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# Absorption and desorption properties of fine lightweight aggregate for application to internally cured concrete mixtures

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## ABSTRACT

Recently, substantial interest has developed in using fine lightweight aggregate for internal curing in concrete. Mixture proportion development for these mixtures requires the specific gravity, water absorption, and water desorption characteristics of the aggregate. This paper presents results from a recent study in which the properties of commercially available expanded shale, clay and slate lightweight aggregates (LWA's) were measured. This research measured the time-dependent water absorption response for the lightweight aggregate. The results indicate that a wide range of 24 h water absorption values exist for commonly used fine lightweight aggregates (e.g., absorption between 6% and 31%). Desorption was measured and it was found that between 85% and 98% of the 24 h absorbed water is released at humidities greater than 93%. These properties can be normalized so that they can be efficiently used in proportioning concrete for internal curing. Normalized plots of absorption and desorption demonstrate benefits for a single function that describes a large class of expanded shale, clay, and slate aggregate for use in internal curing.

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## 1. Introduction

# 1.1. Background on internal curing

The idea that lightweight aggregate can provide moisture to the cement as it hydrates in concrete has been known for over five decades [1]. Bloem [2] reported that "high absorption lightweight aggregate may have the beneficial effect of supplying curing water internally". Philleo [3] discussed the potential for improved strength and durability due to this internal curing. Holm et al. [4] reported anecdotal evidence of reduced plastic shrinkage cracking in LWA mixtures during high rise construction. While these observations have been made on the benefits of LWA for improved curing, it is only recently that the use of LWA has been specifically designed to improve the curing of concrete [5–8]. This development is due primarily to issues associated with increased observances of cracking in higher strength lower water to cement ratio concrete [9,10].

Internal curing has emerged over the last decade as a method to improve the performance of low water to cement ratio (w/c) mix-

tures [7,11,12]. Specifically, internal curing refers to the use of prewetted lightweight aggregate (or other water filled inclusions) that can provide curing water throughout the cross section of the concrete. This differs from conventional curing where water is provided after placement and where the water is applied only at the surface of the concrete. Internal curing was originally promoted to reduce autogenous shrinkage and autogenous shrinkage cracking [5-7,13-15]. However its potential benefits are numerous. Recent work has demonstrated benefits of internal curing for reducing drying shrinkage, drying shrinkage cracking [16,17], reducing the likelihood of thermal cracking [18,19], and improved plastic shrinkage cracking resistance [20]. Internal curing can also improve the freeze-thaw resistance, increase the resistance to fluid absorption [5,21,22] and reduce ion diffusion [23] in concrete. It is becoming increasingly clear that internal curing has great potential for the concrete industry to create a longer lasting more sustainable product; however several aspects of internal curing still require closer examination.

To fully understand how internal curing works we need to first realize that the hydration of cement paste causes a volume reduction which is known as chemical shrinkage [24,25]. While chemical shrinkage starts at the time the water comes in contact with the cement, it has a different impact on the system before and after the paste sets. Before set, the chemical shrinkage causes bulk shrinkage of the cement paste that is equal to the total external volume change. After set, however, the cement paste becomes stiff

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enough to resist a portion of the volume change caused by chemical shrinkage [26,27]. As a result, an under pressure develops in the pore fluid causing, vapor-filled pockets develop inside the cement paste [13,28]. In low *w/c* mixtures, these vapor filled cavities can result in substantial shrinkage since the vapor filled cavities form in relatively small pores with a small radius of curvature [29–31].

Lightweight aggregate can be used to supply water during the curing process to mitigate the effects of self-desiccation in low w/c concrete mixtures. LWA acts as a water reservoir that can provide water to replenish the empty pore volume that is created by the chemical shrinkage occurring during hydration. Since water is removed from large pores first, the ideal lightweight aggregate would have pore sizes larger that the cement paste. The design of concrete for internal curing requires that a sufficient amount of water is placed in the concrete to overcome the effects of self-desiccation. A straightforward approach has been developed to estimate this volume of internal curing water that is needed based on the concept that all the chemical shrinkage volume will be replaced by water [32] as shown in the following equation:

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

where  $M_{LWA}$  (kg/m³) 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,  $C_f(kg/m³)$  is the cement content of the mixture, CS (ml of water per g of cement) is the chemical shrinkage of the cement,  $\alpha_{max}$  (unitless) is the expected maximum degree of hydration (0–1),  $\phi_{LWA}$  (kg of water/kg of dry LWA) is the absorption capacity of the LWA (taken here as the 24 h absorption value), and S (unitless) is the expected degree of saturation of the LWA expressed as a function of the taken absorption value,  $\phi_{LWA}$ .

Eq. (1) is a simple, straightforward easy to use approach for proportioning mixtures. It should be noticed that since the volume chemical shrinkage is dependent on the cement composition and extent of hydration and not the water to cement ratio [33,34], a large value of internal curing is also predicted for high water cement ratio mixtures. Other equations have been proposed for proportioning internally cured mixtures since it is known that the magnitude of autogenous shrinkage decreases as the water to cement ratio increases. These approaches suggest that rather than providing water to fill the entire volume created by chemical shrinkage, water only needs to be provided that replace water lost from the smallest pores. For example, Jensen and Hansen [6,13] developed a mixture design approach that reduces the amount of internal curing water to be provided to only supply the water that is necessary for cement hydration. The design approach may also be extended to account for water lost during evaporation [35] or for higher water to cement ratio mixtures [34] for purposes other than minimizing autogenous shrinkage. Irrespective of the design approach and equation used for proportioning internally cured concrete mixtures, the aggregate properties required for mixture proportioning are the same (i.e., specific gravity, absorption, and saturation factor). This work attempts to provide information on these properties for a range of North American LWA. This paper reports the results of tests that can provide a database for the LWA as well as to provide an input for the proportioning.

It should also be mentioned that the volume of water is not the only critical factor for internal curing. The spatial distribution of this water is also important. For this reason fine lightweight aggregate is generally preferred for internal curing, as compared to coarse lightweight aggregate. This is due to the improvement in the distribution of internal curing water throughout the matrix [5,36,37].

#### 1.2. Background on lightweight aggregate porosity

For over a century [38] it has been known that certain clays, slates, and shales can bloat when they are heated due to a rapid expansion of gas in the material. This expanding gas becomes porosity in the aggregate. While this porosity has historically been seen as beneficial due to the reduced density of the aggregate, it is becoming increasingly clear that this porosity can be used to store moisture for use in internally cured concrete [7,12], or to store some other solutions as shrinkage reducing admixtures or corrosion inhibitors [39]. Quantifying this porosity is critical for determining the water that can be absorbed (and later released) by the lightweight aggregate. Water absorbed by the lightweight aggregate before set does not influence the porosity of the paste and therefore is not considered to be part of the 'water to cement ratio' of a given concrete mixture [5,31,40].

Landgren [41] examined the water absorption properties of lightweight aggregate, and used a 'surface dry' technique to determine that this places the aggregate in a condition where the majority of the "aggregate will neither contribute water to, nor absorb water from, a concrete mix".

Aleksander and Mindess [42] reported that typical absorptions for lightweight aggregates fall between 5% and 15%. Holm et al. [4] advocated that absorption versus time plots should be developed for each lightweight aggregate as LWA differ substantially from more conventional aggregate. Holm et al. [4] conducted absorption tests for one aggregate over two years observing that the rate of water absorption was unique to each aggregate depending on the pore size, continuity, and distribution. Holm et al. [4] reported 24 h absorption values between 5% and 25% of the mass of dry aggregate. Holm noted however that this 24 h value is only a portion of the total water that can be absorbed by the lightweight aggregate. Holm et al. [4] stated that since the pre-wetting of LWA provides water that is only a fraction of the theoretical saturation that "the use of the ASTM expression 'saturated surface dry (SSD)' is inappropriate for LWA, theoretically inaccurate and analytically misleading". The authors fully agree with this statement. and will use the term surface dry (SD), as used by Landgren [41] throughout this paper as this is commonly used in practice to refer to the water absorbed by LWA at 24 h.

Several researchers have investigated the desorption properties of lightweight aggregate. Landgren [43] performed a landmark study that examined the water absorption-desorption characteristics of coarse lightweight aggregate used in the US. This information was used to determine the internal surface area of the lightweight aggregate as well as the susceptibility of these aggregates to fire or freeze-thaw damage. Bentz et al. [32] and Radlinska et al. [31] measured desorption isotherms on a limited number of aggregates using salt solutions to quantify the amount of water that may be released as the concrete experiences self-dessication. This work expands these measurements to a wider range of aggregates (representing a majority of the LWA used in North America) and examines methods to normalize the data.

The effective design of internally cured concrete requires an understanding of the distribution of LWA in the mixture, the time dependent absorption properties, and the desorption properties of the lightweight aggregate. This paper provides a series of measurements on numerous expanded shale, slate and clay fine aggregates commonly used throughout the US for application in internal curing. Results of these tests provide guidance on the values that can be used in Eq. (1). General trends are drawn from this data and provide guidance for developing more detailed proportioning techniques for internally cured concrete. Of special interest is the relationship between absorption and desorption and the time dependent absorption.

# 2. Experimental program

Fifteen fine LWA were selected to represent aggregates commonly used in North America as well as a variety of raw materials. Table 1 provides a listing of the materials that were selected for this study, including the naming convention of the lightweight aggregate, the geographic location of the plant, and the type of raw material used to make the lightweight aggregate. Table 2 presents a sieve analysis performed on these aggregates as received.

# 3. Experimental methods

# 3.1. Determining the surface dry condition

Determining the absorption of the aggregate can be problematic due to difficulties in determining the surface dry (SD) condition for lightweight aggregate. This research discusses three different techniques to evaluate SD. Additional information can be found on Henkefsiefken [44] and Castro et al. [45]. The techniques included the standard cone, the paper towel method, and the use of cobalt chloride.

It should be noted that strictly speaking ASTM C128-07 method [46] is not intended to be used for determining the absorption of LWA. That said 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

**Table 1**List of Lightweight Aggregates (LWA) used in this research.

LWA #	Raw material	LWA name	Plant location
1	Clay	Gravelite	Erwinville, Louisiana
2	Clay	Liapor*	Germany
3	Clay	Livlite	Livingston, Alabama
4	Clay	TXI Frazier Park	Frazier Park, California
5	Shale	Buildex Marquette	Marquette, Kansas
6	Shale	<b>Buildex New Market</b>	New Market, Missouri
7	Shale	Haydite AX	Brooklyn, Indiana
8	Shale	Haydite DiGeronimo	Cleveland, Ohio
9	Shale	Hydrocure	Brooks, Kentucky
10	Shale	Norlite	Albany, New York
11	Shale	TXI Boulder	Boulder, Colorado
12	Shale	TXI Streetman	Streetman, Texas
13	Shale	Utelite	Coalville, Utah
14	Slate	Solite LLC	Buckingham, Virginia
15	Slate	Stalite	Gold Hill, North Carolina

<sup>\*</sup> Liapor is a European aggregate commonly used in research studies as such it is reported here to enable comparison.

**Table 2**Lightweight aggregate gradation shown as the cumulative percentage passing.

LWA #	Sieve #			Fineness			
	4 (%)	8 (%)	16 (%)	30 (%)	50 (%)	100 (%)	modulus
1	94.7	39.7	9.7	3.4	1.7	1.0	4.50
2	100.0	100.0	0.0	0.0	0.0	0.0	4.00
3	100.0	85.6	55.9	28.8	12.7	7.6	3.09
4	85.1	56.3	28.0	11.8	4.9	2.1	4.12
5	98.1	84.8	56.4	34.0	20.4	11.9	2.94
6	95.6	69.1	46.9	30.9	21.2	15.1	3.21
7	98.2	76.2	48.3	28.3	15.9	8.8	3.24
8	100.0	86.4	52.0	23.2	13.6	4.0	3.21
9	99.2	69.2	32.4	14.4	7.5	4.9	3.72
10	97.8	64.2	32.8	18.7	11.2	7.5	3.68
11	99.7	87.0	50.5	31.8	22.9	17.1	2.91
12	95.6	68.5	43.7	28.2	21.4	16.8	3.26
13	100.0	87.7	53.1	27.8	15.4	8.6	3.07
14	88.5	60.1	39.2	27.0	19.6	14.2	3.51
15	99.8	64.8	32.2	16.2	9.0	5.2	3.73

which time the water is decanted and the surface of the aggregate is dried. To determine the surface dry condition of the 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 test arises when angular aggregates that are typically manufactured and crushed are used. This angularity of the aggregate could lock the particles together such that when the cone is lifted, the shape is retained even after surface moisture is removed.

The paper towel method, based on a test procedure from the Department of Transportation for the State of New York [47], involves immersing the aggregate in water for 24 h after which time the water is decanted and the surface of the aggregate is patted dry. The paper towel method spreads the aggregate out and the paper towel is placed across the surface of the aggregates. This process is repeated at different moisture contents (preferable near the SD condition). Once it appears that the paper towel is no longer picking up moisture (as determined by the visual inspection for a change in color from the paper towel) from the aggregate, it is assumed that a surface dry condition has been reached and the aggregate moisture can be determined.

The cobalt chloride method after Kandhal and Lee [48] 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. This process is repeated at different moisture contents (preferably near the SD 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 [44]. 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.

Similar results (in the range of 1.5% absorption) were reported by Castro et al. [45] when these methods were applied to LWA #9. In that research, results from the cone test method provide slightly lower absorption than the paper towel method or the cobalt chloride method. However, it should be noted that the paper towel and the cobalt chloride method work on the same principle (surface moisture). This moisture will change the color of the cobalt or will wet the paper towel. In contrast, the cone test method is more related to friction, which is more susceptible to particle geometry, so it would not be unexpected that would provide a slightly lower absorption. Based on these results and by the simplicity of the test, the paper towel method was chosen to determine the SD condition of the LWA to measure the 24 h water absorption in this research. The absorption is reported as the average of three samples tested.

# 3.2. Absorption as a function of time

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 absorption of water by the lightweight aggregate takes place over time. To evaluate the absorption of

water as a function of time a "volumetric flask test" was conducted. Fig. 1 shows a picture of the setup.

The LWA was prepared by placing it in an oven at  $105 \pm 2$  °C for  $24 \pm 1$  h, and then cooled for 24 h. A  $100 \pm 10$  g of sample was placed in a 250 ml volumetric flask. De-ionized water was added to approximately 80% of capacity of the flask. The flask was then manually agitated for 2–3 min to eliminate entrapped air bubbles between the aggregate particles. Care was taken to keep the aggregate under water all the time. After agitation, the fine particles were allowed to settle to see the level of the fluid on the flask. Additional water was added to bring the water level in the flask to its calibrated capacity. The total mass of the flask, aggregate sample, and water was recorded five minutes after the water was first in contact with the aggregate.

As the aggregate continues absorbing water over time, the level of the fluid will decrease (Fig. 1). Additional water is then added to the flask to keep the fluid at its calibrated capacity at regular intervals (at about 10 min, 20 min, 30 min, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 24 h, 36 h, and 48 h), recording the total mass and the actual time of measurement. Each time before water was added, the flask was agitated for about 30 s to eliminate new air bubbles.

The described procedure does not capture the water absorption during the first five minutes after water is placed in contact with the aggregate. However assuming the absorption value at 24 h is the same as the 24 h absorption obtained by the paper towel method, enables the difference between both values to be used to determine the absorption that occurs during the first 5 min of this test.

Fig. 2 shows the rate at which the lightweight aggregates absorb water during the first 48 h starting from an oven-dry condition. This shows that the rate of water absorbed is higher during the first several hours. The total absorption increases over time.

Table 3 describes the water absorbed by the lightweight aggregate for four conditions: after 6 h, after 24 h, after 48 h and after vacuum. It can be noticed that the absorption at 24 h has a wide range for the aggregate tested (e.g., 6–31%). The expanded slates had a 24 h absorption between 6% and 12%, the expanded shale had a 24 h absorption between 10% and 20%, and the expanded clays had a 24 h absorption between 15% and 31%. The 6 h or 24 h absorption values are approximately 60% and 85% of the vacuum soaked materials, respectively.

It is important to note that the absorption characteristics of an aggregate can also depend on the aggregate size. Large lightweight aggregate have larger voids, and when the aggregate is crushed the large voids are commonly found to coincide with the fracture surface. As a result, these large pores effectively become part of the texture of the aggregate, and then they are no longer considered as internal porosity in the aggregate [36]. It also needs to be noted

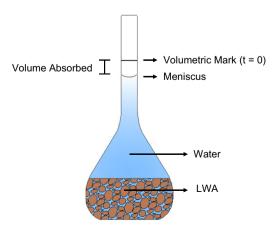


Fig. 1. Setup to measure LWA absorption as a function of time.

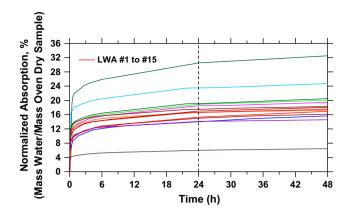


Fig. 2. Water absorption of dry LWA over time during the first 48 h.

**Table 3**Lightweight aggregate absorption at different times.

		•			
LWA #	Specific gravity (OD)	Water absorption at 6 h (%)	Water absorption at 24 h (%)	Water absorption at 48 h (%)	Water absorption after vacuum (%)
1	1.29	13.2	16.0	17.6	26.8
2	1.49	13.1	15.0	16.7	27.0
3	1.10	25.4	30.5	32.6	35.5
4	1.39	15.3	17.7	18.4	19.1
5	1.45	15.6	17.5	18.3	22.5
6	1.50	12.4	14.1	14.6	24.9
7	1.56	7.4	10.0	11.1	20.0
8	1.40	12.9	15.6	16.5	18.6
9	1.51	12.5	15.0	16.1	22.0
10	1.38	16.4	19.1	20.5	25.2
11	1.46	14.9	17.9	19.1	24.9
12	1.48	15.9	18.9	20.1	24.6
13	1.49	15.6	18.5	19.4	23.0
14	1.62	9.9	12.2	13.1	18.6
15	1.51	5.2	6.0	6.5	11.4

that larger un-crushed particles tend to have more porosity simply because they have expanded more than a smaller un-crushed particle. When the same aggregate is being tested using different size particles it is observed that the absorption of the smaller aggregate particle sizes is less than the absorption of larger particle sizes.

# 3.3. Specific gravity measurements

Specific gravity measurements were performed following the ASTM C128-07 standard test method, with the exception that SD condition was estimated using the paper towel method previously described. The second column of Table 3 shows the measured values of the specific gravity for the aggregates used in this research, expressed as in oven dry (OD) condition.

# 3.4. Desorption measurements

The loss of water from the LWA pores during drying can be described through a desorption isotherm (a plot of mass loss as a function of relative humidity at a constant temperature). A vapor sorption analyzer was used to enable the sorption behavior to be evaluated under carefully controlled temperature and humidity conditions.

The LWA was prepared by placing it in an oven at  $105 \pm 2$  °C for  $24 \pm 1$  h, cooled for 24 h and placed in de-ionized water for  $24 \pm 1$  h prior to testing. Once the prewetting of the aggregate was complete the LWA was removed from water and "patted to SD" using

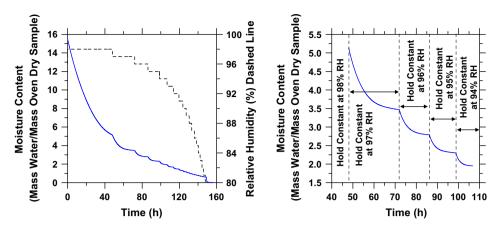


Fig. 3. A typical mass change versus time plot for LWA prewetted with de-ionized water.

absorbent towels. A 40–50 mg sample of aggregate passing sieve #16 and retained on the sieve #30 was placed in a tared 180 mL quartz pan. This particle size was optimized for use in the testing device, and the same aggregate size was tested for all the aggregates. The pan containing the sample was then suspended from the balance ( $\pm 0.001$  mg accuracy) and placed in the relative humidity chamber to equilibrate at  $23.0 \pm 0.05$  °C and  $98 \pm 0.1\%$  RH for up to 48 h or until the sample had achieved a stable mass (less than an 0.001% mass change/15 min). 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 min or 12 h of exposure, unless noted otherwise). After equilibrating at 80% RH, the samples were dried to 0% RH at  $23 \pm 0.05$  °C for up to 48 h or until the sample had achieved a stable mass.

Fig. 3 shows a plot of a typical mass change as a function of time  $(23.0 \pm 0.1 \, ^{\circ}\text{C})$  for a LWA pre-wetted in de-ionized water). As the relative humidity is changed, the sample undergoes a rapid change in mass. The mass change decreases as the sample approaches equilibrium. It can be seen that this general behavior is observed at each change in the relative humidity, however, the magnitude of the mass change is different at each relative humidity and would be consistent with the volume of pores from which water is being lost at each step.

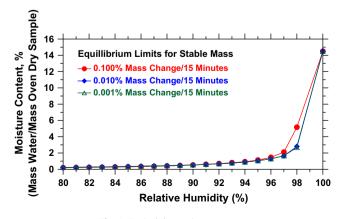
Fig. 4 shows a typical desorption plot created using the data shown in Fig. 3. One of the points that needs to be evaluated in conducting this test is the mass-loss criterion that is used, to assume that a sample has achieved 'equilibrium'. Three different weight loss criteria were evaluated consisting of less than a 0.1%,

0.01%, and 0.001% mass loss over a fifteen minute time interval. The results using the finest criterion were very similar to the 0.01% case criterion with the exception of the 98% RH test point which is approximately 5% lower. The remainder of the tests used the 0.01% mass loss/15 min criteria.

It should be noted that unlike many desorption studies, Fig. 4 is not normalized to the weight of the SD sample but rather it is normalized to the weight of the 0% RH (dry) sample. This was done since it is believed that this value is more easily reproduced and is more useful for mixture proportioning purposes.

Desirable aggregates for use as an internal curing agent are porous so that they hold water. However these aggregates will also need to release the water that is held in the pores at high relative humidities. Fig. 5 illustrates an example of what can be defined as an efficient and less efficient desorption behavior from the internal curing perspective. An efficient aggregate will release the majority of water at high relative humidities (i.e., 93% RH), implying this amount of water is available for reaction with the cement. In contrast, a less efficient internal curing agent (relative to the 15 aggregates considered on this paper) will keep a significant proportion of the absorbed water at lower relative humidities. As such, this water that is not released is unavailable for curing the material.

The measured desorption behavior for the 15 aggregates studied in this research are shown in Fig. 6. It can be noted that all of the aggregates have shown what can be considered a good desorption behavior. These tests were performed using water as the absorbed solution. Information of desorption behavior for other solutions can be found on Castro et al. [49].



**Fig. 4.** Typical desorption response.

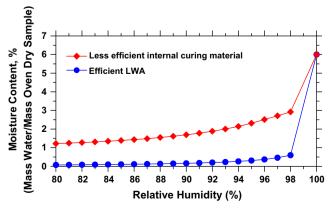


Fig. 5. Example of a good and a bad desorption behavior.

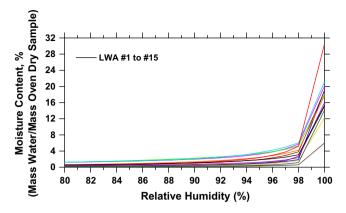


Fig. 6. Measured desorption response for the 15 LWA included in this study.

It should be noted that other methods may be able to be used to improve the resolution of the measurement above 98% RH. For example a pressure plate method [50] shows promise to be used to provide information at high relative humidities.

# 4. Discussion

The absorption of the aggregate at 6 h, 24 h, 48 h, and under vacuum in Table 3 indicates variability in the aggregate pore structure. These results can however be viewed in a slightly different manner if they are normalized as shown in Fig. 7. The y axis can be thought of as the S value from Eq. (1) if the absorption in Eq. (1) is given in terms the 24 h absorption (the most commonly used value in the industry). It is notable that all of the expanded shales, slates, and clays tested demonstrate a similar behavior. As such it may be possible to describe the S factor for Eq. (1) reasonably, using equation:

$$S = t^A \tag{2}$$

where "A" is a fitted constant and "t" represents time (days). In this paper this was fit and is only applicable for the first 48 h. These fitted values and the average value are listed in Table 4. An average value of "A" (A = 0.10) can be used for a general description of the absorption behavior of the entire class of expanded clay, shale and slate, during the first 24 h.

This indicates that when the aggregate is not wetted for at least 24 h, less internal curing water is provided to the mixture. To have a similar volume of curing water in a concrete mixture when the aggregate has not been saturated as long, more LWA would need to be used. It should be noted however that other factors such as water being "pushed" into the aggregate during pumping and var-

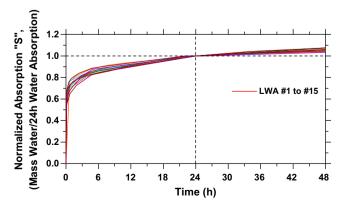


Fig. 7. Time dependent water absorption, normalized by the 24 h water absorption.

**Table 4**Fitted parameter for absorption and desorption curves.

LWA #	Α	D1	D2	D3
1	0.1240	0.0022	0.6230	0.0473
2	0.1320	0.0017	0.6193	0.0314
3	0.1070	0.0029	0.6215	0.0168
4	0.0772	0.0029	0.6190	0.0511
5	0.0726	0.0025	0.6231	0.0312
6	0.0919	0.0010	0.6149	0.0036
7	0.1120	0.0015	0.6243	0.0008
8	0.1171	0.0021	0.6016	0.0005
9	0.1211	0.0022	0.6204	0.0052
10	0.0977	0.0015	0.6223	0.0151
11	0.0878	0.0031	0.6243	0.0712
12	0.1016	0.0026	0.6179	0.0962
13	0.1105	0.0032	0.6215	0.0799
14	0.0879	0.0017	0.6190	0.0061
15	0.0900	0.0012	0.6202	0.0127
Max.	0.1320	0.0032	0.6243	0.0962
Min.	0.0726	0.0010	0.6016	0.0005
Average	0.1020	0.0022	0.6195	0.0313

iation from batch to batch may need to be considered for application in practice [35,51].

Similarly the moisture desorption isotherm from the 15 LWA can be normalized using the 24 h absorption as shown in Fig. 8. It is again notable that all of the expanded shales, slates and clays tested demonstrated a similar behavior.

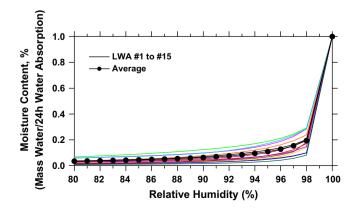
Eq. (3) provides a normalized expression that can be used to describe the desorption response as a function of the relative humidity. The first part of Eq. (3) is a form that is consistent with a BET Type II isotherm, while the second term was added to improve the fit.

$$Description = \frac{D_1 R H}{(1 - R H)(1 + (D_2 - 1) R H)} + D_3 R H^2 \eqno(3)$$

where RH is given from 0 to 1 and  $D_1$  to  $D_3$  are fitted constants. Fitted parameters for all 15 LWA together with statistical parameters can be found on Table 4.

Fig. 9 is presented as an example of how the Eq. (3) fit the experimental data. Fig. 9 shows experimental desorption data from LWA #5 over a wide range of relative humidity (0–98% RH) and its fitted curve using Eq. (3). A logarithmical scale is used for Fig. 9 since if is not the behavior at the lower RH values would be difficult to see.

From Fig. 8 it is possible to determine the proportion of water that will be released from the pores of the LWA at different relative humidities. It can be noticed that all the aggregates release a sub-



 $\begin{tabular}{ll} \textbf{Fig. 8.} Desorption curve on $24$ h saturated LWA, normalized by the $24$ h water absorption. \\ \end{tabular}$ 

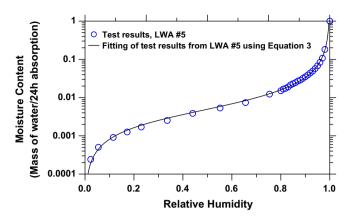


Fig. 9. Fitting of test results using Eq. (3).

stantial portion of water (86–98%) at 93% RH. Table 5 shows the amount of water that has been released from the LWA at 93% RH ( $\psi$ ). It can be noted that desorption results at this RH present a low of variation (COV of 4.5%). For North American expanded clay, slate and shale an average value of  $\psi$  (i.e.,  $\psi$  = 0.93) can be used to provide a general description of the desorption behavior of the aggregate.

As a result, Eq. (1) can be rewritten to describe the role of the aggregate more completely as is presented in the following equation:

$$M_{LWA} = \frac{C_f \times CS \times \alpha_{\text{max}}}{t^A \times \phi_{LWA24h} \times \psi}$$
 (4)

where " $t^{A}$ " is the absorption of the LWA as a function of time relative to its 24 h absorption value, and  $\psi$  is the fraction of water released from the LWA at high relative humidity. The modification of this equation enables mixtures to be proportioned using aggregates with optimal desorption as well as less than ideal desorption (i.e., more LWA is needed in this case).

# 5. Conclusions

This paper describes the time dependent isothermal water absorption and desorption response of commercially available fine

Table 5
Amount of water released from LWA pores at 93% RH.

	•		
LWA #	Water released at 93% RH $\psi$ (mass of water/mass oven dry sample)		
1	0.906		
2	0.936		
3	0.922		
4	0.887		
5	0.919		
6	0.976		
7	0.969		
8	0.958		
9	0.951		
10	0.955		
11	0.869		
12	0.853		
13	0.862		
14	0.960		
15	0.962		
Average	0.926		
COV	4.5%		
Min.	0.853		
Max.	0.976		

lightweight aggregates (LWA). Information is provided about the aggregates that can be used for proportioning internally cured concrete made with fine LWA.

The typical 24 h absorption of different types of fine lightweight aggregates (using a full gradation) was observed to vary between 6% and 31%. The expanded slates has a 24 h absorption between 6% and 12%, the expanded shales had a 24 h absorption between 10% and 20%, and the expanded clays had a 24 h absorption between 15% and 31%. The 6 h or 24 h absorption values are approximately 60% and 85% of the vacuum saturated materials, respectively.

The porosity and absorption decrease as the size fraction of the aggregate decreases due to the crushing process and because larger un-crushed particles tend to have more porosity because they have expanded more than a smaller un-crushed particle.

The time-dependent absorption of LWA was normalized by the 24 h absorption. This normalized absorption showed that different expanded shales, clays and slates fall into a relatively uniform response band.

Desorption tests were performed on aggregates of a single size fraction. The expanded shales, clays, and slates aggregates tested in this study desorbed between 85% and 98% of their 24 h absorbed water at 93% relative humidity. The desorption response of different aggregates from expanded shales, clays and slates falls into a relatively uniform band of behavior across a wide range of raw materials. The normalized desorption results from the single size fraction compare well to tests using a salt solution with aggregates with the full gradation.

The average behavior may be useful for revising the time dependent absorption and desorption values used in the mixture proportioning equations like Eq. (1). For example, the expected degree of saturation "S" can be replaced with a time dependent function "t4", where t is measured in days and "A" can be assumed as 0.10. Further the 24 h absorption should be multiplied by 0.93 to account for the portion of water that is released from the LWA at high relative humidity.

While the actual LWA produced can vary slightly in terms of absorption and gradation, the results of this study show that several normalized properties of commercially produced LWA follows a consistent trend (in time dependent absorption and desorption). This suggests that a generalized behavior for fine expanded shale, slate, and clay LWA can be considered. This would enable mixture proportioning to be done for example using a response curve that reflects the entire class of aggregates rather than one specific aggregate.

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## References

- Klieger P. Early high strength concrete for prestressing. In: Proceeding: world conference on prestressed concrete. San Francisco, CA; 1957.
- [2] Bloem DL. Concrete strength measurements cores and cylinders. In: ASTM proceedings. vol. 65. Philadelphia, PA; 1965.
- [3] Philleo R. Concrete science and reality. In: Skalny JP, Mindess S, editors. Materials science of concrete II. Westerville OH: American Ceramic Society; 1991.
- [4] Holm TA, Ooi OS, Bremner TW. Moisture dynamics in lightweight aggregate and concrete. Expanded Shale Clay and Slate Institute, Publication # 9340; 2004, p. 12.

- [5] Bentz DP, Snyder KA. Protected paste volume in concrete: extension to internal curing using saturated lightweight fine aggregate. Cem Concr Res 1999;29(11):1863-7.
- [6] Jensen OM, Hansen PF. Water-entrained cement-based materials: I. Principles and theoretical background. Cem Concr Res 2001;31(4):647–54.
- [7] RILEM Report 41. Internal curing of concrete state of the art. In: Kovler K, Jensen OM. editors. RILEM Publications S.A.R.L; 2007. p. 161.
- [8] Schlitter J. New methods to quantify the cracking performance of cementitious systems made with internal curing. Master Thesis. Purdue University; 2010.
- [9] Shah SP, Weiss W, Yang W. Shrinkage cracking can it be prevented? Concr Int 1998;20(4):51–5.
- [10] RILEM Early age cracking in cementitious systems. In: Bentur A, editor. Report of RILEM technical committee 181-EAS; 2003. p. 350.
- [11] ACI SP-256. Internal curing of high performance concrete: laboratory and field experiences. American Concrete Institute: Farmington Hills, MI; 2008. p. 110.
- [12] ACI Report on early-age cracking: causes, measurement and mitigation. American Concrete Institute – Committee 231: Farmington Hills, MI; 2010. p. 48.
- [13] Jensen OM, Hansen PF. Autogenous deformation and rh-change in perspective. Cem Concr Res 2001;31(12):1859-65.
- [14] Jensen OM. Autogenous phenomena in cement-based materials. PhD thesis. Aalborg University; 2005.
- [15] Lopez M, Kahn LF, Kurtis KE. Characterization of elastic and time-dependent deformations in high performance lightweight concrete by image analysis. Cem Concr Res 2009;39(7):610–9.
- [16] Henkensiefken R, Nantung T, Weiss WJ. Reducing restrained shrinkage cracking in concrete: examining the behavior of self-curing concrete made using different volumes of saturated lightweight aggregate. In: National concrete bridge conference. St. Louis, MO; 2008.
- [17] Henkensiefken R, Bentz D, Nantung T, Weiss J. Volume change and cracking in internally cured mixtures made with saturated lightweight aggregate under sealed and unsealed conditions. Cem Concr Compos 2009;31(7):427–37.
- [18] Schlitter J, Henkensiefken R, Castro J, Raoufi K, Weiss J, Nantung T. Development of internally cured concrete for increase service life. JTRP Report SPR-3211; 2010. p. 252.
- [19] Byard BE, Schindler AK, Barnes RW. Cracking tendency of lightweight concrete in bridge deck applications. In: Proceedings of the concrete bridge conference. Phoenix, Arizona; 2010.
- [20] Henkensiefken R, Briatka P, Nantung T, Bentz D, Weiss J. Plastic shrinkage cracking in internally cured mixtures made with pre-wetted lightweight aggregate. Concr Int 2010;32(2):49–54.
- [21] Henkensiefken R, Castro J, Bentz D, Nantung T, Weiss J. Water absorption in internally cured mortar made with water-filled lightweight aggregate. Cem Concr Res 2009;39(10):883–92.
- [22] Peled A, Castro J, Weiss J. Atomic force microscopy examinations of mortar made by using water-filled lightweight aggregates. Transport Res Rec 2010;2141:92–101.
- [23] Bentz DP. Influence of internal curing using lightweight aggregates on interfacial transition zone percolation and chloride ingress in mortars. Cem Concr Compos 2009;31(5):285–9.
- [24] Powers T. Absorption of water by Portland cement paste during the hardening process. Ind Eng Chem 1935;27(7):790–4.
- [25] L'Hermite RG. Volume changes of concrete. In: 4th International symposium on the chemistry of cement. Washington DC; 1960.
- [26] Barcelo L, Boivin S, Rigaud S, Acker P, Clauvaud B. Linear vs volumetric autogenous shrinkage measurements: material behavior or experimental artefact. In: Persson B, Fagerlund G, editors. Proceedings of the second international seminar on self-desiccation and its importance in concrete technology. Lund University, Sweden; 1999 p. 109–25.
- [27] Sant G, Lura P, Weiss J. Measurement of volume change in cementitious materials at early ages: review of testing protocols and interpretation of results. Transport Res Rec 2006;1979:21–9.
- [28] Couch J, Lura P, Jensen O, Weiss J. Use of acoustic emission to detect cavitation and solidification (time zero) in cement pastes. In: Jensen O, Lura P, Kovler K, editors. International RILEM conference on volume changes of hardening concrete: testing and mitigation. Denmark, 2006. p. 393–400.
- [29] Bentz DP, Jensen OM. Mitigation strategies for autogenous shrinkage cracking. Cem Concr Compos 2006;26(6):677–85.

- [30] Lura P, Couch J, Jensen OM, Weiss J. Early age acoustic emission measurement in hydrating cement paste: evidence for cavitation during solidification due to self-desiccation. Cem Concr Res 2009;39(10):861–7.
- [31] Radlinska A, Rajabipour F, Bucher B, Henkensiefken R, Sant G, Weiss J. Shrinkage mitigation strategies in cementitious systems: a closer look at sealed and unsealed material behavior. Transport Res Rec 2008;2070:58–67.
- [32] Bentz DP, Lura P, Roberts JW. Mixture proportioning for internal curing. Concr Int 2005;27(2):35–40.
- [33] Geiker M. Studies of Portland cement hydration by measuring of chemical shrinkage and a systematic evaluation of hydration curves by means of the dispersion model. PhD thesis. Technical University of Denmark; 1983.
- [34] Castro J. Moisture transport in cement based materials: application to transport tests and internal curing. Ph.D. thesis. Purdue University; 2011.
- [35] Golias M. Novel techniques to improve concrete performance: the use of soy methyl ester and lightweight aggregate. Master Thesis. Purdue University; 2010.
- [36] Henkensiefken R, Sant G, Nantung T, Weiss J. Comments on the shrinkage of paste in mortar containing saturated lightweight aggregate. In: International RILEM symposium on concrete modelling – CONMOD'08. Delft, The Netherlands; 2008. p. 707–14.
- [37] Suzuki M, Meddah M, Sato R, Kawabata T. Long term shrinkage and stress in ultra high strength concrete using porous ceramic waste for internal curing. In: Jensen OM, Hasholt MT, Laustsen S, editors. Proceedings of the international RILEM conference on use of superabsorbent polymers and other new additives in concrete. Lyngby, Denmark; 2010. p. 263–72.
- [38] ESCSI. Lightweight concrete history, applications and economics. Expanded Shale Clay and Slate Institute. National Press Building: Washington, DC; October 1971.
- [39] Bentz DP. Capitalizing on self-desiccation for autogenous distribution of chemical admixtures. In: Self-desiccation and its importance in concrete technology proceedings of the 4th international research seminar in Lund; June 2005. p. 189–96.
- [40] Henkensiefken R, Nantung T, Weiss J. Saturated lightweight aggregate for internal curing in low w/c mixtures: monitoring water movement using X-ray absorption. Inter J Strain; in press, doi: 10.1111/j.1475-1305.2009.00626.x.
- [41] Landgren R. Water-vapor adsorption-desorption characteristics of selected lightweight concrete aggregates. Portland Cement Association, Research and Development Laboratories, Bulletin 178: Skokie, Illinois; 1964.
- [42] Alexander M, Mindess S. Aggregates in concrete. New York: Taylor and Francis; 2005. p. 439.
- [43] Landgren R. Determining the water absorption of coarse lightweight aggregate for concrete. Portland Cement Association, Research and Development Laboratories, Bulletin 179: Skokie, Illinois; 1964.
- [44] Henkensiefken R. Internal curing in cementitious systems made using saturated lightweight aggregate. Master thesis. Purdue University; 2009.
- [45] Castro J, Henkensiefken R, Bentz D, Nantung T, Weiss J. LWA absorption and desorption: the influence on the microstructure and transport properties of internally cured mortars. In: Sixth international conference on concrete under severe conditions, environment and loading, CONSEC 10. Mexico; 2010. p. 1543-50
- [46] ASTM international, ASTM C128. Standard test method for density, relative density (specific gravity), and absorption of fine aggregate: 2007.
- [47] NYDOT. Test method No.: NY 703-19 E: moisture content of lightweight fine aggregate. New York State Department of Transportation, Materials Bureau: Albany, NY; August 2008.
- [48] Kandhal PS, Lee DY. An evaluation of the bulk specific gravity for granular materials. Highway Research Record 307, HRB, National Research Council: Washington, DC; 1970. p. 44–55.
- [49] Castro J, Lura P, Rajabipour F, Henkensiefken R, Weiss J. Internal curing: discussion of the role of pore solution on relative humidity measurements and desorption of lightweight aggregate, ACI SP-270. Advances in the material science of concrete. Chicago, IL: American Concrete Institute; 2010. p. 89–100.
- [50] Johansson P. Water absorption in a two-layer masonry system properties, profile and predictions. PhD thesis. Lund University; 2005.
- [51] Villarreal VH. Internal curing real world ready mix production and applications: a practical approach to lightweight modified concrete, ACI SP-256. Internal curing of high performance concrete: laboratory and field experiences. Farmington Hills, MI: American Concrete Institute; 2008. p. 45–56.