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Effect of Entrained Air-Void and Supplementary Cementitious Materials on Durability of Concrete

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ABSTRACT

In cold climates, concrete used in pavements and bridges must be able to withstand cyclic freezing and thawing. Durability in these environments is achieved through the production of air-entrained concrete. The admixtures used to achieve an air-void system have changed and current concrete mixtures use more supplementary cementitious materials. This research examined if traditional limits used to describe the air-void system still apply to concrete prepared with these new admixtures and materials. Results show that traditional limits for specifying an air void system (e.g. total air content 4.5-7% and spacing factor less than or equal to 0.2 mm) still constitute a minimum limit, but modern concrete with lower total air contents or spacing factors greater than 0.2 mm are often freeze-thaw durable. With modern mixtures, the microstructure of the hardened cement paste appears to be more durable. Evidence was found for a pessimum level of sorptivity relating to poor freeze-thaw performance.

INTRODUCTION

Background In cold climates, concrete used in highway infrastructure (e.g. pavements, bridges) is exposed to harsh winter conditions, being cyclically frozen and thawed in a saturated state in the presence of chemical deicers. The durability of such concrete is dependant upon many things, including the aggregate component, the hydrated cement paste (HCP) and the presence of a properly entrained air-void system (small, dispersed and closely spaced air bubbles). The research used to establish the current air content requirements as espoused in the *Guide to Durable Concrete* [American Concrete Institute, 2008] was predominantly conducted prior to 1970, and since that time many changes have occurred that significantly affect the quality/characteristics of the HCP as well as the entrained air-void system. Some changes that directly impact the *quality* of the HCP include the use of lower water-to-cementitious ratios (w/cm), finer faster-setting cements, and the extensive use of supplementary cementitious materials (SCMs) such as fly ash, ground blast furnace slag (GBFS), etc. The biggest change in the characteristics of the entrained air-void system has resulted from the introduction of air entraining agents other than those based on vinsol resins.

A major difficulty in predicting the effects to concrete from changes in the HCP quality is that factors contributing to the quality of the HCP, including the entrained air-void system, are diverse and not perfectly understood. It is known, for instance, that lowering the w/cm

will reduce capillary porosity, therefore increasing strength and reducing the permeability of a given concrete. But how changes in cement fineness and/or chemistry, the presence of various SCMs, and the use of the current generation of air entraining admixtures impacts the freeze-thaw (F-T) durability of HCP is not fully understood.

The entrained air-void system is created through the addition of surface-active agents acting at the water-air interface to create stable foams. Historically, naturally derived vinsol resinbased air entraining agents (AEAs) were commonly used and specifications for air-entrained concrete are based on these chemicals. As the use of AEAs derived from synthetic or other natural sources increases, changes in the nature of the resulting air-void system may make past specification practices incorrect for these concrete mixtures. The issue becomes more clouded in that AEAs may interact in an unexpected manner with other concrete constituents (e.g. cement, SCMs, admixtures), making it difficult to anticipate the quality of the HCP and air-void system in advance of construction.

Although the relationship between the F-T durability of concrete and the quality of the hydrated cement paste and the air-void system are thought to be well established, there have been sufficient changes in concrete mixtures (e.g. lower w/cm, the use of SCMs, synthetic versus vinsol resin AEAs, etc.) and problems in the field to warrant a study to re-examine the accepted relationships.

Approach Overall this research examined the relationship(s) between F-T durability and the quality of the HCP, including the air-void system parameters, for various concrete mixtures. The concrete mixtures prepared were thoroughly characterized, including the measurement of the air content, unit weight, air-void system parameters using the Air Void Analyzer (AVA), maturity, calorimetric heat signature, microwave moisture content of fresh concrete, and strength at various ages, sorptivity, and the air-void system parameters of the hardened concrete. In selected cases, an assessment of the HCP porosity using epifluorescence techniques was also performed. This paper presents an overview of only the results regarding air-void system parameters, F-T performance, and sorptivity.

EXPERIMENTAL

To accomplish the research, a combined full- and partial-factorial experimental matrix was established that resulted in a total of 68 different concrete mixtures. Each mixture was prepared in duplicate. The mixture parameters used included:

- Cement type: ASTM Type I/II
- Cement factors: 335 kg/m^3 , 307 kg/m^3 , and 280 kg/m^3
- SCMs: none, Class C fly ash, and Grade 100 ground blast furnace slag
- AEAs: one vinsol resin and one synthetic
- Coarse aggregate: a durable carbonate
- Aggregate grading: gap gradation and optimized
- Fine aggregate volume altered to adjust yield with changes in *w/cm*
- *w/cm*: 0.45, 0.50, and 0.52.
- Two target air contents: $3\% (\pm 1\%)$ and $6\% (\pm 1.5\%)$

A variety of fresh concrete tests were performed to ensure the quality and mixture design for each mixture. The fresh concrete tests included

- Slump: ASTM C143 Standard Test Method for Slump of Hydraulic-Cement Concrete
- Determination of air content: ASTM C231 Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method; ASTM C173 - Standard Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method; and ASTM C138 - Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete
- Air-void system parameters: *Air Void Analyzer (AVA)* Also known as the "Danish Air-Meter" No standard current addresses this instrument or its use.
- Unit weight and yield: ASTM C138

To assess the hardened concrete properties, testing included:

- Determination of hardened air-void system parameters using a flat bed scanner (Peterson, 2009). Calculations in accordance with: ASTM C457 Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete
- Sorptivity after 56 days: ASTM C1585 Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes
- F-T durability: ASTM C666 Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing Method A

RESULTS

The measured air contents for the concrete mixtures with a low target air (i.e. target $3\% \pm 1\%$) and normal target air (i.e. target $6\% \pm 1.5\%$) content, as measured by ASTM C231 (i.e. pressure meter) are shown in Figures 1 and 2, respectively. Figures 3-6 compare the air content measured by ASTM C173 (i.e. volumetric meter), ASTM C138 (i.e. gravimetric air), the AVA, and the flatbed scanner (ASTM C457), to that measured by the pressure meter. Figures 7-10 present the air void system parameters specific surface and spacing factor plotted against each other for both the hardened air void system as measured by the flatbed scanner, and the AVA. Figures 7 and 8 show the relationship between these two parameters as a function of the measured air content. Figures 9 and 10 show the relationship between these two parameters as a function of the durability factor measured by ASTM C666 testing. Table 1 presents a summary of how the prepared concrete mixtures would be classified for F-T resistance, based upon traditional specifications, and also how those same concrete mixtures actually performed when tested in ASTM C666. Figure 11 shows the sorptivity measured by ASTM C1585 for each concrete mixture where each mixture is color-coded to represent the durability factor that corresponds to each mixture.



Figure 1. Measured air contents for the concrete mixtures with low air (i.e. target $3\% \pm 1\%$) as measured by ASTM C231 (i.e. pressure meter).



Figure 2. Measured air contents for the concrete mixtures with normal air (i.e. target $6\% \pm 1.5\%$), as measured by ASTM C231 (i.e. pressure meter).



Figure 3. Air content measured by ASTM C173 (i.e. volumetric meter) compared to that measured by ASTM C231 (pressure meter).



Figure 4. Air content measured by ASTM C138 (i.e. gravimetric air) compared to that measured by ASTM C231 (pressure meter).



Figure 5. Air content measured by AVA compared to that measured by ASTM C231 (pressure meter).



Figure 6. Air content measured by ASTM C457 (flatbed scanner) compared to that measured by (ASTM C231) pressure meter.



Figure 7. Measured air-void system specific surface area versus measured air-void system spacing factor, both measured by ASTM C457, grouped by total air content.



Figure 8. Measured air-void system specific surface area versus measured air-void system spacing factor, both measured by AVA, grouped by total air content.



Figure 9. Measured air-void system specific surface area versus measured air-void system spacing factor, both measured by ASTM C457, grouped by ASTM C666 durability factor.



Figure 10. Measured air-void system specific surface area versus measured air-void system spacing factor, both measured by AVA, grouped by ASTM C666 durability factor.



Figure 11. Bulk sorptivity measured by ASTM C1585 for all concrete mixtures where each mixture is color-coded to represent the ASTM C666 durability factor that corresponds to that mixture.

| Classification | Supplementary Cementitious Material | | |
|---------------------------------------|-------------------------------------|---------|-------|
| | None | Fly Ash | Slag |
| Durable (Spacing Factor < 0.2 mm) | 37.5% | 25.0% | 14.3% |
| Non-durable (Spacing Factor > 0.2 mm) | 62.5% | 75.0% | 85.7% |
| Non-durable - Durability Factor < 80 | 12.5% | 0.0% | 7.1% |

Table 1. Summary of how the low target air mixtures would be classified for F-T durability based upon ASTM C457 results, and also a summary of how many low target air mixtures actually failed ASTM C666 testing with a durability factor less than 80%.

DISCUSSION

As can be seen in Figure 1, the air contents achieved for the low target air mixtures agree well with the target air content. For all mixtures prepared at the low target air content, 85.3% were within the specified target range and 94.1% were within 3 standard deviations of the target air content. For the normal air content mixtures, as shown in Figure 2, 79.4% were within the specified target range and 100% were within 3 standard deviations of the target air content. For both types of mixtures, the mixtures falling furthest from the target air content were generally those prepared using the synthetic AEA, illustrating the difficulty of working with synthetic AEAs relative to the vinsol resin based AEAs.

Figures 3-6 illustrates the range of results that are obtained for measured total air content when using the common methods of measuring fresh air content (i.e. ASM C231, ASTM C173, and ASTM C138) and common methods of determining the complete air-void system parameters (i.e. ASTM C457 and the AVA). As seen in Figure 3, the pressure meter and volumetric meter agree very well and are obviously dependable methods of measuring total air content. However, these methods only provide a measure of air content and other parameters are more important in understanding F-T performance. Figure 4 compares the results of air content determined by gravimetric methods to the pressure meter results. The gravimetric results correlate well but generally under report the total air content. Figure 5 presents the results of measuring the total air content by the AVA as compared to the results obtained from the pressure meter. The AVA performs the worst of all methods when determining the total air content. Not only does it generally under report the total air content, the results are not reproducible. The results shown in Figure 5 do not include results that would plot off the scale used (e.g. results of negative air content). Finally, Figure 6 presents the results of the ASTM C457 analyses performed using a flatbed scanner. The flatbed scanner is currently not an accepted method of performing ASTM C457 but it is currently under consideration for inclusion in the test method. Figure 6 illustrates that the flatbed scanner method correlates well with the air content measured by the pressure meter but slightly overestimates the total air content.

Overall, the mixtures provided a bimodal distribution of mixtures relative to air content and other air-void system parameters. For the low target air mixtures, the effective maximum air content was 4.7% (i.e. 2 of 34 mixtures exceeded this value with total air contents of 5.5% and 5.6%, respectively). For the normal target air mixtures, the effective minimum air content was 4.6% (i.e. 2 of 34 mixtures exceeded this value with total air contents of 3.8% and 4.2%, respectively). Therefore, the dividing line between the two modes can be effectively considered ~4.5%, which is typically considered a minimum air content for F-T durable concrete in most concrete paving specifications. That is, the mixtures prepared

adequately represent concrete with normal air contents and concrete with low air contents, as represented by total air content.

Figures 1-6 demonstrate the mixtures prepared were within the limits of the expected mixture design in terms of total air. A more detailed analysis of the air-void system parameters is presented in Figures 7-10 and incorporates the results of ASTM C666 testing. All graphs included in Figures 7-10 plot the measured specific surface against the measured spacing factor, for each technique (i.e. ASTM C457 and the AVA). This is a good internal check of each technique and the inverse relationship seen is expected. In examining Figure 7, consider the previously presented general conclusion that the lower limit of the normal target air mixtures, in terms of total air content, is consistent with what is commonly considered durable in F-T environments. It is interesting to note that the generally accepted threshold of F-T durability, in terms of spacing factor as measured by ASTM C457 (i.e. 0.2 mm), agrees very well with the distinction of F-T durable and non-durable concrete as measured by total air content. Examining Figure 8 where the same air-void system parameters are determined by use of the AVA, the same trend evident from the ASTM C457 analysis is clearly seen. However, the distinction between the normal target air and low target air mixtures would be a spacing factor of approximately 0.3 mm as determined by the AVA. That is, although the AVA is not dependable at measuring the total air content, it can identify trends in other airvoid system parameters. Although the same threshold of acceptance (i.e. 0.2 mm spacing factor) cannot be used to identify F-T durable concrete, the techniques does, in principal, identify durable and non-durable concrete, albeit with a different threshold.

The plots shown in Figures 9 and 10 present the measured specific surface values plotted against the measured spacing factor for each technique grouped as a function of the F-T durability factor as measured by ASTM C666. The same trends seen in Figures 7 and 8 are evident. That is, the generally accepted threshold of 0.2 mm for spacing factor, as determined by ASTM C457 delineates F-T durable concrete from non-durable concrete. Likewise, the AVA would indicate the same distinction at a spacing factor of 0.3 mm. The interesting thing is that no concrete failed the ASTM C666 F-T test with an ASTM C457 spacing factor of 0.2 mm or less. However many concrete mixtures with a spacing factor greater than 0.2 mm also did not fail the ASTM C666 test. Also, all concrete that failed the F-T test were from the mixtures prepared with low target air contents.

Considering the matrix of concrete mixtures prepared for this study, a key point is the role of SCM replacement on the demonstrated F-T performance. Most concrete mixtures currently being used have some level of SCM replacement and there is concern amongst some state highway agencies that these SCM replacements might compromise F-T performance. To examine this concern it is important to first look at the conventional measures of performance (i.e. the spacing factor as measured by ASTM C457) and then examine the actual performance achieved.

The first two rows of Table 1 present the breakdown of which concrete mixtures examined would be considered durable or non-durable with respect to F-T damage, on the basis of an evaluation of the air-void system parameters. Based upon this conventional measure, the majority of all low target air mixtures would be rejected. However, as seen in the last row of Table 1, few of the concrete mixtures tested actually failed ASTM C666. Although this performance could be attributed to many factors, the important point is that there is no demonstrable loss in performance for a concrete exposed to F-T conditions as a result of the use of common SCMs.

Finally, Figure 11 shows preliminary results from ASTM C1585 sorptivity testing. The sorptivity for each concrete mixture is shown in terms of the ASTM C666 durability factor. What the initial data shows is an apparent "pessimum" sorptivity leading to F-T damage. At low sorptivity, the degree of saturation is low enough to prevent damage from freezing and thawing, At high sorptivity, the water is able to move freely through the concrete and again, F-T damage is avoided. In the pessimum range of sorptivity, where water is able to penetrate and saturate but not readily flow through the concrete, F-T damage occurs.

Overall, modern cements and the use of SCMs lead to a hardened cement paste that can potentially have a higher tensile strength and lower permeability. This in turn creates a situation where a lower total air content, or an air-void system with a larger spacing factor, can in fact be F-T durable. Current limits on both total air content and air void system parameters were established many years ago with different cements, different admixtures, and limited use of SCMs. Evidence indicates that these traditional limits should provide a conservative estimate of performance and incorporating SCMs into concrete mixtures does not necessarily require deviation from these traditional air-void system thresholds.

CONCLUSIONS

There is general agreement between methods of measuring the total air content of a concrete mixture, although the AVA generally does not perform well for this task. With current admixtures, concrete produced with a conventionally accepted level of total air content (e.g. $6 \pm 1.5\%$) can be expected to be F-T durable, but concrete produced with lower air contents can also be durable. The classic limitation of an air-void system spacing factor less than or equal to 0.2 mm is still a safe value to ensure F-T durability, but evidence exists that concrete mixtures with a spacing factor greater than 0.2 mm can also be F-T durable. There appears to be no demonstrable evidence that the use of SCMs reduce F-T durability. Preliminary results indicate that a pessimum level of sorptivity relates to poor F-T performance.

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