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Original article

A comparison of standard and realistic curing conditions of natural hydraulic lime repointing mortar for damp masonry: Impact on laboratory evaluation

Lucie Fusade*, Heather A. Viles

School of Geography and the Environment, University of Oxford, Oxford OX1 3QY, UK

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ABSTRACT

Environmental conditions can affect the curing and performance of lime mortars. Especially in the case of natural hydraulic lime (NHL) mortars to be used for repointing in exposed conditions, it is essential to assess what if any differences these environmental conditions would make to mortar properties through laboratory evaluations before repointing work begins. This study considers a specific historic environment: traditional masonry exposed to high humidity and rainfall, with a particular focus on Devon. Realistic curing conditions (as likely found on-site) of 15 °C, 85% RH, representing an average of summer climate in Devon were compared with standard recommended laboratory conditions of 20 °C, 65% RH. A range of mixes, representing some conservation pointing mortars, was prepared using NHL 2 (St Astier), quartz sand, and crushed Portland limestone in 1:3 and 1:2 binder to aggregate ratios. The influence of curing conditions on carbonation depth, strength development, internal textural structure, pore structure and water uptake at 28 and 90 days is discussed (called here early and medium ages) and the response of NHL mortars to this humid environment during evaporation and salt crystallisation have been assessed. Results show that significant differences are found in laboratory evaluations of mechanical properties of the same NHL mortar exposed to different curing conditions especially at an early age and for mortar made with quartz sand. Laboratory evaluation should be made on samples cured under realistic conditions if information on the early to medium-term (up to 90 days) characteristics of NHL mortar is required. Overall, realistic humid curing conditions help NHL mortars gain good internal structure more quickly, minimising the risk of early failure of pointing mortar exposed in a harsh humid environment.

1. Introduction

1.1. Why repoint damp historic buildings?

Throughout the centuries, lime mortars have been used under a very wide range of environmental conditions. In England, where the climate is often wet, lime mortar is found in many types of historic masonry built before the 1850s. Orientation and topography are key in assessing the impact of wind-driven rain on historic masonry walls [1]. When dealing with historic buildings highly exposed to rainfall and with interior dampness problems, conservation officers first recommend maintenance of the building features, such as the drainage system, conditions of the roof elements, etc. [2,3]. When this has been ensured but interior dampness is still present, conservation work on the masonry is considered, such as rendering, grouting or repointing [4]. Previous research has shown that a render, flat work or harling, often helps to hold most of the water until the rain stops when evaporation will allow the masonry to dry out [5]. However, render can be aesthetically disruptive and therefore repointing has two clear advantages: firstly, it is far less disruptive since it ensures a minimal intervention to the historic masonry [6]; secondly, it is less costly [5].

In a masonry unit, one of the main roles of pointing mortar in a joint is to draw moisture out of the wall to help it dry out [7,8]. Observations from practitioners (such as conservation officers and craftsmen) and scientific research have both identified that failure of weathered original pointing mortar or previous inappropriate interventions lead to rainwater and moisture ingress through the joints [7]. In these cases, mortar needs to be replaced by repointing. The performance of the pointing mortar is therefore critically important to reduce water penetration in the masonry [6,9,10].

* Corresponding author.
Adresse e-mail : lucie.fusade@ouce.ox.ac.uk (L. Fusade).

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In addition to being an efficient solution in dealing with water ingress, it is also important to consider that repointing meets some main conservation principles. Indeed, it is recommended that conservation interventions for repair retain the heritage values of a site by respecting the appearance and ensuring minimal intervention on the original fabric, as well as long-term stability and sustainability [6,11,12]. Research has highlighted that material changes, through weathering or decay, or through intervention replacing the original materials, can be associated with a change of values of the building [13,14]. This is where repointing should be carefully thought through, from philosophical to practical considerations and functional requirements [10]. If new materials are introduced – being different to the originals – this should be for preserving the building as a whole and meeting its current needs and conserving its values. The performance of repointing mortar should therefore be carefully assessed by characterising the mechanical and physical properties of mortars and how they develop and respond to a specific environment, prior to application. With this in mind, and to avoid drawing too general conclusions from one scientific study, we focus here on a specific historic environment: traditional masonry, as for instance found in church towers, exposed to high humidity and rainfall in places such as Devon, in the south west of England.

1.2. The environment (temperature, relative humidity and rainfall) affects hardening of lime mortar

There are several examples of pointing mortar, which have resisted humid environments [15]. All repair interventions on a historic building, such as repointing, have to consider the climatic environment [3,9]. This is especially important in the context of climate change: research has shown that due to predicted increase of rainfall and wind-driven rain in some locations, more water will impact masonry, remaining wetter longer [16] and increasing the wetting and drying cycling [17]; which would impact the overall performance of lime mortar.

Environmental conditions have an impact on the early days of lime mortar, during the setting and hardening process of the fresh mortar. Research has shown that this hardening process, also called carbonation, is governed by the temperature and relative humidity (RH) of the environment [18]. This combination of temperature and relative humidity in the environment in which lime mortar will harden is called the curing condition. As the diffusion of CO_2 in the pores depends on the water content, climate can directly influence the rate of carbonation [19]. In addition, variations in temperature and relative humidity produce different reactions with lime, such as mineral transformation of portlandite [$Ca(OH)_2$] into calcite ($CaCO_3$) that will have an important effects on the internal development and structure of lime mortar [20,21].

However, when testing mortar, recommended laboratory curing conditions of 65% RH, 20 °C [22] are used which are often quite different from the on-site environmental conditions. There is no consensus on the optimum temperature for carbonation, sometimes suggested at 15 °C [18], or 20 °C [19] and researchers suggest that the most favourable RH for carbonation to occur is between 55% and 75%, while carbonation would reduce between 55% to 45% and 75% to 85% [24]. Indeed, diffusion of CO_2 is slower in water than in air, as water blocks the porous system [19,25,26], so research has shown that for carbonation to occur well, masonry exposed to rain should be sheltered [37]. Carbonation is also not the only property that should be considered. Some studies [21,23] have compared the maritime or outside environmental conditions with standardised curing conditions, and identified that higher RH allows a higher rate of carbonation and hydration process [23] and contributes to reducing pore size [21]. However, these environmental conditions were very site specific and no implications for laboratory evaluation was drawn.

Although curing conditions have to be considered on a case by case basis, no research has yet clearly stated whether curing conditions make a significant difference both to the early development of the internal structure and performance of lime mortar, and whether it is important to cure samples under realistic conditions for laboratory characterisation. In general, for humid temperate environments, such as in Devon, the combined effect of low temperature and high humidity can lead over time to the saturation of the mortar, which then presents the risk of failure [18]. This is why prior scientific evaluation and characterisation of how the mortar would develop and perform in such environments is necessary to ensure that the repair mortar will be compatible with the masonry unit [27] and fulfil the technical requirements of a specific environment [28].

2. Research aims

This study compares curing under realistic environmental conditions, such as those found in Devon, with curing under standard laboratory conditions to understand better what, if any, differences these environmental conditions would make to the natural hydraulic lime mortar structure and durability. It focuses on evaluating the development of the internal structure of mortar samples after curing for 28 and 90 days, producing early and medium-aged mortars respectively. Finally, using easily available and commonly used analytical methods it evaluates whether it is of significant importance to use realistic curing conditions for laboratory characterisation, if the mortar is to be applied on a specific site or building.

3. Materials and methods

3.1. Mortar preparation and curing

3.1.1. Compositions of the mortar mixes and properties of the fresh mortars

A range of mixes, representing some conservation pointing mortars, was prepared by a professional mason. Mortars were made using a natural hydraulic limes (NHL) 2 (St Astier). When using lime under humid built environmental conditions, NHLs are often favoured as they allow for a hydraulic set while being feebly hydraulic and without gaining too much strength [29]. They harden through a two-phase process: initially a 'hydraulic set', resulting mainly from the formation of calcium silicate hydrates (C-S-H) and calcium aluminate hydrates (C-A-H), followed by carbonation [30]. Formations of these hydrates gives NHLs their hydraulicity, which means that they need moisture in the air to set and therefore have the capacity to cure under humid environmental conditions [29]. This initial setting also gives them an early strength gain, essential for resisting extreme environmental conditions [31].

Two types of aggregates were used, a well-graded sharp quartz sand (Chardstock) and porous, sand-sized crushed limestone (Portland). The grain size distribution of each aggregate was measured by sieving and showed a similar well-graded distribution (0.76 to 4 mm), with the crushed limestone having more smaller grains (between 0.15 and 0.60 mm) than the quartz sand (Appendix A, Supplementary Fig. 1). Two binder to aggregate ratios by volume were used, a binder-rich one (1:2) and a standard repointing one (1:3). To follow the 1:2 and 1:3 binder to aggregate ratio by volume, the weight needed for each aggregate and binder was adapted for each mortar mix based on bulk densities.

During mixing, water was added based on the experience of the mason to obtain similar consistency [135 mm (± 10) being the

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Table 1
Summary of mortars prepared and properties of fresh mortars.

Mortars	Aggregate	Binder:aggregate ratio (by volume)	Water:binder ratio	Flow (mm)	Initial moisture content (%)
SI3	Quartz sand	1:3	0.79	131	14.74
SI2	Quartz sand	1:2	0.45	132	14.52
CA3	Crushed limestone	1:3	0.11	145	14.58
CA2	Crushed limestone	1:2	0.19	138	16.02

targeted flow for repointing mortar consistency]. Flow tests were carried out on each mix with a flow table (Matest) following BS EN 1015 5-3:1999 [32]. The initial moisture of each aggregate was 10.43% for the quartz sand and 1.38% for the crushed limestones, explaining the need for less water for the CA mixes, which therefore have the lowest water:binder ratio (Table 1). The Initial Moisture Content (IMC) was recorded with a Moisture Analyser (A&D MX50) in %.

3.1.2. Curing conditions and sample sizes

Specimens were divided into two groups and placed in different curing conditions in two environmental chambers (Sanyo-FE 300 H/MP/R20 and Sanyo-MLR-351). The two curing conditions were chosen as follows – one recommended by standards (SC) at 20 °C (± 1 °C), 65% (± 8%) RH until testing [33], and the other chosen to represent realistic conditions (RC) of an average of summer climate in Devon 15 °C, 85 % (these values fluctuate between day and night). Data was taken from the weather station at Chivenor. Calculation shows that these two curing conditions have similar absolute humidities (0.011 kg/m^3). Therefore, any differences of properties between mortars cured under these different conditions is due to the lower temperature or higher RH.

Specimens were made in the laboratory in prisms of 40 mm × 40 mm × 160 mm using polystyrene moulds (Appendix A, Supplementary Table 1). All specimens were demoulded after 5 days and tested at 28 and 90 days (chosen to represent 'early age' and 'medium age' mortars, respectively).

3.2. Mechanical properties of hardened mortars

3.2.1. Carbonation depth

The mean carbonation depth was measured by spraying phenolphthalein on freshly broken specimens. Phenolphthalein is a pH indicator commonly used for determination of carbonation depth, that turns pink above a pH of about 9.3 [34]. When applied on the surface of a freshly broken mortar, the uncarbonated area, composed of calcium hydroxide [$Ca(OH)_2$], is stained pink, whereas the carbonated area, composed of calcium carbonate ($CaCO_3$), remains unstained. The depth of the unstained area was then measured from the four faces with a calliper (0.01 mm), giving a mean value for each of the three replicates.

3.2.2. Compressive strength

The compressive strength was measured according to BS EN 1015-11:1999 [35] on five half prisms (cut from prisms of 40 × 40 × 160 mm) at 28 days and six at 90 days. The testing machine (Matest Unitronic Load Frame Tester) was used with a 10 kN load cell and a loading rate of 50 N/s. Results are reported as the mean of all replicates in N/mm^2.

3.2.3. Ultrasonic pulse velocity

Ultrasonic measurements were carried out on three prisms (40 × 40 × 160 mm) of each mix, with a Portable Ultrasonic Non-destructive Digital Indicating Tester (Pundit Lab, Proceq, UK) with 54 Hz frequency transducers. Direct transmission was used with transducers placed at each end of the specimen on the long axis of the prisms. The propagation velocity (vp) (km/s) was measured and results reported as the mean of three measurements on each three replicates of each mix.

3.3. Pore structure

3.3.1. Mercury intrusion porosimetry (MIP)

The pore size distribution was measured using mercury intrusion porosimetry with a Porosimeter (Quantachrome PoreMaster 33 Hg), with low gas pressure range between 0.2–55 psi and high hydraulic pressure range up to 2110 psi. MIP was performed on one replicate of each mortar mix, previously oven dried at 60 °C.

3.3.2. Pore structure

Optical microscopy of thin-sections impregnated in blue resin was performed using a Olympus BX43 microscope at ×10 magnification with transmitted light. One thin section of each mix was made and observed.

3.3.3. Open porosity

The open porosity (*op*) was evaluated following the gravimetric method adapted from the standard BS EN 1936:2006 [36]. Five half prisms were oven dried for 24 hrs at 70 °C (± 2 °C) to constant mass (*md* in g). Samples were then placed in a desiccator under vacuum at low pressure (less than 15 mm Hg) for one hour and allowed to soak in distilled water for 24 hrs at ambient temperature, enabling determination of the saturated mass (*ms* in g) and immersed mass (*mh* in g). The open porosity (%) was calculated as the mean of five replicates with the formula (1):

$$op = \frac{ms - md}{ms - mh} x100 \quad (1)$$

3.3.4. Capillary absorption

The determination of water absorption coefficient due to capillarity of hardened mortar followed the standard tests BS EN 1015-18:2002 for the design and size of samples[22] and EN 1925: 2000 for the interval of measurements for highly absorbent stone [37]. Samples were oven dried for 24 h at 70 °C (± 2 °C) to constant mass (*md* in g), cut in half, sealed using a moisture and vapour-proof sealant (Parafilm M), and placed in 3 mm of distilled water. The mass of each of the six replicates at defined time was determined using a balance (Sartorius) at 0.01 g precision. The increase in mass (m_1 in g) by the surface immersed (A) (m^2) of each replicate was expressed as a function of the square root of time ($\sqrt{t_1}$) in minutes (mn$^{0.5}$). The water absorption coefficient by capillarity (WACC) (g/m^{2}mn$^{0.5}$) was determined by formula (2) given by the standard as a mean of the six replicates:

$$WACC = \frac{m1 - md}{A.\sqrt{t1}} \quad (2)$$

3.4. Performance in humid environment

3.4.1. Drying behaviour

The drying rate was monitored for 120 hours under two environmental conditions: laboratory at 23 °C (± 2 °C), 55% RH (± 5%), and realistic (Devon) at 15 °C, 85% RH (± 5%). Three replicates of

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Table 2
Summary of the *t*-test results of property development under standard conditions (SC) and realistic conditions (RC).

Conditions	Mortars	Carbonation ($n=12$)	Compressive strength ($n=5$ to 6)	Pulse velocity ($n=9$)	Open porosity ($n=5$)				
		28 d	90 d	28 d	90 d	28 d	90 d	28 d	90 d
SC	SI3	7.2E-09[a]	9.9E-05[a]	2.6E-03[a]	1.1E-01	1.0E-05[a]	9.0E-03[a]	3.5E-02[a]	6.2E-01
RC									
SC	SI2	1.6E-08[a]	5.6E-01	1.7E-03[a]	1.5E-01	4.6E-08[a]	6.1E-04[a]	5.0E-02[a]	2.3E-04[a]
RC									
SC	CA3	7.5E-06[a]	5.5E-02	1.5E-01	2.8E-01	7.2E-06[a]	9.8E-01	2.9E-01	9.0E-03[b]
RC									
SC	CA2	8.7E-04[a]	8.0E-01	5.6E-02	8.3E-01	2.5E-05[a]	9.5E-01	1.8E-03[a]	1.8E-03[b]
RC									

SC: standards conditions; RC: realistic conditions.
N indicates the number of measurements taken. Statistically significant differences occur mostly after 28 days and for both age in SI mortars.
[a] *P*-values < 0.05 indicate that the means are significantly different.
[b] Indicate that one replicate was considered to have failed and not included in the calculation.

each mix in half prisms, having previously been fully saturated under vacuum, were used. The change of mass (m_1 in g) was recorded every 2 to 3 hours for the first 12 hours then every 15 hours (± 3 hrs) using a balance (Sartorius) at 0.01 g precision. For the 28 days test, two drying sets were done, one until 52 hours, the other one from 50 hours until the end. The water content (Wc) (g/cm^2) was calculated as a mean of the three replicates using formula (3):

$$\mathrm{Wc} = \frac{(m1 - md)\,pw}{V} \quad (3)$$

Where md (g) is the oven dry mass of the replicates, ρw (g/cm^3) the density of water at 20 °C and 15 °C degrees, and V (cm^2) the volume of the sample [38].

3.4.2. Salt uptake

The salt uptake and crystallisation test was performed according to Gulotta et al. [39] who followed a version of RILEM MS-A.2 procedure. Specimens (half prisms) were tested at 360 days. Eight replicates of each mortar were used and divided in two groups: four having been cured under standard conditions for 360 days, and four cured under realistic conditions for 90 days and then stored under standard conditions. Specimens were oven dried at 60 °C for 24 hrs and sealed on the long faces with a vapour-proof sealant (Parafilm M). Immersion for 2 hours in a 10% sodium sulphate solution, followed by a drying period of 22 h at 20 °C, 80% RH, constituted a daily cycle. Four daily cycles formed a weekly cycle, after which the weight of each specimen was recorded. Three weekly cycles were performed.

4. Results

In order to determine whether the same mortar mix exposed to different curing conditions would develop different properties, it is important to statistically compare these results. Because all comparisons are paired (comparing one mix, under two curing conditions, for each age and type of testing) paired t-tests were used (Table 2). Realistic curing conditions make a significant difference to the test results for most of the mortars after 28 days of curing. Less significant differences are shown after 90 days of curing. The types of differences and the reasons for them are further explained for each test below.

4.1. Mechanical properties

4.1.1. Carbonation depth

As seen previously, the hardening and setting process, involving a hydraulic set followed by carbonation, is highly influenced by the curing conditions (temperature, RH) of the mortar [19,20], but also by the water:binder ratio [25]. The phenolphthalein test gives an indication of the carbonation depth, highly influenced by porosity, but cannot be directly correlated to the carbonation profile and hydration [26].

Fig. 1 shows that mortars cured under realistic conditions have a significantly higher carbonation depth at 28 days, between 1.10 and 2.70 mm deeper than mortars cured under standard conditions. Although we could argue that higher RH would favour the hydration of the anhydrous calcium silicates and aluminates [24] this may be offset by the reduction in temperature. However, at 28 days, the values are too low and the mortars too young to draw clear conclusion. At 90 days, the carbonation depth is similar or slightly higher under standard conditions. However, as Table 2 shows, these differences are not statistically significant. In addition, binder-rich mortars (SI2 and CA2) experience a lower carbonation depth because in these mixes more binder has to set and carbonate.

4.2. Compressive strength

When water evaporates from the fresh mortar, pores are created giving access to CO_2 which contributes to the mineral transformation of portlandite [$Ca(OH)_2$] into calcite ($CaCO_3$) [38,40]. This process is directly impacted by the environment and especially by the amount of moisture of the air, as water slows down the transport of CO_2. Higher porosity and higher calcite contents often equal higher compressive strength [41].

For quartz sand rich mortars, those exposed to realistic conditions show significantly higher compressive strength at 28 days than those cured under standard conditions (Fig. 2). This is likely due to higher hydration as lower temperature could increase the mineral reaction responsible for strength gain [18]. However, at 90 days, the variation within samples is too high so that no significant difference can be determined (Table 2). The binder-rich mix (SI2) shows higher compressive strength, as noted in previous research [41]: as lime is a porous material, more CO_2 can access lime and carbonate, gaining strength. In mortars made with crushed limestone no significant differences are seen between the two curing conditions and between the two binder:aggregate ratios (Fig. 2, Table 2).

4.2.1. Internal structure – pulse velocity

Research has shown that ultrasonic pulse velocity (UPV) can be used as a non-destructive technique to assess internal material changes or decay in porous building materials used in cultural heritage, such as lime mortars [40,42,43]. UPV gives an indication of the presence of cracks and overall of the internal quality of the material. Previous research has shown the correlation of compressive strength with UPV and that carbonation degree and porosity

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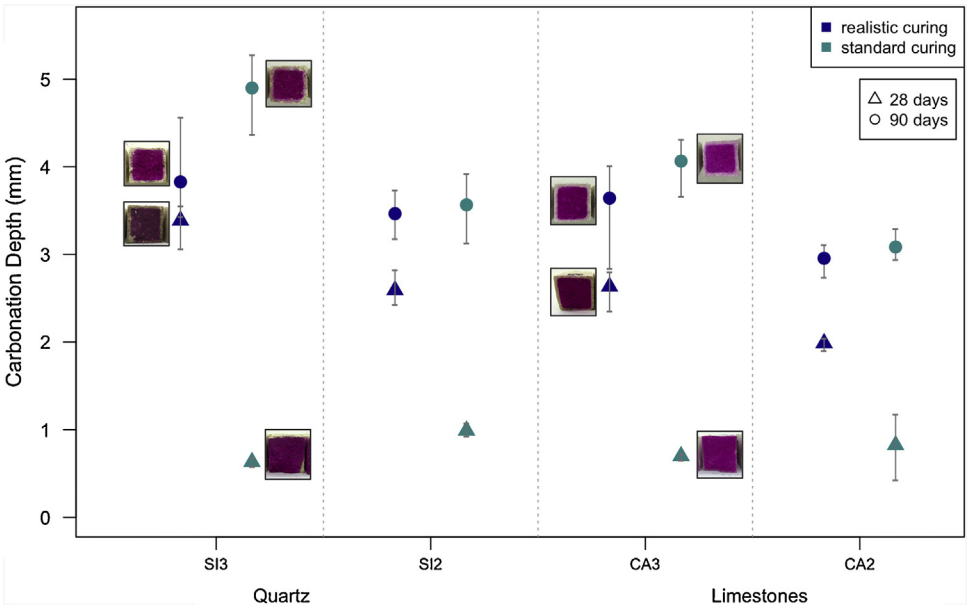

Fig. 1. Comparison of the carbonation depth (mm) of samples cured under standards conditions (SC) and realistic conditions (RC) at 28 and 90 days ($n = 3$). Error bars indicate the minimum and maximum values. The pictures show the uncarbonated area (stained) and the carbonated area (unstained) after phenolphthalein spraying. The same patterns were seen on the SI2 and CA2 samples so are not presented here.

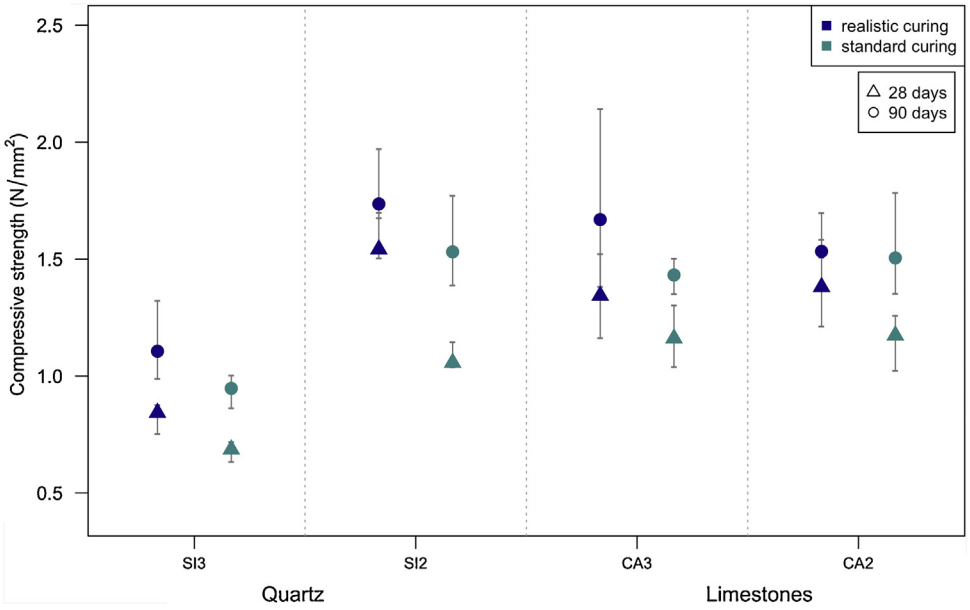

Fig. 2. Development of compressive strength (N/mm^2) under standard and realistic curing conditions ($n = 5$ or 6) showing higher early strength gain under realistic conditions (RC). Error bars indicate minimum and maximum values.

affect UPV the most [40]. In this study, we use UPV as a proxy to assess what the compressive strength and carbonation depth could suggest: a denser internal structure is formed more quickly in the mortar when exposed to realistic humid environmental conditions.

The general trend shows indeed that mortars cured under realistic conditions (RC) have a higher velocity propagation (vp) after both 28 and 90 days of curing (Table 3). A higher propagation velocity represents a denser internal structure, as pores and cracks would slow down the vp. This higher vp for RC mortars, could suggest that their internal structure is denser, more compact, or that their degree of carbonation is higher [42]. This difference is particularly significant at 28 days for all mortars and at both ages for SI mortars (Table 3). In all mortars, the decrease of vp from 28 to 90 days may be a sign of more cracks or more pores.

Overall, CA mortars shows a higher vp than SI mortars, explained by their denser structure and fewer shrinkage cracks. The vp of CA samples under RC does not change from 28 to 90 days, which could imply that their internal structure is mainly formed during the first 28 days of curing. The differences between mortar mixes with different aggregates may be explained by the denser structure of CA mortars which have pore volumes concentrated in the same range.

Fig. 3 highlights that when the compressive strength of samples increases over time it impacts the internal structure of the mortar resulting in a higher propagation velocity (around 1.4 km/s), except for SI3, for which vp decreases. Propagation velocity therefore confirms the hypothesis that mechanical properties develop quicker in mortars exposed to realistic curing conditions.

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Table 3
Summary of the ultrasonic pulse propagation velocity results (km/s) ($n = 9$). A higher vp represents a denser and more intact internal structure.

Conditions	Mortars	28 days		90 days	
		Mean (km/s)	SD	Mean (km/s)	SD
SC	SI3	0.843	± 0.027	0.498	± 0.160
RC		1.020	± 0.063	0.687	± 0.091
SC	SI2	1.100	± 0.032	0.945	± 0.128
RC		1.471	± 0.076	1.399	± 0.204
SC	CA3	1.272	± 0.045	1.405	± 0.080
RC		1.471	± 0.072	1.407	± 0.270
SC	CA2	1.181	± 0.053	1.404	± 0.063
RC		1.410	± 0.093	1.384	± 0.132

SC: standards conditions; RC: realistic conditions.

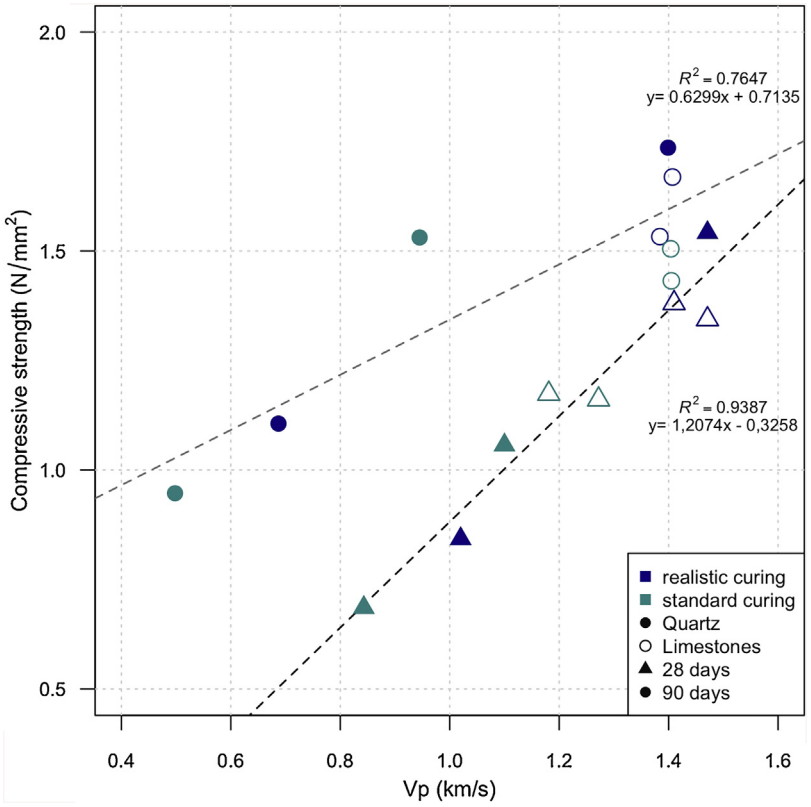

Fig. 3. Relationship between vp, age of testing, and compressive strength. Regression lines show a higher correlation between compressive strength and vp at 28 days (black dashed line) than at 90 days (grey dashed line).

It is interesting to see whether the impact of curing conditions can also be seen in the pore structure, as research has shown a relationship between mechanical properties and pore structure [44].

4.3. Pore structure

4.3.1. Pore size distribution and pore structure

In this study we consider pores between 1 to 1,000 microns (0.001 to 1 mm), in the range of micropores to be mainly relevant to absorption by capillarity [45] and called "capillary pores", and pores from 0.1 to 1 microns to be "smaller capillary pores". Mortars made with quartz sand (Fig. 4a) show a lower overall porosity than the ones made with crushed limestone (Fig. 4b), but with a higher proportion of larger capillary pores (between 8 and 40 microns). Most of the pores in the mortars made with crushed limestone are in the fine capillary pores range. This likely explained their dense structure. Mortars of 1:3 ratio have more of a bimodal pore size distribution, whereas the binder-rich mortars (1:2 ratio) show a unimodal distribution, with few larger pores.

The different curing conditions do not translate into important differences in the same mortar mix, as the pore size distribution remains similar. Cured under realistic conditions, SI3 mortars show a decrease in the volume of smaller capillary pores, between 0.03 to 0.5 μm, and SI2 mortars show a decrease of capillary pore, between 0.08 and 5 μm (Fig. 4a). In mortars made with crushed limestone cured under realistic conditions the proportion of pores between 0.02 to 2 and 5 μm slightly increases, which could show an even denser structure (Fig. 4b).

Fig. 4a′ and b′ illustrate that mortars made with crushed limestone have a denser matrix, fewer shrinkage cracks and no coarser pores than those found in the mortars made with quartz sand. The blue colour of the matrix in Figure 4b' shows that the mortar is more porous. In SI samples, more shrinkage cracks are visible on samples cured under standard conditions, but in general, the curing conditions have no clear influence on the interconnectedness of the pores.

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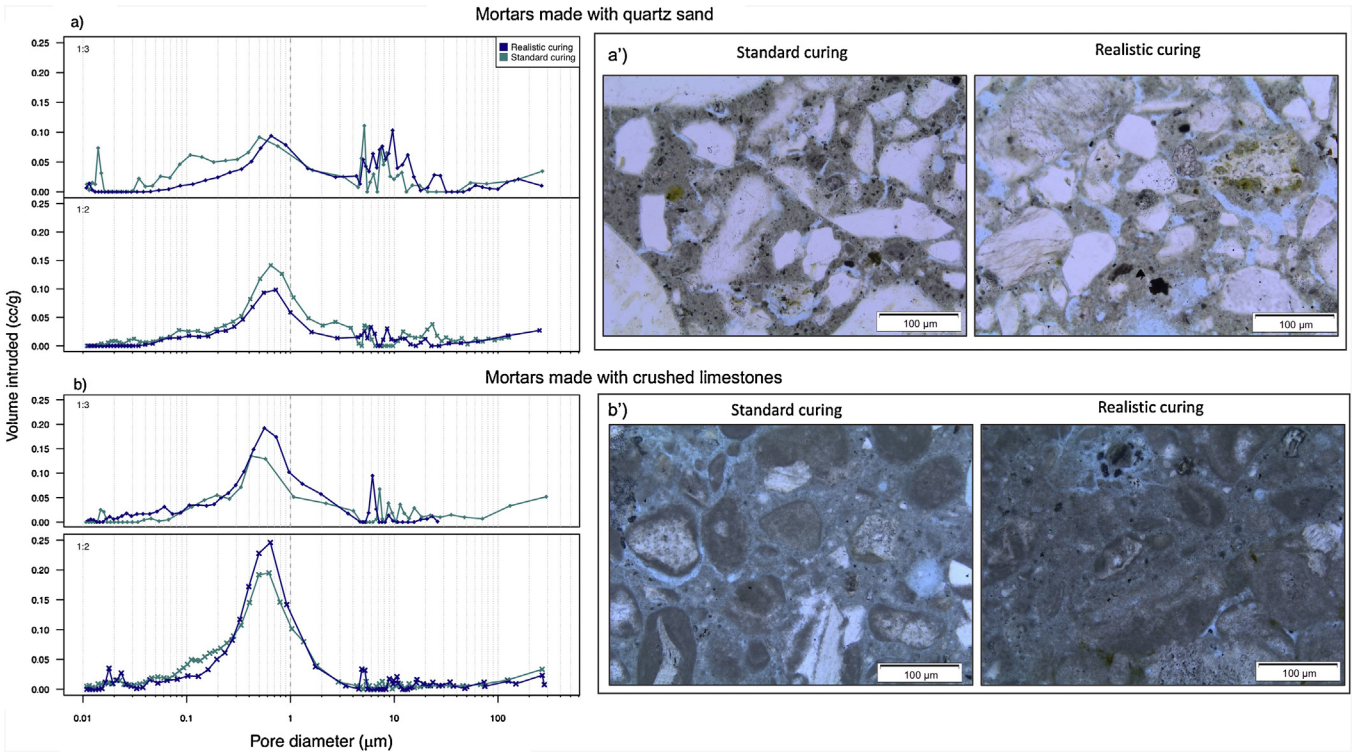

Fig. 4. Pore size distribution with MIP of: a: mortars made with quartz sand; b: mortars made with crushed limestone, the dashed line shows the limit of capillary pores from 1 µm to 1 mm. Thin sections impregnated with blue resin showing the pore structure of: a′: mortars made with quartz sand; b′: mortars made with crushed limestone, under petrographic microscope with transmitted light at ×10 magnification. The scale bar shows 100 microns.

4.3.2. Open porosity

The open porosity is the part of the total porosity connected to the surface of the samples, and so influences water transport properties [38] and gives an indication of the near surface (rather than internal) changes. When mortar carbonates and as minerals transform over times, the porosity also decreases. During the curing and hardening process, evaporation of water leaves pore spaces, and the development of a complex pore structure [38,40]. The expected decreased or unchanged porosity from 28 to 90 days is slightly visible under realistic conditions (Fig. 5). However, under standard conditions the average open porosity seems to be unchanged or increased over time. Significant differences are observed in open porosity between mortars cured under standard and realistic conditions after both 28 and 90 days of curing in most cases, although only at 28 days for SI3, and only at 90 days for CA3 (Table 2). Overall, RC mortars at 28 days have a higher open porosity than SC ones, except SI3, and at 90 days, a significantly lower open porosity (Table 2 and Fig. 5).

Fig. 5 also highlights that mortars made with crushed limestone have a much higher open porosity than those made with quartz sand. Because they required less water to obtain similar consistency (Table 1), the higher porosity cannot be explained by a higher amount of water in the mix, but seems to be brought by the porous calcitic aggregates themselves. This can also be seen in Figure 4b' where no clear pores are found, but where an overall porous matrix is evident.

No clear correlation can be established between the carbonation depth and the open porosity. However, at 28 days, except for SI3, higher carbonation depths are linked to lower open porosity (Appendix A, Supplementary Fig. 1). This could correlate with the fact that RC mortars seem to initially develop more quickly and then slow down after 28 days. It seems however, as expected, that when the open porosity is low, the compressive strength is higher.

4.3.3. Capillary absorption

Fig. 6 indicates that mortars cured under standard vs realistic conditions behave similarly in terms of capillary absorption. However, the calculation of the coefficient of capillary absorption (Appendix A, Supplementary Table 2) shows that in general RC mortars absorb water more slowly, except CA2 at 90 days. This is particularly true for SI3 mortars and CA2 mortars at 28 days. This very slight difference between mortars cured under different conditions suggests that the capillary structure is not as affected by the curing conditions as the mechanical properties are.

In SI mortars, Fig. 6 shows a slight delay in saturation for binder-rich samples (dashed line) at both 28 and 90 days, and higher absorption coefficient for 1:3 ratio mixes (Appendix A, Supplementary Table 2). In CA mortars, the opposite is seen, the binder-rich mix (CA2) has a higher capillary absorption rate. In general, despite being more porous, mortars made with crushed limestone have a slower capillary absorption. Fig. 4a–b show indeed that the pores of CA mortars are in the smaller capillary pores range, explaining their slower capillary uptake [45,46]. No difference of behaviour is seen between 28 and 90 days, which would suggest that most of the capillary structure is formed at the early days of the mortar curing process.

4.4. Performance in humid environments

In humid environmental conditions it is essential to assess how the mortar will behave under wetting and drying cycles - rain and evaporation. As seen previously, with rainfall predicted to increase with shorter cycles in many places [47], the behaviour of mortar during evaporation is critical to the overall preservation of the building.

4.4.1. Drying behaviour under different environments

The ability of mortar to absorb moisture and then to dry out is essential to ensure that moisture will be drawn out of the masonry

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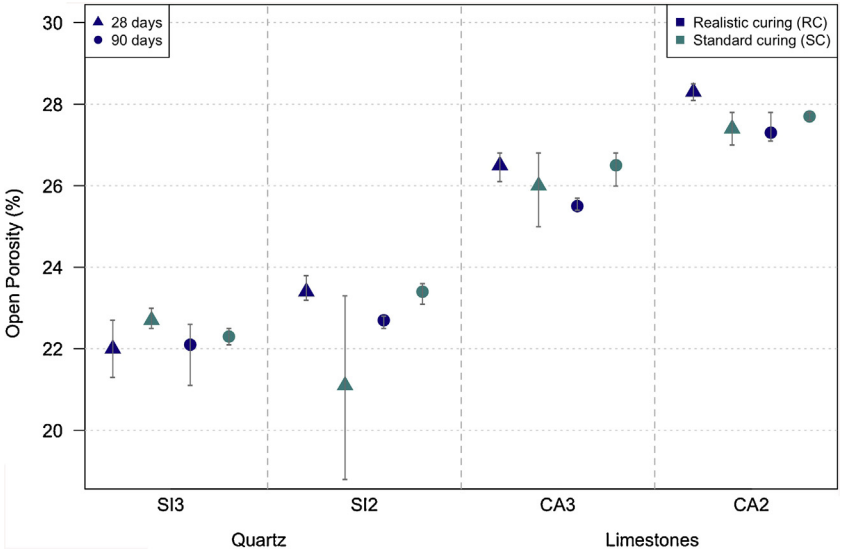

Fig. 5. Comparison of the development of open porosity under realistic conditions (RC) and standard conditions (SC) at 28 and 90 days. Error bars indicate minimum and maximum values.

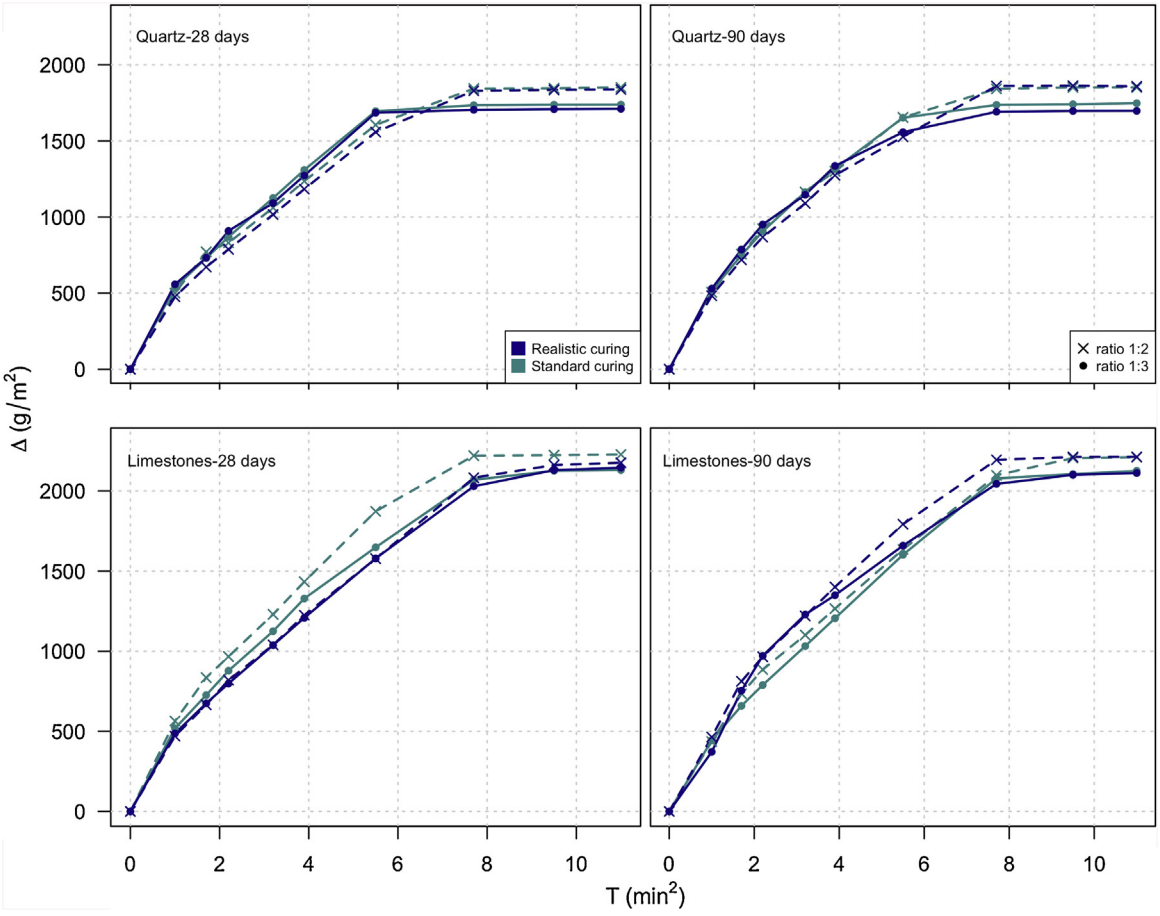

Fig. 6. Capillary absorption of samples under realistic and standard conditions over time. The dashed lines represent the binder-rich mortars (SI2 and CA2).

unit, therefore reducing the threat of ingress of water through the wall to the inside of the building.

Fig. 7 shows no clear differences between drying curves and rates of mortars cured under realistic and standard conditions. The fact that hardly any difference is seen in the drying curves of samples cured under standard or more realistic conditions, suggests that the pore structure responsible for the absorption and desorption of moisture in both liquid and vapour state forms in a similar way when lime mortar is cured under both conditions.

When exposed to realistic humid environment of drying [15 °C, 85% RH (± 5%)], as opposed to drier laboratory conditions, the evaporation rate of all the tested mortars is much slower (Fig. 7). Under laboratory conditions of drying (plain line in Fig. 7), the two stages of drying are visible. For samples analysed after 28 days of curing,

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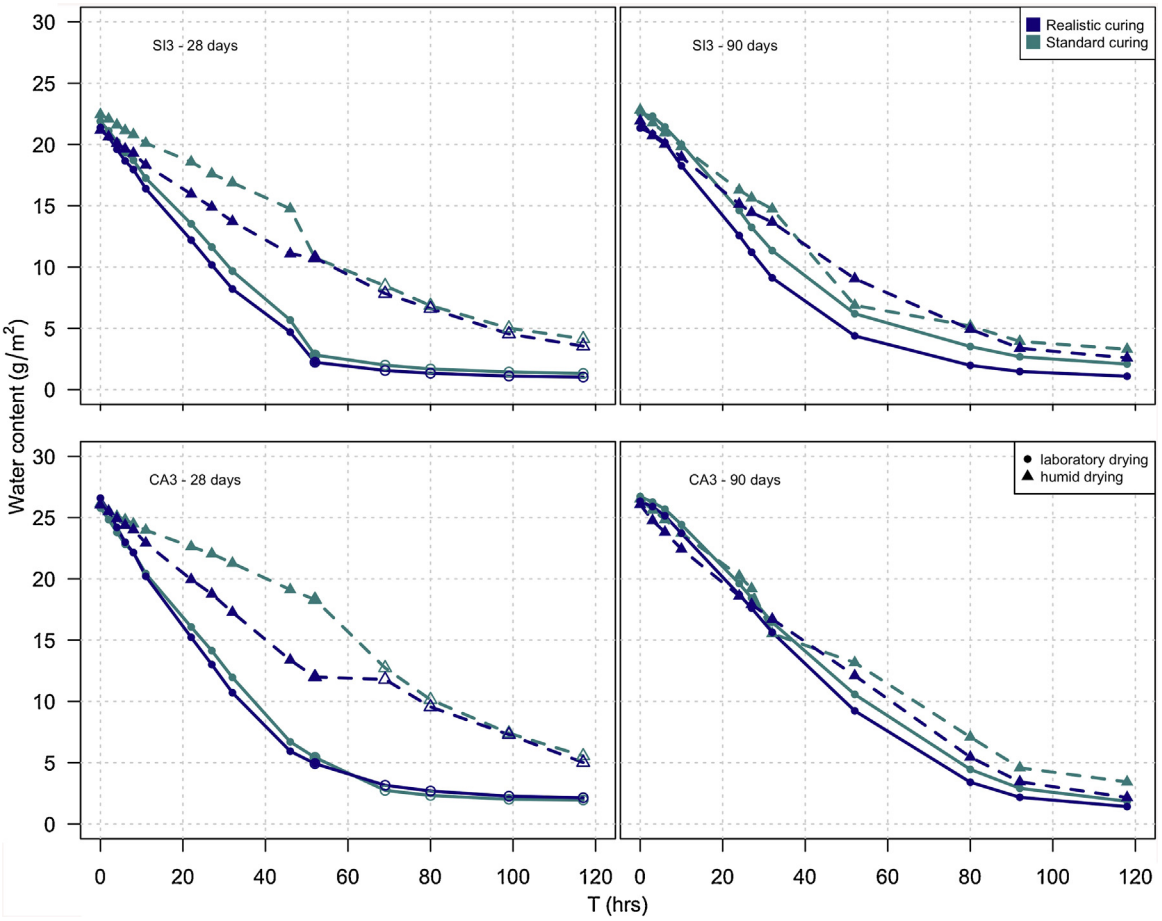

Fig. 7. Drying curves of SI3 and CA3 mortars cured under standard conditions (SC) and realistic conditions (RC) and dried under laboratory (plain line) and humid environment (dashed line). Empty marks at 28 days show the second drying test added to the first one. Data for SI2 and CA2 mortars were similar and are not shown here (Appendix A, Supplementary Fig. 3).

stage 2 starts after approximately 70 hours, and is being delayed to after 80 hours for samples cured for 90 days. Under the two drying conditions, realistic humid environment and drier laboratory conditions, mortars cured for 90 days show a similar drying pattern, especially for CA samples (Fig. 7). The longer stage 1 drying observed in both drying conditions in RC and SC samples at 90 days shows that, in general, longer curing leads to faster drying times. During stage 1, water travels as a liquid to the surface, and so longer stage 1 drying should be beneficial for the whole masonry as the liquid water in the bulk of the wall will have time to travel to the surface and dry out [48]. Finally, CA mortars have a higher water content than SI mortars, and binder-rich mixes as well, which can be related to their general previously observed higher open porosity (Fig. 5).

4.4.2. Salt uptake

In a masonry unit, in order to preserve the historic stones, it is important that the mortar is able to uptake more salt than the surrounding masonry. It is interesting to evaluate whether the different curing conditions of the mortar will make a significant difference to durability when it is exposed to salt crystallization during evaporation. Fig. 8 shows that an increase in mass due to the uptake of both water and salt can be seen over the three weekly cycles for all mortars. The general patterns for all mortars seem to be that those cured under standard conditions, experience a slowing of water and salt uptake at the third weekly cycle, whereas those cured under realistic conditions remain higher, except for SI3 mix (i.e. they take up more water and salt over time). In SI3 samples, the same pattern is seen for SC and RC samples, although RC samples have a higher mass difference.

The increased uptake of salt at the surface of the mortar samples after each weekly cycle is visible in Fig. 9. This is due to the accumulation of salt inside the sample that travels to the surface over time. However, no clear difference is visible between mortar samples which have been subject to standard and realistic curing. However, differences can be seen in the accumulation patterns of mortars made with quartz sand vs those made with crushed limestone, with salt on SI mortars being more granulated around sand grains. For CA mortars, it seems that the salt uptake occurs mainly on carbonated areas: the centres of the samples (uncarbonated area) have indeed less salt visible on the surface. The surface of SI mortars was more affected by weathering due to salt and loss of material.

In general, the salt remained in pores after the test, which decreased the open porosity of samples (Table 4). The decrease in open porosity is more pronounced for mortars cured under standard conditions, except for SI3. This suggests that perhaps for mortars cured under realistic conditions, most of the salt taken up had migrated to the surface, whereas it would have stayed and blocked some pores in the SC mortars.

5. Discussion

5.1. The different curing conditions make a significant difference in some properties

Curing the same lime mortars under two different conditions makes some difference in many properties, especially mechani-

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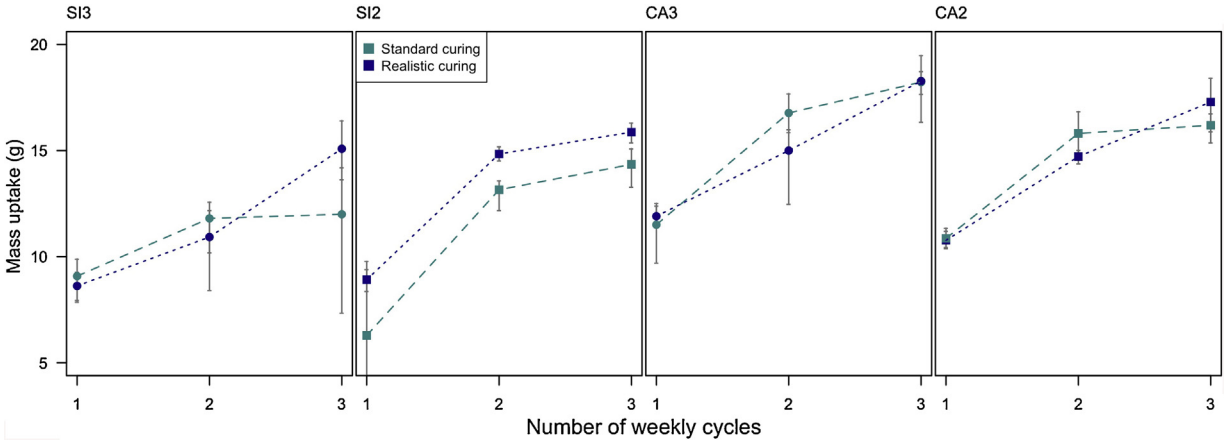

Fig. 8. Mass uptake (in g, calculated as the mean of 4 samples) due to water and salt at each weekly cycle (4 daily cycles) for mortars initially cured under different conditions. Samples cured under realistic conditions (RC) show a more consistent mass uptake.

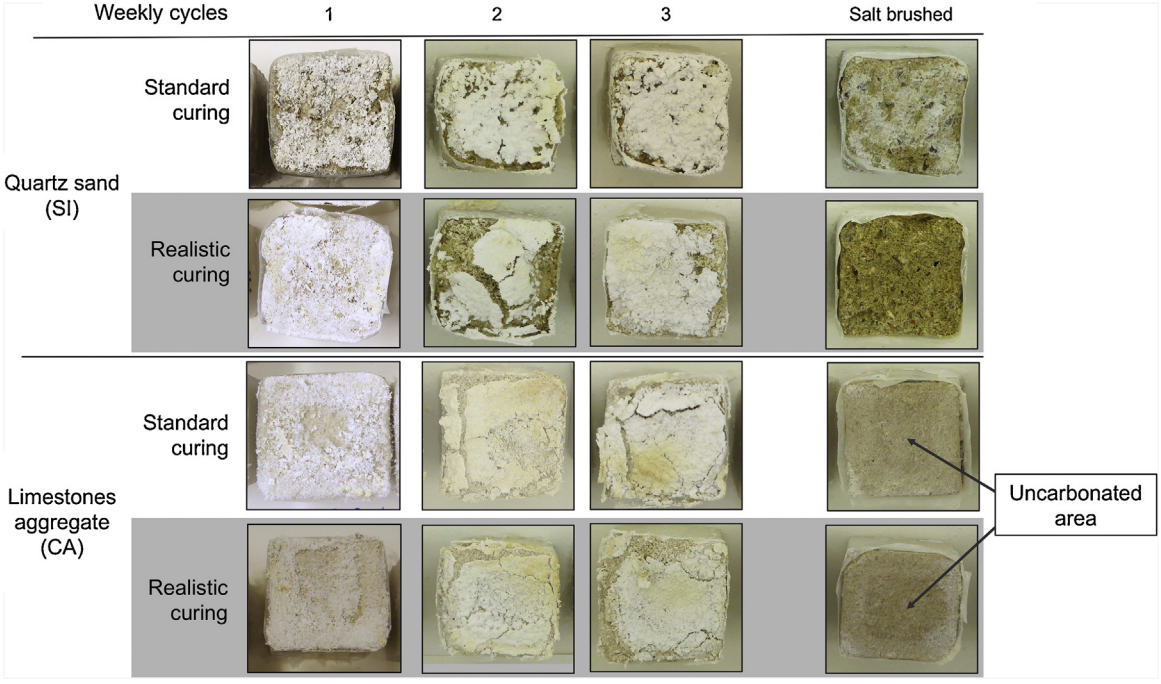

Fig. 9. Patterns of salt uptake over three weekly cycles for mortars (SI3 and CA3) cured under different conditions. Patterns are similar for SI2 and CA2 mortars and are therefore not presented here. Salt was removed after each weekly cycle. The last column shows the effect of salt weathering on the surface of mortars after three weekly cycles, once the salt had been brushed off.

Table 4
Decrease in open porosity after salt uptake and crystallisation ($n = 4$).

Conditions	Mortars	% decrease	
		Mean	SD
SC	SI2	−10.08%	±0.02
RC		−7.44%	±0.02
SC	SI3	−8.01%	±0.02
RC		−10.77%	±0.02
SC	CA2	−14.05%	±0.19
RC		−9.67%	±0.03
SC	CA3	−31.03%	±0.09
RC		−4.96%	±0.04

SC: standard conditions; RC: realistic conditions.

Table 5
Summary of the effect of realistic conditions (RC) as opposed to standard ones (SC) on properties evaluated. ↗ means that samples under RC obtain higher data than SC samples, ↘ lower data and → shows unchanged data. Arrows in blue show significant differences in all binder:aggregate ratios (see Table 1).

Property	Quartz (SI)		Limestones (CA)	
	28 days	90 days	28 days	90 days
Carbonation depth	↗	↘	↗	↘
Compressive strength	↗	↗	↗	↗
Propagation velocity	↗	↗ and →	↗	→
Open porosity	↗ and ↘	↘	↗	↗ and ↘
Capillary absorption	↗ and →	↘	↘	↘
Evaporation				
Salt uptake	↗		↗	

cal and physical properties, at both 28 and 90 days. As Table 5 shows, at 28 days, mortars cured under realistic conditions show higher mechanical properties, a mix of higher and lower porosity and lower or unchanged sorptivity. Indeed, when mortars are cured under realistic humid conditions the carbonation depth at an early age is between 1.1 and 2.7 mm deeper than samples cured under

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standard conditions (Fig. 1), the compressive strength between 14% and 31% higher at 28 days and 2% to 14% at 90 days (Fig. 3), the propagation velocity between 14% and 25% higher at 28 days and 28% to 36% higher at 90 days for SI samples (Table 3), and the capillary absorption decreases up to 8%. The main differences are seen at 28 days and for mortars made with quartz sand. At 90 days, fewer differences are seen.

These results indicate that the mechanical properties and internal structure of mortars cured under realistic humid conditions form quicker – i.e. the values obtained at an early age (28 days) are similar to those of medium age (90 days). This could be explained by the more humid conditions affecting the overall structural development, perhaps preventing the formation of too many shrinkage cracks. It could also suggest that, at an early age, lime mortars are more impacted by, and receptive to, their environment.

The capillary absorption coefficient slows down slightly when mortars are cured under realistic conditions. However, as the capillary absorption and the evaporation curves show, the ability of mortar to uptake and release water does not seem to differ between specimens exposed to standard and realistic curing conditions. This may suggest that the formation of the pore structure is not greatly affected by the outside environment, backed up by the MIP data in Fig. 4. In particular, CA mortars have a denser structure (Fig. 4b′), which could explain why they are less affected by the environmental conditions. Mortars made with quartz sand show the greatest differences in characteristics between those cured under realistic and standard conditions for 28 and 90 days.

5.2. Overall response of mortars in humid environment

Comparing the development at early to medium ages of different natural hydraulic lime mortars under realistic humid conditions and their response to evaporation and salt uptake helps understand how these mortars would perform in humid environments once applied as repointing mortar.

Realistic curing conditions, such as those found in Devon, could be beneficial, as the performance in uptake and release of water is only slightly affected, while their strength develops more quickly than those cured under standard conditions. This quicker internal development could benefit the performance of mortar exposed to rainfall. Indeed, a mortar reaching its hardened state quicker (Fig. 3) while having a greater ability to take up salt (Fig. 8) could perform well, and with less danger of failure, under a harsh environment experiencing series of intense rainfall events and ongoing masonry dampness. In addition, a lower capillary absorption would slow down the ingress of moisture. This would be greatly beneficial for the preservation of the overall masonry unit exposed to wind-driven rain and dampness.

5.3. Implication for laboratory evaluation of lime mortar

Characterisation of mortar mixes in the laboratory prior to on-site application is necessary to ensure mortars fit-for-purpose are chosen to be used. This study has shown that if the development of the mortar over different ages is to be assessed, in particular early to medium-term, using realistic curing conditions similar to the ones in which the mortar is going to be applied is important. Indeed, in the early curing period (up to 28 days), the mechanical properties of the mortar have been shown to develop at a different rate. Curing under realistic conditions (close to the ones on-site) gives a better insight of how natural hydraulic lime mortars would develop during the early stages and enables the practitioners to understand how mortars would respond to this specific environment. However, if the overall performance of the mortar, beyond medium-term curing (i.e. more than 90 days), is being assessed, standard curing conditions should be adequate.

6. Conclusions

In this study, realistic curing conditions, based on an average summer climate in Devon, were used to understand the effect of this humid environment on the development at early and medium-term curing ages of selected natural hydraulic lime mortars. This study has shown that although using realistic curing conditions can have an impact on some mechanical properties, the differences become less pronounced over time. Three main findings are:

- for the same natural hydraulic lime mortar exposed to different curing conditions significant differences are found, in particular on the mechanical properties and internal structure formation. Over the early to medium-term (up to 90 days curing time) natural hydraulic lime mortars are affected by the surrounding humid environment (RH and temperature). The main difference is seen in mortars made with quartz sand, for which realistic conditions have a more significant impact than those made with crushed limestone;
- the above results imply that the design of appropriate laboratory conditions (close to realistic ones) to characterise natural hydraulic lime mortar before repointing is particularly important for an assessment of the early to medium-term behaviour of the mortar. Over longer time scales, and an evaluation of the overall performance after 90 days, standard conditions are likely to be adequate;
- finally, humid curing conditions seem to be beneficial for minimising the early risk of failure of the repointing mortar and to preserve the overall masonry unit exposed to cycles of wetting and drying. Indeed, the study has shown that during curing under humid conditions natural hydraulic lime mortars can gain a quicker good internal structure more quickly, while retaining the ability to take up salt and water. These characteristics should help preserve the masonry units from deterioration.

Further testing on mortars made with other materials, under different conditions, and at earlier and later ages would also be interesting and provide a fuller assessment of the significance of curing conditions to reporting mortar performance.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at https://doi.org/10.1016/j.culher.2018.11.011.

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