

Freeze-Thaw Resistance of Concrete With Marginal Air Content

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Foreword

In 1994, the Strategic Highway Research Program (SHRP) published results from a research study on freezing and thawing of concrete, in which a number of concretes containing 2.5 to 3 percent total air performed adequately in freeze-thaw tests. These results seemed surprising in light of common minimum specification limits of 4 to 6 percent. The work reported here began as a followup study to the SHRP work, an attempt to corroborate the earlier results.

This report describes a laboratory investigation of the behavior of concrete with “marginal” air void systems, in which the air content and other air void system parameters do not consistently meet commonly accepted thresholds for freeze-thaw durability. Some of the concretes did provide good durability—but others did not. The type of air-entraining admixture played a major role in performance. In addition to measuring air-void parameters by the linear traverse technique, special programmed equipment at Turner-Fairbank Highway Research Center (TFHRC) was used to measure and record each individual chord length across the air voids traversed. The air-void chord length distributions are presented and analyzed in this report. The research that is the subject of this paper was funded by the Federal Highway Administration (FHWA) and conducted entirely at FHWA’s TFHRC.

The results of this research will be of interest to engineers involved in the construction and acceptance of both concrete pavements and structures built in climates with below-freezing temperatures. The report will also be of interest to concrete researchers studying the factors affecting concrete durability.

Gary L. Henderson
Director, Office of Infrastructure
Research and Development

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16. Abstract Freeze-thaw resistance is a key durability factor for concrete pavements. Recommendations for the air void system parameters are normally: 6 ± 1 percent total air, and spacing factor less than 0.20 millimeters. However, it was observed that some concretes that did not possess these commonly accepted thresholds presented good freeze-thaw resistance in laboratory studies. This study evaluated the freeze-thaw resistance of several "marginal" air void mixes, with two different types of air-entraining admixtures (AEA)—a Vinsol resin and a synthetic admixture. This study used rapid cycles of freezing and thawing in plain water, in the absence of deicing salts. For the specific materials and concrete mixture proportions used in this project, the marginal air mixes (concretes with fresh air contents of 3.5 percent or higher) presented an adequate freeze-thaw performance when Vinsol resin based air-entraining admixture was used. The synthetic admixture used in this study did not show the same good performance as the Vinsol resin admixture.			
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SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
AREA				
in ²	square inches	645.2	square millimeters	mm ²
ft ²	square feet	0.093	square meters	m ²
yd ²	square yard	0.836	square meters	m ²
ac	acres	0.405	hectares	ha
mi ²	square miles	2.59	square kilometers	km ²
VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft ³	cubic feet	0.028	cubic meters	m ³
yd ³	cubic yards	0.765	cubic meters	m ³
NOTE: volumes greater than 1000 L shall be shown in m ³				
MASS				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
TEMPERATURE (exact degrees)				
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
ILLUMINATION				
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m ²	cd/m ²
FORCE and PRESSURE or STRESS				
lbf	poundforce	4.45	newtons	N
lbf/in ²	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
AREA				
mm ²	square millimeters	0.0016	square inches	in ²
m ²	square meters	10.764	square feet	ft ²
m ²	square meters	1.195	square yards	yd ²
ha	hectares	2.47	acres	ac
km ²	square kilometers	0.386	square miles	mi ²
VOLUME				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m ³	cubic meters	35.314	cubic feet	ft ³
m ³	cubic meters	1.307	cubic yards	yd ³
MASS				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact degrees)				
°C	Celsius	1.8C+32	Fahrenheit	°F
ILLUMINATION				
lx	lux	0.0929	foot-candles	fc
cd/m ²	candela/m ²	0.2919	foot-Lamberts	fl
FORCE and PRESSURE or STRESS				
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in ²

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.
(Revised March 2003)

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CHAPTER 1: INTRODUCTION

In 2004, the value of concrete production for highway construction and maintenance has been estimated to be more than 9 billion dollars. Nevertheless, 34 percent of the United States' major roads are still in poor or mediocre condition.⁽¹⁾ Although in cold climate regions, the most persistent problem is the concrete deterioration caused by freezing and thawing;⁽²⁾ it is an issue not completely resolved.

Since the late 1930s, air-entraining cements and admixtures have been used to impart freeze-thaw resistance to concrete. Because air detracts from some other concrete properties (particularly strength), the goal of air entrainment is to provide sufficient air in the concrete to ensure freeze-thaw resistance, but no more than is required for that purpose. In non-freeze-thaw exposures some air is often used for economy or improved workability.

Research from the 1940s through 1960s by Gonnerman,⁽³⁾ Powers,⁽⁴⁾ Klieger,⁽⁵⁾ Cordon and Merrill,⁽⁶⁾ and others sought to establish air requirements for frost-resistant concrete. These initial research efforts concluded that at least 3 percent of air, by volume, in the fresh concrete was necessary to protect concrete from freezing and thawing (see figure 1, for example). Further research indicated that, since the air voids protect the paste, the required air content depended on the paste content, which is largely a function of aggregate size and gradation and of minimum cement content requirements. Therefore, 3 percent air per unit of concrete volume may be sufficient for a lean mix but not for a richer mix.

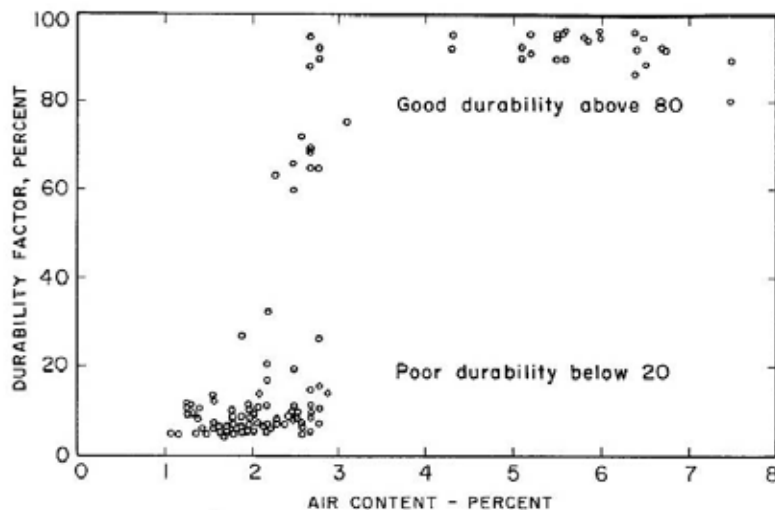


Figure 1. Graph. Freeze-thaw durability factor for different levels of total air contents.⁽⁶⁾

The air bubbles can be classified as entrapped or entrained. Entrapped air voids are relatively large, typically 1 to 10 millimeters (mm) or more in size. Air-entrained concrete contains much smaller voids that range from 0.01 mm to 1 mm in diameter⁽⁷⁾ and that are stabilized in fresh cement paste through the action of the air-entraining admixture (AEA) (see chapter 2). The amount of entrapped air in concrete is also a function of aggregate size and gradation (especially fine aggregate gradation). Entrapped air usually comprises 1 to 2 percent of the concrete volume,

but in some cases can comprise as much as 3 or 4 percent.⁽⁵⁾ When air-entraining admixture or air-entraining cement is used to produce air-entrained concrete, the air void structure is usually smaller, with fewer larger air voids.

The American Concrete Institute (ACI) 211.1 *Standard Practice for Selecting Proportions for Concrete*⁽⁸⁾ guidelines for air content reflect the factors discussed above, and over time certain recommendations (ACI 201.2R⁽⁹⁾), specifications (American Society for Testing and Materials (ASTM) C 94⁽¹⁰⁾ and ACI 301⁽¹¹⁾), and codes (ACI 318⁽¹²⁾) regarding air content and other air void system parameters have evolved. Most State departments of transportation (DOTs) where concrete is exposed to significant freezing and thawing specify target air contents of 5 to 7 percent in the fresh concrete for aggregate maximum sizes of 50 mm down to 12.5 mm (often with tolerances of ± 2 percent).⁽¹³⁾ Usually this specification is based on results of fresh concrete testing by either ASTM C231⁽¹⁴⁾ and American Association of State Highway and Transportation Officials (AASHTO) T 152⁽¹⁵⁾ (pressure method) or ASTM C173⁽¹⁶⁾ and AASHTO T196⁽¹⁷⁾ (volumetric method). Unfortunately, these methods provide only a measurement of the total air volume, not the size or distribution of the air voids. Furthermore, these tests are often performed before the completion of construction operations (such as placing, consolidating, and finishing) that can alter the air void system. Therefore, the actual in-place hardened air content and other air void system parameters may differ significantly from those in the fresh concrete.

Another commonly accepted hardened concrete parameter for freeze-thaw resistance is an air-void spacing factor (ASTM C 457⁽¹⁸⁾) of 0.200 mm or less (spacing factor is defined and discussed in chapter 2). A number of early research studies reported that a spacing factor of approximately 0.250 mm or less signified adequate freeze-thaw resistance. Although Powers first advocated void spacing as a means of specifying air entrained concrete in the 1950s,⁽¹⁹⁾ few States have actually used a spacing factor specification. Until the recent advent of the Air Void Analyzer™ (AVA), the only means of determining the spacing factor was the labor-intensive ASTM C457,⁽¹⁸⁾ which involves microscopical examination of a polished specimen of hardened concrete. The AVA method estimates the spacing factor from measurements on fresh concrete, which makes it a faster and more practical quality control test than ASTM C457.⁽¹⁸⁾ Recently, some States have begun to specify spacing factor based on the AVA measurement. However, since the AVA and ASTM C457⁽¹⁸⁾ methods are different, it is not clear whether a limit of 0.200 mm for the spacing factor determined by the AVA is appropriate for assuring freeze-thaw durability.

It is also very important to highlight that the current recommendations were established based mostly on data of concretes containing neutralized Vinsol® resin as an air-entraining admixture (AEA). On the other hand, the scarcity of Vinsol resin admixture is responsible for the increasing use of synthetic admixtures. Nevertheless, an extensive comparison of the freeze-thaw performances of Vinsol and synthetic air-entrained concretes with marginal air content has not yet been performed.

In 1994, the Strategic Highway Research Program (SHRP) published results from a research study on freezing and thawing of concrete, in which a number of concretes containing 2.5 to 3 percent total air performed adequately in freeze-thaw tests. These results seemed surprising in light of common minimum specification limits of 4 to 6 percent. The work reported here began as a followup study to the SHRP work, an attempt to corroborate the earlier results.

This report describes a laboratory investigation of the behavior of concrete with “marginal” air void systems, in which the air content and other air void system parameters do not meet commonly accepted thresholds for freeze-thaw durability.

The effect of deicing agents on concrete durability will not be covered in this document. Only evaluations using freezing and thawing in plain water were used in this study (AASHTO T 161⁽²⁰⁾ and ASTM C 666, Procedure A,⁽²¹⁾ using freezing in water and thawing in water).

OBJECTIVES

The objectives of this study are as follows:

- To evaluate the water-cement (w/c) ratio influence on the freeze-thaw resistance of the mixes with Vinsol resin air-entraining admixture (chapter 3).
- To compare the performance of the mixes with Vinsol resin and synthetic air-entraining admixtures (chapter 4).

ORGANIZATION AND SCOPE

The report contains five chapters. Chapter 1, the introduction, defines the objectives and scope of the study. Chapter 2 provides background information on freeze-thaw behavior of concrete, air entrainment, and freeze-thaw testing. Chapters 3 and 4 describe laboratory experiments performed as part of this research and discuss the experimental results. Chapter 5 provides a summary of findings, conclusions, and future research needs.

There are four appendices to the report. Appendix A contains the properties of the materials used in the project. Appendix B contains the complete test data for the experiments described in chapters 3 and 4 of the report. Appendix C presents the analyses of variance of the test results. Appendix D describes the equipment and method used to obtain time-domain data from ASTM C 215⁽²²⁾ (impact method) testing of freeze-thaw test specimens.

CHAPTER 2: BACKGROUND

CONCRETE MICROSTRUCTURE

Hardened concrete is composed of coarse and fine aggregate particles embedded in a matrix of hardened cement paste. The hardened paste, which comprises approximately 25 to 30 percent of the concrete volume, consists primarily of calcium silicate hydrate (CSH) gel, calcium hydroxide (CH), calcium sulfoaluminate and capillary pore space (space originally filled with water in excess of that required for hydration of the cement). The CSH gel is itself porous, with an intrinsic porosity of approximately 28 percent.⁽²³⁾ A schematic diagram of concrete paste microstructure at the boundary of an air void is shown in figure 2. The solid portion of the hydrated cement gel is depicted as small black spheres. The interstitial spaces between the spheres are the gel pores. The capillary pores are denoted with a “C.”

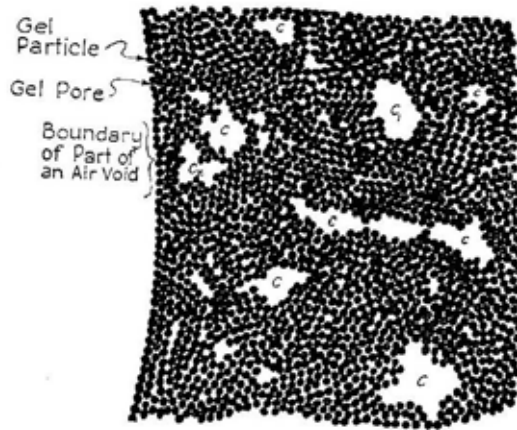


Figure 2. Illustration. Concrete paste microstructure.⁽²⁴⁾

The gel pore diameters range in size from 5×10^{-7} to 25×10^{-7} mm. The temperature at which water freezes is a function of the pore size; the gel pores are so small that water cannot freeze inside them at temperatures above -78 °C.⁽²⁴⁾ The capillary pores are considerably larger and vary in size, typically ranging from 10×10^{-6} to 50×10^{-6} mm in well hydrated pastes of low water-cement ratio, whereas in pastes of high water-cement ratio at early ages, size may vary from 3×10^{-3} to 5×10^{-3} mm. Figure 2 also shows the boundary of an air void, indicating that air voids are usually several orders of magnitude larger than capillaries and gel pores.

ORIGIN OF AIR IN CONCRETE

All concretes contain natural or entrapped air that is incorporated into concrete during mixing operations.⁽²⁵⁾ It is relatively large, often irregularly shaped voids, typically 1 to 10 mm or more in size. Entrapped air can comprise about 1 to 3 percent of the volume in concrete. Air-entrained concrete also contains much smaller, spherical air voids ranging from 0.01 mm to 1 mm in diameter. A typical average size of entrained air voids is about 0.10 mm.⁽⁷⁾ Entrained air is incorporated into the concrete in the same way as entrapped air (mixing); however, entrained air

is stabilized as small bubbles in the fresh cement paste through the action of AEA. A properly air-entrained concrete may contain 300,000 air voids per cubic centimeter of paste.

Air-Entraining Admixtures (AEA)

Air-entraining agents are admixtures used to stabilize the air entrapped during the mixing in the form of very small, discrete bubbles known as entrained air.⁽²⁶⁾

The air-entraining admixtures are surfactants that possess a hydrocarbon chain terminating in a hydrophilic polar group. The other end of the chain is hydrophobic and does not mix well with water. Not all organic materials are suitable for use as air-entraining agents. One of the first used and most effective is Vinsol resin, which is extracted from pinewood.⁽²⁶⁾ The earliest work on freeze-thaw resistance, as well as recommendations for air void systems, were based on research on concretes containing Vinsol resin admixtures.

Currently, a large number of admixtures are based on synthetic chemicals. They can be divided into anionic and nonionic. The first, anionic group is composed of alkylarylsulfonates and alkylsulfates such as sodium dodecyl benzene sulfonate. An example of a nonionic agent is nonylphenoethoxylate.

FREEZE-THAW DAMAGE MECHANISMS

If the aggregates used in concrete are frost-resistant, the freeze-thaw resistance of the cement paste determines the overall resistance of the concrete to freezing and thawing, as described below. If the aggregate is susceptible to freeze-thaw damage, it can contribute to concrete deterioration. Since most aggregates are freeze-thaw resistant and the aggregates used in this research were durable, this mechanism is not covered in this research.

Several theories have been proposed to explain concrete damage due to freezing and thawing.

Critical Saturation

The critical saturation theory, proposed by Powers,⁽²⁷⁾ states that concrete will only suffer damage from freezing when the capillaries in the cement paste are more than 91.7 percent full of water. This theory is based on the fact that water expands in volume by approximately 9 percent when it freezes. If the capillary pores are saturated with water and the water freezes, stresses will be generated. If the pores are only partially filled, the expansion resulting from ice formation may be accommodated. Critical saturation can occur in localized zones within the cement paste.

Hydraulic Pressure

Powers⁽⁴⁾ also put forth the hydraulic pressure theory, which states that damage from freezing is caused by a buildup of hydraulic pressure from the resistance to flow of unfrozen water in cement paste capillaries. As water freezes, if the cement paste does not expand to accommodate it, unfrozen water will be pushed through the capillary pores, away from the sites of freezing, like water through a pipe. Powers applied Darcy's Law to illustrate the factors influencing the pressure generated by this flow:

$$\Delta h = \frac{\eta}{k} Q \frac{l}{A}$$

Figure 3. Equation. Pressure gradient.

In the above equation, Δh is the pressure gradient, η is the fluid viscosity, k is the permeability, Q is the flow rate, l is the length of the flow path, and A is the flow area.. The pressure generated increases as fluid viscosity, flow rate, or length increase, and as permeability or area decrease. The fluid viscosity (viscosity of the pore solution (water with dissolved ions)) may not vary much. Flow rate is related to the rate of freezing (the faster the ice forms, the faster water is pushed through the capillaries). Permeability and flow area (i.e., size of the capillary pore) depend on the particular cement paste microstructure. For a given cement paste, pore fluid viscosity, and flow rate, a maximum length of flow can be calculated by setting the pressure gradient equal to the tensile strength of the cement paste (i.e., the maximum pressure for which damage to the paste will not occur):

$$l = \Delta h \frac{k}{\eta} \frac{A}{Q}$$

Figure 4. Equation. Spacing factor.

Powers called this distance the theoretical permissible spacing between bubbles and estimated its magnitude to be on the order of 0.20 mm,⁽⁴⁾ based on Vinsol resin admixtures.

Ice Accretion and Osmotic Pressure

Powers and Helmuth⁽²⁴⁾ proposed the ice accretion/osmotic pressure theory to explain experimental results that were inconsistent with the hydraulic pressure theory. The osmotic pressure theory stated that, during freezing, water moves from the gel pores to the capillary pores according to the laws of thermodynamics (diffusion from high to low free energy) and the theory of osmosis (diffusion along concentration gradients).

As stated before, the freezing temperature of the water depends on the size of the pore. Gel pores are so small that water cannot freeze in them at temperatures above -78 °C. Water, at temperatures below 0 °C, has a higher free energy than ice; therefore, water will flow from gel to capillaries along a free energy gradient in order to freeze. If sufficient water flows to capillaries and freezes, the capillary will become full and pressure will develop. This pressure increases the free energy of the ice (or ice and water) in the capillary. Water will continue to flow to the capillary until the free energy of the ice and water in the capillary equals the free energy of water in the gel pores.

Water flow along ion concentration gradients can also occur during freezing. The water in capillary pores is not pure water but a solution of various ions dissolved in water. Ice, on the other hand, is pure water. Therefore, when ice forms in a capillary, the concentration of the remaining unfrozen pore solution increases, thus creating a concentration gradient. Even if the capillary is full of ice and water, water will flow from the gel (less concentrated) to the capillary (more concentrated) to equalize the pore solution concentration. This osmotic movement of water generates pressure.

ROLE OF AIR VOIDS

The theories of damage due to freezing and thawing identify stresses due to excessive pressure buildup as the cause of damage.

Under the ice accretion theory, water and ice in a capillary pore has a higher free energy than water or ice in an air void because capillaries are sufficiently small to inhibit the normal growth of ice crystals, whereas air voids (even the smallest ones) are large enough for ice crystals to form normally. Therefore, if air voids are present, water will diffuse from both the gel and capillaries to the air voids. Instead of filling the capillaries and generating pressure, water flows to the air voids, where (unless the concrete is completely saturated with water) ample space should be available to accommodate ice formation without pressure buildup. Air voids provide a similar protective function in the case of osmotic pressure.

In either case, air voids act as pressure relief sites, with each air void protecting a zone of hardened cement paste surrounding it. Figure 5 illustrates (in two dimensions) air voids protecting a zone (or shell) of paste. The limit of the protective shell is the maximum distance from an air void in which excessive pressure (i.e., that exceeding the tensile strength of the concrete) will not be generated. Thus, the goal of air entrainment is to provide a sufficient number of well-distributed air voids in the cement paste to ensure that most or all of the paste is within the required distance of an air void.

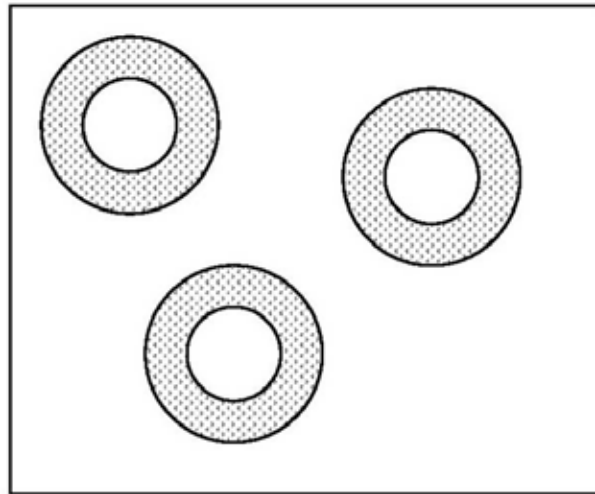


Figure 5. Illustration. The darker area shows the air void's protection zone of concrete.

AIR VOID PARAMETERS

The air void system in concrete can be described using several characteristics, or parameters, such as volume, number of bubbles, bubble size distribution, and spatial distribution within the paste. Air volume is the most commonly specified parameter; however, air volume alone is not a sufficient determinant of freeze-thaw resistance.

In theory, the volume of space needed for the expansion of ice formation is quite small. In a concrete containing 30 percent paste, and assuming 40 percent capillary porosity in the paste, only 1.3 percent total air volume is required to accommodate the expansion if the concrete is fully saturated. Even non-air-entrained concretes routinely contain this much air. However, the theories of freeze-thaw damage described previously illustrate that it is not the total volume of air, but rather the presence of a sufficient number of well-distributed air voids, that is critical. These characteristics are most commonly described in terms of specific surface and spacing factor.

Specific surface, the ratio of total bubble surface area to total air volume, normally expressed in mm^2/mm^3 or $\text{inches}^2/\text{inches}^3$, reflects the relative number and sizes of the air voids. For a given volume of air, a greater number of smaller air voids results in a higher specific surface area. Figure 6 shows the same volume of air as figure 5 in smaller voids. These figures indicate that for a given volume of air, smaller air voids provide more protection than larger voids, as the number of bubbles will be higher and the distance between them will be less. Specific surface, then, is an indicator of air void system effectiveness.

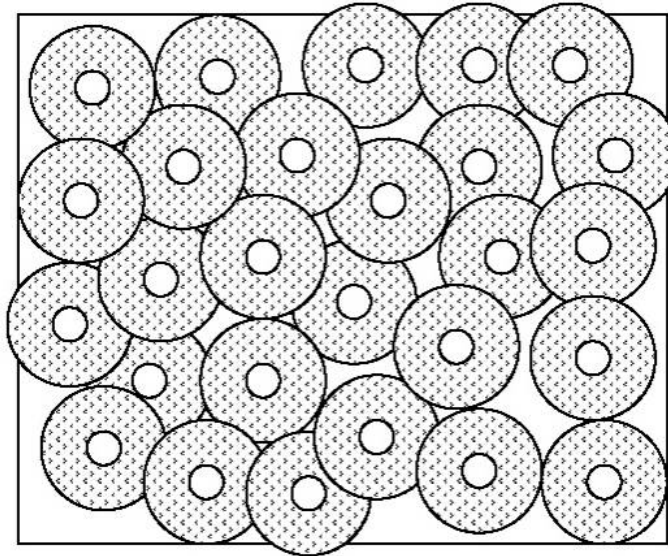


Figure 6. Illustration. Smaller air voids have higher specific surface and a greater number of bubbles than larger air voids, and offer more protection.

The spacing factor⁽⁴⁾ is an empirical quantity intended to represent the maximum distance that water would have to travel in the cement paste to reach an air void. If this distance is less than the critical maximum distance at which excessive stresses develop, the concrete should be adequately protected. The spacing factor was derived from a hypothetical air void system consisting of single-sized air voids arranged in a cubic lattice. In reality, voids are multisized and distributed randomly through the cement paste. Therefore, a distribution of spacings (distances from different points in the paste to the nearest air void) exists.⁽²⁸⁾

Nevertheless, much research has shown a relationship between spacing factor and concrete freeze-thaw durability (Klieger studies), at least for concretes containing admixtures available at the time of the research (Vinsol resin). Specific surface and spacing factor can be determined from ASTM C457⁽¹⁸⁾ measurements.

Often-quoted rules of thumb for these parameters are: 6 ± 1 percent air, specific surface $\geq 24 \text{ mm}^2/\text{mm}^3$, and spacing factor $\leq 0.20 \text{ mm}$. However, air content and specific surface cannot be viewed as independent. A specific surface of $24 \text{ mm}^2/\text{mm}^3$ at an air volume of 6 percent may be adequate, but a specific surface of $24 \text{ mm}^2/\text{mm}^3$ for an air volume of 4 percent may not be adequate. This concept is illustrated in figure 7, which shows a concrete volume containing two-thirds the volume of the air of figure 6 in identically sized voids (and therefore, with identical specific surface). Even with the voids spaced so their protected shells do not overlap, the unprotected area is significantly greater at the lower air void content.

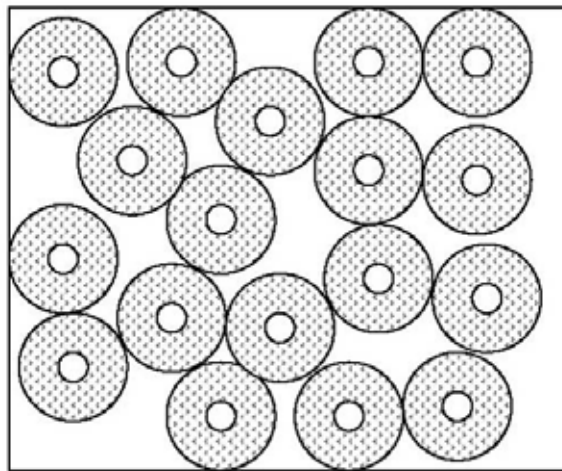


Figure 7. Illustration. Protection zone for a lower air void content.

Because air volume and specific surface must be considered in tandem, total bubble surface area (the product of air volume and specific surface) has been proposed as an appropriate parameter.⁽²⁹⁾

In practice, air content is usually the only parameter that is specified. That is because until the recent advent of AVA, which estimates the spacing factor from measurements on fresh concrete, the only means of determining the spacing factor was ASTM C457⁽¹⁸⁾ (hardened concrete). Air content is usually tested in the field using the pressure-meter method (ASTM C231⁽¹⁴⁾ and AASHTO T 152⁽¹⁵⁾) or the volumetric method (ASTM C 173⁽¹⁶⁾ and AASHTO T196⁽¹⁷⁾), or it can be also calculated gravimetrically (ASTM C 138⁽³⁰⁾ or AASHTO T 121⁽³¹⁾) from concrete density (unit weight). However, the air content alone may not indicate the adequacy of freeze-thaw protection. As previously stated, it is the volume, number, and size distribution (taken together) that determine the quality of the air void system. Furthermore, the stability of the air void system (and thus, the air content and other parameters) may be affected by a number of materials, as well as environmental and construction variables—mix proportions, mixing time, haul time, pumping, spreading, vibration, and finishing. Thus there is no guarantee that the air

void system in the concrete, as placed, is the same as the air void system of the concrete sampled at the truck chute or from the grade.

FREEZE-THAW TESTING

The AASHTO T-161⁽²⁰⁾ and ASTM C666 *Resistance of Concrete to Freezing and Thawing*⁽²¹⁾ are the standard laboratory test methods for assessing concrete's resistance to freezing and thawing. The test method uses concrete specimens (of widths and depths from 76 mm to 127 mm and lengths between 279 mm to 406 mm) that are subjected to temperature cycling from 4 °C to -18 °C. The duration of the cycles is 2 to 5 hours. The freezing portion of the cycle is accomplished by air cooling (similar to air conditioning); the thawing portion, by submersion in water.

Freezing rate is an important factor. In pastes of ordinary porosity, where hydraulic pressure is the main mechanism of deterioration, fast freezing in the laboratory promotes more severe concrete degradation than in the field. In dense pastes, where growth of capillary ice is the main cause of damage, fast freezing in the laboratory promotes milder degradation than in the field.⁽³²⁾

There are two standard procedures, designated Procedure A and Procedure B. In Procedure A, the prisms are placed in containers (usually stainless steel) with approximately 3 mm space between the prism and the bottom and sides of the container. The container is filled with water, thereby surrounding the specimen on all sides (including the top) with water for the duration of testing. Figure 8 shows a photograph of a vertical container. Some equipment provides for horizontal containers. This study uses vertical containers. In Procedure B, the specimens are placed directly into the freeze-thaw chamber with no container or other covering.



Figure 8. Photo. Vertical container for freeze-thaw concrete specimen (ASTM C 666 Procedure A).

DAMAGE ASSESSMENT USING MODAL TESTING

Modal testing is a nondestructive method for assessing the dynamic response of structures. This method uses sinusoidal excitation for the input signal and forces the specimen to vibrate at a frequency as the response of the specimen is monitored with an accelerometer.⁽³³⁾

ASTM C215⁽²²⁾ uses modal testing to assess damage to beams undergoing freeze-thaw testing. A natural frequency of vibration is a characteristic (dynamic property) of an elastic system. Assuming a homogeneous, isotropic, elastic material, the dynamic modulus of elasticity is related to the resonant frequency and density.

Resonance manifests itself as a great increase in amplitude of oscillation when a system is driven at a certain frequency. The natural frequency (resonant frequency) is the characteristic frequency at which maximum response (amplitude) occurs.

Two methods for measuring resonant frequency are described in ASTM C215⁽²²⁾: sinusoidal excitation (forced oscillation) and impact excitation. The classic ASTM C215⁽²²⁾ forced resonance setup uses either transverse or longitudinal resonance. In the longitudinal mode, the oscillator is at one end and the pickup is at the other. In the transverse mode, the oscillator is in the middle of the top surface, and the pickup is at one end of the top surface.

The ASTM C215⁽²²⁾ impact method uses a modally tuned impact hammer to excite vibrations in the beam and an accelerometer attached to the beam to record the response. ASTM C215⁽²²⁾ shows schematics of the impact resonance test setup. Modal tuning enables the isolation of the hammer's response from the structural response, thus providing an accurate measurement of the specimen response and not the combined system (impact hammer and structure) response.⁽³⁴⁾

The resonant frequency of a concrete prism varies depending on the testing mode and the orientation of the prism. The resonant frequency in the longitudinal direction is typically much higher (on the order of 5000 Hz) than the transverse resonant frequency (on the order of 2500 Hz). The resonant frequency for a prism with a rectangular cross section is lower when the prism is supported on the wider edge.

In the present study, the impact test method was used to measure transverse frequency, following the setup used by Clarke.⁽³⁵⁾ Appendix D presents more details about this method. The test setup is shown in figure 9. The rectangular test prisms (75 mm by 100 mm by 400 mm) were placed with the narrow edge (75 mm) down on piano wire supports located at the nodal points (0.224 by prism length from the end of the specimen).

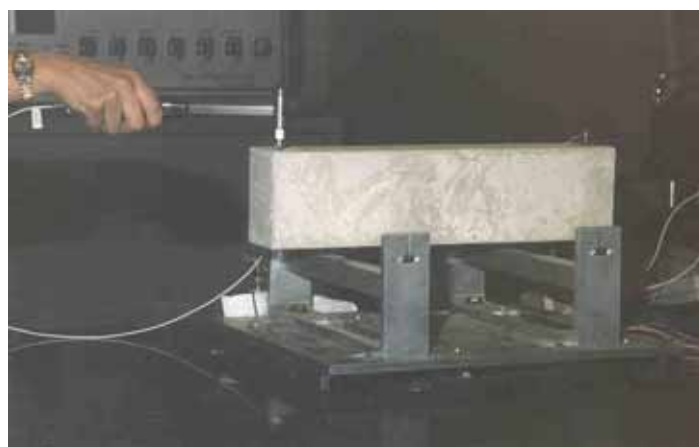


Figure 9. Photo. ASTM C 215 test setup.

The procedure used to assess damage begins by removing the beam from the freeze-thaw chamber, and after the thawing cycle is finished, towel drying (to saturated, surface dry (SSD)) the beam and weighing it. The specimen is placed on the piano wire supports, and an accelerometer (output signal) is attached to one end of the beam using vacuum grease. Using the impact hammer, the beam is tapped at its opposite end, and the time domain response data (impulse versus time and response versus time) are recorded using appropriate equipment. Figures 10 and 11 are examples of time domain impulse and response data, respectively.

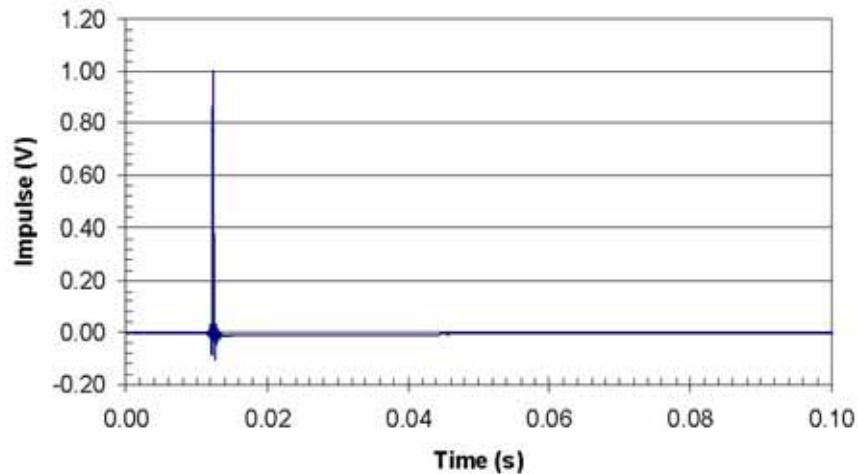


Figure 10. Graph. Time domain impulse data.

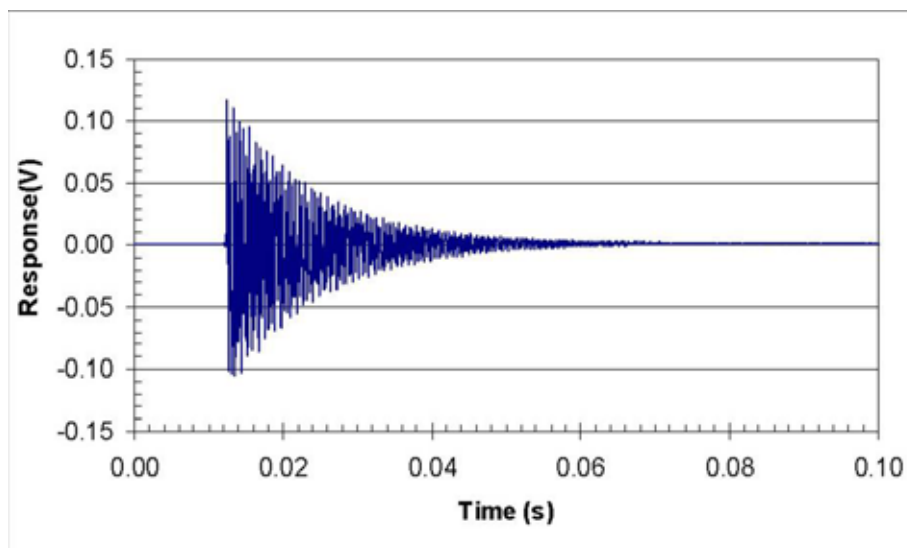


Figure 11. Graph. Time domain response data.

Dynamic signal analysis software (Virtual Bench Dynamic Signal Analyzer (DSA)) can then be used to transform the time domain data to the frequency domain (using the Fast Fourier Transform (FFT)) to determine the frequency response curve and to determine the resonant

frequency. A typical frequency response curve is shown in figure 12. The resonant frequency is the frequency (x-axis value) at the maximum amplitude of the frequency response curve.

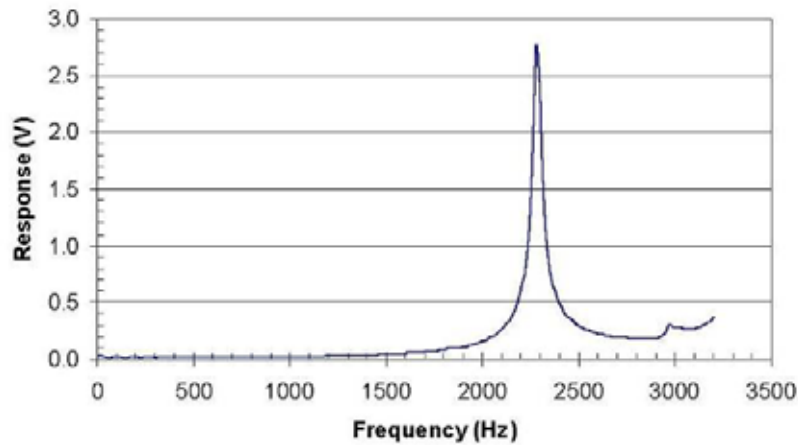


Figure 12. Graph. Frequency response curve.

Testing is repeated at regular intervals—usually every 10 to 30 cycles (depending on expected freeze-thaw behavior). The relative dynamic modulus (RDM), expressed in percentage, is calculated as follows:

$$P_c = \frac{n_c^2}{n^2} \times 100$$

Figure 13. Equation. Relative dynamic modulus.

where c is the number of cycles of freezing and thawing, n_c is the resonant frequency after c cycles, and n is the initial resonant frequency (at zero cycles).

The durability factor (DF) is defined as:

$$DF = \frac{N}{M} \times P_c$$

Figure 14. Equation. Durability factor.

where P_c is the relative dynamic modulus, N is the number of cycles completed, and M is the planned duration of testing (usually 300 cycles). Testing is usually halted when the relative dynamic modulus falls below 50–60 percent of its initial value.

If the concrete is not adequately protected by air entrainment, microcracking occurs with each cycle of freezing and thawing. Microcracks increase damping in the beam, thereby reducing the vibration amplitude and flattening the frequency response curve. Cracking also causes the resonant frequency to decrease—waves take longer to travel through the concrete when it contains cracks (figure 15).

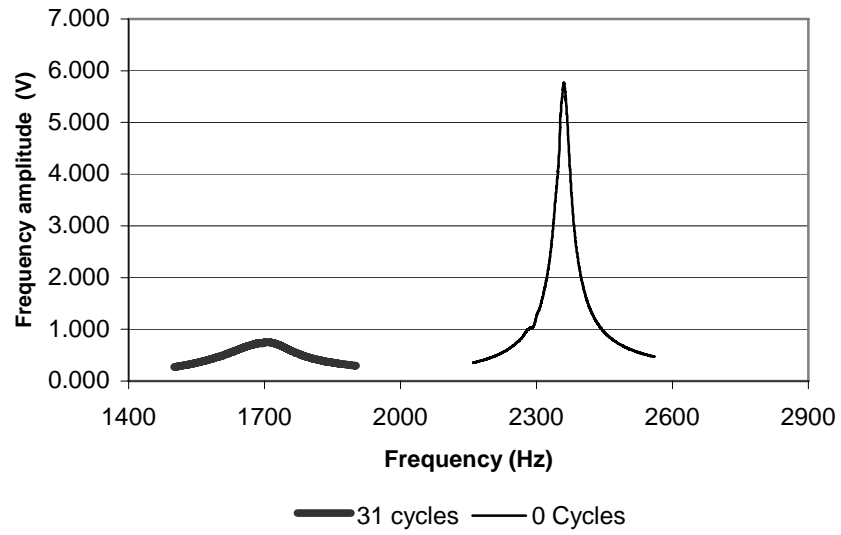


Figure 15. Graph. Effect of freeze-thaw cycling on the resonant frequency curve of a non-air-entrained concrete after 31 cycles when concrete failure was achieved (mix 302—beam A, see chapter 5).

CHAPTER 3: EFFECT OF AIR CONTENT AND W/C ON FREEZE-THAW RESISTANCE

In order to evaluate the relation between concrete microstructure and its freeze-thaw resistance, an experiment was designed for concretes with fresh air contents (total air) ranging from 2.5 to 4.5 percent and w/c ranging from 0.4 to 0.5; freeze-thaw testing was performed using ASTM C 666,⁽²¹⁾ Procedure A. Specimens were monitored for changes in resonant frequency (ASTM C 215⁽²²⁾) and mass at regular intervals. ASTM C457⁽¹⁸⁾ air void system evaluations (both modified point count and linear traverse) were conducted on hardened specimens from each mix.

EXPERIMENTAL INVESTIGATION

The experiment was designed as a central composite design in the two variables, with a total of 13 mixes (4 factorial points, 4 axial points, and 5 center points). Central composite design, (which is enhanced factorial design), is widely used for fitting a second-order response surface. It allows estimation of a full quadratic model for each response. Response surface methodology consists of a set of statistical methods normally applied in situations where several factors (for instance, the proportions of individual materials in concrete)—in this case w/c ratio and air entrained—influence one or more performance characteristics, or responses (freeze-thaw resistance, for example).

The experiment design of this study consists of 2^k factorial points, $2*k$ axial points, and 5 center points, where k is the independent variable (in this case 2). The design is shown in table 1. Shaded mixes represent the control mix (center point), which was repeated several times.

Table 1. Experiment design for mixes 115–118.

Run #	Mix ID	W/C	Total fresh air (%)	Point type
1	115-1	0.45	3.5	Center
2	115-2	0.45	3.5	Center
3	115-3	0.40	4.5	Factorial
4	116-1	0.45	3.5	Center
5	116-2	0.40	2.5	Factorial
6	116-3	0.45	2.5	Axial
7	117-1	0.50	3.5	Axial
8	117-2	0.50	2.5	Factorial
9	117-3	0.45	4.5	Axial
10	117-4	0.45	3.5	Center
11	118-1	0.50	4.5	Factorial
12	118-2	0.45	3.5	Center
13	118-3	0.40	3.5	Axial

Materials included Type I Portland cement (ASTM C 150⁽³⁶⁾), #57 crushed limestone coarse aggregate, ASTM C33⁽³⁷⁾ natural sand (quartz), and tap water (material properties can be found in

appendix A). The air-entraining admixture was a Vinsol resin-based admixture meeting ASTM C 260⁽³⁸⁾ (AASHTO M154⁽³⁹⁾). Concrete was mixed in a 0.25 m³ drum mixer according to ASTM C192.⁽⁴⁰⁾ The batch size was 0.07 m³. Mix proportions actually used are shown in table 2.

Fresh concrete tests included slump (ASTM C 143⁽⁴¹⁾), fresh air content (ASTM C231⁽¹⁴⁾), and unit weight (ASTM C 138⁽⁴¹⁾). Five 75- by 100- by 400-mm prisms (for freeze-thaw testing) and eight 100- by 200-mm cylinders (for strength testing and ASTM C457 evaluations⁽¹⁸⁾) were cast for each mix. Admixture dose is given in liters (L) per 100 kg of cement.

Table 2. Mixture proportions for mixes 115–118.

Mix ID	W/C	Coarse agg (SSD) kilogram (kg)/m ³	Fine agg (SSD) kg/m ³	Cement kg/m ³	Water kg/m ³	AEA L/100 kg	WRA† L/100 kg
115-1	0.45*	976	866	355	163	0.033	0.260
115-2	0.45	978	870	355	160	0.033	0.260
115-3	0.40	978	891	355	142	0.072	0.260
116-1	0.45	970	889	352	159	0.007	0.319
116-2	0.40	979	944	356	142	0.003	0.260
116-3	0.45	980	898	356	160	0.008	0.260
117-1	0.50	979	825	356	178	0.023	0.260
117-2	0.50	978	850	355	178	0.002	0.260
117-3	0.45	975	854	355	160	0.046	0.260
117-4	0.45	976	869	355	160	0.028	0.260
118-1	0.50	978	797	355	178	0.043	0.260
118-2	0.45	978	870	355	160	0.023	0.260
118-3	0.40	981	920	357	143	0.036	0.260

* Actual as-batched w/c was 0.46 for this mix .

† WRA—water reducing admixture

RESULTS

The fresh concrete properties for each mix are shown in table 3.

Slumps were quite low at w/c of 0.40 and 0.45 (13 mm or less) but increased to 50 mm or more at w/c=0.50. Slump also increased slightly with air content at w/c=0.50.

The mean 28-day strengths (21-day strengths for mixes 115-1, 115-2, and 115-3) and standard deviations are shown in table 4.

Table 3. Fresh concrete properties for mixes 115–118.

Mix ID	W/C	Slump (mm)	Total air content (%)	Unit weight (kg/m ³)
115-1	0.46	0	3.6	2361
115-2	0.45	5	3.6	2368
115-3	0.40	0	4.6	2374
116-1	0.45	15	3.4	2379
116-2	0.40	0	2.5	2401
116-3	0.45	15	2.4	2416
117-1	0.50	65	3.5	2390
117-2	0.50	50	2.7	2366
117-3	0.45	0	4.4	2371
117-4	0.45	15	3.8	2358
118-1	0.50	70	4.6	2352
118-2	0.45	5	3.6	2377
118-3	0.40	0	3.3	2387

Table 4. 28-Day strength results for mixes 115–118.

Mix ID	W/C	Total fresh air content (%)	Mean 28-day strength* (megapascals (MPa))	Std. dev. (MPa)
115-1*	0.46	3.6	39.9	0.3
115-2*	0.45	3.6	43.1	1.6
115-3*	0.40	4.6	43.1	1.7
116-1	0.45	3.4	50.3	1.4
116-2	0.40	2.5	53.2	0.2
116-3	0.45	2.4	50.3	0.8
117-1	0.50	3.5	38.6	5.8
117-2	0.50	2.7	40.8	1.0
117-3	0.45	4.4	44.3	1.5
117-4	0.45	3.8	46.3	4.8
118-1	0.50	4.6	41.0	1.3
118-2	0.45	3.6	49.0	0.8
118-3	0.40	3.3	49.3	0.0

* 21-day strengths are reported for 115-1, 115-2, and 115-3. All results based on 2 tests of 100- by 200-mm cylinders.

The results of freeze-thaw testing are summarized in table 5. DF ranged from 3.3 to 94.8 percent. With one exception (mix 116-1), mixes with greater than 3.0 percent fresh air content performed well (DF > 80) through more than 300 cycles of freezing and thawing. All specimens suffered some mass change (loss) during testing. Mass losses ranged from 0.61 to 3.66 percent. The mass loss can be attributed to surface scaling, which occurred on all beams. Any mass gain resulting from water entering the concrete through cracks was obscured by the losses due to scaling.

Table 5. Summary of freeze-thaw test results for mixes 115–118.

Mix ID	W/C	Fresh air (%)	Cycles	Final RDM (%)	DF (%)	Mass change (%)
115-1	0.46	3.6	300	84.2	84.2	−3.66
115-2	0.45	3.6	300	80.3	80.3	−3.28
115-3	0.40	4.6	300	85.7	85.7	−2.63
116-1†	0.45	3.4	103	51.5	17.7	−0.66
116-2	0.40	2.5	132	48.0	21.1	−0.61
116-3	0.45	2.4	191	57.7	36.7	−2.60
117-1	0.50	3.5	303*	89.6	90.5	−3.53
117-2	0.50	2.7	38	26.4	3.3	0.13
117-3	0.45	4.4	303*	90.9	91.8	−3.39
117-4	0.45	3.8	303*	90.3	91.2	−1.88
118-1	0.50	4.6	300	94.8	94.8	−1.73
118-2	0.45	3.6	300	92.7	92.7	−1.18
118-3	0.40	3.3	300	92.4	92.4	−0.92

* The values of DF are corrected to 300 cycles. The actual relative dynamic modulus is shown in final RDM column.

† Not included when averaging center mixes.

Tables 6 and 7 summarize the results of ASTM C457⁽¹⁸⁾ modified point count (MPC) and linear traverse (LT) evaluations on polished surfaces cut axially from 100- by 200-mm cylinders.

The significantly different freeze-thaw resistance of mix 116-1, when compared to the other center mixes, and its low durability factor can be explained by its air void system. It seems that the fresh air content of mix 116-1 was not properly determined, so although the percentage of fresh air showed to be within the target range, both modified point count and linear traverse results show a different scenario. Not only was the air content much lower than the other center mixes, but also the specific surface was much lower and the spacing factor was much higher. As a result, when averaging center mixes, mix 116-1 is disregarded.

Table 6. Modified point count (MPC) results for mixes 115–118.

Mix ID	Fresh air (%)	Air (%)	Paste (%)	Voids counted	MCL (mm)	Voids per m	Specific surface (mm ⁻¹)	Spacing factor (mm)
115-1	3.6	4.0	28.0	313	0.302	138	13.6	0.406
115-2	3.6	3.7	25.5	219	0.385	96	10.6	0.521
115-3	4.6	4.1	27.0	321	0.286	142	14.5	0.381
116-1	3.4	2.5	28.5	109	0.525	49	7.6	0.876
116-2	2.5	3.7	28.4	127	0.660	55	6.1	0.940
116-3	2.4	2.4	28.1	132	0.404	59	10.0	0.686
117-1	3.5	6.2	25.4	400	0.353	175	11.3	0.368
117-2	2.7	3.8	26.8	105	0.822	47	4.9	1.118
117-3	4.4	4.6	30.7	394	0.264	173	15.2	0.343
117-4	3.8	5.0	29.5	300	0.386	132	10.7	0.483
118-1	4.6	7.2	26.2	658	0.249	289	16.2	0.229
118-2	3.6	3.9	27.1	264	0.330	116	12.5	0.445
118-3	3.3	3.9	26.6	243	0.364	106	10.5	0.483

Notes: All results are averages of two tests on two different polished surfaces.
MCL=Mean chord length

Table 7. Linear traverse (LT) results for mixes 115–118.

Mix ID	Fresh air (%)	Air (%)	Voids counted	MCL (mm)	Voids per m	Specific surface (mm ⁻¹)	Spacing factor (mm)
115-1	3.6	4.8	386	0.282	169	14.1	0.363
115-2	3.6	6.1	300	0.465	130	8.6	0.533
115-3	4.6	5.3	463	0.262	201	15.3	0.320
116-1	3.4	3.8	140	0.612	63	6.5	0.879
116-2	2.5	4.9	192	0.587	83	6.8	0.744
116-3	2.4	3.6	178	0.460	79	8.7	0.673
117-1	3.5	5.4	393	0.315	173	12.7	0.381
117-2	2.7	4.1	127	0.732	55	5.5	1.011
117-3	4.4	5.3	473	0.257	209	15.6	0.315
117-4	3.8	4.9	327	0.345	142	11.6	0.437
118-1	4.6	5.6	581	0.221	256	18.1	0.264
118-2	3.6	4.2	276	0.345	122	11.5	0.472
118-3	3.3	3.9	221	0.399	98	10.0	0.564

Notes: All results are based on one test.
Spacing factors were calculated using paste content from MPC.
MCL=Mean chord length

DISCUSSION AND ANALYSIS

Figures 16–18 show the influence of the air content (based on fresh air content) on durability. It can be observed that the mixes with fresh air content in the levels of 3.5 percent and 4.5 percent

present similar freeze-thaw resistance. They last at least 300 cycles and their durability factors are higher than 80 percent, except for mix 116-1. On the other hand, the mixes with air content around 2.5 percent present much lower freeze-thaw resistance. A correlation of 0.78 was obtained between fresh air content and durability factor. The legends in the figures indicate the mix ID for the plotted points, and text boxes in the figure provide a summary of air content, and DF for each mix. The center mixes are represented by their average RDM (mix 116-1 was not included).

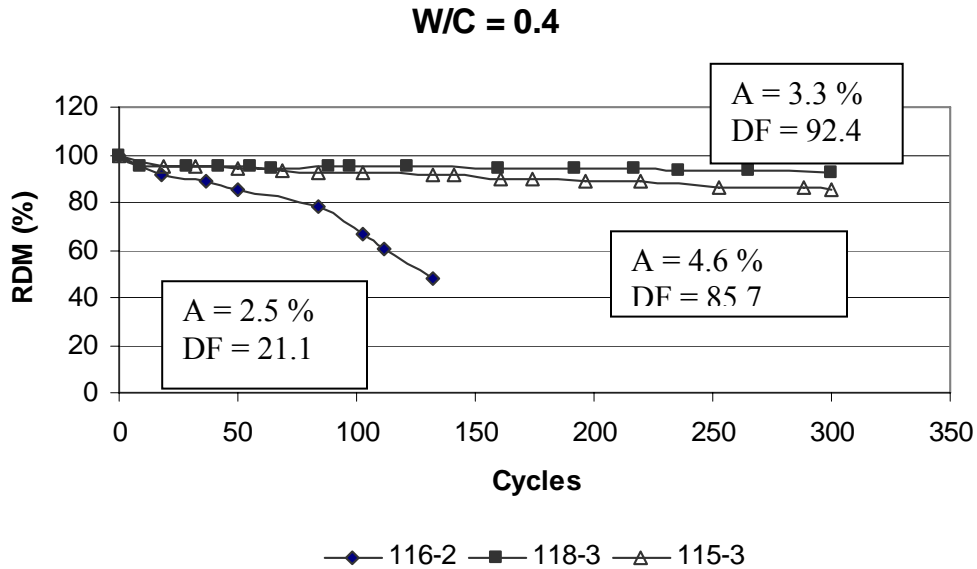


Figure 16. Graph. Relative dynamic modulus versus cycles for mixes with water-cement ratio=0.40.

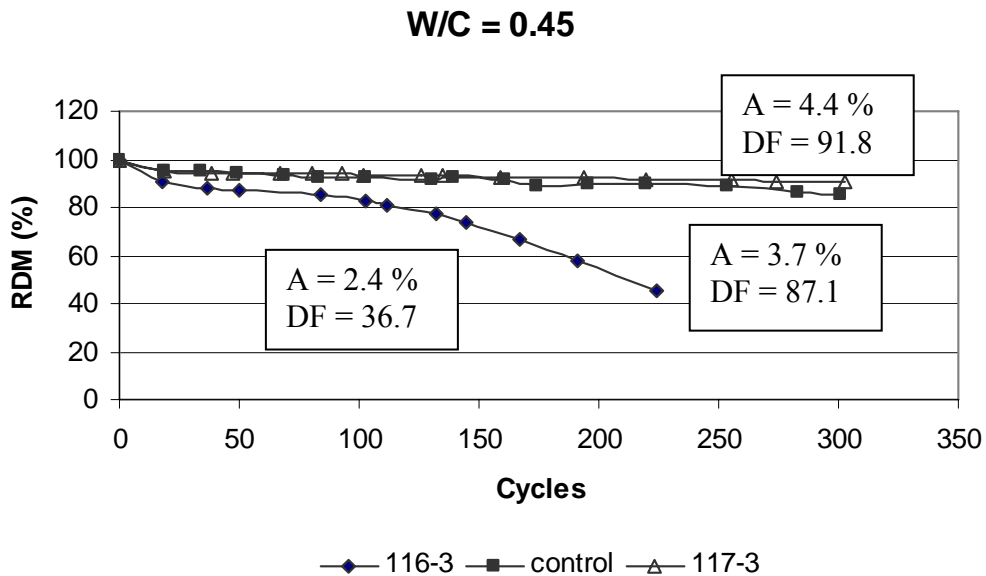


Figure 17. Graph. Relative dynamic modulus versus cycles for mixes with water-cement ratio=0.45.

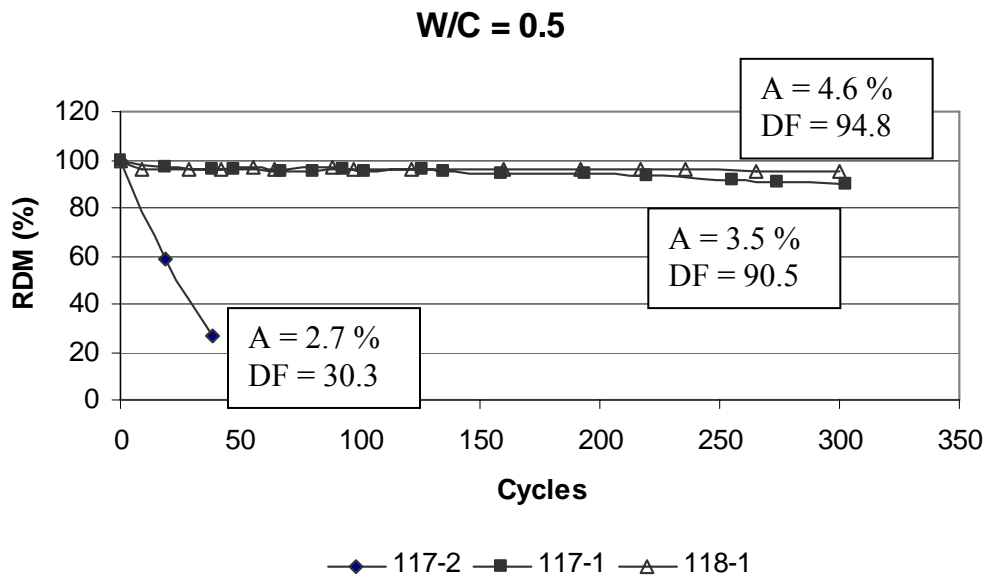


Figure 18. Graph. Relative dynamic modulus versus cycles for mixes with water-cement ratio=0.50.

The water-cement ratio (within the range tested) does not appear to play a significant role on the freeze-thaw resistance (figures 19 and 20). The correlation between water-cement ratio and durability factor was 0.04. Only for mixes with designed air content of 2.5 percent (figure 21), the mix with w/c=0.5 (117-2) shows a much lower freeze-thaw resistance. Nevertheless, this difference in performance seems to be much more related to the air void system (low specific surface and high spacing factor) than to the w/c ratio. It is confirmed if mixes 116-2 and 116-3 are compared, where the latter presents higher specific surface, lower spacing factor, and as a result, better freeze-thaw resistance.

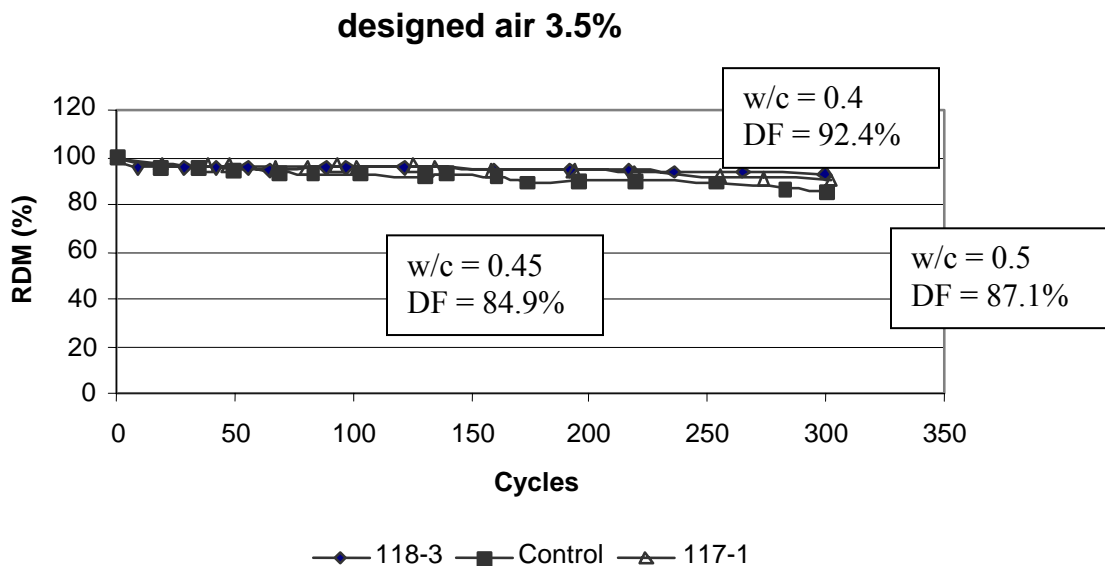


Figure 19. Graph. Relative dynamic modulus versus cycles for mixes with designed air void content of 3.5 percent.

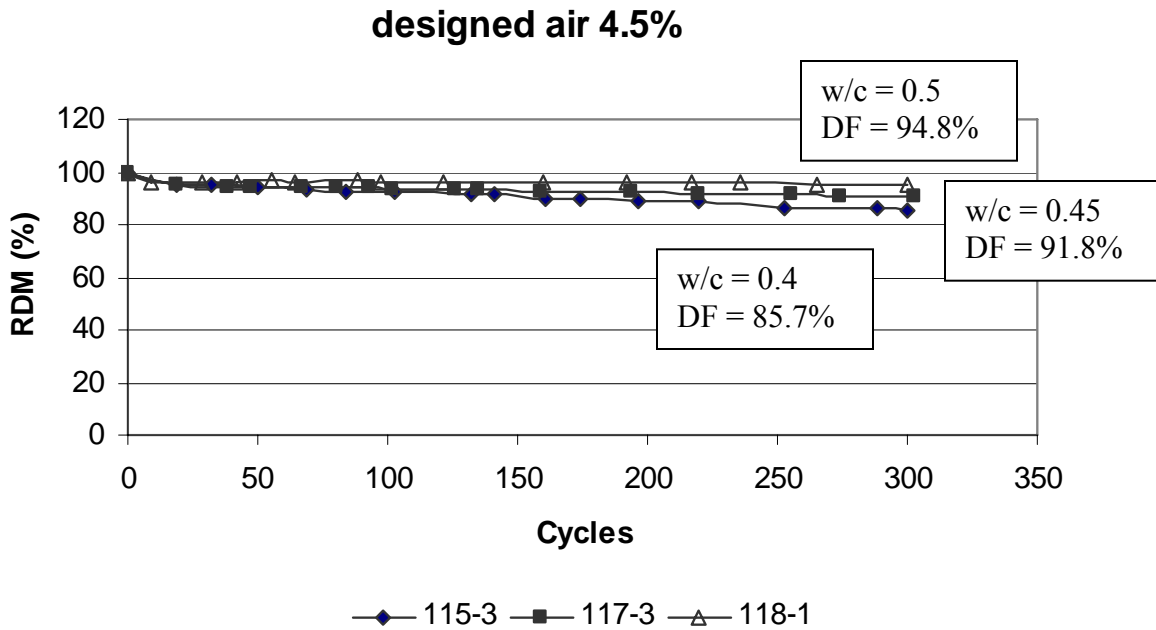


Figure 20. Graph. Relative dynamic modulus versus cycles for mixes with designed air void content of 4.5 percent.

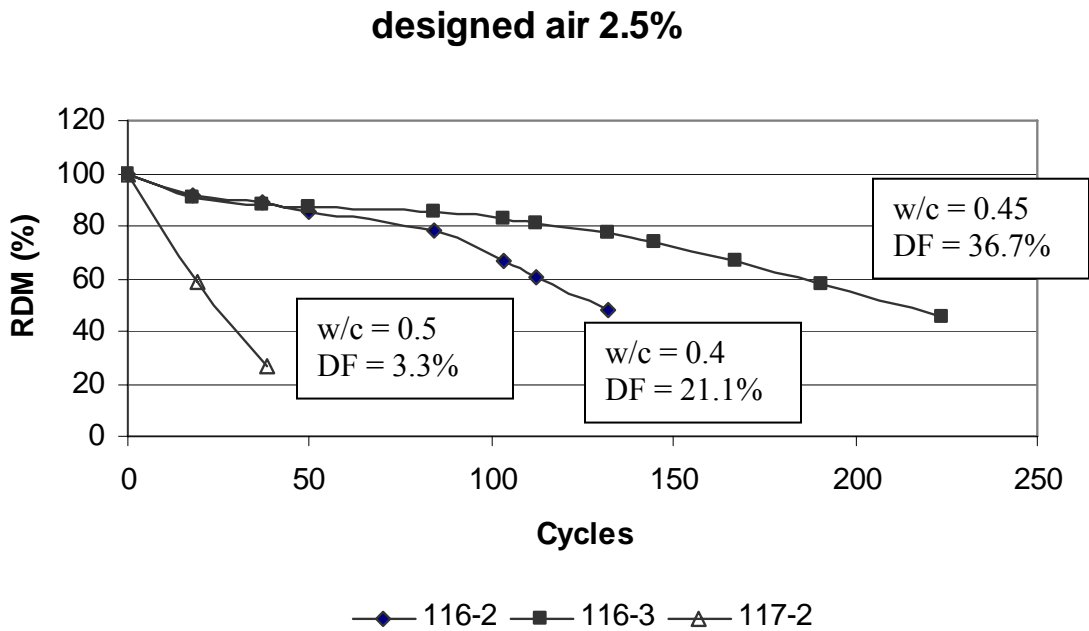


Figure 21. Graph. Relative dynamic modulus versus cycles for mixes with designed air void content of 2.5 percent.

The mass change over cycles can be associated with concrete deterioration. A mass gain can be an indication of cracking formation and water absorption through the cracks. On the other hand,

the mass loss can also be related to concrete deterioration in the case where concrete specimens scale significantly during testing. This set of mixes (115–118) did not show any mass gain. Also, the mass loss did not present any trend in relation to air void system parameters (figures 22–24).

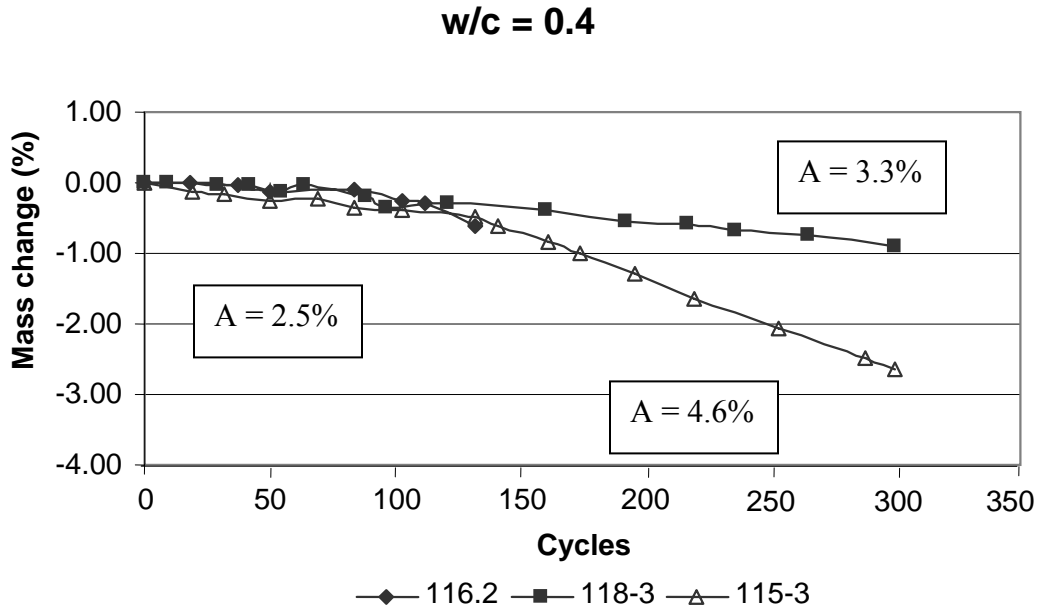


Figure 22. Graph. Mass change versus cycles for mixes with water-cement ratio=0.40.

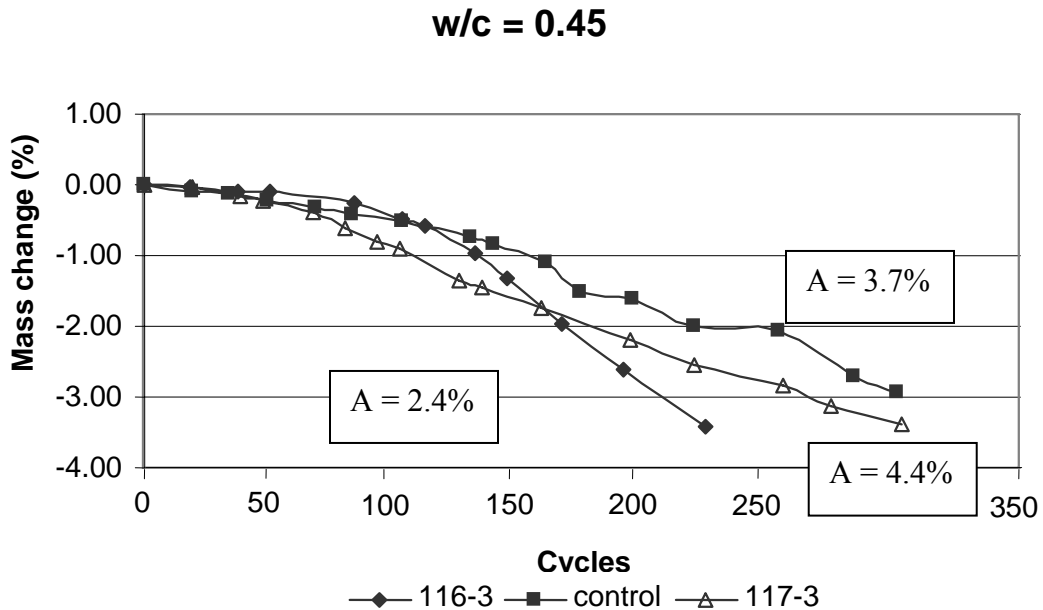


Figure 23. Graph. Mass change versus cycles for mixes with water-cement ratio=0.45.

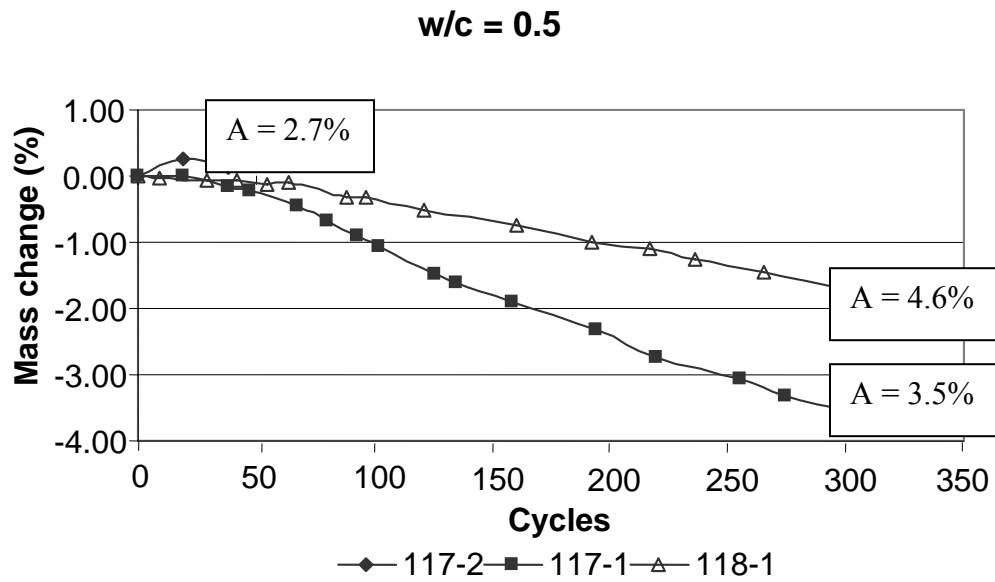


Figure 24. Graph. Mass change versus cycles for mixes with water-cement ratio=0.50.

In terms of air measurement, the fresh air void content, when measured according to ASTM C 231,⁽¹⁴⁾ was always lower than the linear traverse air volume (measured according to ASTM C 457⁽¹⁸⁾) and, in most of the cases, lower than the modified point count, as well (figure 25).

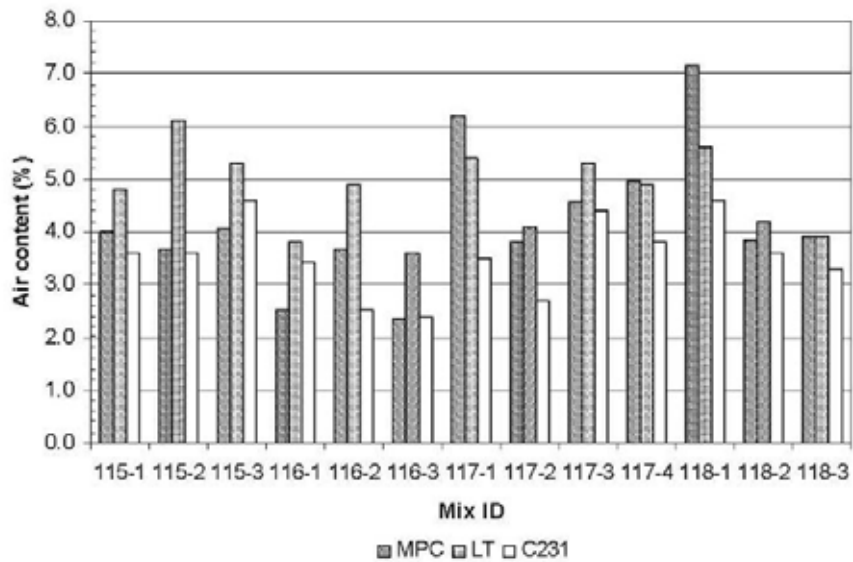


Figure 25. Graph. Comparison among modified point count test, linear traverse test, and fresh air content.

The spacing factor versus the relative dynamic modulus (figure 26) shows a clear trend (with correlation of 0.91): the higher the spacing factor, the lower the RDM. The specific surface shows the same trend (figure 27) but with a lower correlation (0.77).

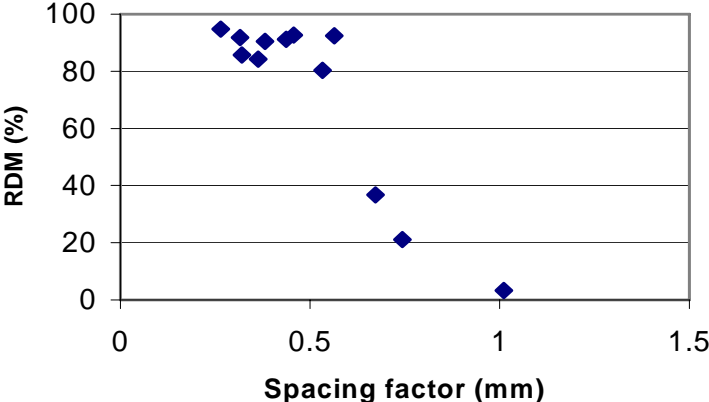


Figure 26. Graph. Relation between spacing factor and relative dynamic modulus.

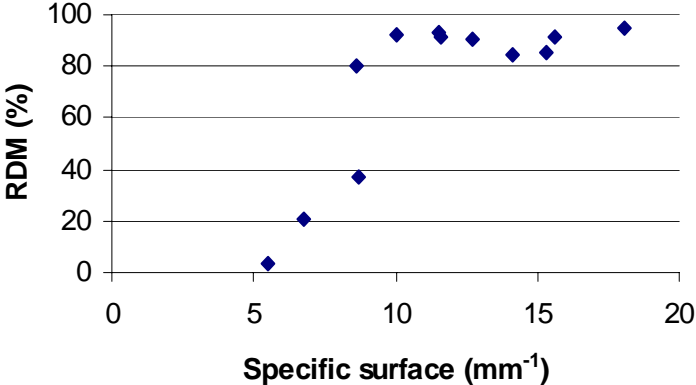


Figure 27. Graph. Relation between specific surface and relative dynamic modulus.

CHAPTER 4: USE OF SYNTHETIC AIR-ENTRAINING ADMIXTURE

The scarcity of Vinsol resin admixture is responsible for the increasing use of synthetic admixtures. The freeze-thaw performance of marginal air mixes containing synthetic admixtures was investigated in a comparison with Vinsol resin. This experiment was made up of concretes with a wide range of air contents, batched with two different air-entraining admixtures (AEA), Vinsol and a synthetic. Freeze-thaw testing was performed using ASTM C 666⁽²¹⁾, Procedure A. Specimens were monitored for changes in resonant frequency (ASTM C 215⁽²²⁾) and mass at regular intervals. ASTM C457⁽¹⁸⁾ air void system evaluations (both modified point count and linear traverse) were conducted on hardened specimens from each mix.

EXPERIMENTAL INVESTIGATION

Two sets of tests were performed—one for each of the two air-entraining admixtures: set 1 containing Vinsol resin air-entraining admixture and set 2 containing synthetic air-entraining admixture. The mix proportion of the two sets was the same. Each set consisted of five concrete mixtures proportioned with w/c ratios of 0.45 and target fresh air contents of 2.5 to 4.5 percent, in increments of 0.5 percent. In set 1, an additional non-air-entrained concrete mixture was also proportioned. The materials used are shown in table 8.

Table 8. Materials used for set 1 (mixes 223–302—Vinsol resin (VR) AEA) and set 2 (346–350—(synthetic) SYN AEA).

Component	Set 1 (223–302—VR AEA))	Set 2 (346–350—SYN AEA))
Water	Municipal tap water	Municipal tap water
Cement	Type I	Type I
Fine aggregate	Natural sand	Natural sand
Coarse aggregate	#57 Crushed limestone	#57 Crushed limestone
AEA type	Vinsol resin	Synthetic
WRA	ASTM C 494 Type A* ⁽⁴²⁾	ASTM C 494 Type A* ⁽⁴²⁾

* The water reducer admixture used in both sets was the same and meet the requirements of ASTM C 494⁽⁴²⁾ Type A, water-reducing, Type B, retarding, and Type D, water-reducing and retarding, admixtures.

The mixture proportions used for sets 1 and 2 are shown in tables 9 and 10.

Table 9. Mixture proportions for set 1 (223–302)—w/c=0.45.

Mix ID	Target air (%)	Coarse agg (SSD) kg/m ³	Fine agg (SSD) kg/m ³	Cement kg/m ³	Water kg/m ³	Vinsol AEA L/100kg	WRA L/100kg
223	4.0	1015	836	356	160	0.033	0.210
224	3.5	1015	836	356	160	0.026	0.210
225	3.0	1015	836	356	160	0.035	0.210
226	4.5	1015	836	356	160	0.048	0.210
227	2.5	1015	836	356	160	0.013	0.210
302	—	1015	876	356	160	0.000	0.415

Table 10. Mixture proportions for set 2 (mixes 346–350)—w/c=0.45

Mix ID	Target air (%)	Coarse agg (SSD) kg/m ³	Fine agg (SSD) kg/m ³	Cement kg/m ³	Water kg/m ³	Synthetic AEA L/100kg	WRA L/100kg
348	2.5	1015	857	356	160	0.028	0.266
346	3.0	1015	861	356	160	0.039	0.266
347	3.5	1015	857	356	160	0.016	0.266
349	4.0	1015	805	356	160	0.079	0.266
350	4.5	1015	805	356	160	0.138	0.266

The concrete was mixed in batches of 0.042 m³ in a drum mixer with 0.125 m³ capacity. From each mix in set 1 (223–302), three 100- by 200-mm cylinders for compressive strength and five 75- by 100- by 400-mm beams for freeze-thaw testing were cast. Two cylinders 150- by 300-mm were cast for air void system analysis (ASTM C 457⁽¹⁸⁾). From each mix in set 2, the same number and type of cylinders were cast along with four beams (same size as set 1).

For set 1, using the Vinsol resin AEA, the mix sequence was as follows:

1. The coarse and fine aggregates were added to the mixer and mixed for 30 seconds.
2. The AEA was added to part of the water, the AEA and water were added to the mixer, and the materials were mixed for 30 seconds.
3. The WRA was added to the remaining water, the WRA and water were added to the mixer, the cement was added to the mixer, and the materials were mixed for 4 minutes.
4. The mixer was stopped for a 2-minute rest period.
5. The materials were mixed for 2 additional minutes.

For set 2, using the synthetic AEA, various trial mix sequences were carried out until the target air content was achieved. The final mix sequence used was quite different from the regular mix procedures used, including the use of warm water and the addition of the two admixtures at the same time:

1. The coarse and fine aggregates were added to mixer and mixed for 30 seconds.
2. The AEA and WRA were added to the entire amount of water, the AEA, WRA, and water were added to the mixer, and the materials were mixed for 30 seconds.
3. The cement was added to the mixer and the materials were mixed for 3 minutes.
4. The mixer was stopped for a 3-minute rest period.
5. The materials were mixed for 2 additional minutes.

In set 1, the admixtures were added to the mix separately (AEA first, then WRA); the water was at room temperature. In set 2, however, both admixtures were added at the same time (in accordance with the manufacturer’s recommendations), and the mixing water was warm (around 38 °C), in order to reduce the amount of air entrained. This procedure was necessary because, even when very small amounts of AEA were used, the air content produced exceeded the target values. In both sets of mixes, the coarse aggregates were batched dry, while the sand was batched moist. For each mix, the mixing water contents were adjusted accordingly.

All test specimens were cured in saturated limewater at 23±2 °C. Compressive strength cylinders were cured for 28 days and freeze-thaw specimens for 14 days. The cylinder ends were ground in a concrete end grinder prior to strength testing per ASTM C 39.⁽⁴³⁾

The specimens were tested in accordance with ASTM C666, Procedure A.⁽²¹⁾ The specimens were monitored for changes in resonant frequency in accordance with ASTM C 215⁽²²⁾ and for mass changes (to the nearest 1 g) at regular intervals. ASTM C457⁽¹⁸⁾ air void system evaluations (both modified point count and linear traverse) were conducted on hardened specimens from each mix.

RESULTS

The fresh concrete properties for sets 1 and 2 are shown in tables 11 and 12, respectively. A tolerance of 0.2 percent (deviation from target value) for fresh air content was considered acceptable.

Table 11. Fresh concrete properties for set 1 (VR AEA).

Mix ID	Slump (mm)	Air content (%)	Unit weight (kg/m ³)
223	44	4.0	2368
224	51	3.6	2379
225	44	3.1	2397
226	25	4.7	2349
227	44	2.7	2393
302	25	2.0	2400

Table 12. Fresh concrete properties for set 2 (SYN AEA).

Mix ID	Slump (mm)	Air content (%)	Unit weight (kg/m ³)
346	25	3.2	2363
347	19	3.5	2360
348	19	2.3	2390
349	25	4.0	2345
350	25	4.3	2339

The strength test results are shown in tables 13 and 14.

Table 13. 28-Day strengths for set 1 (VR AEA).

Mix ID	W/C	Fresh air content (%)	Mean 28-day strength (MPa)	Std. dev. (MPa)
223	0.45	4.0	36.4	2.0
224	0.45	3.2	39.4	1.6
225	0.45	3.1	38.5	1.8
226	0.45	4.7	38.1	1.3
227	0.45	2.7	43.2	0.1
302	0.45	2.0 (Non-air-entrained)	49.5	4.1

All results based on 3 tests of 100- by 200-mm cylinders.

Table 14. 28-Day strengths for set 2 (SYN AEA).

Mix ID	W/C	Fresh air content (%)	Mean 28-day strength (MPa)	Std. dev. (MPa)
346	0.45	3.2	44.8	1.0
347	0.45	3.5	35.0	1.2
348	0.45	2.3	42.5	1.0
349	0.45	4.0	38.1	0.4
350	0.45	4.3	32.6	1.8

All results based on 3 tests of 100- by 200-mm cylinders

Table 15 presents the air void system for set 1 (223–227). The air void parameters were determined according to ASTM C 457⁽¹⁸⁾ (linear traverse) and represent the average of two measurements. The combined linear traverse and point count results can be found in appendix B. The air system of mix 302 (set 1) (non-entrained-air concrete) was not determined.

Table 15. Air void system of set 1 (VR AEA) measured by linear traverse.

Mix	Fresh air (ASTM C 231)	Air (%) ASTM C 457	Chord length (mm)	Voids counted	Mean chord length (mm)	Voids per m	Specific surface (mm ² /mm ³)	Spacing factor (mm)
223	4	2.4	55	276	0.22	120	19.9	0.38
224	3.6	2.8	64	215	0.30	93	13.5	0.49
225	3.1	4.2	94	288	0.33	126	12.2	0.45
226	4.7	4.7	106	495	0.21	215	18.7	0.28
227	2.7	3.3	74	212	0.35	93	11.5	0.54

All mixes of set 1 presented marginal air void contents. The spacing factors were higher than the maximum value (0.2 mm) normally required for a good freeze-thaw resistance (most of them were above 0.36 mm) and the specific surface areas were lower than the normally desired

(24 mm^{-1}) for the total air volume in the range of the mixes for this study. Some of the mixes had specific surface area half of that, for example mix 227.

One could expect that the freeze-thaw performance of those mixes would not be adequate. Nevertheless, table 16 shows that DFs were above 80 percent, excepting for the non-air-entrained mix 302, which could be considered a satisfactory performance. All the air-entrained mixes withstood at least 300 cycles, excluding beam 224-A5 that suffered some damage during the handling of the specimen not related to testing. The tables and the graphs of the RDM over cycles can be found in appendix B.

**Table 16. Durability factor—results for set 1 (VR AEA).
Results are sorted by percent fresh air content.**

Mix	Fresh air (%)	Durability factor						
		A1	A2	A3	A4	A5	Proc A avg	Proc A std dev
302	2.0 non A/E	14.4	14.5	16.7	12.1	15.4	14.4	1.9
227	2.7	86.7	88.1	86.8	89.9	76.8	87.9	1.5
225	3.1	89.4	90.5	90.0	90.8	88.2	90.2	0.6
224	3.6	85.7	87.2	85.4	84.4	00.0*	85.7	1.2
223	4.0	89.6	88.5	89.7	84.4	92.0	88.9	2.8
226	4.7	92.1	94.0	93.0	95.0	95.3	93.5	1.3

* The DF for 224-A5 was not included when calculating averages and standard deviations.

Figure 28 shows the RDM versus cycles for one of the air entrained concretes (mix 225), which is representative of the mixes of set 1, excepting mix 302 (figure 29).

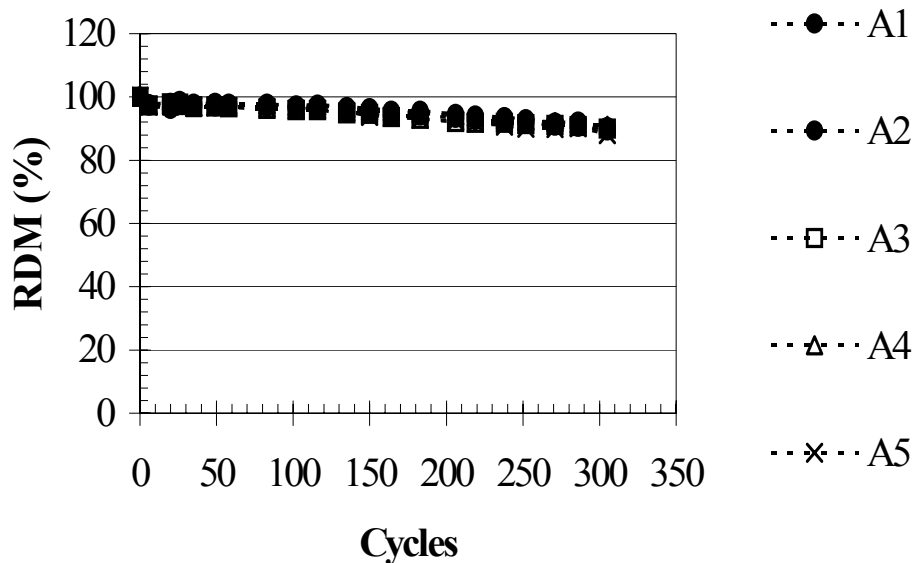


Figure 28. Graph. Relative dynamic modulus versus cycles for mix 225 (VR AEA—3.1 percent fresh air content). Individual specimens are shown. “A” stands for specimens tested according to Procedure A.

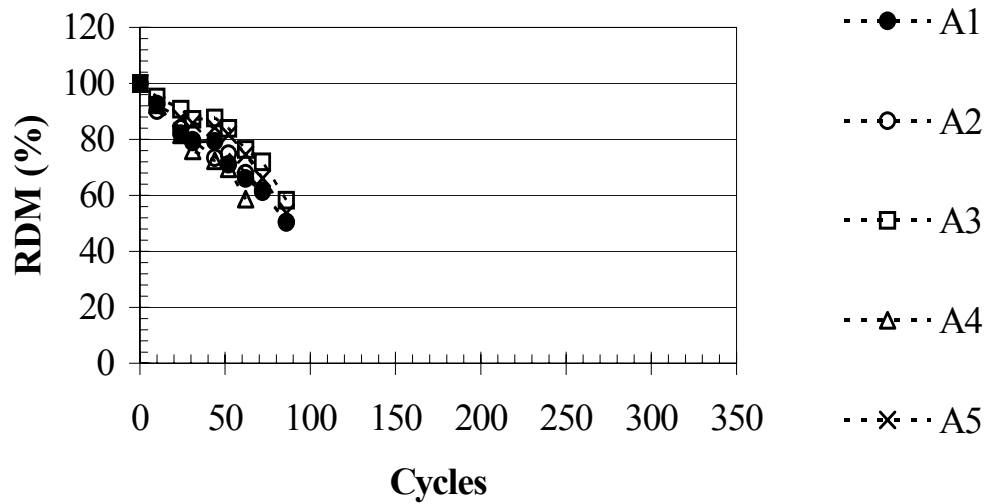


Figure 29. Graph. Relative dynamic modulus versus cycles for mix 302 (non-air-entrained). Individual specimens are shown. “A” stands for specimens tested according to Procedure A.

Set 2 presented a much better air system, with respect to spacing factor and specific surface area, but most of the mixes remained in the range of marginal air void parameters (table 17). The air void parameters were determined according to ASTM C 457⁽¹⁸⁾ (linear traverse) and represented the average of two measurements. Figures 30 and 31 show the differences in the air void chord size distributions from linear traverse results for the two different admixture types, sets 1 and 2, respectively. It is important to mention that the air was well distributed and no clustering was observed.

Table 17. Air void system of mixes 346–350 (set 2—SYN AEA) measured by linear traverse.

Mix	Fresh air (ASTM C 231)	Air (%) ASTM C 457	Accum. chord length (mm)	Accum. voids counted	Mean chord length (mm)	Voids per m	Specific surface (mm ² /mm ³)	Spacing factor (mm)
346	3.2	4.4	101	632	0.16	280	25.2	0.21
347	3.5	4.6	104	642	0.16	280	25.0	0.22
348	2.3	4.2	95	352	0.27	154	15.0	0.37
349	4	4.5	101	887	0.11	388	35.3	0.15
350	4.3	5.0	114	966	0.12	423	33.8	0.15

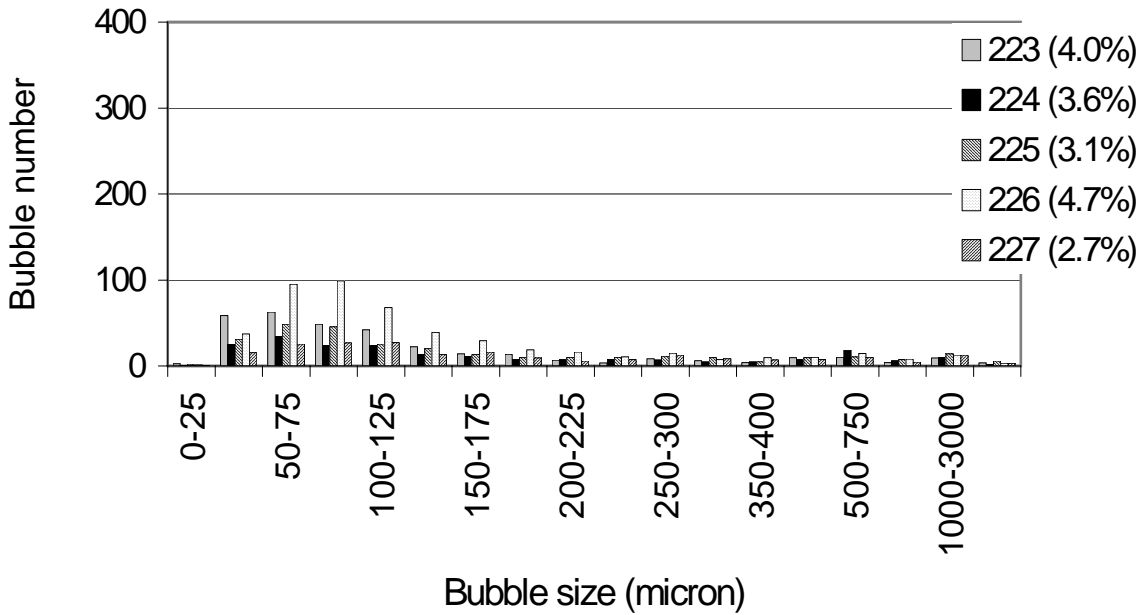


Figure 30. Graph. Bubble size distribution by C 457 (linear traverse) of set 1 with Vinsol resin admixture.

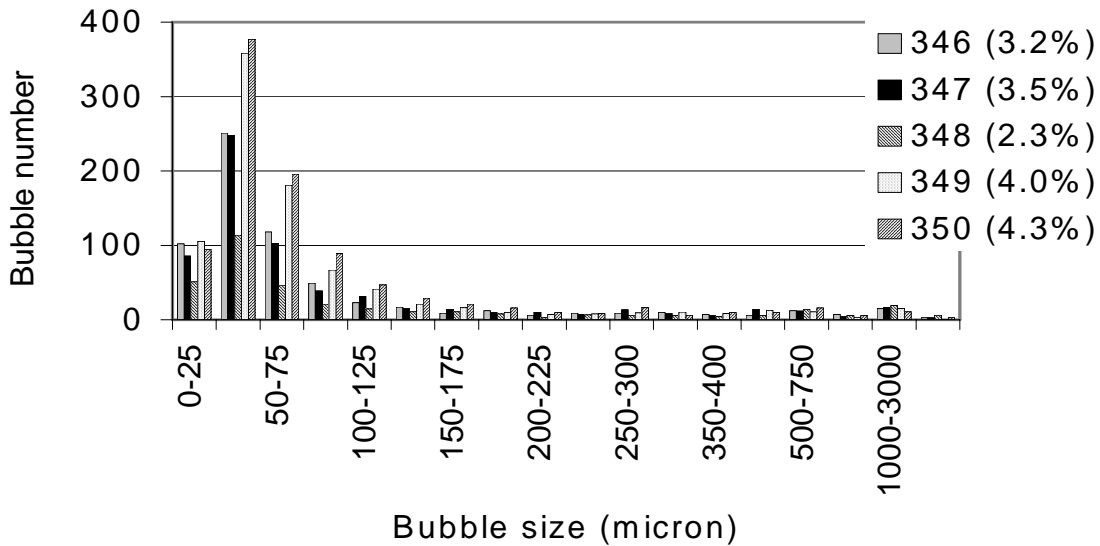


Figure 31. Graph. Bubble size distribution by C 457 (linear traverse) of set 2 with synthetic air-entraining admixture.

However, the freeze-thaw performance of set 2 was worse than that of set 1 (table 18 and figures 32–35). Only mix 350 (the highest air volume, lowest spacing factor, and highest specific surface) had a DF above 80 percent. The tables of the RDM over cycles and the combined linear traverse and point count results can be found in appendix B.

Table 18. Durability factor—results for set 2 (SYN AEA).
Results are sorted by percentage of fresh air.

Mix	Fresh air (%)	A1	A2	A3	A4	Proc A avg	Proc A std dev
348	2.3	38.3	22.2	29.4	24.9	28.7	7.1
346	3.2	66.2	46.0	56.9	53.4	55.6	8.4
347	3.5	68.0	78.3	77.1	78.8	75.6	5.1
349	4.0	82.4	62.5	50.6	66.9	65.6	13.2
350	4.3	76.6	86.2	83.1	83.5	82.3	4.1

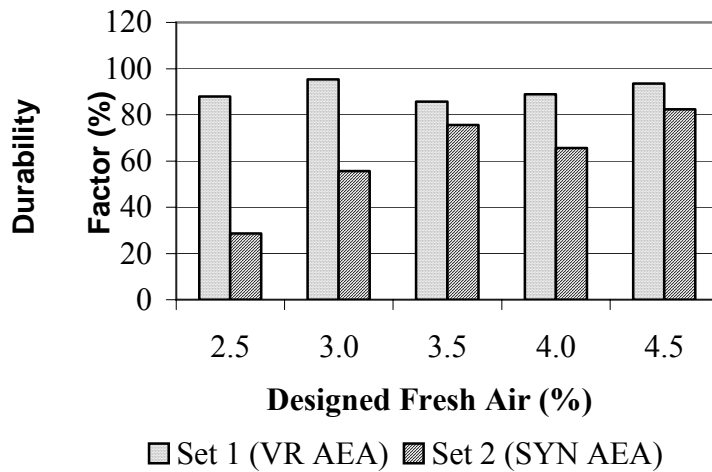


Figure 32. Graph. Comparison between mixes prepared with Vinsol resin air-entrained admixture (set 1) and synthetic air-entrained admixture (set 2).

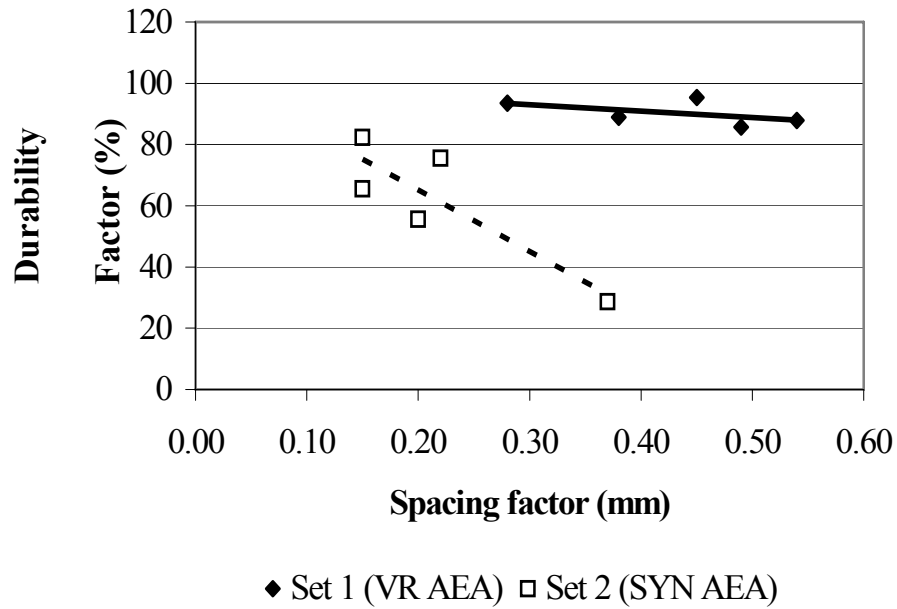


Figure 33. Graph. Relation between durability factor and spacing factor of mixes with Vinsol resin admixture (set 1) or synthetic admixture (set 2).

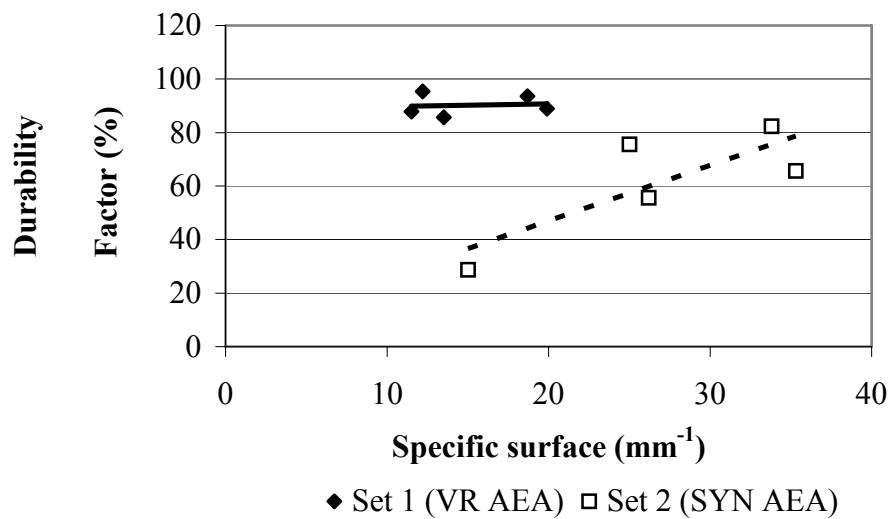


Figure 34. Graph. Relation between durability factor and specific surface of mixes with Vinsol resin admixture (set 1) or synthetic admixture (set 2).

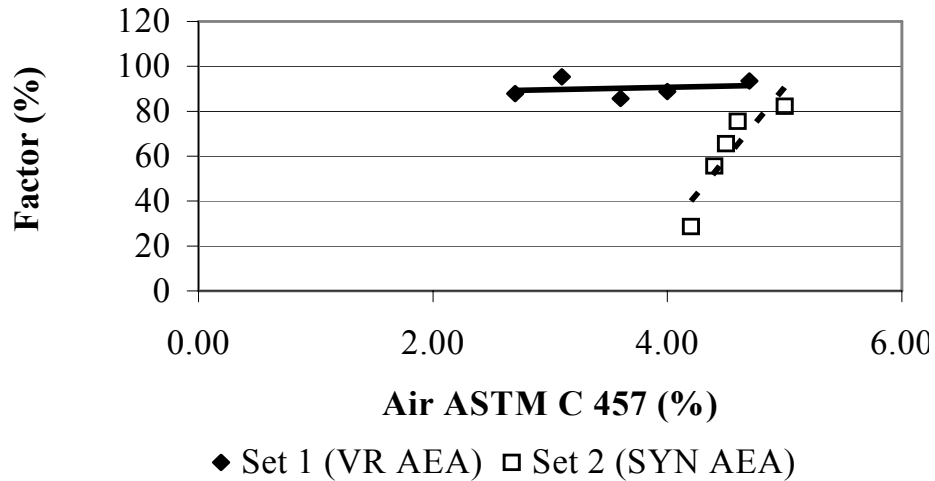


Figure 35. Graph. Relation between durability factor and hardened air content of mixes with Vinsol resin admixture (set 1) or synthetic admixture (set 2).

Figure 36 shows the DF versus fresh air for sets 1 and 2. For set 1, it can be observed that the marginal air void concretes had similar freeze-thaw resistance, but if no air entrainment is provided, the freeze-thaw resistance of the concrete is much poorer. For set 2, the higher the fresh air content (ASTM C 231⁽¹⁴⁾), the higher the DF.

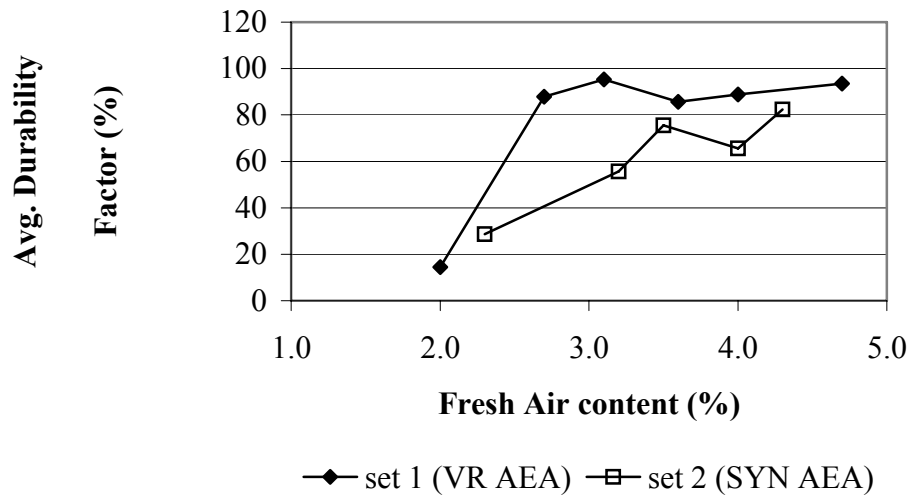


Figure 36. Graph. Relation between durability factor and fresh air content of mixes with Vinsol resin admixture (set 1) or synthetic admixture (set 2).

All specimens suffered some mass change (loss) during testing. The mass loss is a good indication of the scaling of the specimen (figure 37). Mass losses ranged from 0.32 to 4.03 percent (figure 38). Any mass gain due to water entering the concrete through cracks was obscured by the losses due to scaling. No correlation was observed between mass loss and the

freeze-thaw performance of sets 1 and 2. The mass change versus cycles can be found in appendix B.

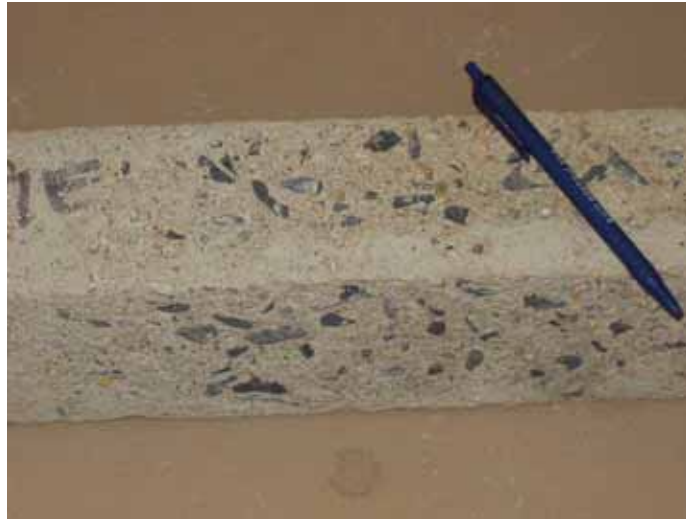


Figure 37. Photo. Scaling of typical specimen. The specimens tended to scale toward the center region of the beam specimens corresponding to the area where the metal containers bulged due to ice formation between the concrete and the container.

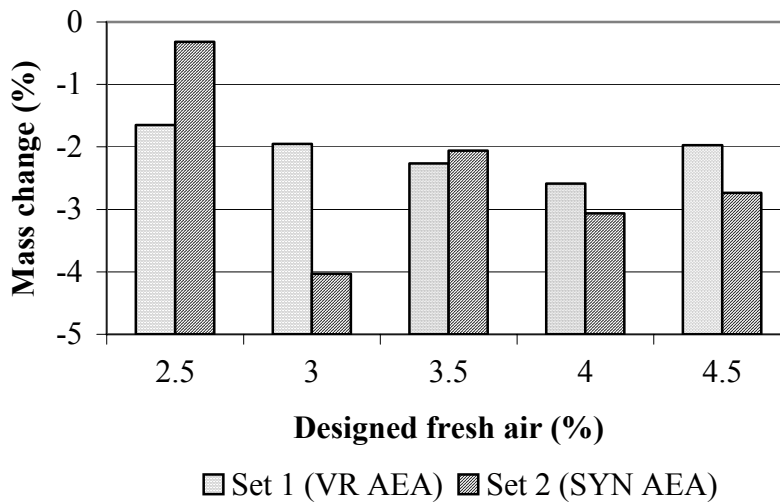


Figure 38. Graph. Mass change of mixes with Vinsol resin admixture (set 1) or synthetic admixture (set 2).

It must be pointed out that set 1 and set 2 differ only in the type of air-entraining admixture—set 1 has Vinsol resin and set 2, synthetic. For the mixes prepared in this study and for the specific

admixtures used, the Vinsol resin mixes exhibited better freeze-thaw resistance although they had a worse air void system.

The reasons for this unexpected observation are not known. It is possible that the water reducer or the cement used had an influence in the efficiency of the air void system. Another possibility is that the air-entraining admixture contains nonionic surfactants, which could result in a lack of a hydrophobic “tail” oriented towards the interior of the air bubbles, preventing water intrusion as pressure develops during freezing.⁽²⁶⁾ A previous study⁽⁴⁴⁾ showed that the cement-alkali level may have a negative impact on the air void system, and as a consequence for the freeze-thaw performance, on concretes with synthetic air-entraining admixture.

There are well-established thresholds for the air void parameters, which date from the time when only Vinsol resin admixtures were available. Experience shows that these limits ($> 6 \pm 1$ percent air, specific surface $\geq 24 \text{ mm}^2/\text{mm}^3$, and spacing factor $\leq 0.20 \text{ mm}$) would be expected to give good concrete freeze-thaw resistance. The test data presented in this chapter suggest these limits may not be adequate to assure durability for some air entrained concrete containing synthetic admixtures.

There is insufficient data in this study to generalize this finding for all Vinsol resin and synthetic admixtures and all levels of air content. More research is needed in order to confirm this finding.

CHAPTER 5: CONCLUSIONS

This study investigated the freeze-thaw resistance of several marginal air void mixes in the absence of deicing salts. The influence of water-cement ratio and the type of air-entraining admixture were evaluated.

The project was divided into two phases: 1) the first phase, designed to evaluate the w/c ratio influence on the freeze-thaw resistance of the mixes containing Vinsol resin air-entraining admixture; and 2) the second phase, designed to compare the performance of marginal air content mixes containing either Vinsol resin or synthetic air-entraining admixture.

In phase 1, the mixes had air contents that varied from 2.4 percent to 7.2 percent (measured according to ASTM C 457⁽¹⁸⁾) or 3.5 percent to 4.5 percent fresh air content (ASTM C 231⁽¹⁴⁾). The spacing factors ranged from 0.23 mm to 1.12 mm. The w/c ratios used were 0.40, 0.45, and 0.50. The mixes with fresh air contents of 3.5 percent or higher, except for mix 116-1, showed satisfactory freeze-thaw resistance, with DFs above 80 percent and lasting at least 300 cycles. No trend was observed in terms of the effect of w/c ratio on freeze-thaw resistance for the mixes investigated.

In phase 2, all mixes were designed to be in the range of marginal air (2.5 percent to 4.5 percent). Some had spacing factors and specific surface areas higher than the minimum recommended for a good freeze-thaw resistance. set 1 (with Vinsol resin admixture) showed a better freeze-thaw performance than set 2 (with synthetic admixture), although in most of the mixes of set 1 the air void system was much poorer, when measured by ASTM C 457⁽¹⁸⁾ linear traverse, with higher spacing factors and lower specific surface areas for the same levels of air contents.

In set 1 (VR AEA), all the air-entraining mixes lasted at least 300 cycles and had a DF above 80 percent. The DF did not increase with increasing specific surface, decreasing spacing factor, or increasing air content, as expected.

In set 2 (SYN AEA), only mix 350 had a DF above 80 percent. In this set, the expected trends were confirmed, i.e., the higher the spacing factor, the lower the DF; the higher the specific surface, the lower the DF. Nevertheless, no trend was found for hardened air content and DF.

For the specific materials and mix proportions used in this project, the marginal air mixes presented an adequate freeze-thaw performance when Vinsol resin based air-entraining admixture was used. The synthetic admixture used in this study did not show the same good performance as the Vinsol resin admixture. A different behavior may occur when other Vinsol and synthetic admixtures are used and higher levels of air entraining are present. The reasons for this unexpected observation could not be explained.

There are well-established thresholds for the air void parameters that would be expected to give good concrete freeze-thaw resistance. The test data presented in this study suggest these limits may not be applicable in all cases to air entrained concrete containing synthetic admixtures.

There is insufficient data in this study to generalize these results for all the Vinsol resin and synthetic air-entraining admixtures and all levels of air content. More research is needed in order to confirm this finding.

APPENDIX A

Table 19. Coarse aggregate gradations mixes 115–118.

Sieve size	Limestone (% passing)	ASTM C33
1 inch	100.0	95–100
¾ inch	92.7	
½ inch	44.1	25–60
⅜ inch	18.1	
No. 4	3.0	0–10
No. 8	1.1	

Table 20. Coarse aggregate gradations mixes 223–302.

Sieve size	Limestone (% passing)	ASTM C33
1-inch	100.0	95–100
¾-inch	92.7	
½-inch	44.1	25–60
⅜-inch	18.1	
No. 4	3.0	0–10

Table 21. Coarse aggregate gradations mixes 346–350.

Sieve size	Limestone (% passing)	ASTM C33
1-inch	92.4	95–100
¾-inch	34.9	
½-inch	9.5	25–60
⅜-inch	1.5	
No. 4	0.3	0–10

Table 22. Fine aggregate gradation mixes 115–118.

Sieve size	Percent passing	ASTM C33
#4	98.1	95–100
#8	80.4	80–100
#16	63.4	50–85
#30	33.1	25–60
#50	10.2	5–30
#100	3.4	0–10
#200	0.7	

Table 23. Fine aggregate gradation mixes 223–302.

Sieve size	Percent passing	ASTM C33
#4	98.1	95–100
#8	80.4	80–100
#16	63.4	50–85
#30	33.1	25–60
#50	10.2	5–30
#100	3.4	0–10
#200	0.7	

Table 24. Fine aggregate gradation mixes 346–350.

Sieve size	Percent passing	ASTM C33
#4	97.8	95–100
#8	86.7	80–100
#16	72.9	50–85
#30	46.3	25–60
#50	16.7	5–30
#100	4.2	0–10
#200	1.4	

Table 25. Other aggregate properties mixes 115–118.

Property	Sand	Limestone
Bulk SG (dry)	2.48	2.71
Bulk SG (SSD)	2.54	2.72
Apparent SG	2.65	2.73
Absorption (%)	1.4	0.4

Table 26. Other aggregate properties mixes 223–302.

Property	Sand	Limestone
Bulk SG (dry)	2.57	2.72
Bulk SG (SSD)	2.62	2.73
Apparent SG	2.66	2.74
Absorption (%)	1.1	0.4

Table 27. Other aggregate properties mixes 346–350.

Property	Sand	Limestone
Bulk SG (dry)	2.58	2.72
Bulk SG (SSD)	2.61	2.73
Apparent SG	2.67	2.74
Absorption (%)	1.36	0.28

Table 28. Cement composition (values in percent unless otherwise indicated) mixes 115–118.

SiO ₂	20.5
Al ₂ O ₃	4.9
Fe ₂ O ₃	3.3
CaO	62.2
MgO	3.5
SO ₃	3.0
Na ₂ O eq.	0.6
Loss on ignition (LOI)	1.5
Insoluble residue	0.25

Table 29. Additional cement properties mixes 115–118.

Potential compounds	C ₃ S (%)	51
	C ₃ A (%)	7
Fineness, Blaine (m²/kg)		375
Soundness, autoclave expansion (%)		0.120
Time of setting, Vicat (minutes)		
Initial		160
Final		265
Air content (%)		7.3
Compressive strength (MPa)		
3 days		27.0
7 days		36.3
28 days		48.6

Table 30. Cement composition (values in percent unless otherwise indicated) mixes 223–302.

SiO ₂	20.5
Al ₂ O ₃	4.9
Fe ₂ O ₃	3.3
CaO	62.2
MgO	3.5
SO ₃	3.0
Na ₂ O eq.	0.60
LOI	1.5
Insoluble residue	0.25

Table 31. Additional cement properties mixes 223–302.

Potential compounds	C ₃ S (%)	51
	C ₃ A (%)	7
Fineness, Blaine (m²/kg)		375
Soundness, autoclave expansion (%)		0.10
Time of setting, Vicat (minutes)		
Initial		150
Final		270
Air content (%)		6.8
Compressive strength (MPa)		
3 days		23.3
7 days		31.0
28 days		43.5

Table 32. Cement composition (values in percent unless otherwise indicated) mixes 346–350.

SiO ₂	20.9
Al ₂ O ₃	4.4
Fe ₂ O ₃	3.0
CaO	62.8
MgO	3.5
SO ₃	3.0
Na ₂ O eq.	0.58
LOI	1.1
Insoluble residue	0.23

Table 33. Additional cement properties mixes 346–350.

Potential compounds	C₃S (%)	54
	C₃A (%)	7
Fineness, Blaine (m²/kg)		366
Soundness, autoclave expansion (%)		0.12
Time of setting, Vicat (minutes)		
	Initial	160
	Final	265
Air content (%)		7.3
Compressive strength (MPa)		
	3 days	27.0
	7 days	36.3
	28 days	48.6

APPENDIX B

Table 34. Mix 115-1—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	94.8	95.6	94.0	96.5	95.2	1.09
32	94.0	91.3	94.8	94.8	93.7	1.65
50	93.1	93.0	93.1	93.0	93.1	0.05
69	93.1	93.0	92.3	92.2	92.6	0.48
84	92.3	91.3	91.4	91.3	91.6	0.45
103	91.4	89.6	90.6	89.7	90.3	0.86
132	91.4	88.8	89.8	89.7	89.9	1.10
141	91.4	88.8	90.6	89.7	90.1	1.14
161	90.6	88.8	90.6	88.8	89.7	1.03
174	90.6	87.9	89.8	88.8	89.3	1.14
196	77.7	86.3	88.9	88.0	85.2	5.13
220	91.4	85.5	88.9	88.0	88.4	2.46
253	88.1	83.8	87.3	87.2	86.6	1.89
288	88.1	82.2	86.5	84.7	85.4	2.52
300	88.1	80.6	84.0	83.9	84.2	3.07

Table 35. Mix 115-2—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	94.8	94.8	94.0	94.0	94.4	0.48
32	94.0	93.1	94.0	97.4	94.6	1.91
50	92.3	92.3	92.3	93.1	92.5	0.44
69	91.4	91.4	92.3	93.1	92.1	0.82
84	90.6	91.4	92.3	91.5	91.4	0.69
103	90.6	90.6	92.3	91.5	91.2	0.81
132	89.8	88.9	92.3	90.6	90.4	1.43
141	90.6	89.8	93.1	92.3	91.4	1.54
161	88.9	88.9	92.3	89.8	90.0	1.58
174	88.1	87.3	92.3	89.0	89.2	2.19
196	88.1	87.3	91.4	87.3	88.5	1.96
220	88.1	87.3	91.4	87.3	88.5	1.96
253	84.0	83.2	89.8	85.7	85.7	2.91
288	81.6	79.3	86.5	81.7	82.3	3.01
300	80.1	75.4	84.0	81.7	80.3	3.64

Table 36. Mix 115-3—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	94.9	94.9	94.9	94.9	94.9	0.03
32	95.7	94.1	94.1	94.9	94.7	0.79
50	94.9	93.3	93.3	94.0	93.9	0.78
69	93.2	92.4	92.4	94.0	93.0	0.76
84	92.4	91.6	92.4	94.0	92.6	1.01
103	92.4	91.6	92.4	94.0	92.6	1.01
132	91.6	90.8	91.6	93.2	91.8	1.00
141	91.6	90.8	91.6	93.2	91.8	1.00
161	89.9	89.2	89.2	91.5	89.9	1.10
174	89.1	88.3	90.0	92.3	89.9	1.73
196	89.1	86.7	90.0	91.5	89.3	1.99
220	89.1	86.7	90.0	88.2	88.5	1.38
253	88.3	84.4	80.5	90.7	85.9	4.49
288	85.9	82.0	87.5	89.8	86.3	3.30
300	85.1	81.2	87.5	89.0	85.7	3.40

Table 37. Mix 116-1—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
18	87.4	89.1	89.8	87.5	88.5	1.20
37	84.2	88.3	84.2	80.4	84.3	3.23
50	94.9	82.7	77.1	70.6	81.3	10.28
84	63.4	69.9	63.3	56.3	63.2	5.59
103	48.0	55.6	55.9	46.4	51.5	5.00

Table 38. Mix 116-2—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
18	90.1	91.8	92.5	92.5	91.7	1.13
37	87.7	90.2	89.3	89.3	89.1	1.01
50	85.4	85.4	85.3	86.1	85.6	0.37
84	80.0	75.6	75.5	80.7	78.0	2.79
103	70.5	61.1	61.4	72.5	66.4	5.98
112	65.6	52.2	56.3	69.0	60.8	7.84

Table 39. Mix 116-3—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
18	89.0	89.9	90.6	92.3	90.5	1.41
37	85.8	88.2	88.9	90.7	88.4	2.03
50	85.0	87.4	87.3	89.0	87.2	1.67
84	84.2	85.8	84.8	85.8	85.1	0.80
103	82.6	83.4	80.1	83.4	82.4	1.58
112	77.1	76.5	84.8	86.6	81.3	5.20
132	74.9	79.5	74.7	79.4	77.1	2.73
145	71.1	76.5	69.4	76.4	73.4	3.60
167	64.7	71.3	59.6	70.4	66.5	5.45
191	52.7	62.1	51.1	64.7	57.7	6.74
224		43.9		46.6	45.2	1.93

Table 40. Mix 117-1—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	94.6	103.8	94.6	94.6	96.9	4.61
38	93.7	101.9	93.8	93.7	95.8	4.08
47	92.9	103.8	93.8	93.7	96.1	5.21
67	94.6	101.0	93.8	92.9	95.5	3.67
80	94.6	100.0	93.8	93.7	95.5	3.01
93	94.6	101.9	93.8	93.7	96.0	3.96
102	93.7	100.0	93.8	92.9	95.1	3.30
126	94.6	101.0	93.8	93.7	95.8	3.48
135	93.7	100.0	93.8	92.9	95.1	3.30
159	93.7	100.0	93.8	91.1	94.7	3.77
194	92.9	100.0	92.9	90.3	94.0	4.19
220	92.0	99.0	92.0	88.5	92.9	4.42
255	91.1	97.2	92.0	87.7	92.0	3.92
274	92.0	96.2	90.3	85.1	90.9	4.59
303	91.1	95.3	90.3	81.8	89.6	5.65

Table 41. Mix 117-2—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	63.9	55.6	64.4	50.7	58.6	6.68
38	34.2	24.6	26.6	20.1	26.4	5.88

Table 42. Mix 117-3—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	94.6	95.6	94.7	95.6	95.1	0.52
38	93.8	94.7	93.9	94.7	94.3	0.53
47	93.8	94.7	93.9	95.6	94.5	0.86
67	94.6	94.7	93.0	95.6	94.5	1.08
80	93.8	93.8	93.0	94.7	93.8	0.71
93	94.6	94.7	93.0	95.6	94.5	1.08
102	93.8	93.8	91.3	94.7	93.4	1.47
126	93.8	93.8	92.2	79.0	89.7	7.16
135	92.9	93.0	91.3	95.6	93.2	1.78
159	92.0	92.1	90.5	94.7	92.3	1.77
194	92.0	93.0	90.5	95.6	92.8	2.16
220	91.2	91.3	89.6	94.7	91.7	2.16
255	91.2	91.3	88.8	94.7	91.5	2.45
274	91.2	91.3	87.9	93.9	91.1	2.43
303	91.2	91.3	87.1	93.9	90.9	2.79

Table 43. Mix 117-4—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
19	95.6	96.5	96.5	96.5	96.3	0.43
38	94.8	95.6	96.5	96.5	95.8	0.83
47	95.6	95.6	96.5	96.5	96.0	0.51
67	94.8	95.6	95.6	96.5	95.6	0.71
80	94.8	95.6	95.6	96.5	95.6	0.71
93	94.8	95.6	95.6	97.4	95.8	1.10
102	93.9	95.6	94.7	97.4	95.4	1.49
126	94.8	94.7	94.7	98.2	95.6	1.76
135	93.9	95.6	93.8	97.4	95.2	1.67
159	93.9	94.7	93.0	96.5	94.5	1.50
194	93.9	95.6	92.1	96.5	94.5	1.93
220	93.0	94.7	90.4	95.6	93.5	2.29
255	92.2	93.0	93.0	95.6	93.5	1.51
274	91.3	93.0	87.9	94.8	91.8	2.93
303	90.5	91.3	85.4	93.9	90.3	3.57

Table 44. Mix 118-1—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
9	95.4	97.3	96.4	95.5	96.1	0.87
29	95.4	97.3	96.4	95.5	96.1	0.87
42	95.4	97.3	96.4	95.5	96.1	0.87
55	96.3	97.3	97.3	95.5	96.6	0.86
64	95.4	97.3	96.4	95.5	96.1	0.87
88	96.3	98.2	97.3	95.5	96.8	1.17
97	94.5	97.3	97.3	95.5	96.1	1.36
121	95.4	97.3	97.3	95.5	96.4	1.05
160	95.4	97.3	97.3	95.5	96.4	1.05
192	94.5	96.4	97.3	96.4	96.1	1.14
217	94.5	96.4	97.3	96.4	96.1	1.14
236	93.7	96.4	97.3	95.5	95.7	1.54
265	93.7	95.5	97.3	95.5	95.5	1.48
300	92.8	95.5	96.4	94.6	94.8	1.54

Table 45. Mix 118-2—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
9	95.7	96.5	96.5	96.5	96.3	0.41
29	95.7	95.7	95.7	95.7	95.7	0.02
42	95.7	96.5	96.5	95.7	96.1	0.49
55	96.6	96.5	96.5	96.5	96.5	0.01
64	95.7	96.5	95.7	96.5	96.1	0.49
88	95.7	96.5	96.5	96.5	96.3	0.41
97	95.7	96.5	95.7	95.7	95.9	0.43
121	94.9	95.7	93.1	93.1	94.2	1.29
160	94.9	95.7	94.8	94.8	95.0	0.43
192	94.0	95.7	94.8	94.0	94.6	0.81
217	94.0	96.5	94.0	94.0	94.6	1.28
236	93.2	95.7	94.0	94.0	94.2	1.06
265	92.3	95.7	93.1	93.1	93.6	1.46
300	91.5	94.8	92.3	92.3	92.7	1.46

Table 46. Mix 118-3—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
9	95.8	95.0	95.0	95.8	95.4	0.47
29	95.8	95.8	95.0	95.0	95.4	0.49
42	95.8	95.8	95.0	94.1	95.2	0.81
55	95.8	95.8	95.0	94.1	95.2	0.81
64	94.2	95.8	94.2	94.1	94.6	0.82
88	95.8	96.7	94.2	95.0	95.4	1.06
97	95.8	96.7	94.2	95.0	95.4	1.06
121	96.7	96.7	92.6	95.0	95.2	1.93
160	95.8	97.5	92.6	92.5	94.6	2.48
192	95.0	96.7	91.8	92.5	94.0	2.26
217	95.0	97.5	91.0	93.3	94.2	2.75
236	94.2	97.5	90.2	92.5	93.6	3.08
265	93.4	96.7	89.4	92.5	93.0	2.99
300	92.6	96.7	89.4	90.9	92.4	3.14

Table 47. Mix 223—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	100.0	0.00
9	96.7	94.1	97.7	96.3	97.5	96.5	1.45
23	96.3	95.9	96.8	95.9	96.6	96.3	0.39
32	95.9	95.2	96.8	95.0	95.9	95.8	0.71
42	95.9	94.9	96.6	94.7	95.8	95.6	0.79
59	94.2	93.0	94.8	91.7	95.4	93.8	1.51
70	95.1	93.8	95.6	92.5	95.3	94.5	1.30
84	95.2	94.5	96.1	92.9	95.7	94.9	1.27
98	95.3	94.3	95.8	92.6	95.6	94.7	1.31
112	95.5	94.3	95.8	92.5	95.6	94.7	1.41
121	95.7	94.3	96.0	92.5	95.7	94.8	1.47
146	95.4	94.2	95.6	92.2	95.9	94.7	1.51
165	94.6	93.8	94.8	92.0	95.2	94.1	1.27
179	94.6	93.5	95.2	91.7	95.6	94.1	1.53
198	93.5	93.0	94.2	90.0	94.5	93.0	1.82
213	92.7	91.7	92.7	88.7	93.3	91.8	1.86
227	92.0	90.8	91.6	88.1	92.9	91.1	1.85
246	91.6	90.3	91.6	87.7	92.8	90.8	1.96
269	90.7	89.3	90.6	86.2	92.6	89.9	2.38
282	90.3	88.6	90.0	85.2	92.3	89.3	2.61
301	89.6	88.5	89.7	84.4	92.0	88.9	2.79

Table 48. Mix 224—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	100.0	0.00
9	96.2	96.8	97.2	97.6	96.8	97.0	0.57
23	96.1	96.4	96.8	96.6	96.8	96.5	0.30
32	95.8	96.4	95.9	96.0	95.9	96.0	0.25
42	95.7	95.6	95.6	95.6	95.8	95.6	0.06
59	95.7	94.9	95.1	94.4	95.5	95.0	0.53
70	95.1	94.5	94.2	94.4	94.8	94.5	0.37
84	95.1	94.6	94.6	93.9	95.0	94.6	0.52
98	95.1	94.6	93.9	93.2	94.4	94.2	0.82
112	95.1	94.0	93.9	93.1	94.5	94.0	0.84
121	95.1	93.7	94.0	92.7	94.0	93.9	1.01
146	94.5	92.9	93.2	91.6	94.1	93.0	1.20
165	94.4	93.2	92.9	90.6		92.8	1.57
179	92.6	92.8	92.4	90.1		92.0	1.28
198	93.0	91.9	91.5	88.5		91.2	1.91
213	91.8	90.8	90.8	88.0		90.4	1.62
227	90.3	89.5	89.4	86.9		89.0	1.50
246	89.5	89.1	88.8	86.6		88.5	1.30
269	87.7	87.7	86.9	86.2		87.1	0.76
282	86.6	87.3	86.5	85.3		86.4	0.86
301	85.7	87.2	85.4	84.4		85.7	1.18

Table 49. Mix 225—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	100.0	0.00
6	97.2	97.8	97.3	97.5	97.3	97.4	0.24
20	96.2	98.1	97.9	97.4	97.4	97.4	0.73
26	97.5	98.7	97.8	97.5	97.5	97.8	0.52
35	96.9	97.9	97.0	96.8	96.8	97.1	0.47
49	96.9	98.1	97.0	97.3	96.9	97.2	0.50
58	97.0	97.9	96.7	97.5	96.8	97.2	0.49
83	96.8	97.9	96.1	97.6	96.6	97.0	0.72
102	96.4	97.4	95.8	97.1	96.1	96.5	0.68
116	96.3	97.6	95.8	97.3	96.0	96.6	0.81
135	95.1	96.8	94.7	96.5	95.3	95.7	0.93
150	95.0	96.5	94.6	95.5	94.0	95.1	0.92
164	94.1	95.6	93.6	95.4	93.7	94.5	0.98
183	94.1	95.7	93.1	94.9	93.1	94.2	1.13
206	93.4	94.6	92.1	94.1	92.5	93.3	1.06
219	92.8	94.2	91.8	93.7	91.7	92.8	1.09
238	92.3	93.6	91.9	92.9	90.9	92.3	1.01
252	92.0	92.9	91.4	92.4	90.2	91.8	1.04
271	90.9	91.9	91.1	92.0	90.1	91.2	0.80
286	90.5	92.1	90.8	91.5	90.3	91.0	0.76
305	89.4	90.5	90.0	90.8	88.2	89.8	1.05

Table 50. Mix 226—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	100.0	0.00
6	94.9	97.0	96.2	97.3	97.1	96.5	1.01
15	94.2	96.3	97.1	96.8	96.7	96.2	1.18
29	94.5	96.8	97.0	96.8	97.1	96.4	1.09
38	94.9	96.7	96.8	97.0	97.1	96.5	0.92
63	94.9	97.0	97.0	97.2	97.3	96.7	1.02
82	94.7	97.1	96.6	97.1	97.3	96.5	1.07
96	95.1	97.3	96.6	97.2	97.4	96.7	0.96
115	94.2	96.4	95.9	96.8	96.8	96.0	1.08
130	93.3	95.5	95.4	96.3	96.5	95.4	1.26
144	93.2	95.4	94.9	95.9	96.2	95.1	1.17
163	93.5	95.9	95.5	96.0	96.2	95.4	1.09
186	93.4	96.0	94.8	95.8	96.3	95.3	1.19
199	93.5	95.8	94.4	95.6	96.1	95.1	1.09
218	93.4	95.8	94.5	95.7	96.2	95.1	1.15
232	93.0	95.3	94.1	95.6	95.7	94.8	1.19
251	93.3	95.6	94.1	95.4	96.0	94.9	1.13
266	92.9	95.5	93.7	95.2	95.7	94.6	1.23
285	92.9	95.0	93.4	95.0	95.8	94.4	1.23
304	92.1	94.0	93.0	95.0	95.3	93.9	1.36

Table 51. Mix 227—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.00	100.00	100.00	100.00	100.00	100.0	0.00
6	97.04	96.44	96.84	97.56	96.41	96.9	0.48
15	96.61	96.06	96.33	96.59	95.98	96.3	0.29
29	96.44	96.52	96.16	96.93	95.90	96.4	0.39
38	96.35	95.81	95.91	96.42	95.10	95.9	0.53
63	96.31	96.02	96.16	96.84	95.90	96.2	0.37
82	96.18	95.60	96.04	96.50	96.02	96.1	0.33
96	96.10	95.56	95.91	96.50	96.15	96.0	0.35
115	95.25	94.93	95.03	95.70	95.18	95.2	0.30
130	94.45	94.27	94.40	94.99	94.47	94.5	0.28
144	93.49	93.44	93.56	94.28	94.17	93.8	0.40
163	93.28	93.15	93.48	94.36	93.71	93.6	0.48
186	92.19	92.70	92.94	93.40	93.42	92.9	0.51
199	91.78	92.41	92.40	92.90	93.04	92.5	0.50
218	90.99	92.12	91.49	92.49	92.92	92.0	0.77
232	90.66	91.38	90.92	91.87	68.91	86.7	9.98
251	89.92	91.30	90.79	91.83	74.61	87.7	7.35
266	89.27	90.85	89.49	91.17	70.85	86.3	8.69
285	88.90	90.32	88.07	90.65	80.66	87.7	4.08
304	86.67	88.06	86.82	89.86	76.75	85.6	5.13

Table 52. Mix 302—RDM versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	100.0	0.00
10	92.6	90.2	95.1	92.4	92.2	92.5	1.74
24	82.0	83.9	90.8	81.6	87.6	85.2	3.92
31	79.0	79.7	87.1	75.9	85.7	81.5	4.76
44	79.3	73.4	87.6	72.3	84.2	79.4	6.65
52	71.1	74.9	83.9	69.5	81.7	76.2	6.35
62	66.0	67.9	76.4	58.6	74.6	68.7	7.11
72	61.2	61.6	71.9		66.0	65.2	4.99
86	50.3	50.5	58.2		53.7	53.2	3.73

Table 53. Mix 346—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
9	94.3	95.2	94.7	94.9	94.8	0.38
22	94.1	94.0	94.0	93.7	94.0	0.17
31	93.9	93.2	93.5	93.9	93.6	0.34
43	93.0	91.2	93.4	93.8	92.9	1.15
53	92.5	91.6	92.9	93.5	92.6	0.82
69	90.9	90.3	91.7	93.3	91.5	1.32
83	90.1	89.8	90.4	92.2	90.6	1.10
92	90.6	87.9	90.1	91.6	90.1	1.54
101	89.6	87.7	89.5	90.7	89.4	1.22
119	87.9	86.8	87.8	88.8	87.8	0.82
133	87.6	83.7	87.5	86.8	86.4	1.84
150	86.8	83.3	85.3	86.8	85.6	1.64
165	85.2	83.2	84.0	86.1	84.6	1.26
179	83.6	81.4	81.6	85.4	83.0	1.90
194	83.0	81.2	79.2	83.1	81.6	1.81
204	81.5	76.8	77.0	81.6	79.2	2.71
216	80.0	78.7	77.8	82.7	79.8	2.15
230	76.1	73.5	71.2	77.2	74.5	2.70
248	74.9	71.9	70.3	76.1	73.3	2.69
266	70.1	67.2	66.1	69.9	68.3	1.99
287	69.0	62.2	62.5	66.3	65.0	3.24
305	66.2	46.0	56.9	53.4	55.6	8.38
311	61.8				61.8	
329	58.9				58.9	

Table 54. Mix 347—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
17	95.1	95.4	94.9	94.7	95.0	0.30
26	94.9	95.2	94.9	94.5	94.9	0.29
38	94.8	94.7	94.6	94.0	94.5	0.36
48	94.4	94.7	94.3	94.3	94.4	0.19
64	94.3	94.7	93.7	92.7	93.9	0.87
78	92.4	94.1	92.6	93.1	93.1	0.76
87	93.2	92.9	92.7	88.9	91.9	2.03
96	93.0	92.9	91.9	92.2	92.5	0.54
114	92.4	92.9	93.1	91.9	92.6	0.54
128	91.8	91.9	90.0	91.0	91.2	0.88
145	91.9	91.0	90.0	90.0	90.7	0.91
160	91.1	91.0	89.4	88.7	90.1	1.19
174	90.2	89.9	88.8	88.5	89.4	0.83
189	89.5	89.4	87.4	88.1	88.6	1.02
199	87.7	88.5	86.4	87.2	87.5	0.88
211	87.4	88.1	88.3	86.5	87.6	0.81
225	83.6	86.9	84.3	85.3	85.0	1.43
243	81.0	84.1	83.7	84.0	83.2	1.48
261	78.0	83.8	82.6	82.9	81.8	2.60
282	75.6	82.1	80.6	81.6	80.0	2.98
300	68.0	78.3	77.1	78.8	75.6	5.08
306	67.5	78.2	77.7	78.3	75.4	5.29
324	62.8	73.9	75.2	77.0	72.2	6.41
337	59.4	71.0	70.8	76.1	69.3	7.06
355		66.6	70.6	50.4	62.5	10.70
369		66.9	65.6		66.3	0.92
384		64.5	65.8		65.2	0.92
403		58.7	61.6		60.2	2.05
412			60.2		60.2	
437			48.2		48.2	

Table 55. Mix 348—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
11	94.7	95.3	94.7	96.0	95.2	0.64
25	94.6	94.5	92.6	95.4	94.3	1.20
34	94.3	94.2	93.1	93.9	93.9	0.56
43	94.3	92.4	93.1	93.3	93.3	0.79
61	94.0	87.1	90.4	90.8	90.6	2.82
75	93.1	82.8	87.3	90.8	88.5	4.48
92	91.8	78.1	82.6	88.9	85.4	6.17
107	88.8	72.6	80.8	86.3	82.1	7.19
121	86.1	55.1	78.6	80.5	75.1	13.69
136	82.0		70.9	63.2	72.0	9.45
146	77.7		65.1	51.1	64.7	13.29
158	76.1		59.3		67.7	11.91
172	67.7		51.3		59.5	11.61
190	62.2				62.2	
208	55.3				55.3	

Table 56. Mix 349—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
11	96.2	95.1	95.8	96.3	95.8	0.55
25	96.1	94.1	95.6	100.0	96.5	2.48
34	96.0	95.0	95.9	95.9	95.7	0.45
43	95.7	94.3	95.3	95.1	95.1	0.57
61	95.9	94.1	95.7	95.7	95.4	0.83
75	95.8	94.3	95.5	95.0	95.1	0.64
92	95.3	93.6	95.0	93.7	94.4	0.87
107	95.0	92.9	94.5	93.8	94.1	0.92
121	94.8	92.3	92.1	92.7	93.0	1.24
136	93.5	91.5	90.6	91.4	91.7	1.21
146	92.3	89.7	88.9	90.4	90.3	1.45
158	91.6	90.2	88.3	90.2	90.1	1.34
172	86.2	89.1	86.5	89.0	87.7	1.58
190	85.1	83.8	83.0	89.4	85.3	2.83
208	85.4	79.4	76.3	84.5	81.4	4.35
229	85.7	75.5	71.7	80.4	78.3	6.07
247	85.2	68.3	63.5	76.5	73.4	9.56
253	85.8	66.2	60.0	73.8	71.5	11.14
271	85.3	63.9		71.9	73.7	10.79
284	84.5	63.8		66.7	71.7	11.22
302	82.4	62.5		66.9	70.6	10.44
316	83.4	62.4		66.9	70.9	11.04
331	82.2	64.4		60.9	69.2	11.39
350	80.1	63.4		59.7	67.7	10.84
359	79.9	62.6		59.4	67.3	11.02
384	72.4	60.7			66.6	8.24
393	67.3	53.8			60.6	9.54
403	66.0				66.0	

Table 57. Mix 350—RDM versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	100.0	100.0	100.0	100.0	100.0	0.00
18	97.0	95.4	96.3	96.1	96.2	0.62
27	97.3	96.0	96.6	96.3	96.6	0.54
36	96.4	95.6	96.0	95.9	96.0	0.36
54	96.2	96.6	96.2	95.3	96.1	0.54
68	96.6	96.1	94.9	94.3	95.5	1.06
85	96.6	96.4	94.2	94.1	95.3	1.38
100	96.4	96.0	92.2	93.7	94.6	1.97
114	95.0	95.3	91.6	93.4	93.8	1.73
129	93.7	95.2	90.5	92.9	93.1	1.99
139	92.5	93.7	89.4	92.0	91.9	1.78
151	91.8	93.6	89.6	92.1	91.8	1.65
165	87.6	92.5	87.5	90.8	89.6	2.49
183	85.6	91.9	86.1	90.0	88.4	3.06
201	85.0	91.3	86.9	90.6	88.5	2.99
222	84.2	90.6	86.5	89.4	87.7	2.89
240	83.4	89.3	85.2	87.6	86.4	2.62
246	83.6	89.0	85.6	87.3	86.4	2.31
264	81.3	87.5	85.5	85.9	85.0	2.64
277	81.0	86.5	84.3	84.5	84.1	2.28
295	77.7	83.8	83.0	84.4	82.2	3.08
309	76.6	86.2	83.1	83.5	82.3	4.07
324	75.4	85.9	82.6	81.5	81.3	4.37
343	72.7	85.2	80.9	81.4	80.0	5.27
352	71.7	82.8	80.8	81.1	79.1	5.01
377	60.3	79.9	75.9	77.3	73.4	8.87
386	52.0	77.5	73.1	77.4	70.0	12.15
396		76.2	72.6	77.3	75.4	2.45

Table 58. Mix 115-1—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	0.00	-0.34	-0.04	-0.06	-0.11	0.16
32	-0.06	-0.44	-0.11	-0.13	-0.18	0.17
50	-0.17	-0.65	-0.27	-0.27	-0.34	0.21
69	-0.26	-0.84	-0.40	-0.46	-0.49	0.25
84	-0.36	-0.98	-0.52	-0.64	-0.62	0.27
103	-0.47	-1.25	-0.66	-0.77	-0.79	0.33
132	-0.57	-1.47	-0.91	-1.06	-1.00	0.37
141	-0.79	-1.55	-0.97	-1.12	-1.11	0.33
161	-1.03	-1.90	-1.33	-1.52	-1.44	0.37
174	-1.21	-2.19	-1.55	-1.80	-1.69	0.41
196	-1.49	-2.58	-2.08	-2.26	-2.10	0.46
220	-1.86	-3.12	-2.56	-2.83	-2.59	0.54
253	-1.49	-2.58	-2.08	-2.26	-2.10	0.46
288	-2.53	-4.02	-3.69	-3.71	-3.49	0.65
300	-2.68	-4.22	-3.87	-3.87	-3.66	0.67

Table 59. Mix 115-2—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	-0.11	-0.13	-0.03	-0.10	-0.09	0.04
32	-0.16	-0.17	-0.06	-0.18	-0.14	0.05
50	-0.30	-0.33	-0.12	-0.25	-0.25	0.09
69	-0.44	-0.41	-0.19	-0.32	-0.34	0.11
84	-0.52	-0.44	-0.16	-0.44	-0.39	0.16
103	-0.63	-0.42	-0.26	-0.54	-0.46	0.16
132	-0.84	-0.69	-0.55	-0.78	-0.72	0.13
141	-0.91	-0.74	-0.73	-0.84	-0.80	0.08
161	-1.17	-0.95	-1.12	-1.27	-1.13	0.13
174	-1.31	-1.12	-1.37	-1.55	-1.34	0.18
196	-1.86	-1.39	-1.74	-1.98	-1.74	0.25
220	-2.19	-1.82	-2.16	-2.44	-2.15	0.25
253	-2.75	-2.18	-2.43	-2.94	-2.57	0.34
288	-3.33	-2.61	-2.82	-3.50	-3.07	0.42
300	-3.51	-2.82	-3.04	-3.74	-3.28	0.42

Table 60. Mix 115-3—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	-0.08	-0.15	-0.02	-0.20	-0.11	0.08
32	-0.10	-0.28	-0.04	-0.26	-0.17	0.12
50	-0.14	-0.36	-0.10	-0.37	-0.24	0.14
69	-0.25	0.01	-0.16	-0.46	-0.21	0.20
84	-0.27	-0.40	-0.19	-0.51	-0.34	0.14
103	-0.34	-0.45	-0.20	-0.55	-0.38	0.15
132	-0.50	-0.36	-0.37	-0.74	-0.49	0.18
141	-0.70	-0.60	-0.38	-0.80	-0.62	0.18
161	-0.96	-0.78	-0.60	-0.97	-0.83	0.18
174	-1.26	-0.87	-0.74	-1.18	-1.01	0.25
196	-1.71	-1.08	-1.01	-1.40	-1.30	0.32
220	-2.34	-1.29	-1.29	-1.69	-1.65	0.49
253	-2.74	-1.95	-1.61	-1.99	-2.07	0.48
288	-3.09	-2.45	-1.95	-2.44	-2.48	0.47
300	-3.19	-2.64	-2.11	-2.59	-2.63	0.44

Table 61. Mix 116-1—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
18	-0.01	0.06	-0.01	0.01	0.01	0.04
37	-0.04	-0.04	-0.08	0.00	-0.04	0.03
50	-0.15	-0.14	-0.17	-0.06	-0.13	0.05
84	-0.30	-0.15	-0.20	-0.16	-0.20	0.07
103	-0.72	-0.52	-0.65	-0.76	-0.66	0.11

Table 62. Mix 116-2—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
18	0.03	-0.02	-0.01	0.00	0.00	0.02
37	0.00	-0.08	-0.01	-0.01	-0.03	0.04
50	-0.36	-0.11	0.03	-0.05	-0.12	0.17
84	-0.34	-0.17	0.06	0.01	-0.11	0.18
103	-0.49	-0.47	-0.02	-0.08	-0.26	0.25
112	-0.51	-0.53	-0.03	-0.12	-0.30	0.26

Table 63. Mix 116-3—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
18	-0.01	-0.02	-0.05	-0.02	-0.02	0.02
37	-0.10	-0.12	-0.14	-0.07	-0.11	0.03
50	-0.21	-0.20	0.25	-0.28	-0.11	0.24
84	-0.33	-0.33	-0.28	-0.06	-0.25	0.13
103	-0.56	-0.55	-0.53	-0.23	-0.47	0.16
112	-0.70	-0.65	-0.62	-0.30	-0.57	0.18
132	-1.19	-1.00	-1.07	-0.55	-0.96	0.28
145	-1.61	-1.42	-1.44	-0.81	-1.32	0.35
167	-2.26	-2.11	-2.12	-1.33	-1.95	0.42
191	-2.95	-2.94	-2.75	-1.76	-2.60	0.57
224		-4.06		-2.80	-3.43	0.89

Table 64. Mix 117-1—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	0.02	-0.01	-0.01	0.00	0.00	0.01
38	-0.11	-0.23	-0.15	-0.13	-0.15	0.05
47	-0.20	-0.33	-0.20	-0.23	-0.24	0.06
67	-0.32	-0.63	-0.35	-0.49	-0.45	0.14
80	-0.44	-0.95	-0.50	-0.79	-0.67	0.24
93	-0.53	-1.28	-0.69	-1.12	-0.90	0.35
102	-0.61	-1.46	-0.88	-1.36	-1.08	0.40
126	-0.90	-2.16	-1.18	-1.74	-1.50	0.56
135	-0.97	-2.30	-1.28	-1.88	-1.61	0.59
159	-1.23	-2.68	-1.48	-2.23	-1.91	0.67
194	-1.53	-3.08	-1.93	-2.75	-2.32	0.72
220	-1.93	-3.51	-2.28	-3.25	-2.74	0.76
255	-2.15	-3.78	-2.57	-3.71	-3.05	0.82
274	-2.35	-4.17	-2.80	-3.99	-3.33	0.89
303	-2.51	-4.36	-2.94	-4.33	-3.53	0.95

Table 65. Mix 117-2—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	0.24	0.23	0.23	0.31	0.25	0.04
38	0.18	0.02	0.11	0.21	0.13	0.08

Table 66. Mix 117-3—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	-0.04	-0.02	0.01	-0.02	-0.02	0.02
38	-0.20	-0.19	-0.08	-0.18	-0.16	0.06
47	-0.29	-0.24	-0.16	-0.20	-0.22	0.05
67	-0.45	-0.44	-0.34	-0.33	-0.39	0.06
80	-0.64	-0.69	-0.55	-0.51	-0.60	0.08
93	-0.83	-0.92	-0.72	-0.70	-0.79	0.10
102	-0.94	-0.99	-0.90	-0.82	-0.91	0.07
126	-1.45	-1.52	-1.38	-1.11	-1.36	0.18
135	-1.56	-1.60	-1.44	-1.14	-1.44	0.21
159	-1.77	-1.92	-1.75	-1.47	-1.73	0.19
194	-2.21	-2.35	-2.35	-1.89	-2.20	0.21
220	-2.58	-2.78	-2.68	-2.21	-2.56	0.25
255	-2.75	-3.00	-3.03	-2.60	-2.85	0.21
274	-3.19	-3.30	-3.24	-2.75	-3.12	0.25
303	-3.39	-3.52	-3.64	-3.02	-3.39	0.27

Table 67. Mix 117-4—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	-0.04	-0.05	-0.04	-0.04	-0.04	0.01
38	-0.23	-0.04	-0.07	-0.04	-0.10	0.09
47	-0.27	-0.06	-0.08	-0.07	-0.12	0.10
67	-0.39	-0.06	-0.17	-0.05	-0.17	0.16
80	-0.67	-0.08	-0.30	-0.07	-0.28	0.28
93	-0.79	-0.10	-0.36	-0.03	-0.32	0.34
102	-0.85	-0.11	-0.39	0.00	-0.33	0.38
126	-1.15	-0.16	-0.70	-0.18	-0.55	0.47
135	-1.18	-0.18	-0.73	-0.21	-0.57	0.48
159	-1.45	-0.22	-1.04	-0.28	-0.75	0.60
194	-1.67	-0.38	-1.36	-0.44	-0.96	0.65
220	-1.94	-0.61	-1.81	-0.66	-1.25	0.72
255	-2.23	-0.71	-2.13	-0.84	-1.48	0.81
274	-2.28	-0.86	-2.34	-0.87	-1.59	0.83
303	-2.67	-1.02	-2.63	-1.20	-1.88	0.89

Table 68. Mix 118-1—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
19	-0.04	-0.05	-0.04	-0.04	-0.04	0.01
38	-0.23	-0.04	-0.07	-0.04	-0.10	0.09
47	-0.27	-0.06	-0.08	-0.07	-0.12	0.10
67	-0.39	-0.06	-0.17	-0.05	-0.17	0.16
80	-0.67	-0.08	-0.30	-0.07	-0.28	0.28
93	-0.79	-0.10	-0.36	-0.03	-0.32	0.34
102	-0.85	-0.11	-0.39	0.00	-0.33	0.38
126	-1.15	-0.16	-0.70	-0.18	-0.55	0.47
135	-1.18	-0.18	-0.73	-0.21	-0.57	0.48
159	-1.45	-0.22	-1.04	-0.28	-0.75	0.60
194	-1.67	-0.38	-1.36	-0.44	-0.96	0.65
220	-1.94	-0.61	-1.81	-0.66	-1.25	0.72
255	-2.23	-0.71	-2.13	-0.84	-1.48	0.81
274	-2.28	-0.86	-2.34	-0.87	-1.59	0.83
303	-2.67	-1.02	-2.63	-1.20	-1.88	0.89

Table 69. Mix 118-2—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
9	-0.02	-0.03	0.06	-0.02	0.00	0.04
29	-0.06	-0.05	-0.04	-0.05	-0.05	0.01
42	0.02	-0.05	-0.03	-0.03	-0.02	0.03
55	0.00	-0.01	-0.01	-0.04	-0.01	0.02
64	0.04	0.04	0.00	0.01	0.02	0.02
88	-0.15	-0.17	-0.27	-0.20	-0.20	0.05
97	-0.17	-0.18	-0.29	-0.24	-0.22	0.06
121	-0.29	-0.26	-0.52	-0.38	-0.36	0.12
160	-0.42	-0.37	-0.63	-0.52	-0.49	0.11
192	-0.61	-0.54	-0.82	-0.72	-0.68	0.12
217	-0.64	-0.54	-0.86	-0.81	-0.71	0.15
236	-0.80	-0.65	-0.96	-0.97	-0.84	0.16
265	-0.97	-0.73	-1.07	-1.05	-0.95	0.16
300	-1.20	-0.92	-1.27	-1.34	-1.18	0.19

Table 70. Mix 118-3—Mass change versus cycles.

Cycles	A1	A2	A3	A4	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00
9	0.03	0.02	-0.03	0.01	0.01	0.03
29	0.01	0.00	-0.13	-0.05	-0.04	0.06
42	0.06	0.02	-0.17	-0.04	-0.03	0.10
55	-0.07	-0.01	-0.22	-0.20	-0.13	0.10
64	0.00	0.08	-0.13	-0.12	-0.04	0.10
88	-0.19	-0.04	-0.32	-0.25	-0.20	0.12
97	-0.88	-0.02	-0.25	-0.21	-0.34	0.38
121	-0.37	-0.01	-0.42	-0.34	-0.29	0.19
160	-0.57	-0.08	-0.50	-0.44	-0.40	0.22
192	-0.72	-0.12	-0.69	-0.67	-0.55	0.29
217	-0.80	-0.11	-0.76	-0.72	-0.60	0.33
236	-0.89	-0.16	-0.87	-0.83	-0.69	0.35
265	-1.05	-0.17	-0.91	-0.84	-0.75	0.39
300	-1.24	-0.21	-1.14	-1.08	-0.92	0.47

Table 71. Mix 223—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
9	-0.01	-0.01	0.00	-0.01	-0.03	-0.01	0.01
23	-0.11	-0.09	-0.08	-0.03	-0.10	-0.08	0.03
32	-0.14	-0.15	-0.13	-0.06	-0.13	-0.12	0.03
42	-0.25	-0.24	-0.21	-0.10	-0.24	-0.21	0.06
59	-0.43	-0.41	-0.41	-0.29	-0.40	-0.39	0.06
70	-0.46	-0.48	-0.46	-0.38	-0.50	-0.45	0.05
84	-0.56	-0.57	-0.58	-0.49	-0.61	-0.56	0.04
98	-0.71	-0.75	-0.77	-0.70	-0.85	-0.76	0.06
112	-0.75	-0.76	-0.80	-0.79	-0.99	-0.82	0.10
121	-0.80	-0.85	-0.94	-0.90	-1.12	-0.92	0.12
146	-0.96	-1.05	-1.11	-1.21	-1.27	-1.12	0.12
165	-1.06	-1.27	-1.30	-1.45	-1.49	-1.32	0.17
179	-1.20	-1.36	-1.41	-1.62	-1.58	-1.43	0.17
198	-1.31	-1.57	-1.56	-1.82	-1.74	-1.60	0.19
213	-1.43	-1.68	-1.63	-1.91	-1.84	-1.70	0.19
227	-1.58	-1.89	-1.76	-2.05	-1.97	-1.85	0.19
246	-1.79	-2.08	-1.90	-2.21	-2.16	-2.03	0.18
269	-1.98	-2.33	-2.21	-2.50	-2.40	-2.29	0.20
282	-2.05	-2.31	-2.30	-2.57	-2.46	-2.34	0.20
301	-2.26	-2.66	-2.49	-2.85	-2.68	-2.59	0.22

Table 72. Mix 224—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
9	-0.03	0.00	0.03	-0.03	-0.01	-0.01	0.02
23	-0.04	-0.03	-0.01	-0.03	-0.01	-0.03	0.01
32	-0.05	-0.01	0.00	-0.03	-0.03	-0.02	0.02
42	-0.05	-0.03	-0.04	-0.07	-0.03	-0.05	0.02
59	-0.17	-0.13	-0.21	-0.25	-0.17	-0.19	0.05
70	-0.18	-0.10	-0.21	-0.22	-0.12	-0.18	0.05
84	-0.27	-0.30	-0.47	-0.39	-0.32	-0.36	0.09
98	-0.42	-0.52	-0.68	-0.56	-0.49	-0.55	0.11
112	-0.50	-0.64	-0.86	-0.69	-0.65	-0.67	0.15
121	-0.58	-0.78	-1.02	-0.78	-0.74	-0.79	0.18
146	-0.75	-1.11	-1.39	-1.10	-0.99	-1.09	0.26
165	-0.87	-1.22	-1.55	-1.35	0.00	-1.25	0.29
179	-1.00	-1.35	-1.66	-1.52		-1.38	0.28
198	-1.09	-1.50	-1.89	-1.72		-1.55	0.35
213	-1.14	-1.59	-2.02	-1.88		-1.66	0.39
227	-1.32	-1.76	-2.23	-2.04		-1.83	0.40
246	-1.46	-1.86	-2.41	-2.21		-1.98	0.42
269	-1.73	-2.11	-2.74	-2.51		-2.27	0.45
282	-1.79	-2.21	-2.85	-2.53		-2.35	0.46
301	-2.02	-2.41	-3.09	-2.72		-2.56	0.45

Table 73. Mix 225—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
6	0.01	-0.04	0.00	0.00	-0.04	-0.01	0.02
20	0.01	-0.04	0.00	-0.01	0.13	0.02	0.07
26	-0.04	-0.03	-0.08	-0.03	-0.09	-0.05	0.03
35	-0.06	-0.07	-0.09	-0.09	-0.13	-0.09	0.03
49	-0.10	-0.08	-0.06	-0.08	-0.13	-0.09	0.03
58	-0.13	-0.05	-0.17	-0.12	-0.25	-0.14	0.07
83	-0.30	-0.17	-0.31	-0.14	-0.44	-0.27	0.12
102	-0.40	-0.22	-0.44	-0.22	-0.58	-0.37	0.15
116	-0.49	-0.22	-0.48	-0.21	-0.68	-0.42	0.20
135	-0.62	-0.30	-0.62	-0.28	-0.85	-0.53	0.24
150	-0.69	-0.36	-0.73	-0.43	-0.95	-0.63	0.24
164	-0.83	-0.46	-0.87	-0.52	-1.11	-0.76	0.27
183	-0.91	-0.56	-0.98	-0.58	-1.22	-0.85	0.28
206	-1.16	-0.73	-1.22	-0.77	-1.50	-1.08	0.32
219	-1.23	-0.75	-1.31	-0.81	-1.54	-1.13	0.34
238	-1.40	-0.87	-1.50	-1.00	-1.84	-1.32	0.39
252	-1.51	-0.99	-1.59	-1.09	-1.99	-1.43	0.40
271	-1.69	-1.16	-1.79	-1.21	-2.17	-1.60	0.42
286	-1.87	-1.26	-2.05	-1.31	-2.32	-1.76	0.46
305	-2.00	-1.43	-2.23	-1.50	-2.60	-1.95	0.50

Table 74. Mix 226—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
6	-0.03	-0.03	0.00	0.00	0.00	-0.01	0.01
15	-0.08	-0.08	-0.05	-0.03	-0.03	-0.05	0.03
29	-0.10	-0.04	-0.04	-0.03	-0.01	-0.04	0.04
38	-0.13	-0.13	-0.13	-0.08	-0.05	-0.11	0.04
63	-0.30	-0.22	-0.25	-0.17	-0.16	-0.22	0.06
82	-0.39	-0.32	-0.38	-0.24	-0.25	-0.32	0.07
96	-0.46	-0.33	-0.42	-0.24	-0.25	-0.34	0.10
115	-0.56	-0.43	-0.60	-0.35	-0.41	-0.47	0.10
130	-0.61	-0.49	-0.69	-0.43	-0.50	-0.54	0.10
144	-0.74	-0.62	-0.86	-0.54	-0.59	-0.67	0.13
163	-0.82	-0.68	-1.01	-0.64	-0.63	-0.76	0.16
186	-1.07	-0.93	-1.23	-0.83	-0.83	-0.98	0.17
199	-1.12	-0.95	-1.23	-0.88	-0.83	-1.00	0.17
218	-1.34	-1.18	-1.48	-1.05	-1.04	-1.22	0.19
232	-1.47	-1.26	-1.58	-1.17	-1.13	-1.32	0.19
251	-1.56	-1.41	-1.77	-1.24	-1.32	-1.46	0.21
266	-1.72	-1.52	-1.87	-1.30	-1.33	-1.55	0.25
285	-1.89	-1.75	-2.16	-1.53	-1.60	-1.79	0.25
304	-2.07	-1.95	-2.38	-1.69	-1.78	-1.97	0.27

Table 75. Mix 227—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
6	0.01	0.01	0.01	0.05	0.00	0.02	0.02
15	-0.01	-0.01	0.03	0.05	0.00	0.01	0.03
29	-0.03	0.01	0.01	0.03	-0.03	0.00	0.02
38	-0.05	-0.08	-0.06	-0.01	-0.32	-0.11	0.12
63	-0.13	-0.18	-0.09	-0.12	-0.13	-0.13	0.03
82	-0.22	-0.26	-0.13	-0.21	-0.22	-0.21	0.05
96	-0.23	-0.28	-0.15	-0.26	-0.26	-0.24	0.05
115	-0.34	-0.35	-0.23	-0.33	-0.37	-0.32	0.05
130	-0.43	-0.41	-0.31	-0.36	-0.41	-0.38	0.05
144	-0.54	-0.49	-0.40	-0.51	-0.59	-0.51	0.07
163	-0.62	-0.54	-0.44	-0.59	-0.67	-0.57	0.09
186	-0.79	-0.69	-0.63	-0.76	-0.81	-0.74	0.07
199	-0.85	-0.76	-0.67	-0.77	-0.87	-0.78	0.08
218	-1.00	-0.92	-0.84	-1.00	-1.09	-0.97	0.09
232	-1.05	-1.00	-0.86	-1.07	-1.17	-1.03	0.11
251	-1.18	-1.14	-1.02	-1.26	-1.32	-1.18	0.12
266	-1.31	-1.20	-1.07	-1.36	-1.45	-1.28	0.15
285	-1.52	-1.41	-1.34	-1.49	-1.64	-1.48	0.11
304	-1.72	-1.56	-1.48	-1.69	-1.81	-1.65	0.13

Table 76. Mix 302—Mass change versus cycles.

Cycles	A1	A2	A3	A4	A5	Mean	Std dev
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	0.05	0.05	0.04	0.08	0.09	0.06	0.02
24	0.15	0.17	0.04	0.11	0.10	0.11	0.05
31	0.12	0.09	0.08	0.15	0.08	0.10	0.03
44	0.09	0.05	0.08	0.10	0.04	0.07	0.03
52	0.06	0.10	0.12	0.14	0.06	0.10	0.03
62	0.12	0.14	0.13	0.14	0.08	0.12	0.03
72	0.18	0.18	0.17		0.13	0.16	0.03
86	0.13	0.15	0.13		0.11	0.13	0.02

APPENDIX C. STATISTICAL ANALYSIS—DURABILITY FACTOR

Table 77. Durability factor sets 1 and 2—sorted by fresh air content.

Mixes	Admixture	Mean	s	n _i	Pooled s ²	S.E.	t ₀	t _(.05,n1+n2-2)	Prob (t)	Conclusion
227–348	Vinsol	87.85	1.48	5	22.59	3.19	-18.55	2.365	0.0000003	Different
	Synthetic	28.72	7.06	4						
225–346	Vinsol	90.17	0.64	5	30.32	3.69	-9.35	2.365	0.0000332	Different
	Synthetic	55.62	8.38	4						
224–347	Vinsol	85.69	1.18	5	11.89	2.31	-4.38	2.365	0.0032219	Different
	Synthetic	75.55	5.09	4						
223–349	Vinsol	88.87	2.79	5	78.59	5.95	-3.92	2.365	0.0057823	Different
	Synthetic	65.58	13.15	4						
226–350	Vinsol	93.53	1.26	5	8.02	1.90	-5.88	2.365	0.0006102	Different
	Synthetic	82.35	4.07	4						

Table 78. Mass change sets 1 and 2—sorted by fresh air content.

Mixes	Admixture	Mean	s	n _i	Pooled s ²	S.E.	t ₀	t _(.05,n1+n2-2)	Prob (t)	Conclusion
227–348	Vinsol	-1.61	0.11	5	0.05	0.16	8.34	2.365	0.0000697	Different
	Synthetic	-0.32	0.33	4						
225–346	Vinsol	-1.79	0.39	5	0.18	0.28	-7.89	2.365	0.0000996	Different
	Synthetic	-4.03	0.47	4						
224–347	Vinsol	-2.56	0.45	5	0.12	0.23	2.16	2.365	0.0680295	Different
	Synthetic	-2.06	0.06	4						
223–349	Vinsol	-2.59	0.22	5	0.48	0.46	-1.02	2.365	0.3426067	Not significantly different
	Synthetic	-3.06	1.03	4						
226–350	Vinsol	-2.02	0.29	5	0.11	0.23	-3.16	2.365	0.0159507	Different
	Synthetic	-2.74	0.39	4						

