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Effect of sample conditioning on the water absorption of concrete

Javier Castro^{a,*}, Dale Bentz^{b,1}, Jason Weiss^{c,2}^a Pontificia Universidad Catolica de Chile, School of Engineering, Casilla 306, Correo 22, Santiago, Chile^b National Institute of Standards and Technology, Materials and Construction Research Division, Engineering Laboratory, 100 Bureau Drive, Stop 7313, Gaithersburg, MD 20899-7313, USA^c School of Civil Engineering, Purdue University, 550 Stadium Mall, West Lafayette, IN 47907-2051, USA

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ABSTRACT

ASTM C1585 is commonly used to determine the absorption and rate of absorption of water in unsaturated hydraulic cement concretes. Unfortunately, a wide range of relative humidities can exist in the samples after this relatively short conditioning period and such variation may considerably influence the test results. Three main variables were studied in this program: the water to cement ratio, the paste volume fraction, and the effect of sample conditioning. The results confirm that water absorption testing is considerably influenced by sample preparation. Samples conditioned at 50% relative humidity can show up to six times greater total absorption than similar samples conditioned at 80% relative humidity. Samples that were conditioned in the oven at 105 °C do not appear to follow a similar trend when compared with specimens conditioned in chambers at lower temperatures for a longer duration. The absorption is also influenced by the volume of paste in the samples. The experiments show that a lack of control on moisture content or lack of consideration of the material composition may lead to a misunderstanding of the actual absorption behavior.

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1. Introduction

The durability of concrete subjected to aggressive environments depends largely on transport properties, which are influenced by the pore system [1–7]. Three main mechanisms govern transport in cementitious systems: permeability, diffusion and absorption. Permeability is the measure of the flow of fluids under a pressure gradient, while diffusion is the movement of ions due to a concentration gradient. Absorption can be described as the ability to take in water by means of capillary suction. All three mechanisms are heavily influenced by the volume of pores as well as the connectivity of the pore network. A large fraction of concrete in service is only partly saturated and the initial ingress of water and dissolved salts is influenced, at least in part, by capillary absorption [7]. As such, water absorption has been used as an important factor for quantifying the durability of cementitious systems [4–11]. Water absorption is the primary focus of this study since it is being increasingly used by specifiers and in forensic studies to provide a parameter that can describe an aspect of concrete durability. It is also important that these properties be adequately described for use in service life models [6,11].

1.1. Water absorption test

ASTM C1585 [1] is commonly used to determine the absorption and rate of absorption (commonly referred to as sorptivity) of water in unsaturated hydraulic cement concretes. This test method, based on work reviewed by Hall [12], consists of preconditioning cylindrical samples (2" (51 mm) in thickness and 4" (102 mm) in diameter) to a known moisture content, then exposing the bottom surface of the sample to liquid water and measuring the increase in mass resulting from water absorption. According to the standard conditioning procedure, samples are conditioned for 18 days. This conditioning period begins by first placing the sample in a 50 °C and 80% relative humidity (RH) environment for 3 days. The samples are then removed from this environment and placed in individually sealed containers where they remain for a minimum 15 days at 23 °C, to allow internal moisture to redistribute throughout the specimens before the test begins.

The absorption test involves recording incremental mass change measurements at relatively frequent intervals during the first 6 h after the sample comes in contact with water and subsequently taking one measurement every day for the next 8 days. The amount of absorbed water is normalized by the cross-section area of the specimen exposed to the fluid using Eq. (1):

$$i = \frac{m_t}{(a \cdot \rho)} \quad (1)$$

* Corresponding author. Tel.: +56 2 354 4245.

E-mail addresses: jecastro@ing.puc.cl (J. Castro), dale.bentz@nist.gov (D. Bentz), wjweiss@purdue.edu (J. Weiss).¹ Tel.: +1 301 975 5865.² Tel.: +1 765 494 2215.

where i is the normalized absorbed fluid volume, m_t is the change in specimen mass at time t ; a is the area of the specimen exposed to the fluid (i.e., that of the bottom face), and ρ is the density of the absorbed fluid (taken to be 1000 kg/m^3 at 23°C for water).

These absorbed fluid volumes are then plotted as a function of the square root of time. The initial sorptivity is determined as the slope of the curve during the first 6 h, while secondary sorptivity is determined using the slope of the same measurements between 1 and 8 days, as outlined in ASTM C1585 [1]. It should be noted that these times work well for water though they may not work as well for other fluids with different surface tension and/or viscosity [13].

The initial and secondary sorptivities can be used to evaluate the connectivity of the pore network [9]. Additionally, the secondary sorptivity, combined with exposure conditions, has been used for performing service life predictions [11].

1.2. The role of the relative humidity

Water ingress in unsaturated concrete is dominated by capillary suction upon initial contact with water [7,12–18]. Capillary absorption can be related to the volume of the pores and pore size distribution, as well as the size (i.e. radius) of the partially empty capillary pores (Fig. 1a). The relation between the equilibrated relative humidity and the radius of the smallest empty pore is given by the Kelvin-Laplace equation (Eq. (2)).

$$\ln(RH) = \frac{2\sigma V_m}{r_m RT} \quad (2)$$

where: RH is the relative humidity, σ is the surface tension of water (pore solution), V_m is the molar volume of water, r_m is the average radius of curvature, R is the universal gas constant, and T is the absolute temperature.

It should be noted that this expression is simplified as it does not consider the effect of water that is absorbed on the walls of the pores. Largely the concrete community has considered two sizes of pores as introduced by Powers [19]. The gel pores are considered to be small pores (<10 nm diameter) that are a part of the hydration products. Capillary pores are larger pores that occur due to excess water. Capillary porosity is particularly of concern in transport, as is the interconnectivity of the capillary pores.

Fig. 1b shows a conceptual illustration based on Powers [19] that uses a desorption isotherm to illustrate the volume of water located in the different size pores at different relative humidities.

The relative humidity used to condition the sample prior to the sorption test can have a significant impact on the results [1]. Previous test results by Parrot [20,21] indicated that the

water absorption rate was very sensitive to the moisture content of the concrete, particularly at relative humidities above 60% which were common for field exposure. Water leaves the largest accessible pores first. It can be seen from Fig. 1b that capillary pores occupy the range of humidity from approximately 80% to 100% RH. As such, initially upon drying water leaves the capillary pores. The lower the relative humidity, the greater the total volume of pores that are empty and available to be filled with water during the sorption test. Further, the lower humidity will empty smaller pores, creating a higher suction force in the materials and resulting in a greater sorption rate and a larger overall total absorption.

According to ASTM C1585, the standardized test conditioning will generally provide an internal relative humidity similar to relative humidities found near the surface in some field concrete structures [1,22,23]. This range of relative humidities can represent what is found in samples in the field; however, it is wide enough to considerably affect the test results.

Castro et al. [24] shows that the relative humidity of samples that were kept in the field under different exposure conditions was in the range of 80–100% depending on the type of exposure, which is somewhat higher than what is mentioned in ASTM C1585.

1.3. Research objectives

The objectives of this research are threefold. First, this research will examine the influence of conditioning relative humidity (oven dry, 50%, 65% and 80% RH) on the results of sorption tests performed on mortars with different w/c, containing a fixed volume of aggregate. Second, this research will examine the influence of the volume of aggregate (or equivalently the paste content) on the results of sorption testing. Third, this research will examine the effect of the conditioning method specified in ASTM C1585-04.

2. Materials

An ASTM C150 Type I ordinary portland cement (OPC) was used in this study, with a Blaine fineness of $370 \text{ m}^2/\text{kg}$ 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.

A polycarboxylate-based high-range water-reducing admixture (HRWRA) was added in varying rates as indicated in Table 1, depending on the mixture proportions, to maintain similar consistencies (i.e., workability). The sand used was natural river sand with a fineness modulus of 2.71, an apparent specific gravity of 2.58, and a water absorption of 1.8% by mass.

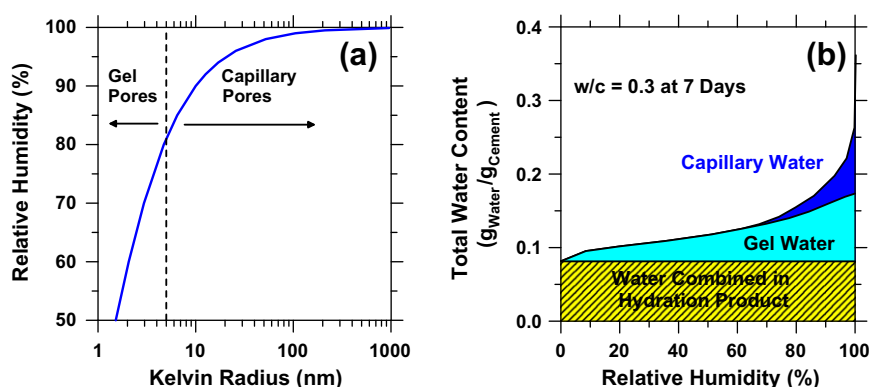


Fig. 1. Relation between relative humidity and partially empty pores in cement paste.

Table 1

Mixture proportions in saturated surface dry (SSD) conditions.

Material	55/0.35	55/0.40	55/0.45	55/0.50	45/0.50	35/0.50
Volume fraction of aggregate	55%	55%	55%	55%	45%	35%
w/c	0.35	0.40	0.45	0.50	0.50	0.50
Cement (kg/m ³)	673	626	585	549	671	793
Water (kg/m ³)	235	250	263	275	336	397
Fine aggregate (kg/m ³), SSD	1442	1442	1442	1442	1180	918
HRWRA (g/100 g cement)	0.60	0.40	0.20	0.00	0.00	0.00

2.1. Mixture proportioning

Six different mixtures were prepared in total. Four of the mixtures were mortars with a single volume fraction of fine aggregate (55% of the total volume) and different w/c (0.35, 0.40, 0.45, and 0.50). These mixtures were designated as 55/0.35, 55/0.40, 55/0.45 and 55/0.50, with the number on the left representing the volume fraction of fine aggregate and the number on the right representing w/c. Additionally, two other mortars were prepared with w/c of 0.50, but with different volume fractions of fine aggregate (35% and 45% of the total volume). They were designated as 35/0.50, 45/0.50. A list of the mixture proportions can be found in Table 1.

2.2. Mixing procedure

The mixing procedure used for the mortar was in accordance with ASTM C192-06 [25]. The aggregate was oven dried and cooled for 24 h before mixing. The volume of water was corrected by the absorption of the aggregate. The water and cement were conditioned for 24 h at room temperature prior to mixing.

3. Experimental method

Six 100 mm × 200 mm cylinders were cast for each mixture. After 1 day of curing, the samples were demolded and then sealed in double plastic bags for sealed curing. Bags were stored in a room at $23 \pm 1^\circ\text{C}$ until samples reached an age of 28 d. After that, cylinders were removed from bags and three 50 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw using water as the cooling fluid.

After cutting, samples were conditioned by placing them in environmental chambers at $23 \pm 0.5^\circ\text{C}$. Specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were placed in environmental chambers at three different relative humidities ($50 \pm 1\%$, $65 \pm 1\%$ and $80 \pm 1\%$). Specimens from mixtures 35/0.50 and 45/0.50 were placed in an environmental chamber at $50 \pm 1\%$ relative humidity. Samples were kept in the environmental chamber until they reached mass equilibrium, defined as a mass change of less than 0.02% over a 15 day period. Mixture 55/0.35 placed at $50 \pm 1\%$ relative humidity required the longest period of time (14 months) to reach mass equilibrium. However, all samples were maintained in the chambers for 14 months to test them all at the same age.

Additional specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were placed at $50 \pm 1\%$ RH. After the 14 months, these specimens were dried in an oven at $105 \pm 2^\circ\text{C}$ until they reached mass equilibrium.

Once the samples were removed from the chambers or from the oven, the side surface (i.e. outer circumference) was sealed with epoxy. A section of plastic is clamped to the surfaces during the application of epoxy to keep the surfaces clean. This also helped to limit drying during the curing process. After the epoxy was dry, 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 [1].

Specimens from mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50 were tested over a period of 90 days. Specimens from mixtures 35/0.50 and 45/0.50 were tested over a period of 8 days.

Two additional 100 mm × 200 mm cylinders were cast for each mortar mixture. After 1 day of curing, the samples were demolded and then sealed in double plastic bags for sealed curing. Bags were stored in a room at $23 \pm 1^\circ\text{C}$ until samples reached an age of 28 d. After that, cylinders were removed from bags and 10 mm ± 2 mm thick samples were cut from the central portion of each cylinder with a wet saw. After cutting, mortar samples were vacuum saturated for 24 h. After that, specimens were placed in environmental chambers at six different relative humidities ($93 \pm 1\%$, $87 \pm 1\%$, $80 \pm 1\%$, $75 \pm 1\%$, $65 \pm 1\%$ and $50 \pm 1\%$) to determine their desorption isotherms.

4. Experimental results and discussion

4.1. Desorption isotherms

Fig. 2 shows the desorption isotherm curves measured using 10 mm thick samples. Mass change was monitored at regular intervals until it reached equilibrium, defined as a mass change of less than 0.02% over a 15 day period. At the end, all samples were oven dried to express water absorption in terms of the dry mass of the sample.

It can be noticed that while the values of the moisture content are similar at 50% and lower RH (lower RH results not shown in Fig. 2), as it refers to the small gel pore system [19], the capillary pores at high RH are strongly influenced by the w/c.

4.2. Effect of initial conditioning on water absorption tests

4.2.1. Effects of relative humidity on sorption test

Fig. 3 shows the absorbed water during the 90 days of testing performed on mortars conditioned at different relative humidities

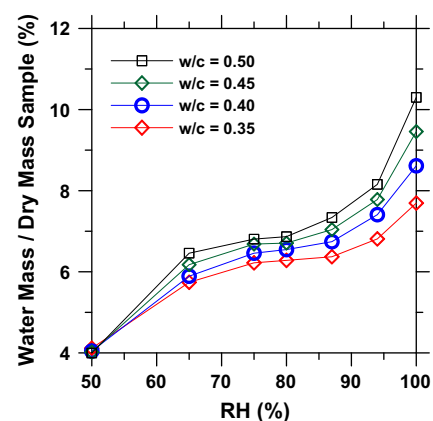


Fig. 2. Desorption curves for 14 months mortar samples (typical standard deviation in the average of three samples is lower than 0.2%).

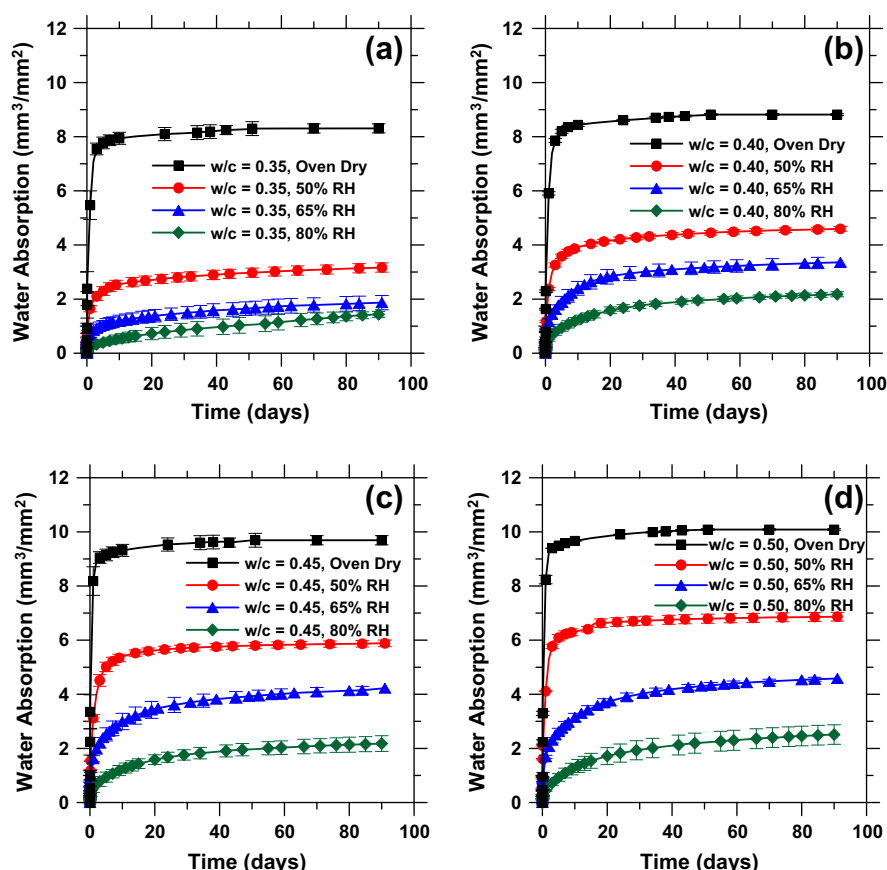


Fig. 3. Absorbed water in mortars as a function of relative humidity (a) mixture 55/0.35, (b) mixture 55/0.40, (c) mixture 55/0.45, (d) mixture 55/0.50. Error bars represent the standard deviation for the average of three samples.

(mixtures 55/0.35, 55/0.40, 55/0.45 and 55/0.50). It can be noticed that the water absorption is very sensitive to the relative humidity at which the specimens were pre-conditioned before testing. In each case, as the conditioning relative humidity increases, more water is retained in the pore system and thus the absorption decreases.

These results can be viewed in a slightly different manner if they include the initial amount of water held in the pores before the test. In order to do this, samples were oven dried at the end of the sorption test to calculate the amount of water they held before starting the test. Additional specimens that were kept at $50 \pm 1\%$ RH during the 14 months were oven dried and then saturated by the procedure described in ASTM C642-07 [26] to measure the total amount of interconnected porosity in the systems. Results from Fig. 3 were then normalized by the total amount of pores in the system, which can be viewed as the degree of saturation of the sample as a function of time. This is presented in Fig. 4. Fig. 5 shows the total degree of saturation for the samples after 90 days.

Figs. 4 and 5 show that samples prepared at different relative humidities with a low w/c (e.g. $w/c = 0.35$) do not reach values near to saturation even after 90 days of being in contact with water. It may be attributed to the refined pore network of this low w/c system which makes it difficult for water to move through the sample to fill all the pores. This is commonly referred to as depercolation, which occurs after different hydration times for different w/c [27].

In contrast after 90 days, samples prepared with a higher w/c (e.g. $w/c = 0.50$) reach much higher levels of saturation. It can be noted from Fig. 4 that samples conditioned at 50% RH reach values near saturation after about 40 days of testing, similar to what is obtained with oven dry samples. Again this may be attributed to the

connectivity of the pore network and the size of these pores. In this case, a more interconnected pore network will facilitate the movement of water to the interior of the specimens and the diffusion of water vapor out of the sample. However, when these samples were conditioned at higher relative humidities (65 and 80% RH), the amount of initially retained water is high enough to reduce the diffusion of vapor out of the sample. As a result, this may explain why the level of saturation of these specimens is lower.

4.2.2. Effects of relative humidity on the amount of absorbed water after 8 days

Fig. 6 shows the cumulative water that was absorbed after 8 days of testing performed on mortars conditioned at different relative humidities, expressed as a function of w/c (Fig. 6a) and as a function of the relative humidity (Fig. 6b).

Fig. 6a shows that mixture 55/0.50 can exhibit six times higher absorption when the samples are conditioned at 50% RH compared with similar samples conditioned at 80% RH.

Fig. 7 shows a normalization of the data presented in Fig. 6. In Fig. 7a the normalization is made with respect to the absorption of samples with $w/c = 0.35$ (mixture 55/0.35). In Fig. 7b the normalization is made with respect to the absorption of samples conditioned at 50% relative humidity. It can be seen that the values follow a consistent trend in each case, except for the oven dry samples. This is in general agreement with the parallel nature of the desorption isotherms for the mortars provided in Fig. 2.

4.2.3. Effects of relative humidity on initial sorptivity

Fig. 8 shows the initial sorptivity calculated as the slope of the absorption vs. the square root of time during the first 6 h of test [1].

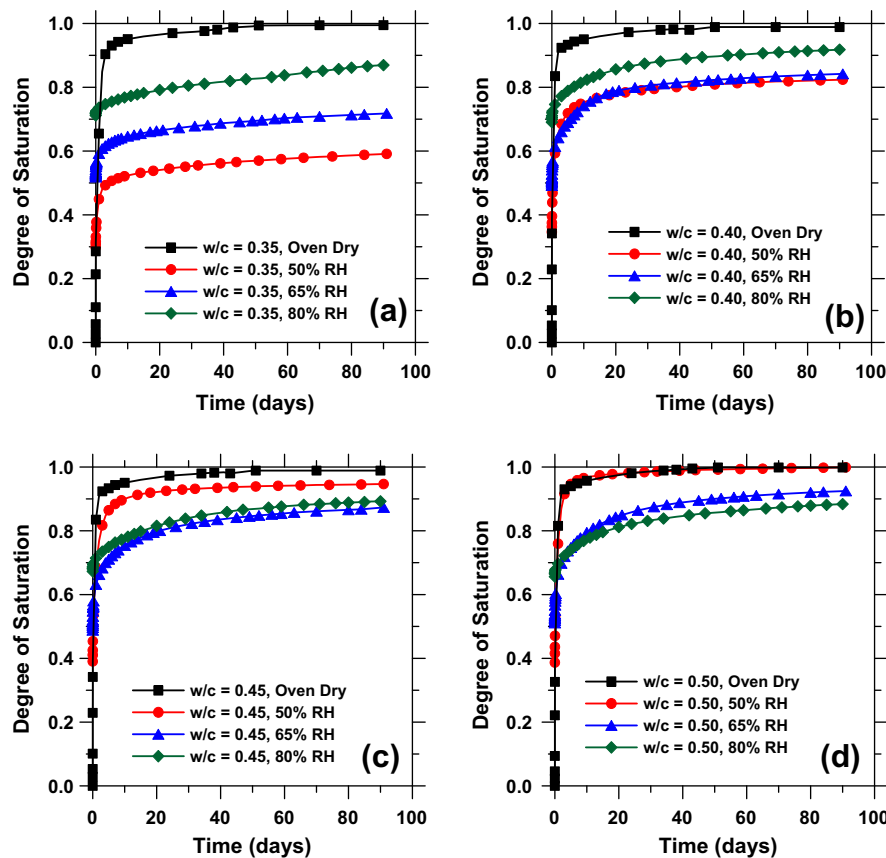


Fig. 4. Degree of saturation as a function of time during the water absorption test: (a) mixture 55/0.35, (b) mixture 55/0.40, (c) mixture 55/0.45, (d) mixture 55/0.50. Typical standard deviation of the average of three samples is lower than 0.02 points in the degree of saturation.

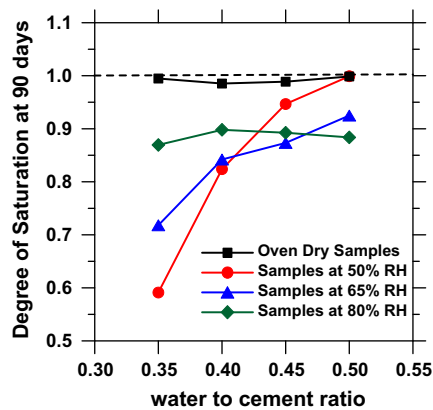


Fig. 5. Degree of saturation after 90 d in contact with water as a function of the w/c.

Fig. 8a shows that mixture 55/0.50 can exhibit a ten times higher initial sorptivity when the samples are conditioned at 50% RH compared with similar samples conditioned at 80% RH.

It needs to be noted that the oven dry samples show a much higher initial sorptivity, due to the fact that the gel's capillary pores are empty. It is also possible that the samples exhibit microcracking around the aggregates due to differential thermal expansion and contraction which may result in the aggregate restraining paste movement. This would be in addition to any microcracking caused by moisture gradients that may occur in the samples that

have dried. During drying, the paste may contract much more than the aggregates that do not really lose moisture or at least not as much moisture loss. Even with some moisture loss from the aggregate, they would likely shrink much less than the surrounding paste due to their higher modulus. While the increase in sorptivity is observed to be linear for the specimens conditioned at 50%, 65% and 80% relative humidity, this trend appears to break down for the oven dry samples which may be attributed to increased micro-cracking generated during the sample preparation [28–32].

4.2.4. Effects of relative humidity on secondary sorptivity

Fig. 9 shows the secondary sorptivity calculated as the slope of the absorption vs. the square root of time between 1 d and 8 d of testing. Trends are similar to those observed for the initial sorptivity. However, it needs to be noted that samples that were oven dry prior to the test present a considerably lower secondary absorption with respect to the samples conditioned in environmental chambers. This may be explained by the high initial absorption of the oven dry samples shown in Fig. 8. During this initial absorption it can be noticed that since a majority of the water was already absorbed in the first hours of the test, the secondary rate of absorption will be much lower. It can also be expected that microcracking enabled a more rapid ingress of water [33].

Fig. 9 shows a similar trend to what was noted in the case of total absorption and initial sorptivity, namely that the secondary sorptivity of samples conditioned in chambers exhibits a consistent trend when the results are plotted against the w/c or the relative humidity at which samples were conditioned. However, samples that are conditioned by drying them in an oven at 105 °C do not follow the same tendency.

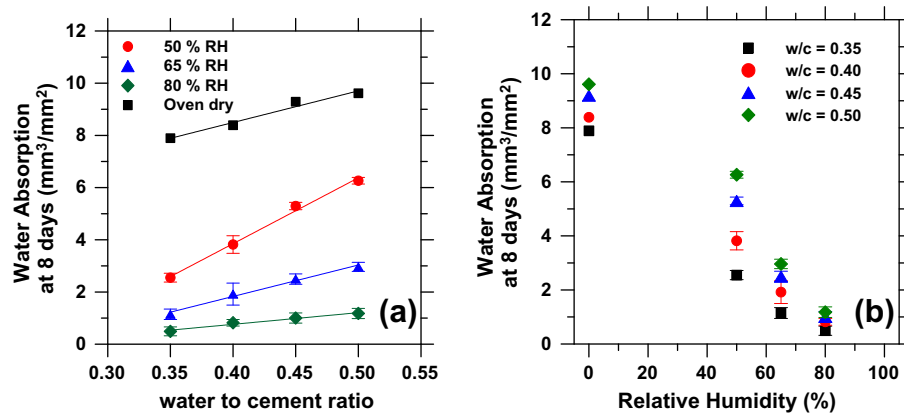


Fig. 6. Cumulative absorption at 8 d for mortars with 55% aggregates versus: (a) w/c, (b) relative humidity. Solid lines are provided only to show a general tendency in the data. Error bars represent the standard deviation on the average of three samples.

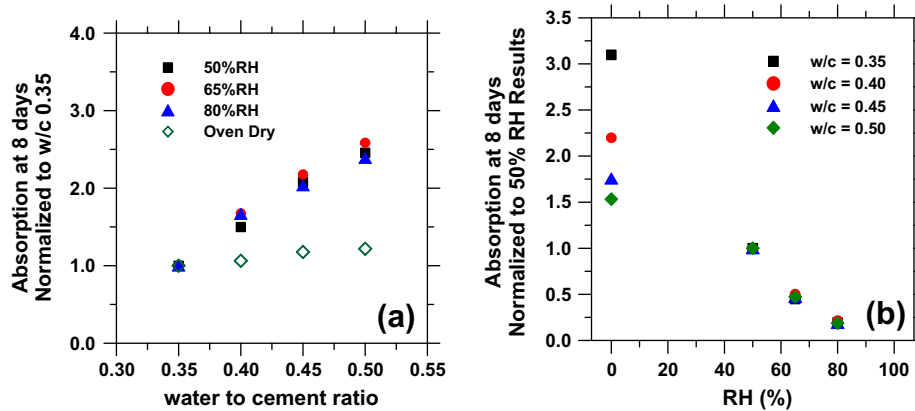


Fig. 7. Cumulative absorption at 8 days versus w/c and relative humidity: (a) normalized to absorption of mixture 55/0.35, (b) normalized to absorption at 50%RH.

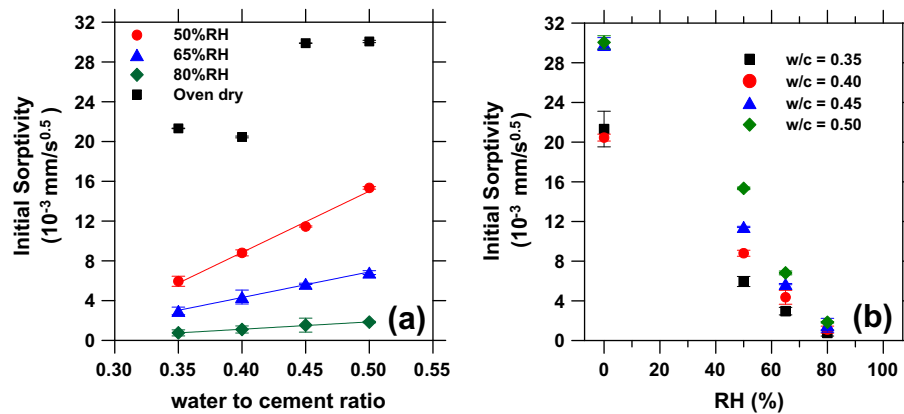


Fig. 8. Initial absorption of the 55% aggregate mortars conditioned at different RH as a function of: (a) w/c, (b) relative humidity. Solid lines are provided to show a general tendency in the data. Error bars represent the standard deviation on the average of three samples.

4.3. Effects of initial moisture of samples on ASTM C1585 conditioning method

At the age of 24 months, samples from each mixture conditioned at the three different relative humidities were removed from the chambers. The side surface was sealed with epoxy to be then “re-conditioned” using the 18 day procedure described in ASTM C1585. In addition, three other samples from each mixture

were saturated following the procedure described in ASTM C642 [26], to then be “re-conditioned” following the same 18 day procedure. While such a resaturation procedure was employed in the initial sorption testing upon which the ASTM C1585 standard was based [34], it was subsequently omitted from the standard. After samples were fully prepared, testing was performed in accordance with ASTM C1585 over a period of 8 days, with results provided in Fig. 10. In addition, Fig. 11 shows the calculated initial and

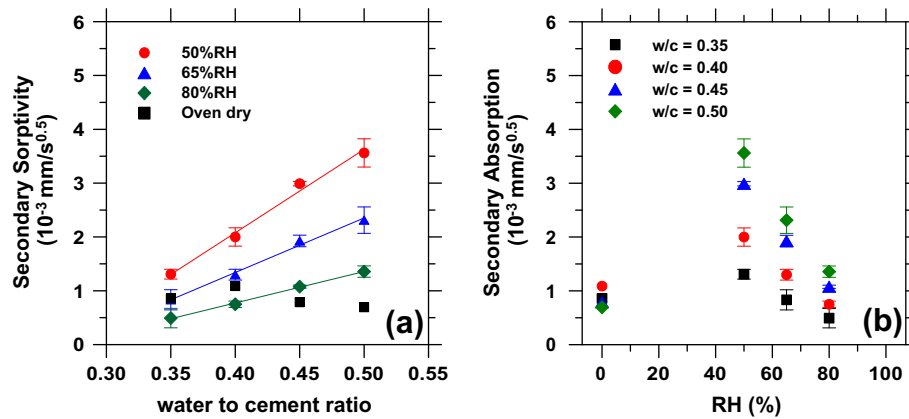


Fig. 9. Secondary absorption on mortars with 55% aggregates conditioned at different RH as a function of: (a) w/c, (b) relative humidity. Solid lines are provided to show a general tendency in the data. Error bars represent the standard deviation on the average of three samples.

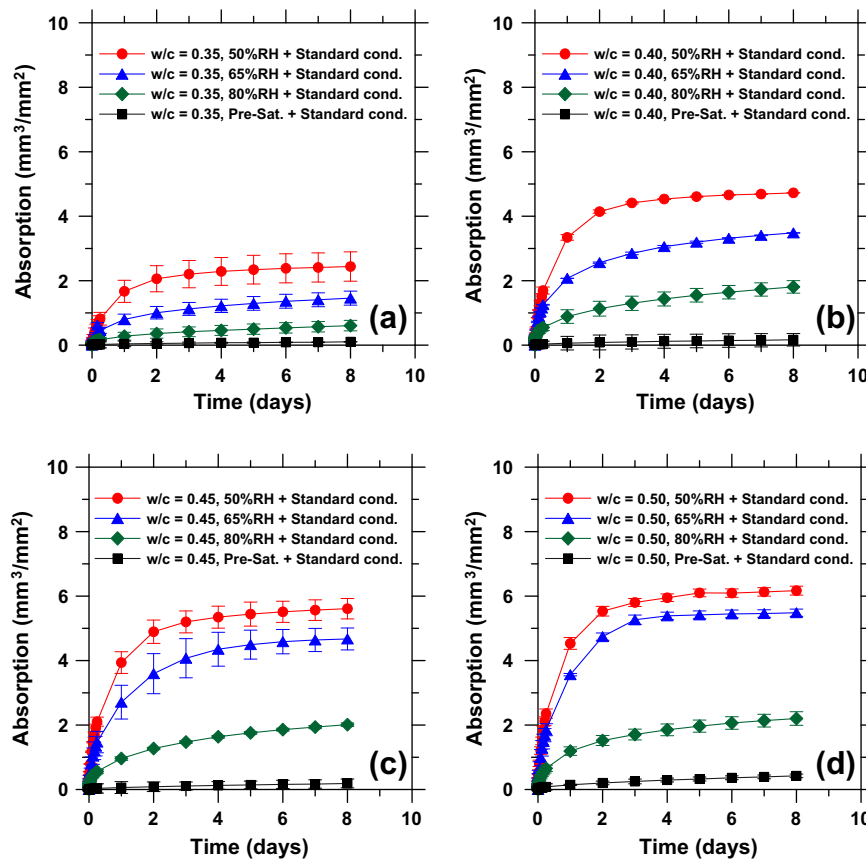


Fig. 10. Effect of initial moisture on the conditioning procedure established in ASTM C1585-04 (a) mixture 55/0.35, (b) mixture 55/0.40, (c) mixture 55/0.45, (d) mixture 55/0.50. Error bars represent the standard deviation for the average of three samples.

secondary sorptivities from these tests. Secondary sorptivity values are not reported when the correlation coefficient is lower than 0.98.

Figs. 10 and 11 show that the 3 days of controlled drying at $50 \pm 2^\circ\text{C}$ and 80% RH followed by the 15 days for internal moisture equilibration is not capable of eliminating the effects of the “moisture history”. These results suggest that the ASTM C1585 preparation method does not prepare all the samples to the same water content before a water absorption test. As such this accelerated method can make a substantial difference in how the data is interpreted. This may be due to a moisture hysteresis effect [35]. It

should be noted that this can be a concern for field samples evaluated using this method, as their as-received relative humidities may easily vary between the extremes examined in this study.

4.4. Effects of volume of aggregate on sorption test

Fig. 12 shows the absorbed water during 8 days of testing performed on mortars containing different volumes of aggregate (mixtures 55/0.50, 45/0.50 and 35/0.50) conditioned at 50% relative humidity. In Fig. 12a the effect of a higher volume of paste is observed as the mixture containing the lower volume of aggregate

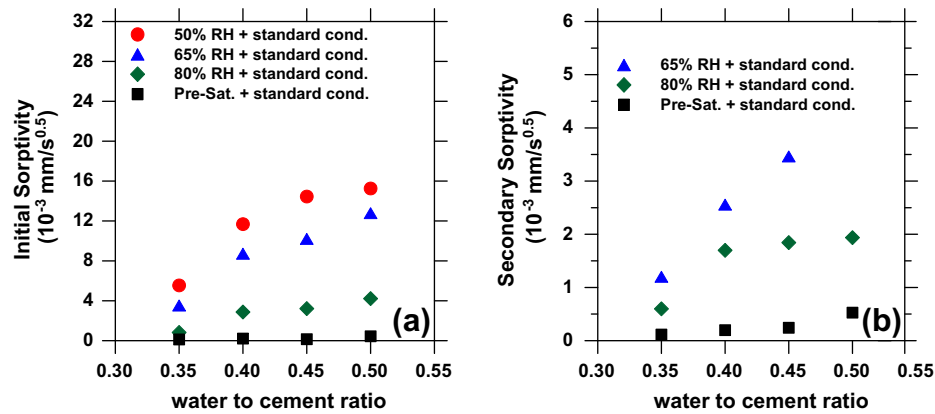


Fig. 11. Initial and secondary sorptivities on mortars with different initial moisture contents, conditioned with the procedure established in ASTM C1585-04.

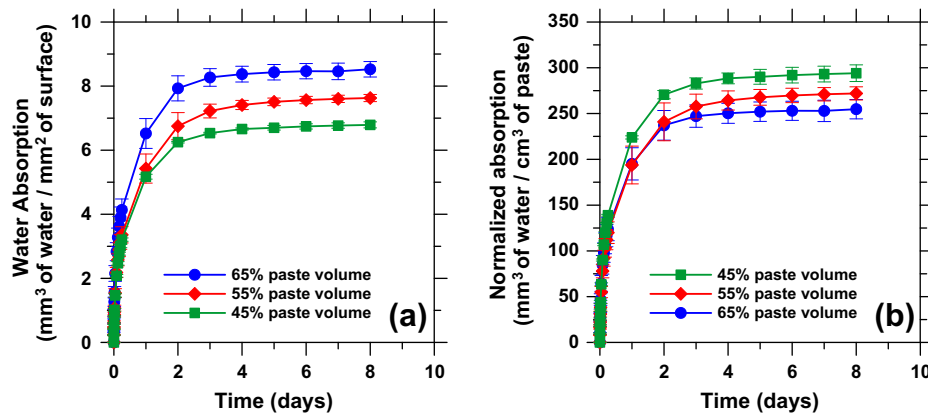


Fig. 12. Water absorption in mortars containing different volume of aggregates: (a) normalized by surface in contact with water, (b) normalized by volume of paste.

has the higher absorption. However, when the results are normalized by the volume of paste (volume of the main absorbent material), a reversal in the order of the samples is observed (Fig. 12b). The samples with the higher volume of aggregates have a higher absorption.

Water absorption is typically reported without considering the effect of the absorption of the aggregate in the samples. To better understand its effect, Fig. 13 was calculated assuming five different sand absorptions (0.0%, 0.6%, 1.2%, 1.8%, and 2.4%) to then subtract

these values from the absorption in Fig. 12b. When the sand absorption is assumed to be 0.0%, the resulting absorption at 8 days will be the same as the absorption presented in Fig. 12b. From Fig. 13, it can be noticed that for the assumed 1.8% sand absorption, the normalized water absorbed for the sample is the same after 8 days, independent of the amount of aggregate in the sample.

Fig. 14 shows a desorption isotherm for the sand used in these mixtures. It can be noted that at 50% RH (humidity at which the samples were conditioned), the amount of water on the sand is

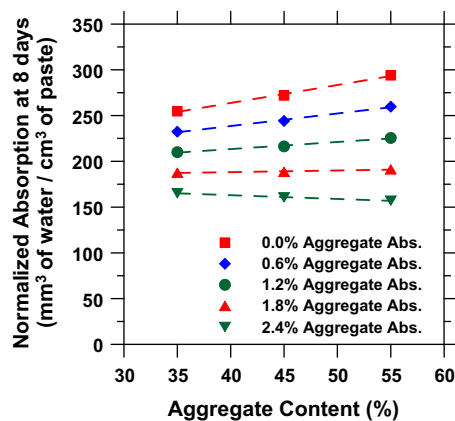


Fig. 13. Water absorption at 8 d normalized by volume of paste, corrected by different values of aggregate absorption.

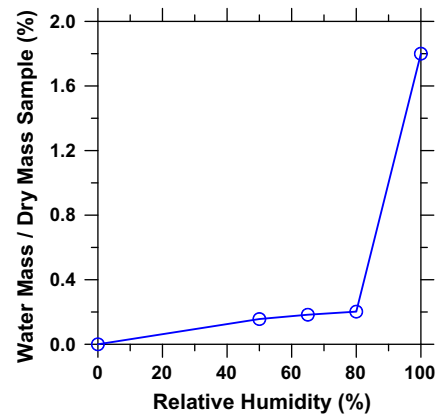


Fig. 14. Desorption isotherm for the sand used in this research.

about 0.2%. Considering that the aggregate used in this study has a 24 h absorption of 1.8%, this would imply that an effective aggregate desorption of 1.6% would have occurred in the samples at 50% relative humidity. This is reasonably consistent with the difference in water absorption of samples containing different amounts of aggregate and their absorption can be explained mainly by the amount of water absorbed by the aggregates.

5. Conclusions

This paper has described the absorption behavior of mortars conditioned at different relative humidities. As was shown in previous works by Hall [12], Hooton et al. [4,7] and Martys and Ferraris [16,34], the water absorption test is considerably affected by the relative humidity of the samples before starting the test, which if not properly accounted for can lead to a misunderstanding of the actual absorption behavior. Samples conditioned at a 50% relative humidity can show a total absorption that is approximately six times greater than similar samples conditioned at 80% relative humidity. This is consistent with expectations based on the mortars' desorption curves.

Initial sorptivity, secondary sorptivity and total absorption at 8 days for samples conditioned in chambers show a linear trend related to the w/c and the relative humidity at which samples were conditioned. Samples that are conditioned by drying in an oven at 105 °C do not follow the same trend as samples conditioned in other approaches. This is attributed to two factors: (1) emptying of a wider range of pores, and (2) the potential for microcracking. The conditioning procedure described in ASTM C1585-04 is not able to eliminate the "moisture history" of the samples, and thus can lead to a misunderstanding of the water absorption test results, especially in field samples which have obtained a lower relative humidity. It is recommended that field samples be pre-saturated prior to being exposed to the conditioning regimen of ASTM C1585.

Comparing samples containing different volumes of aggregate can also lead to a misunderstanding of the actual absorption behavior. Samples containing higher volumes of cement paste will absorb more water. When the results are normalized by the volume of cement paste, the sample containing lower volumes of cement paste will absorb more water. However, for the materials examined in this study, this difference can be mainly explained by the amount of water absorbed by the aggregates in the sample.

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