Chapter 1
Analysis of the Natural Hydraulic Lime Mortar's Properties Under Accelerated Tropical Climate Conditions

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1.1 Introduction

The worldwide physical and literature evidence of lime mortar since the ancient civilization clearly states the favourable character of its setting properties and strength (Hughes and Válek 2003; Holmes and Wingate 2002). Current literature demonstrates the scientific advantages of lime over modern cement, which proves lime is greener than ordinary Portland cement (OPC). Lime requires a lower firing temperature during manufacturing and emits fewer greenhouse emissions per unit weight than OPC (Imbabi et al. 2012; Mahasenan et al. 2003). In addition, during the setting process, lime sequester carbon dioxide (CO_2), thus reducing its carbon footprint impact.

Understanding climatic conditions is a critical 'first-order' concern in planning and building functional and efficient buildings because climate substantially impacts the function, operation, life duration, and overall durability of a building/structure

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(Forster et al. 2014; Jelle 2012; Rathore et al. 2020). However, material deterioration from poor weather circumstances such as temperature fluctuations, solar radiation, atmospheric gases, humidity, rainfall, and the wind is less considered in the construction guidelines (Oliver 2008; Lomas and Porritt 2017; Mizzi et al. 2018). Lime mortar is temperature sensitive, affecting the development of hydration products and the creation of calcite compounds (Schroder and Ogletree 2010). These outputs are important in defining the strength and long-term durability and, hence, resistance to deteriorating features.

Due to changes in the construction industry landscape and lime mortar's sensitivity towards the atmosphere, most contemporary buildings in tropical Asia use Portland cement (PC) (Rasiah et al. 2017). Further widespread use of PC-based mortars is related to the benefits of the comparatively dense materials' impermeability or "waterproofing" qualities (Prezant et al. 2008). This viewpoint is largely criticized, particularly in historic structures (Banfill and Forster 1999). Lime mortars are crucial in these nations because they allow the structure to "breathe" by preventing the wall from holding moisture (Lawrence 2006). Numerous buildings and homes are constructed with PC-bound concrete because it is resistant to severe rains. In a hot-humid climate, biological growth from penetrating rain, evaporation, condensation, air pollution, dehydration, and thermal stress typically causes faults in rendering and mortars (de Oliveira et al. 2021).

1.2 Methodology

In this study, the mortar specimens were made from St. Astier NHL 3.5 lime, sand aggregates, and purified water. In order to guarantee that any salts and other contaminants were eliminated from the water, distillation was performed on it.

1.2.1 Specimens Manufacturing

The rheological characteristics of the mortars were determined using a manually controlled flow table device (BS EN 459-2:2010). The binder: aggregate volume ratio of 1:3 was chosen, and the mixing was designed based on the relative bulk density (RBD) principles, as shown in Table 1.1 (Razali et al. 2020).

Table 1.1 Mortar manufactures constituents and flows value

NHL density (g/L)	Constituent amounts of materials in the mortar used in the slump test			Flow value (mm ± 2 mm)
	Mass of NHL (g)	Mass of sand (g)	Mass of water (g)	
650	207.9	1564.0	332.0	165

The 'dry mixing' approach was chosen, in which the binder and aggregates were mixed for three minutes using a mechanical mixer before adding water and mixing for another three minutes (Allen et al. 2003; Ball et al. 2009). The mixture then being poured into 160 × 40 × 40 mm moulds. Afterwards, an automated vibrating table was utilized to compact the samples and remove trapped air (BS 459-2: 2010).

1.2.2 Curing Process

The curing process was conducted in an environmental chamber using accelerated tropical climatic simulations of temperature (°C) and relative humidity (RH) of (i) 20 °C–60%RH; (ii) 27 °C–90%RH; and (iii) 33 °C–90%RH. Seven days after being placed inside the curing chamber, the samples were de-moulded. The samples were covered with damp hessian fabric to minimize moisture loss during the early phases of the specimen set. A CO_2 injection system was devised to ensure enough carbonation, permitting a continuous CO_2 concentration of 450–500 ppm.

1.2.3 Chemical and Physical Testing

As recommended by Hanley and Pavia (2008), a 56-day test period was chosen as an acceptable alternative to the 365-day primary test duration.

1.2.3.1 Potential Hydrogen—pH

The pH value is important to ascertain the alterations that took place during the hydration process. Rasanen and Penttala (2004) used the approach to determine the hardened mortar. In order to prevent measurement errors caused by carbonation and variations in moisture content gradient, the mortar samples were manually cut at a depth of 40 mm from the surface. Afterwards, the samples were run through a crushing press to turn them into fine powdery particles. Following the crushing step, 15 g of the specimen was mixed into 15 mL of purified water.

1.2.3.2 Carbonation Depth

The samples were sprayed with 0.1% phenolphthalein solution, in which the colour pink denotes uncarbonated mortar. The stained area was measured using a digital calliper (± 0.01 mm) and photographed with a digital single-lens reflex camera for the record.

1.2.3.3 Mortar Strength Tests

The mortar samples were tested for three-point flexural strength (flexural response) using universal testing machine at low loading rates of 2 mm/min (Apostolopoulou et al. 2020). On the other hand, the compressive strength test applied load compresses the sample between the platens of the compression testing apparatus (BS EN 459-2:2010). The results' data were calculated using the standard flexural and compressive strength test formulas.

1.2.3.4 Sorptivity Test (Capillary Absorption)

Evaluating capillary absorption by adopting the direct gravimetric technique provides insight into porous structure and volume (Benachour et al. 2009; Hall and Hoff 2012). The surface of the specimens was then coated with resin except for the bottom surface (the surface that will come into contact with water). The specimen was immersed in water that had a height of 5 mm. Five weight gain readings are taken within the specified time interval. The data was then graphed, and the sorptivity value was calculated using the slope gradient, as shown in Eq. (1.1) (Dias 2000);

$$\text{Sorptivity}, i = \mathrm{k}\sqrt{\mathrm{t}} \tag{1.1}$$

A study by de Oliveira et al. (2006) measured the sorptivity coefficient, k, in kg/m^2/$min^{0.5}$. In this study, however, k values are given in terms of volume; volume (mm^3) absorbed across section (mm^2) per min = mm/$mm^{0.5}$.

1.2.3.5 Physical Pore Structure Analysis

This study used an electron microscope to study further the pore structure of the mortar specimen. The scanning electron microscope (SEM) is superior to conventional microscopes in several ways. The SEM has a broad field depth, allowing a greater portion of a specimen to be in focus simultaneously. Additionally, the SEM has a lot better resolution, allowing for much greater magnification of closely spaced specimens.

1.3 Results and Discussions

1.3.1 *Potential Hydrogen—pH*

Figure 1.1 shows that the entire specimens read a high amount of alkalis of pH 14 on the day of casting and read pH 10–12 after a week. After 14 days, the pH of each specimen fell to 7. As a mortar exterior chemically reacts with CO_2 in the atmosphere, the $Ca(OH)_2$ compound carbonates into calcite. Principally, the carbonation process, with time, lowers the pH value in the mortar by maximizing the number of Ca^{2+} ions. Although the pH values change is small, a clear trend can be distinguished.

Based on the data obtained, it was determined that the neutralization process with its corresponding exothermic reaction had occurred per Eqs. (1.2) and (1.3) (Lottermoser 2010). The interaction between calcium hydroxide and carbon dioxide produces a mortar that hardens rather fast. However, the process can be reversed if the temperature, water loss, or carbon dioxide loss changes. The pH will decrease with the continued re-precipitation of carbonate, which releases hydrogen ions.

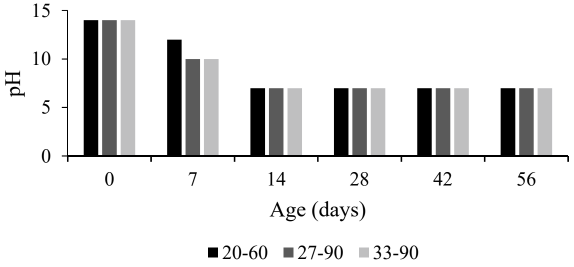
$$CaCO_{3(s)} + H^{+}_{(aqueous)} \Leftrightarrow Ca^{2+}_{(aqueous)} + HCO^{-}_{3(aqueous)} \tag{1.2}$$

$$CaCO_{3(s)} + 2H^{+}_{(aqueous)} \Leftrightarrow Ca^{2+}_{(aqueous)} + H_2CO^{-}_{3(aqueous)} \tag{1.3}$$

The pH value will decrease as the calcium inside the mortar solubility rises. The mortar exterior surface exhibited limited moisture vapour movement, resulting in greater pH variation and carbonation depth variation compared to the sample's opposite side. In this experiment that involves a high-humidity atmosphere, excessive moisture will transport more hydroxides (alkalis) to the surface, thus increasing the pH. This reaction may weaken the mortar since calcium ions (Ca^{2+}) are necessary to form hydrates.

Then again, the pH changes might be related to differences in material features and qualities, which had a commensurate effect on the degree of carbonation in the mortars. Carbonation is responsible for the overall decrease in pH over time. During the initial week of curing, the surfaces of all specimens were exposed to atmospheric

Fig. 1.1 pH of mortars



carbonation. The optimal CO_2 concentration promotes quick carbonation of mortar, hence lowering the pH levels.

The rate of hydration is accelerated by a high temperature, which also affects the pH. The extended exposure of the specimens probably resulted in higher pH alterations. Continuous measurements were conducted till the conclusion of the investigation to detect any pH value rise.

1.3.2 Carbonation Depth

This study's carbonation depth (Fig. 1.2) reveals that climate and curing time are vital variables determining the mortar carbonation rate. This creates an environment rich in carbonic acid, resulting in portlandite ($Ca(OH)_2$) transition into calcite. The process results in the formation of calcium carbonate ($CaCO_3$), which aids in the mortar's hardening. Intriguingly, mortars cured at 27 °C–90%RH demonstrated better carbonation than other studied climates, in which the mortars were fully carbonated in 14 days. In a lower-studied climate, the time required for complete carbonation was much longer (doubling to 28 days). On the other hand, a steady linear trend was observed during the 33 °C–90%RH climate.

Due to lime's intrinsic thermodynamic features (high carbonation temperature, high CO_2 carrying capacity), carbonation rates increase as the temperature rises (Materic and Smedley 2011). As the temperature rises to 33 °C, the water's contact with the solid surface increases, and the carbonation process begins swiftly. After prolonged contact with high temperatures, the materials become dehydrated, preventing the carbonation of $Ca(OH)_2$. The carbonation process was continued in an atmosphere of 90%RH. This was a result of the detrimental effect of excessive humidity on carbonation. Importantly, the pace and magnitude of carbonation were generally independent of time but temperature-dependent (Materic and Smedley 2011). Lime hydration is reactivated when exposed to humid air at room temperature.

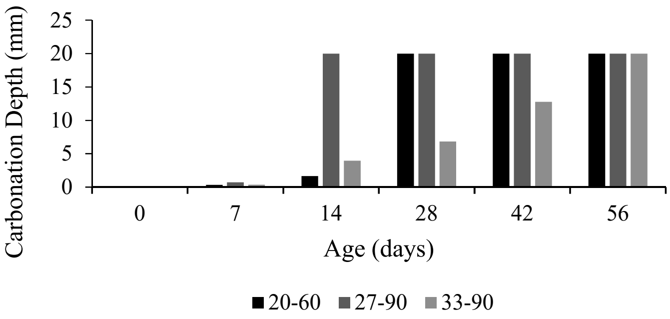


Fig. 1.2 Carbonation depth of mortars

The pace of carbonation is affected by several environmental conditions, including relative humidity, temperature, and the amount of CO_2 in the air. High CO_2 enhances calcite formation from $Ca(OH)_2$. The presence of water adsorbed from the surface of calcium hydroxide (as a result of the spraying regime) significantly impacts calcium hydroxide's carbonation. The presence of water adsorbed by mortar controls the kinetics and significantly affects the carbonation progression of $Ca(OH)_2$. Carbonation relies on the presence of water and happens fast near the surface. This is accentuated in a carbon dioxide-saturated environment.

1.3.3 Flexural Strength

Figure 1.3 demonstrates that the specimens' optimal curing conditions for flexural growth were at 20 °C–60%RH. This data illustrates both quicknesses of early strength (at 7 days) and the consistency of strong growth across 56 days. On the other hand, the strength developments in the 27 °C–90%RH specimens were atypical, in which the specimens had a positive early strength development but suffered a 61% loss at 28 days. Even though curing at 33 °C–90%RH experienced an early linear flexural strength growth of up to 42 days, the specimens had the weakest flexural strengths relative to specimens from different climate conditions.

The mortars improved their mechanical strength with extended curing times/ longer carbonation processes. Due to the rising binder content and porosity, which facilitates the transport of CO_2 into the mortar where portlandite ($Ca(OH)_2$) is still available to undergo the carbonatation process, the carbonation response was stronger with longer curing durations. This aligns with the generally acknowledged body of knowledge in this field (Hughes and Válek 2003; Holmes and Wingate 2002; Forster et al. 2014). This is true for specimens cured at 27 °C–90%RH. Mortars cured at the higher temperature of 33 °C–90%RH exhibited the worst flexural strength, most likely due to a delayed carbonation rate impeded by very high and prolonged moisture levels that impeded CO_2 diffusion. Nonetheless, Cizer et al. (2007, 2008)

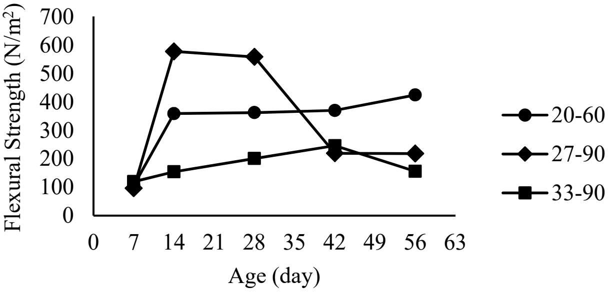


Fig. 1.3 Flexural strength of mortars

indicated that the flexural strength of modified mortar depended heavily on alternative ingredient(s), seeded material(s) and porosity.

In contrast, the flexural strengths found at the initial curing period of 28 days are inconclusive due to the fact that the specimens were greatly impacted by the water content used in the mix and humidity in the atmosphere, as there is a direct correlation between water ratio and strength. Lanas and Alvarez-Galindo (2006) developed a hypothesis that helps to explain this phenomenon by demonstrating that mortars have the best strength when a particular proportion of $Ca(OH)_2$ stays uncarbonated. Observations corroborated this hypothesis on different testing days in all studied climates. A modest drop in uncarbonated $Ca(OH)_2$ results in a small decrease in mortar strength. However, analytical analysis cannot precisely identify $Ca(OH)_2$'s quantity.

1.3.4 Compressive Strength

Figure 1.4 illustrates the influence of temperature and curing time on the mortar's compressive strength. All specimens show an increase in strength with respect to time, even though the strength development varies according to the studied climates. It is reasonable that, while analyzing the growth of compressive strength, the variables impacting the quality of flexural strength would also be prominent. As discussed in flexural strength, carbonation rate determines strength development. The compressive strength improves over time by converting $Ca(OH)_2$ to $CaCO_3$ (calcite). This is predicted and consistent with the recognized knowledge of the mechanics behind the development of strength in lime mortars during curing. According to Moropoulou et al. (1997), because crystalline calcite is present in the mortar, the porosity of the material is reduced, while at the same time, its strength is increased.

Mortars lost strength due to prolonged, high-temperature curing over extended periods because they exhibited a non-uniform distribution of hydration products due to rapid hydration. This resulted in an increase in porosity and a high compressive strength in the early stage. However, a decrease in longer strength development

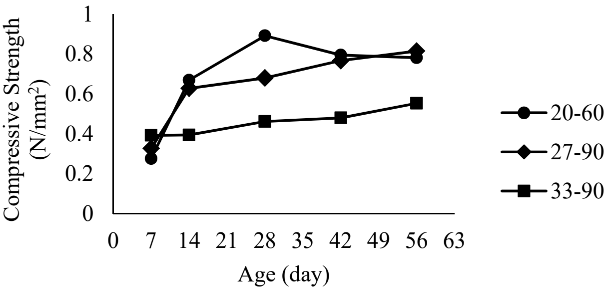


Fig. 1.4 Compressive strength of mortars

was observed in most accelerated climates. These findings correlated with Ezziane et al. (2007), who observed the strength loss evolution in elevated temperatures and proposed mineral admixtures in the mortar as a plausible solution. Some mineral admixtures in the mortar change the hydration kinetics, resulting in a decrease in heat evolution and the production of extra C–S–H.

1.3.5 Sorptivity

A substantial correlation between lime mortar and sorptivity highlights the significance of a mortar's absorption and transmission properties. Figure 1.5 demonstrates that the water absorption rate and sorptivity coefficient significantly affect the curing environment and time. The samples cured at the lower climate had a denser structure, as shown by their high sorptivity values. Additional research revealed that the 33 °C–90%RH specimens had the greatest *k* values, indicating an abundance of linked pores.

The mortar specimens in 20 °C–60%RH climate displayed the lowest sorptivity values. It is believed that the intricacy of matrix development in the structure is a consequence of composition and hygrothermal conditions that either promote or inhibit growth; an increase in sorptivity does not necessarily cause by a decrease in relative humidity. Based on microscopy investigation, the specimens cured at this climate observed crystal formations of unusual forms and sizes.

Even though the specimens were exposed to the same high humidity level, it was shown that the temperature impacts affected pore structure, resulting in increased sorptivity. This was likely the result of a conducive atmosphere for the development of hydration products. Once exposed outside the curing box, the temperature differential between the specimens and the ambient room temperature may have further altered the sorptivity. In order to achieve thermal balance with the surrounding air, the specimens would emit a certain quantity of moisture into the environment. As a result, moisture release and loss during equilibrium attempts would reduce the moisture content of the specimens. This would affect their absorption throughout the sorptivity

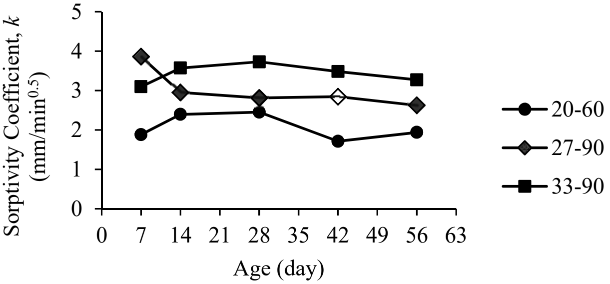


Fig. 1.5 Sorptivity coefficient, *k* of mortars

tests. Specimens treated at 33 °C–90%RH had a crystal structure that was smaller but more uniform and regular-shaped than those cured at 20 °C–60%RH. It is fair to conclude that these tests resulted in size discrepancies in the crystal formations, which led to a decrease in water absorption. Variations in the physical properties linked with cure led to variances in sorptivity. These findings are comparable to those of Kerr (2008), who proposed probable parameters that may influence the deterioration of the pore structure due to the formation of additional hydration products.

1.3.6 Physical Pore Structure Analysis

Figure 1.6 depicts SEM photographs of specimens. Clusters of needle-shaped (acicular) and hexagonal plate-shaped crystals can be found embedded in various places throughout the set matrix. Crystals that resemble needles (acicular) are indicative of the C–S–H morphology. Ettringite formation was also identified. Through its hydration process, ettringite hydrates fast, creating early strength and generating significant heat. Its creation might partially explain the strong outcomes of all lime mortars cured in tropical climates gaining rapid strength development but subsequently experiencing a strength loss afterwards.

1.4 Conclusion

The study shows that lime mortar specimens cured in accelerated tropical climates exhibited enhanced early-age strength development, but the strength qualities and pore structure deteriorated with time. These findings demonstrate the challenges of using NHL materials in high-temperature and high-humidity conditions, such as tropical climates. Specimens cured in accelerated tropical climates exhibited clusters of needle-shaped acicular and hexagonal plate-shaped crystals that led to unstable matrix formation. These results are critical because the pore structure's growth significantly impacts the hardened material's strength, permeability, and durability. This can potentially compromise the lime mortar's properties, leading to structural failure that could result in user safety. However, further studies should be made by studying the new mixture of NHL using other admixtures or addictives to overcome the deficiency of lime mortar quality used in tropical climates.

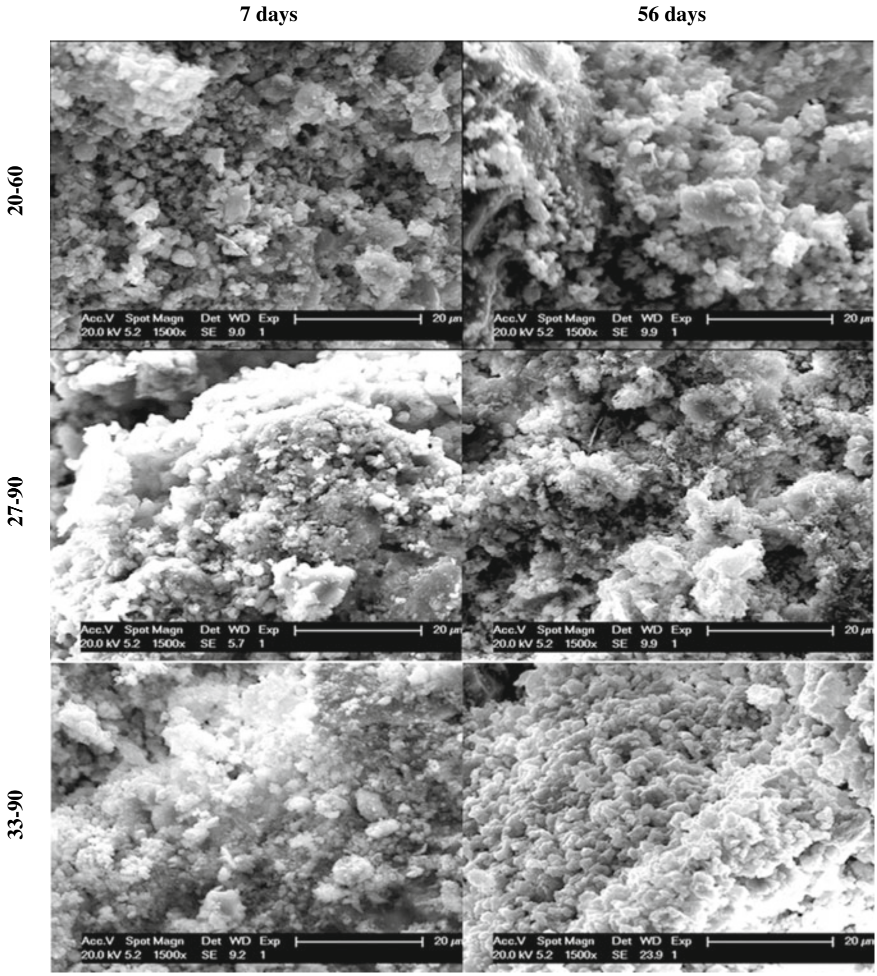


Fig. 1.6 SEM images of mortars

References

Allen G, Allen J, Elton N, Farey M, Holmes S, Livesey P, Radonjic M (2003) Hydraulic lime mortar for stone. Donhead Publishing Ltd., Dorset, UK, Brick and Block Masonry

Apostolopoulou M, Asteris PG, Armaghani DJ, Douvika MG, Lourenço PB, Cavaleri L, Bakolas A, Moropoulou A (2020) Mapping and holistic design of natural hydraulic lime mortars. Cem Concr Res 136:106167–106187

Ball RJ, El-Turki A, Allen GC (2009) Influence of carbonation on the load dependent deformation of hydraulic lime mortars. In: Walker P et al (eds) 11th international conference on non-conventional materials and technologies (NOCMAT 2009), Bath, UK

Banfill PFG, Forster AM (1999) A relationship between hyraulicity and permeability of hydraulic lime'. In: Bartos P, Groot C, Hughes JJ (eds) Proceedings PRO012: international RILEM workshop on historic mortars: characteristics and tests Paisley, Scotland, Edinburgh. RILEM, 11, United Kingdom

Benachour Y, Davy CA, Skoczylas F, Houari H (2009) Effect of high calcite filler addition upon micro-structure and freeze/thaw resistance of a mortar. In: Tanabe T, Sakata K, Mihashi, H, Sato R, Maekawa K, Nakamura H (eds) Creep, shrinkage and durability mechanic of concrete and concrete structure. Taylor & Francis Group, London, UK

Cizer O, Van Balen K, Van Gemert D, Elsen J (2007) Carbonation and hydrations of mortars with calcium hydroxide and calcium silicates binders. Taylor & Francis Group, Coventry, UK

Cizer O, Van Balen K, Van Gemert D (2008) Blended lime-cement mortars for conservation purposes: microstructure and strength development structural analysis and historic construction. Taylor & Francis Group, London

de Oliveira VC, Godinho JP, Grings KJO, de Oliveira RA, dos Santos EGF (2021) Performance as for water tightness of rendering mortars using admixtures. Int J Build Pathol Adapt 6(1):1–9

de Oliveira LAP, de Castro Gomes JP, Gonilho Pereira CN (2006) Study of sorptivity of self-compacting concrete with mineral additives. J Civil Eng Manag 12(3): 215–220. https://doi.org/10.1080/13923730.2006.9636395

Dias WPS (2000) Reduction of concrete sorptivity with age through carbonation. Cem Concr Res 30(8)

Ezziane K, Bougara A, Kadri A, Khelafi A, Kadri E (2007) Compressive strength of mortar containing natural pozzolan under various curing conditions. Cem Concr Compos 29(8):587–593

Forster AM, Szadurski EM, Banfill PFG (2014) Deterioration of natural hydraulic lime mortars, I: effects of chemically accelerated leaching on physical and mechanical properties of uncarbonated materials. Constr Build Mater 72:199–207

Hall C, Hoff WD (2012) Water transport in brick. Spon Press, Oxon, UK, Stone and Concrete

Hanley R, Pavia S (2008) A study of workability of natural hydraulic lime mortars and its influence on strength. Mater Struct 41(2):373–381

Holmes S, Wingate M (2002) Building with lime: a practical introduction. ITDK, London

Hughes JJ, Válek J (2003) Mortars in historic buildings: a review of the conservation, technical and scientific literature. Historic Scotland, Edinburgh

Imbabi MS, Carrigan C, McKenna S (2012) Trends and developments in green cement and concrete technology. Int J Sustain Built Environ 1(2):194–216

Jelle BP (2012) Accelerated climate ageing of building materials, components and structures in the laboratory. J Mater Sci 47(18):6475–6496

Kerr EA (2008) Damage mechanism and repairability of high strength concrete exposed to heat, Unpublished thesis. The University of Notre Dame

Lanas J, Alvarez-Galindo JI (2006) Masonry repair lime-based mortar: factors affecting the mechanical behaviours. Cem Concr Res 33(11):1867–1878

Lawrence RMH (2006) A study of carbonation in non-hydraulic lime mortars, unpublished thesis. The University of Bath

Lomas KJ, Porritt SM (2017) Overheating in buildings: lessons from research. Build Res Inf 45(1–2):1–18

Lottermoser BG (2010) Mine wastes: characterization. Springer-Verlag, Berlin, Treatment and Environmental Impacts

Mahasenan N, Smith S, Humphreys K (2003) The cement industry and global climate change: current and potential future cement industry CO_2 emissions. In: Greenhouse gas control technologies-6th international conference, Pergamon

Materic V, Smedley SI (2011) High-temperature carbonation of $Ca(OH)_2$. J Am Chem Soc 50(10):5927–5932

Mizzi B, Wang Y, Borg RP (2018) Effects of climate change on structures; analysis of carbonation-induced corrosion in reinforced concrete structures in Malta. IOP Conf Series Mater Sci Eng 442(1):12023–12038

Moropoulou A, Biscontin G, Bakolas A, Bisbikou K (1997) Technology and behaviour of rubble masonry mortars. Constr Build Mater 11(2):119–129

Oliver JE (ed) (2008) Encyclopedia of world climatology. Springer Science & Business Media

Prezant B, Weekes DM, Miller JD (eds) (2008) Recognition, evaluation, and control of indoor mold. AIHA Publishing, Virginia

Rasanen V, Penttala V (2004) The pH measurement of concrete and smoothing mortar using a concrete powder suspension. Cem Concr Res 34(5):813–820

Rasiah R, Ahmed A, Al-Amin AQ, Chenayah S (2017) Climate change mitigation: comparative assessment of Malaysian and ASEAN scenarios. Environ Sci Pollut Res 24(3):2632–2642

Rathore PKS, Shukla SK, Gupta NK (2020) Yearly analysis of peak temperature, thermal amplitude, time lag and decrement factor of a building envelope in tropical climate. J Build Eng 31:101459–101474

Razali N, Azizan MA, Pa'ee KF, Razali N, Jumadi N (2020) Preliminary studies on calcinated chicken eggshells as fine aggregates replacement in conventional concrete. Mater Today Proc 31:354–359

Schroder L, Ogletree V (2010) Adobe homes for all climates: simple, affordable, and earthquake-resistant natural building techniques. Chelsea Green Publishing, Vermont