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Porosity, Water Absorption and Abrasion Resistance of Concrete Dosed with Multi-Crystalline (MCE) Waterproofing Admixture: Comparing Two MCE dosages with Control Concrete

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***Abstract** - Concrete durability issues are associated with water penetration and concrete porosity in addition to its strength. This paper 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 compared to control concrete. 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. Furthermore, the standard testing method described in ASTM C779 was used to measure abrasion resistance for reference and MCE-dosed concrete specimens. These experiments were performed 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, density and abrasion resistance 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 and a significant increase in the abrasion resistance. MCE functions very well with 1% dosage, however, additional performance is obtained with 2% dosage. At high w/cm ratio, 2% MCE is recommended.*

Keywords: Concrete, Moisture, Pores, Absorption, Waterproofing, Crystallization, Durability, ASTM C642.

1. Introduction

The durability of concrete is governed by its structure and its mechanical and transport properties [1]. Water penetration controls the intrusion of various harmful chemical materials that causes various durability issues. The study of porosity and water absorption as well as concrete hardness has a major reflection on evaluating the potential durability of concrete.

The structure of concrete, as a porous composite material, consists of mineral aggregates bonded together by hydrated cementitious paste [2, 3]. The theoretical required amount of water needed for full cement hydration, is about 23% of water by weight of cement [4]. However, water physically adsorbed within the porous structure of the produced hydration products, demanding about 15% of cement weight [4]. Consequently, a minimum of water-to-cement (w/c) ratio of 0.38 is essential for the complete cement hydration reactions and saturation of the hydration products [4]. Furthermore, to account for unavoidable water evaporation due the exothermic cement hydration, a w/c=0.40 is usually needed for complete cement hydration and saturation [4, 5]. When, the w/c ratio is less than such a minimum, the hydration yield is expected to be small. Thus, investigating concrete with w/c ratio below 0.38 (or below 0.4), should be considered as a special case with a different trend that

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typical w/c ratios. Typically, concrete made of normal Portland cement is more porous than concrete modified with the addition of supplementary cementitious materials (SCMs) such as fly ash. The addition of fly ash to the mix design decreases the porosity and refines the pore structure [6]. Class C fly ash leads to more compact and denser structure with lower total porosity than control specimens [7]; as it consumes part of the cement hydration by-product, $Ca(OH)_2$ (portlandite) through hydration.

The structure of the paste involves a network of hydration solid products and voids involving bound and free water [4, 8]. The structure of pores and voids is complex ranging from less than 1 nm to 1 μm, with six orders of magnitude variations [9]. 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 [10, 11]. The pores, involve various types and sizes as listed in Table 1 [12]. 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.

Table 1. Types of pores and voids in cementitious pastes [12].

Type	Position	Description	Size
Fine gel pores	Within hydration layers	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

Water penetration into concrete structure is usually achieved by permeation and wicking flow. Permeation is driven by hydrostatic pressure to which concrete is subjected; while wicking flow is driven by capillary suction, which is controlled by surface tension and the intermolecular forces between water molecules and the concrete pores [13].

Hereafter, 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 [2])). The effective flow channel comprises 20-50% of the total porosity [9]. The porosity decreases with curing age [14]. Furthermore, the pore size decreases by decreasing w/c ratio, i.e. pore refining [6]. 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 [15]. Furthermore, the paste structure includes much larger air voids (macrospores) [6]. These include (1) air entrained spherical voids that are intentionally created by adding admixtures (for controlling freezing and thawing) (10 μm -1 mm [2]) and (2) air entrapped irregular voids that are unintentionally created due to mixing and handling of fresh mixtures (larger than 1 mm) [9]. The impact of air voids on water saturation is not totally settled in the literature: There is a believe that their impact is insignificant, while air voids can get saturated with water (at a slower rate than the capillary pores) and they can be interconnected through the network of the capillary pores [8, 10].

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) [9]. On the other hand, the apparent density is the mass of the solid material divided by the volume of solid material plus impermeable pores [2]. ASTM C642 [16] 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 correlated to various concrete properties such strength, permeability, absorptivity, diffusivity, shrinkage and durability [6]. Furthermore, the integrity of concrete structure controls its surface hardness. Abrasion resistance (measured according to ASTM C779) is a crucial parameter in waterproofed concrete subjected to eroding surroundings such as traffic, moving machines and water hydrodynamic (e.g. pavements and bridge decks) [17]. For enhancing abrasion resistance, surface hardeners such as silicate solutions are typically used in responding to the wear effects [18].

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) [12]. The ACI Concrete Terminology [19] 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" [19]. 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 [6, 9, 20]. In fact, pore refining reduces various transport properties (including permeability) and enhances durability in concrete [1, 12, 17], while it increases the capillary action [6].

Waterproofing through pore blocking and pore lining of concrete have different effects on water absorption and permeability [21]. Consequently, the efficacy of waterproofing is evaluated based on measuring the water absorption by capillary action [22] and on measuring the permeability under a high hydrostatic pressure [23-27]. Muhammad et al. [22] 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 [17, 18, 28-30]. 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 [23, 31, 32]. According to ACI 212.3R-16 [23], 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 [33]; but it can increase water absorption due to pore refining [18]. Hence, reducing water absorption requires pore lining functionalities in addition to pore blocking. Combined pore blocking and pore lining crystalline waterproofing materials [17] are available in the industry involving triple functionalities of hydrophobicity with hygroscopic and hydrophilic crystallinity. These include Multi-Crystalline Waterproofing (MCE) Admixture [30] and Dual Crystalline Engineered (DCE) Sealer [29]. They are two patented materials for internal waterproofing by pore-blocking (crystallinity) and pore lining (hydrophobicity) [34, 35]. MCE is chlorine free and ammonia free, water-based Permeability Reducing Admixture (PRAH) (ACI 212.3R-10 [23]). Various aspects of waterproofing and durability performances of MCE and Pavix materials have published in previous papers [27, 36-41].

In a recent publication [42], the efficiency of such a technology for reducing water permeability under a hydrostatic pressure has been shown to be above 99%, due to significant pore blocking. The main aim of this experimental paper is to study the effect of MCE on water absorption, porosity and to relate such a performance with abrasion resistance. Limited experimental data on these parameters were published in a previous paper [37]. The previous data did not offer a comparative evaluation of MCE-dosed concrete with reference concrete. There was no estimation of relative performance of MCE-dosed concrete compared to control concrete. This paper is an expansion of such previous work; it provides a complete comparative analysis at various water to cement ratios. 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, various types of densities and abrasion resistance.

2. Experimental

Reference and crystalline-waterproofed concrete specimens were cast 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 [43].

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 ratio of water to cementitious materials (w/cm) was varied between 0.37 to 0.54. For tests with w/cm=0.37 (which is below the theoretical minimum value), they are considered in this work as special cases. 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, cast 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% [35]. The MCE was added to the mixing water according to the required dosage (1% or 2%), by mass of the cementitious material content. The amounts of the constituents (per cubic yard of concrete) and their types and sources are listed in Table 2.

Table 2: Iowa DOT C4-WRC20 mix proportions for one cubic yard of concrete [43].

Component	British Units	SI units
Portland Cement (Type I)	491 lb	222.9 kg
Coarse aggregates	1490 lb	676.5 kg
Fine aggregates	1479 lb	671.5 kg
Fly ash	123 lb	55.8 kg
Air entrainment	6%	6%
Water reducer	4.0 lq oz	118 ml
w/c range	0.37-0.54	0.37-0.54

Abrasion resistance of fully cured reference and MCE-dosed concrete was measured according to testing method described in ASTM C779. The obtained data are presented as plots of wear depth as function of elapsed time for the investigated w/cm ratios. Water absorption, densities and volume fraction of permeable pores were measured according to ASTM C642 [16]. 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 [16]. Then, the ASTM C642 experimental variables were determined according to the following equations:

Absorption after immersion (%)

$$= [(m_s - m_d)/m_d] \times 100\% \quad (1)$$

Absorption after immersion and boiling (%)

$$= [(m_b - m_d)/m_d] \times 100\% \quad (2)$$

Bulk density (ρ_{bd}), dry (g/cm^3)

$$= [m_d/(m_b - m_w)] \times \rho \quad (3)$$

Bulk density after immersion (ρ_{bi}), (g/cm^3)

$$= [m_s/(m_b - m_w)] \times \rho \quad (4)$$

Bulk density after immersion and boiling (ρ_{bib}), (g/cm^3)

$$= [m_b/(m_b - m_w)] \times \rho \quad (5)$$

Apparent density (ρ_a), (g/cm^3)

$$= [m_d/(m_d - m_w)] \times \rho \quad (6)$$

Volume of permeable voids (%)

$$= [((\rho_a - \rho_{bd})/ r_a] \times 100 \quad (7)$$

Also, volume of permeable voids (%)

$$= [(m_b - m_d)/(m_b - m_w)] \quad (7a)$$

Where ρ is the density of water at 4 ^{o}C (1 g/cm^3).

3. Results and Discussion

The results of the effects of w/cm ration on the measured experimental dependent variables for reference (control) concrete 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 which is a special case), 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≥ 0.44. However, for the special case of 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) [3]. 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%) [4]. 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 [4, 5]. 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).

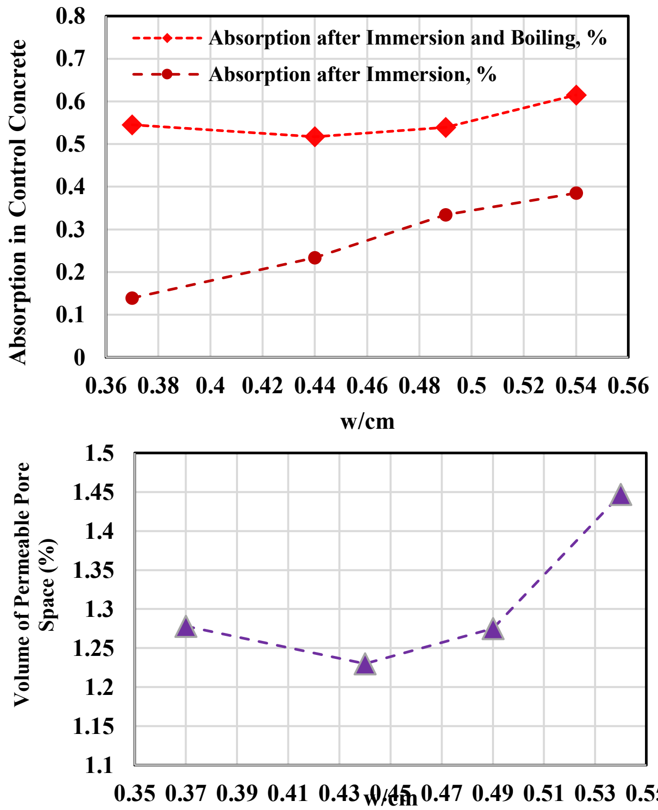

Figure 1: The effect w/cm ratio on the water absorption (after immersion and after immersion and boiling) and on the volume fraction of permeable pores for reference concrete (Iowa DOT C4-WRC20).

Figure 2 shows the effect of w/cm ratio on the densities and the abrasion in terms of depth of wear 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) (the special case), 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. The abrasion curves show the expected (hypothesized) trend: the depth of wear increases with elapsed time. Furthermore, increasing the w/cm ratio increases the level of wear as a result of decreasing concrete integrity and decreasing its strength.

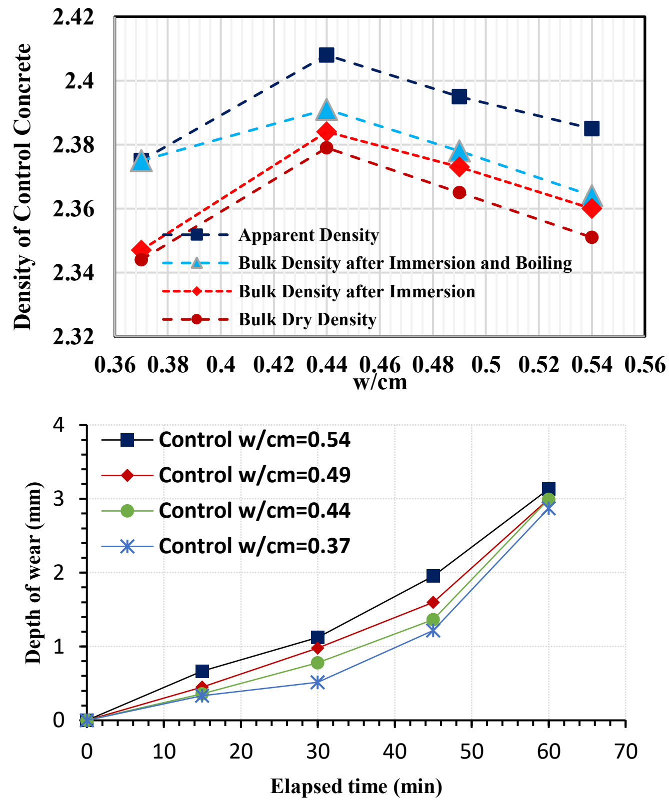

Figure 2: The effect w/cm ratio on densities and abrasion resistance (in terms of depth of wear) for reference concrete (Iowa DOT C4-WRC20).

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) decreases 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) (the special case, 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 [44]. MCE is usually applied in cases with w/cm ≥ 0.40.

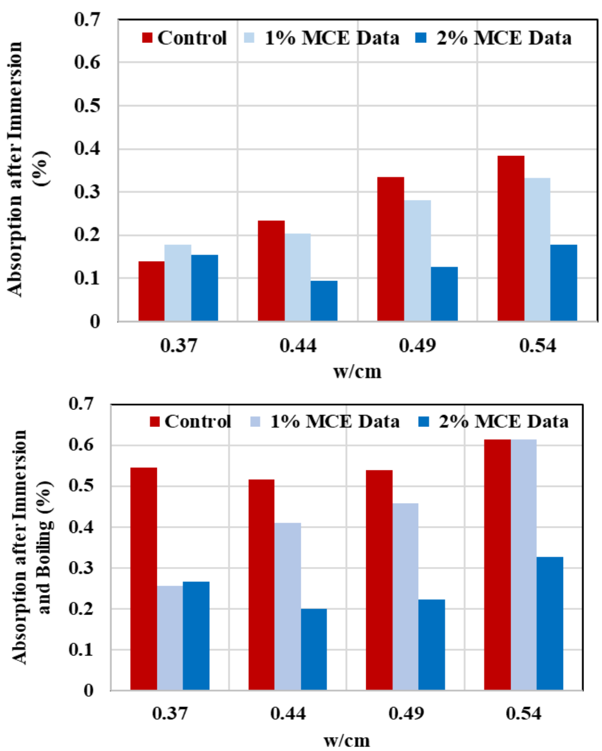

Figure 3: The effect of adding MCE admixture to concrete mix design (for 1% and 2% MCE) on the absorption at various w/cm ratios.

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.

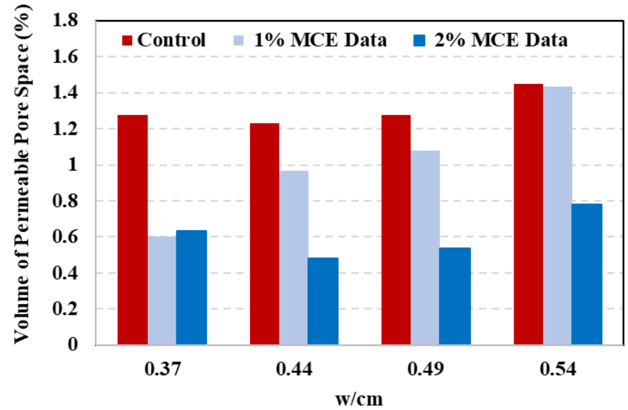

Figure 4: The effect of adding MCE admixture to concrete mix design (for the dosages of 1% and 2%) on the fraction of permeable pores at various w/cm ratios.

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. 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. Table 3 lists the density increase for the 2% dosage relative to that of the control concrete. In fact, MCE is not labelled as a typical densifying additive, thus, the densifying effect of 2% MCE is around 1% for most of the cases. 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 with 1% MCE; the yield of crystals with 1% MCE are lower than those with 2% MCE.

Table 3: Results of densities enhancement for 2% MCE dosage over the control concrete

Parameter	2% MCE Relative Performance (%) over the control concrete			
w/cm	0.37	0.44	0.49	0.54
Bulk Dry Density	1.1%	1.2%	1.5%	1.6%
Bulk Density after Immersion	1.1%	1.0%	1.3%	1.4%

Bulk Density after Immersion and Boiling	0.0%	0.9%	1.2%	1.4%
Apparent Density	0.4%	0.5%	0.8%	1.0%

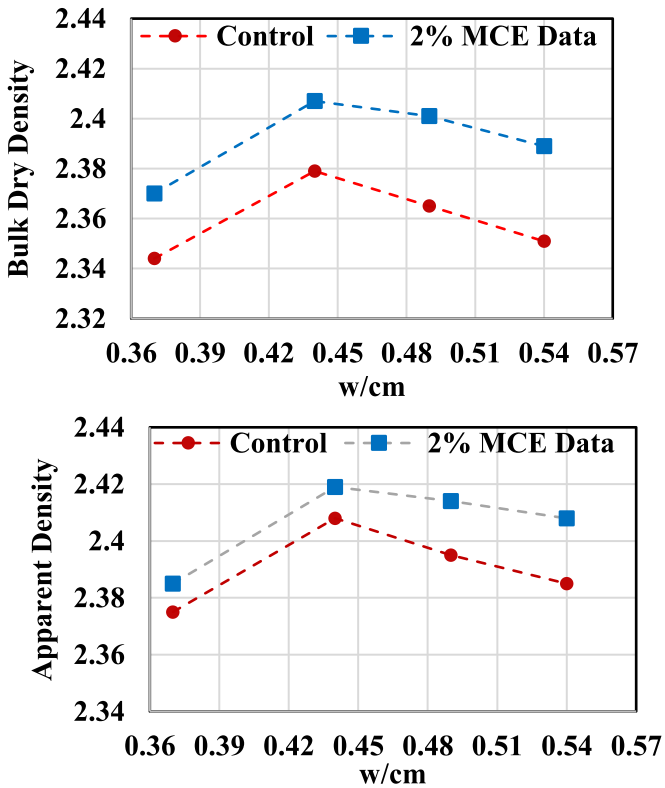

Figure 5: The effect of adding MCE admixture to concrete mix design (at 2% dosage) on the bulk dry density and apparent density at various w/cm ratios.

3.4 Effect of MCE Admixture on Abrasion Resistance of Concrete

Figure 6 compares the abrasion curves for control, 1%-MCE and 2%-MCE concrete for the lowest and the highest w/cm ratios investigated in this work. These trends for MCE-dosed concrete are similar to those for the control concrete (See Figure 2). However, it is obvious that the addition of MCE to concrete increases its abrasion resistance (by lowering the depth of wear) regardless of the dosage. ASTM C779 classifies failure when the depth of wear (at 60 seconds) reaches 3 mm (or greater). In this case, the reference concrete is failure while the 1% and 2% MCE conform to the standard and pass. Such an enhancement in the abrasion resistance provides evidence of the capability of MCE for enhancing mechanical strength. Such strength improvement is attributed to the increase in concrete integrity due to better cement hydration and due to the pore blocking mechanism of the crystallization products. This behavior is in agreement with that reported in a previous paper [40] for a similar crystallization system (called DCE), which is used as a surface treatment for concrete.

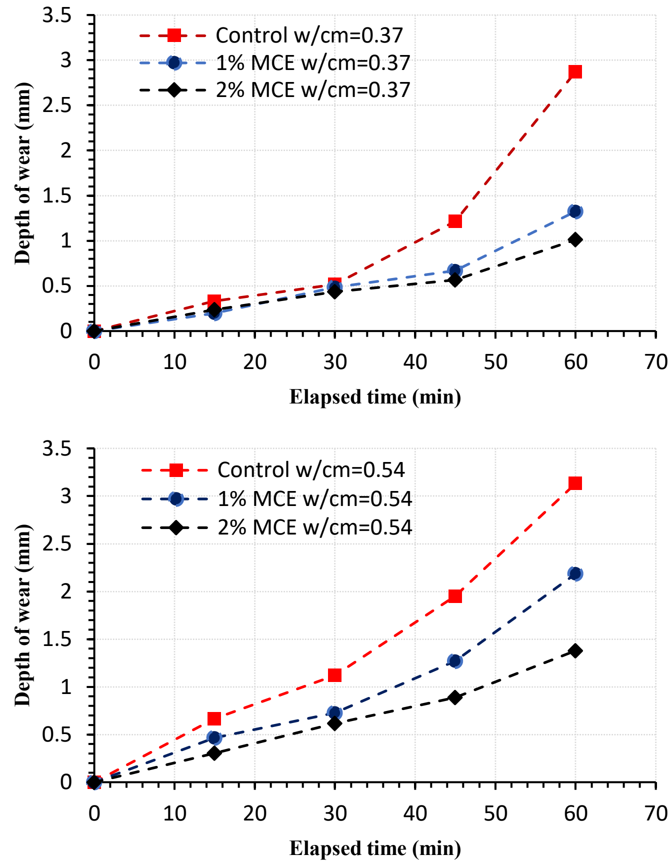

Figure 6: The abrasion curves for control, 1%-MCE and 2%-MCE concrete for w/cm=0.37 (top) and w/cm=0.54 (bottom).

Table 4 compares the percentage enhancement of the abrasion resistance for the 2% MCE dosage at various w/cm ratios. It is clear that at all the elapsed time, the addition of 2%-MCE increases the abrasion resistance for all investigated w/cm ratios. At the standard time of 60 seconds, the enhancement of the 2%-MCE is in the range of 56-65%. Larger percentage enhancements are obtained at lower w/cm ratios.

Similar trends are obtained for the 1%-MCE, but with slightly lower range. Table 5 shows these results. At the standard time of 60 seconds, the enhancement of the 1%-MCE is in the range of 30-54%. As in the case of 2%MCE, larger percentage enhancements are obtained at lower w/cm ratios. The 2%-MCE has an additional performance over 1%-MCE due to larger yield of hygroscopic crystallization and better integrity with 2% MCE compared to 1%. Such an additional benefit becomes more obvious at the high w/cm ratio than that at the low w/cm ratio, due to more availability of pores to occupy with crystals.

Table 4. The percentage enhancement of the abrasion resistance for the investigated w/cm ratios.

Elapsed Time, min	2% MCE w/cm= 0.37	2% MCE w/cm= 0.44	2% MCE w/cm= 0.49	2% MCE w/cm= 0.54
15	28.5%	28.7%	34.1%	54.4%
30	15.7%	36.0%	42.6%	45.1%
45	53.3%	44.5%	48.7%	54.5%
60	64.8%	62.7%	58.1%	56.0%

Table 5. The percentage enhancement of the abrasion resistance for the 1% MCE dosage at various w/cm ratios.

Elapsed Time, min	1% MCE w/cm= 0.37	1% MCE w/cm= 0.44	1% MCE w/cm= 0.49	1% MCE w/cm= 0.54
15	41.1%	33.1%	30.4%	30.2%
30	6.0%	27.9%	37.0%	35.1%
45	44.9%	27.7%	30.6%	35.0%
60	53.9%	48.0%	40.5%	30.2%

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. The performance and validity of both of the investigated dosages (1% and 2%) are confirmed experimentally. 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. For typical w/cm (≥ 0.44), the MCE reduces water absorption by a percentage within a range of 47-62% for the 2% dosage and by 13-16% for the 1% dosage. Furthermore, MCE strengthens the concrete with a significant increase in the abrasion resistance for the two dosages of 1% and 2%. For the typical dosage of 2% MCE, 56-65% reduction in wear depth can be obtained at 60 seconds of wear. Based on these findings, MCE provides a significant waterproofing performance and strength that enhances durability of concrete and hence extends its service life.

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