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Is BS EN 459-1:2015 fit for purpose in the context of conservation?

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Abstract

Natural hydraulic lime (NHL) binders consist mainly of portlandite, reactive silicates, and aluminates formed from the reaction of crushed limestone, containing clays or other impurities, during calcination. By their nature, these binders have a variable chemical and mineral composition, depending on the geographical location of the limestone extraction (initial composition) and the manufacturing process. The NHL classification, as specified in BS EN 459-1:2015, does not consistently give a representative indication of the properties of mortars made with particular NHL binders, because different limes in the same classification often exhibit very different properties and behaviour, thereby hindering the ease of user specification for mortars. A representative selection of binders was characterised using calorimetric analysis, X-ray diffraction, X-ray fluorescence, and particle size analysis. The varying proportions of reactive mineral phases between the limes were related to the kinetics of the hydraulic reaction of the NHL binders and their chemical and physical properties. A relationship has been established between the chemical and physical properties of the binders and the NHL characteristics. These results will be used to validate a model predicting the long-term behaviour of NHL mortars for conservation interventions on heritage and historic buildings.

Introduction

Lime-based mortars are one of the most ancient man-made construction materials and they can be found in a large proportion of surviving buildings constructed before the 20th century.

The current standard for building limes in the UK is BS EN 459-1:2015,¹ hereafter referred to as BS EN 459-1, which divides limes into two separate families: air lime and lime with hydraulic properties. Lime with hydraulic properties is further divided into three sub-groups: natural hydraulic lime (NHL), formulated lime, and hydraulic lime. This research is concerned with NHLs, which are those in which the hydraulic properties derive from the natural raw material, without any additions, other than grading agents up to 0.1 per cent.

NHLs are widely used in the conservation process, presenting – when compared to cement mortars – some of the beneficial characteristics of an air-lime-based mortar, such as better pore structure, increased water vapour diffusion, and improved ability to accommodate movement through flexibility. Unlike air-lime mortars, they have a faster initial set and the ability to harden in damp conditions or even under water.^{2,3,4,5,6}

Formed by calcining crushed limestone that contains clay, at temperatures between 950°C and 1050°C, NHLs consist mainly of portlandite (calcium hydroxide, $\text{Ca}(\text{OH})_2$) and reactive silicates and aluminates. The material's ability to set under water and the faster setting time are dependent on the amount of hydraulic phases, which in turn derives from the composition of the raw material and the calcination temperature. The rapid initial set of hydraulic lime mortars is attributed to hydraulic reactions; the calcium silicates and aluminates react with water, forming calcium silicate hydrates and calcium aluminate hydrates. Further strength develops from the continued process of hydration and, over the longer term, carbonation of free lime (calcium hydroxide). Carbon dioxide (CO_2) diffuses through the porous structure reacting with the calcium hydroxide and the hydration products, forming calcium carbonate (CaCO_3), amorphous silica, and alumina. Hydration and carbonation depend on the amount of hydraulic phases and the calcination temperature of the original limestone.^{7,8,9,10,11,12,13}

NHL binders are classified according to BS EN 459-1; classification is based on the compressive strength of a standard mortar mix at 28 days and the minimum weight content of available lime (calcium hydroxide; Table 1). Mortar samples for compliance testing are made using a standardised sand, following a standard binder/aggregate mass ratio of 1:3. The quantity of water is based on the bulk density of the binder and the intended classification (Table 2).

Lime type	Available lime as $\text{Ca}(\text{OH})_2$ (%)	Minimum compressive strength at 28 days – tolerance values in brackets (MPa)
NHL 2	≥ 35	2 (2–7)
NHL 3.5	≥ 25	3.5 (3.5–10)
NHL 5	≥ 15	5 (5–15)

Table 1 NHL classification and tolerances according to BS EN 459-1:2015

Lime type	Bulk density (kg/dm^3)	Water (g)	Water/binder w/b
NHL 5	> 0.6	225	0.5
NHL 2; NHL 3.5	> 0.6	248	0.55
NHL 2; NHL 3.5; NHL 5	≤ 0.6	270	0.6

Table 2 NHL classification and tolerances according to BS EN 459-1:2015

The wide overlap of the three classifications allows limes with significantly different properties to be classified as the same type. In addition, the standard mix for the compliance test with BS EN 459-1 is not representative of the mix ratios commonly used in building or conservation works. The test at 28 days can also be misleading when characterising and classifying limes of low hydraulicity, where the majority of strength is gained through carbonation over the longer term.^{14,15} For these reasons, there is a concern among some conservation professionals that BS EN 459-1 is not an appropriate standard by which to select binders for conservation mortars. A better knowledge of the binders and how their characteristics influence the final properties of the mortars is needed, hence this research is being developed.

The hydration process of lime-based binders is studied frequently using diffraction and spectroscopic techniques.¹⁶ In this study, isothermal calorimetry is used to relate the reactivity of NHL binders to the mechanical properties of corresponding lime-based mortars. Better understanding of the variability of NHL binder properties and how these characteristics influence mortar performance will facilitate more informed selection of materials, particularly in conservation where the preservation of the historic fabric is one of the most important factors.

Materials and methods

In order to study how the presence of hydraulic phases affects the reactivity of NHL binders, three binders from a single manufacturer and one from a different producer, presenting a diverse range of physical and chemical properties, were selected.

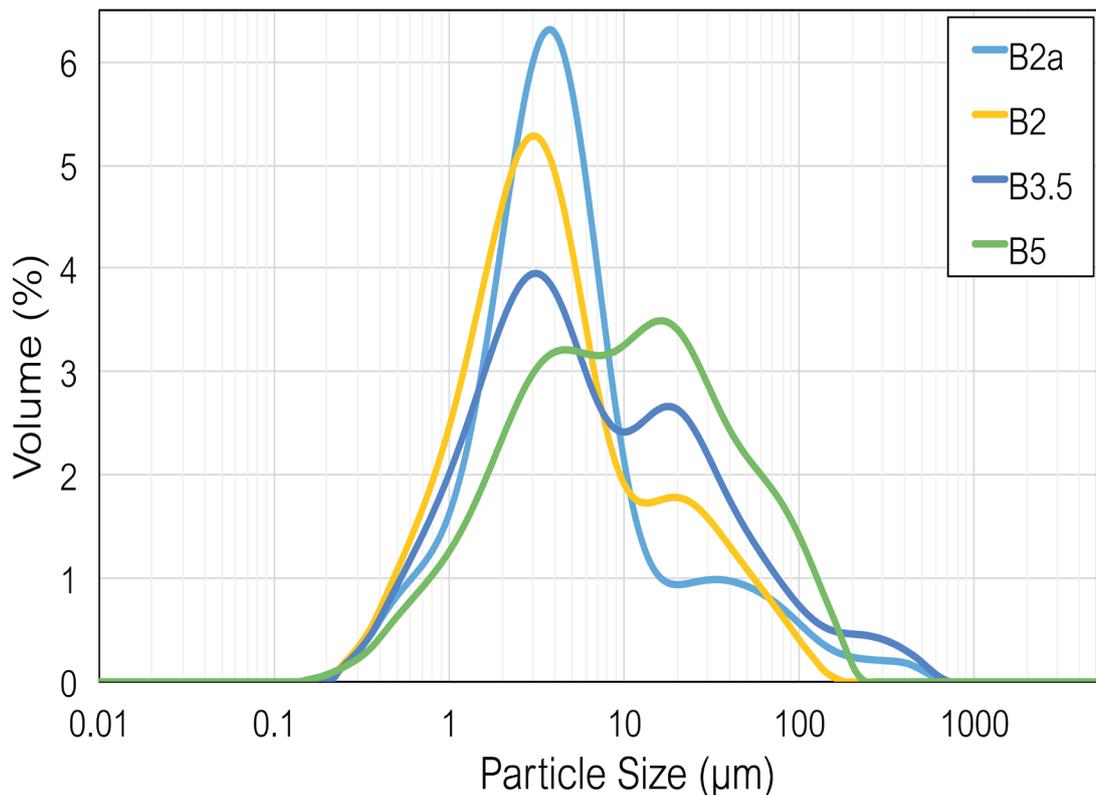
Binder characterisation

Bulk density was determined using the process described in BS EN 459-2:2010,¹⁷ and the surface area was determined by BET (Brunauer, Emmett and Teller) nitrogen adsorption analysis using a Micromeritics 3Flex (Table 3). Particle size distribution was obtained by testing the dry powder in a Malvern Mastersizer 2000 and obtaining the frequency of the particle size related to the volume (Figure 1).

Binder	NHL type	Bulk density (g/cm ³)	Surface area (m ² /g)
B2	NHL 2	0.58	10.04
B3.5	NHL 3.5	0.67	6.24
B5	NHL 5	0.79	4.14
B2a	NHL 2	0.64	5.46

Table 3 Physical properties of the binders: bulk density and surface area

Fig. 1 (Right) Particle size distribution of the four binders in the 'as received' form.



Binder	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	LOI
B2	9.3	0.4	0.4	66.0	0.4	0.5	22.0
B3.5	15.2	1.0	0.5	61.4	0.6	0.5	19.8
B5	15.6	0.9	0.6	60.7	0.9	0.6	19.7
B2a	7.8	1.6	2.1	66.4	2.4	0.4	18.0

Table 4 XRF main oxide composition in % of weight

The oxide composition of the binders was examined by X-ray fluorescence (XRF) analysis (Table 4), performed on 40 mm diameter pellets (thickness 2 mm) pressed from the binders and analysed using an EDAX Eagle II Energy Dispersive XRF spectrometer with rhodium X-ray, operated at 25kV and 1mA for 100 seconds in multiple spots per sample for the spectrum acquisition. The loss on ignition (LOI) – to determine the chemically combined and free water content and the carbon dioxide content derived from the carbonates – was determined by burning 1g of binder (± 0.001) at 950°C until the sample mass had stabilised (when all water and carbon dioxide was driven off).

X-ray diffraction analysis (Table 5) of the mineral phase composition of the binders was performed at ambient temperature using a Bruker-AXS D8 powder X-ray diffractometer. The equipment was operated at 40kV, 40mA, and the source of radiation was Cu-K α X-rays. The step was 0.02°, from 4 to 75° (2 θ).

Using a Calmetrix I-Cal 4000 with F/P-Cal logger software, the isothermal calorimetric tests were performed on samples of the binders mixed with water. The reaction between the hydraulic components of binders and water is exothermic, and the heat produced (hydration heat) is related to the speed and intensity of the hydraulic reaction. The heat of the reaction was recorded for 72 hours.

Binder	Portlandite	Calcite	Belite	Alite
B2	++	+	+	+
B3.5	++	++	+	+
B5	++	++	++	++
B2a	++	+	++	+

Table 5 XRD qualitative mineral composition

++ Strong signal, identified by 3 or more major peaks

+ Moderate signal, identified by 3 peaks of intensity <20% of maximum

Mortar testing

Mortar prisms 160 x 40 x 40 mm were prepared with a binder/aggregate volumetric ratio of 1:2, mixed to a consistency to give a spread of 165 \pm 10 mm, measured by flow table (BS EN 1015-3:1999¹⁸). Table 6 shows the characteristics of the mass of the constituents in each of the mortar samples, the binder/aggregate (b/a) ratio by mass, the flow table spread, and the water/binder (w/b) ratio by mass. Specimens were prepared and cured following the recommendations of BS EN 1015-11:1999,¹⁹

Mortar	Binder	Binder (kg)	Sand (kg)	Water (kg)	b/a (w/w)	Spread (mm)	w/b (w/w)
MB2	B2	2.42	11.83	2.70	1:4.89	160	1.12
MB3.5	B3.5	2.75	11.56	3.28	1:4.20	174	1.19
MB5	B5	3.30	11.72	2.97	1:3.55	174	0.90
MB2a	B2a	2.64	11.68	2.51	1:4.42	160	0.95

Table 6 Formulations characteristics (1:2 binder/aggregate volumetric formulation)

Compressive and flexural strength testing was carried out on the prisms at 7, 14, 28, 91, 180, and 360 days according to BS EN 1015-11:1999.

Results and discussion

Calorimetric results (Figure 2) show the specific hydration heat power per 1g of dry binder. The initial high values at the beginning of the test (1) for all the binders represent the first contact of the binder with water. After that point, B2 and B3.5 have almost identical curves, and demonstrate a slower decrease of heat flow than B5 and B2a, indicating that in these mortars hydration is initiated faster than in the other two samples. B5 shows a small change in the curve, with a local maximum at around 8 hours of testing (2), related to the alite content, which is faster to react than the other constituents. B2a presents a local maximum at 13 hours (3) that can be related to the belite phase.²⁰

The specific heat of hydration, where the slope of the curve is related to how fast the reaction is happening (Figure 3), demonstrates that all the binders continue to have hydraulic reactions until the end of the calorimetric test (72 hours). The curves for B2 and B3.5 are similar, and indicate that the reaction process is initially faster than B5 and B2a, but tends to slow down after 10 hours. For B2a, reaction at around 13 hours (related to the hydration of the belite phase) is also detectable in these data. The B5 reaction process is initially slower than B2 and B3.5, but after 10 hours it demonstrates a faster and more energetic reaction.

Fig. 2 (Right)
Calorimetric analysis
for the first 20 hours.

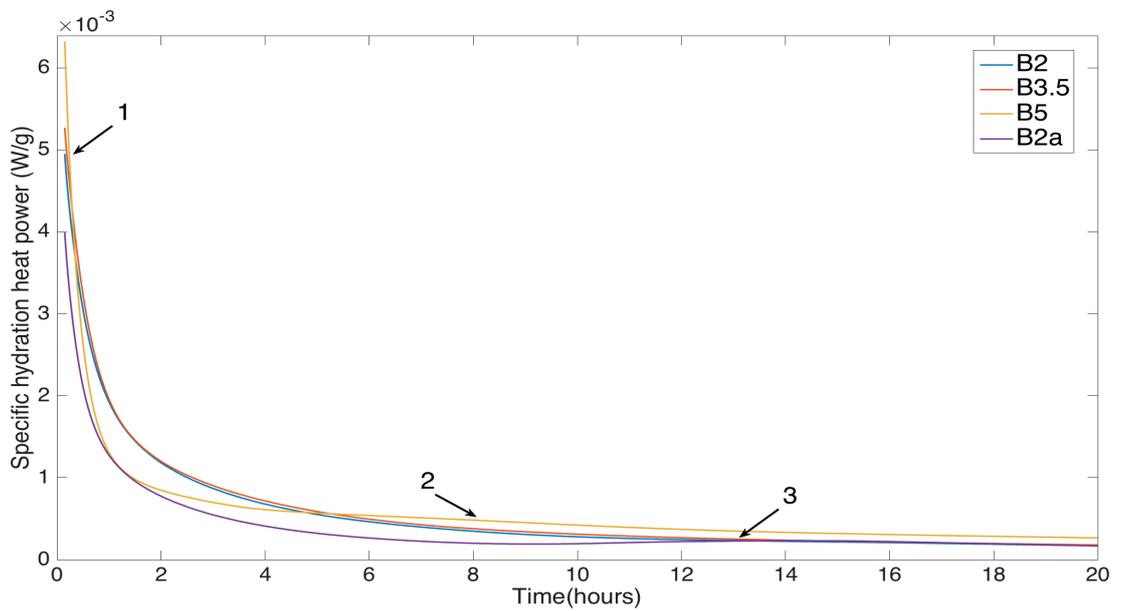
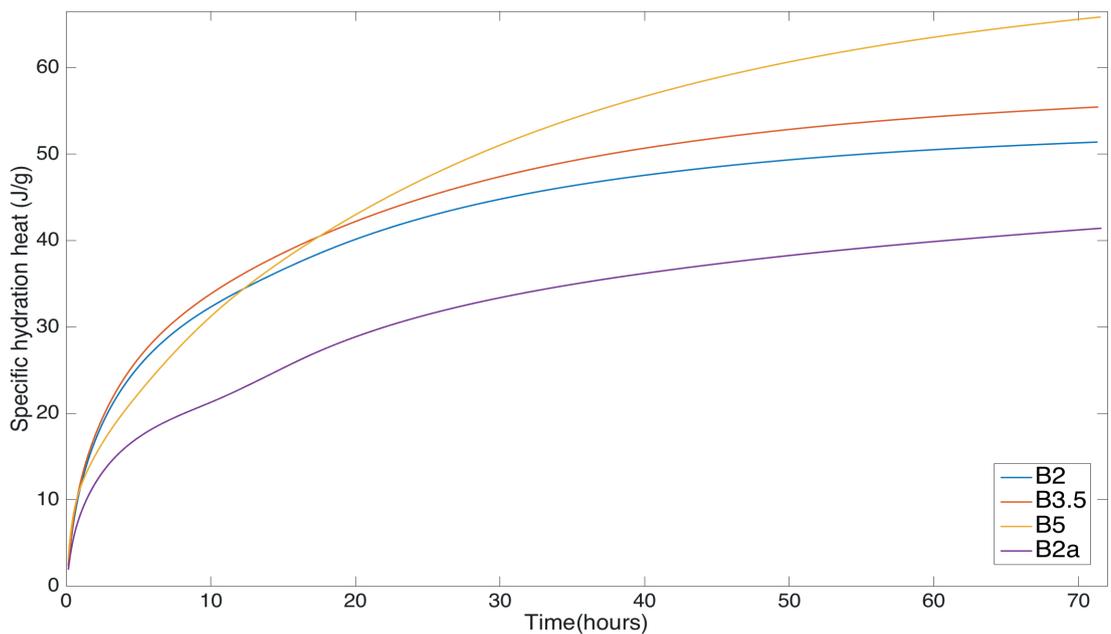


Fig. 3 (Right)
Specific hydration
heat per 1g
of binder.



It appears that for the three binders produced by the same manufacturer (B2, B3.5, and B5), the initial speed of hydration reaction is related to surface area (shown in Table 3): the larger the surface area, the faster the hydration speed. Despite the differences in their surface areas, B2 and B3.5 show similar reaction kinetics initially (up to 10 hours). This is probably due to the higher silicon dioxide (SiO_2) content of B3.5, which increases its reactivity, compensating for its smaller surface area. The slower reaction, for the first 10 hours, of B5 is probably related to the low surface area and it is therefore potentially less reactive at the beginning of the calorimetric test.

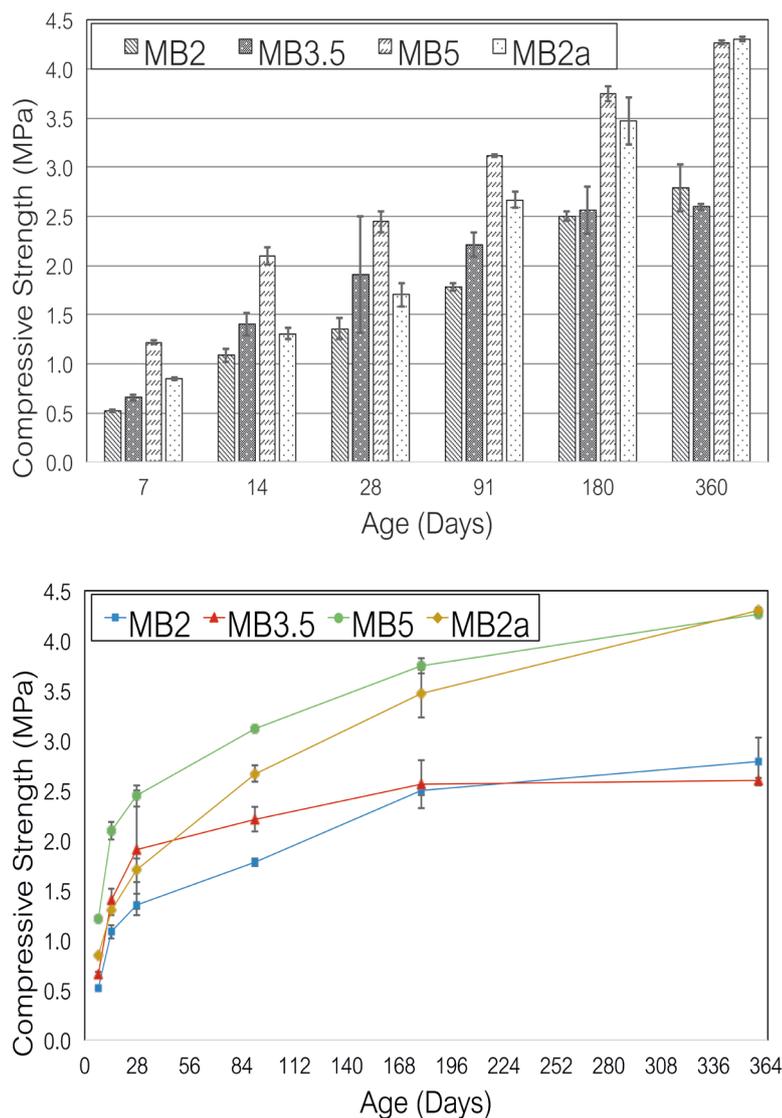
Compressive strengths are shown in Figure 4, where the evolution of the compressive strength with time is also indicated. As in the calorimetric tests for B2 and B3.5, there are similarities with MB2 and MB3.5; both have similar compressive strength at all ages.

MB5 is consistently the strongest mortar, but MB2a achieves similar strength to MB5 at 360 days. This demonstrates the importance of carbonation in hydraulic lime-based mortars, where the magnesium oxide (MgO) in the form of dolomite also contributes to the gain of strength in MB2a, along with the calcium oxide (CaO) in the form of portlandite.

XRF analysis showed that B5 and B3.5 have similar chemical compositions, but the calorimetric tests and compressive test results of the respective mortars (MB5 and MB3.5) highlight the effect of differences in mineral phase composition and surface area on mortar strength. B5 contains more alite (which is known to contribute to faster strength gain and higher mortar strength) and has a lower specific surface area, so demands less water for the same workability, thereby helping to achieve a stronger mortar.²¹

The slightly higher specific heat of hydration after 7 hours of B3.5 compared to B2 can be related to the slightly higher content of silicon dioxide in B3.5, potentially having higher reactivity. The mortars produced with these binders showed that the higher water/binder ratio for MB3.5 can offset the higher hydraulicity of B3.5, thus reducing mortar strength. Mortar MB5, made from the B5 binder that has a higher content of alite and belite, achieved higher strengths. Furthermore, long-term carbonation in MB2 can lead to very similar compressive strength results at later ages (from 180 days onwards) compared to those in mortars made with binders classified in higher strength categories in BS EN 459-1, such as sample B5 analysed here. MB3.5 does not show any strength gain between 180 and 360 days, appearing to have stabilized.

Binders B2 and B2a are both classified as NHL 2 under BS EN 459-1, although they display very different behaviour in the calorimetric analysis, and mortars made with them (MB2 and MB2a) have very different compressive strengths at all the testing ages. B2 and



B3.5 have different BS EN 459-1 classifications, but they achieve similar calorimetric results, and mortars MB2 and MB3.5 have similar compressive strengths at all testing ages. Despite B5 and B2a being designated in different BS EN 459-1 classes, MB5 and MB2a achieved the same compressive strength at one year.

These results show the importance of the chemical and mineral composition, but also demonstrate that the physical properties of the binders should be taken into consideration when predicting the behaviour of mortars. The variability of the final properties of the mortars made from binders with similar chemical characteristics is determined by their physical properties. Therefore, a model to predict mortar properties over long-term periods should consider both chemical and physical parameters. It is anticipated that, with further research, a model considering these properties will be achieved, which will in turn aid industry professionals to make an appropriate selection of materials for intervention in historic fabric and new build.

Fig. 4 (Top and above) Compressive strength results.

Conclusions

Hydraulic reactions are dependent not only on the chemical and mineral characteristics but also on the physical properties of the binders.

The calorimetric tests can be useful to study the hydraulic reactions of NHL binders and to establish the differences and similarities between the binders.

The BS EN 459-1 NHL designation assigned to different binders does not reflect the long-term properties of mortars made with such binders. Consequently, caution should be exercised when using this standard to predict mortar properties. Mortars that show lower strength at early ages (until 28 days) can achieve much higher strengths for later periods (after 90 days) and may potentially be too strong for use with historic or weak fabric. On the other hand, some mortars appear to gain lower strength, relative to others, than might be expected based on the BS EN 459-1 classification of the binder. Because MB3.5 does not continue to gain strength beyond 180 days, after 360 days it is no

stronger than MB2, whereas it is a natural assumption that the NHL 3.5 designation implies that mortar made with this binder should be stronger than one made with NHL 2.

For these reasons, it is recommended that the designation of a lime binder product into one of the three classes under BS EN 459-1 should not be used as the sole criterion for selecting a binder for conservation purposes. Research is continuing to develop a model to predict the long-term behaviour of NHL mortars based on the chemical and physical properties of the binders.

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