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Doubling the service life of concrete structures. II: Performance of nanoscale viscosity modifiers in mortars

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ABSTRACT

A new approach for increasing the service life of concrete structures is evaluated in a series of mortar specimens. The new approach consists of employing nanoscale viscosity modifiers to increase the viscosity of the concrete pore solution and concurrently and proportionally decrease the diffusion rates of deleterious ions such as chlorides and sulfates. In part I of this series, viscosities of bulk solutions of the admixtures in water and electrical conductivities of admixture solutions also containing potassium chloride were examined to verify the viability of this new technology. In the current paper, these studies are extended to quantifying the performance of one of these admixtures in mortars by measuring the penetration depth of chloride ions in cylindrical specimens exposed to a 1 mol/L chloride ion solution for up to 1 year. While significant reductions in the 1 year penetration depth are produced when the viscosity modifier is utilized via conventional addition to the mixing water, the best performance is achieved when a solution of the viscosity modifier is utilized to pre-wet fine lightweight aggregates that are then added to the mortar mixture. A scaling function appropriate for radial diffusion was used to estimate the relative effective diffusion coefficients. Compared to a reference mortar, the best mixture reduced the effective diffusion coefficient by a factor of 2.7, consistent with the overall objective of doubling concrete service life.

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1. Introduction

Recent emphasis on sustainability has reinforced the critical importance of service life of construction materials. No sustainability analysis can be complete without a quantitative and accurate prediction of service life, and extending the service life of a material or system can greatly enhance its "sustainability index" [1]. In part I of this series [2], a new paradigm for enhancing (doubling) the service life of concrete structures was presented based on the application of nanoscale viscosity modifiers to reduce diffusion rates of deleterious ions such as chlorides and sulfates. The viability of this approach was examined via measurements of solution viscosities and electrical conductivities, by which it was demonstrated that significant transport reductions could only be obtained with nanoscale molecules (molecular masses of 1000 or less) and not with much larger viscosity modifiers such as xanthum gum and high molecular mass cellulose ethers, in agreement with previous studies [3,4]. In the current paper, this effort is extended to examining the performance of one of these nanoscale viscosity modifiers in a series of mortars that have been exposed to 1 mol/L chloride ion solutions for 1 year. Three approaches for introducing the viscosity modifier into the mortar mixture have been examined: conventional introduction as an admixture during mixing, topical introduction as a curing solution, and introduction via fine lightweight aggregates (LWA) that have been saturated with a solution of the admixture [5]. Control mixtures consist both of a conventional mixture with no viscosity modifier and no LWA and a mixture with no viscosity modifier but containing LWA saturated only with water, the latter effectively comprising a mortar with internal curing. These approaches are strictly applicable for decreasing transport through uncracked material, although it is worth noting that both the reduced surface tension of the viscosity modifier investigated in this study and the incorporation of internal curing via the fine lightweight aggregates should additionally contribute to a reduction in the early-age cracking of these materials [6].

2. Materials and experimental procedures

For this study, a single Type I/II portland cement was used for the different mortar mixtures. The measured oxide composition and Bogue potential phase composition, as provided by the cement manufacturer, are listed in Table 1. A Blaine fineness of 380 m^2/kg was reported by the manufacturer. The cement powder specific

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Table 1
Cement oxide composition and Bogue potential phase mass fractions as provided by the cement manufacturer.

Oxide or property	Mass fraction
CaO	0.630
SiO_2	0.203
Al_2O_3	0.047
Fe_2O_3	0.035
SO_3	0.029
MgO	0.032
Na equivalent	0.54
Bogue – C_4AF	0.11
Bogue – C_3A	0.07
Bogue – C_3S	0.58
Bogue – C_2S	0.15

gravity was measured at the National Institute of Standards and Technology (NIST) using ASTM C188 [7]; two replicates each gave a value of 3220 kg/m^3. The measured particle size distribution (via laser diffraction) of the cement is given in Fig. 1.

The LWA, an expanded shale, was a commercially available product. It has a saturated-surface-dry (SSD) specific gravity of 1.80 ± 0.05 (one standard deviation) and a measured desorption of approximately 20% when exposed to a slurry of potassium nitrate (relative humidity of 93%). The total absorption capacity of the LWA was approximately 22% by mass, as measured by drying an SSD sample. The size distribution of the LWA was determined by sieving in order to replace the same volume and a similar size distribution of normal weight sand in the mortar mixtures with LWA.

Mortars were prepared with a water-to-cement mass ratio (w/c) of 0.4 and a sand volume fraction of 55%. Besides the viscosity modifier described below, no other chemical admixtures were added to the mortars. For the mortar with the viscosity modifier added directly to the mixing water, a 10% solution prepared from a commercially available polyoxyalkylene alkyl ether was employed. This additive increased the viscosity of the initial mixing solution to 1.5 times that of distilled water [2]. For the mortar with the viscosity modifier added via saturated LWA, a 50:50 (mass basis) solution of the additive in distilled water was employed to pre-wet the LWA in a sealed jar. Sufficient LWA replaced the normal weight sand on an equal volume basis to provide an extra 0.08 mass units of this solution per unit mass of cement, effectively achieving the same volume fraction of the viscosity modifier for both of these addition methods. The same mixture proportions were employed for the control mortar with LWA saturated with only water, simply by replacing the 50:50 solution with distilled water. Air contents of the fresh mortars, as determined by unit weight measurements, ranged between 2.3% and 4%. Each mortar mixture was used to cast a series of 50 mm diameter by 100 mm length cylinders for further evaluation.

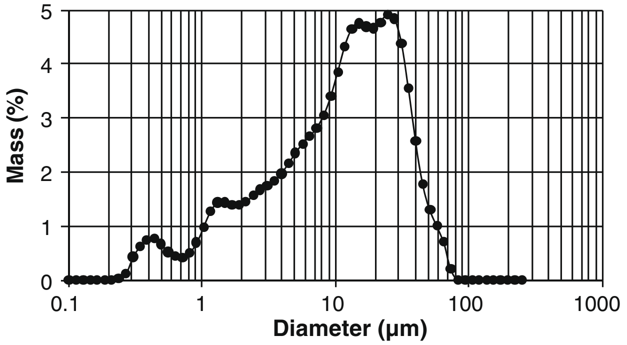

Fig. 1. Measured differential particle size distribution for the cement. The *x*-axis indicates an equivalent spherical particle diameter and the *y*-axis the mass% at each given diameter. The shown results are the average of six individual measurements and the error bars (one standard deviation) would fall within the size of the shown symbols.

For curing, three different procedures were employed after demolding the cylinders at an age of 1 day. The cylinders produced without LWA were cured for either an additional 6 days or 27 days in a solution of NaOH/KOH/$Ca(OH)_2$ [0.031 mol/L NaOH, 0.165 mol/L KOH, and saturated with $Ca(OH)_2$] to prevent leaching of alkalis from the cylinders or were cured for the same time periods in a solution of this same alkali hydroxide mixture along with the viscosity modifier (10% by total mass of water and viscosity modifier) to mimic topical addition of the viscosity modifier. The mortars with LWA, that effectively employed internal curing, were sealed in double plastic bags until their time of being exposed to chlorides.

After curing times of 7 days and 28 days, each mortar cylinder was submerged in 600 g of 1 mol/L chloride ion solution stored in individual sealed plastic bottles. After chloride ion exposure times of 28 days, 56 days, 180 days, and 365 days, two cylinders from each mixture were removed from their exposure solutions and split lengthwise using a universal testing machine. For each specimen, one of the exposed faces was sprayed with a 0.1 mol/L solution of silver nitrate ($AgNO_3$) [8,9], photographed, and image processing software employed to assess the average depth of chloride ion penetration. To do this, the area of the specimen that is brown (indicating no chloride penetration) was determined using the software and compared to the area occupied by the complete (2-D) specimen face. An example of these images for three of the mortar specimens first cured for 28 days and then exposed to the 1 mol/L chloride ion solution for 365 days is provided in Fig. 2.

In addition to measuring chloride ion penetration depths, hydration kinetics of the various mortars were evaluated using both isothermal and semi-adiabatic calorimetry. For isothermal calorimetry, the heat of hydration was measured during the first 7 days of hydration on pre-mixed (as opposed to being mixed in situ in the calorimeter cells) sealed mortar samples with a mass between 6.6 g and 7.6 g using a TAM Air Calorimeter.[1] To provide an indication of variability, two specimens from the same batch were evaluated in neighboring calorimeter cells for each experiment; in the results to follow, heat flow values are normalized per unit mass of cement in the sample. For semi-adiabatic calorimetry, the semi-adiabatic temperature of the specimen was measured during the first 3 days of hydration on a single sealed mortar specimen with a mass of approximately 380 g using a custom-built semi-adiabatic calorimeter unit [10]; replicate specimens from separate batches have indicated a standard deviation of 1.4 °C in the maximum specimen temperature achieved during a 3 day test.

3. Results and discussion

It is well recognized that a 10% solution of a chemical admixture can significantly impact the hydration kinetics of a cement-based material [2]. For this reason, hydration kinetics of the various mortar mixtures were assessed using both isothermal and semi-adiabatic calorimetry. The isothermal calorimetry results for the four mortar mixtures are provided in Fig. 3. The addition of the viscosity modifier either via a conventional route or via pre-wetted LWA retards the hydration reactions, shifting the main hydration peak, but by less than 2 h. This mild retardation is likely linked to the increased viscosity of the pore solution slowing down dissolution

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[1] Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification imply endorsement by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

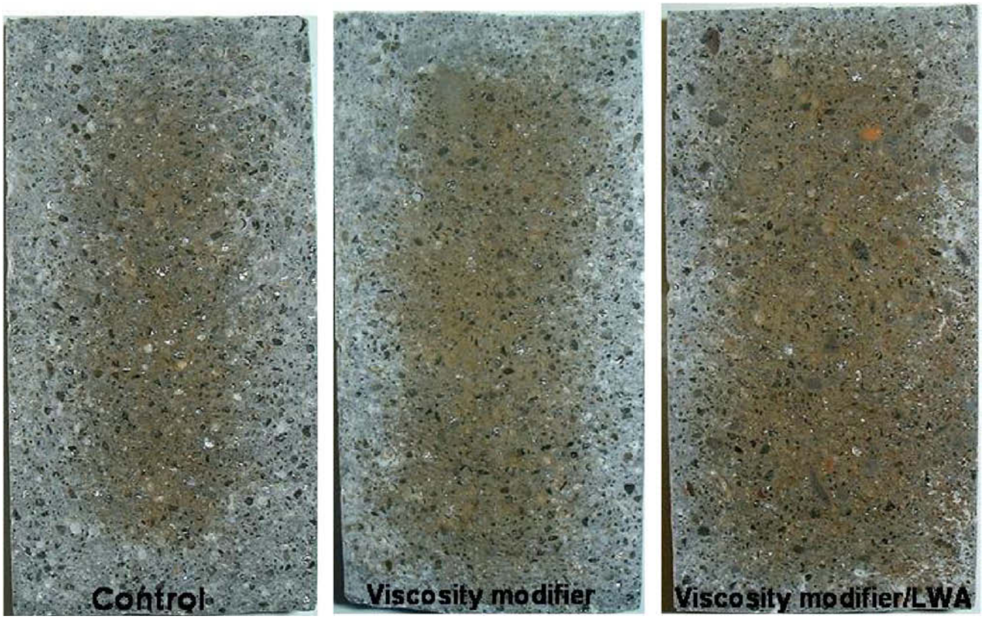

Fig. 2. Unprocessed images for three mortar specimens (control, viscosity modifier in the mixing water, and viscosity modifier solution in the LWA) that had been cured for 28 days and then exposed to chlorides for 365 days. Outer grey region indicates depth of chloride ion penetration into each specimen.

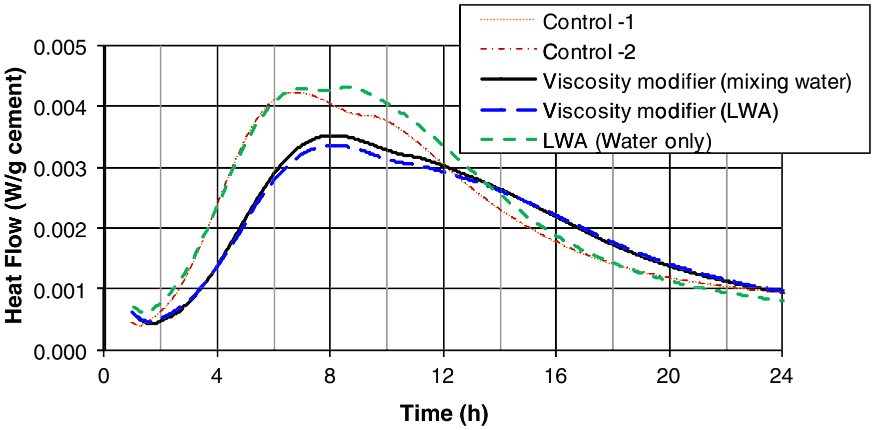

Fig. 3. Heat flow vs. time during the first 24 h of hydration for the mortar mixtures. Two replicates (basically overlapping one another) are shown for the control mortar to give some idea of measurement uncertainty.

and diffusion processes that are contributing to the early-age hydration reactions [11]. While not necessarily desirable, this mild retardation could be acceptable from a field performance viewpoint or might be offset by employing an accelerator. The mortar mixture with LWA saturated with only water exhibits a slight acceleration of the hydration reactions relative to the control mortar at ages beyond 6 h, as has been noted previously [12]. Similar performance trends are observed in the results for semi-adiabatic calorimetry shown in Fig. 4, with the mortars with the viscosity modifier once again exhibiting about a 2 h delay with respect to the control mortar, along with a slight acceleration of less than 1 h for the mortar with internal curing.

Results for the measured penetration depths for the various mortars are shown in Figs. 5 and 6, for the 7 days and 28 days curing times, respectively. As would be expected, the penetrations in Fig. 6 are slightly smaller than those in Fig. 5, due to the densification of the mortar microstructure provided by the additional curing/hydration from 7 days to 28 days. For exposure to chloride ions at these early ages, it must be recognized that ion transport into the specimens will occur by multiple mechanisms, including both diffusion of ions in the solution-filled porosity and sorption (imbibition) of the surrounding fluid to satisfy the ongoing demand created by the chemical shrinkage that accompanies the cement hydration reactions [5,13]. This influence of sorption is clearly observed for the specimens only cured for 7 days in Fig. 5, where a significant increase in penetration depth during the first 28 days of exposure to chloride ions is followed by no discernible further increase between 28 days and 56 days. Due to significant hydration occurring between 7 days and 35 days of age, the external 1 mol/L chloride ion solution is drawn into the cylinder. This has the effect of redefining the “zero” depth of penetration to be approximately 6 mm. At later ages, this sorption process becomes much less significant due to the substantial decreases in both the hydration (chemical shrinkage) and the sorption rates of the specimens. As this sorption generated front of chlorides later disperses by diffusion, the local concentration at the originally measured (28 days exposure) penetration depth may fall below that detectable by the silver nitrate spray technique, leading to an observed decrease in penetration depth at 56 days (Fig. 5). Such disparities from expected behavior are not generally observed for the specimens that

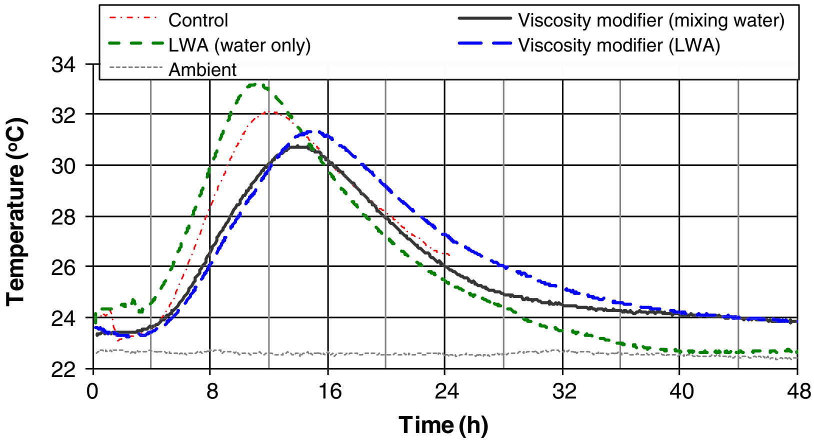

Fig. 4. Semi-adiabatic temperature response for the mortar mixtures. Data for the control mixture is only shown for the first 24 h, due to instability of laboratory temperature conditions beyond that point.

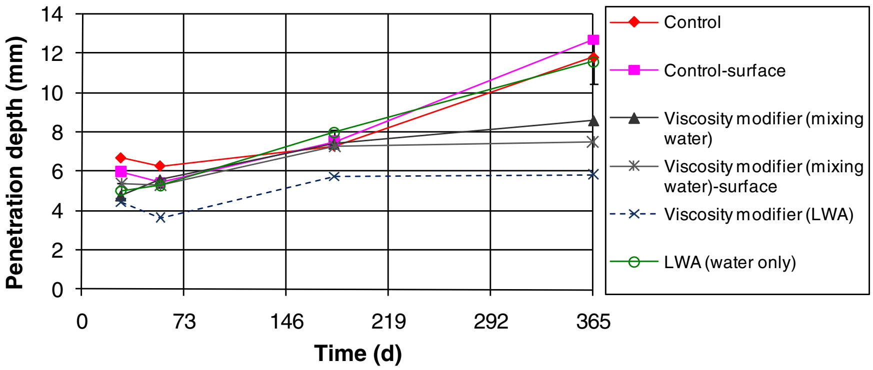

Fig. 5. Measured chloride ion penetration depths vs. chloride exposure time for the mortar mixtures first cured for 7 days and then exposed to 1 mol/L chloride ion solutions. Bars on LWA (water only) data provide an indication of measurement uncertainty (standard deviation) for two replicate specimens. In the legend, the designation of surface indicates those specimens that were cured in a solution of the viscosity modifier/alkali hydroxide mixture.

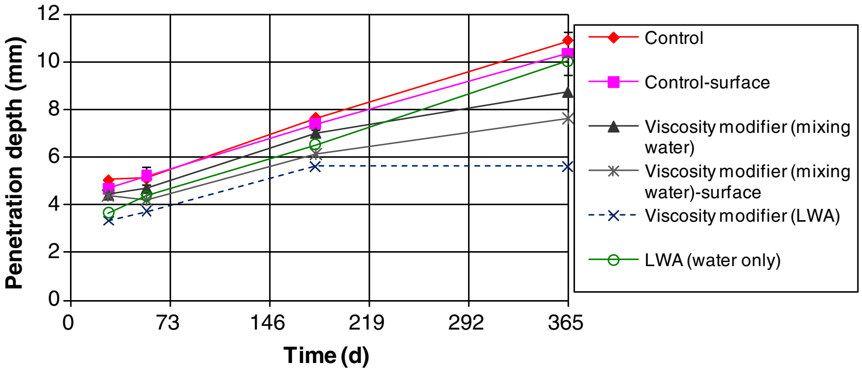

Fig. 6. Measured chloride ion penetration depths vs. chloride exposure time for the mortar mixtures first cured for 28 days and then exposed to 1 mol/L chloride ion solutions. Bars on control-surface data provide an indication of measurement uncertainty (standard deviation) for two replicate specimens.

were first cured for 28 days (Fig. 6), as by that point in time, the hydration reactions have proceeded to a point where the subsequent chemical shrinkage-induced sorption is less significant. In support of this, mass measurements of all cylinders after demolding, after curing, and after chloride exposure indicated that the mass gain during the first 28 days of chloride ion exposure for the specimens first cured for 28 days was less than half of that measured for those specimens cured for only 7 days prior to a 28 day chloride exposure.

Focusing on the results in Fig. 6 (in the hopes that most concretes will not be exposed to chloride ions during their first 28 days), while all systems with the viscosity modifier offer some advantages by reducing chloride ion penetration depths, the best performance is clearly achieved by the mortar in which the viscosity modifier is added by pre-wetting LWA. This approach provides the lowest penetration depth at all assessed ages and exhibits a relative performance that improves with time.

The reasons for the improvement in performance over longer exposures could be several. First, the internal curing provided by the solution in the pre-wetted LWA should result in enhanced hydration of the cement paste and the formation of denser interfacial transition zone (ITZ) microstructures, perhaps leading to depercolation of the more porous ITZ regions surrounding the normal weight sand particles [12]. This first cause is supported by the positive results obtained in this study for the mortar containing LWA pre-wetted with only water and cured under sealed conditions for 28 days, and is consistent with recent measurements of diffusion coefficients of lightweight concrete by Thomas [14], who found that while short term diffusion coefficients were reduced by 15–25% by the incorporation of LWA, long term (3 years of exposure) values could be decreased by as much as 70%. To obtain these benefits from internal curing, the necessity of an adequate sealed curing period is demonstrated by the significant difference in long term performance of the mortars with LWA saturated with only water and cured under sealed conditions for either 7 days or 28 days. A 7 day sealed curing period may be insufficient to depercolate the capillary porosity in the mortar, so that the LWA will quickly resaturate when exposed to the chloride solution and then contribute to diffusive pathways through the microstructure. Conversely, after 28 days of sealed curing, a dense microstructure has formed around the LWA such that they are not easily (if at all) resaturated upon exposure to the chloride solution. In this latter case, the pores in the LWA should remain vapor-filled and will not substantially contribute to diffusive transport. This hypothesis is supported by the results of Pyc et al., whose mass measurements suggested that once the pores in LWA empty during internal curing of concretes with intermediate size LWA, they are not subsequently resaturated, even upon complete immersion [15].

A second contribution to the substantial reduction in measured penetration depths could be that as hydration occurs over time, the volume of pore solution (porosity) is decreased so that the concentration of the viscosity modifier will be concurrently increased, neglecting any absorption of the viscosity modifier by hydration products. Results in the part I paper of this study indicated that if the concentration of the viscosity modifier used in the present study is doubled from 10% to 20%, the corresponding relative viscosity (relative to distilled water) increases from 1.5 to 2.6 [2]. This substantial increase in solution viscosity should be accompanied by a proportional decrease in ionic diffusion rates, consistent with the observed change in slope between 180 days and 365 days for the specimens with the viscosity modifier vs. the control specimens in Fig. 6.

The relative effective diffusion coefficients among the mixtures can be estimated from the measured penetration depths. For one-dimensional diffusion in Cartesian coordinates, the ratio of the diffusion coefficients of two materials is proportional to the ratio of the squares of the penetration depths at the same exposure times. However, for pure radial diffusion in cylindrical coordinates, the relationship is more complex as described below.

Deriving the relationship between penetration depth and diffusion coefficient for radial diffusion requires first studying the concentration profiles in a cylinder [16]. For purely radial diffusion into a cylinder having radius R, and assuming the external concentration C_o is constant, the concentration C profile is shown in Fig. 7 as a function of the scaled radius r/R. A constant external concentration is a valid approximation for this experiment because the mortar capillary porosity is a small fraction of the external chloride solution volume. In support of this, for specimens exposed to chlorides for 6 months, the measured chloride ion concentration in the external solution was typically on the order of 0.8 mol/L, in comparison to the initial 1 mol/L concentration. The curves are parameterized by the dimensionless quantity Dt/R^2, where D is the diffusion coefficient and t is the exposure time. After spraying a fractured surface with silver nitrate, the visual inspection of the sample reveals a reaction boundary where the chloride concentration is above a critical value C_{crit}. Because this critical concentration ratio is relatively constant for a particular experiment [8,9], observations of silver nitrate staining are made along horizontal lines in Fig. 7, such as the two marked dashed lines.

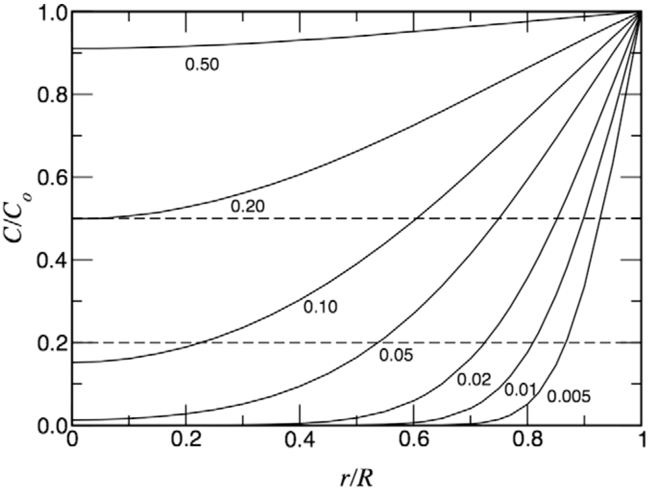

Fig. 7. The concentration profiles for radial diffusion in a cylinder. The concentration C is scaled by the constant external concentration C_o, and the radius r is scaled by the cylinder radius R. The numbers indicate the value of the dimensionless parameter Dt/R^2.

In Fig. 7, two horizontal dashed lines are drawn to represent two possible C_{crit}/C_o values for consideration: 0.20 and 0.50; given the continuous progression of observed penetration depths in Fig. 6, a larger C_{crit}/C_o is unlikely. Moreover, Baroghel-Bouny et al. have reported values ranging between 0.13 and 0.44 for this ratio (considering total chlorides) for exposures to solutions with a nominal concentration of 1 mol/L [8,9]. The values of Dt/R^2 as one moves horizontally along each of the two dashed lines in Fig. 7 are plotted in Fig. 8 as thick solid lines. Here, the data are plotted as a function of the relative penetration p, the ratio of the penetration depth divided by the cylinder radius R. The data are plotted on a log–log graph, so the slope of the line equals the exponent of the penetration that scales the diffusivity. Also shown in the figure are two thin straight solid lines denoting lines with a slope equal to two. Note that the only point where the curves have a slope equal to two is at the limit of very small penetrations. As the chlorides penetrate farther into the cylinder, the slope de-

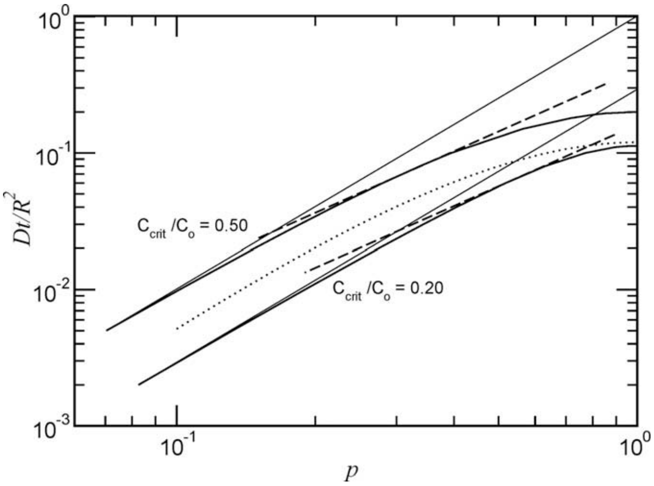

Fig. 8. The dimensionless parameter Dt/R^2 as a function of the relative penetration p for two values of C_{crit}/C_o (thick curved solid lines). The thin solid lines have a constant slope equal to 2, and the dashed lines have a slope of 3/2. The dotted line is proportional to the scaling parameter s used in this study.

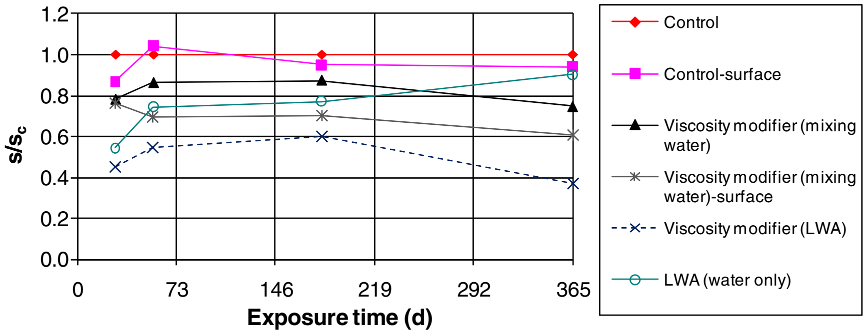

Fig. 9. The ratio s/s_c vs. exposure time for the mortar mixtures first cured for 28 days and then exposed to 1 mol/L chloride ion solutions.

creases, ultimately achieving a value of zero at complete penetration. For comparison purposes, the dashed lines have slope 3/2, so the decrease in slope is not dramatic for values of relative penetration p up to 0.5.

The two curves in Fig. 8 represent the functional relationship between penetration depth and diffusion coefficient. The dotted line in Fig. 8 is an analytical approximation to these curves. This curve is proportional to a scaling function s that has the following dependence on the relative penetration depth p:

$$s = p^{2(1-\sqrt{p})} \tag{1}$$

Therefore, for measurements at a fixed time on cylinders having the same radius, the ratio D/s is a constant, and from this, the ratio of two different diffusivities can be estimated from the relative penetration depths:

$$\frac{D_1}{D_2} = \frac{p_1^{2(1-\sqrt{p_1})}}{p_2^{2(1-\sqrt{p_2})}} \tag{2}$$

When the values of p_1 and p_2 are small, the Cartesian scaling is recovered where the ratio of the diffusivities is proportional to the ratio of the penetration depths squared.

The data in Fig. 6 are replotted in Fig. 9, showing the ratio of s/s_c vs. exposure time, where s_c is the value of s for the control mortar. The values thus plotted indicate the relative diffusivities (compared with the control) for each mortar mixture. For the mortar prepared with the conventional addition of the viscosity modifier, the 1 year value of s is reduced to 75% of the control value. For the mortar prepared with the viscosity modifier added via the LWA, the 1 year value of s is reduced to 37% of the control value. For applications where service life is directly inversely proportional to diffusivity, this would imply a service life that is increased by a factor of 2.7. Thus, this technology still appears to be a promising route for doubling the service life of concrete, as first proposed in the part I paper of this study [2]. Future research will focus on extending these validation studies to field concrete mixtures and on continuing the search for alternative viscosity modifiers that may offer better performance, be more economical, or both.

In addition to reducing diffusion rates, these viscosity modifiers should also offer benefits with regards to flow under pressure and transport via sorption [17]. While an increase in the pore solution viscosity does not change the permeability of the concrete microstructure, the flow rate of a fluid within the concrete due to a pressure gradient will be inversely proportional to this viscosity. The sorptivity coefficient of a concrete (or any porous material in general) is proportional to the inverse of the square root of viscosity [18], so that a higher pore solution viscosity will produce a lower sorptivity coefficient. The reduced surface tension provided by the viscosity modifier examined in this study will further reduce the sorptivity coefficient (and also likely reduce autogenous and drying shrinkage) [4,18]. Additionally, the enhanced hydration achieved in the concrete in which the viscosity modifier is introduced by pre-wetting LWA will produce a denser concrete with lower permeability and sorptivity coefficients [19].

4. Conclusions

This study has validated a new approach for reducing the rate of diffusion into cement-based materials for a series of mortar specimens. By appropriately increasing the viscosity of the mortar pore solution using nanoscale viscosity modifiers, significant reductions in the penetration depth of chloride ions are achievable. A particularly efficacious approach to introducing the viscosity modifier into the mixture is to pre-wet fine lightweight aggregates with a concentrated solution of the admixture in water. This approach produced the greatest reductions in measured chloride ion penetration depths, due to the beneficial influences of both the viscosity modifier and the internal curing provided by employing pre-wetted fine LWA. An analysis of radial diffusion revealed a scaling function from which the effective diffusion coefficient could be inferred based on the radial penetration depth. Compared to a reference mortar mixture, the factor of 2.7 reduction in the effective diffusion coefficient through the combined use of a viscosity modifier and LWA may be a viable approach to doubling service life of reinforced concrete structures.

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