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FREEZE/THAW DURABILITY OF DRY MASONRY CONCRETE

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ABSTRACT

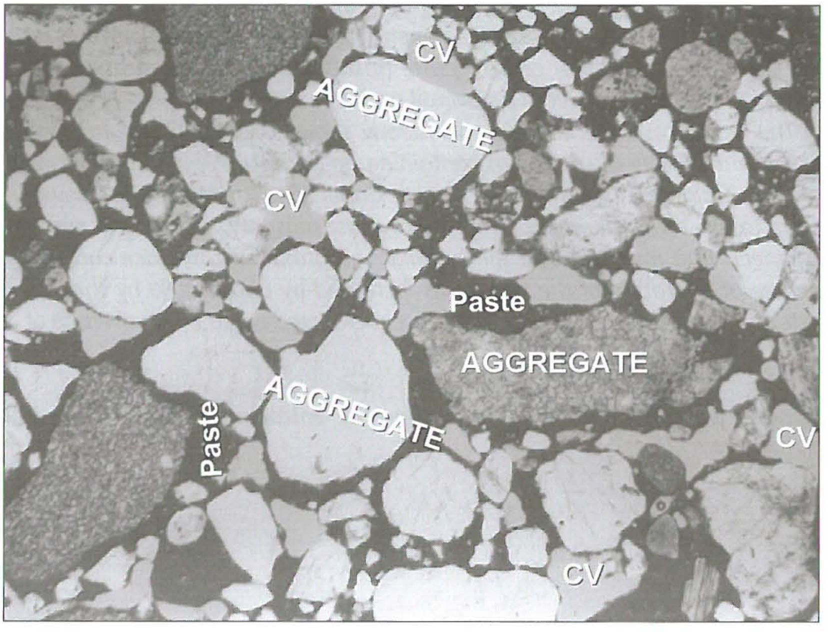
The increasing use of dry cast masonry products such as blocks, paving units or segmental retaining walls, especially in areas experiencing freezing and thawing environments, has created a need for demonstrating their long-term durability. In the present paper, the freeze/thaw durability performance of dry cast masonry products in water and in 3% sodium chloride solution is summarized. Mixes containing cement –to-aggregate ratios typically used in masonry products, ranging from 11% to 18% by weight, were studied. The relatively open and porous microstructure of masonry units and the poor quality of cement paste (insufficient cement hydration and inhomogeneity of the paste) result in an overall poor freeze-thaw performance in masonry units. The work presented in this paper clearly shows the significant increase in the freeze/thaw durability of specimens containing an admixture which both provides efficient air-entrainment and improved cement dispersion. The use of the admixture entrains up to 3% total volume of air in the dry cast masonry, resulting in spacing factors between 200 mm and 300 mm, similar to normal cast-in-place concrete. Also, the degree of hydration of the paste was increased by about 30% by the use of the admixture. Freeze/thaw durability was consequently increased by an average of about 95%.

Key words: *Masonry, durability, freeze-thaw, air-entrainment, degree of hydration.*

I. INTRODUCTION

Conventional concrete comprises the following components: aggregate and cement paste. Each of those components is porous. Over the last decades, it has been widely established that the engineering properties of cement-based materials are predominantly influenced by their pore structure characteristics. Several models have been proposed to describe the structure of hardened cement paste (Feldman *et al.* 1970; Jennings *et al.* 1994). Despite its shortcomings, the classical model proposed by Powers and Brownyard (1948) remains one of the few theoretical formulations that can be used to predict the distributions of the various classes of pores in the hydrated cement paste. Besides the air voids, the model distinguishes two different classes of pores in the cement paste: capillary pores, remnants of the water-filled space between the hydrating cement grains; and the gel pores, a fine-scale porosity present in the C-S-H gel. The size division between capillary and gel pores is, to a large extent, arbitrary. Based on a classification of pores made by Mindess and Young (1981), capillary pore radius ranges from 50 Å to 5 μm and gel pore radius ranges from 3 Å to 60 Å. However, the internal structure of a machined, no-slump masonry product is very different from conventional cast-in-place concrete. Figure 1 shows a picture of a thin section of masonry unit (magnification 16X). As seen from the picture, the structure of this material is not only formed by aggregates and paste, but also by an additional type of large pore called a compaction void. The size of compaction voids can be on the order of a few millimeters. Compaction voids are irregularly-shaped and in most cases are interconnected throughout the masonry unit. The volume of com-

Figure 1. Micrograph of an masonry thin section showing the compaction voids (CV), the aggregate and thin strips of paste holding the matrix together (magnification 16X).



paction voids is a function of the distribution of the granular structure in the mix (including cement) and of the energy of compaction during production.

The role of compaction voids in the durability of dry, no-slump material is still not well understood. There is a substantial volume of literature published over the last 50 years on the freeze-thaw durability of normal cast-in-place concrete (Powers *et al.* 1953; Helmuth 1960; Litvan 1980). But very few studies have addressed the durability issues related to dry masonry products. Some research results tend to indicate that, when compaction voids are small and isolated due to the high cement content of the mix and the high compaction energy provided during the production of the material, they can act as efficient escape boundaries and provide protection against freeze-thaw deterioration (BRITE project 1993). Others show that the compaction voids do not contribute to the protection against frost in masonry units (Pigeon *et al.* 1995). These pores can get saturated very fast; they can provide a source of free water to the bulk of the specimen and therefore contribute to the fast saturation of the paste portion of the material. As described by Fagerlund (1979), when specimens reach a critical degree of saturation, freeze-thaw damage occurs within a few cycles. Water can easily escape from compaction voids and ice can form without inducing high stresses in the material. On the other extreme, gel pores are extremely small (smaller than 60 Å) and the water they contain is adsorbed on the surface of calcium silicate hydrate particles and thus cannot freeze unless extremely low temperatures (lower than –78°C) are reached (Powers *et al.* 1947). It is therefore the presence of the intermediate size of pores, the capillary pores (size approximately between 50 Å to 5 µm), that can make the cement paste susceptible to frost damage. More than 20% of the volume of the paste can be occupied by capillary pores containing a relatively important amount of water that can start to freeze at –3 °C. Due to the dry nature of these materials, the water content of masonry mixes are low. Due to the lack of sufficient water and short curing times, the cement is generally not properly hydrated, resulting in an unhomogeneous and highly permeable paste structure that is more susceptible to freeze-thaw damage compared to conventional concrete.

It is also well known that besides the pore structure, other parameters can affect the freeze-thaw durability of dry masonry material (Pigeon, 1995). If the cooling rate and the number of freezing and thawing cycles are kept constant, as in the case of laboratory testing, the severity of the freeze-thaw damage is directly related to the water content in the porous system and the presence of deicing salts (i.e. NaCl or $CaCl_2$).

Low porosity materials, where very little freezable water is available, can be very resistant to frost damage. On the other extreme, very high porosity can be beneficial to frost protection since the material becomes saturated only at very high humidities and any water can easily escape the pore structure without inducing high stresses. Based on these observations, it seems that any condition that reduces permeability without decreasing drastically the porosity could be detrimental to the freeze-thaw durability of a cement-based material.

It is well known that, during freeze-thaw testing where the specimen is in contact with water, the water content of the specimen increases with increasing numbers of cycles. Vanderhorst *et al.* (1980) showed that the rate of water absorption of specimens during freeze/thaw testing is substantially higher than specimens subjected to prolonged periods of immersion without freezing. So while products that reduce the permeability of the material under atmospheric pressure do not necessarily prevent the accumulation of water during freezing and thawing cycles in the pores, they hinder the egress of water causing more frost damage. Integral water repellent admixtures can be classified as such products. They are used frequently in dry masonry products to reduce water movement in the units and/or to reduce the appearance of efflorescence at the surface of the material. The effect of integral water repellents on the freeze-thaw durability of masonry units is not yet fully understood. Our own laboratory data (figure 2) show that integral water repellents reduce but do not prevent the ingress of water into the pores during the freezing and thawing cycles. As shown in figure 2, when subjected to freezing and thawing cycles in this test, the specimen containing an integral water repellent absorbs water a slightly lower rate as its counterpart containing no integral water repellent.

Another factor contributing to the saturation of the cement-based material is the presence of deicing salts. Figure 3 shows the relationship between relative humidity and

Figure 2. Water uptake under freezing and thawing cycles.

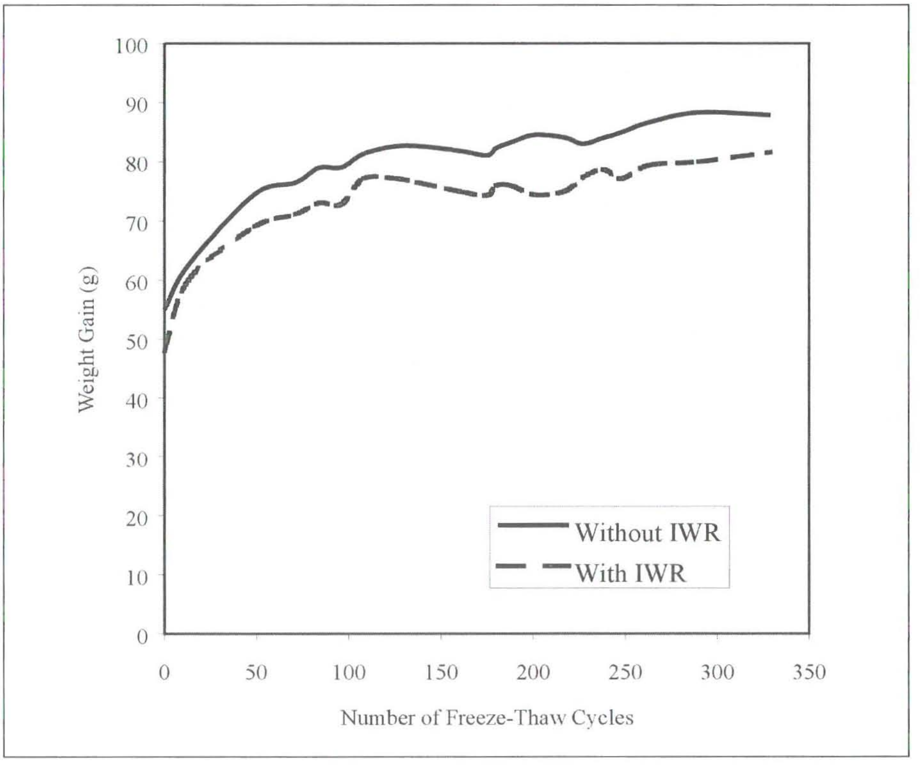
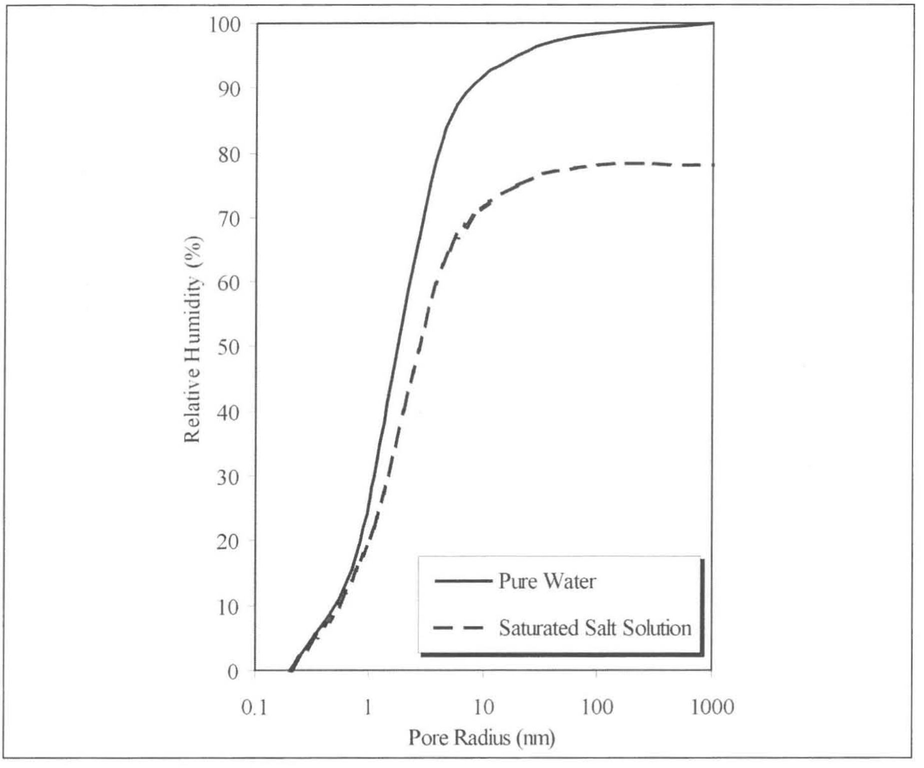


Figure 3. Relationship between relative humidity and pore radius for pure water and saturated salt solution (Dullien 1979).



pore radius obtained by the Kelvin equation (Dullien 1979), for both pure water and saturated salt solution. The relative humidity corresponding to any pore radius, is the minimum vapor pressure required to prevent evaporation and loss of liquid from that pore. Thus if pure water is filling the pores, no less than 100% relative humidity is required to maintain the saturation. However, if the pores are filled with saturated salt solution, a relative humidity of 76% is sufficient to prevent evaporation from all cement paste pores (gel and all capillary pore sizes). The presence of salt is likely to increase the rate of saturation and thus increase the risk of premature damage on freezing. Salt can have other effects contributing to the frost damage. The presence of salt could result in: decreasing the temperature of ice formation, creating more movement of water in the bulk material because of osmotic effects, or increasing the surface tension of water and thus increasing its ability to fill capillary pores in the paste (Dorner 1984). These subjects are beyond the scope of this paper.

Over the last 20 years, intentionally entrained spherical air-voids (sizes approximately from 10 to 1000 μm) have been successfully employed to protect the paste in conventional concrete against freeze-thaw damage. However, as indicated before, masonry products are inherently different than cast-in-place concrete. Primarily, it is very difficult to entrain air in masonry units because of their dry nature.

Recently, a newly developed admixture has exhibited an excellent potential in improving the freeze-thaw durability of dry masonry material by not only efficiently dispersing cement but also by allowing the entrainment of air-voids in the paste. In this report, the freeze-thaw durability of typical masonry unit mix designs containing the novel freeze-thaw admixture is examined.

II. EXPERIMENTAL PROCEDURES

In achieving the objective of this research program, the following steps of investigation were followed:

1. Evaluation of the bulk properties such as compressive strength, water absorption and unit weight. These properties were determined in accordance with ASTM C140 '*Standard Method of Sampling and Testing Concrete Masonry Units'.*

2. Determination of the freeze-thaw durability of SRW specimens both in water and 3% sodium chloride solution according to ASTM C1262 '*Standard Test Method for Evaluating the Freeze-Thaw Durability of Manufactured Concrete Masonry Units and Related Concrete Units*'. The acceptable requirements for freeze-thaw durability that the weight loss of test specimens at the conclusion of 100 cycles in water and 40 cycles in saline solution should not exceed 1% of their initial weight.

3. Determination of the freeze-thaw durability of specimens both in water and 3% sodium chloride solution according to ASTM C1262 test method.

4. Determination of the paste and the air void structure characteristics using different analytical techniques such as:

   - Optical microscope observations to determine the characteristics of the air-void system. The tests was done in accordance with ASTM C457 *'Modified Point Procedure–Count Method'*.This method allows the seperation of the spherical air voids from the irregularly shaped compaction voids.

   - Fluorescent impregnated thin sections combined with the use of an image analysis system to determine the degree of hydration of the paste.

III. RESULTS AND DISCUSSION

Bulk properties

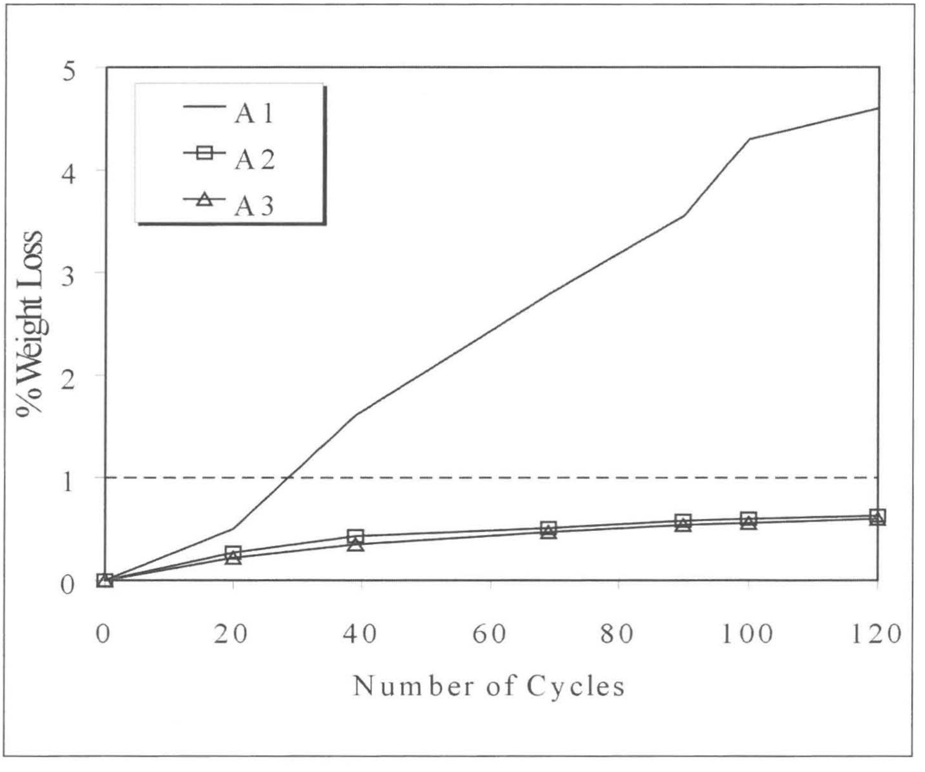
Three different sets of material were used, and identified as A, B and C. Some details of the mix designs, as well the results of the bulk properties of the mixes presented in this paper are given in Table 1. Specimens A1, B1 and C1 are the reference material containing no freeze-thaw durability enhancing admixture. The

Table 1. Mix design and physical properties.

Sample #	Cement Content (%)*	Integral Water Repellent (IWR)	Novel Freeze-Thaw Admixture (ml/100 kg cement)	28 day Compressive Strength (MPa)	Absorption (%)	Density (kg/m³)
A1	12	yes	-	29	8.7	2020
A2	12	yes	847.6	26	9.6	2023
A3	13	yes	652.0	25	10.1	2011
B1	13	yes	-	42	5.2	2203
B2	13	yes	652.0	46	4.8	2235
B3	11	yes	847.6	46	4.8	2227
C1	18	yes	-	58	3.9	2273
C2	18	no	-	49	5.8	2216
C3	18	yes	1304	38	5.7	2212
C4	15	yes	1304	42	5.7	2206

* Cement content is given as a percentage of the amount of aggregate used in the mix.

Figure 4. Freezing and thawing test results of set A in presence of water.



results of compressive strength, % absorption and density represent the average of the results of three specimens. For such systems, it is difficult to assess the exact water-to-cement ratio since the amount of water added to the mix is usually judged by the surface texture of the material after demolding. However an estima-

Figure 5. Freezing and thawing test results of set B in presence of 3% saline solution.

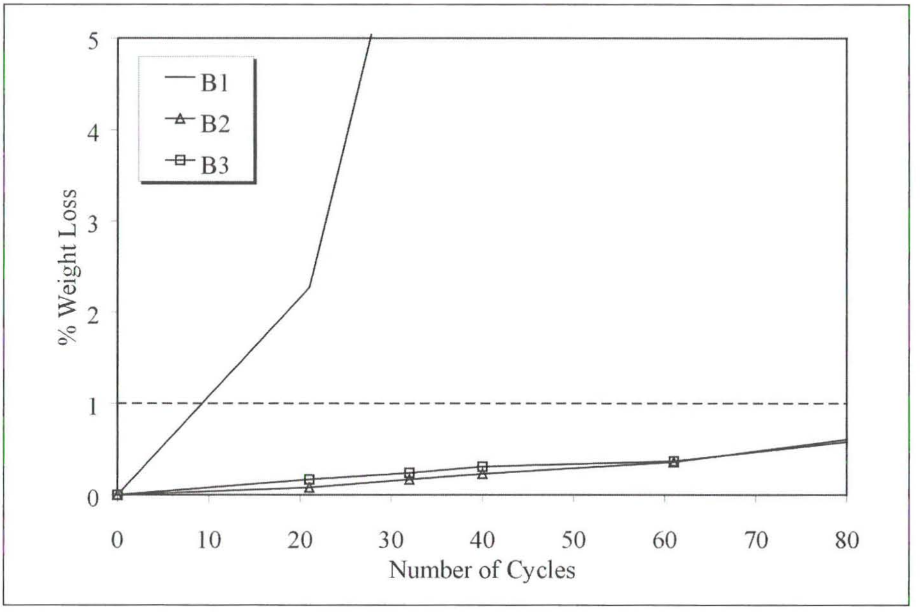
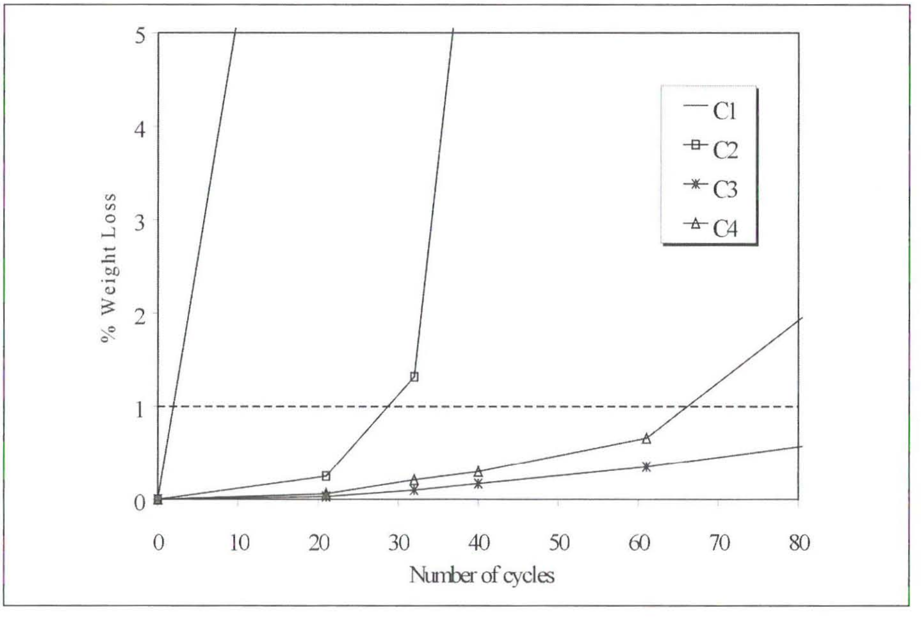


Figure 6. Freezing and thawing test results of set C in presence of 3% saline solution.



ted water to cementitious ratio of 0.30 to 0.40 can be expected. The freeze-thaw admixture studied in this paper is a novel freeze-thaw durability-enhancing admixture developed in our laboratories and based on proprietary technology.

Freeze-thaw durability

Figures 4 to 6 show the results (average of three specimens) of freeze-thaw testing for the three different sets of material. A specimens were tested only in water and B and C specimens were tested in saline solution.

As can be seen from figure 4, the mix A1, containing only an integral water repellent, starts to lose a lot of residue at around 20 cycles. Whereas in the case of specimens A2 and A3, the addition of about 650 to 850 ml/100kg of the freeze-thaw enhancing admixture reduced quite significantly the weight loss even after 120 cycles.

Again in the case of sets B (figure 5) tested in 3% saline solution, the sample containing only an integral water repellent and no freeze-thaw enhancing admixture shows a very high weight loss before even reaching 10 cycles. On the other hand, for the same mix design but this time with the addition of 652 ml/100kg of the freeze-thaw enhancing admixture (sample B2), the % of weight loss has dropped significantly at 80 cycles. In the case of sample B3, the cement content was dropped by 2% (from 13% to 11%) and still a great resistance to freeze-thaw damage was observed past 80 cycles.

Figure 6 shows the results of set C specimens, which contain a high amount of cement at 18%. Even in this case the mix containing only an integral water repellent (C1) failed very badly before even reaching 10 cycles. As seen in table 1, this mix design had a very high compressive strength result (58 MPa). Samples C3, containing the same cement content and the freeze-thaw enhancing admixture, has a compressive strength around 33% lower, but shows a remarkable freezing and thawing performance even up to 80 cycles in a 3% saline solution. These results clearly show that there is no real correlation between the physical performance of the masonry units and their freeze-thaw durability. As shown by the sample C4, when the proper freeze-thaw protection is obtained, even a reduction of cement does not reduce the freeze-thaw durability of the masonry units. Finally, the poor performance of specimen C2, containing no integral water repellent, shows clearly that the freeze-thaw damage occurs in the abscence of an integral water repellent.

In all cases the mode of failure was both in the form of scaling and/or bulk macrocracking. Figure 7 shows an example of the failure mode of a specimen containing no freeze-enhancing admixture.

Air void characteristics and paste structure

Air-voids characteristics

Figure 8 shows a polished section of masonry product specimen containing the freeze-thaw enhancing admixture. In the picture the compaction voids and the spherical entrained air-voids can be clearly distinguished. In most cases, it was possible to entrain from 1.5% to 3% air.

Figure 7. Photograph showing sample from a masonry unit without any freeze-thaw enhancing admixture before being subjected to freeze/thaw test (left) and after being subjected to 40 freeze/thaw cycles in the presence of water following ASTM C1262 test procedure (right)

Figure 8. Polished section of masonry unit containing a freeze-thaw enhancing admixture. Compaction voids are labeled as CV and the spherical entrained air voids are indicated with the arrows.

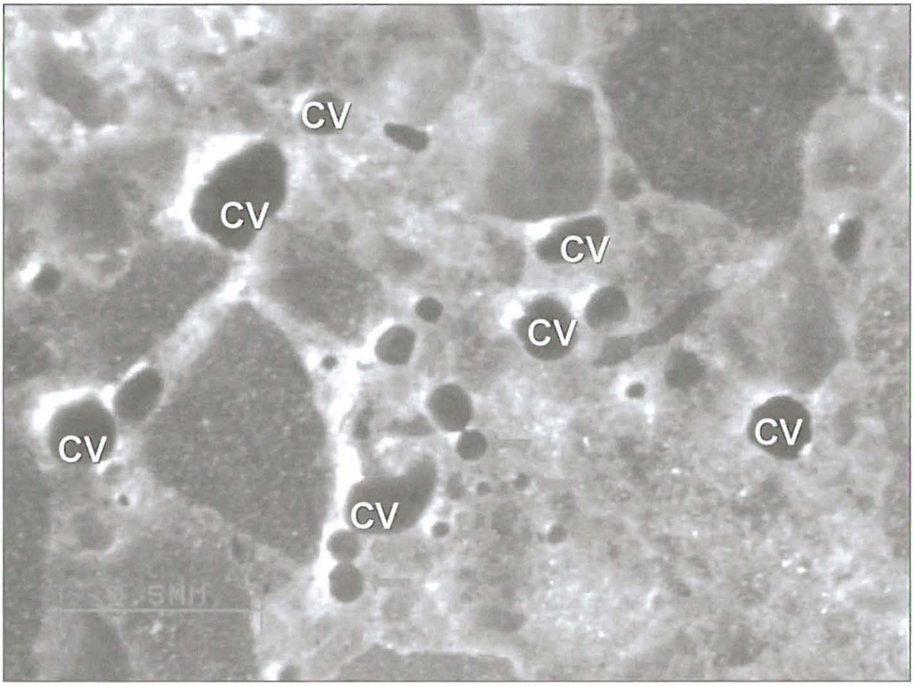


Figure 9. Correlation between spacing factor and freeze-thaw durability in presence of water.

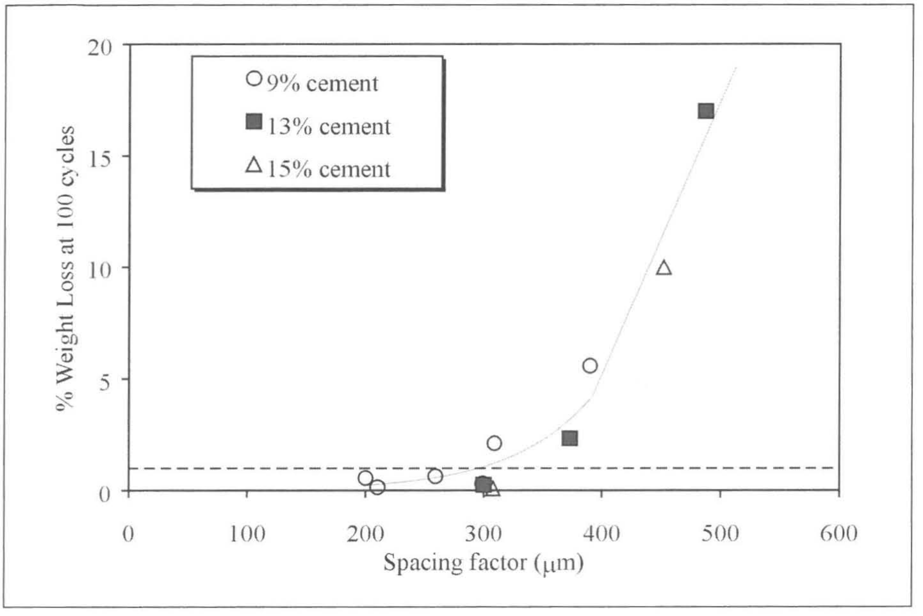
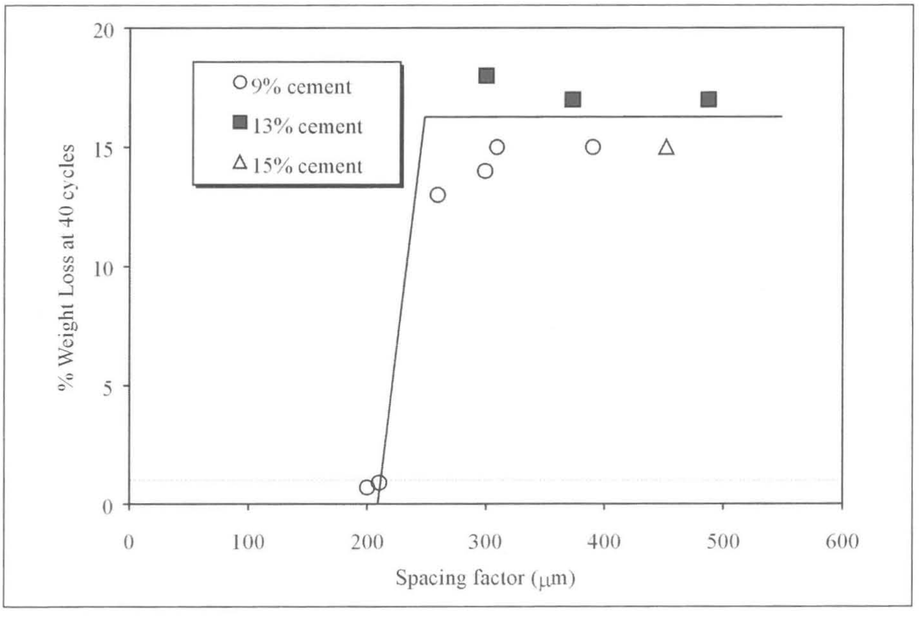


Figure 10. Correlation between spacing factor and freeze-thaw durabilityin presence of saline solution.



The work on the microscopic determination of air-void content and parameters of air-void system is still on-going. Based on the limited data that we have at this stage of the project, the spacing factors for non-air entrained and air-entrained

Figure 11. Fluorescent dye impregnated thin section of mix C1 without a freeze-thaw enhancing admixture. The arrows show clusters of unhydrated cement showing the unhomogeneity of the paste. No spherical entrained air-voids are present. Evaluated degree of hydration = 46% (magnification 25X).

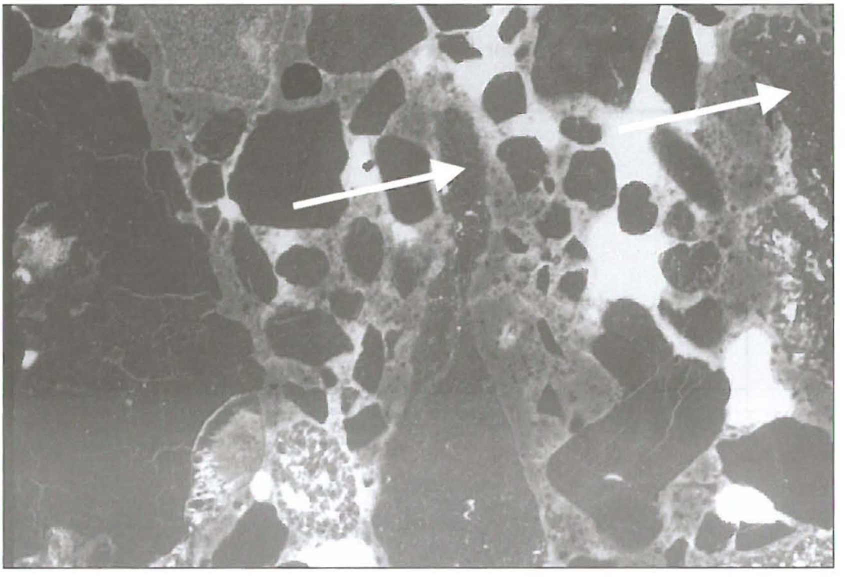
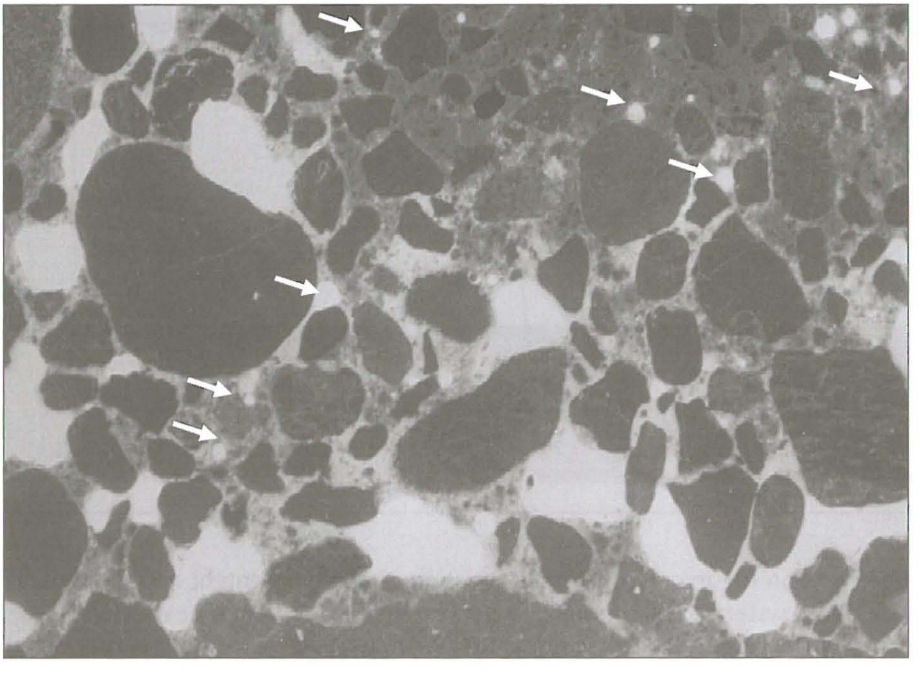


Figure 12. Fluorescent dye impregnated thin section of mix C3 containing a freeze-thaw enhancing admixture. Spherical entrained air voids are indicated with the arrows and no cluster of unhydrated cement grains are observed. Evaluated degree of hydration = 66% (magnification 25X).



mixes ranged from 450 μm to 550 μm and from 200 μm to 300 μm respectively. It is well established by now that in normal concrete, satisfactory freeze/thaw durability is obtained with a spacing factor less than 200 μm. But in the case of dry no-slump masonry material no data is available in the literature on this relationship. The results of our preliminary findings on the correlation between air-void characteristics and freeze-thaw durability are summarized in figures 9 and 10 for both freeze-thaw tests conducted in the presence of plain water and in the presence of a 3% saline solution. In both graphs the % weight loss at a fixed number of freeze-thaw cycles (100 cycles in water and 40 cycles in saline solution) are shown as a function of air void spacing factor at varying cement content. Because of the harsher nature of the saline freeze-thaw test, the freeze-thaw performance of the specimens exposed to saline solution is studied at a lower number of cycles. In both graphs the overwhelming effect of entrained air and the smaller effect of cement content on the freeze-thaw performance of masonry units can be seen. Again, based on our limited data, it can be said that in the presence of water (figure 9), spacing factors up to 300 μm are sufficient to obtain a durable material, and in saline solution (figure 10), a much harsher test 200 μm is absolutely necessary to reduce the % of weight loss below 1% at 40 cycles.

Paste structure

In order to increase the ability of the paste to withstand the freeze-thaw stresses, it is crucial, in the case of dry masonry products, to improve the hydration of cement to obtain a homogeneous paste. The very nature of dry masonry products and the fast curing methods used, make the hydration of the cement in masonry units very difficult to acheive. In general not enough water is available and the presence of clusters of unhydrated cement grains are common, as in the case of sample C1, shown in figure 11. Based on results obtained by image analysis on thin fluorescent dye impregnated sections, the degree of hydration of mixes containing only an integral water repellent is, in most cases, below 50%. The unhydrated cement grains can create weak regions where freeze-thaw damage can start. Besides entraining air, the other important attribute of the novel freeze-thaw enhancing admixture studied in this paper seems to be its efficiency in dispersing the cement grains. As shown in figure 12, the mix C3 containing a freeze-thaw enhancing admixture shows the absence of unhydrated cement grain clusters, and the degree of hydration was significantly increased up to 66%.

IV. CONCLUSIONS

The results clearly show that the use of the novel admixture studied in this paper increases significantly the freeze-thaw durability of dry masonry specimens, both in water and in saline solution. In general, the % weight loss was reduced by an average of about 95%.

The freeze-thaw admixture has the combined effect of entraining suficient air-voids and effectively dispersing the cement. This performance was achieved with mixes containing anywhere between 9% and 18% cement over aggregate.

It was possible to entrain a total volume of 2 to 3% air, resulting in spacing factors between 200 to 300 μm.

The addition of the freeze-thaw admixture allowed an increase in the degree of hydration of about 20% compared to the reference material. Moreover the efficient use of the cement could permit a reduction in cement content.

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