Impact of Moisture Curing Conditions on Mechanical Properties of Lime-Cement Mortars in Early Ages

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Abstract: Blended lime-cement mortars, which are frequently used in masonry construction, mature as a result of two different phenomena, namely lime carbonation and cement hydration. At any given temperature, these two processes require different moisture conditions for optimal contribution to the mechanical performance of mortar. Since mortars have an impact on the non-linear behavior of masonry from the time of application, it is necessary to optimize their performance with regard to composition and curing conditions. It is expected that a suitable choice of mortar in conjunction with the unit will provide better performance of masonry by reducing risk of cracking and facilitating durability of masonry.

This work aims at studying the impact of environmental relative humidity (RH) in the curing process of lime-cement mortars, focusing on their early age behavior. Two mixes with 25% and 67% lime in the binder (by volume), binder-aggregate ratio of 1:3 and target workability of 175±10 mm were chosen for the study. Mechanical properties like compressive strength, flexural strength, open porosity and density have been studied at 2, 4 and 7 days of curing age. Temperature was kept constant at 20°C while three distinct environmental humidity conditions were tested: sealed environment, 90% RH, and 60% RH. Results have been explored to understand how the evolution of basic mechanical properties changed as a function of curing RH. Curing in sealed conditions and 90% RH did not result in similar values of strength, in either of the two blended mixes. For the mix with 25% lime - 3C1L12S (25%), hardening appeared to be guided by cement hydration. For the mix with 67% lime - 1C2L9S (67%), curing in RH of 60% and 90% resulted in almost the same strength at day 7, demonstrating that lime carbonation may be important earlier than 7 days.

Keywords: Mortars, moisture curing conditions, early ages, mechanical strength, lime and cement, relative humidity

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INTRODUCTION

Lime and cement are often mixed together in varying proportions to form mortar for brick masonry, in order to overcome the individual shortcomings of either type of binder (Arandigoven and Alvarez 2007). The two main mechanisms that contribute to the hardening of such type of mortars are carbonation of lime and hydration of cement (Cizer 2009). It is commonly accepted that in order to promote cement hydration, the relative humidity (RH) in the environment must be more than 80% at the very least (Hayri and Baradan 2011, Spears 1983). The European standards for cement mortars recommend covered curing for the first 24 hours, followed by immersion in a water bath for the remaining duration, up to the point of testing (EN 196-1, 2005). In the case of carbonation of air lime mortars, it is reported that carbonation generally does not take place in saturated conditions because the diffusion of carbon dioxide gets hindered (Cizer et al. 2008). This is because diffusivity of carbon dioxide drops by almost 10,000 times in water compared to that in air (Richardson 1988). However, some amount of moisture is required in the mortar, since the controlling factor of carbonation reaction has been found to be the dissolution of calcium hydroxide crystals in the aqueous medium of the reaction (Balen 2005). Furthermore, interdependence between carbonation of the mortar and its moisture content has been reported, since water is also released during carbonation (Cizer et al. 2008). Balen and Gemert (1994) proposed mathematical formulations to express carbonation using non-linear coupled differential equations taking into account the effects of drying of mortar along with the water produced by carbonation. Research stating a numerical value of requisite RH for carbonation of lime over time, was not found (Balen 2005, Balen and Gemert 1994).

Cizer (2009) studied the competition between carbonation of lime and hydration of cement that results from combining air lime and Portland cement together. It was concluded that at a given temperature of 20°C, if RH is maintained at 90%, then the bulk of cement hydration is mostly rendered complete by 28 days. Furthermore, by employing XRD and TGA for lime-cement mortars, it was concluded that in an environment with RH of 60% carbonation of free lime begins to get promoted only after 7 days (Cizer 2009). However, a comparison of mechanical strength of lime-cement mortars has not been done for ages less than 28 days in different moisture conditions of curing. The European standards recommend curing in 20°C and 65±5% RH after 7 days of age (EN 1015-11 2007). It has been verified from literature that lime-cement mortars do indeed require dry curing conditions (~60% RH) in the long term in order to promote carbonation (Cizer 2009). For the first 7 days however, the standard recommends curing in 20°C and 95±5% RH (EN 1015-11, 2007). But no study was found to compare results of mechanical strength from different moisture curing conditions at ages less than 7 days, which makes it difficult to conclude whether 95±5% RH would be beneficial for all lime-cement mortars in the early ages, especially for mortars in which the binder is mostly composed of lime (Arandigoyen and Alvarez 2007, Cizer 2009).

This study aims at carrying out research on two lime-cement mortars, one with a majority of cement in the binder and the other with majority of lime in the binder to compare the difference in impact that moisture curing conditions may have on them. Basic mechanical parameters like strength and density have been studied in an attempt to understand how moisture curing conditions impact the mixes from the time of casting up to 7 days, i.e. before the EU Standards impose $65\pm5\%$ RH as the requisite moisture curing condition (EN 1015-11 2007, Cizer et al. 2010). Both mixes were subjected to three different moisture curing conditions namely, 60% RH, 90% RH and a sealed environment and tested for compressive strength, flexural strength, ultrasound velocity and change in density at 2, 4 and 7 days of curing. Porosity has been studied at 7 days of curing age. The first reason for choosing a sealed environment over a range of RH between 20%-30% was that it would not aid cement hydration and would also possibly hinder carbonation

(Hayri and Baradan 2011, Balen 2005). The second reason was that the European standards recommend $95\pm5\%$ RH or 'wrapping in polythene bags' as moisture curing for lime-cement mortars in the first 7 days (EN 1015-11 2007). The two conditions may not necessarily lead to similar mechanical properties, especially in the wide range of mixes with (10-90) % cement in the binder.

RESEARCH PROGRAM

Materials

The binder material used for both mixes was composed of lime and cement. The lime used was air lime of type CL-90S, according to EN 459-1 (2010) and the cement used was Portland cement type CEM I – 42.5 R according to EN 197-1 (2000). The choice of the latter was made to ensure constant properties and repeatability in the testing program, despite CEM II being more commonly used in real applications. The binders were pre-conditioned in sealed boxes in an environment of 20°C temperature and 60% relative humidity for up to 7 days before casting of the mixes. The aggregate used was sand in accordance with the standard EN 13139 (2013), with customized particle size range of 0/4 mm. Before casting the mixes, the aggregates were heated to 105°C and cooled down to room temperature. This was done for constant moisture content in each mix.

Mortar compositions

The notations C, L, S have been used to denote the proportions of cement, lime and sand used, by volume. For example 3C1L12S denotes 3:1:12 (Cement: Lime: Sand) by volume. Furthermore, the number in the parenthesis next to the notation of the mix, denotes the percentage of lime in the binder, by volume, for example 3C1L12S (25%). Two different mix compositions were chosen for this study, namely 3C1L12S and 1C2L9S with 25% and 66.7% lime in the binder (by volume) respectively [Table 1]. The choice of these two mortars was based on masonry mixes used on field, and also to ensure the same demolding time (i.e. 2 days) for both according to the European standards, which is based on the quantity of lime in the mix (EN 1015-11 2007). The choice of mortars was also guided by the objective of the research, i.e. to test the impact of moisture curing conditions on two lime-cement mortars which were very different with respect to the composition of their binder. And therefore, one mortar with 'a little' lime (>25% by volume in binder) and one mortar with 'a lot' of lime (>50% by volume in binder) were chosen.

Table 1: Composition of blended lime cement mortars (For every 1 m ³ of mortar produced)							
Nomenclature of mixes		Cement: Lime: Sand	Cement	Lime	Water	Water-Binder ratio	
Notation	Lime in binder (Volume %)	(Ratio by volume)	(kg)	(kg)	(kg)	(By weight)	
3C1L12S	25	3:1:12	262.7	33.4	295.6	1.00	
1C2L9S	67	1:2:9	116.8	89.0	325.0	1.58	

To ensure consistency in casting of different mixes, measurement of raw materials were carried out by converting volume to mass by employing their apparent densities. Workability of the mixes was decided keeping in mind the industrial application of these mortars; according to EN 1015-3 (2007), a flow table value of 175 ± 10 mm was targeted for all mixes. Prismatic specimens of size $40\times40\times160$ mm were cast for all the tests according to standard EN 196-1 (2005). According to recommendations from EN 1015-11 (2007), demolding of specimens of both mix compositions was carried out two days after casting. Three

moisture curing conditions were used with temperatures of 20 ± 1 °C, namely $60\pm5\%$ RH, $90\pm5\%$ RH and sealed conditions. The first two conditions were achieved by air curing, i.e. the specimens were placed in a climatic chamber with the corresponding conditions. The latter was achieved by keeping the specimens sealed in plastic bags in 20 ± 1 °C (Hayri and Baradan 2011). Each of these curing conditions was imposed on the specimens from the time they were cast, up to the point of testing.

Experimental campaign

Three-point bending (flexural strength) test was performed at 2, 4 and 7 days of curing age (EN 1015-11 2007). The final value was obtained from an average of three specimens, each of which was preloaded with 150 N and tested with displacement control at the rate of 0.006 mm/s. Unconfined uniaxial compressive strength test was performed at a rate of 50 N/s on the 6 parts resulting from the 3 specimens subjected to flexural tests. The loading conditions were chosen to ensure that even the specimens with the lowest strength could be tested without shock and were subsequently kept uniform for all specimens. Each value shown is representative of an average obtained from tested specimens that were cast and cured in the same mold. Standard error of each average value has been shown as error bar in the graphs. Furthermore, reproducibility from the experimental procedure was tested by casting the same mix on different days and repeating some tests. The difference in values of average compressive strength was found to be between 2% to 5%. Prior to each of the flexural tests, change in weight of each specimen was measured along with ultrasonic pulse velocity (UPV). UPV was measured with P-waves of frequency 150 kHz, along the length (i.e. 160 mm) of the specimens. Additionally, open porosity was measured on the 7th day for both mix compositions cured in the three different moisture conditions. The procedure adopted was based on the RILEM recommendations by TC 25-PEM (1980) for deterioration of stone. However, the duration of subjecting the specimens to vacuum and immersion in water was modified to 3 hours from 24 hours based on the recommendations (tailored for mortars) from University of Beira Interior, often used in Portugal.

RESULTS

Mechanical strength

Values of compressive strength obtained for the mix 3C1L12S (25%) exhibit an increase in strength with time for all curing conditions (Figure 1). It is also possible to suppose that the evolution of compressive strength seems to be guided mostly by cement hydration as on the 7th day, curing in 60% RH leads to the least amount of strength. Furthermore, it is also possible to note that on day 7, there is a difference of more than 10% in the values of compressive strength obtained from sealed conditions and 90% RH. Interestingly, this difference manifests between day 4 and day 7 of curing, because in the first 4 days strength for the sealed condition and 90% RH appear to be similar. The rate of evolution of strength appears to be similar for all three conditions, with (50-60) % strength being attained in the first 2 days and (80-90) % strength by the 4th day, normalized with respect to day 7.

For the mix 1C2L9S (67%), it is interesting to note that cement hydration does not seem to be playing the same role as it did in the mix 3C1L12S (25%). The sealed conditions result in the lowest compressive strength on day 7 (Figure 2). Curing with 90% RH consistently results in higher strength than sealed conditions, with the difference amounting to 16% by day 7.



Curing with 60% RH exhibits relatively different kinetics. It results in the lowest strength on day 2, increasing to be the highest on day 4 and subsequently increases just a little bit by day 7. However, if the standard errors of the values of strength are taken into account, the strength on day 7 may be considered the same for 60% and 90% RH curing conditions. Compared to curing in 60% RH, while sealed conditions do result in 11% higher strength on day 2, continued curing in a sealed environment results in 29% lower strength on day 4 as and 13% on day 7. It may thus be concluded that in a mix, with a significant amount of lime, cement hydration may not be the major or only driving factor even between 2-7 days of curing. Carbonation may be beginning to contribute to hardening of the mortar earlier than 7 days of curing, but other phenomena arising from drying and capillary forces may also be envisaged. Additionally, it may be noted that regardless of the amount of lime in the binder, curing in 90% RH and sealed conditions do not result in comparable values of compressive strength after 7 days of curing.

For the mix 3C1L12S (25%), flexural strength exhibits trends similar to those observed in compressive strength (Figure 3). Curing in sealed conditions lead to the highest strength, followed by curing in 90% RH and then by 60% RH. Once again, cement hydration seems to be the driving mechanism of hardening, since there is a difference of almost 35% in the values of strength obtained at day 7, between curing in sealed conditions and 60% RH. In the case of curing in 90% RH, it appears that strength drops slightly between day 4 and day 7, but if the standard errors of both values are taken into account, the strength may be considered similar. The same conclusion may be drawn for change in strength between day 4 and day 7, in sealed curing conditions, i.e. taking standard errors into account results in similar values of strength. It must also be noted that by day 7, curing in sealed conditions and 90% RH lead to difference of almost 25% in the results.

In the case of the mix 1C2L9S (67%), behavior observed in flexural strength is similar to that of compressive strength as well (Figure 4). While curing in 60% results in the lowest strength on day 2, it rises to the highest on day 4 and subsequently increases further by day 7. It may be noted that on day 4, similar to the pattern in compressive strength, 60% RH leads to the highest value, followed by 90% RH and then by sealed conditions. In this case however, all curing conditions result in almost the same value of strength by day 7. Once again, it may be concluded that in the time between 2-7 days of curing, cement hydration may not be the sole hardening mechanism for mixes with significant quantities of lime in them.



Ultrasonic pulse velocity and change in density

UPV was measured for all specimens at of mix 3C1L12S (25%) and has been displayed below (Figure 5). For these measurements, the coefficient of variation in each case was less than 1% and therefore does not reflect as significant standard error on the graph. The trend follows that of compressive strength and flexural strength, with the highest velocity obtained from curing in sealed conditions, followed by 90% RH, and then 60% RH. All curing conditions result in an increase in UPV with time.

For the mix 1C2L9S (67%) UPV could not be measured on day 2 and day 4 of curing in sealed conditions, possibly due to low strength and saturated conditions (Figure 6). Furthermore, UPV displays a trend very different from that of compressive strength or flexural strength (Figure 6). The highest velocity occurs from curing in sealed conditions, followed by 90% and then 60% RH. The plausible reason for these unexpected trends of UPV observed could be that ultrasound waves travel faster in saturated or moist specimens (Pakosy 2006, Lencis et al. 2014). Additionally, in the case of concrete, this impact is reported to be higher for low strength concrete (Blitz and Simpson 1996). These preliminary test results indicate that in the case of mixes with a significant quantity of lime in the binder, UPV is not a direct indication of the trends observed in compressive or flexural strength.



In order to evaluate the percentage change in density of hardened prismatic specimens, the average change in density of the mixes was evaluated at 4 and 7 days for different curing conditions - 90% and 60% RH (Table 2). The reference adopted was the density measured after demolding the specimens cured in sealed conditions. It may be noted that curing in sealed conditions resulted in negligible loss of weight between day 2 and day 7 for both the mixes.

Table 2: Loss in density of mortar specimens cured in different moisture conditions							
Percentage loss in density of	Mix (Cement: Lime: Sand) – (% volume of lime)						
specimens - $\%(\Delta \rho)$	3C1L12S (25%)			1C2L9S (67%)			
Sealed – reference (kg/m ³)		2186.8		2168.1			
Curing condition - RH	Day 2	Day 4	Day 7	Day 2	Day 4	Day 7	
90 %	3.4	3.8	4.7	2.3	8.2	7.9	
60 %	2.7	5.8	6.8	8.0	10.7	12.0	

Table 2: Loss in density of mortar specimens cured in different moisture conditions

The first evident difference is that after demolding on the 2nd day up to 7 days of curing, the mix 3C1L12S (25%) has greater density than 1C2L9S (67%) in sealed conditions. This is because of the greater quantity of cement in the binder which is denser than lime and the resulting hydration of cement. Secondly, the loss in density is greater when the specimens are cured in 60% RH, compared to 90% RH. This is expected since RH indicates the amount of vapor in the air compared to the total amount of vapor it can contain and lower the RH, the more moisture can be held by the atmosphere, promoting more intense drying of the mortar specimens. Additionally, loss in density increases for both mixes with time, consistently remaining a little higher for curing in 60% RH. The third observation is that the loss in density in the mix 1C2L9S (67%) remains almost always higher than its counterpart mix 3C1L12S (25%). This could be explained with the help of chemistry since cement hydration uses water while lime carbonation produces water (Cizer 2009). Furthermore, cement hydration causes a denser, less porous skeleton compared to lime carbonation (Mosquera et al. 2006), which could be responsible for the consistently higher losses in density of the mix 1C2L9S (67%).

By correlating change in density or ultrasound velocity with compressive strength, no pattern is evident. However, if all three are correlated and expressed as a function of RH, it is possible to observe that the square of UPV (m/s, with y-axis = UPV²) increases with an increase in a function of density (kg/m³) and compressive strength (MPa, with x-axis = $\rho^{0.5} f_c^{1.5}$). For the mix 3C1L12S (25%) this pattern may be observed after 2 and 4 days of curing in different moisture conditions (Figure 7). The relationship is not linear, possibly because the moisture conditions (expressed in RH) considered do not vary linearly either. For the mix 1C2L9S (67%), the correlations could not be observed at day 2 or day 4 because of lack of data from UPV measurements. However, it was possible to compare data obtained on day 7 from both mixes, cured in different conditions (Figure 8). It is easy to conclude from Figure 8, that after 7 days, the mix 3C1L12S (25%) exhibits greater UPV, density and compressive strength than the mix 1C2L9S (67%) in all of the three moisture curing conditions. More experiments are required to understand this relationship better; between the compressive strength of a mortar, its density and UPV measured, taking into account RH in curing.



Figure 7: Correlation between density, compressive strength and UPV for mix 3C1L12S (25%)

Figure 8: Correlation between density, compressive strength and UPV at day 7 for lime-cement mixes

Open porosity

Open porosity was measured for both mixes after 7 days of curing in the three different moisture conditions (Table 3). One-way ANOVA was performed for the results of the mix 3C1L12S(25%) (p-value = 1.65e-3, F-value = 22.38) and it was found that the values obtained in different curing conditions were (statistically) significantly different from each other. The lowest value of porosity was obtained from curing in sealed conditions, possibly due to sealed curing conditions aiding cement hydration. However, it was not possible to identify a meaningful pattern, with respect to the level of RH. For the mix 1C2L9S (67%), one-way ANOVA (p-value = 9.94e-5, F-value = 61.77) resulted in statistically significant difference in the mean values of open porosity obtained in different moisture conditions. It was observed that the highest open porosity was in sealed curing conditions, followed by curing in 90% RH and subsequently in 60% RH (Table 3). While the range of variation is small, a pattern does seem evident. It may be that cement hydration is not the main factor causing hardening, since otherwise the pattern would have been reversed, with the lowest value of open porosity in sealed conditions since greater levels of moisture in the ambience would promote cement hydration (Hayri and Baradan 2011), leading to a reduction in both pore volume and size (Mosquera et al. 2006). It may also be observed that regardless of amount of moisture in different curing condition, 3C1L12S(25%) always exhibits lower values of open porosity, compared to 1C2L9S(67%).

This is consistent with data found in literature; mixes with greater quantities of cement in the binder exhibit lower values of open porosity, because more cement is available in the binder for hydration (Cizer 2009).

Nomenclature of mixes		Porosity at day / in different curing conditions (RH)					
Notation	Lime in binder (Volume %)	Ratio	Sealed	90 %	60 %		
3C1L12S	25	3:1:12	24.0	25.7	24.6		
1C2L9S	67	1:2:9	28.0	27.4	25.9		

Table 3: Open porosity of lime-cement mortars measured after 7 days of curing in different moisture conditionsNomenclature of mixesPorosity at day 7 in different curing conditions (RH)

CONCLUSIONS

The research carried out in this paper is intended to be an introductory study. Since, it is established from literature (Cizer 2009) and the European standards (EN 1015-11 2007) that after 7 days of curing, 60% RH is required to promote carbonation; the focus of this work was the period up to 7 days of curing. The main conclusion from this work is that in order to optimize mechanical behavior, different moisture curing conditions may be required for blended mortars with significantly different quantities of lime and cement in their binder. Furthermore, the results of mechanical strength and open porosity indicate that in mixes with a significant amount of lime in the binder, lime carbonation may be playing a role in hardening earlier than 7 days of curing. While this may be an important consideration in the development of new recommendations, further research is required to confirm the trends observed in this preliminary study.

- 1) Based on evolution of flexural and compressive strength up to day 7, the main hardening mechanism in the mix 3C1L12S (25%) appears to be cement hydration. Sealed curing conditions result in maximum strength (on day 2, 4 and 7), followed by 90% RH and then by 60% RH. Curing in sealed conditions and 90% RH do not result in similar values of strength.
- 2) For the mix 1C2L9S (67%) however, sealed conditions result in the lowest values of compressive and flexural strength on day 7. Curing in 60% RH results in higher or almost the same values of strength as curing in 90% RH does on day 4. By day 7, 60% and 90% RH result in almost the same strength, demonstrating that lime carbonation may be important earlier than 7 days.
- 3) Open porosity values for the mix 3C1L12S (25%) do not exhibit a pattern with regard to moisture in curing conditions. However, for the mix 1C2L9S (67%) on day 7, open porosity was found to increase with increase in RH of the curing condition.

It must be stated that all results obtained in this work, were from mortars that were cast and cured in laboratory conditions. The mechanical properties obtained from mortars cast on field, may vary due to difference in dimensions, curing conditions, suction from masonry units and so on. Therefore, the results presented in this research may not be reflective of how mortar may actually perform in masonry.

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