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Porosity and Water Absorption of Concrete with Multi-Crystalline Waterproofing Admixture: The Effect of Admixture Dosage and Ratio of Water to Cementitious Materials

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Abstract – Concrete durability issues are associated with water penetration and concrete porosity. This submission investigates the performance of liquid multi crystalline waterproofing admixture (MCE) manufactured by International Chem-Crete Corporation (Dallas, TX, USA). The investigation included the two dosages of 1% and 2% MCE by the weight of cementitious materials. The standard testing methods described in ASTM C642 was used to measure density, water absorption and volume fraction of permeable pores for reference and MCE-dosed concrete specimens. This was done using the mix design of Iowa Department of Transportation (Iowa DOT) with various ratios of water to cementitious materials (w/cm) ranging from 0.37 to 0.54. The results are presented as curves of water absorption, fraction of permeable pores and density for reference and 1% and 2% dosed concrete as functions of w/cm ratio. The findings demonstrate the efficiency of each of the investigated dosages of MCE in pore blocking waterproofing. MCE reduces the fraction of permeable pores and water absorption at the two dosages, with a slight densifying effect at 2% MCE. At high w/cm ratio, 2% MCE is recommended.

Keywords**:** concrete, moisture, pores, absorption, waterproofing, crystallization, durability, ASTM C642.

1. Introduction

Concrete structure is a porous composite material consisting of mineral aggregates bonded together by hydrated cementitious paste (1, 2). The structure of the paste involves a network of hydration solid products and voids involving bound and free water (3, 4). The structure of pores and voids is complex ranging from less than 1 nm to 1 μm, with six orders of magnitude variations (5). The paste porosity (about 5–6% of the paste volume) is a structural characteristic that is controlled by the size and distribution of the pores and their connectivity (6, 7). The pores, involve various types and sizes as listed in Table 1 (8). These includes (1) fine gel enclosed that are generally isolated within the cement hydration layers (within the C-S-H crystals or gel) and (2) large capillary pores surrounding the hydration crystals and forming connected networks that allows water penetration. Hence, from the perspective of permeability, the pore structure is divided into permeable pores creating flow channels and impermeable pores (isolated pores that are not filled with water when the concrete is submerged (According to ASTM C125-15b (1))). The effective flow channel comprises 20-50% of the total porosity (5). The porosity decreases with curing age (9). Furthermore, the pore size decreases by decreasing w/c ratio, i.e. pore refining (10). The interfacial transition zone between the aggregates and the cement paste (ITZ) is more porous than the bulk paste; it has cracks resulting from shrinkage (11). Furthermore, the paste structure includes much larger air voids (macrospores) (10).

Table 1: Types of pores and voids in cementitious pastes (8).

Type	Position	Description	Size
Fine gel pores	Within the hydration layers (within gel)	Interlayer micropores	< 0.5 nm
		Micropores	0.5–3 nm
Large permeable capillary pores	Within hydration layers	Small capillaries	3 – 10 nm
	Between C-S-H crystals	Medium capillary	10 – 50 nm
		Large capillaries	50 nm – 0.5 μm

The pore structure and the level of saturation control the concrete density. Several types of concrete densities are defined: the skeleton density is the mass of the solid material divided by the volume of solid material only (without the volume of pores) (5). On the other hand, the apparent density is the mass of the solid material divided by the volume of solid material plus impermeable pores (1). ASTM C642 (12) defines 4 types of densities. These include (1) Bulk dry density which is the mass of the dry material divided by its total volume, (2) Bulk density after immersion which measures the density of a sample saturated with water (filling most of the pores), (3) Bulk density after immersion and boiling which accounts for all voids filled by water, including those in the permeable pores, by boiling the sample and (4) Apparent density specifically measures the density of the solids and the non-permeable voids.

Pore structure is usually corelated to various concrete properties such strength, permeability, absorptivity, diffusivity, shrinkage and durability (10). Water penetration into concrete structure is driven by pore structure. Porosity is a structural property while water permeability and absorptivity are intrinsic properties. Water penetrates concrete by permeation flow (under a hydrostatic pressure) and/or by capillary action (wicking) (under no or minor hydrostatic pressure) (8). The ACI Concrete Terminology (13) defines capillary action (suction) as "the movement of a liquid in the interstices of concrete, soil, or other finely porous material due to surface tension" (13). Furthermore, it defines the permeability as "the movement of water due to a pressure gradient, such as water in contact with a concrete structure installed underground". It is well established in the literature permeability is exponentially correlated to the porosity (5, 10, 14). In fact, pore refining reduces various transport properties (including permeability) and enhances durability in concrete (8, 15, 16), while it increases the capillary action (10).

Waterproofing through pore blocking and pore lining of concrete have different effects on water absorption and permeability (17). Consequently, the efficacy of waterproofing is evaluated based on measuring the water absorption by capillary action (18) and on measuring the permeability under a high hydrostatic pressure (19-23). Muhammad et al. (18) indicated in their survey that the water absorption is the most commonly used test in evaluating waterproofing. Traditional pore blocking/ crystalline waterproofing technologies fill the pores by precipitates or crystals from the reaction with hydration products (16, 24-27). On the other hand, pore lining waterproofing (or dampproofing) technologies create water-repelling molecular layer through the reaction with silicone sites at the cementitious internal walls. Then, they reduce water (capillary) absorption or wicking flow (19, 28, 29). According to ACI 212.3R-16 (19), hydrophobic waterproofing materials are not effective if there is a possibility of a hydrostatic pressure devolvement. Furthermore, creating flow obstacle through pore blocking/filling may not reduce capillary suction (30); but it can increase water absorption due to pore refining (24). Hence, reducing water absorption requires pore lining functionalities in addition to pore blocking. Combined pore blocking and pore lining crystalline waterproofing materials (16) are available in the industry involving triple functionalities of hydrophobicity with hygroscopic and hydrophilic crystallinity. These include Multi-Crystalline Waterproofing (MCE) Admixture (27) and Dual Crystalline Engineered (DCE) Sealer (26). They are two patented materials for internal waterproofing by pore-blocking (crystallinity) and pore lining (hydrophobicity) (31, 32). MCE is chlorine free and ammonia free, water-based Permeability Reducing Admixture (PRAH) (ACI 212.3R-10 (19)). Various aspects of waterproofing and durability performances of MCE and Pavix materials have published in previous papers (23, 33-38).

In a recent publication [under review], the efficiency of such a technology for reducing water permeability under hydrostatic pressure has been shown to be above 99%, reflecting large pore blocking. The main aim of this experimental submission is to study the effect of MCE on water absorption and porosity. Some researchers believe that the capillary absorption controls the long-term durability (8). The independent experimental variables are MCE dosage and the ratio of water to cementitious materials (w/cm). The dependent experimental variables are water absorption, volume fraction of permeable pores and various types of densities.

2. Experimental

Reference and crystalline-waterproofed concrete specimens were casted and investigated according to the procedures of ASTM C642. All specimens were made and tested by the independent laboratories of Construction Material Testing (CMT, Des Moines, Iowa, USA). The concrete mix design was according to Iowa Department of Transportation (Iowa DOT C-4 WRC20); commonly used in infrastructure applications such as pavements and bridge decks (39). The mix design was composed of total cementitious content of 593 lb, while 20% replacement of the cementitious content (119 lb) was replaced by Class C fly ash. The amounts of the constituents (per cubic yard of concrete) and their types and sources are listed in Table 2. The ratio of water to cementitious materials (w/cm) was varied between 0.37 to 0.54. The air entrainment admixture was GRT SA-50 and the water reducing admixture was GRT 400-NC (from Euclid Admixture, Cleveland, Ohio, USA). The target air content was 6%. The specimens, casted as cylinders 15.2x 30.4 cm (6x12 inches), were demolded after 24 hours, then cured for 28 days under normal laboratory conditions. The crystalline waterproofing admixture was MCE from International Chem-Crete Corporation (Dallas, Texas, USA). The total solid content in MCE is about 15% (32). The MCE was added to the mixing water according to the required dosage (1% or 2%), by mass of the cementitious material content.

Table 2: Iowa DOT C4-WRC20 mix proportions for one cubic yard of concrete.

Component	**Type/ Source**	**Amounts**	
		Addition	**Volume (ft^3)**
Cement	Continental (Type IL)	474 lb	2.44
Fly ash	Boral Resources (Class C)	119 lb	0.71
Coarse aggregates	D-57 Concrete Stone – A85006 (Martin Marietta – Ames)	1470 lb	8.99
Fine aggregates	Concrete Sand – A77534 (Martin Marietta – Saylorville)	1486 lb	8.99
Water	Local/ Municipal	266 lb	4.27
Air entrainment	GRT SA-50	6%	1.62
Water reducer (MRWR)	GRT 400 NC (Mid-Range)	4.0 lq oz	---
MCE admixture	International Chem-Crete Co.	1% or 2%	

Water absorption, densities and volume fraction of permeable pores were measured according to ASTM C642 (12). After curing for 28 days, the specimens were initially weighed and then dried in oven at 100°C for at least 24 hours. Then, after allowing the specimens to cool in a desiccator, the oven-dry mass (m_d) was measured. Such drying, weighing, cooling process was continued until the difference between successive values of m_d was <0.5%. Then, the specimens were immersed in water for 48 hours to achieve saturation, then, the saturated concrete mass (m_s) was measured. Then, the specimens were immersed and boiled in water for five hours, then allowed to cool for at least 14 hours to reach room temperature (20-25°C). Then, the saturated mass after boiling (m_b) was measured. Finally, the mass of the specimen suspended in water (m_w), after immersion and boiling, was measured. Then, experimental variables were determined according to the equations defined in the ASTM C642 (12).

3. Results and Discussion

The results of the effects of w/cm ration on the measured experimental variables for reference (control) concrete (absorption, fraction of permeable pores and density) are shown in Figures 1 and 2. Figure 1 shows that increasing the w/cm ration increases the water absorption after immersion as expected due to the increase in the porosity. A reasonably similar trend is observed for the water after absorption and boiling, however, a higher absorption curve is obtained; due to a larger amount of water that reaches more pores through saturation after boiling. For very low w/cm ratio (0.37), a slightly higher water absorption is obtained than those for w/cm 0.44 and 0.49. In a similar trend, Figure 1 also shows that increasing the w/cm ration increases the fraction of permeable pores, for w/cm $\geq$ 0.44. However, for very low w/cm ratio (0.37), a higher fraction of permeable pores is obtained than those for w/cm 0.44 and 0.49. The w/cm=0.37 is lower than the typical theoretical value (minimum 0.38) (2). The theoretical limit of w/cm is set to account for the required water needed for full cement hydration (about 23%) plus the amount of water physically adsorbed within the porous structure (about 15%) (3). In fact, such a limit does not account to the unavoidable water evaporation from heat release from cement hydration and hence the typical theoretical w/cm is set at about 0.40 (3, 40). Based on this analysis, the trends for reference concrete are well interpreted for w/cm ≥ 0.44 while the off-trend values at w/cm=0.37 are related to insufficient cement hydration, when growing below the theoretical minimum of w/cm; due to less water availability for reaction, and hence leads to a larger porosity. The focus of this work is for cases with w/cm > 0.40 (For MCE application).

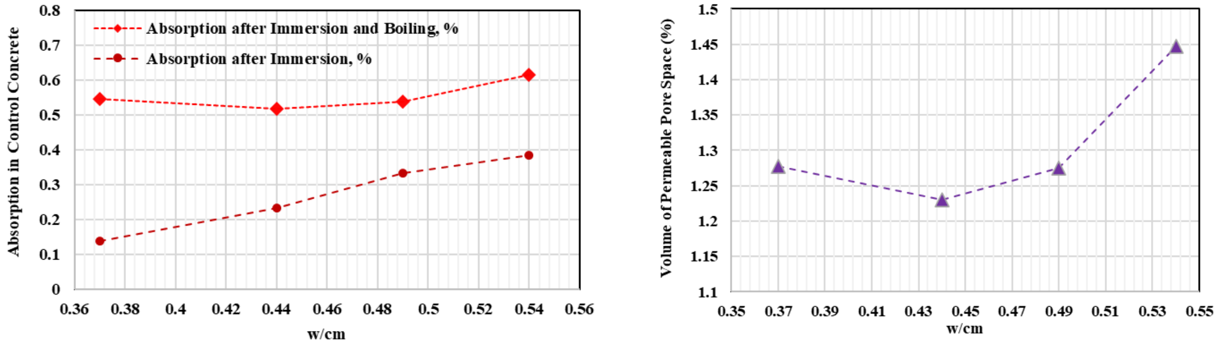

Fig. 1: The effect w/cm ratio on the water absorption (after immersion and after immersion and boiling) and on the volume fraction of permeable pores for reference concrete (Iowa DOT C4-WRC20).

Figure 2 shows the effect of w/cm ratio on the densities of reference concrete. In general, all types of densities have nearly the same trend. The density decreases with increasing w/cm for w/cm ≥ 0.44; due to the increase the porosity (see Figure 1). However, for very low w/cm ratio (0.37), lower densities are obtained than those for w/cm ≥ 0.44, due to insufficient hydration when going below the theoretical limit; as explained above. The bulk dry density has the lowest value among all other densities, since it includes empty voids in the concrete volume. The curve of bulk density after immersion is above that for the bulk dry density because the saturation of the pores with water increases the density. Furthermore, the values of the bulk density after immersion and boiling are higher than those for bulk density after immersion since a larger fraction of voids is saturated upon boiling. The apparent density has the highest value as it is very close to the absolute density: it is nearly the density of "net" solid, but does not exactly have the same since it includes the gel/ isolated pores within the concrete volume.

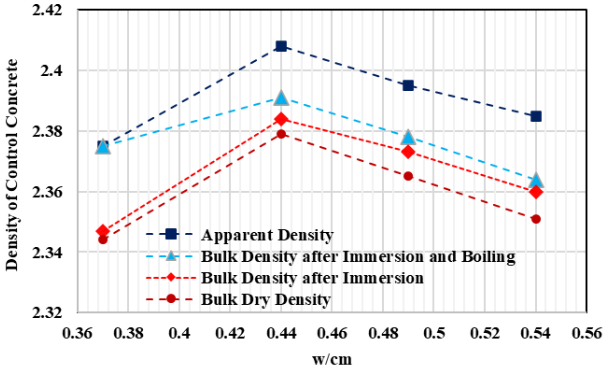

Fig. 2: The effect w/cm ratio on reference concrete densities.

3.1 Effect of MCE Admixture on Concrete Absorptivity

Figure 3 shows the effect of adding MCE admixture to concrete mix design (for the dosages of 1% and 2%) on the water absorption at various w/cm ratios. The addition of MCE (at any of the two investigated dosages) deceases the absorption after immersion and the absorption after immersion and boiling for w/cm ≥ 0.44. Such significant reductions in absorptivity are achieved by the crystallinity of the MCE (pore blocking) and its hydrophobicity (pore lining). The reductions for the 2%

MCE dosage are in the range of 47-62%. The 1% MCE dosage achieves lower levels of reductions than the 2% MCE, as expected due to lower yield of crystallization and pore filling than that for the 2% MCE. For very low w/cm ratio (0.37), (which is lower than the minimum theoretical w/cm), an inconstant trend is observed as explained above for the reference concrete. It has been reported that the crystalline admixtures become insignificant for decreasing the water absorption at low w/cm ratio (41). MCE is usually applied in cases with w/cm ≥ 0.40.

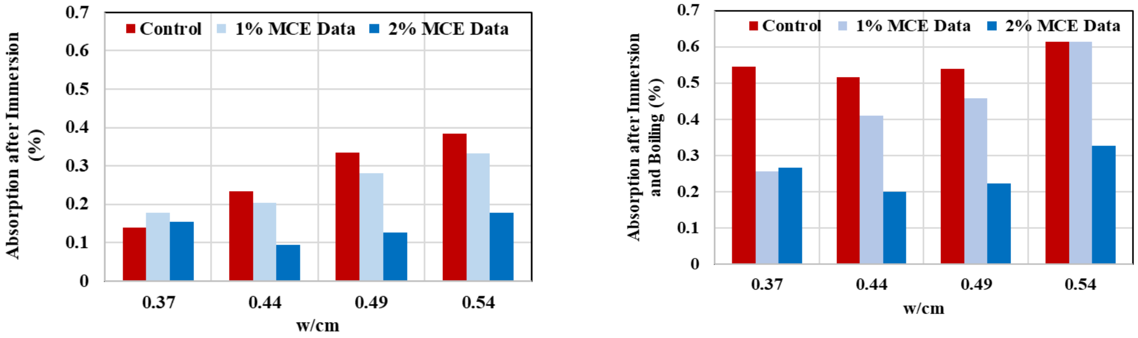

Fig. 3: The effect of adding MCE admixture to concrete mix design (for 1% and 2% MCE) on the absorption at various w/cm ratios.

3.2 Effect of MCE Admixture on Concrete Porosity

Figure 4 shows the effect of adding MCE admixture (for the dosages of 1% and 2%) on the fraction of permeable pores of concrete at various w/cm ratios. Obviously, adding MCE reduces the fraction of permeable pores (at each of the two investigated dosages). This is achieved from MCE pore filling functionality resulting from the formation of the crystals within the pores. The dosage of 2% MCE leads to reductions in the porosity in the range of 46-61%. Obviously, the 2% MCE dosage leads to larger reductions in porosity that the 1% MCE; this is due to larger yield of crystal formations, as expected.

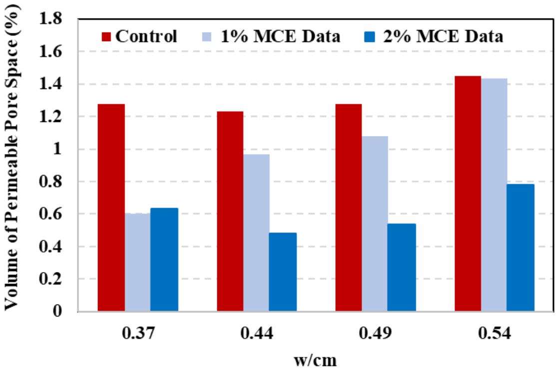

Fig. 4: The effect of adding MCE admixture to concrete mix design (for the dosages of 1% and 2%) on the fraction of permeable pores at various w/cm ratios.

3.3 Effect of MCE Admixture on Concrete Density

Figure 5 shows the effect of adding MCE admixture (at 2% dosage) on the bulk dry density and apparent density at various w/cm ratios. They are shown as two sample curves: nearly similar trends are obtained for the other two types of densities with 2% MCE dosage. Table 3 lists all density results for the two dosages and for the control specimens. Figure 5 shows that the 2% MCE has a slight densifying effect due to the formation of the crystals within the pores and from the enhanced cement hydration. In fact, MCE is not labelled as a typical densifying concrete additive, thus, the densifying effect of 2% MCE is only around 1% only. This is because the fractional volume of pores in the reference concrete is relatively very low (in the range of 1.23-1.45%), and the pore filling effect is a partial filling of such a percentage. For the 1% MCE dosage, no noticeable densifying effect could be measured. In fact, a small reduction in the density (around to 0.5%) is observed; the yield of crystals with 1% MCE are lower than those with 2% MCE.

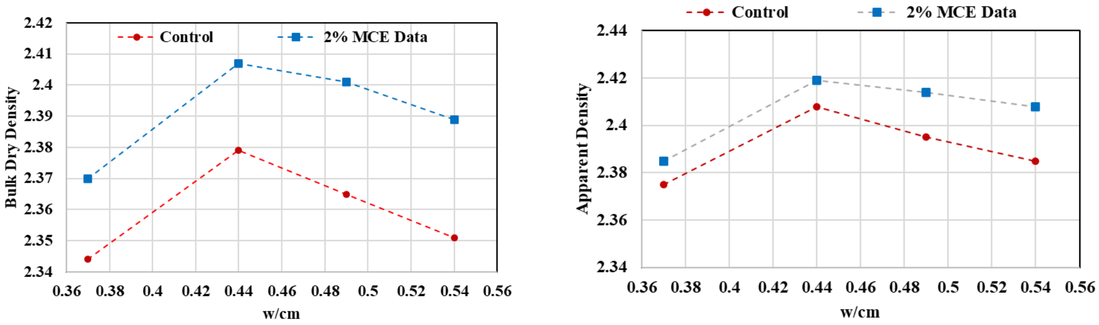

Fig. 5: The effect of adding MCE admixture to concrete mix design (at 2% dosage) on the bulk dry density and apparent density at various w/cm ratios.

Table 3: Results of densities for reference concrete and for 1% MCE and 2% MCE dosages.

Parameter	**Control**				**1% MCE Data**				**2% MCE Data**			
w/cm	**0.37**	**0.44**	**0.49**	**0.54**	**0.37**	**0.44**	**0.49**	**0.54**	**0.37**	**0.44**	**0.49**	**0.54**
Bulk Dry Density	2.344	2.379	2.365	2.351	2.374	2.369	2.35	2.335	2.37	2.407	2.401	2.389
Bulk Density after Immersion	2.347	2.384	2.373	2.36	2.378	2.374	2.357	2.343	2.373	2.409	2.404	2.393
Bulk Density after Immersion and Boiling	2.375	2.391	2.378	2.364	2.38	2.379	2.361	2.349	2.376	2.412	2.406	2.397
Apparent Density	2.375	2.408	2.395	2.385	2.388	2.392	2.376	2.369	2.385	2.419	2.414	2.408

4. Conclusion

The experimental program for testing MCE admixture shows that MCE reduces the concrete porosity and the water absorptivity. Such a pore blocking waterproofing performance is confirmed experimental for the two dosages of 1% and 2%. A slight densifying effect occurs with the 2% MCE dosage. The performance of MCE in terms of water absorption and fraction of permeable pores is dependent on its dosage and the w/cm ratio. At high w/cm ratio, the 2% MCE dosage is recommended; as it yields a reduction in porosity in the range of 46-58% for all w/cm ratios. The 2% MCE reduces water absorption by a percentage within a range of 47-62% for w/cm ≥ 0.44. The 1% MCE dosage shows a reduction in the water absorption after immersion by 13-16% for w/cm ≥ 0.44. Based on these findings, MCE provides a significant waterproofing performance that enhances durability of concrete and hence extends its service life.

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