LWA Absorption and Desorption: The Influence on the Microstructure and Transport Properties of Internally Cured Mortars

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Abstract

The increased propensity for shrinkage cracking in low water-to-cement ratio (*w/c)* concrete has resulted in the development of new methods that can reduce the risk of early-age cracking, including internal curing. Internal curing can use water-filled lightweight aggregate (LWA) to supply 'curing water' to low *w/c* paste as it hydrates. Significant research has been performed to determine the effects of internal curing on shrinkage and stress development; however, relatively little detailed information exists about the effects of internal curing on transport properties such as water absorption or ion diffusivity. This study examines the influence of internal curing on absorption of water and the electrical conductivity in mortar specimens. In addition, procedures for determining the absorption and desorption of LWA are also discussed, as this can play a substantial role in determining the volume of LWA that is needed and the amount of curing water that can be supplied to the paste   The results indicate that the inclusion of water-filled LWA can increase the long term degree of hydration of the cement paste and reduce both the total porosity and the interconnection of the pore structure of mortar specimens. The sorptivities and electrical conductivities exhibited by these mortars were better than expected. For example, results for an internally-cured mortar with a *w/c* of 0.30 indicate performance that is consistent with a conventional mortar prepared with a *w/c* of 0.23 or a paste with a *w/c* of 0.30.

1. Introduction

The propensity for shrinkage cracking in low water-to-cement ratio (*w/c*) concrete has resulted in the development of methods that can reduce the risk of early-age cracking. One of these methods is referred to as internal curing. Internal curing uses water-filled inclusions (typically lightweight aggregate, (LWA)) to provide sacrificial water-filled reservoirs to supply water on demand to the hydrating cement paste from the time of mixing until the time when moisture equilibrium is achieved between the reservoirs and the surrounding cement paste [1-5]. This allows the fluid in the lightweight aggregate to be transferred to the paste thereby reducing self-desiccation in the paste along with its accompanying autogenous shrinkage, and thus avoiding early-age cracking [6-10].

Previous work [5, 11-13] has shown that the use of LWA for internal curing will increase the long term degree of hydration of sealed cured mortars, producing a denser microstructure. Bentz et al. [14, 15] suggested that this densification around the aggregates results in a system with less percolated normal weight aggregate (NWA) interfacial transition zones (ITZs) and therefore could provide a further reduction in sorptivity and fluid transport. Elsharief et al. and Peled et al. [16, 17] used scanning electron microscopy (SEM) to show a reduction in the porosity of the zone surrounding the LWA in comparison with NWA. Peled et al. [16] used an atomic force microscopy (AFM) technique to further confirm the densification of the ITZ and the reduction in calcium hydroxide near the surface of the LWA.

One fundamental concept in the proportioning of mixtures with internal curing is determining the volume of the lightweight aggregate (i.e., volume of the water reservoirs that are needed). A straightforward approach has been developed to estimate this volume of water based on the concept that all of the created chemical shrinkage volume will be replaced by water. This concept has been proposed by Bentz et al. [4, 18] and is shown in equation 1:

$$M_{LWA} = \frac{C_f \times CS \times \alpha_{max}}{S \times \phi_{LWA}}$$
Equation 1

where: M_{LWA} (kg/m^3) is the mass of LWA (in a dry state) that needs to be saturated to provide sufficient water to fill in the voids created by chemical shrinkage, C_f (kg/m^3) is the cement content of the mixture, CS (g 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 (0 to 1) and was taken to be 1 in this study when the dry LWA was soaked for 24 h, and ϕ_{LWA} (kg of water/kg of dry LWA) is the absorption capacity of the LWA (taken here as the 24 h absorption value). It has been stated that it may be more appropriate to use the desorption of the LWA down to 92 % RH rather than the absorption [18].

It can be noted that to assess correctly the effect of internal curing agents (as LWA) an accurate determination of absorption capacity is crucial. It may also be argued that in addition to just knowing the absorption capacity (i.e., how water can be absorbed by the aggregate), it is also vital to know how water is lost from the aggregate during curing (i.e., the desorption response). For these reasons, this research will focus on the analysis of available methods to characterize the absorption and desorption behavior of LWA. In addition, the effect of internal curing will be measured with respect to transport using electrical impedance (conductivity) and water sorption measurements.

2. Research Significance

Specifically this paper considers two main aspects associated with the LWA that is used for internal curing.

- First, the paper will focus on the challenges and importance of an accurate determination of absorption and desorption in the LWA. The need for accurately determining the water that can be absorbed and more importantly desorbed (i.e. given off by the aggregate) can clearly be seen in the value of ϕ_{LWA} described in Equation 1.
- Second, this paper will also discuss the beneficial effects of internal curing in terms of increased hydration of the mixture and the densification of the microstructure of the ITZ around the aggregates. This will be discussed in terms of water absorption, electrical impedance, and degree of hydration.

3. A Description of the Materials and Mixtures Used in this Investigation

Eight different mixtures were prepared (Table 1). Three mixtures were cement paste with different *w/c* (designated as P-0.25, P-0.30, and P-0.35) with the number on the right representing *w/c*. Three mixtures were plain mortar with a single volume fraction of fine

aggregate (55 % of the total volume) and different *w/c* (designated as M-0.25, M-0.30, and M-0.35) with the number on the right representing *w/c*. Two mortar mixtures were prepared with different volumes of sand replaced by a saturated rotary kilned-expanded shale (from here on, LWA) and a *w/c* of 0.30. These mixtures were designated as 11.0 % and 23.7 %, with the number representing the total volume of the mixture occupied by the LWA (in percentage). It is important to note that though the designations are on a total volume basis, the volume of aggregate (LWA and sand) remained constant at 55 % since only the sand was replaced with LWA. It should also be noted that 23.7 % volume replacement corresponds to the amount of LWA necessary to compensate for the anticipated chemical shrinkage according to equation (1).

Table 1 Mixture Proportions in saturated surface dry (SSD) condition

Material	P-0.25	P-0.30	P-0.35	M-0.25	M-0.30	M-0.35	11.0%	23.7%
Cement (kg/m^3)	1761	1619	1498	793	728	674	728	728
Water (kg/m^3)	440	486	524	198	218	236	218	218
Fine Aggregate (kg/m^3)	0	0	0	1418	1418	1418	1135	808
Dry LWA (kg/m^3)	0	0	0	0	0	0	172	369
HRWRA (g/ 100 g cement)	0.60	0	0	0.75	0.50	0.60	0.50	0.66
Additional Water Provided by LWA (kg/m^3)	0	0	0	0	0	0	18	39

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 phase composition of 56 % C_3S, 16 % C_2S, 12 % C_3A, 7 % C_4AF and a Na_2O equivalent of 0.68 % by mass.

A polycarboxylate-based high-range water-reducing admixture (HRWRA) was added in varying rates depending on the mixture, as indicated in Table 1. Different rates of HRWRA addition were employed to maintain similar consistencies (i.e., workability) of these mortar mixtures.

The normal weight sand used was natural river sand with a fineness modulus of 2.71, an apparent specific gravity of 2.58, and an absorption of 1.8 % by mass. Portions of the normal weight sand were replaced with LWA with a fineness modulus of 3.10 and a specific gravity of 1.56. The 24-hour absorption of the LWA was determined to be 10.5 % according to ASTM C128-07 [19].

The mixing procedure used for the mortar was in accordance with ASTM C192-06 [20] while the procedure for the paste was in accordance with ASTM C305-06 [21]. The LWA was oven dried, air cooled, and then submerged in water for 24 h ± 1 h prior to mixing. The volume of water used to submerge the LWA included both mixing water and the water that 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.

4. Aggregate Absorption and Desorption

Determining the absorption of the aggregate can be problematic due to difficulties in determining the saturated surface dry (SSD) condition for lightweight aggregate. This paper used three

different techniques to evaluate SSD and compared the results. The techniques included the standard cone method [19], the paper towel method, and the use of cobalt chloride [22].

It should be noted that ASTM C128 is not currently intended to be used for determining the absorption of LWA, as stated in sections 1.3 and 5.5 of the standard [19]. However, in lieu of a more appropriate method for determining the absorption of LWA, ASTM C128 is commonly used. In this test, the sample is immersed in water for 24 h, after which time the water is decanted and the surface of the aggregate is dried. The difficulty lies in determining the saturated surface dry condition, i.e., when there is no more surface moisture on the aggregate. To determine the saturated surface dry condition of fine lightweight aggregate, the damp aggregate is placed in a cone, tamped 25 times, and the cone is removed. The cone (and provisional cone) tests rely on the principle of surface tension for determining when the surface moisture disappears. When moisture is still on the surface of the aggregate, surface tension of the water will hold the particles in the form of the cone after the cone is removed. When no moisture is present on the surface, slight slumping of the aggregate cone will occur. The question that arises from using the cone tests is that the LWA is typically manufactured and crushed, resulting in angular aggregate particles. The angularity of the aggregate can lock the particles together such that when the cone is lifted, the shape is retained even after surface moisture is gone.

The paper towel method involves immersing the aggregate in water for 24 h, after which the water is decanted and the surface of the aggregate is patted dry using a paper towel. This process is repeated at different moisture contents (preferably near to the SSD condition). Once it appears that the paper towel is no longer picking up moisture from the aggregate, it is assumed that a surface dry condition has been reached and that the aggregate moisture content can be determined.

The cobalt chloride method [22] also involves immersing the aggregate in water for 24 h after which time the water is decanted and the surface of the aggregate is dried. At that time, a small amount of cobalt chloride powder is sprinkled on the surface of the aggregate. Cobalt chloride changes color in the presence of moisture from blue in the anhydrous (i.e., dry) form to pink when it reacts with water [22]. This process is repeated at different moisture contents (preferably near the SSD condition). After a photo is taken, the cobalt chloride is removed from the surface of the aggregate and the aggregate is placed in the oven to determine the moisture content. When there is a higher moisture content, more water is on the surface of the aggregate and therefore more water can react with the cobalt chloride, resulting in a deeper red color. As the aggregate dries and the moisture content decreases, a lower extent of reaction occurs, resulting in the cobalt chloride appearing bluer.

The images of cobalt chloride on the surface of the aggregate were analyzed in image processing software (ImageJ©, freely available at http://rsbweb.nih.gov/ij/). The original RGB images were split into three separate images according to their red, green and blue colors. The area of the cobalt chloride on the surface of the aggregate of the blue image was selected and the average hue of the cobalt chloride was determined.

Figure 1 shows the average hue of blue obtained from the image analysis as a function of the moisture content in the aggregate. The average hue will increase as the aggregate dries since unreacted (i.e., dry) cobalt chloride is blue and only the blue image was processed. An image was also captured of cobalt chloride in the unreacted state to determine the upper bound of the hue of blue. The upper bound represents the theoretical maximum hue that could be reached. The aggregate begins to reach this value at low moisture contents. This is because no water is on the surface of the aggregate to react with the cobalt chloride. A lower bound was also determined by combining cobalt chloride with water and letting a complete reaction take place.

The results from the paper towel and standard cone methods are also shown along with the cobalt chloride method on Figure 1. The color change appears to begin to stabilize (which would indicate no water is on the surface) at approximately 11 %. This is close to the 10.5 % indicated by the standard cone method. It should be noted that some difference is observed between each method which can influence the mixture design. The impact of these differences will be presented later in this section.
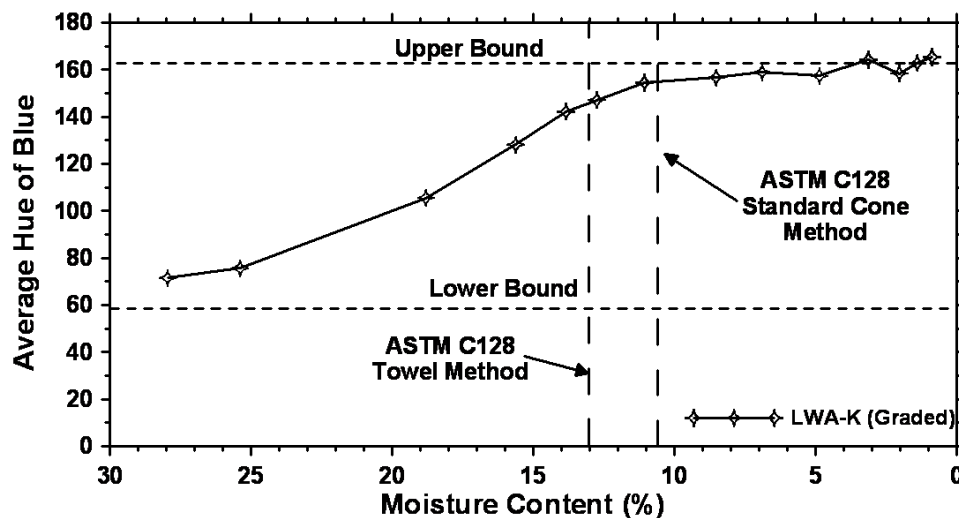

Figure 1: Average hue of blue at different moisture contents for LWA using the cobalt chloride method and comparison with the standard cone and towel paper methods. The average standard deviation for the cobalt measurements was 5, and the average standard deviation of the moisture contents was 0.5 %.

The aforementioned methods rely on saturating a specimen for 24 h and then determining the disappearance of surface moisture for determining the absorption capacity of the LWA. It is important to note, however, that the further absorption of water by the lightweight aggregate takes place over time. Figure 2(a) shows the rate at which LWA absorbs water during the first 48 h, starting from an oven-dry condition. This shows that while the rate of water absorbed during the first several hours is very high, the total absorption will continue to increase for several days, weeks, or months. As a result it is very important to know that any extra absorption during the time elapsed between mixing and set will be conditioned by the amount of time that the LWA was presoaked.

Figure 2(b) evaluates the effect of a change in the value of absorption assumed for mixture proportioning. This difference can be produced because of a miscalculation of the actual absorption or because of additional water absorption produced after mixing and before set. From Figure 2(b), one can see that a variation in the reported absorption value of ± 2 % (similar to the variation measured with the three test methods) will produce a change in the actual *w/c* in the range of ± 0.01 (*w/c* between 0.29 and 0.31). In other words, if an LWA absorption of 10.5 % is assumed, but the actual absorption is 8.5 %, the mixture will have additional water resulting in a higher initial mixing *w/c*, because the LWA is pre-mixed with the total water as described earlier. This example is based on the Mixture 23.7 % from Table 1, assuming 10.5 % absorption for the LWA at 24 h.
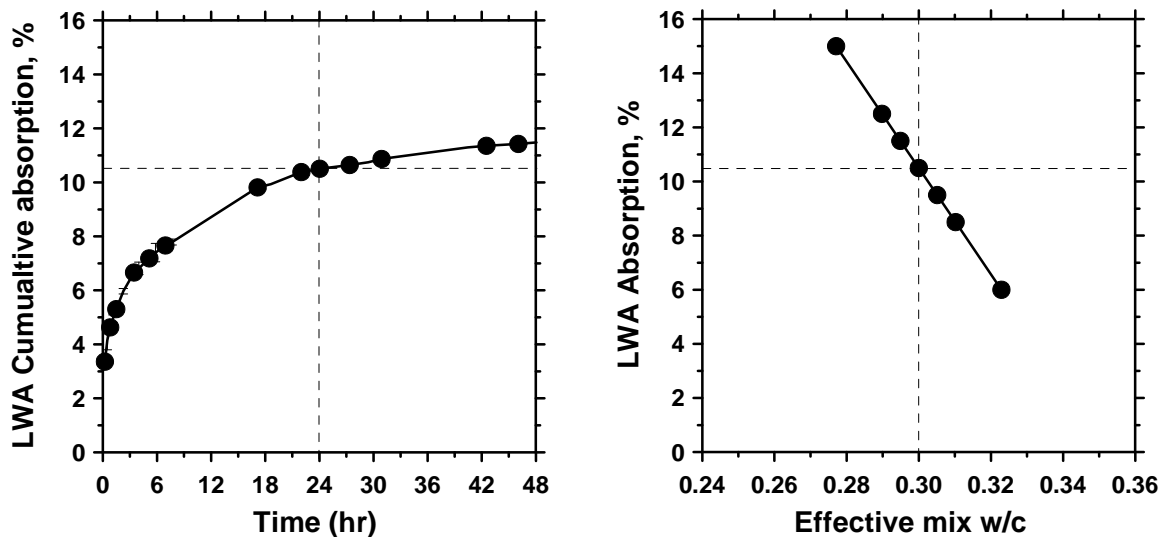
Figure 2: (a) Absorption of LWA over time, (b) evaluation of the effect of a change in the absorption on the effective *w/c* for mixture 23.7 % (in Figure 2(a) typical variability in the average of 3 samples is 0.1 %).

It is important to note that the absorption characteristics of an aggregate can also depend on the aggregate size. Larger LWA generally have larger voids, and when the aggregate is crushed the large voids will be lost. The different pore structures will affect both the absorption rate and the absorption capacity of LWA. Figure 3a shows the mass percent of water absorbed as a function of time for different LWA particle diameters. As the particles become smaller, they take on water at a slower rate and absorb less water than their larger counterparts. Figure 3b shows the mass percent of water absorbed normalized by the particle diameter. From this, it can be seen regardless of the size of the particle, the absorption is nearly the same when normalized by the particle diameter.

Moisture desorption may be even more important than absorption capacity, because it will determine how much of that available water (absorption capacity) will actually participate in the hydration of the cement. A moisture desorption isotherm is an established technique for evaluating the effects of temperature and relative humidity on the moisture content of a material.
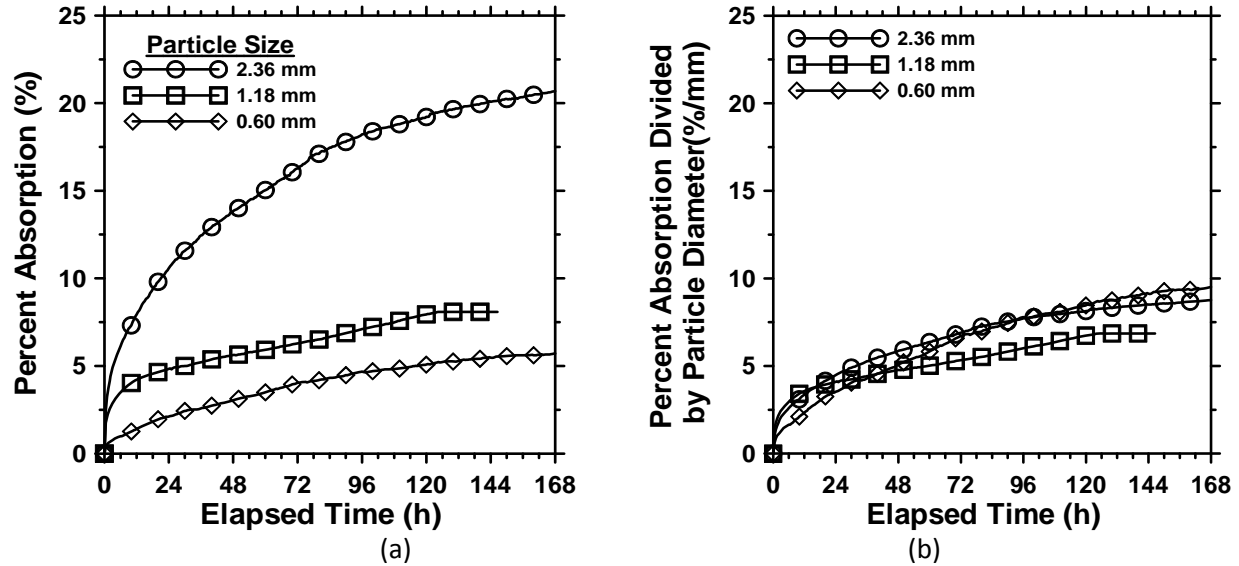
Figure 3: (a) The absorption rate of different particle diameters of the same LWA. (b) The absorption rate of different particle sizes normalized by the particle diameter.

The desorption response was also measured for LWA after it was oven dried for 24 h ± 1 h, cooled, and placed in deionized water for 24 h ± 1 h prior to testing, to prewet the aggregate. The prewetted LWA was removed from water and "patted to SSD" using absorbent towels. A 70 mg to 100 mg sample was placed in a tared 180 mL quartz pan. The pan containing the sample was then suspended from the balance (± 0.001 mg accuracy) and placed in the relative humidity chamber to equilibrate at 23.0 ± 0.1°C and 98 ± 1% RH for up to 48 h or until the sample had achieved a stable mass (less than an 0.001 % mass change/15 minutes). After the sample equilibrated, the relative humidity in the chamber was changed in 1 % RH steps to 80 % RH, allowing the sample to equilibrate at the new humidity (less than an 0.001 % mass change/15 minutes or 12 h of exposure, unless noted otherwise). After equilibrating at 80 % RH, the samples were dried to 0 % RH for up to 48 h or until the sample had achieved a stable mass.

Figure 4 shows the desorption response of LWA samples that were conditioned as mentioned in the previous paragraph. This LWA appears to be a good aggregate for use in internal curing, since a large volume of the water leaves the aggregate at high (i.e., greater than 90 % or 95 %) relative humidity, exactly when this water is most needed by the hydrating cement paste.

After the absorption-desorption behavior of the LWA has been accurately characterized, it is possible to study the effect of using LWA as internal curing agents. In this research, three test methods will be use to assess the effect of LWA on performance: electrical impedance spectroscopy (conductivity), sorption or water absorption (ASTM C1585), and degree of hydration by measuring the heat of the reaction. The results of these tests are presented in the next section.
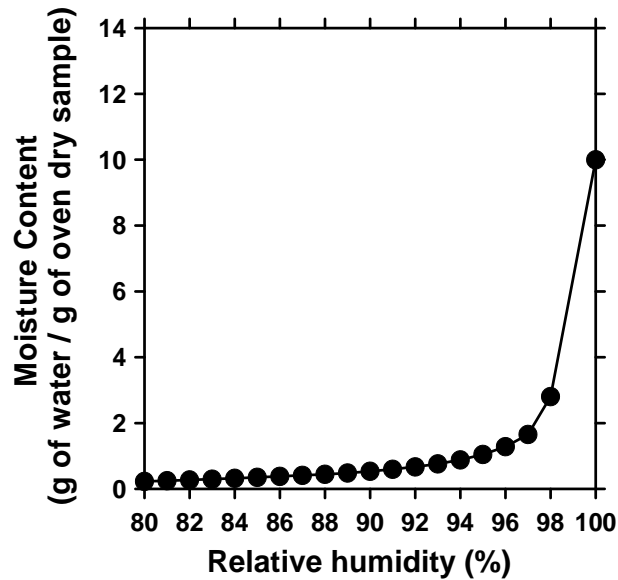
Figure 4: A Typical Desorption Response for LWA (typical variability in the average of 2 samples is 0.2 %).

5. Evaluation of Hydration and Densification of Microstructure around the Aggregate

5.1 Experimental methods

5.1.1 Heat of Hydration

Internal curing supplies additional water that can increase hydration of the cement and densify the microstructure at the surface of the lightweight aggregate. To measure the potential increase in hydration, an isothermal calorimeter was used (TAM Air by TA Instruments[1]). The cumulative heat of hydration was measured during the first seven days after mixing for three mortar mixtures with a *w/c* of 0.30 and three volumes of LWA 0 %, 11.0 % and 23.7 % (mixtures M-30, 11.0 % and 23.7 %). Approximately 20 g of externally mixed mortar were placed into each vial, with two replicates for each mixture.

5.1.2 Sorptivity

The effect of aggregate interfaces and additional hydration was further evaluated by performing the sorption test as specified in ASTM C1585 [23] on samples containing LWA and plain mortars used as references. Three 100 mm × 200 mm cylinders were cast for each mixture. After one day of curing, the samples were demolded and three 50 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a masonry saw, using water as a lubricant. The samples were sealed in two plastic bags until they reached an age of 28 d. After curing in the plastic bags was complete, samples were conditioned by placing them in a desiccator over a saturated potassium bromide (KBr) salt solution that provided an 80 % relative humidity

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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 or recommendation by the National Institute of Standards and Technology or Purdue University, nor does it indicate the products are necessarily the best available for the purpose.

environment at 50 °C. The desiccator was placed in an oven at 50 °C for 3 d. The samples were removed and then stored in sealed containers for 15 d to allow the moisture to equilibrate within the samples. Once the samples were removed from the sealed containers, the side surface was sealed with aluminum tape and the top surface was covered with plastic to avoid evaporation from the sample during testing. After the samples were prepared, testing occurred in accordance with ASTM C1585-04 [23].

5.1.3 Electrical Impedance Spectroscopy

After the sorptivity measurements were completed, the samples were vacuum saturated for 24 h using tap water and the conductivity of the samples was measured using electrical impedance spectroscopy (EIS). Electrodes were placed on the top and bottom surface of the samples; wet sponges were placed between the samples and the electrodes to avoid contact issues between them [11]. The impedance measurements were made over a frequency range from 10 MHz to 10 Hz, using a 500 mV AC signal.

5.2 Results and analysis

5.2.1 Results from Heat of Hydration

Figure 5 shows the measured heat of hydration normalized by the theoretical maximum heat of hydration. This maximum theoretical value (476 J/g of cement) was calculated using the heat of hydration of the cement compounds (measured in cement) reported by Gartner et al. [24].

The mixtures containing the pre-wetted LWA show an increase in heat that can be related to an increase in the degree of hydration. The effect of internal curing can be seen even at early ages due to the supply of additional water from the LWA. A similar increase in heat released at 7 d has been observed in w/c=0.4 mortars prepared with internal curing [15].

Mixtures 23.7 % and 11.0 % show an increase of 4 % and 3 % respectively on the heat of hydration at six days compared with the plain mortar (M-0.30). It can be seen that the addition of LWA slightly increases the degree of hydration, but doubling the amount of LWA (from 11.0 % to 23.7 %) did not have a significant impact on the overall hydration of the mixture during the first 6 d.
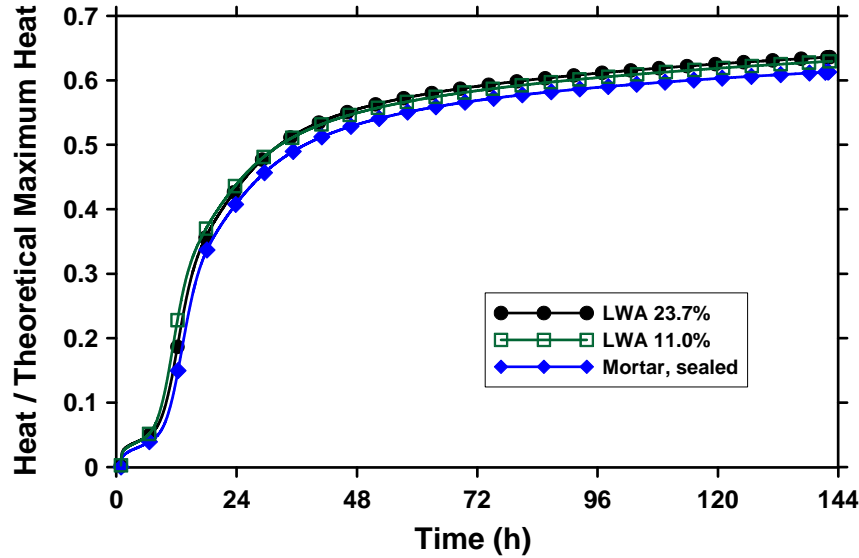
Figure 5: Normalized heat of hydration for *w/c*=0.30 mortars
(typical variability at 6 days in the average of 2 samples is 0.2 J/g of cement)

5.2.2 Results from Sorptivity Testing

Figure 6 show partial results of the sorption tests performed in this research. Additional information about these results can be found in Henkensiefken et al. [11].

Figure 6(a) shows the relationship between the amount of absorbed water at 8 d and the *w/c*, for samples first cured for 28 d. Because of the continued hydration of the LWA mixtures, they exhibit behavior equivalent to that of a conventional mortar with a lower *w/c*. A "mortar equivalent *w/c*" (i.e., the *w/c* of a mortar sample that would exhibit a similar absorption to the absorption in the LWA mixtures) is shown in Figure 6(a). The 11.0 % LWA mortar with *w/c* = 0.30 has a performance similar to a mortar with a *w/c* of 0.28, while the 23.7 % LWA mixture exhibits a sorption performance equivalent to a mortar with *w/c* of 0.23.

Figure 6(b) shows the same data as Figure 6(a), but now is used to determine a "cement paste equivalent *w/c*". The 55/0.30 mixture has a performance similar to a paste sample with *w/c* of 0.37. The 11.0 % LWA mortar has a performance similar to a paste with a *w/c* of 0.35, while the 23.7 % LWA mixture exhibits a performance equivalent to a paste with *w/c* of 0.30. In general, a conventional mortar of a specific *w/c* such as 0.30 exhibits a higher absorption per unit volume of paste than an equivalent cement paste, suggesting the porous and well connected ITZs surrounding each NWA are contributing to increased sorption relative to a plain paste where no such ITZs are present.

Different from what was found with the heat of hydration, a higher amount of LWA was required to produce significant effects on the water absorption results. This suggests that the reduction in the overall water absorption may be related to a reduction in the porosity around the aggregates (ITZ) and a depercolation of the ITZ system, more than with an increase in the overall degree of hydration of the cement.
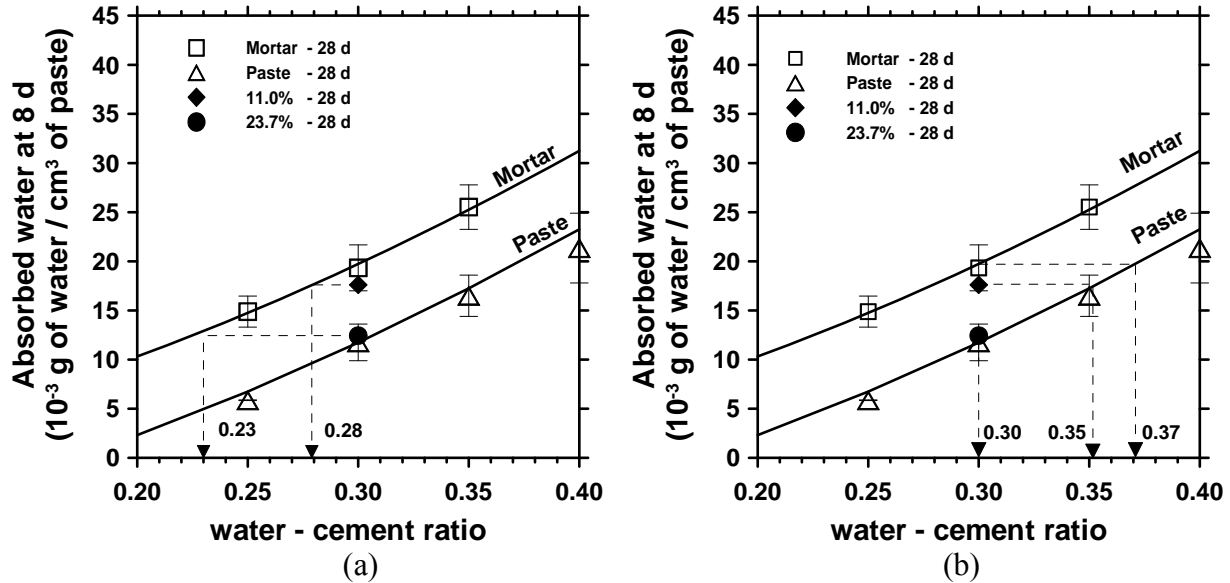
Figure 6: (a) Mortar equivalent *w/c* and (b) cement paste equivalent *w/c*, both determined from cumulative absorbed water at 8 d on samples cured for 28 d. Solid lines are provided to show a general tendency in the data.

5.2.3 Results from Electrical Impedance

Figure 7 shows the relationship between the electrical conductivity and *w/c* for samples cured for 28 d. Similar to the results for absorbed water, a "mortar equivalent *w/c*" was calculated. The 11.0 % LWA mortar has a performance similar to a mortar with a *w/c* of 0.29, while the 23.7 % LWA mixture exhibits a performance equivalent to a mortar with *w/c* of 0.23, consistent with the sorptivity results from Figure 6 (a).
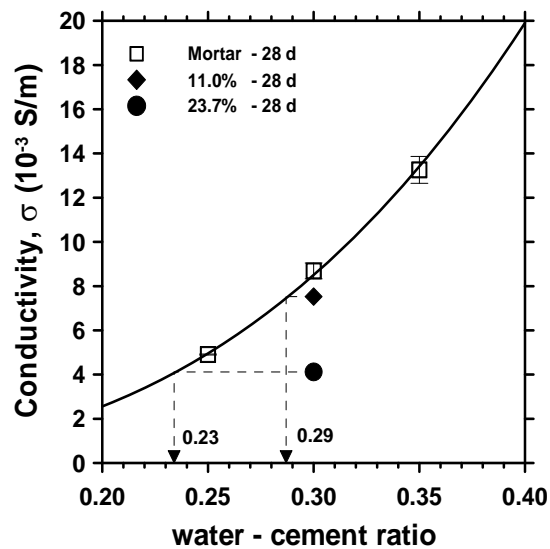

Figure 7: Mortar equivalent *w/c* determined from electrical conductivity on samples cured for 28d. Solid line shows a general tendency in the data.

6. Conclusions

This paper examines the difficulties of an accurate determination of LWA absorption and the effect on increasing durability when LWA is properly used. Specifically, this paper has shown that:

- The available test methods to evaluate the absorption capacity of LWA (standard cone test, provisional cone test, paper towel test and cobalt test) can obtain values that vary on the order of 2 % - 3 % absolute absorption.

- Eventual additional absorption of water by LWA after casting is not considerable when the LWA is first pre-soaked for 24 h. This extra absorption will have a negligible influence on the effective w/c of the mixtures.

- The addition of LWA as an internal curing agent increases the degree of hydration, producing a denser microstructure. The increased density of the microstructure results in less water absorption and reduced ionic (electrical) transport.

- From a sorptivity and conductivity perspective, the $w/c = 0.3$ mixture with 23.7 % LWA performs better than the conventional mortar mixture with a $w/c = 0.25$.

- A reduction in electrical conductivity was observed on vacuum-saturated specimens, indicating that the influences of increased hydration and denser bulk and ITZ microstructures overwhelm those of any increased water-filled porosity of the vacuum-saturated LWA. Thus, isolated water-filled inclusions do not substantially increase conductive/diffusive transport in a well-hydrated dense mortar microstructure.

- Comparing results of heat of hydration, sorption and electrical conductivity, it can be deduced that this densification occurs preferentially around the aggregate interfaces.

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