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## Hygrothermal properties of NHL mortars

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**Abstract** – Hygrothermal simulation software enables designers to assess the impact of insulation retrofits on solid walls before a retrofit project starts and help to avoid the negative effects on solid wall performance. However, the material databases used by the software lack information on the material properties of the traditional masonry used in Scotland. The hygrothermal properties of uncarbonated and carbonated lime mortar made with St Astier NHL2, NHL3.5 and NHL5 and Loanleven sand have been determined by laboratory testing. Thermal conductivity, sorptivity or water absorption coefficient by partial immersion, water vapour permeability (dry and wet cup), hygroscopic sorption, density and porosity results are presented.

Keywords – Natural hydraulic lime; mortar; hygrothermal properties.

### 1. INTRODUCTION

Water can cause building fabric deterioration and create unhealthy indoor environments. Its transport is closely linked to heat transfer and traditional solid masonry walls breathe. This moisture transfer ability determines their performance and durability. Installing more insulation to reduce energy consumption risks upsetting the equilibrium within a wall because energy efficiency retrofits almost always change the moisture performance of masonry. Hygrothermal simulation software enables designers to assess the impact of insulation retrofits on solid walls before a retrofit project starts and helps to avoid negative effects [1,2]. However, the material databases used by the software lack information on the material properties of traditional masonry. The aim of this work was to contribute to a dataset of material properties which can be used by designers to ensure that retrofits meet the needs of traditional buildings as well as achieve energy efficiency. This paper focuses on mortars made with contemporary natural hydraulic limes (NHLs) since these are common in northern Europe: it reports the hygrothermal properties of uncarbonated and carbonated NHL mortars. Air limes (CL90) and selected building stones will be reported at a later stage.

### 2. EXPERIMENTAL PROCEDURE

#### 2.1 MATERIALS AND SPECIMEN PREPARATION

Three mortars comprising NHL2, NHL3.5 and NHL5 (St Astier, France) and oven-dry Loanleven concreting sand in the proportions shown in table 1 (binder:sand 1:3 by volume) were prepared. These NHLs are the most commonly used in Scotland, which lacks indigenous lime manufacture, and the proportions are those recommended by Historic Environment Scotland [3]. Table 2 gives the particle size distribution of the sand. 12 litres of each mortar were mixed in a Hobart 20 litre mixer for five minutes and tested using a standard (BS EN 459-2) flow table apparatus. The water was adjusted to achieve 150mm flow (NHL2 and NHL3.5) and 160mm flow (NHL5). Mortars were then cast into 100mm cubes (steel moulds) and 360 x 240 x 12mm thick tiles (timber moulds lined with cling film). Before the mortar had set the tiles were cut into 90mm diameter discs using a simple cylindrical steel “cookie cutter” device and both cubes and tiles were allowed to harden in the moulds, covered in polythene sheet, for 7 days. The specimens were demoulded and separated into two groups. Upon demoulding, the use of the cookie cutter had made it easy to separate the discs

from the surrounding fragments. Half of the cubes, discs and fragments were stored at 20°C in airtight drums to ensure they remained saturated. The other half were transferred to a TAS Series 3 controlled environment chamber and stored at 20°C, 60% relative humidity (RH) and 600ppm CO<sub>2</sub> until 56 days of age. These curing and exposure conditions had been used previously to ensure full carbonation of the specimens [4]. The extent of carbonation was confirmed by spraying freshly fractured surfaces with 1% phenolphthalein solution in alcohol as indicator. At the end of this process a set of uncarbonated and fully carbonated mortars with each NHL was available for further testing.

Table 1. Batch weights of mortar mixes

Binder type	NHL2	NHL3.5	NHL5
Mass of binder g	2090	2356	2700
Mass of sand g	15200	15200	15200
Mass of water g	2812	2640	2718
Flow mm	150	150	160

Table 2. Particle size distribution of Loanleven sand

Sieve size mm	0.063	0.125	0.250	0.5	1.0	2.0	4.0
% passing	4.0	18.5	42.5	66.5	80.5	95.5	100

## 2.2 THERMAL CONDUCTIVITY

Thermal conductivity was measured by the thermal probe method according to ASTM D5334-14, using the Thermtest TLS-100 instrument (Thermtest inc, Fredericton, Canada). It proved to be impracticable to drill a 3mm test hole 100mm deep in the centre of a specimen so a 3x3mm groove was cut in the surface of a cube of each mortar, using a stone cutting disc mounted in a hand-held angle grinder. The cubes were conditioned by exposure to 60% RH until constant mass and then the test probe was laid in the groove, surrounded in silicone thermal grease (RS Components, UK, thermal conductivity 5W/mK), and covered with a second cube of the same mortar. This ensured that the required test condition of a minimum of 50mm depth of mortar around the probe was met. The test was replicated on three cubes.

## 2.3 SORPTIVITY AND WATER ABSORPTION COEFFICIENT

Water sorptivity and water absorption coefficient were determined gravimetrically by immersing one cast face and the trowelled face of cubes 2mm deep in water and weighing at intervals over a 24 hour period or until water was visible on the top face, according to BS EN ISO 15148:2002. The test was replicated on three cubes.

## 2.4 WATER VAPOUR PERMEABILITY

The water vapour permeability was determined using both dry cup and wet cup methods according to BS EN ISO 12572:2001. The 90mm diameter discs were cured or carbonated as described above

and then conditioned by exposure to 60% RH and sealed into aluminium cups. For the dry cup tests the cups contained desiccant (anhydrous  $\text{CaCl}_2$ ) and for the wet cup tests they contained saturated  $\text{KNO}_3$  solution (93% RH). Having first trimmed any irregularities in the circumference, discs were placed (cast face down) onto a narrow bead of silicone sealant (B&Q plc) to create an air- and liquid-tight seal. The residual gap between the irregular circumference and the wall of the cup was sealed using molten paraffin wax (Akros Organics, Belgium). The assembled discs/cups were placed in the environment chamber at 20°C and 60% rh and weighed at the same time every week. At least three discs were tested for each combination of NHL class and carbonation state in both dry and wet cup tests.

## 2.5 HYGROSCOPIC SORPTION

The moisture absorption and desorption curves were determined according to BS EN ISO 12571:2013. Fragments of each mortar, weighing approximately 30g, were oven dried to constant mass then placed successively in airtight boxes containing saturated salt solutions, giving 33% ( $\text{MgCl}_2$ ), 53% ( $\text{Mg}(\text{NO}_3)_2$ ), 75% ( $\text{NaCl}$ ), 85% ( $\text{KCl}$ ) and 93% ( $\text{KNO}_3$ ) RH. At each RH the fragments were weighed at intervals until constant mass (achieved in typically 2-3 days). The absorption curves were determined at successively increasing RH, after which the desorption curves were obtained at successively decreasing RH until a final oven dried mass gave a confirmation value. The test was replicated on three fragments of each combination of NHL class and carbonation state.

## 2.6 DENSITY

The density of hardened mortar was determined by displacement of water. The cubes saturated after completion of the sorptivity test were suspended beneath a balance and then weighed both in air and when immersed in water. The cubes were then oven dried to constant mass and the test repeated, taking care to complete the weighing quickly before significant absorption had occurred. The density of the water used in the calculation was that for the measured temperature [5].

## 2.7 POROSITY

Porosity and pore size distribution were determined by mercury intrusion porosimetry using a Quantachrome PoreMaster33 instrument, using a sample cell of 8 mm × 20 mm and capillary volume of 0.5 ml, with 33000 psi final pressure, on fragments of each mortar of mass approximately 1g.

# 3. RESULTS

## 3.1 THERMAL CONDUCTIVITY

Table 3 shows the thermal conductivity of the NHL mortars. Each entry is the mean of a single measurement on each of three cubes. The standard error, estimated from the variances about the mean values, is about 0.35. This suggests that only the differences between dryness and saturation are significant and that the trends of increasing thermal conductivity with binder hydraulicity and decreasing thermal conductivity upon carbonation are within experimental error.

Table 3. Thermal conductivity of NHL mortars, W/mK (means of 3 cubes)

Binder type		NHL2	NHL3.5	NHL5
Uncarbonated	Dry	0.71	0.73	0.91
	Saturated	1.16	1.30	1.79

Carbonated	Dry	0.62	0.69	0.68
	Saturated	1.03	1.22	1.29

### 3.2 SORPTIVITY AND WATER ABSORPTION COEFFICIENT

Fig. 1 shows a typical set of sorptivity data, exemplifying the linear relationship with  $\sqrt{\text{time}}$ : the points at 24 hours, and often those at 6 hours, fall below the line because the water front has reached the free top surface of the specimen and no further mass gain can take place. Tables 4 and 5 show the sorptivity and water absorption coefficient, respectively. In all cases, the trowelled face has a lower sorptivity than the cast face, and the uncarbonated mortars have a lower sorptivity than the carbonated mortars. The sorptivity of uncarbonated mortars decreases with increasing hydraulicity of the binder, but that of the carbonated mortars decreases from NHL2 to a minimum value with NHL3.5 and reverts to a similar value of sorptivity with NHL5.

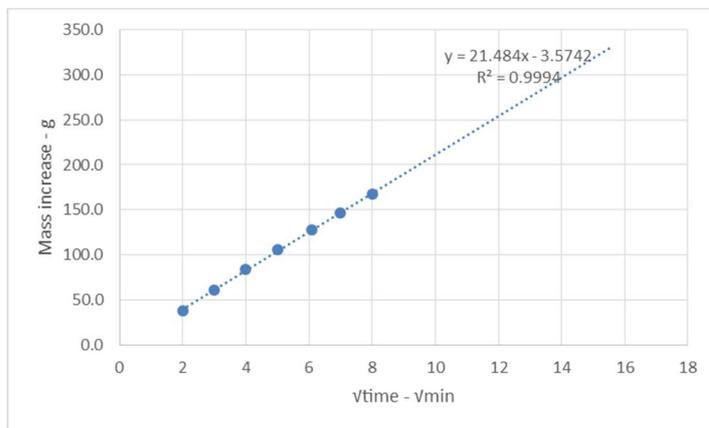


Figure 1. Typical sorptivity raw data for a carbonated NHL2 mortar cube

Table 4. Sorptivity (mm/ $\sqrt{\text{sec}}$ ) and water absorption coefficient ( $\text{kg}/\text{m}^2\sqrt{\text{sec}}$ ) of mortars (means of 3 cubes)

Binder type		NHL2		NHL3.5		NHL5	
		sorptivity	wac	sorptivity	wac	sorptivity	wac
Uncarbonated	Trowelled face	1.25	9.7	0.62	4.8	0.41	3.2
	Cast face	1.64	12.7	0.89	6.9	0.79	6.1
Carbonated	Trowelled face	1.59	12.3	0.95	7.4	1.70	13.2
	Cast face	2.04	15.8	1.55	12.0	2.51	19.4

### 3.3 WATER VAPOUR PERMEABILITY

Fig. 2 shows graphs of mass gain or loss obtained from the dry cup and wet cup tests, respectively. The examples chosen have the lowest values of  $R^2$ , the coefficient of determination, shown by any sample in the two test methods. The values of  $R^2$  were somewhat lower (0.9931-0.9986) for the dry cup tests than for the wet cup tests (0.9984-1.0), suggesting that the dry cup tests are more sensitive to experimental conditions. Mass changes were recorded over 8 weeks and the rate of change of

mass taken as that calculated by linear regression using the 6 consecutive points that gave the highest value of  $R^2$ . BS EN ISO 12572:2001 defines an acceptance criterion for the data, which requires five successive changes in mass (i.e. six consecutive points) to be within  $\pm 5\%$  of the average change. This corresponds to a value of  $R^2$  of 0.9998 and was achieved by 11 out of the 42 specimens tested.

Table 5 shows the coefficient of water vapour permeability for all the mortars tested. The wet cup test gives a consistently higher value than the dry cup test. The difference between carbonated and uncarbonated NHL2 and NHL3.5 mortars is not significant but for NHL5 the carbonated mortar has a lower permeability than the uncarbonated one.

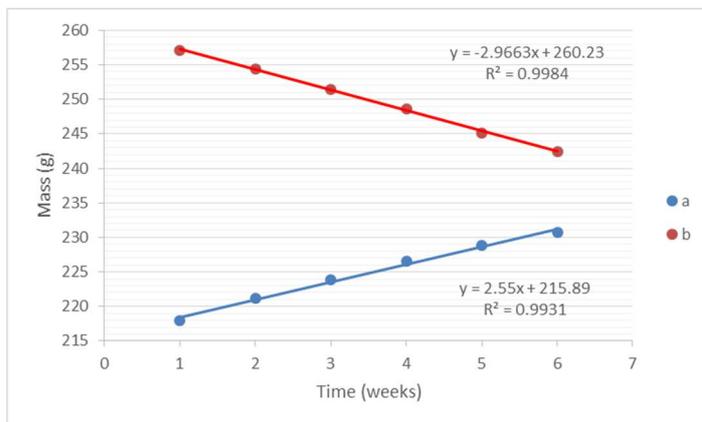


Figure 2. Examples of mass gain/loss curves for wet and dry cup tests: (a) wet cup, carbonated NHL3.5, (b) dry cup, uncarbonated NHL5 mortar

Table 5. Water vapour permeability of NHL mortars, 10-12 kg/(m sec Pa) (means of 4 or 3\* discs)

Binder type	NHL2	NHL3.5	NHL5
Uncarbonated			
Dry cup	10.3	11.1	9.6*
Wet cup	19.6*	22.4*	20.0*
Carbonated			
Dry cup	11.9	11.2	7.1*
Wet cup	23.6	25.7	16.3*

### 3.4 HYGROSCOPIC SORPTION

Fig. 3 and 4 show curves of moisture content against relative humidity for uncarbonated and carbonated NHL mortars, respectively. Every curve exhibits hysteresis, with the desorption curve at higher moisture content than the absorption curve and there is a consistent dip in the desorption curve at 85% relative humidity, which may be an experimental artefact caused by replenishing the solution in the chamber during the test. The moisture content of the uncarbonated mortars is consistently higher than that of the carbonated mortars at all relative humidities but there is no significant difference between the binder types.

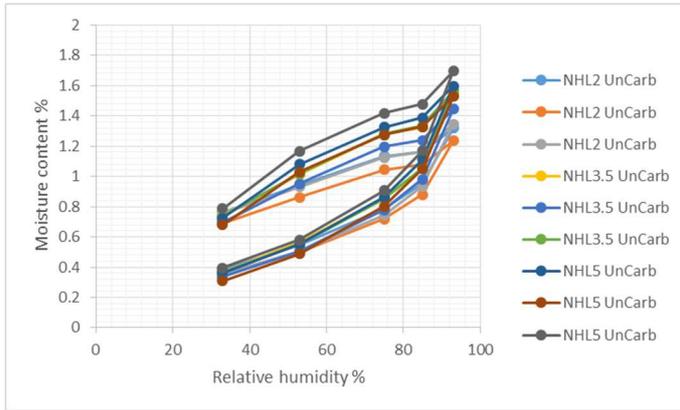


Figure 3. Variation of moisture content with relative humidity for uncarbonated mortars

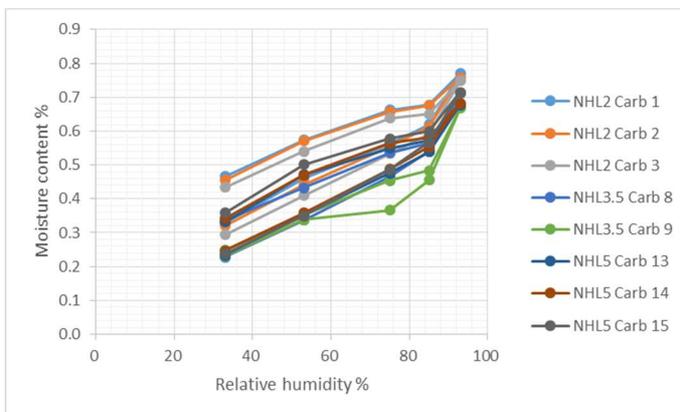


Figure 4. Variation of moisture content with relative humidity for carbonated mortars

### 3.5 DENSITY

Table 6 shows the density of all the mortars in the saturated and oven-dry states, together with the corresponding saturated moisture contents (mc). The carbonated mortars are slightly denser than the uncarbonated mortars.

Table 6. Density of NHL mortars, kg/m<sup>3</sup> (means of 3 cubes)

Binder type		NHL2	NHL3.5	NHL5
Uncarbonated	Oven dry	1911	1811	1949
	Saturated	2110	1989	2148
	% mc at saturation	10.4	9.8	10.2
Carbonated	Oven dry	1937	1806	1971
	Saturated	2121	1992	2166
	% mc at saturation	9.5	10.3	9.9

### 3.6 POROSITY

Fig. 5 shows the pore size distributions of the uncarbonated and carbonated mortars. The uncarbonated mortars show a broadly bimodal distribution with two peaks at around 0.08-0.11 and 1.2-1.4  $\mu\text{m}$  (NHL3.5 and NHL5) and at 0.4 and 3.6  $\mu\text{m}$  (NHL2), whereas in the carbonated mortars the distribution is strongly unimodal with a peak at 1.3-1.6  $\mu\text{m}$  (NHL3.5 and NHL5) and 0.7  $\mu\text{m}$  (NHL2). Table 7 shows the total porosity of the mortars. The porosity of the carbonated mortars is consistently 2-3% higher than that of the uncarbonated mortars.

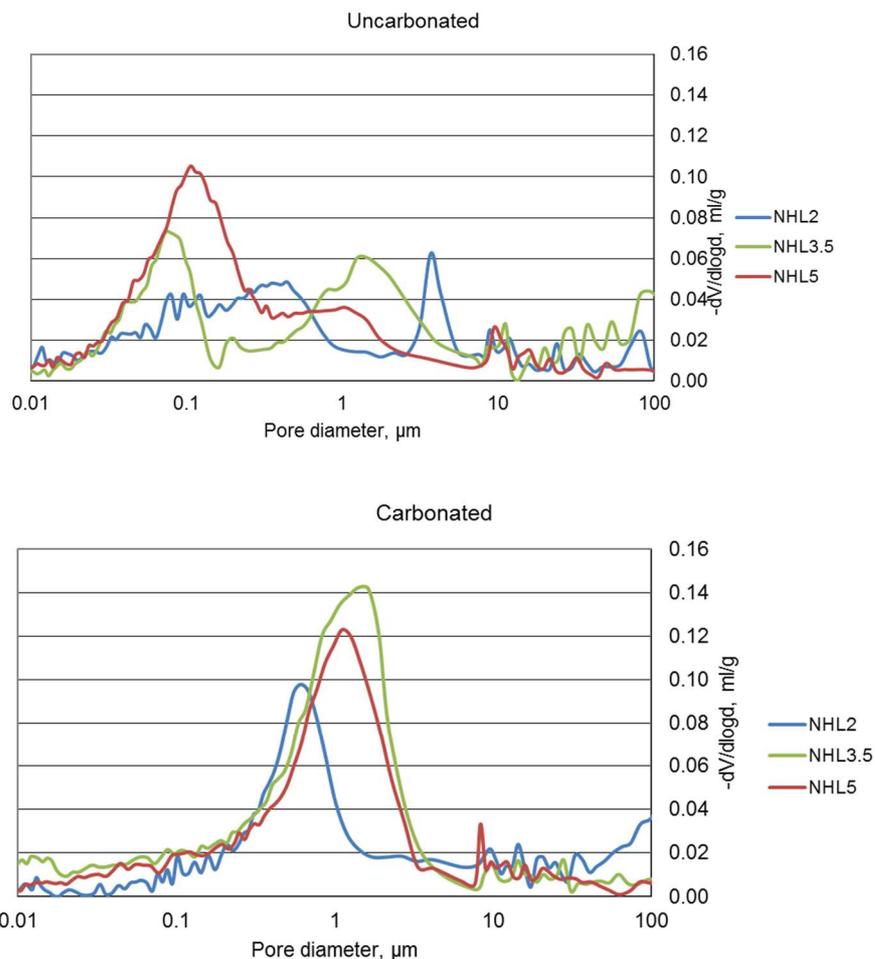


Figure 5. Pore size distributions of (upper) uncarbonated and (lower) carbonated mortars

Table 7. Total porosity of NHL mortars, %

Binder type	NHL2	NHL3.5	NHL5
Uncarbonated	19.1	24.0	24.1
Carbonated	21.8	27.7	25.2

### 4. DISCUSSION

The results are consistent with the known effect of carbonation of NHL. Large tabular hexagonal crystals of  $\text{Ca}(\text{OH})_2$  exhibiting a bimodal pore size distribution transform into small cubic crystals of the denser  $\text{CaCO}_3$  which fill the pores [6]. The resulting pore size distribution has a single peak at

around 1  $\mu\text{m}$ , as is clearly visible in scanning electron micrographs [6]. This accounts for the resulting increase in sorptivity and water absorption coefficient, which are both dominated by the larger capillaries. The slight increase in mortar density as a result of carbonation is explained by the uptake of  $\text{CO}_2$ . The insignificant differences in thermal conductivity and water vapour permeability suggest that these are not affected by the pore size distribution. That the hygroscopic sorption is larger in uncarbonated than in carbonated mortars is probably due to the stronger affinity between  $\text{Ca}(\text{OH})_2$  and water than between  $\text{CaCO}_3$  and water.

In the context of assessing the thermal performance of historic masonry these results are important because, even though a wall is referred to as stone masonry, the mortar accounts for as much as 40% of the material [7], so whilst the properties of building stones are widely available there is a significant contribution made by the mortar. Additionally, the differences between uncarbonated and carbonated mortars may need to be taken into account because it is likely that the core of a wall may remain uncarbonated (and exhibiting a greater degree of hygroscopicity) for a very long time, whilst the surface zones may be carbonated in a few years, depending upon the climatic conditions they experience. This issue is likely to require care in the choice of values for simulation and justifies further experimental investigations on the materials used historically, including the air limes used in other regions.

## 5. CONCLUSIONS

Relevant hygroscopic properties of NHL mortars have been determined and can contribute to their successful utilisation in hygrothermal simulation of the effect of retrofitting insulation in historic buildings.

## 6. ACKNOWLEDGEMENTS

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