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Supplementary Notes				
<p>Abstract</p> <p>The air-void systems produced by two commercially available air-entraining admixtures (AEA), one a vinsol resin formulation and the other a tall oil formulation, were studied in mortars. Mortars were composed of four different portland cements and two Class F fly ashes (20 percent by mass) with concrete sand. The mortar proportions were based on Virginia Department of Transportation Class A4 concrete with a water-cementitious material ratio of 0.45. Foam index tests were conducted on all cementitious combinations, and the results were used to determine the dosage of AEA in the mortars.</p> <p>The air content of the mortars was determined gravimetrically, and specimens were cast for subsequent linear traverse analysis of the air-void system. With both AEAs, mortar air contents in the target range produced spacing factors much lower than necessary to provide resistance to freezing and thawing, suggesting that the ranges for air content currently used in VDOT specifications could be reduced to lessen the potential for acceptance problems associated with excessive air content. Further study is recommended to verify that the relationships observed in this study are also observed in concrete and to define improved air content specifications.</p>				

FINAL REPORT

**LABORATORY INVESTIGATION OF AIR-VOID SYSTEMS PRODUCED
BY AIR-ENTRAINING ADMIXTURES IN FRESH AND HARDENED MORTAR**

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Virginia Transportation Research Council
(A partnership of the Virginia Department of Transportation
and the University of Virginia since 1948)

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ABSTRACT

The air-void systems produced by two commercially available air-entraining admixtures (AEA), one a vinsol resin formulation and the other a tall oil formulation, were studied in mortars. Mortars were composed of four different portland cements and two Class F fly ashes (20 percent by mass) with concrete sand. The mortar proportions were based on Virginia Department of Transportation Class A4 concrete with a water-cementitious material ratio of 0.45. Foam index tests were conducted on all cementitious combinations, and the results were used to determine the dosage of AEA in the mortars.

The air content of the mortars was determined gravimetrically, and specimens were cast for subsequent linear traverse analysis of the air-void system. With both AEAs, mortar air contents in the target range produced spacing factors much lower than necessary to provide resistance to freezing and thawing, suggesting that the ranges for air content currently used in VDOT specifications could be reduced to lessen the potential for acceptance problems associated with excessive air content. Further study is recommended to verify that the relationships observed in this study are also observed in concrete and to define improved air content specifications.

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LABORATORY INVESTIGATION OF AIR-VOID SYSTEMS PRODUCED BY AIR-ENTRAINING ADMIXTURES IN FRESH AND HARDENED MORTAR

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INTRODUCTION

For more than 50 years, small air voids have been purposely introduced into concrete to provide resistance to freezing and thawing. Research conducted in the 1940s had indicated that the parameter of the air-void system most important to freezing and thawing resistance was the spacing of the air voids (Powers, 1949, 1954). Void spacing has traditionally been determined on specimens of hardened concrete by petrographic examinations. In the 1940s and 1950s, relationships were developed between the desired spacing factor for durability and the volume of air necessary to achieve it. It was recognized that the volume of air was directly proportional to the volume of paste in the concrete and the severity of the freezing and thawing conditions. This information was used to provide guidance in standards and specifications on the total air content required in fresh concrete for acceptance.

Although this practice has served fairly well for 50 years, changes in concrete technology have taken place that warrant investigation. In particular, the relationship between air content and spacing factor was developed when the type of material used to entrain air in concrete was limited primarily to admixtures based on vinsol resin (VR). Today, other materials such as tall oils (TO) and synthetic detergents are used that provide more stable air-void systems (Whiting and Nagi, 1998). However, the size of the voids produced is smaller, and thus the relationship between total air and spacing factor may have changed.

With the thrust toward performance specifications, attention has been refocused on the spacing factor achieved in the concrete rather than simply the total air content. Spacing factor requirements are appearing on new construction projects such as Wacker Drive in Chicago (Kaderbek et al., 2002) and the Confederation Bridge from the mainland to Prince Edward Island (Holley et al., 1999). These projects have specified spacing factors measured on hardened concretes. The Ministry of Transportation Ontario has instituted an end-result specification for air-void characteristics based on analysis of hardened concrete (Schell and Konecny, 2003). This practice, although well-founded, has the disadvantage that the information is not available until several weeks after the concrete is placed. In Europe, the application of performance specifications with spacing factor requirements led to the development of an apparatus that can measure the spacing factor in fresh concrete. The apparatus provides the concrete producer with a tool to evaluate concrete proportions and mixing sequences quickly for their impact on the air-void system. This apparatus, the Air-Void Analyzer (AVA) is a focus technology of the AASHTO Technology Implementation Group. As initially conceived, AVA testing was to be

included in the current project; however, because of difficulties in obtaining the equipment, this aspect was deferred.

PURPOSE AND SCOPE

The objective of this research was to evaluate the differences in air-void systems produced by different commercially available AEAs and to assist in the development of performance-based specifications for air-entrained concrete. This project was planned with a limited scope to provide the necessary information for a subsequent, more comprehensive evaluation of air-void systems for durable concrete.

METHODS

Overview

This was a laboratory study in which the testing was performed on mortars. This approach was used because air is entrained in the mortar fraction of concrete, the movement of sand grains in the mortar during mixing affects the generation of the air-void system, and mortar testing is far more efficient with respect to time and materials than is concrete testing.

Materials

Two commercially available AEAs with differing formulations were evaluated: VR, a neutralized visol resin marketed primarily for use in concrete pavements, and TO, a tall oil formulation for use in structural concrete.

A stock laboratory natural sand was used for all mortar batches. The fineness modulus of the sand was between 2.7 and 2.8, and the bounds of its grading are shown in Figure 1.

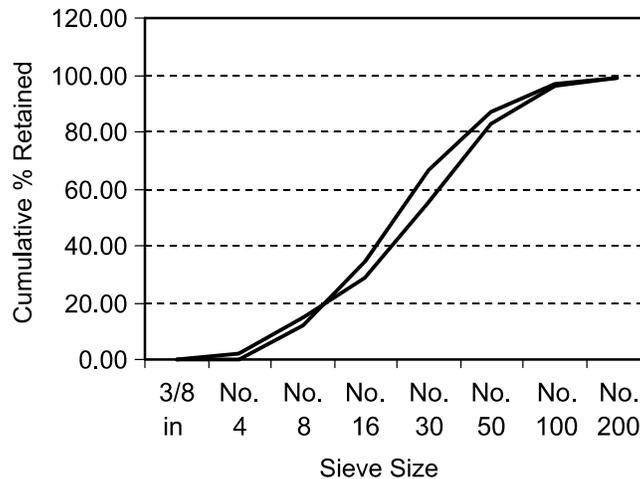


Figure 1. Grading Band of Sand Used in Mortar Tests

Six portland cements and two Class F fly ashes (ASTM C 618) were used to produce portland cement–fly ash blends with 20% fly ash. The characteristics of the cements are shown in Table 1. Fly ashes F1 and F2 had loss-on-ignition values of 2.06% and 2.55%, respectively.

Table 1. Cement and Fly Ash Characteristics

Component	Portland Cements					
	C1	C2	C3	C4	C5	C6
SiO ₂ , %	19.00	21.78	----	21.10	21.34	21.16
Al ₂ O ₃ , %	5.60	4.66	---	4.90	4.68	4.63
Fe ₂ O ₃ , %	2.60	3.13	---	4.00	2.84	3.15
CaO, %	61.60	63.61	---	64.10	64.2	63.64
MgO, %	2.61	2.44	---	1.20	2.2	2.58
SO ₃ , %	4.50	2.68	---	2.30	2.7	3.25
Na ₂ O, %	---	---	0.42	---	0.22	0.20
K ₂ O, %	---	---	0.20	----	0.77	0.77
Na ₂ Oequiv., %	1.04	0.48	0.48	0.18	0.72	0.70
LOI, %	1.60	1.22	---	2.00	1.01	0.81
Insol Res., %	0.14	0.26	---	0.31	---	---
C ₃ S, %	53.0	50.0	---	55.0	55.7	53.4
C ₂ S, %	14.0	24.7	---	19.0	19.4	20.6
C ₃ A, %	10.0	7.1	---	6.0	7.6	6.9
C ₄ AF, %	---	9.5	---	12.0	8.6	9.6
Blaine, m ² /kg	372	367	---	403	386	541
-No. 325, %	96.9	95.7	---	90.1	98.0	99.7

Note: LOI = loss on ignition; Insol Res. = insoluble residue.

Testing

The testing was conducted in four steps:

1. *Foam index (FI) tests were performed for all cementitious blends with each admixture to estimate the dosage requirement.* The procedure is described in Table 2.

Table 2. Foam Index Procedure

-
1. Place 50 mL tap water, 16 g cement, and 4 g fly ash in a 1-pint widemouth glass jar.
 2. Cover and shake for 15 sec, then place on countertop.
 3. Allow the covered jar to rest undisturbed for 45 sec.
 4. At 1 min from start, remove the lid and introduce an increment of diluted (1:20) air-entraining admixture (AEA).
 5. Cover and shake for 15 sec, then place on countertop.
 6. Allow the covered jar to rest undisturbed for 45 sec.
 7. At 2 min from start, remove lid and estimate the percentage of liquid surface covered by foam. The foam index is achieved with essentially 100% foam coverage.
 8. If the coverage is less than 100%, add an increment of AEA and repeat steps 2-7 until the foam index is achieved. If the initial dosage of AEA produces 100% coverage, repeat the test with a smaller increment of AEA
-

2. *Mortars were mixed in accordance with ASTM C 305 using an amount of AEA equivalent to the foam index. The mortar proportions were based on the requirements of VDOT Class A4 concrete (635 lb/yd³ cementitious material and 0.45 water-cementitious material ratio [w/cm], target air content of 6.5%). Because air is entrained in the mortar fraction of concrete and the typical coarse aggregate content for Class A4 concrete is 40%, the target air content for the mortars was 10.8%. The mortar proportions are given in Table 3.*
3. *The air system in fresh mortars was tested gravimetrically in general accordance with ASTM C 185 except that the sand was a commercially available concrete sand and the calculations to determine the gravimetric air content were adjusted accordingly. Cylindrical specimens were cast from each batch and moist cured for a period of time sufficient to develop adequate maturity to allow preparation of petrographic specimens for analysis in accordance with ASTM C 457.*
4. *The analysis (ASTM C 457) of the air-void systems in the mortars was conducted following the linear traverse procedure using a magnification of 80X. An assumed paste content of 52%, based on the mortar proportions, was used in the calculations determining the parameters of the air-void system.*

Table 3. Mortar Proportions per Batch

Cement, g	504
Fly ash, g	116
Sand, g	1048
Water, mL	280
Air-entraining admixture, mL	Per foam index

RESULTS AND DISCUSSION

The results of the FI tests were used to establish the dosage of AEA for the mortars and are shown in Figure 2. The dosages reported are based on the amount of undiluted AEA used. The FI test is a simple test developed primarily to track the effect of a particular fly ash on the AEA dosage (Whiting and Nagi, 1998). For the suite of materials tested, the cement seems to exert the most influence on the AEA FI, with cements C1, C2, and C3 generally having a higher FI than cements C4, C5, and C6, with the exception of C1-F1-VR. This effect was more pronounced with VR than with TO. The cement alkali content, in particular, the initial water-soluble alkali content, is the parameter that is generally understood to affect AEA dosage, with higher alkali content generally increasing the effectiveness of the AEA. Thus, a higher alkali cement would be expected to have a lower FI. These results were somewhat mixed with respect to cement alkali content; for instance, C4, with the lowest total alkali content, tended to have an unexpected low FI, and C1, with the highest alkali content, had an unexpectedly high FI when coupled with F2. The low influence of fly ash on the FI is to be expected, as the primary fly ash characteristic affecting AEA dosage is carbon content, which is similar for the two tested materials as indicated by their loss-on-ignition values.

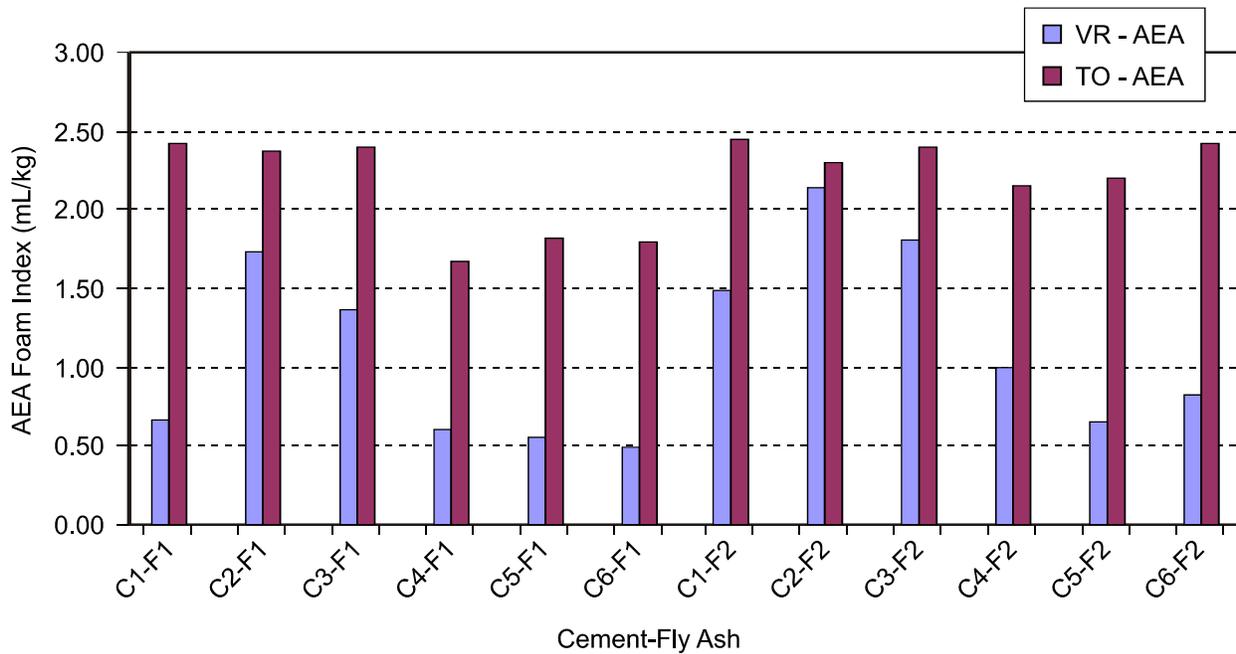


Figure 2. Results of Foam Index Tests. Values are based on amount of undiluted air-entraining admixture (AEA) used. VR = vinsol resin, TO = tall oil.

Mortars were mixed using the amount of AEA suggested by the FI for the various cement-fly ash combinations. The air content of the plastic mortars was determined gravimetrically, and specimens were then cast and cured for subsequent microscopic air-void determinations. The targeted range for total air content was approximately 8.0% to 13.5% (allowing a 25% fluctuation around the target of 10.8%). Comparisons of the total air content determined gravimetrically and by linear traverse (LT) analysis for the VR and TO mortars are shown in Figures 3 and 4, respectively. Some discrepancy between gravimetric and LT measurements are to be expected because water-filled pockets will not be accounted for gravimetrically, but will occur as voids following curing. This tendency was more pronounced in the VR mortars.

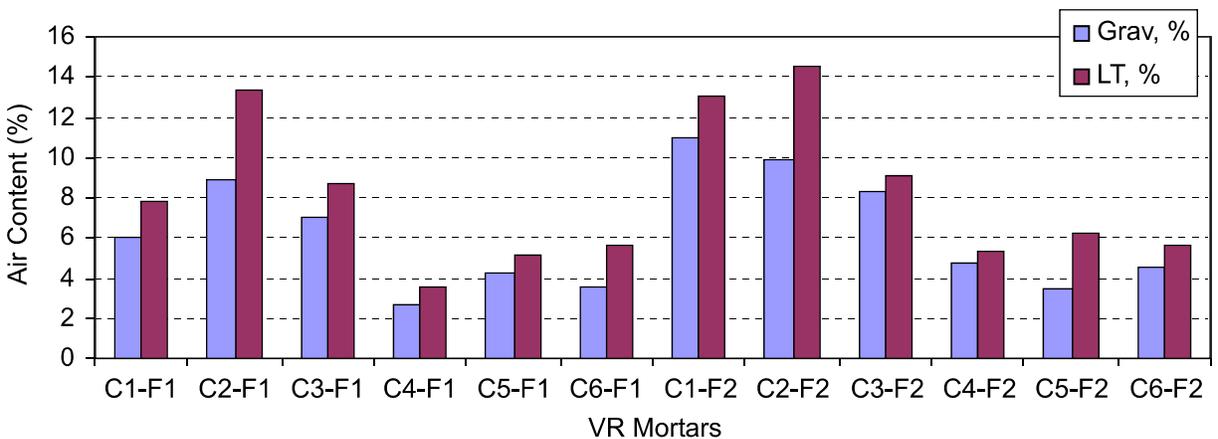


Figure 3. Comparison of Vinsol Resin (VR) Mortar Air Contents Determined Gravimetrically (Grav) and by Linear Traverse (LT) Analysis

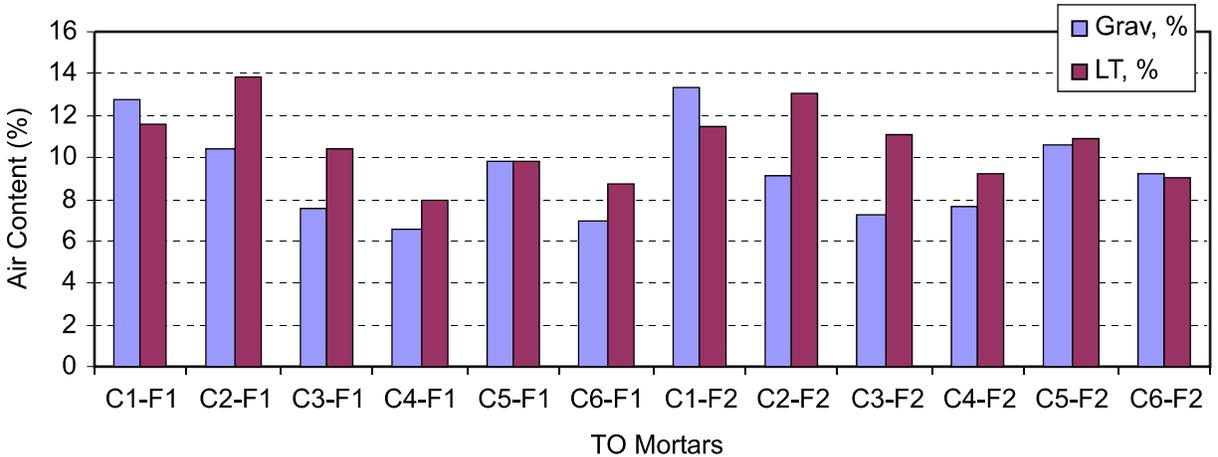


Figure 4. Comparison of Tall Oil (TO) Mortar Air Contents Determined Gravimetrically (Grav) and by Linear Traverse (LT) Analysis

For the VR mortars (Figure 3), air contents were within (or slightly below) the targeted range for mortars with C1, C2, and C3; C4, C5, and C6 mortars were below the targeted range. Air contents essentially mirrored the AEA dosages determined from the FI (Figure 2). With the TO mortars (Figure 4), values were within or slightly above the targeted range, depending on the determination method. Air contents for mortars made with C1, C2, and C3 were somewhat higher than those for C4, C5, and C6 mortars, again mirroring the relative AEA dosages.

Although total air content is useful in controlling the production of air-entrained concrete, the relevant characteristic of the air-void system that provides freeze-thaw resistance is the spacing factor (SF), the theoretical average distance from a point in the paste to the nearest air void. The SF is calculated from data collected during an ASTM C 457 analysis including an element of void size (see Lane and Stutzman [2004] for a description of linear traverse data analysis). Figures 5 and 6 illustrate the void size distribution of the C3-F1 mortars with VR and TO, respectively. The specific surface is another useful descriptor of air-void systems; it is related to the relative size of the voids, with larger values associated with smaller voids. As a general rule of thumb, entrained voids have been considered to be those with sizes under 1 mm (Lane and Stutzman, 2004). The distributions in these figures suggest that a further division can be made below 1 mm since it is really the small voids that are generated by the AEA and will control the spacing factor. Figures 7 and 8 show the relationship among the total air content, air content in voids below 0.30 mm, and the spacing factor for the VR and TO mortars, respectively. In these figures, the offset between the total air content and the voids < 0.30 mm is simply the air contained in the larger voids; thus the spacing factor is principally a function of those small voids. This explains the European practice of using the percentage of air contained in voids with chord lengths under 0.30 mm as a focal point with regard to providing frost resistance (Wilk et al., 1974).

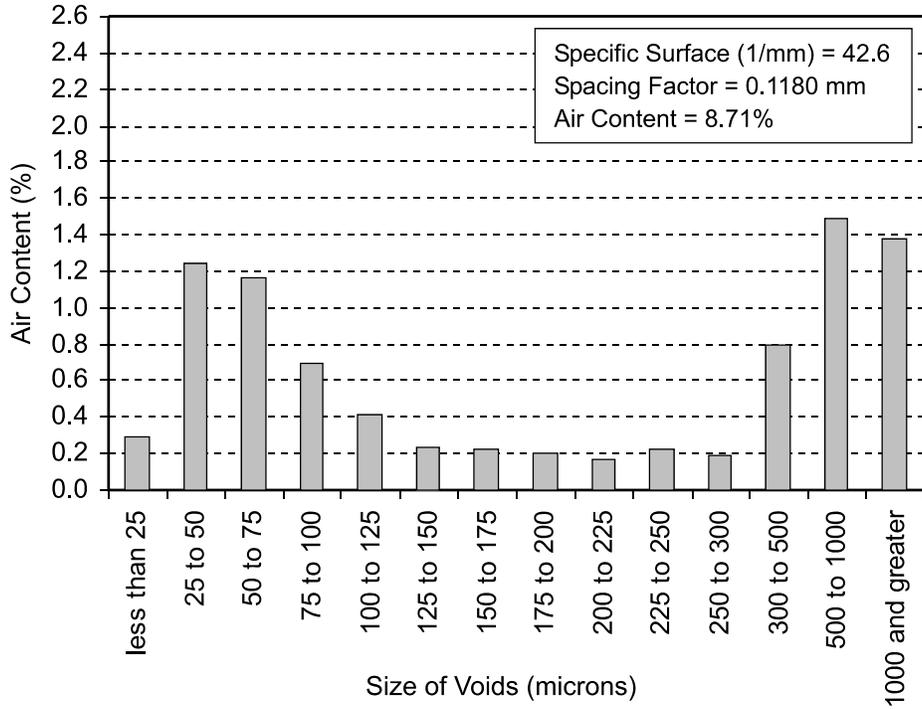


Figure 5. Void Size Distribution for C3-F1-VR Mortar

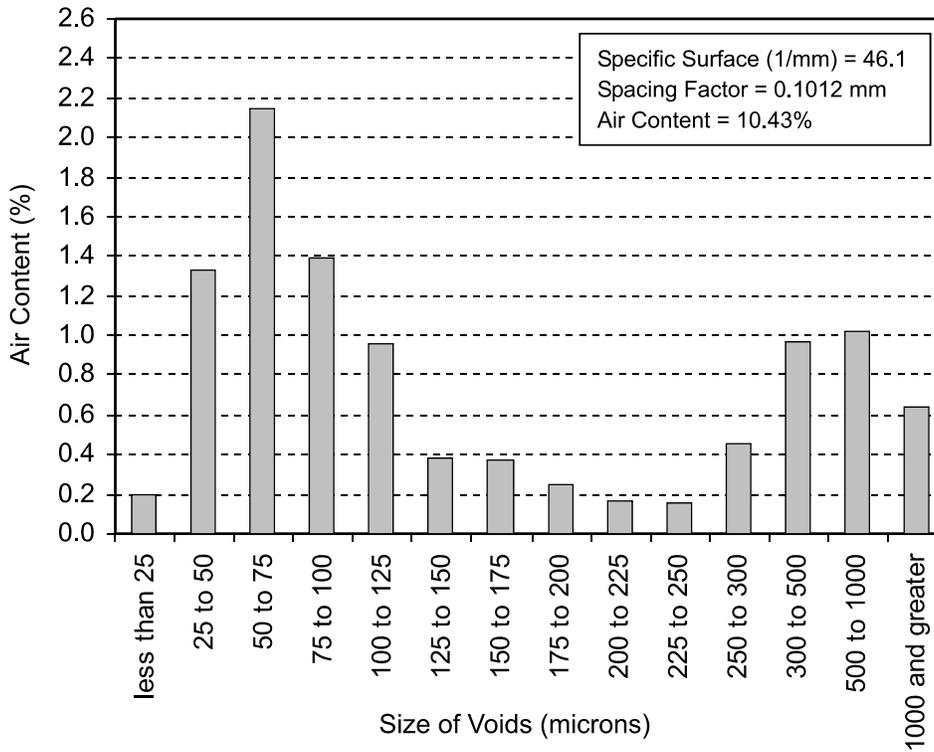


Figure 6. Void Size Distribution for C3-F1-TO Mortar

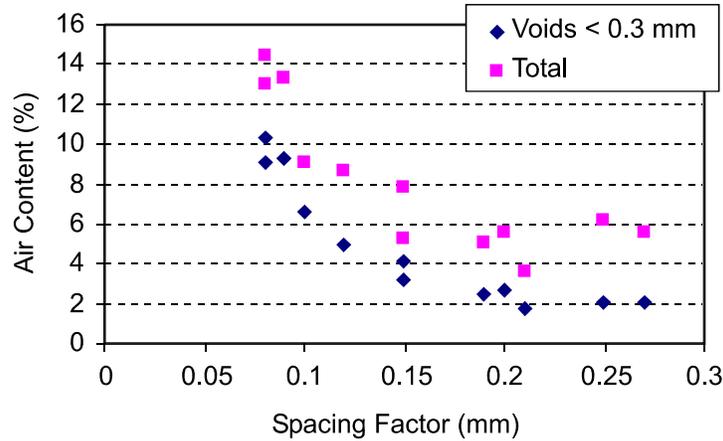


Figure 7. Relationship of Spacing Factor to Total Air Content and Air Content of Chords Less Than 0.30 mm for Vinsol Resin Mortars

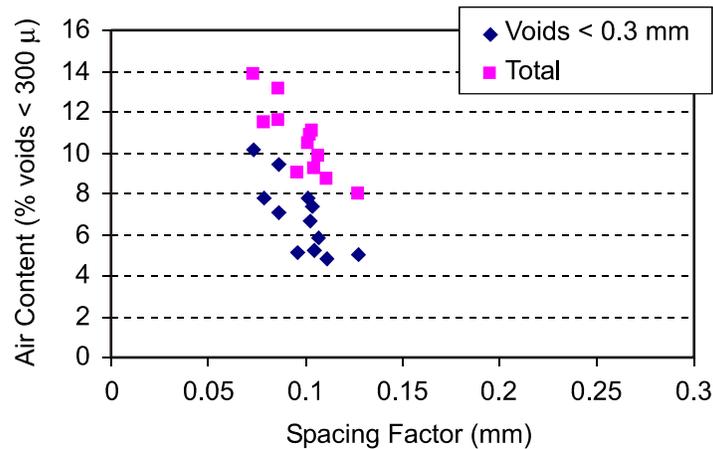


Figure 8. Relationship of Spacing Factor to Total Air Content and Air Content of Chords Less Than 0.30 mm for Tall Oil Mortars

Customarily, a spacing factor of 0.20 mm has been used as the maximum value to ensure frost resistance (Mather, 1990). Hover (1994) discussed the issues that led to the selection of this value despite indications that a maximum value of 0.25 mm (0.01 in) as proposed by Powers (1949) would be satisfactory for field exposures. The principal reason for using the 0.20 mm value was its correlation with satisfactory durability in rapid freezing-thawing tests. In two recent high-profile projects, both in severe environments, Wacker Drive in Chicago (Kaderbek et al., 2002) and the Confederation Bridge (Holley et al., 1999), a maximum spacing factor of 0.25 mm was used in construction specifications, recognizing that conditions in the rapid freezing-thawing test are far more extreme than those encountered even in severe field environments. Although it is clear that a maximum spacing factor can provide assurance of frost resistance, Pigeon and Pleau (1995) commented that spacing factors lower than 0.20 mm do not greatly improve frost resistance. Although minimum spacing factors have not appeared in specifications, it is apparent that spacing factors of around 0.10 mm and below are associated with low concrete strengths. Consequently, a spacing factor target range of 0.15 to 0.25 mm will

be used to examine Figures 7 and 8 further with the assumption that it will provide satisfactory frost resistance.

For the VR mortars (Figure 7), the target spacing factor range is achieved with total mortar air contents between 4% and 7%, below the anticipated target air content range. This range is equivalent to concrete air contents in the range of 2.5% to 4.2%. Mortar air contents above 9% total, equivalent to 5.5% for concrete, were associated with spacing factors below the desired minimum. In general, the FI provided a good first estimate of the VR AEA dosage necessary to produce a satisfactory air-void system.

Prior to the linear traverse examinations, tests of the six VR mortars with below-target total air contents (gravimetric) with the FI dosage of AEA were repeated with an increased dosage. The three mortars corresponding to those with the highest initial spacing factors (C4-F1, C5-F2, and C6-F1) were subjected to ASTM C 457 analysis. In each case, the dosage was doubled, and the gravimetric air contents increased by 130%, 100%, and 70%, respectively, but each was still below the original gravimetric target minimum of 8%. Figure 9 shows the linear traverse results in terms of void chords < 0.30 mm and spacing factor for these mortars. The increased AEA dosage pushed the marginal spacing factors of C6-F1 and C5-F2 into the desired range. For the VR AEA, a dosage that produced mortar air contents of approximately 2.5% to 5% (equivalent to concrete air contents of 1.5% to 3%) in voids with chords less than 0.30 mm resulted in air-void systems with the spacing factor in the desired range of 0.15 to 0.25 mm. Assuming a large void content of 1.5% to 2% in well-consolidated concrete (as expected in an air test), a spacing factor in the desired range should be achieved with a concrete air content in the range of 3% to 5%. In a recent study, Tanesi and Meininger reported that concrete produced using a VR admixture and having a total air content as low as 3.5% provided good performance in rapid freezing and thawing tests.

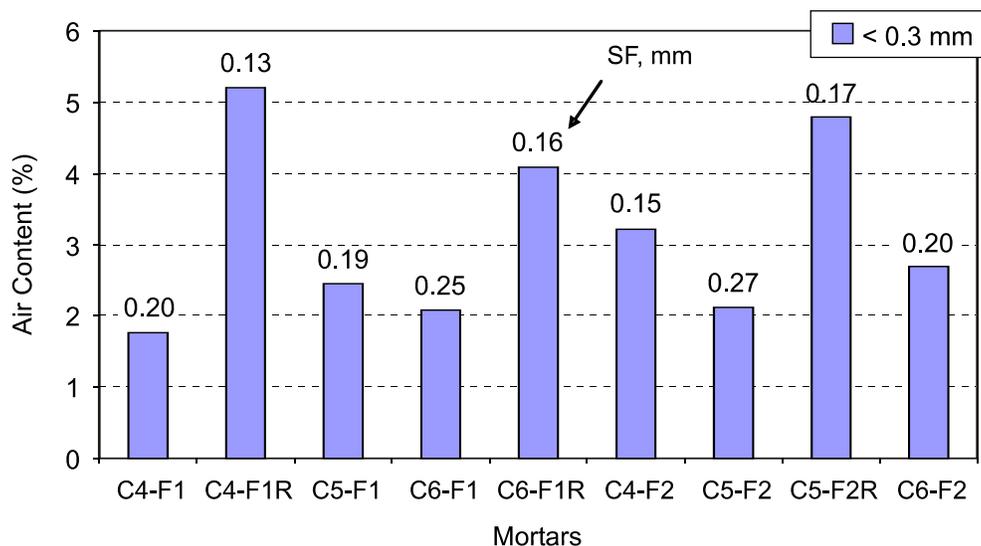


Figure 9. Air Content and Spacing Factor of VR Mortars for Cements C4, C5, And C6. VR dosage was the FI except for mortars labeled R, for which the dosage was doubled.

With regard to the results for the TO mortars (Figure 8), the total air contents were all in the desired range (8% to 13.5%) except one (13.9%), and equivalent concrete air contents for the

range are 5.8% to 8.1 %. Based on total air content, they fall essentially within the commonly specified range, but the spacing factors are much lower than necessary. Such air-void systems are often observed in concretes with lower than expected compressive strength and illustrate that the current specified range is set higher than necessary for concretes produced with TO AEA. Further work is necessary to define the appropriate target range for TO admixtures and to see if it is the same or different from that for VR AEA. In the case of the TO AEA, the FI test was not effective in predicting a suitable dosage for a good air-void system; however, the results can be used to reduce dosages to produce systems with spacing factors in the desired range. As with the VR AEA, it is expected that a lower target range can be identified. However, Tanesi and Meininger recently reported that concretes with very low air contents and satisfactory spacing factors (0.15 to 0.25 mm) produced with an AEA believed to be similar to the TO used here did not necessarily provide satisfactory performance in the rapid freezing and thawing test.

CONCLUSIONS

- *The FI test provides a good first approximation of the dosage of VR AEA to produce a satisfactory air-void system but overestimates the required dosage of TO AEA.*
- *Spacing factor is primarily a function of voids with chords less than 0.30 mm, and this can be used to define target ranges of small voids to assure adequate frost resistance.*
- *Air contents within the target range for both AEAs produced spacing factors lower than necessary for resistance to freezing and thawing. This suggests that lower target ranges for air content could be established that would provide satisfactory protection from freezing and thawing while avoiding concrete acceptance problems associated with excessive air content.*

RECOMMENDATIONS

1. The Virginia Transportation Research Council should initiate a new study to verify that the relationships between air content and spacing factor observed in this study carry over to concrete mixtures. The work should include rapid freezing and thawing tests of concrete to assure adequate durability. Study results would provide a basis for refinement of VDOT specifications for air-entrained concrete.
2. The Air-Void Analyzer should be included in the study to determine how this technology can be used by VDOT.

ACKNOWLEDGMENTS

The successful completion of this project rests on the strong contributions of Marshall Davis, who conducted the foam index and mortar tests, prepared specimens for petrographic examinations, and assisted with data analysis; Mike Burton and Bill Ordell, who performed mortar testing; and Bobby Marshall, who performed the linear traverse analyses. The comments and suggestions of Larry Lundy, David Mokarem, and Celik Ozyildirim were a benefit to the author in improving the report.

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