens tested or, in the event that one specimen or one result has been discarded, it shall be the average of the test results of the remaining specimens.

9. TEST SPECIMENS OF HARDENED CONCRETE

9.1 *Number of Specimens*-Make three or more test specimens for each type of test and age of test specified in Table 1.

9.2. *Types of Specimens*-Prepare specimens made from concrete with the tested air-entraining admixture and reference concrete under test in accordance with the following:

9.2.1. Compressive Strength-Prepare specimens in accordance with T126. Exposed surfaces of specimens shall be covered by wet burlap and polyethylene sheeting to prevent evaporation of water from the concrete during the first 18-24 hours after casting. The specimens shall be then demolded and placed in a moist room maintained at 73.4 ± 3°F) until the time of test.

9.2.2. *Flexural Strength*- Prepare specimens in accordance with T126. Exposed surfaces of specimens shall be covered by wet burlap and polyethylene sheeting to prevent evaporation of water from the concrete during the first 18-24 hours after casting. The specimens shall be then demolded and placed in a moist room maintained at 73.4 ± 3°F) until the time of test.

9.2.3. *Split Tensile Strength*- Prepare specimens in accordance with T126. Exposed surfaces of specimens shall be covered by wet burlap and polyethylene sheeting to prevent evaporation of water from the concrete during the first 18-24 hours after casting. The specimens shall be then demolded and placed in a moist room maintained at 73.4 ± 3°F) until the time of test.

9.2.4. *Air-Void Analysis*- Prepare specimens in accordance with T126. Exposed surfaces of specimens shall be covered by wet burlap and polyethylene sheeting to prevent evaporation of water from the concrete during the first 18-24 hours after casting. The specimens shall be then demolded and placed in a moist room maintained at 73.4 ± 3°F) until the time of test.

9.2.5. *Resistance to Freezing and Thawing*-Test specimens shall consist of prisms made and cured in accordance with the applicable requirement of T 126. Test specimen dimensions shall be as required by T 161. Make one set of specimens from the concrete mixture containing the air-entraining admixture under test and from the reference concrete mixture, the air content of each mixture being as specified in Section 5.2.

9.2.6. *Deicer Scaling Test*- Prepare specimens in accordance with T126. Exposed surfaces of specimens shall be covered by wet burlap and polyethylene sheeting to prevent evaporation of water from the concrete during the first 18-24 hours after casting. The specimens shall be then demolded and placed in a moist room maintained at 73.4 ± 3°F) for 28 days.

10. TESTS ON HARDENED CONCRETE

10.1. Test specimens of hardened concrete in accordance with the following methods:

10.1.1. *Compressive Strength*-T 22. Test specimens at the ages of 7 and 28 days. Calculate the compressive strength of the concrete containing the admixture under test as a percentage of the compressive strength of the reference concrete as follows:

10.1.1.1. Divide the average compressive strength of the specimens made from the concrete containing the admixture under test at a given age of test by the average compressive strength of the specimens made from the reference concrete at the same age of test and multiply the quotient by 100.

10.1.2. *Flexural Strength*-T 97. Test specimens at the ages of 7 and 28. Calculate the flexural strength of the concrete containing the admixture under test as a percentage of the flexural strength of the reference concrete as follows:

10.1.2.1. Divide the average flexural strength of the specimens made from the concrete containing the admixture under test at a given age of test by the average flexural strength of the specimens made from the reference concrete at the same age of test, and multiply the quotient by 100.

10.1.3 *Split Tensile Strength*-T 198. Test specimens at the ages of 7 and 28 days. Calculate the split tensile strength of the concrete containing the admixture under test as a percentage of the split tensile strength of the reference concrete as follows:

10.1.3.1. Divide the average split tensile strength of the specimens made from the concrete containing the admixture under test at a given age of test by the average split tensile strength of the specimens made from the reference concrete at the same age of test, and multiply the quotient by 100.

10.1.4. *Resistance to Freezing and Thawing*-Procedure A of T 161. Place specimens under test at the age of 14 days.

10.1.5. *Deicer Scaling Test ASTM C 672*. Test the specimens at the age of 28 days (9.2.6)

PROPOSED STANDARD SPECIFICATION FOR AIR-ENTRAINING ADMIXTURES FOR CONCRETE

1. Scope

1.1 This specification covers materials proposed for use as air-entraining admixtures to be added to concrete mixtures in the field.

2. Referenced Documents

2.1 *AASHTO Standards*

- T 127 Practice for Sampling and the Amount of Testing of Hydraulic Cement
- T 137 Test Method for Air Content of Hydraulic Cement Mortar
- *Method for Air-Entraining Admixture for Concrete (Proposed)*

3. Terminology

3.1 *Description of Term Specific to This Standard:*

3.1.1 A*ir-entraining admixture – for the purpose of this specification*, a material that is used as an ingredient of concrete, added to the batch immediately before or during its mixing, for the purpose of entraining air.

4. General Requirements

4.1 At the request of the purchaser, the manufacturer shall state in writing that the air-entraining admixture supplied for use in the work is essentially identical in concentration, composition, and performance to the air-entraining admixture tested under this specification.

NOTE 1 – It is recommended that, whenever practicable, tests with the air-entraining admixture be made using all of the ingredients of the concrete proposed for the specific work, because the effect produced by the air-entraining admixture may vary with the properties of the other ingredients of the concrete.

4.2 Requirements for establishing compositional or chemical equivalence of a subsequent lot relative to a previous lot that was subjected to quality tests and found to comply with the requirements of 5.1 may be determined by agreement between the purchaser and the manufacturer. At the request of the purchaser, the manufacturer shall recommend appropriate test procedures, such as infrared spectrophotometry (I.R.), pH value and solids content, for establishing the equivalence of materials for different lots or different portions of the same lot.

NOTE 2 – Ultraviolet light absorption (U.V.) of solutions and infrared spectroscopy of dried residues have been found to be valuable for these purposes. The specific procedures to be employed and the criteria to establish equivalence should be stipulated with due regard to the composition and properties of the sample.

4.3 At the request of the purchaser, the manufacturer shall state in writing the chloride content of the air-entraining admixture and whether or not chloride was added during its manufacture.

NOTE 3 – Admixtures that contain chlorides may accelerate corrosion of embedded metals.

5. Optional Uniformity Requirements

5.1 A series of two or more samples from a manufacturing lot will be considered sufficiently uniform to be properly composited into a single sample for quality testing provided they do not differ more than the amounts indicated in 5.4.

5.2 A single sample from a subsequent lot or a composite sample prepared by combining two or more samples from a subsequent lot that do not differ by more than the amounts indicated in 5.4, may be considered sufficiently similar to a sample from a previous lot that was subjected to quality tests and found to comply with the requirements of 6.1, so that it may be regarded as also in compliance with these requirements, provided it does not differ from the sample so tested by any additional optional, appropriate tests, such as I.R. and U.V.

5.3 Determinations of uniformity shall be made in accordance with the procedures given in the sections “Check Tests for Uniformity” and “Procedure for Residue by Oven Drying” of Test Method ASTM C233.

5.4 Allowable differences in results of uniformity determinations shall not exceed the following amounts:

5.4.1 The manufacturer shall provide an acceptable range of pH not to exceed a range of 2.0. The pH of samples tested shall fall within this range.

5.4.2 The air content in percent of Test Method ASTM C185 mortars prepared from successive lots shall not differ by more than 2.0 from that for the acceptance sample.

5.4.3 The manufacturer shall provide acceptable limits of residue content not to exceed ± 12% of the midpoint of the limits. The residue content of samples tested shall fall within these limits (Note 4).

NOTE 4 – As an example, an admixture may commonly be produced with residue content ranging from 5.0 to 6.5%. The manufacturer would provide acceptable limits of 5.06 to 6.44%, representing ± 12% of the midpoint of the limits which is 5.75%.

6. Performance Requirements

6.1 The air-entraining admixture shall conform to the requirements in Table 1. Determination of properties listed in this Table shall be made in accordance with the “Methods for Air-Entraining Admixture for Concrete (proposed)”

Table 1 Physical Requirements

Time of setting, allowable deviation from control (minutes)	± 75 minutes
Compressive strength (7 and 28 days), minimum % of Control	75
Flexural strength (7 and 28 days), minimum % of Control	75
Split tensile Strength (7 and 28 days), minimum % of Control	75
Average spacing factor range (in.)	0.002 to 0.008
Durability factor, (%) more than (Freeze/thaw test, 300 cycles)*	80
Scaling rate (Deicer scaling test, 50 cycles)*	0 to 1

** Applicable only when the compressive strength is more than 75% and spacing factor did not conform to the requirements.*

6.2 The air-entraining admixture can be certified for one or both proposed application protocols. Details on the protocols are presented in the Test Method for Air-Entraining Admixture for Concrete (proposed).

6.3 In order for an admixture to be certified for pavement applications, concrete prepared with the admixture following the two mix designs listed under Protocol A shall confirm the requirements listed in 6.1.

6.4 In order for an admixture to be certified for Structural applications, concrete prepared with the admixture following the two mix designs listed under Protocol B shall confirm the requirements listed in 6.1.

7. Sampling

7.1 Opportunity shall be provided the purchaser for careful sampling and inspection, either at the point of manufacture or at the site of the work, as may be specified by the purchaser.

7.2 Samples shall be either ‘grab’ or ‘composite’ samples, as specified or required by this specification. A grab sample is one obtained in a single operation. A composite sample is one obtained by combining three or more grab samples.

7.3 For the purpose of this specification, it is recognized that samples will be taken for the two following reasons:

7.3.1 *Quality Tests* – A sample taken for the purpose of evaluating the quality of a source or lot of admixture will be required to meet all the applicable requirements of this specification. Samples used to determine conformance with the requirements of this specification shall be composites of grab samples taken from sufficient locations to ensure that the composite sample will be representative of the lot.

7.3.2 *Uniformity Tests* – A sample taken for the purpose of evaluating the uniformity of a single lot or of different lots from the same source will generally be subjected to a limited number of tests as the result of agreement between the purchaser and manufacturer (see Section 4). Such samples shall be composite samples from individual lots when different lots from the same source are being compared. When the uniformity of a single lot is being determined, grab samples shall be used.

7.4 *Liquid Air-Entraining Admixture* – Liquid admixtures shall be agitated thoroughly immediately prior to sampling. Grab samples taken for quality or uniformity tests shall represent not more than 9500 L (2500 gal) of admixture and shall have a volume of at least 1 L (1 qt). A minimum of three grab samples shall be taken. Composite samples shall be prepared by thoroughly mixing the grab samples selected and the resultant mixture sampled to provide at least 4 L (1 gal) for quality tests. Grab samples shall be taken from different locations well distributed throughout the quantity to be represented.

7.4.1 Admixtures in bulk storage tanks shall be sampled equally from the upper, intermediate, and lower levels by means of drain cocks in the sides of the tanks or a weighted sampling bottle fitted with a stopper that can be removed after the bottle is lowered to the desired depth.

7.4.2 Samples shall be packaged in impermeable, airtight containers that are resistant to attack by the admixture.

7.5 *Nonliquid Air-Entraining Admixture* – Grab samples taken for quality or uniformity tests shall represent not more than 2 metric tons (2 tons) of admixture and shall have a mass of at least 1 kg (2 lb). A minimum of four grab samples shall be taken. Composite samples shall be prepared by thoroughly mixing the grab samples selected and the resultant mixture sampled to provide at least 2.5 kg (5 lb) for the composite sample. Grab samples shall be taken from different locations well distributed throughout the quantity to be represented.

7.5.1 Samples of packaged admixtures shall be obtained by means of a tube sampler as described in Practice ASTM C183.

7.5.2 Samples shall be packaged in moisture-proof, airtight containers.

7.6 Samples shall be thoroughly mixed before testing to assure uniformity. When recommended by the manufacturer, the entire sample of a nonliquid admixture shall be dissolved in water prior to testing.

8. Test Methods

8.1 Determine the properties enumerated in Section 7 in accordance with Test Method ASTM C233. It is recommended that, whenever practicable, tests be made in accordance with the section on Materials for Tests for Specific Uses in Test Method ASTM C233, using the cement proposed for the specific work.

9. Rejection

9.1 The air-entraining admixture shall be rejected if the purchaser desires when it fails to meet any of the applicable requirements of this specification.

9.2 After completion of tests, an admixture stored at the point of manufacture for more than 6 months prior to shipment, or an admixture in local storage in the hands of a seller for more than 6 months, shall be retested before use when requested by the purchaser. It shall be rejected, if the purchaser desires, when it fails to meet any of the applicable requirements of this specification.

9.3 Packages or containers varying more than 5% from the specified weight or volume shall be rejected if the purchaser desires. If the average weight of volume of 50 packages or containers taken at random is less than that specified, the entire shipment shall be rejected if the purchaser desires.

10. Packaging and Marking

10.1 The proprietary name of the air-entraining admixture and the net quantity in pounds or gallons (kilograms or liters) shall be plainly indicated on the packages or containers in which the admixture is delivered. Similar information shall be provided in the shipping advices accompanying packaged or bulk shipments of admixtures.

11. Keywords

11.1 air content; air entraining admixtures; cement concrete pH; residue; specific gravity

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