4th Portuguese Congress on Mortars and ETICS

Mechanical Properties of Hydraulic Lime Mortars

Geoffrey Allen
University of Bristol
UK
g.c.allen@bristol.ac.uk

Richard Ball
University of Bath
UK
r.j.ball@bath.ac.uk

Abstract: The effect of dewatering, time dependent deformation under load and wetting and drying of NHL3.5 natural hydraulic lime mortar has been investigated. Both dewatering and wetting and drying cycles were found to increase mortar strength by enhanced carbonation. An apparatus was constructed to measure the dimensional changes over a period of 180 days. The rate of deformation during the first two weeks of sample monitoring was proportional to the applied load. A logarithmic creep expression of the form \( \varepsilon = A \ln (t) + B \) gave the best fit to the data where, \( \varepsilon \) is strain, \( t \) is time and \( A \) and \( B \) are constants. A linear relationship was found between the coefficient \( A \) and applied load.

Keywords: Hydraulic lime, dewatering, wetting and drying, creep

1. INTRODUCTION

Greater environmental awareness of the advantages of lime mortars in terms of carbon dioxide emissions released during manufacture and the adsorption of carbon dioxide during carbonation is promoting their use. A number of different limes are available for building, differing by their strength and chemistry [1-4]. Calcium limes harden by carbonation but hydraulic lime mortars do so through a combination of hydration and carbonation. In this study natural hydraulic lime (NHL), produced by burning a mixture of naturally occurring argillaceous limestone or chalk has been used. For hydraulic mortars, the initial hydration reaction results in the formation of a calciumsilicate-hydrate (C-S-H) structure, which provides an initial set [4-6]. Subsequently the remaining bulk calcium hydroxide then reacts with atmospheric carbon dioxide. The increase in volume associated with carbonation fills surface pores and cracks, reducing the number of diffusion paths.
and restricting the diffusion of carbon dioxide to greater depths [7]. In thick-walled structures this can result in the mortar located at the centre of the wall remaining un-carbonated for many years. If the mortar is fully carbonated, dissolution and re-precipitation of calcium carbonate by the movement of moisture through the structure may also contribute to strength [8]. These mechanisms are commonly associated with autogenous healing.

Here we report studies of the mechanical behaviour of hydraulic lime mortars in masonry units or stones. The role of the masonry in extracting water from the mortar depends on its open porosity [9-10]. Measurements of the change in resistivity of the mortar with time have been carried out using impedance spectroscopy to monitor the dewatering process. Dimensional changes of mortar within a structure, over time, will lead to changes in the magnitude and direction of forces both within the mortar and masonry units. Measurement of creep and shrinkage in lime mortars allow the significance of deformation to be determined in a building context.

In practice, it is not possible to determine how wetting and drying affect the strength of a mortar joint on site. Repeated dampening by spraying is often used as a method of protecting mortar however there is no simple rule to determine whether an appropriate number of cycles have been undertaken. If the mortar or render dries too rapidly binding properties of the lime are lost and the mortar is likely to fail. In an attempt to verify the most appropriate procedures a NHL3.5 hydraulic lime mortar was exposed to wetting and drying cycles to simulate the effect of environmental conditions.

2. EXPERIMENTAL METHOD

2.1 Raw materials and sample specimen designs
Natural hydraulic lime mortars of classification 3.5 (comprising 30% dicalcium silicate, 48% calcium hydroxide, 14% calcium carbonate, 2% gypsum, 2% calcium alumino ferrite, 2% tricalcium aluminate and 2% calcium aluminosilicate by weight) was mixed with well rounded sand with particle size 0.5mm. Energy dispersive analysis of the sand indicated the presence of silicon, aluminium, potassium oxygen, iron, titanium and calcium in order of decreasing signal intensity, (see figure 1).

![Energy dispersive x-ray analysis from the surface of a Croxden sand grain showing the elemental composition.](image-url)
The raw materials were stirred for 10 minutes using sufficient water to provide a mix flow between 180-200mm. The mould was coated with release oil prior to casting cylinders 18 mm in diameter and 36 mm in length. These samples were of suitable dimensions for dewatering, wetting and drying, creep and compressive testing. To reduce the presence of air pockets within the specimens and to ensure an even distribution of lime mix within the mould, each mould was half filled and vibrated for 1 minute before filling to just below the top and continuing vibration for a further 1 minute. To ease extraction of the specimens, the mould had a removable bottom and was split along the specimen length. After one day, the bottom of the mould was removed.

2.2 Compressive testing
The compressive strength of the mixes was determined using a Zwick/Rowell testing machine. Each specimen was mounted between two parallel stainless steel platens. The top platen was allowed to pivot on a ball joint to accommodate specimen surface irregularities. Loading was applied at a constant stress rate of 0.079 Nmm-2s-1 until failure.

2.3 Dewatering
Mortar samples were dewatered by inverting the cylindrical moulds to allow the mortar to contact a high sorptivity brick substrate. A time of 15 minutes was allowed for dewatering, substantially more than that recorded in practice. Dewatered and non-dewatered mortar specimens were exposed to atmospheres containing either pure nitrogen or nitrogen containing 400 ppm carbon dioxide. Under both gas compositions the relative humidity and temperature was maintained at 65% and 20°C respectively. Samples were removed for mechanical testing after 14, 28 and 56 days of exposure.

2.4 Wetting and drying
A specially designed wetting and drying chamber was used to condition the samples. This consisted of a spraying device capable of saturating the samples within 60 seconds. Two fans, positioned to suck air through the chamber, dried the samples over a period of several hours. Both spraying and drying operations were electronically controlled wetting for 10 minutes and drying for 20 hours, repeatedly for the duration of exposure within the chamber. Natural hydraulic lime mortars were manufactured with mix ratios by volume 1:1, 1:2, 1:3 and 1:4 (lime:sand) and examined after 28, 56, 90 and 180 days exposure [11].

2.5 Deformation and shrinkage
A creep rig containing eight individual locations for simultaneous sample monitoring was constructed as shown in Figure 2. Displacements were logged using a PC interfaced with Solartron Metrology linear displacement transducers via an Orbit Measurement System with resolution 50 nm. Readings were recorded at intervals of 10 s. Following an initial post-mixing period of 7 days, samples were inserted into the creep rig. Creep loads of 0, 0, 108, 108, 162, 162, 225 and 234N were applied to 8 samples respectively [12-14].
2.6 Scanning electron microscopy
The structure of the mortar was characterised using SEM and the elemental composition of the sand by EDX after coating with a layer of gold for 180 seconds in an Edwards sputter coater. Examination was carried out in a Hitachi S-2300 SEM using a secondary electron detector. Energy dispersive x-ray analysis was performed using an Oxford Instruments detector and electronics with Thomson Scientific WinEDX software.

2.7 Impedance measurements
The application of impedance spectroscopy to cementitious systems is well documented [15]. The impedance response of the wet mix was monitored using a Solartron 1260 impedance analyser over the frequency range from 10 MHz - 100 Hz. Each sweep between these frequencies contained 5 steps per decade and were carried out at a potential of 100 mV. 200 sweeps each lasting 70 seconds were repeated continuously over a time period of 225 minutes. The test cell [16] was constructed using two rectangular stainless steel electrodes 25 mm by 30 mm spaced 25 mm apart. These were positioned in a square section tube of the same dimensions as the brick prism to allow unidirectional flow of liquid between the wet mix and substrate. The electrodes were positioned 15 mm above the brick surface.

3. RESULTS
3.1 The dewatering process
The dewatering process was monitored in real time using impedance spectroscopy. The measured resistivity was plotted against time and the slope of the line corresponded to water movement into the absorbent substrate (see figure 3). The change in slope was considered to be a consequence of the formation of silicate crystals at the mortar/substrate interface, as shown in figure 4. Figure 5 shows the average compressive strengths of both non-dewatered and dewatered mortars after 14, 28 and 56 days of curing in atmospheres of 100% nitrogen and nitrogen containing 400 ppm carbon dioxide. The results presented
show that, for NHL3.5, the failure stress increased when the mortars were cured in the environment containing 400 ppm carbon dioxide. Previous results indicated that 28 days was a sufficient period for the carbonation process to have initiated throughout the entire volume of the sample [8]. The NHL3.5 lime mortars showed a clear increase in strength at all curing times for dewatered mortar over the non-dewatered equivalent.

Figure 3. Plot of resistivity versus time for NHL3.5 mortar on a porous brick substrate indicated transition from absorption into the brick to drying.

Figure 4. SEM image showing silicate formation on the surface of a brick substrate following dewatering of a wet mix. (Image by Dr Adel El-Turki, University of Bristol)
3.2 Effect of wetting and drying on strength
For each mix the most dramatic increase in strength was observed within the first 28 days following 33 wetting and drying cycles. The average strength (28 – 180 day) of the cycled mortar in comparison to an un-cycled control, for each of the mix ratios tested, is shown in figure 7 [11].

3.3 Time related deformation of NHL3.5
The deformation of 1:2 NHL3.5 to sand mortar was monitored over a period of 180 days. During the initial 14 day period there was a large change after which the rate of deformation was observed to reach a steady value. Figure 6 shows a plot of creep rate against time calculated by subtracting the averaged rate of deformation of the unloaded samples from that of the samples loaded to 225 and 234N [12].

4. Discussion
During the life cycle of a lime mortar a number of significant processes occur. First amongst these is the rapid process of dewatering when the mortar is placed upon porous masonry. Climate changes in humidity and temperature may then play a part in the rate of the stiffening process and subsequent changes in physical and chemical structure. Figure 5 shows the average compressive strengths of both non-dewatered and dewatered mortars after 14, 28 and 56 days of curing in 100% nitrogen and nitrogen containing 400 ppm carbon dioxide. The results presented show that, for all mixes, the failure stress increased when the mortar was cured in the environment containing 400 ppm carbon dioxide. Furthermore, the strength increased with curing time, as well as with binder hydraulicity.
Figure 6. Plot of creep rate against time calculated by subtracting the averaged rate of deformation of the unloaded samples from that of the samples loaded to 225 and 234N.

Figure 7. Enhanced development of compressive strength resulting from wetting and drying of natural hydraulic mortars of mix ratio 1:1, 1:2, 1:3 and 1:4 (lime:sand) over a period of 28–180 days.

Previous results indicate that 28 days is sufficient for the carbonation process to have initiated throughout the entire volume of the sample [8]. At all curing times the dewatered hydraulic lime mortar was consistently stronger in compression when compared to its non-dewatered equivalent. Subjection of a lime mortar to wetting and drying of a lime
mortar may also significantly influence its compressive strength. The magnitude of this effect was determined by a study of NHL3.5 mortars subjected to alternate cycles of wetting and drying, figure 7 [11]. Much of the increase in strength was found to occur during the initial 28 days of exposure. The observations from this study were consistent with a mechanism for sample strength increase which relies on the transport of water through the structure. Hydraulic lime mortars harden by the combined effect of hydration and carbonation. The hydration reaction is rapid when compared to the carbonation process and involves the formation of a silicate phase which is represented by equation 1.

\[
2\text{Ca}_2\text{SiO}_4 + 4\text{H}_2\text{O} \rightarrow \text{Ca}_3\text{Si}_2\text{O}_7 \cdot 3\text{H}_2\text{O} + \text{Ca(OH)}_2
\]

Silicate phases were most readily identified in cycled samples compared to the equivalent control batch. This suggested that the cycling regime encouraged hydration and consequently an increase in strength. The phenomenon of autogeneous healing has been reported in concrete exposed to wetting and drying. The main cause was attributed to secondary hydration of non or partially reacted cement particles and the formation of calcium carbonate as a result of the reaction of unhydrated cement and migrant carbon dioxide dissolved in the pore water [17,18]. The observation of silicates within pores of the cycled lime mortar suggested that cycling promoted the precipitation and growth of these phases [8].

Concurrently with hydration, and over a longer period, carbonation of calcium hydroxide provides long term strength. Wet and dry cycles provide the optimum conditions for carbonation to prevail [19,20]. Here this is illustrated in figure 8 a to c. Figure 8a represents the pores in a dry mortar, diffusion of carbon dioxide can occur but there will be insufficient water present to promote carbonation. During wetting the sample first absorbs water and then becomes saturated as shown in figure 8b. Water now blocks the pores and the diffusion rate of carbon dioxide is approximately 10,000 times slower than that in air, thus the rate of carbonation is low [11]. During the drying process the pore water gradually evaporates. At a critical point the pores will contain a thin film of water and allow the diffusion of carbon dioxide. This critical point also occurs during the initial process of wetting and absorption. These are the optimum conditions for carbonation,
figure 8c. The formation of silicates within the structure is also expected to limit the water permeability which may reduce the rate of carbonation. The results reported in this paper provide evidence that the process of wetting and drying influences both reactions. The creep measurements show that the process of deformation of a NHL3.5 1:2 mortar over a period of 180 days can be divided into a number of stages, figure 9 [21]. During the first stage, a very high rate of deformation occurred within two days of loading. This was believed to be associated with bedding-in of the sample as asperities on the sample surface were flattened by the smooth loading platens. Following the initial bedding-in period a deformation in the form of two essentially linear stages identified as primary and secondary creep was observed [22,23].

![Graph showing creep stages](image)

Figure 9. General form of strain-time curve for material subject to deformation [19].

Attempts have been made to apply a mathematical expression to describe the data in this study. The highest correlation coefficients of 0.96 to 0.97 were obtained from the logarithmic equation (2) where $\varepsilon$ is creep strain, $t$ is time and $A$ and $B$ are coefficients. Values of $A$ and $B$ for logarithmic equations fitted to each of the mortar specimens tested are given in Table 1.

$$\varepsilon = A \ln t + B$$  \hspace{1cm} (2)

Here $A$ may be related to the degree of carbonation and $B$ is a physical property of the mortar. Creep is defined as the time dependent deformation of a specimen under load therefore the difference between the strain rates in the loaded and unloaded samples represents the creep rate. This is illustrated in figure 9. However the creep rate was observed to decrease rapidly with time during stage I, and was essentially zero during stage II suggesting that the deformation exhibited during the latter stage is due to a shrinkage process.
Table 1 Coefficients A and B from exponential equations fitted to creep/shrinkage data

<table>
<thead>
<tr>
<th>Load, N</th>
<th>Load as percentage of 7 day strength</th>
<th>Coefficients from logarithmic equation</th>
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<tr>
<td></td>
<td></td>
<td>A</td>
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<tr>
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<tr>
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<tr>
<td>234</td>
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<td>-0.2959</td>
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5. CONCLUSIONS
The following conclusions have been drawn:

1. Dewatering results in a dramatic increase in strength of the hydraulic mortars when comparisons were made at 14, 28 and 56 days with non-dewatered equivalent samples.
2. It is suggested that dewatering of mortars prepared with hydraulic binders increases the rate of crystallisation of hydrated silicate phases within the matrix, influencing the mechanical strength.
3. Wetting and drying increases the compressive strength of natural hydraulic lime mortars of classifications 3.5 by increasing the rate of hydration and carbonation.
4. Time dependent deformation was observed in NHL3.5 lime mortars attributed to a load dependent creep mechanism and a shrinkage mechanism which is independent of load. From figure 8 the load dependent creep component was most significant during the first fourteen days of stiffening.
5. A logarithmic creep expression of the form $\varepsilon = A \ln (t) + B$ gave the best fit to the data, where $\varepsilon$ is strain, $t$ is time and $A$ and $B$ are constants. A linear relationship was found between the coefficient $A$ and applied load suggesting that this constant is load dependent.

6. ACKNOWLEDGEMENTS
The authors would like to thank Hydraulic Lias Limes and Hanson Cement for supplying the raw materials and sample manufacture.

7. REFERENCES


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