Evaluating Full and Partial Internal Curing in Terms of Internal Relative Humidity, Hydration and Performance

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Abstract

Internal curing is being increasingly considered for use in concrete pavements. Internal curing frequently uses prewetted lightweight aggregate (LWA) to provide water to the cement paste as it hydrates. While internal curing has many benefits, many of these benefits can be traced to the additional hydration or a reduction in autogenous shrinkage that occurs as a result of the water that is stored in the aggregate. Many times internally cured mixtures are designed to provide a volume of internal curing water that is equivalent to the volume of chemical shrinkage; some have asked if there are benefits to only supplying a portion of this water. If only a portion of this water is needed it would reduce the volume of LWA used and make the pavement more cost-efficient. This paper examines the additional hydration that occurs in internally cured concrete systems with varying levels of internal curing (i.e., varying volumes of water that are equal to chemical shrinkage as well as partial volumes of this water) at early ages using isothermal calorimetry. In addition, internal relative humidity and the densification of the ITZ are measured for the plain and internally cured concrete. Internal curing is beneficial as the additional hydration can improve strength and reduce fluid transport. Internal curing, relative humidity, cement hydration, degree of hydration, calorimetry, SEM, Scanning Electrical Microscopy

Introduction

Relative humidity and cement reaction. In a saturated cement paste the hydration of cement will continue until all the cement has reacted or there is no available space for hydration products to form (Powers 1948, Jensen and Hansen 2001a). In systems that are not fully saturated with water, the hydration reaction may be slowed or inhibited by the lack of moisture (Powers and Brownyard 1947, Snyder and Bentz 2004). Several researchers have evaluated the effect of the degree of saturation (i.e., moisture content or relative humidity) on cement hydration (Powers 1948, Carrier and Cady 1970, Spears 1983, Ho et al. 1988, Snyder and Bentz 2004). Powers (1948) landmark research is often quoted as a general relationship between relative humidity and the extent of cement hydration that can occur. Powers stored un-hydrated cement powder at vapor pressures of 0.1, 0.2, 0.3, 0.4, 0.5, 0.7, 0.8, 0.89 and 1.00 (similar to storing samples at different relative humidities). The non-evaporable water of the samples stored at each vapor pressure was determined after six months and used as a measure of reaction. The results from Powers study showed that cement hydration is influenced by the relative humidity in which the samples are stored (Figure 1). The cement reaction is almost nonexistent at

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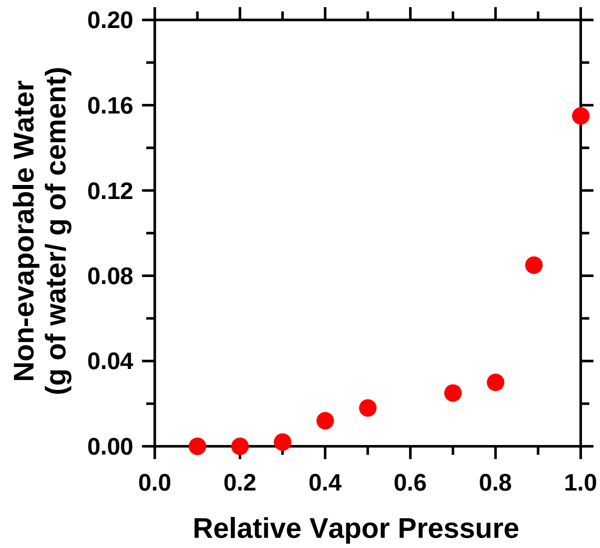
relative humidity lower than 30%. The reaction increases slightly as the relative humidity increases in the range of 30% to 80%; however the reaction still occurs at a very low rate. In the high relative humidity range (80% to 100%) the rate of cement reaction increases.

Figure 1. Amount of water taken up by dry cement exposed to water vapor for six months (after Powers 1948)



Internal curing. Internal curing is being increasingly used as a method to improve the performance of concrete (RILEM 2007, ACI 2012, ASCE 2012, Barrett et al. 2015). In North America internally cured concrete is typically made by replacing a portion of the volume of fine aggregate with prewetted lightweight aggregate (LWA). This prewetted LWA releases water to the paste after set which helps to reduce autogenous shrinkage (Bentz and Snyder 1999, Jensen and Hansen 2001a-b, Cusson and Hoogeveen 2008, Henkensiefken et al. 2009c, Lopez et al. 2009), reduce restrained shrinkage cracking (Radlinskla et al. 2008; Henkensiefken et al. 2009a), increase strength (Bentz 2007), reduce fluid transport (Bentz and Snyder 1999, Henkensiefken et al. 2009b, Peled et al. 2010, Castro et al. 2012a, Golias et al. 2012), and improve robustness to thermal cracking during construction (Schlitter et al. 2010, Raoufi et al. 2011, Byard et al. 2010, Barrett et al. 2015). The majority of benefits have been reported in low w/c mixtures; however the use of internal curing in more moderate w/c systems (0.39 – 0.45) had shown benefits also (Castro et al. 2012a, Espinoza-Hijazin and Lopez 2011, Barrett et al. 2014). It has been hypothesized that internal curing supplies additional water that can increase hydration of the cement and densify the microstructure at the surface of the lightweight aggregate (Henkensiefken 2009b, Lura et al. 2010, Peled et al. 2013).

Research Objective. The objective of this research was to quantify the effect of the addition of pre-wetted lightweight aggregate on the relative humidity of mortars and its effect on the degree of hydration of cement and densification of the interfacial transition zone. This paper will examine the potential benefits of internal curing in mortar systems with *w/*c of 0.30, 0.33, 0.36, 0.39, 0.42 and 0.45. Further, the paper will evaluate the benefits of providing a volume of water that is equivalent to the chemical shrinkage (a full replacement level) as well as partial replacement levels.

Materials. An ASTM C150 Type I ordinary portland cement (OPC) was used in this study, with a Blaine fineness of 370 m^2/kg and an estimated Bogue composition of 56 % C_3S, 16 % C_2S, 12 % C_3A, 7 % C_4AF and a Na_2O equivalent of 0.68 % by mass. The sand used was natural river sand with a fineness modulus of 2.71, an apparent specific gravity of 2.58, and water absorption of 1.0 % by mass. Portions of the normal weight sand were replaced with an expanded shale (e.g., the producer of the lightweight aggregate was Buildex Marquette) with a fineness modulus of 3.10 and a specific gravity of 1.45. The 24 hour absorption of the fine lightweight aggregate was determined to be 17.5 % and 18.8% according to the paper towel (NYDOT 2008, Castro et al. 2011) and centrifuge respectively (Miller et al. 2014).

Twenty one different mixtures were prepared. The mortars had a single volume fraction of fine aggregate (55% of the total volume) and six different *w/c*'s (0.30, 0.33, 0.36, 0.39, 0.42, and 0.45). For each system fine lightweight aggregate (LWA) was used as a replacement of normal weight aggregate (NWA) sand at three different levels: 0 %, 50 % and 100 % of the volume of LWA required to supply the water to fill the porosity created by chemical shrinkage, as determined using the Equation 1 (Bentz et al. 2005). Three additional mortars were prepared: a plain mortar with a *w/c* of 0.48, a plain mortar with a *w/c* of 0.51 and an internally cured mortar (following Eq 1) with a *w/c* of 0.27. A list of the mixture proportions can be found in Table 1. The replacement is on a total volume basis, e.g., the volume of aggregate (LWA and NWA) remained constant at 55 % of the total volume.

$$M_{LWA} = \frac{C_f \times CS \times \alpha_{\max}}{S \times \phi_{LWA}} \qquad \textbf{Eq. 1}$$

where M_{LWA} (kg/m^3) is the mass of LWA (in a dry state) that needs to be water filled to provide water to fill in the voids created by chemical shrinkage, C_f (kg/m^3) is the cement content of the mixture, CS (ml of water per g of cement) is the chemical shrinkage of the cement, α_{max} (unitless) is the expected maximum degree of hydration (0 to 1), S (unitless) is the expected degree of saturation of the LWA (here as 1 at 24 hr), and Φ_{LWA} (kg of water/kg of dry LWA) is the absorption capacity of the LWA (here the 24 h absorption).

Mixing was performed in accordance with the ASTM C305 procedure (ASTM 2006). The LWA was oven dried, air cooled, and then submerged in water for 24 ± 1 h prior to mixing. The volume of water used to submerge the LWA included both mixing water and the water the LWA would absorb in 24 h. The excess water (water not absorbed in 24 h) was then decanted and used as the mixing water. The normal weight sand was oven dried and cooled for 24 h before mixing. For the plain mortar mixtures and the pastes, the water and cement were conditioned for 24 h at room temperature.

Experimental method procedures

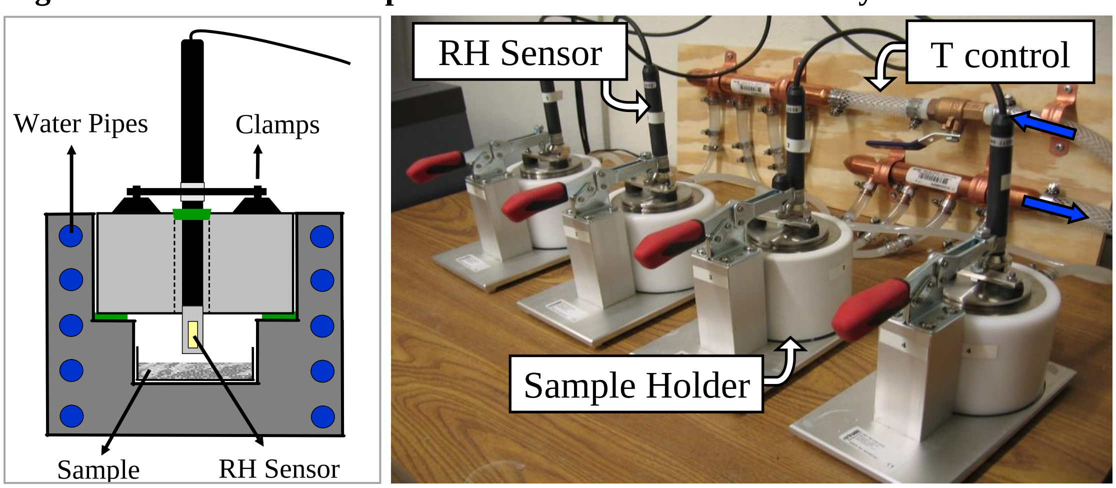
Relative Humidity of the Mortars. Relative humidity measurements were performed on mortar samples using HygrClip2S sensors (± 0.8% RH and ± 0.1 °C at 23 °C) which were connected with a HygroFlex5 transmitter. Samples were cast into cylindrical containers (50 mm height and 22 mm diameter) where they were kept until they reached an age of 10 h. After casting, the samples were demolded and crushed and approximately

10 g of sample was placed in 12 mm deep cups with a 43 mm diameter. The probes were mounted in a 75 mm x 68 mm stainless steel cylinder that was placed over a water jacketed sample cup holder. The water jacket was connected with a programmable water bath. For this study the samples were maintained at a constant temperature (23.0 ± 0.1 °C). A picture of the setup can be seen in Figure 2. After conditioning the signal, the data was transmitted to a computer where it was recorded at 5 minute intervals.

Table 1. Mixture Proportion of Mixtures used in this Research

Mixture Designation	w/c	Cement (kg/m^3)	Water (kg/m^3)	NWA, SSD (kg/m^3)	LWA, Dry (kg/m^3)	Water in LWA (kg/m^3)	Water Reducer (g/ 100 g cem.)
Plain Mortar 0.30	0.30	727.0	218.1	1429.7	-	-	0.6
Plain Mortar 0.33	0.33	693.3	228.8	1429.7	-	-	0.4
Plain Mortar 0.36	0.36	662.6	238.6	1429.7	-	-	0.2
Plain Mortar 0.39	0.39	634.5	247.5	1429.7	-	-	-
Plain Mortar 0.42	0.42	608.7	255.7	1429.7	-	-	-
Plain Mortar 0.45	0.45	584.9	263.2	1429.7	-	-	-
Plain Mortar 0.48	0.48	562.9	270.2	1429.7	-	-	-
Plain Mortar 0.51	0.51	542.5	276.7	1429.7	-	-	-
LWA Mortar 0.30, 50%	0.30	727.0	218.1	1230.6	110.8	19.4	0.6
LWA Mortar 0.33, 50%	0.33	693.3	228.8	1220.9	116.2	20.4	0.4
LWA Mortar 0.36, 50%	0.36	662.6	238.6	1212.0	121.2	21.2	0.2
LWA Mortar 0.39, 50%	0.39	634.5	247.5	1221.2	116.1	20.3	-
LWA Mortar 0.42, 50%	0.42	608.7	255.7	1229.7	111.3	19.5	-
LWA Mortar 0.45, 50%	0.45	584.9	263.2	1237.5	107.0	18.7	-
LWA Mortar 0.27, 100%	0.27	764.2	206.3	1053.1	209.6	36.7	0.8
LWA Mortar 0.30, 100%	0.30	727.0	218.1	1031.5	221.6	38.8	0.6
LWA Mortar 0.33, 100%	0.33	693.3	228.8	1012.0	232.4	40.7	0.4
LWA Mortar 0.36, 100%	0.36	662.6	238.6	994.2	242.3	42.4	0.2
LWA Mortar 0.39, 100%	0.39	634.5	247.5	1012.7	232.1	40.6	-
LWA Mortar 0.42, 100%	0.42	608.7	255.7	1029.7	222.6	39.0	-
LWA Mortar 0.45, 100%	0.45	584.9	263.2	1045.3	213.9	37.4	-

Figure 2. Picture of the setup used to measure relative humidity on crushed mortars



Each sensor was calibrated using reference salts with well know relative humidity of equilibrium (potassium sulfate: 97.42 ± 0.48 % RH at 23°C; potassium chloride: 84.65 ± 0.27 % RH at 23°C; sodium chloride: 75.36 ± 0.13 % RH at 23°C; and sodium bromide: 58.20 ± 0.42% RH at 23°C). A list of additional salts that can provide other useful points for calibration may be found on Greenspan (1977). An example of the calibration results for one of the used sensor is shown on Figure 3.

Figure 3. An example of typical calibration for relative humidity measurements.

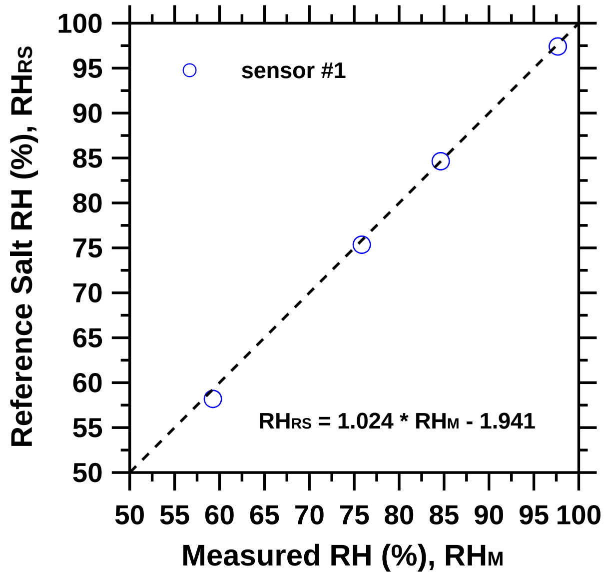


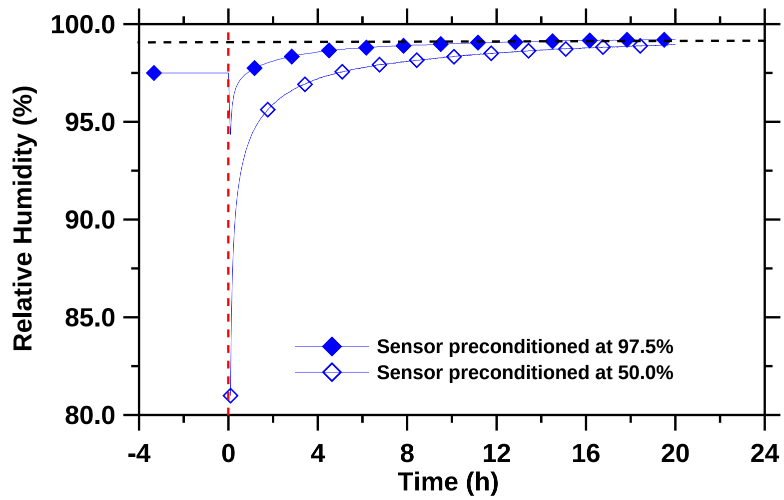
Figure 4 shows an example of relative humidity measurements over a synthetic pore solution corresponding to a cement paste at an age of 24 h (Castro et al. 2010). It should be noted that care needs to be exercised to insure that the sample temperature is properly accounted for (in this case maintained constant at 23 °C ± 0.1 °C). It should also be noted that the sensor requires time to equilibrate with the solution. Figure 4 shows the effect of using sensors preconditioned at different relative humidities before the test. A time of 2 h to 3 h is needed to attain equilibrium when the sensors were preconditioned for 4 h prior to the test at a relative humidity that is similar to what is expected from the test (i.e., 97.42 ± 0.48 % RH using potassium sulfate at 23 ± 0.1 °C When the sensors were preconditioned at 50 ± 0.5 % RH (to simulate room RH) it was observed that the sensors could take much longer to equilibrate with their surroundings (8 h to 10 h). As a result, a period of preconditioning is recommended for RH measurements where rapid equilibrium may be needed. It can also be observed that the repeatability between samples is 0.8 % RH.

Cement Hydration. To measure the potential increase in hydration at early ages, an isothermal calorimeter (TAM Air by Thermometric) was used as previously reported by Pane and Hansen (2005), Poole et al. (2007), Johansen et al. (2009), Lura et al. (2010), and Castro et al. 2012b). The cumulative heat of hydration was measured during the first seven days after mixing. Approximately 10 g of externally mixed mortar were placed into each vial, with two replicates for each mixture. The measured heat of hydration is normalized by the theoretical maximum heat of hydration. This maximum theoretical value (522 J/g of cement) was calculated using the heat of hydration of the cement compounds (measured in cement) reported by Gartner et al. (Bensted and Barnes 2002).

An SEM was used to assess the degree of hydration of cement and porosity near the NWA and LWA surface to measure the potential increase in hydration at later ages.

The area of the un-hydrated cement particles at the ITZ were analyzed using image acquisition following the procedure used by Diamond (2004).

Figure 4. Typical relative humidity measurement over a simulated pore solution



Experimental Results and Discussion.

Relative Humidity Measurements. Figure 5 shows the relative humidity measurements as a function of time during the first seven days after casting. As it can be expected, the relative humidity decreases as a function of time for all mixtures, however the rate at which the relative humidity change is highly influenced by the w/c and the amount of pre-wetted LWA that was used (0, 50% or 100% LWA). For the system prepared with a w/c = 0.30, there is a difference of 10% RH after seven days between the plain mixture and the internally cured mixture. That difference is reduced to 3% for the system prepared with a w/c = 0.45.

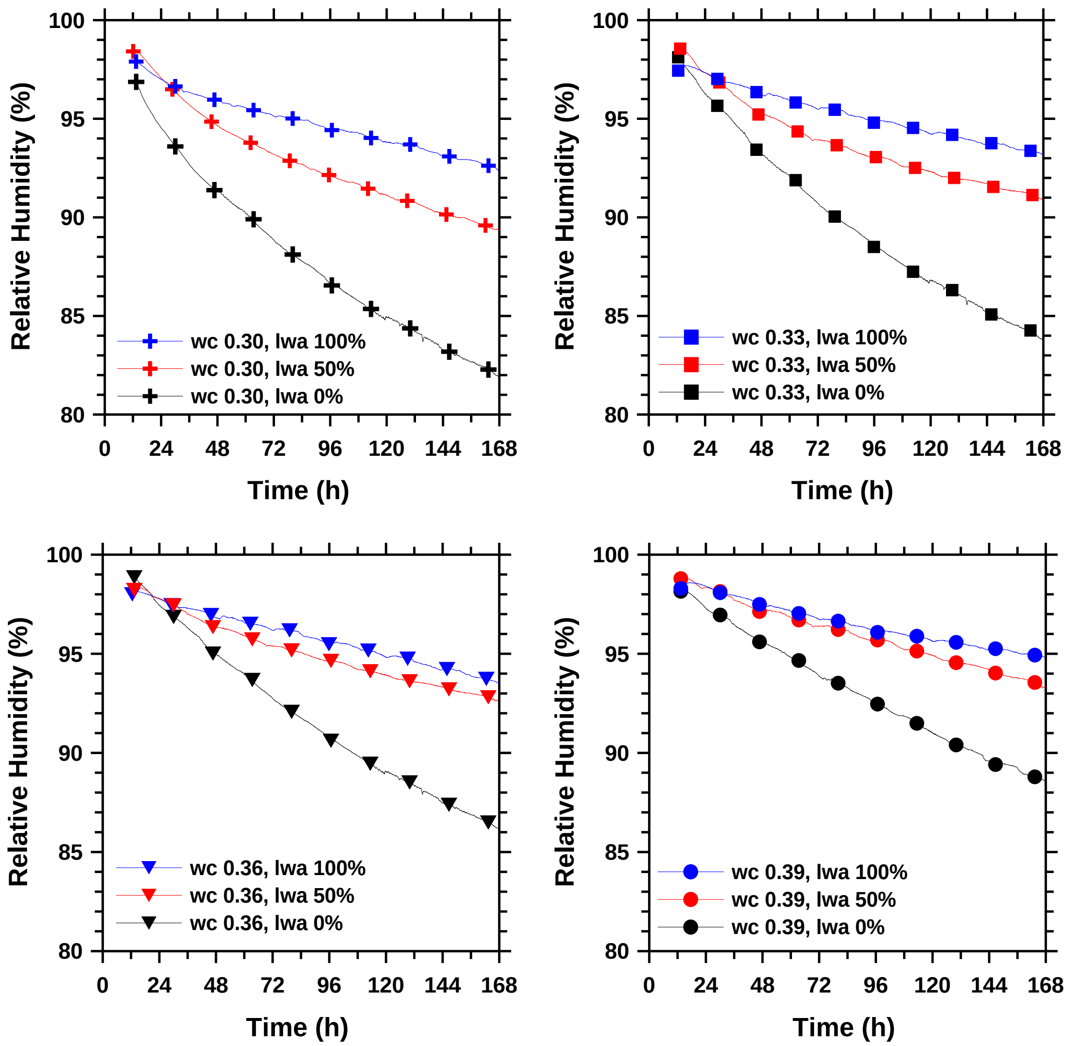
From Figure 5 it can be noted that even in a systems with larger pores and a higher degree of saturation (in theory systems at early ages or for a system with a high *w/c*), the internal RH is lower than 100%. This can be explained by the presence of dissolved salts in the pore fluid, as the amount of water molecules that are present at any time in the gas phase is proportional to their amount in the liquid phase. This concept is expressed in Raoult's law, Eq. 2.

$$RH_s = \frac{p_g}{p_{sat}} = X_l \qquad \textbf{Eq.2}$$

where X_l is the molar ratio of water in the pore solution, p_g is the partial vapor pressure of water and p_{sat} is the partial vapor pressure at saturation. It should be noted however that Raoult's law is only strictly valid for dilute solutions. For concentrated solutions, the interaction between the ions in the solution starts to play a role and the RH drop should be calculated based on the activity of the water in the solution (Castro et al. 2011).

At 24 h there is not visible difference between the systems containing 50 % and 100 % of the amount of LWA calculated using the Bentz and Snyder (1999) approach. This is also expected as the demand for water at early ages is low enough as the system containing the 50 % LWA can supply all the needed water. At a later age however, it is possible to observe a considerable reduction on the relative humidity on the systems prepared without LWA.

Figure 5. Relative humidity measurements as a function of the time after casting.



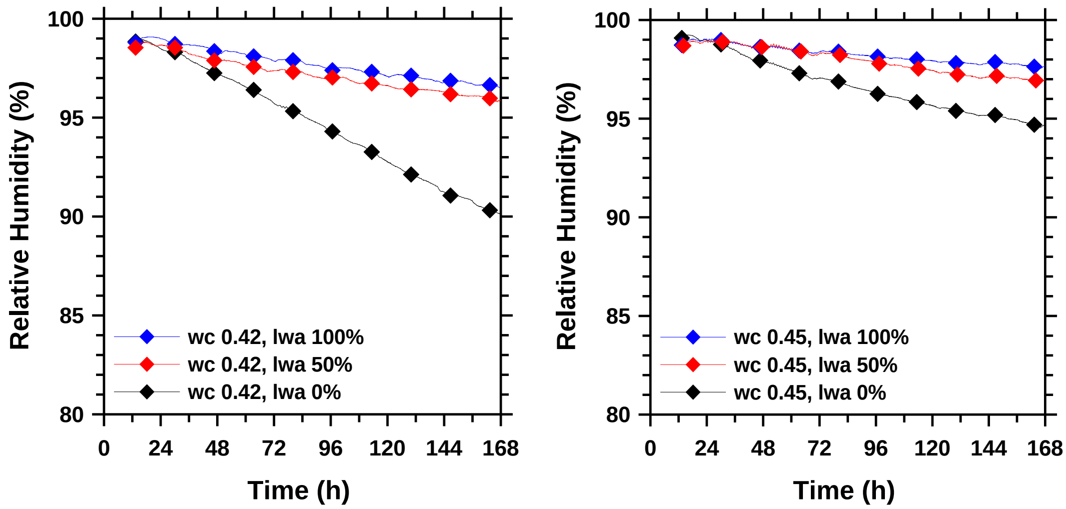


Figure 6 shows the relative humidity measurements as a function of the *w/c*. The relative humidity has been plotted at six particular times after casting (1, 2, 3, 4, 5 and 7 days after casting). At early ages (24 h after casting), the inclusion of LWA particles do not have a substantial effect except for the systems with the lowest w/c (w/c of 0.30 and 0.33). This benefit becomes more substantial and for higher w/c over time. It can be noticed however supplying 50% LWA provides greater than half the benefits of the 100% LWA system. While the exact benefits will depend on the pore sizes that are refilled by the internal curing water, the amount of water in the LWA can be used with the pore size distribution to determine the relative humidty that can be expected (Barrett et al. 2014).

The pores in the higher water to cement ratio cement paste are larger. In the higher w/c mixtures the smaller pores remain filled with water and the water leaves the largest pores which according to the Kelvin Young-Laplace equation has little reduction in the relative humidity.

Some of the samples that were cast into cylindrical containers (50 mm height and 22 mm diameter) were kept sealed in the container until reach the age of 1 year. After that, samples were demolded and relative humidity measurements were taken following the same procedure. From Figure 7 it can be seen that little difference exists for the low w/c systems (w/c = 0.30) in the measured relative humidity at 1 year after casting. However, at higher w/c, there is up to 8 % of increase in the relative humidity of internally cured system compared with the plain system. This may reflect additional water in the system that can be still available for further hydration.

Degree of Hydration by Calorimeter. Figures 8 and 9 show the measured heat of hydration for mortars made using the normal weight aggregate and internally cured mortars made using the prewetted lightweight aggregate that have been sealed since the time of casting. In Figures 8 and 9 the y-axis on the left hand side shows the results in term of the heat released while the y-axis on the right hand side shows the same results but normalized by the theoretical maximum heat that can be released by a gram of the cement used in this research (522 J/g of cement). This normalized value can be interpreted as the degree of hydration of the sample.

The results show that initially all the mixtures demonstrate a similar rate of heat release irrespective of the *w/c*. Also, it can be observed that after the first 24 hours a difference begins to be noticed between the different *w/c* mixtures. Mixtures with a higher water to cement ratio show a greater heat release. This suggests that lower *w/c* mixtures have a reduction in their hydration due to either a lack of water to participate in the hydration (or relative humidity) or a lack of space for the particles to hydrate. It can be noticed that specimens with internal curing show a slightly higher degree of hydration for each mixture due to the use of the LWA.

Figure 10 shows the degree of hydration of the mixtures at the time of 24, 72, and 168 hours. While the cumulative heat of hydration increases steadily for lower w/c mixtures, a plateau begins to develop for higher w/c mixtures. This plateau would be less pronounced at a later age. From the data shown in Figure 10 it appears that the cumulative heat of hydration is related to the water to cement ratio of a mortar and the amount of free water available for the mixture. This effect is most pronounced for low water to cement ratio materials.

Figure 6. Relative humidity measurements as a function of the w/c

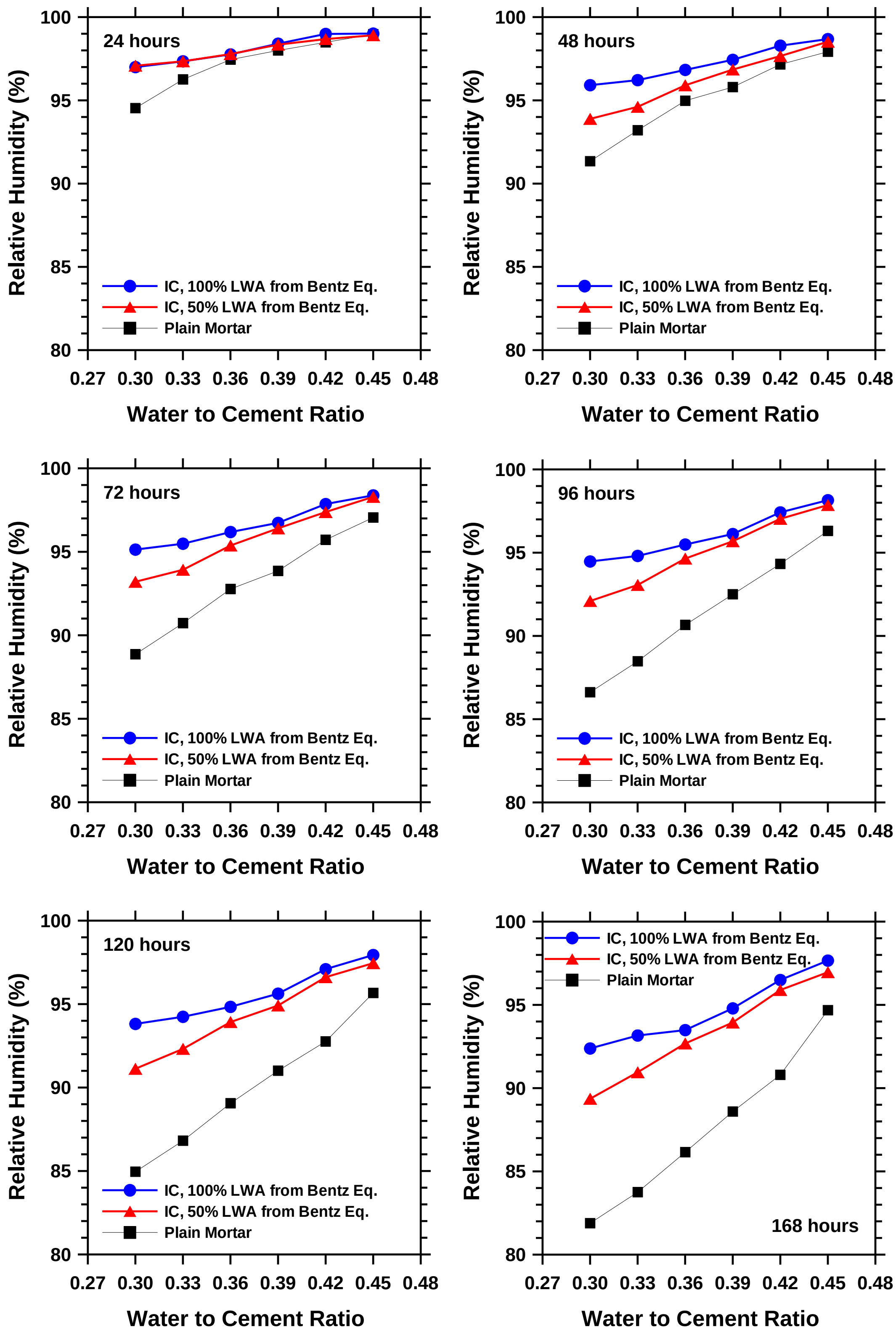


Figure 7. Relative humidity measurements at the age of 1 year.

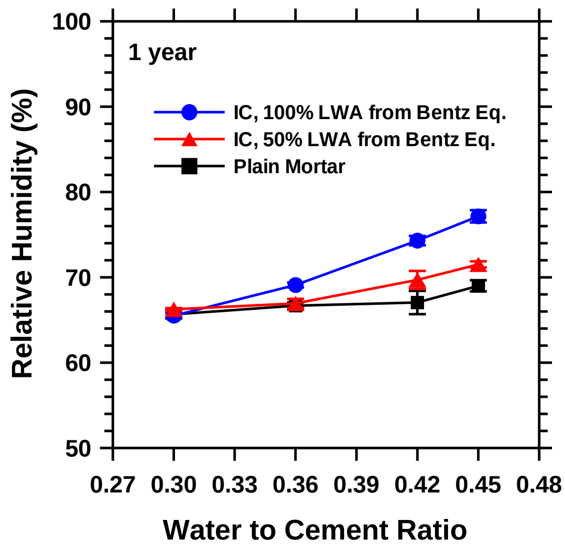
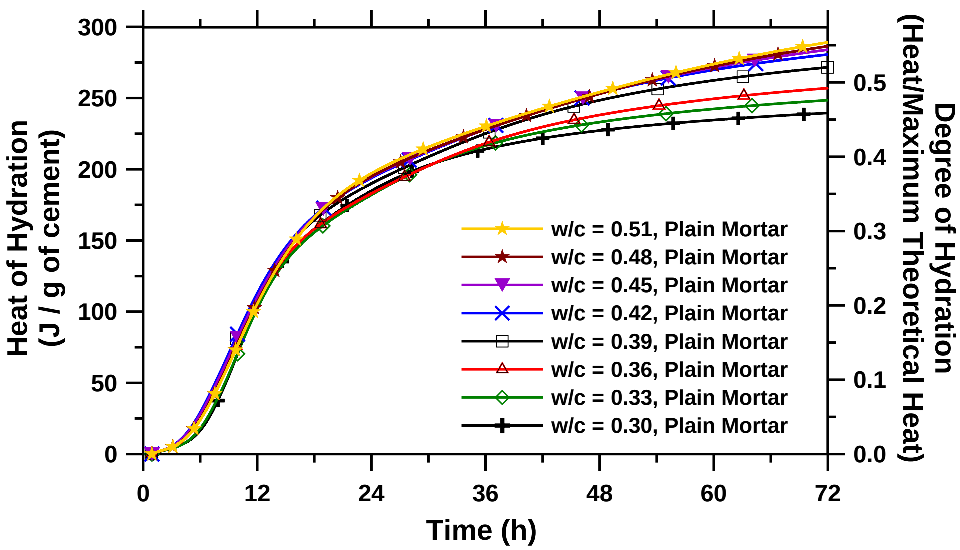


Figure 8. Effect of water to cement ratio on hydration on plain mortar systems.



Relative Humidity vs Degree of Hydration. Figure 11 shows the relationship between the relative humidity and the degree of hydration of cement expressed as the total heat released. The addition of LWA produces that the curves shift to result in a higher degree of hydration. In practical terms, this can be interpreted as the inclusion of LWA is equivalent to increase the w/c of the mixtures (in terms of hydration), which generate a higher degree of hydration but not the higher permeability or lower strength provided by a higher water to cement ratio. The curve of the internally cured mixture with w/c 0.30 behaves similarly to the curve of the plain mixture with w/c 0.36. The curve of the internally cured mixture with w/c 0.33 behaves similarly to the curve of the plain mixture with w/c 0.39. The curve of the internally cured mixture with w/c 0.39 behaves similarly to the curve of the plain mixture with w/c 0.42.

Figure 9. Effect of water to cement ratio on the hydration on internal cured mortar

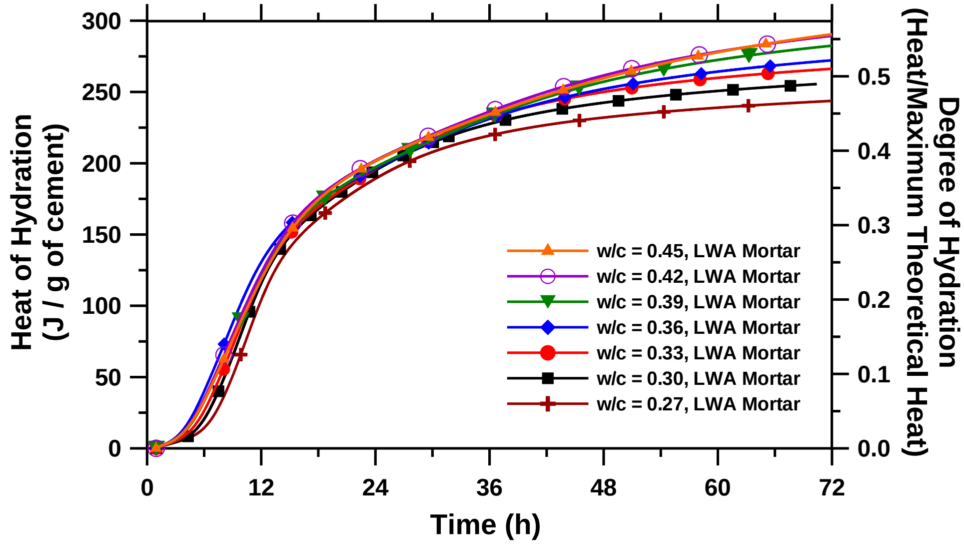
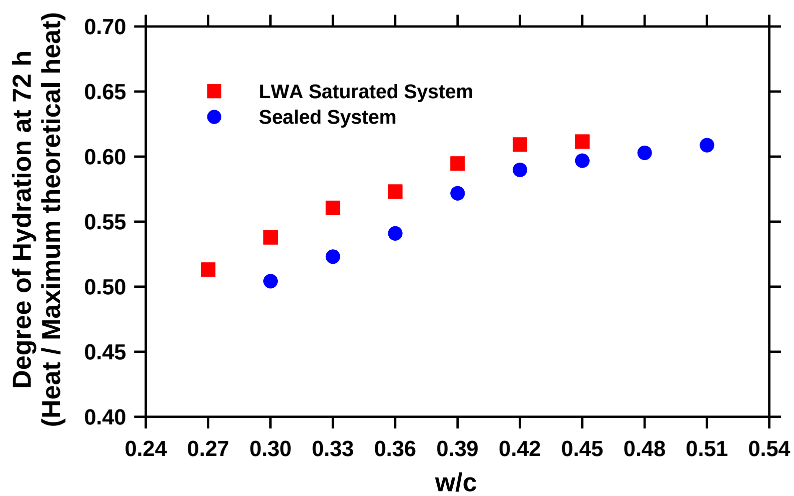


Figure 10. A plot of the cumulative heat released at 72 hours as a function of the *w/c*.



Degree of Hydration by SEM at Later Ages: Figure 12 shows the results from the image analysis performed on SEM images from samples with a w/c of 0.30 system at a) 1 day, b) 28 days, and c) 365 days. Figure 12 d) show the results of the image analysis of the w/c 0.42 system at 365 days. Figure 12 a) through 12 c) show that the volume of un-hydrated cement particles decrease with age due to hydration. It can also be seen that the use of internal curing reduces the amount of un-hydrated cement particles in the vicinity of the lightweight aggregate at all the different ages tested. This may be related to less calcium hydroxide being deposited on the surface of the aggregate which may encourage more cement hydration due to local chemistry. However, the benefits of internal curing appear to decrease with the age of the sample. This is likely due to the fact that a majority of the empty space is filled in for both systems. Figure 12 d) shows that at 1 year after casting, systems with a higher w/c (w/c = 0.42) can also be beneficiated by the use of pre-wetted LWA.

Figure 11. Influence of internal curing on the internal relative humidity of mortars and in the rate of hydration of cement.

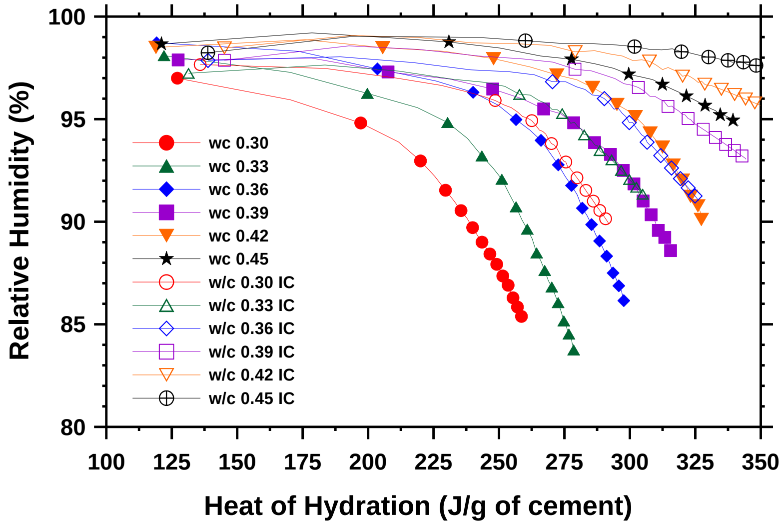
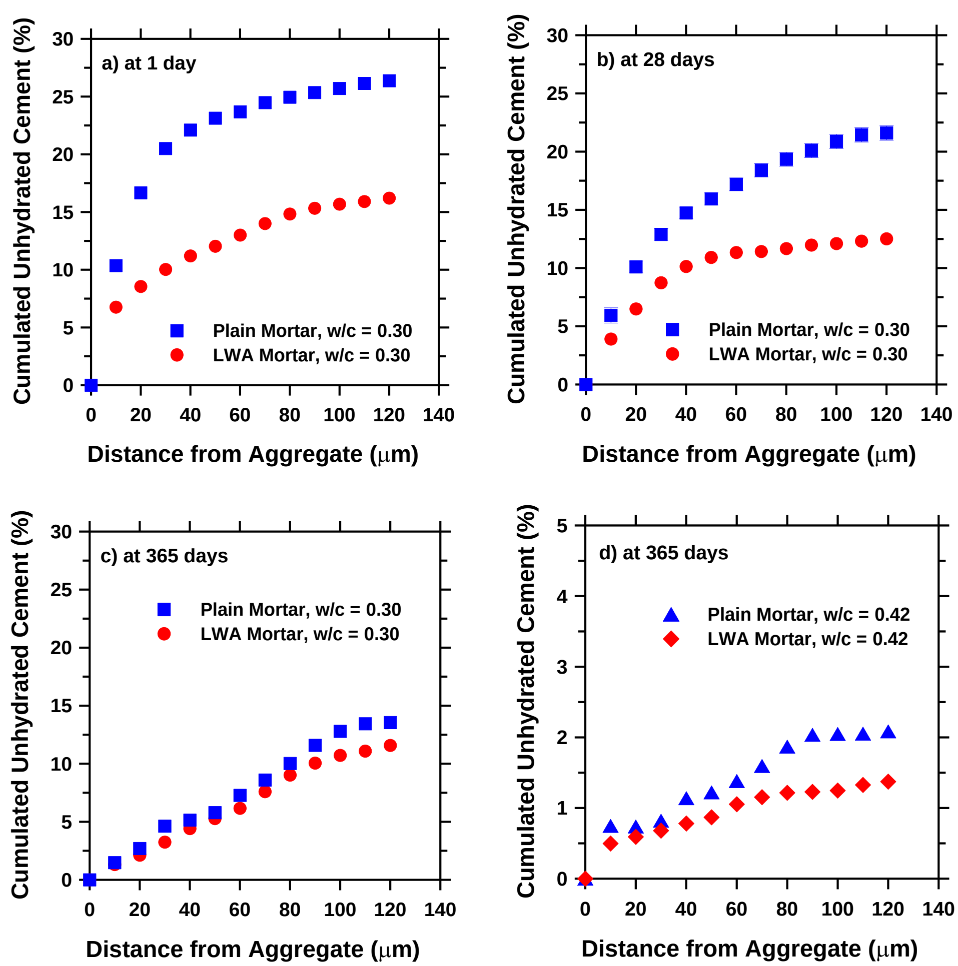


Figure 12 Un-hydrated Cement Particles Measured by SEM Image Analysis



Conclusions

Internal curing is being increasingly considered for use in concrete pavements. Internal curing frequently uses prewetted lightweight aggregate (LWA) to provide water to the cement paste as it hydrates. Questions exist however as to the amount of LWA that should be used in these mixtures. While other applications have simply add a supply of water that is equal to the chemical shrinkage volume or to account for the chemical shrinkage volume and water lost due to drying this paper evaluates the benefit of using lower volumes of LWA replacement.

This paper described a series of tests that evaluate the degree hydration of plain and internally cured mortars using isothermal calorimetery and measurements of relative humidity. The calorimetery measurements performed using plain mortars prepared with a w/c between 0.27 and 0.51 showed that while the cumulative heat release curves are similar irrespective of water to cement ratio at early ages (before 24 hours), they begin to diverge at a later age with higher water to cement ratios exhibiting a higher heat release which is due to the additional space for hydration as well as the additional amount of fluid available for hydration. It was shown that the inclusion of pre-wetted LWA supplies additional water that increases the degree of hydration especially for mortars with low water to cement ratios.

Scanning electron microscope images were used to evaluate the hydration of cement at later ages. Measurements performed at 28 days and 365 days on systems prepared with water to cement ratio of 0.30 shows that the effect of additional hydration produced by internal curing increases with time. Measurements performed on mortar systems prepared with water to cement ratio of 0.42 shows that the benefits of internal curing on the cement hydration can be extended to higher w/c systems. However, internal curing requires longer periods of time in order to demonstrate these improvements in high w/c systems.

Relative humidity measurements are dependent on the *w/c* of the system as well as the amount of water used for internal curing. Using a volume of water that is equivalent to the volume of chemical shrinkage is an efficient way to increase the hydration and internal relatively humidity; however lower amounts of internal curing water also have benefits. While the use of internal curing water that replaces only a fraction of the chemical shrinkage water is also beneficial, these benefits are highly related to the pore size distribution of the cement paste. The approach discussed by Barrett et al. (2014) could be used to tailor the amount of water to the size of the pore that empties, the relative humidity and the corresponding shrinkage.

When the internal relative humidity is plotted against the degree of hydration of the cement at early ages, it can be observed that at similar relative humidity, the internally cured mortar shows a higher degree of hydration. This provides a more dense system, especially around the LWA particles. This can explain increases in strength as well as reductions in fluid transport properties. The results obtained in this work can provide valuable data for use in life cycle models for predicting the long term performance of internally cured concrete. The results show that for w/c typical for concrete paving partial replacement levels may be very successful.

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