Kiwa CMT

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Report on the XRD and supporting chemical analysis of 1No. sample of hardened potential lime mortar

Client: **Ecoright Limited**
Unit 2, Paddock Road
Industrial Estate
Caversham
Reading
Berkshire
RG4 5BY

Attention: **Roger Shroff**

Site: **Comet Hotel, Hatfield**

October 2018

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Client: Ecoright Limited
Unit 2, Paddock Road
Industrial Estate
Caversham
Reading
Berkshire
RG4 5BY

Date: 22nd October 2018

Originator: Roger Shroff

Order ref.: Letter ref. RS/09/10/2018

Our ref.: 58467/CH

Site: Comet Hotel, Hatfield

Samples: 1No. sample of "yellow/buff" coloured hardened potential lime mortar, delivered to KCMT by Royal Mail Parcel Post on Wednesday the 10th October 2018. The sample consisted of a single piece of hard, well compacted mortar of approximate 232g mass with a "brick frog" impression. The sample was allocated KCMT chemistry laboratory prefix 58467.

Requirements: Carry out X-ray diffraction (XRD), together with appropriate chemical analysis in order to identify the binder type, provide other relevant information on composition and indicative mix proportions.

Test method: XRD analysis

The sample was subjected to an initial visual examination using magnifications up to x20 and indicative testing with reagents and indicator solutions to assist with identifying their composition and condition.

After the initial examination, a binder rich sub-specimen was obtained from the sample. The specimen was prepared for XRD analysis by disrupting a representative sample in an impact mortar before grinding in an agate mortar and pestle and sieving the resulting material over a 63µm test sieve to remove as much of the aggregate component as possible. Care was taken to keep the crushing of aggregate particles to a minimum to avoid masking of binder components which may only be present in trace quantities.

The powdered specimen of mortar was backpacked into a proprietary sample holder for subsequent presentation in the diffractometer. The sample preparation described above was adopted in the interests of ensuring, as near as possible, the completely random orientation of the crystalline components required to give true peak intensities in the diffractograms.

The prepared samples were analysed in a Philips X-ray Diffractometer fitted with a single crystal monochromator set to run over the range 3° to 60° 2ɵ in steps of 1°2ɵ/minute using CuKα radiation.

The digital output from the diffractometer was analysed by 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°.

Chemical analysis – mortar sample

A sub-sample of the mortar was prepared with reference to clause 7.4.5 of BS 4551:2005 + A2 : 2013 and analysed for lime (CaO), soluble silica (SiO_2), acid soluble sulphate, insoluble residue and LOI using methods described by this standard.

Note : The procedure used for the lime (CaO) determinations deviates from the British Standard only in terms of the indicator used.

Date of analysis: Testing & analysis carried out during October 2018.

Results:

Comments and observations

XRD:

Mortar sample 558467/1

The mortar within the sample is well compacted and hard such that it could not be broken using persistent firm finger pressure; hammer impact was needed to break and disrupt the sample in preparation for analysis.

There was no evidence of any lime inclusions within the mortar and the mortar fabric was notably uniform throughout. No evidence of air entrainment was found.

The colour of the mortar was assessed by comparing a freshly broken surface to the Munsell Soil Colour Charts and found to be 2.5YR 7/4 “Pale Yellow ”.

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Tests carried out via application of phenolphthalein indicator to freshly broken surfaces of the sample showed the specimen to be fully carbonated. Testing with water droplets applied to fracture and cast surfaces of the mortar showed that drops were absorbed quickly and diffused throughout the thickness of the mortar suggesting a well-connected pore structure.

The aggregates within the mortar have the appearance of a natural quartz rich sand with a maximum grain size of 1.0mm, although most are finer than 0.5mm. The particles are mostly sub-angular to sub-round in shape and all exhibit what appear to be water worn surfaces.

XRD results are presented in appendix 3 in the form of a labelled diffractogram

Abbreviations used on the traces to identify peak positions are as follows:

cc = Calcite ($CaCO_3$) Calcium carbonate, carbonated lime from the binder;

va = Vaterite ($Ca(CO)_3$), another crystalline form of Calcium Carbonate, commonly associated with leached and redeposited lime from binder;

br = Brownmillerite ($Ca_2(Al,Fe)_2O_5$), Calcium Aluminium Iron Oxide, hydraulic clinker component in some forms of hydraulic lime;

gy = Gypsum ($CaSO_4$) Calcium Sulphate Hydrate, sulphate reaction product from a reaction between environmental sulphates and lime in the binder ;

qz = Quartz (SiO_2), Silica from the quartz aggregate present within the sand;

For clarification, the proportion of crystalline components found were quantified via Rietveld Refinement. Findings are as follows:

Component	58467/1 % by mass
Calcite	66.4
Vaterite	0.2
Brownmillerite (C_4AF)	1.5
Gypsum	3.2
Quartz	28.7
Total	100.0

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Conclusions:

On the basis of the XRD analyses and bearing in mind the absence of Alit and Belite within the binder, indications are that the mortar was most probably made using a processed quartz rich sand and a hydraulic lime binder, the latter being in hydrate form, possibly an early modern NHL.

The aggregates are consistent with a water transported natural sand and there are similarities with sands available from the Leighton Buzzard region of the UK.

Chemical analysis

On the basis of the XRD examination, it is assumed that mortar sample was made using a hydraulic lime.

Using the above and the basic chemical analyses carried out on the sample, the mix proportions of the mortar mortars are calculated to be of the order of:

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1 : 0.5 to 1.5 (hydraulic lime : sand)

It should be noted that the above is given as "indicative/tentative only guidance" based on the examination and analyses carried out and the stated assumptions made. It should be noted that certain factors, including impacts of in-service conditions may have a bearing on the calculated results.

General record photographs may be found in appendix 1. Detailed analytical results are included in appendix 2 and the X-ray diffractogram forms appendix 3.

Note. comments and interpretations expressed above are based on analytical results obtained from the mortar sample provided to Kiwa CMT Testing by the client and relate only to this sample.

The original XRD lab. report remains on our records.

Signed, checked and approved by

M. Barham

M. Barham
(Chief Chemist)

For and on behalf of Kiwa CMT Testing

Appendix 1

Record photographs

58467/1 "as received" sample showing brick contact surfaces

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Freshly fractured surface through the thickness of the sample. Although the mortar seems to have been made using a very fine sand, a single coarse (sandstone) aggregate particle (5.3mm size) was noted within the sample

Appendix 2

Detailed Analytical Results

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Analytical Data – chemical analysis

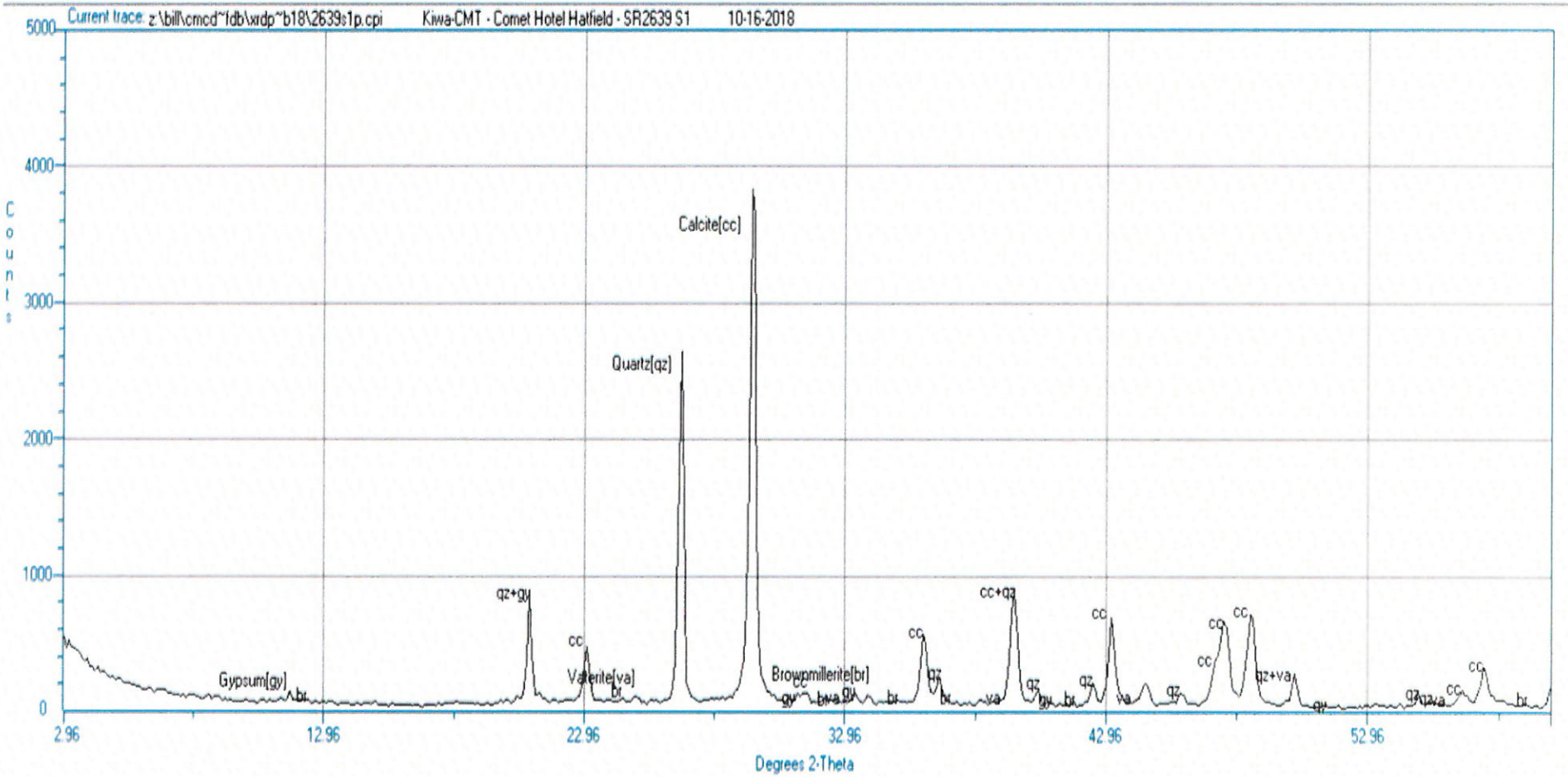
Sample reference:		**58467/1**
Lime (CaO)	(%)	18.15
Soluble silica (SiO)	(%)	4.00
Insoluble residue	(%)	57.26
Loss on ignition	(%)	16.23
Total sulphate	(%)	0.48

Assumptions:

1. The binder is a early modern hydraulic lime
2. The lime determined by the analysis is derived only from the lime binder
3. The hydraulic lime was used in hydrate form
4. The densities of hydraulic lime and the sand/fine aggregate are 675 and 1675 kg/m^3 respectively

Appendix 3

X-ray diffractogram



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