



Construction Materials Consultants Ltd.

Wallace House, Whitehouse Road, Stirling, FK7 7TA
Tel 01786 434708 Fax 01786 475133
E-mail mail@cmcstirling.co.uk

Report on the Examination and Analysis of a Mortar Sample from a Stepped Footing in a Medieval Town Wall

Near the former Beverley Gate, Hull

Prepared and Approved By

W A Revie

Prepared for

The Earth Stone & Lime Company
Hall Farm
Maltongate
Thornton Dale
Pickering
North Yorkshire YO18 7SA

Date Issued

9th January 2018

Our Ref: M/1826/17
Your Ref: Town Wall, Hull
Doc. Ref: M182617R1



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1.0 Introduction

A sample of material stated to be mortar from a stepped footing in a brick built Medieval Town Wall, at the former Beverley Gate in Hull, was received in CMC's Stirling Laboratory on the 22nd November 2017.

The sample was received with a request for it to be examined and analysed to identify the type of binder, and the form in which it was used, in the mortar at time of construction. In addition, an attempt was to be made to establish the mix composition of the mortar.

This report details observations from a macroscopic and microscopic examination of the sample along with the results of an analysis carried out on the sample received. The report concludes with comment on the composition of the mortar and the binder type and the form in which the mortar was prepared and used.

2.0 Sample

A sample was received in CMC's Stirling laboratory on the 22nd November 2017, with the sample submitted by Nigel Copsey of The Earth, Stone & Lime Company. On receipt in the laboratory the sample details were entered into the sample register and the unique sample reference SR2525 allocated, with the laboratory and Client sample reference reproduced below:

CMC Sample Ref.	Location/Comment
SR2525 – S1	Bedding mortar from the stepped brick footing in a Medieval Town Wall, adjacent to the former Beverly Gate, Hull

3.0 Methods of Examination and Analysis

Following an initial examination of the sample received was prepared and submitted to the following laboratory programme for examination and analysis:

The sample was initially submitted to a detailed examination with the aid of a stereo-binocular microscope, at magnifications up to x 20 to assist in assessing it's as-received condition. During this examination, small sub-sample was exposed to a range of indicator solutions and reagents to assist in assessing the sample composition.

Following the initial examination, a sub-sample was prepared from the sample for analysis by X-ray Powder Diffraction (XRD). This form of analysis was employed to permit identification of the crystalline components present in the materials under examination. This to aid identification of the binder type employed in the mortar.

As the quantity of mortar present in the sample was too small to permit mix composition by acid digestion, a petrographic thin section was prepared from the largest intact area of mortar, adhering to one of the brick fragments. The thin section was submitted to a microscopic examination, to assist in clarifying the form of the lime used and permit the mix composition of the mortar to be determined by modal analysis.

4.0 Macroscopic Examination

Observations from the examination of the sample are presented below, along with a summary of the properties of the sample, as received. The mass and dimensions relate to pieces of brick with thin coatings of adhering mortar, and not purely the mortar:

CMC Sample Ref	Client Ref	Mass Received (grams)	Size of Intact Piece (mm)	Colour ¹ Sample Munsell Chart
SR2525-S1	Bedding Mortar	275.5	71.1 x 41.7 x 50.3	Mortar - 7.5YR 8/1 "White" Brick - 2.5YR 6/6 "Light Red"



Plate No. 1: The above shows the sample as received with the mortar coated face of the brick fragments shown. The inner surfaces were of fractured soft brick and are shown below.



Plate No. 2: The fractured, mortar free surfaces, of the brick pieces are shown above.

¹ The colour was assessed by comparison against the Munsell Soil Colour Charts.



Plates No. 3 & 4: Both of the above show examples of the mortar adhering to pieces of the brick, which was typical of all of the pieces received.



Plates No. 5 & 6: The left plate shows a view of the surface of the mortar adhering to the brick fragment in Plate No. 4 above. This being the thickest depth of mortar on any of the pieces received was used for thin section preparation. The right plate shows a close-up of one of the large lime inclusions observed in the mortar.

The mortar and brick pieces were received in a damp condition and the mortar was noted to be soft and very greasy to touch, which was typical of a high calcium air lime binder. On drying the mortar was found to be compact but locally friable. However, mortar could only be separated from the dried brick pieces under firm finger pressure, but could be removed under moderate nail pressure and once separated it could be powdered with relative ease.

The mortar was noted to be strongly bonded to the brick to which it adhered, and it had the appearance and texture of a typical lime mortar, which contained a number of lime inclusions. These were sub-round to sub-angular in shape, with the largest observed being 9.3mm in size.

The mortar was found to be fully carbonated and from tests using water droplets, it was indicated that the mortar contain a well-connected pore structure, into which water was rapidly absorbed and diffused through the full thickness of the mortar.

The aggregates in the mortar had the appearance of a natural quartz rich sand, containing flint fragments, fine sandstone and siltstone fragments along with indeterminate lithic fragments and a low abundance of shell. Brick fragments and ash clinker were also noted to be present. The sub-rounded shape of the majority of the natural sand grains and the presence of shell would infer an estuary or beach source. The aggregate also contained a proportion of charcoal and ash, perhaps included with the binder or a contaminant of the aggregate, or perhaps added as a pozzolan.

The brick fragments to which the mortar adhered had the appearance of low fired, or sun-dried clay brick, they were highly porous, and although firm they could be broken and disrupted under firm finger pressure.

5.0 Microscopic Examination

A petrographic thin section was prepared from a slice sawn from the surface of the piece of brick retaining the greatest thickness of mortar, with the slice aligned to permit the maximum area possible on the slide.

The sample was prepared for thin sectioning by initially drying the slice to a constant weight at 60°C prior to impregnating the dried sub-sample with an epoxy resin containing a fluorescent blue dye. One side the impregnated slice was polished and mounted onto a glass slide (50mm x 75mm). The mounted sample was then ground and polished to give a thickness of approximately 30microns.

The microscopic examination of the thin section was undertaken using an Olympus BH2 Polarised light microscope, fitted with a Digital Camera, to permit the recording of images of areas of note for record purposes.

The presence of dyed epoxy resin enables detailed analysis of void distribution, an assessment of microporosity and a clear indication of any crack patterns present, in plane polarised light.

The sample was also mounted into a 'Swift automatic stage' to permit the proportion of the components present to be determined by modal analysis, permitting the determination of the mix composition. The observations from the examination of the sample are presented below:

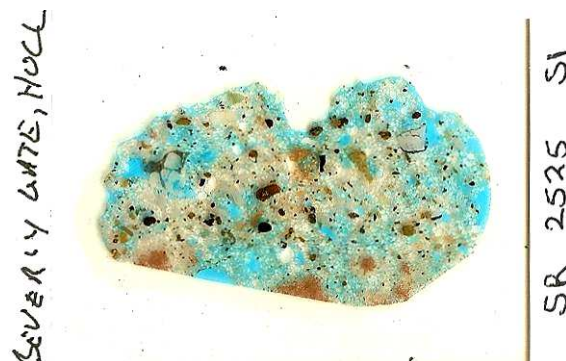


Plate No. 7:

Thin section prepared from a piece of mortar for microscopic examination and modal analysis.

Aggregate

This aggregates in the mortar sample contains a mixed suite of rock types, including quartz, quartzite, flint, igneous rock fragments, sandstone, siltstone, along with brick fragments, shell fragments, opaque materials (coal/charcoal), with a small quantity of ash clinker.

The aggregates are rounded to sub-round in shape, with a low proportion of elongated aggregate particles also present. In addition there were small smooth rounded brick fragments, and coal, which along with shell fragments may infer that the aggregates were won from an estuary or beach source.

The aggregates range in size from 0.04mm to 3.7mm (coarse silt to very coarse sand and fine gravel) in the section examined. With no clay or fine silt material observed, it is again inferred that a water transported material was used as the aggregate.

The aggregates are mostly well bound within the paste. Although peripheral microcracks and localised areas of high microporosity, with localised binder depletion by leaching were noted, they do not appear to have been detrimental to the performance of the mortar.

Binder

The binder is typical of a lime mortar that was made with a high calcium, non-hydraulic, lime. Although generally dense, the paste displays random patches of high microporosity and locally it is transected by a network of fine shrinkage cracks. This is consistent with binder rich mixes that have undergone early plastic and long term drying shrinkage.

Locally there were small orange brown coloured to black and amorphous particles observed to be present, some of which appear to display reaction rims or have partially altered. However, these appear to be either residue from ash from the kiln or from ash added with the aggregate, rather than have formed within the limestone that was calcined. And although some of this material may be potentially pozzolanic, in nature, most show no reaction and it will, therefore, have contributed little, if anything, to the properties of the binder, which is basically that of a high calcium air lime.

The paste is fully carbonated and unhydrated lime inclusions were not observed. The lime inclusions apparent, include both rounded to sub-round and angular to sub-angular inclusions, with the majority having the appearance of having formed from unmixed putty at the time of placing.

The more angular inclusions, however, are typical of particles of incompletely slaked quicklime, at the time of placing. Lime inclusions range in size from 0.2mm to 3.4mm in the section examined, with only a small number of these showing well defined margins with little evidence of diffusion into the surrounding paste, but without perimeter cracking. Whereas, most of the rounded inclusions (putty) display partial diffusion into the surrounding paste.

From the examination of this sample it is indicated that the mortar was probably mixed with the aggregate, in the form of a quicklime, but it was well slaked prior to placing. However, in the absence of significant shrinkage crack development and the patchy microporosity observed, the mortar may have been made in the form of a "hot lime mortar" in which most of the quicklime had slaked, at the time of mixing, with the mortar placed after remixing, and likely to have been placed cold. These practices being typical of those followed in early brick masonry construction.

Voids and microcracks

Voids are abundant as both entrapped air voids, which are typically round to sub-round in shape and up to 3.5mm in size, but mostly <1.6mm, and those formed from leaching and binder depletion, which are commonly irregular, sub-angular or elongated/flaky in shape. Many of the voids are locally lined by fine fringes of calcite, with uncommon and very localised clusters of gypsum crystals.

Cracks are rare and occur as localised features peripheral to, and linking; larger aggregate particles. The cracks are very fine, ranging in width from <0.02mm to 0.13mm, and are typical of drying shrinkage features.

The results of a point count (modal) analysis are presented in the following table:

Sample Ref:	SR2525-S1	
Constituents	%	
Aggregate	Inclusions as binder	Inclusions as Aggregate
Quartz & Flint	24.4	24.4
Lithic Fragments	9.8	9.8
Shell	2.4	2.4
Amorphous/Ash/Brick	7.6	7.6
Lime inclusions & Clinker	-	13.0
Total Aggregate	44.2	57.2
Binder (Lime)	35.8	35.8
Clinker	0.8	0
Lime inclusions	12.2	0
Secondary products/Calcite	6.3	6.3
Reaction Products/Gypsum	0.7	0.7
Total Binder	55.8	42.8
Total Constituents	100.0	100.0
Cracks/Voids	14.3	14.3
Binder: Aggregate Ratio	Total	Effective
	1.0 : 0.79	1.0 : 1.34

Table No. 1: Modal Analysis carried out on thin section prepared from sample S1.

The effective binder content is calculated on the basis that the inclusions are acting as aggregate rather than as binder and is probably a truer measure of the binder content of the mix, relating to its performance as a mortar. Whereas, the total lime content is a reflection of the mix at the time the mortar was made and placed, including the inclusions (both fully slaked and unslaked) as part of the added lime binder.

Photomicrographs:

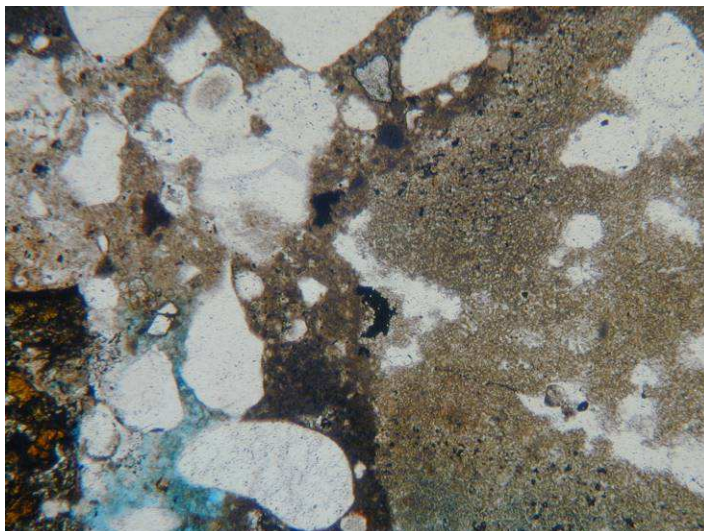


Plate No. 8:

A view in plane polarised light (ppl) showing an area paste within the mortar adjacent to a fully slaked angular lime inclusion.

The left side of the plate shows fine aggregate particles encapsulated in paste. Whereas the right side of the plate contains the inclusion, which is fully slaked and locally leached, but it does not diffuse into the mortar paste at its margins.

An ash clinker fragment can be seen in the lower left.

Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

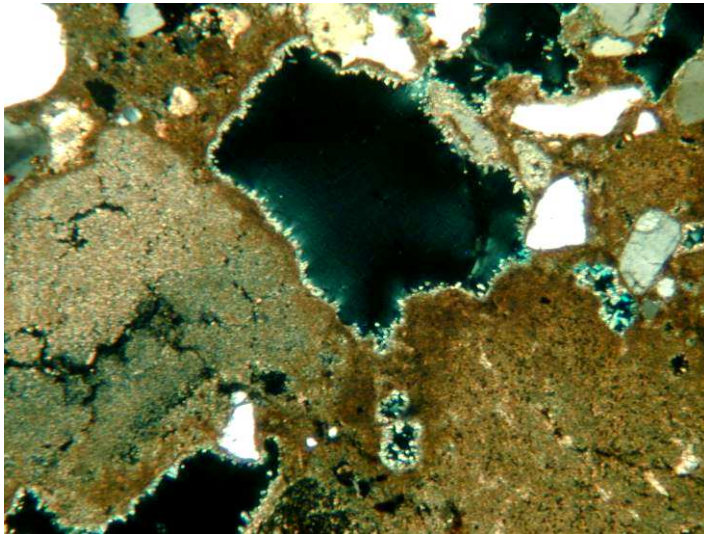


Plate No. 9:

A view in cross polarised light (xpl), of a paste rich area of the mortar, where an angular lime inclusion can be seen in the lower left with a putty inclusion in the lower right. The latter showing the typical globular structure with a fine shrinkage crack pattern throughout. The void in the centre of the plate (black) is rimmed with redeposited calcite crystals. The aggregates in the upper part of the plate are dominated by quartz grains.

Voids, impregnation resin, and amorphous material appear dark in xpl. Field of view 2.4mm.

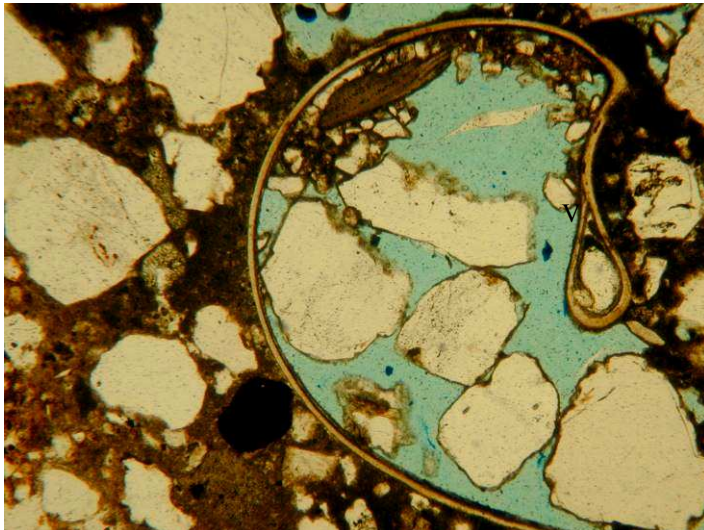


Plate No. 10:

Another view in ppl, of a voided area within a shell fragment surrounded by a dense area of mortar fabric. The sand grains within the void formed by the shell are quartz grains, as are most of the grains encapsulated within the paste to the left.

The paste is dense and fully carbonated, with the small black fragments being of coal and the small sub-rounded and vesicular particles being ash.

Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.

6.0 Analysis by X-Ray Diffraction

To assist in the identification of the composition of the binder in the mortar sample a binder rich sub-sample was prepared and submitted to analysis by X-ray Powder Diffraction (XRD).

To achieve this, a representative sub-sample of the mortar was obtained, from several brick fragments, with this ground in an agate mortar and pestle, taking care to minimise crushing of the aggregate, with a concentrated sample of the binder obtained by sieving the sample over a 63 μ m sieve. The powdered sample collected was back-packed into a proprietary sample holder for presentation in the X-Ray Diffractometer.

The prepared sample was analysed in a Philips X-ray Diffractometer fitted with a single crystal monochromator, set to run over the range 3° to 60° 2 θ in steps of 0.1° 2 θ at a rate of 1° 2 θ /minute using CuK α radiation.

The digital output from the diffractometer was analysed using a computer program, which matched the peak positions against the JCPDS International Standard Mineral Data-base sub-files using a search window of 0.1° .

The result obtained is shown in the following figure, which is appended to this report, in the form of a labelled X-ray diffractogram:

Figure No. 1 – SR2525-S1 – Binder from Bedding mortar in a stepped footing, in a brick Medieval Town Wall.

The most abundant mineral components identified are indicated in the appended diffractogram using the following short-hand notation:

cc = Calcite (CaCO_3) Calcium Carbonate, carbonated lime component, also the dominant component of any limestone in the aggregate,

ba = Bassanite ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) Calcium Sulphate Hemihydrate, most likely present as reaction product between lime in binder and environmental sulphates,

fs = Feldspar, mostly of the plagioclase group, Albite, with minor Orthoclase, common rock forming minerals, components aggregate and brick fragments,

qz = Quartz (SiO_2) dominant component of the sand grains, present as a contaminant.

From the analysis it is confirmed that the binder used in the mortar was a non-hydraulic Calcium lime. There was no evidence from the analysis of any hydraulic components or pozzolanic materials in the sample analysed.

The low abundance of calcium sulphate detected would infer that the mortar has been affected by sulphates, perhaps from groundwater or leached from the brick itself. However, the quantity is such as not to have been detrimental to the mortar analysed. This is consistent with the condition of the mortar, as represented by the sample received.

The sulphate detected in the form of Bassanite, may infer limited reaction, or the possibility of loss of moisture from gypsum, the form commonly found, in response to drying and processing in the laboratory prior to analysis, with conversion of Gypsum (calcium sulphate hydrate) to Bassanite (calcium sulphate hemi-hydrate).

8.0 Discussion

On the basis of the examination and analyses carried out on the mortar in the sample submitted it is concluded that the mortar is a lime rich mortar, where the lime is a high calcium air lime, with no evidence of any hydraulicity detected in the lime, or from pozzolanic components in the mortar.

The mortar, from as the appearance of the mix, was prepared as a Hot Mixed Mortar (HMM), where the lime was used in the form of a quicklime and was mixed with the sand prior to slaking. The slaking of the lime occurring with the sand.

However, given the abundance of putty inclusions in the mix it is likely that the mortar was remixed, perhaps after screening to remove over, and under, burnt lime particles, and oversized aggregates. With the mortar placed cold rather than it having been placed as a hot mix.

The mortar is fully carbonated and shows some evidence of leaching of binder, due to water percolation, with redeposition of lime, observed as calcite fringes, both within voids and lining fine water percolation channel ways. Locally, fine gypsum crystals were observed, but these are rare and most likely have formed in response to a reaction between the lime and water borne sulphate, perhaps leached from the brick itself.

The aggregates appear to have been obtained from a beach, estuary or river source. This assumption is made as most of the grains display sub-rounded features and water worn surfaces. Which along with a proportion of weathered rounded brick fragments, coal, ash and shell fragments in the sand would also be consistent with sands from such sources.

9.0 Quality Statement

We confirm that in the preparation of this report we have exercised reasonable skill and care.

The observations and analysis results reported and comments offered relate only to the sample of mortar adhering to brick fragments, received from Nigel Copsey of the Earth, Stone & Lime Company. Which was stated to be from the stepped footing of a Medieval Town Wall, close to the former Beverley Gate, in Hull, and was received on the 22nd November 2017.

