Natural hydraulic limes for masonry repair: hydration and workability
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ABSTRACT
Lime mortars are preferred for repair of historic masonry because their greater flexibility enables them to accommodate expansion and contraction in service without damaging the stone but there is little information on properties of mortars made with commercially available limes. Binder hydration rate and workability of mortars using Natural Hydraulic Limes of grades NHL2, NHL3.5 and NHL5 from four manufacturers is reported. Significant differences in the hydration rates between different limes offer the possibility of a better classification system. However, the variation in the workability between different products could pose challenges to users and specifiers of mortars for masonry repair.

1. INTRODUCTION
Lime binders have been used in masonry for centuries. The development of Portland cement in the 19th century, with its superior setting and strength gain, even under water, led to a severe decline in the use of lime. However, when the use of cement mortar for repair and repointing of stone masonry was found to cause severe damage and loss of historically-important fabric, interest in the use of lime binders in repair materials was rekindled. Lime mortars are more flexible and better able to accommodate expansion and contraction in the masonry, while the much harder cement mortars concentrate the stresses into the stone masonry units, which subsequently deteriorate. Additionally, lime binders are perceived as more environmentally friendly than cements [1,2].

Commercially available Natural Hydraulic Limes are classified according to their hydraulicity, conferred by the presence of C₂S, and are assigned to grades NHL2, NHL3.5 and NHL5 according to their compressive strength in an arbitrarily defined standard laboratory mortar [3]. However, these grades do not necessarily align with performance in use and specifiers of limes for masonry repair are hindered by a lack of information on the mortars used in practice. The objective of this work, as part of a Knowledge Transfer Partnership between Heriot-Watt University and Historic Scotland, was to compare the properties of alternative natural hydraulic limes from four manufacturers, commercially available in the UK. This paper reports the rate of heat evolution in a conduction calorimeter and the workability of mortars made with two sands.

2. MATERIALS
NHL2, NHL3.5 and NHL5 lime binders were obtained from four UK suppliers – Hanson (www.hanson.com), originating from France, Otterbein (http://www.zkw-otterbein.de), Germany, St Astier (www.stastier.co.uk) France, and Singleton Birch (http://www.lime-mortars.co.uk) Portugal - and typical properties are shown in Table 1. These vary between suppliers but it can be seen that as the strength class increases the C₂S content increases and the free Ca(OH)₂ decreases. There is essentially no C₃S and the aluminate content is around 1-2% in these products. Hanson do not supply an NHL5 lime, so the testing programme used 11 products.

<table>
<thead>
<tr>
<th>Table 1. Range of properties of Natural Hydraulic Limes</th>
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<tr>
<td>Binder</td>
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<tr>
<td>Bulk density kg/m³</td>
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<tr>
<td>% Free Ca(OH)₂</td>
</tr>
<tr>
<td>% Unburnt CaCO₃</td>
</tr>
<tr>
<td>% Insoluble residue</td>
</tr>
<tr>
<td>% C₂S</td>
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Two mortar sands were used – Loanleven, Perthshire, a mainly quartzitic sand, and Cloddach, Morayshire, predominantly granitic. Figure 1 shows that Cloddach’s particle size distribution is slightly finer. Additionally,
Cloddach sand has a slightly lower bulk density and higher voids ratio than Loanleven sand.

![Particle size distribution of the sands.](image)

**Figure 1.** Particle size distribution of the sands.

3. METHODS

Using the Wexham Developments (Reading, UK) JAF conduction calorimeter, 20g of each of the 11 NHLs was mixed with 14g of DI water by kneading in a sealed polythene bag which was placed carefully around the heat sensor. The calorimeter was sealed and held at constant temperature of 20°C in a water bath for 72 hours. Each experimental run was calibrated in accordance with the manufacturer’s instructions and the data processed using Excel®.

Due to time restrictions, mortars were prepared using only the NHL3.5 limes at a binder/sand ratio 1:2.5 by volume, calculated using the bulk densities of each material. Each mortar was prepared at several water/binder ratios, by successive addition of water to the parent mix, following a strict timetable to ensure comparability of the results. These repeated tests enabled a first assessment of the relationship between workability and water content for each binder to be made. The weighed amounts of lime and sand were placed in the bowl of a mixer complying with BS EN 196-1 and mixed dry by hand for 1 minute. Half of the water was added and mixed at low speed for 2 minutes, followed by the remainder and mixed for 7.5 minutes. When stationary, the bowl was then scraped to move any mortar adhering to the walls back into the body of the material, and mixing restarted for a further 7.5 minutes. The mortar was then tested and returned to the mixing bowl and more water added to achieve the new water content. This was repeated on a strict 20 minute time interval to give 4-5 results.

The workability of each mortar was determined with the Flow test according to BS EN 459-2 and the rheology measured in the Viskomat NT (Schleibinger, Germany) apparatus following the two-point principle [4,5]. The torque $T$ exerted on a stationary paddle during rotation of a cylinder containing the mortar was measured as the speed of rotation $N$ increased to 200 rev/min over 2 minutes and decreased to zero again over 2 minutes. Mortars conform to the Bingham model, with the intercept $g$ corresponding to the yield stress and the slope of the line $h$ corresponding to the plastic viscosity:

$$T = g + hN.$$

4. RESULTS AND DISCUSSION

4.1 Heat of hydration

The rate of heat evolution over 72 hours for each of the natural hydraulic limes is shown in Figure 2.

![Rate of heat evolution of NHL2s (top), NHL3.5s (middle) and NHL5s (bottom).](image)

**Figure 2.** Rate of heat evolution of NHL2s (top), NHL3.5s (middle) and NHL5s (bottom).

All the curves showed relatively high initial values due to the heat of wetting and the initial dissolution of free lime, although the rate of heat evolution is much less than is observed in cement. After this the rate of heat evolution decreases over time but with thermal events which are more pronounced in the NHL3.5 and NHL5 binders. Otterbein 3.5 and 5 showed a peak at about 5 hours, while St Astier 5 peaked at about 8 hours and Singleton Birch 3.5 and 5 peaked at 20-25 hours. The absence of thermal peaks in the NHL2 binders but their presence in the NHL3.5 and 5 binders is consistent with the hydraulicity differences: there is insufficient hydraulic matter in the NHL2s to show any
detectable hydration in the conduction calorimeter. The time of the later thermal events is consistent with the slow reaction of dicalcium silicate.

The cumulative heat evolution over 72 hours is shown in Figure 3. The results are not consistent with the grades assigned by BS EN 459-1. The cumulative heat evolved from limes of the same grade covers a range from a 2.5-fold difference (NHL2) through 1.9-fold (NHL3.5) to 1.7-fold (NHL5). Singleton Birch NHL3.5 and 5 are significantly more reactive than the others of the same grade, due to the heat evolution at 20-25 hours. Finally, Otterbein’s cumulative heat evolved decreases from NHL2 to 3.5 to 5, but as expected from the heat evolution rate the cumulative trace rises rapidly at first and then more slowly.

4.2 Workability – flow test

Figure 4 shows the relationship between flow and water/binder ratio for NHL3.5 limes with the two sands. Whilst the measured flow increases with increasing water/binder ratio, there is a complex interaction between the effect of type of binder and type of sand on the water/binder ratio needed for a particular flow. Hanson and Singleton Birch require less water when used with Cloddach sand than with Loanleven, whereas Otterbein and St Astier require more water with Cloddach sand than with Loanleven. As noted above, Cloddach is slightly finer with a lower bulk density and higher voids ratio than Loanleven sand. Table 2 gives the measured bulk density of each of the NHL3.5 limes but the differences do not explain the differences in water/binder ratio.

Figure 4. Effect of water/binder ratio on flow (NHL3.5).

Table 2. Bulk density of NHL3.5 limes

<table>
<thead>
<tr>
<th>Lime</th>
<th>Hanson</th>
<th>Otterbein</th>
<th>St Astier</th>
<th>Singleton Birch</th>
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<tbody>
<tr>
<td>Bulk density kg/m³</td>
<td>710</td>
<td>610</td>
<td>776</td>
<td>863</td>
</tr>
</tbody>
</table>

4.3 Rheology – two-point test

Figure 5 shows the general form of the flow curve of a lime mortar tested as described above in the Viskomat NT. The hysteresis loop between the up- and down-curves is due to the breakdown of structure formed by the flocculation of particles in water, and the parameters g and h, corresponding to yield stress and plastic viscosity respectively, are obtained by the best fit straight line through the points on the downcurve.

Figure 6 shows the relationship between yield stress, plastic viscosity and water/binder ratio for NHL3.5 limes with the two sands. In most cases the yield stress follows the expected
decreasing trend with increasing water/binder ratio [5] but only two plastic viscosity results show this effect. In part this is because some of the stiffer mortars tended to stick around the impeller and slip against the wall of the container: this tends to make the value unreliable. The same complex interaction between the effect of type of binder and type of sand on the water/binder ratio needed for a particular yield stress is visible.

5. DISCUSSION

The rate of heat evolution of lime binders is considerably less than that of cements, for which the conduction calorimeter was designed, but differences between the limes are clearly visible. The total (cumulative) heat evolved over 72 hours hydration is at variance with the NHL classification assigned in the BS EN 459-1 hydraulicity test and this raises the important question of whether heat evolution would be a more discriminating test of hydraulicity.

Within an overall relationship with water/binder ratio, workability depends in a complex way on the physical properties of the binder and sand. This is particularly important because in practice mortars are proportioned by volume and the different bulk densities of the binder interacts with the voids ratio in the sand. Controlling workability by water content, as done in practice, may mean that specifying mortar by grade of binder may not achieve the expected properties: a binder of low bulk density may produce mortar of high water/binder ratio and poor strength and durability. Clearly data on mortars using NHL2 and 5 binders is needed to complete the story.

6. CONCLUSIONS

These preliminary results confirm that not all natural hydraulic limes show the same performance in mortar, even when they are graded the same by BS EN 459-1. This has significant implications for the use of lime mortar in practice.

REFERENCES