CONSTRUCTION MATERIALS CONSULTANTS, INC.

Laboratory Investigation of A Masonry Mortar

PROJECT NAME
PROJECT ADDRESS

Prepared for:
CLIENT

DATE
CMC NO

Construction Materials Consultants, Inc.

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DATE

Client Contact
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RE: PROJECT NAME

Dear CLIENT:

Construction Materials Consultants, Inc. (CMC) is pleased to provide the enclosed comprehensive report on 'Laboratory Investigation of A Masonry Mortar," for the Project: PROJECT NAME.

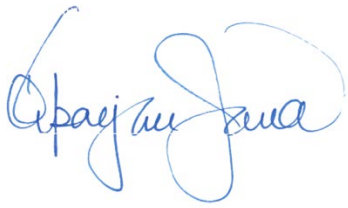
Results, opinions, and conclusions presented herein are based on the information and sample provided at the time of this investigation. We reserve the right to modify the report as additional information becomes available. Neither CMC nor its employees assume any obligation or liability for damages, including, but not limited to, consequential damages arising out of, or in conjunction with the use, or inability to use this resulting information.

Sample will be disposed after submission of the report. All reports are the confidential property of clients, and information contained herein may not be published or reproduced pending our written approval.

Please feel free to contact us with any additional questions. We look forward to providing our service again for your future projects.

Sincerely Yours,

CONSTRUCTION MATERIALS CONSULTANTS, INC.



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President, Petrographer

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Abbreviations:
PPL: Observations in plane polarized light mode in a petrographic microscope;
XPL: Observations in crossed polarized light mode in a petrographic microscope; most thin-section photomicrographs are taken in both PPL and corresponding XPL modes in a Nikon Eclipse 600 POL petrographic microscope.
FW: Field width of a photomicrograph measured in millimeters; the bars at the base of most thin-section photomicrographs show the scale in microns, taken by using a Jenoptik Progres GRYPHAX camera;
XRD: X-ray diffraction.

EXECUTIVE SUMMARY

The present study involves laboratory examination of a masonry mortar retrieved from PROJECT NAME, located in CITY, STATE. The mortar was examined *a la* ASTM C 1324 to determine the composition and ingredients, mix proportion, and, subsequently, suggest an appropriate tuck-pointing mortar for renovation.

The ASTM C 1324 procedures followed consisted of detailed petrographic examinations including optical microscopy, scanning electron microscopy and energy-dispersive X-ray microanalyses (SEM-EDS), X-ray diffraction (XRD), X-ray fluorescence (XRF), and various chemical analyses, including determination of acid-insoluble residue content (by hydrochloric acid digestion), bulk oxide composition of mortar (by XRF), soluble silica content to determine the proportions of cement component, if any, in the binder, free and combined water contents and degree of carbonation (from losses on ignitions at 110°C, 550°C, and 950°C, respectively). Petrography and chemistry have not only provided detailed composition, mineralogy, and microstructure of the mortar but also compositions, mineralogies, and proportions of various ingredients used in formulation of the mortar. Additionally, a portion of the mortar was digested in hydrochloric acid to extract its sand for determination of size, shape, color(s), and grain-size distribution of sand.

Based on these studies, the mortar is determined to be a cement-lime type, made using Portland cement, lime, and natural siliceous sand. Sand contains major amounts of quartz and quartzite, subordinate amounts of feldspar, granite, and ferruginous particles. No calcareous component is detected in the sand. Sand used was, clean, well graded (compares within the upper and lower limits of size distribution of masonry sand specifications in ASTM C 144 for natural sand), well-distributed, and present in sound condition with no evidence of any deleterious alkali-aggregate reaction of sand.

The binder contains a Portland cement and hydrated lime (or lime putty) where the former is present as residual cement particles in a dominantly carbonated matrix of lime and hydration products of cement. The overall matrix is dominated by slaked lime of possible high-calcium origin as indicated by porous, fine-grained carbonated cryptocrystalline to microcrystalline calcite masses of carbonated lime matrix, nominal magnesia content of bulk mortar, and negligible magnesia content of paste. Residual Portland cement particles show well hydration with a few remnants and relicts of cement particles (mostly dark interstitial ferrite matrix of cement particles).

Based on detailed laboratory studies and calculated mix proportions, the mortar is, therefore, determined to be ***a cement-lime mortar having 1-part Portland cement to 7-part lime to 18.5-part sand (2.3 times sand of the sum of separate volumes of cement and lime).***

A possible tuck-pointing mortar could be a modern ASTM C 270 Type N cement-lime or a masonry cement mortar made using Portland cement in conformance to ASTM C 150 and hydrated lime in conformance to ASTM C 207 for a cement-lime mortar, or, a Type N masonry cement as shown below in conformance to ASTM C 91, and a masonry sand in conformance to ASTM C 144.

Existing Mortar	Estimated Mix Proportions	Suggested Tuckpointing Mortar	Suggested Mix Proportions (to be verified by trial and error over a small test area)	Suggested Sand
Cement-lime mortar	1 part cement to 7-part lime to 18.5-part sand (2.3 times sand of the sum of separate volumes of cement and lime) – under-sanded	ASTM C 270 Type N cement-lime or masonry cement mortar	1-part Portland cement to 1 to 1 1/2-part lime to 3 times the sum of separate volumes of cement and lime	Masonry sand in conformance to ASTM C 144

The final choice of binder and sand ingredients would depend on the match in appearance, compositions, and properties with the existing mortar. The chosen mortar should bond well to the masonry unit as well as to the existing jointing mortar and preferably be softer than the present mortar without introducing any undue stress from any compositional or proportional mismatch to the existing mortar or mismatch to the masonry units.

INTRODUCTION

Reported herein are the results of detailed laboratory studies of a masonry mortar sample reportedly collected from PROJECT, located in CITY, STATE. CLIENT CONTACT of CLIENT, provided the sample.

The purposes of the laboratory investigation are to determine:

a) The overall composition of the mortar, including the type, size, and composition of sand used in the mortar;
b) The type of the binder(s) used;
c) Evidence of any physical and/or chemical deterioration of the mortar;
d) Detailed chemical and mineralogical compositions of the sand and the binder(s) used in the mortar;
e) Extraction of sand from the mortar and determination of grain-size distribution of sand used in the mortar;
f) Based on the determined composition, an estimation of volumetric proportions of various binder and sand components used in the mortar mix; and,
g) Based on the determined compositions and mix proportion, suggestions for a suitable tuck-pointing mortar that can be used during renovation at the location of the extracted mortar.

PROJECT NAME

Figure 1 shows photographs of the subject building. No additional background information about construction of the building was provided.

MORTAR SAMPLE

Table 1 and Figures 2 and 3 provide weight, dimensions, appearance, and integrity of the mortar sample, as received.

Sample ID & Location	Weight (grams)	Largest Piece Dimensions (mm)	Appearance	Integrity
Existing Mortar	32.7	75 mm × 25 mm × 10 mm	Light beige, moderately soft to moderately hard, dry, uniform appearance of pieces (Figures 2 and 3)	Three unequal pieces (Figures 2 and 3)

Table 1: Weight, dimensions, color, hardness/softness, and integrity of the mortar sample as received.

Figure 1: Photograph of PROJECT building located at ADDRESS, CITY, STATE – the subject building from where mortar sample for the present study was reportedly retrieved.

Figure 2: Shown are the few small fragments of mortar as received in a plastic Ziploc bag. All pieces were used for laboratory testing.

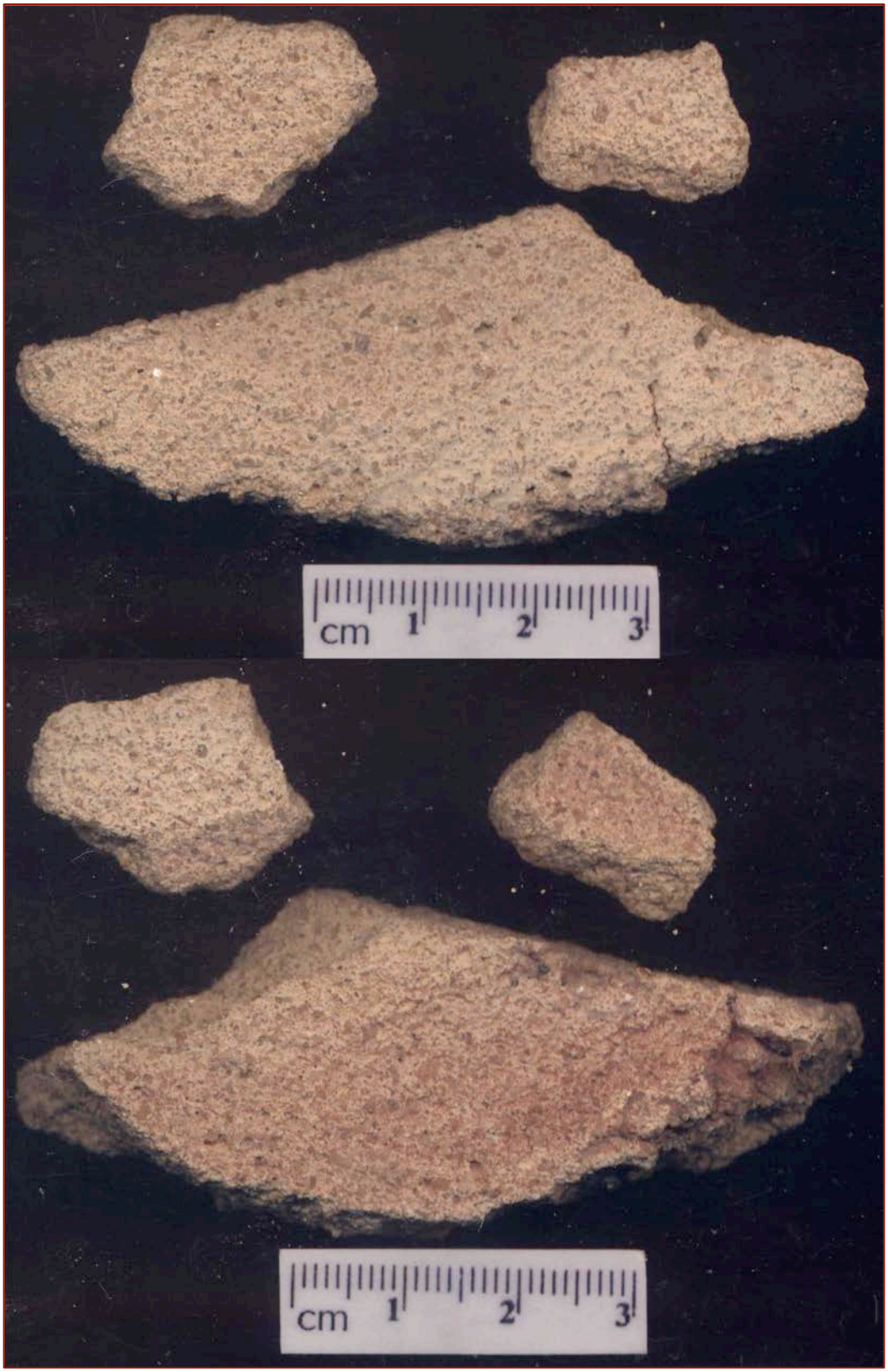


Figure 3: Overall appearance of individual pieces of mortar, as received (scanned by a flatbed scanner with a black background).

Figure 4 shows blue dye-mixed epoxy-impregnated thin section of a representative piece of the mortar and the residue of thin section. The purpose of using a blue dye-mixed epoxy is to highlight all pore and void spaces as well as microcracks in the mortar. Both the thin section as well as the residue shows deep and complete penetration of dyed epoxy inside the mortar indicating an inherently porous nature of the mortar that was receptive of the dyed epoxy.

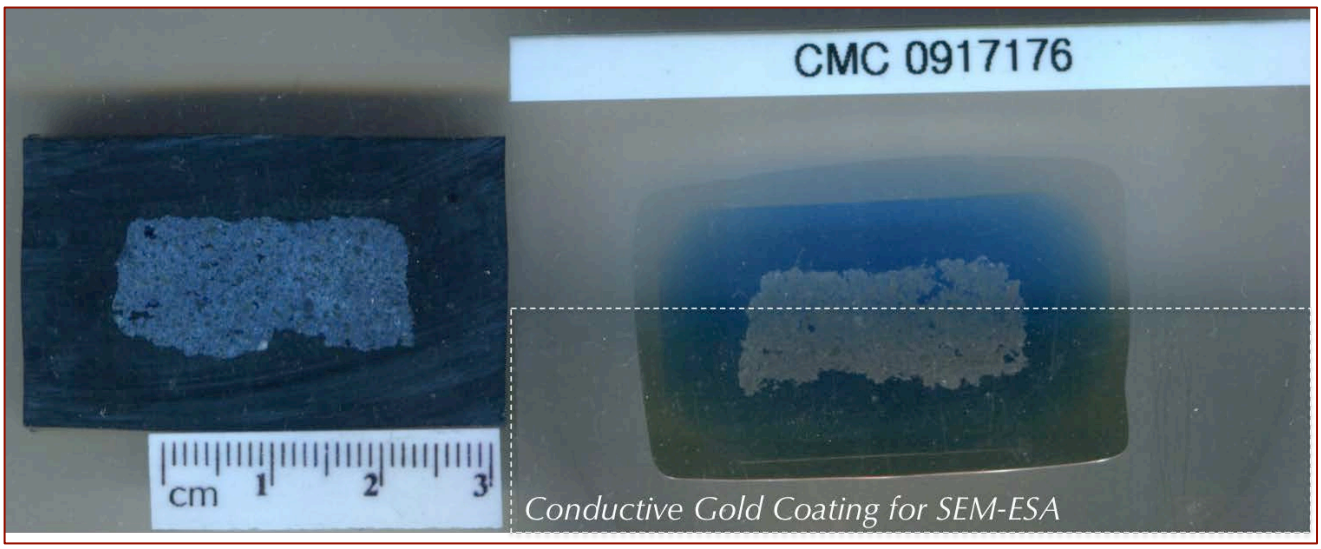


Figure 4: Photographs of blue dye-mixed epoxy-impregnated thin section of a piece of the mortar on right, and the residue left after thin section preparation on left.

METHODOLOGIES

Appendix A1 provides detailed methodologies for laboratory testing of masonry mortar that are followed by the laboratories of CMC. The mortar sample was tested by following the methods of ASTM C 1324 "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar," which includes detailed petrographic examinations, followed by chemical analyses, along with various analytical methods to test masonry mortars as described in various literatures, e.g., Erlin and Hime 1987, Doebley and Spitzer 1996, Chiari et al. 1996, Middendorf et al. 2005 a and b, Elsen 2006, Bartos et al. 2000, Valek et al. 2012, Jana 2005, 2006, and Goins 2001 and 2004.

PETROGRAPHIC EXAMINATIONS

POROSITY, SAND SIZE, & GRADING FROM IMAGE ANALYSES OF THIN SECTION PHOTOMICROGRAPHS

Figure 5 shows photomicrographs of blue dye-mixed epoxy-impregnated thin section of mortar, which were used for image analyses in Adobe Photoshop to highlight the sand particles and voids separately, and, then calculate proportions of sand and void volumes by Image J. Figures 5 through 7 show:

a) Photomicrographs of blue dye-mixed epoxy-impregnated thin section of mortar in Figure 5 from a mosaic of 6 photomicrographs taken over 6 different areas in thin section from a transmitted-light stereo-zoom microscope (field width of each photomicrograph is 4.4 mm);
b) Corresponding black-and-white binary image in Figure 6 of thin section photomicrographs in Figure 5 to highlight the open spaces in black against everything else in white to calculate estimated pore volume; and,
c) Corresponding black-and-white binary images in Figure 7 of thin section photomicrographs in Figure 5 to highlight the sand particles in black against everything else in white, thus evaluate the size, shape, angularity, sphericity, gradation, and distribution of sand particles, and calculate sand volume.
d) The black and white binary images highlighting the sand or pore spaces were derived from Adobe Photoshop, and, volumetric proportions of sand and pore spaces were calculated from Image J, an open-source image analysis software developed by the National Institute of Health (www.imagej.nih.gov).

Sample ID	Pores, Voids, Cracks, Separations				Sand		
	Estimated (%) Volumes	Proportion of Irregular Voids	Shrinkage Microcracks	Total Volume is Contributed From:	Nominal Maximum Size (mm)	Shape, Angularity, Grading, Distribution	Estimate Volumes in Images (%)
Mortar	11.6 (Figure 6)	Few Interstitial voids between sand	Very few elongated	Interstitial Voids	2.0	Equidimensional, Subangular to Well-rounded, Well-graded, Well-distributed	40.6 (Figure 7)

Table 2: Estimated porosity, types of pore spaces, nominal maximum size (determined from photomicrographs) of sand aggregate, and angularity and grading of sand in the thin section photomicrographs of mortar determined from image analysis by Image J.

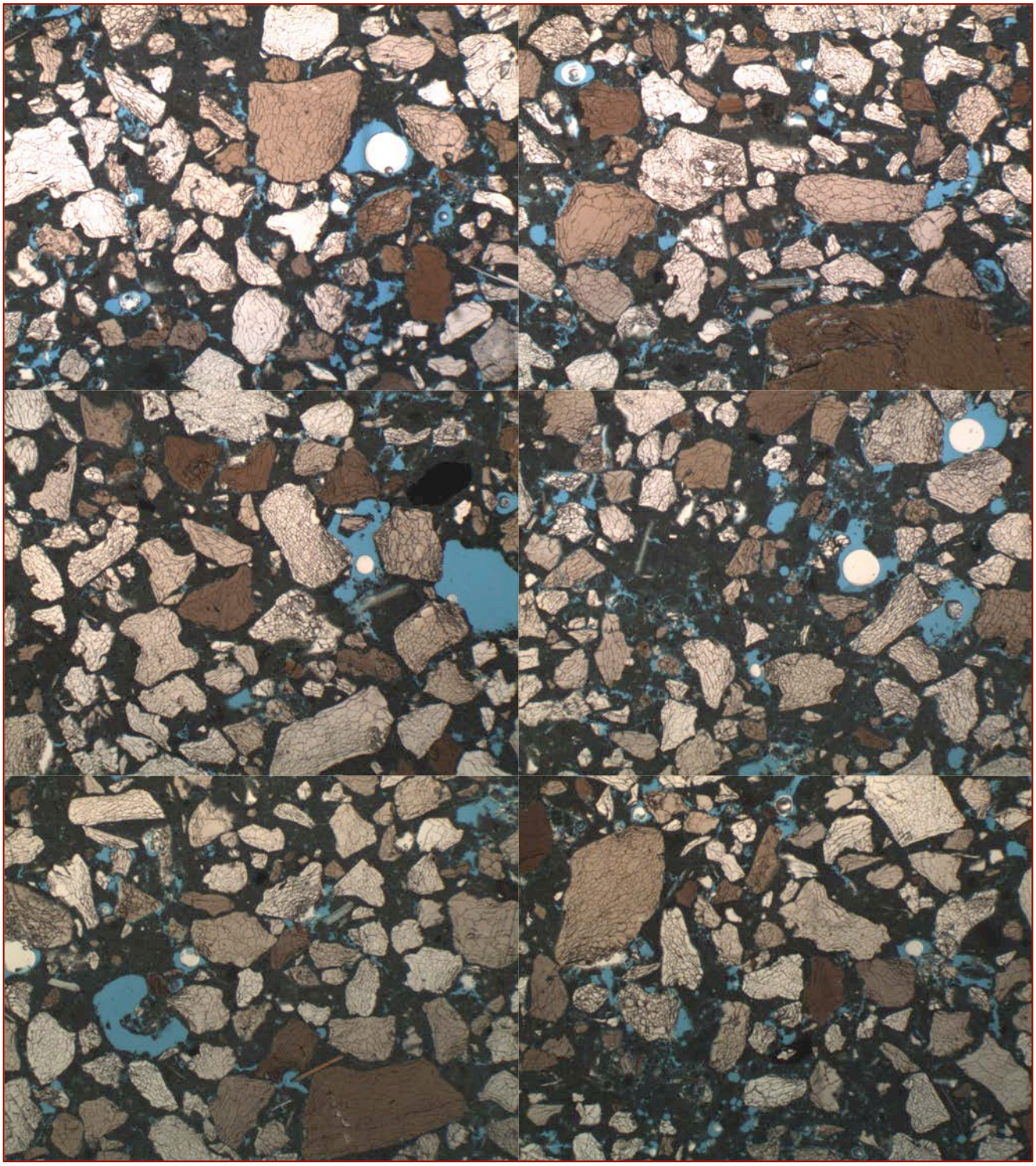


Figure 5: Mosaic of 6 photomicrographs of blue dye-mixed epoxy-impregnated thin section of mortar shown in Figure 4. Field width of each photo is 4.4 mm. Total width is 8.8 mm. This mosaic of six photomicrographs was used to obtain black and white color image in Figure 6 highlighting pore spaces from blue here to black in Figure 6.

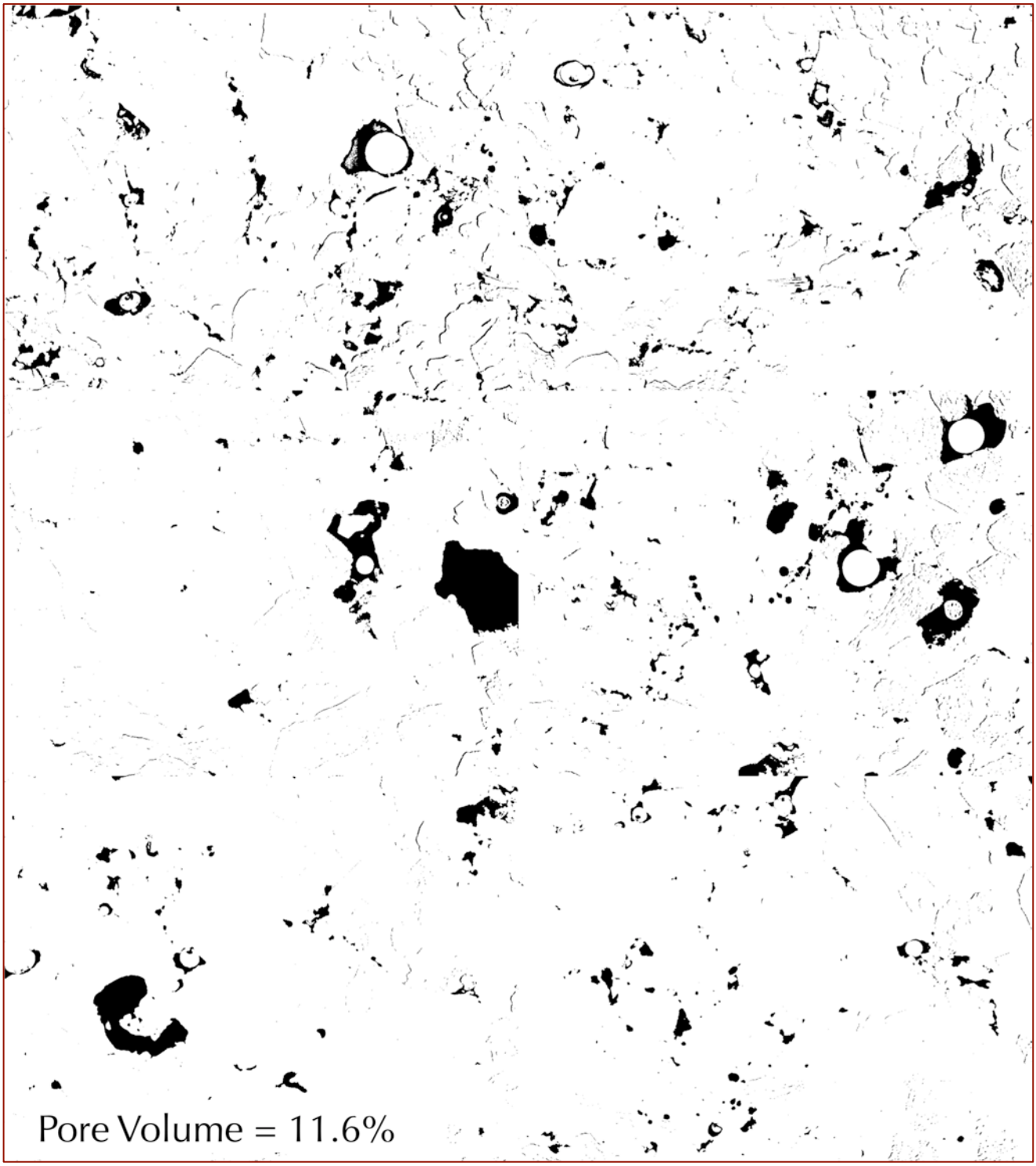


Figure 6: Corresponding black and white images of thin section photomicrographs of mortar shown in Figure 5 that only highlight interstitial void spaces in the mortar in black. Image analysis of entire mosaic of six photos determined 11.6 percent voids. Total width of this combined image is 8.8 mm.

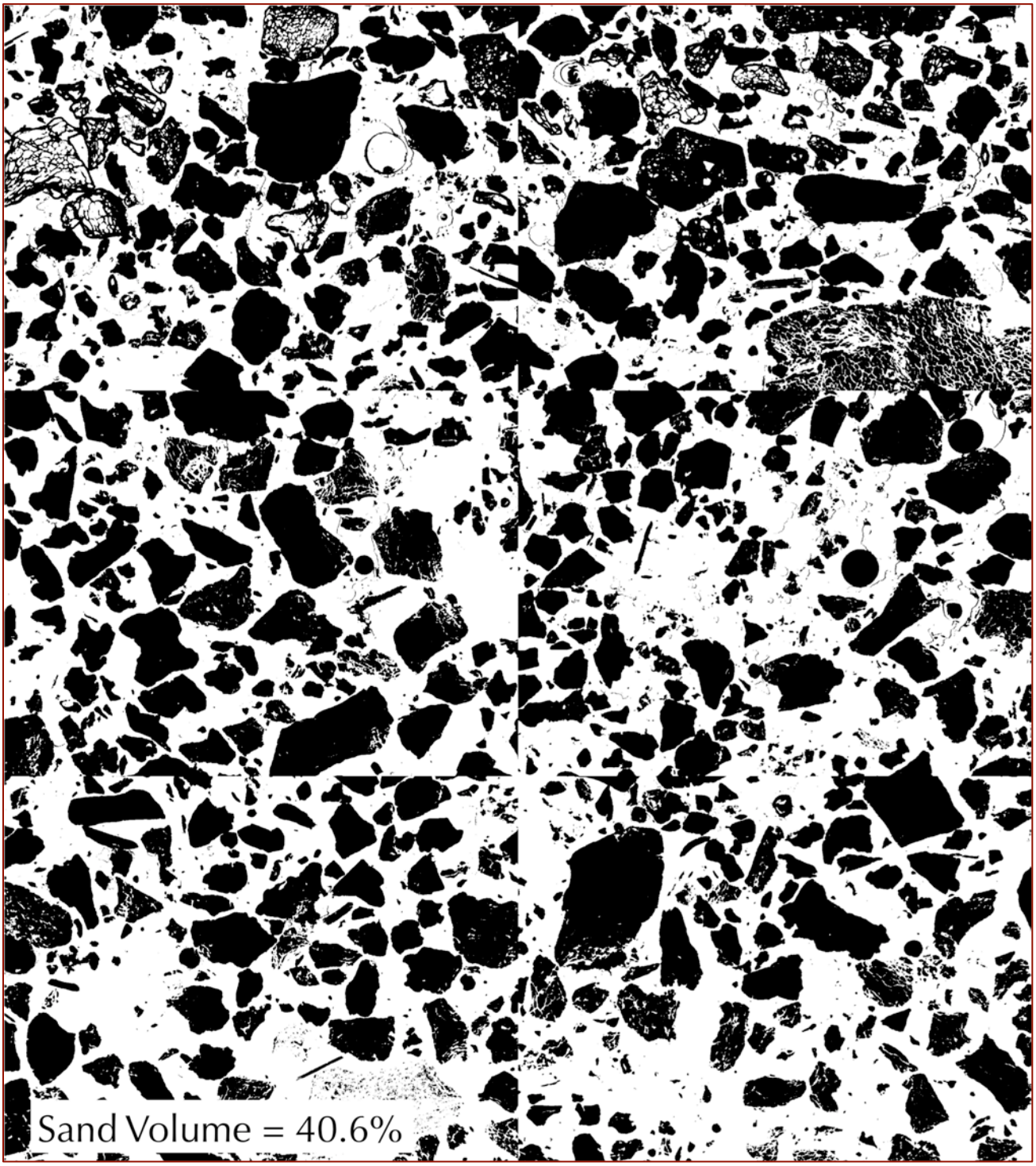


Figure 7: Corresponding black and white images of thin section photomicrographs of mortar shown in Figure 5 that only highlight sand particles in the mortar in black. Image analysis of entire mosaic of six photos determined 40.6 percent sand volume. Total width of this combined image is 8.8 mm.

Results of volumes of sand and pore spaces obtained from such practices are only considered as rough estimates, since adequate numbers of such photomicrographs across the entire examined surface area of a mortar sample are needed for better accuracy of these parameters. Care is needed to select pore spaces and void areas that are

inadequately filled with blue dyed epoxy, or of aggregate-paste gaps during the process of highlighting not only just the easily detectable pore spaces and voids, but also any not so distinct voids. Selection of all voids, open spaces, microcracks, etc. are done in Adobe Photoshop in the final processed blue-dyed photomicrographs, which are then transferred to Image J to calculate the volumes of pore spaces not only from entrained and entrapped air-voids, but also from cracks, microcracks, and aggregate-paste separations.

MORTAR SAND – TYPES, COMPOSITIONS, MINERALOGY, SIZE, GRADING

Optical Microscopy of Sand

Following Figures 5 and 7, Figure 8 shows the size, shape, angularity, distribution, and grading of sand particles in the mortar. Sand is of siliceous composition consisting of major amounts of quartz and quartzite, subordinate amounts of feldspar, granite, and ferruginous particles. No calcareous component is detected in the sand. Sand used was, clean, well-graded, well-distributed, and present in sound condition with no evidence of any deleterious alkali-aggregate reaction of sand. As seen in Figures 5, 7, and 8, sand has a nominal maximum size of 2 mm, where particles are angular to subangular, well-graded, and well-distributed. Figure 8 shows reflected-light photomicrographs of thin section of mortar showing size, shape, angularity, and size distribution of sand particles. Photos in Figures 13 and 14 show mineralogy, texture, rock and mineral-types of individual sand grains in thin section under a petrographic microscope. Sand particles are characteristic of crushed sand (mainly due to angularity of sand particles), which is dense, massive and crystalline-textured.

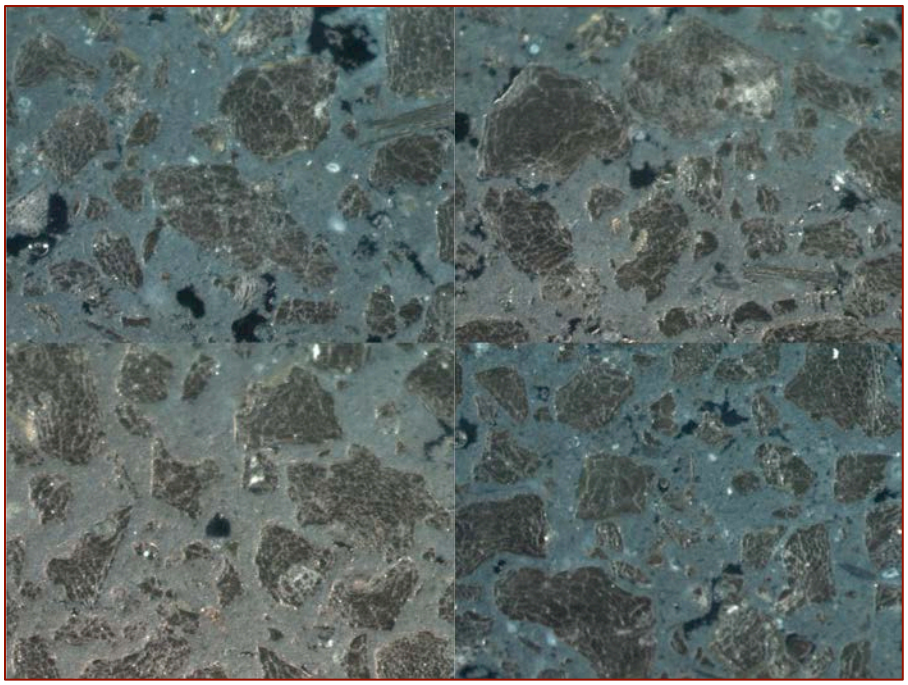


Figure 8: Reflected-light photomicrographs of blue dye-mixed epoxy-impregnated thin section of mortar showing size, shape, angularity, gradation, and distribution of sand particles.

Aggregate Gradation & Mineralogy After Extraction From Mortar & Sieve Analysis

A representative fragment of mortar was selected for digestion in (1+3) dilute hydrochloric acid to dissolve away all binder fractions and extract, wash, and dry the acid-insoluble component of aggregate which, based on optical microscopy, is determined to be the entire mass of sand since sand is siliceous in composition and does not contain any acid-soluble, e.g., carbonate components.

The bulk mortar sample (one representative piece) was first gently broke down into small pieces in a porcelain mortar and pestle making sure not to reduce inherent grain-size of sand during this size reduction process of bulk mortar. Subsequent smaller pieces were then placed in 250-ml glass beakers with a magnetic stirring rod over a stirrer and stirred for a period of 24 hours to assure complete digestion of binder fractions and settlement of sand only at the bottom of beakers. Sand particles thus extracted were then filtered out, washed in distilled water, oven dried and prepared for sieve analysis in a small automatic (Gilson) sieve shaker with 2-in. (50 mm) diameter US Sieves Nos. 4, 8, 16, 30, 50, 100, 200, and pan as shown in Figure 11.

Figure 9: Gilson mini sieve shaker used for sieve analysis of sand extracted from mortar.

Sand, thus extracted by acid digestion of intact mortar (without any mechanical pulverization or fracturing of mortar to obtain the original sand used), and sieved through various size fractions to examine conformance to the standard specification of masonry sand *a la* ASTM C 144 are then weighed in each sieve and fraction of each size retained on various sieves are then calculated and provided in the Table in Figure 12.

Figures 10 and 11 show the sand particles thus extracted from the mortar and size, shape, angularity, and color of sand retained on various sieves.

Size distribution of sand in the mortar is plotted in Figure 12, which shows that for most size fraction, sand conforms within the upper and lower limits of ASTM C 144 size gradation for the natural sand. The bottom 'percent retained' histogram shows normal size distribution of sand without any enrichment of coarse or fine fractions. Therefore, sand is judged to be overall in conformance to an ASTM C 144 masonry sand.

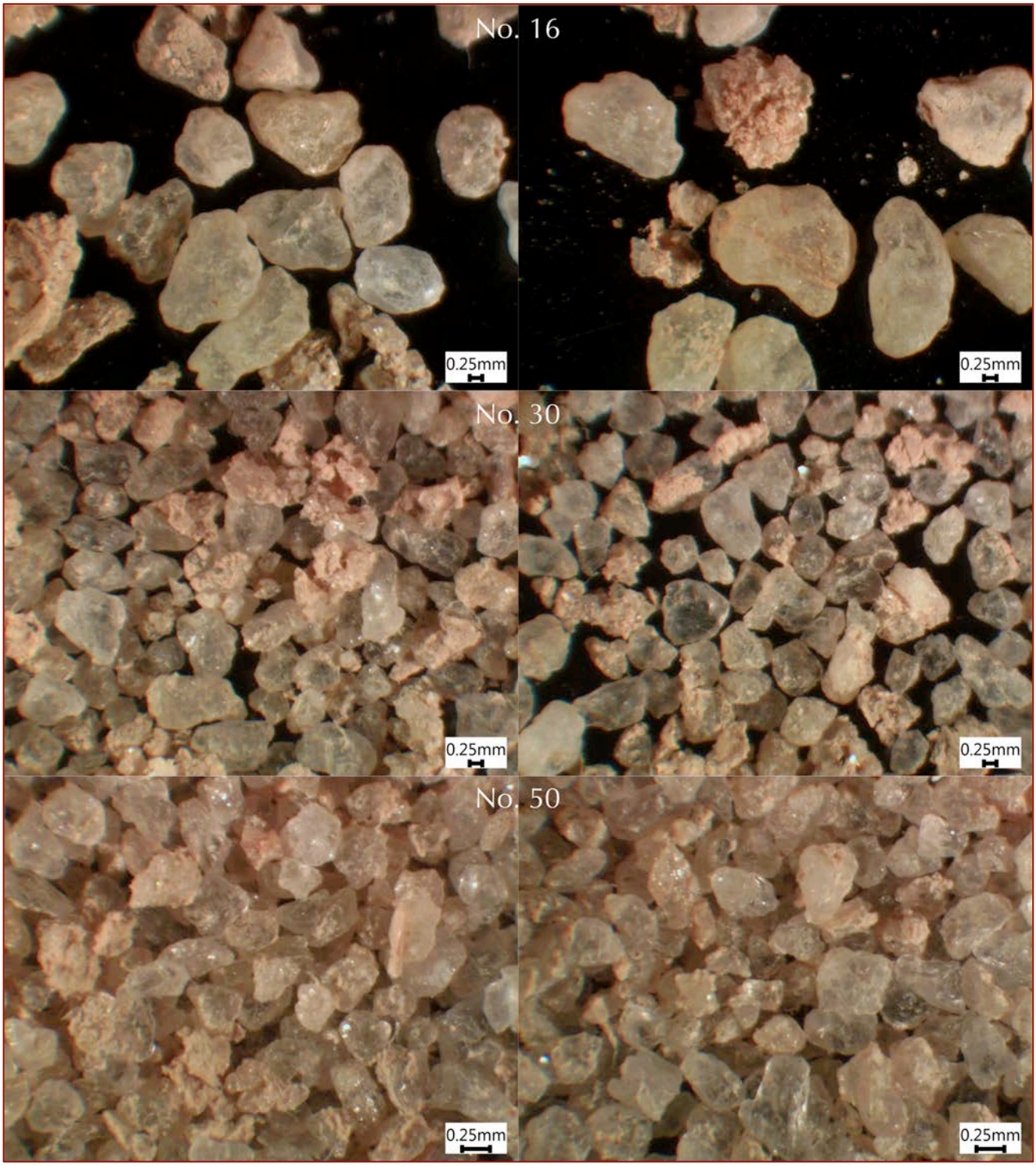


Figure 10: Size, shape, color, angularity, and size distribution of sand particles extracted after digestion of mortar. Each photomicrograph (taken with a Stereozoom microscope) shows particles retained on each sieve (sieve sizes are marked). Results of sieve analyses are shown in the Table and particle size distribution graph in Figure 12.

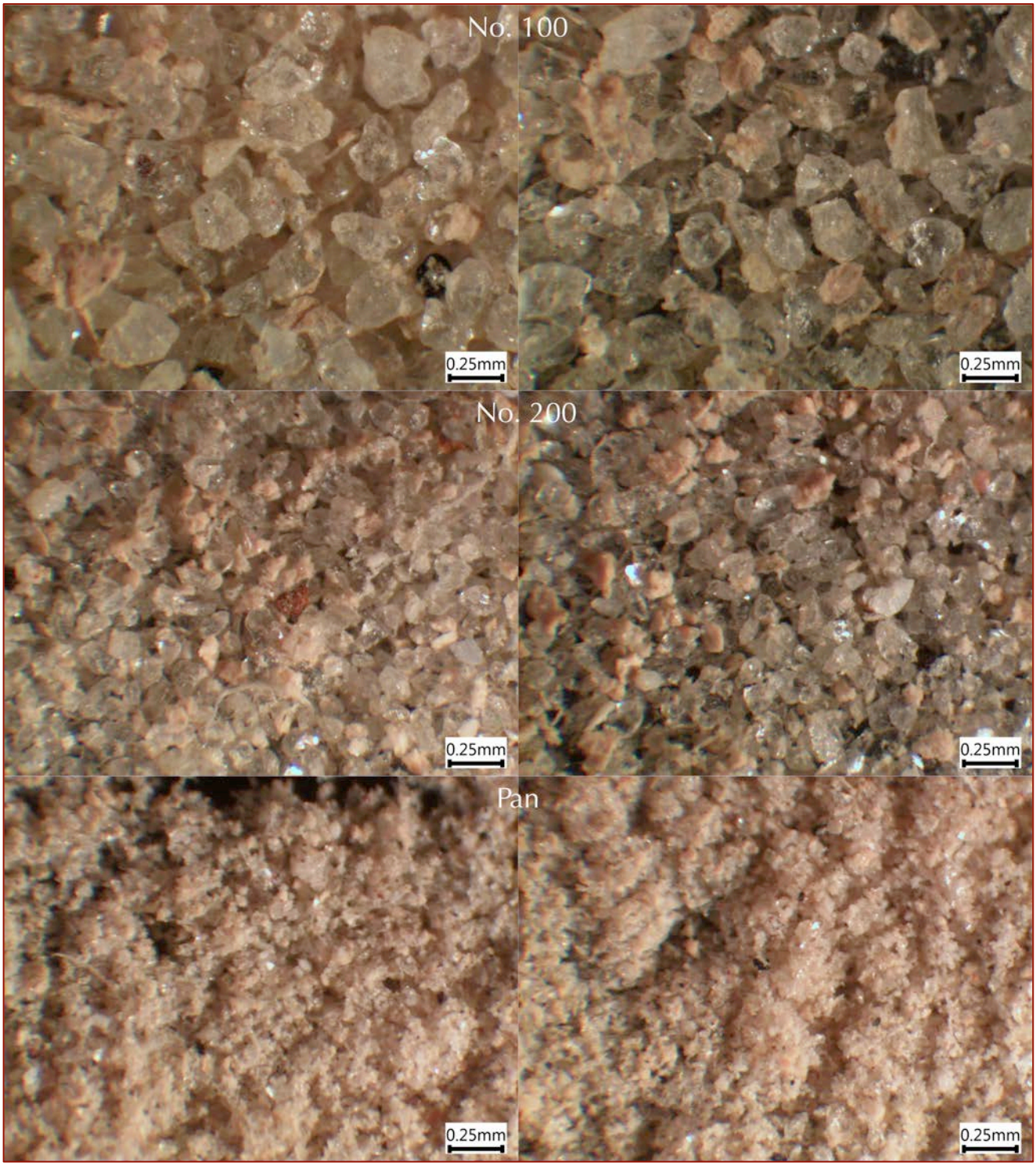
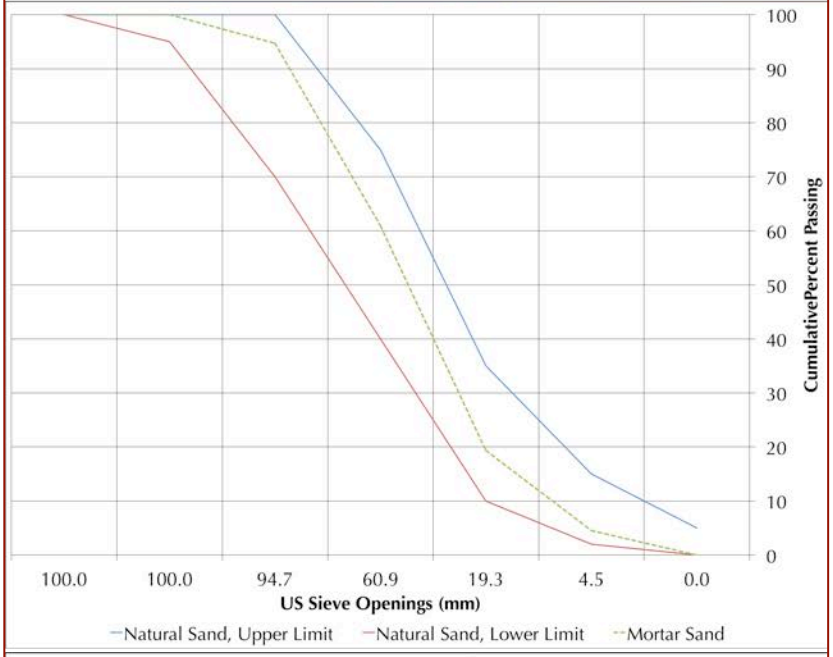


Figure 11: Size, shape, color, angularity, and size distribution of sand particles extracted after digestion of mortar. Each photomicrograph (taken with a Stereozoom microscope) shows particles retained on each sieve (sieve sizes are marked). Results of sieve analyses are shown in the Table and particle size distribution graph in Figure 12.

Sand Sieve Sizes		Percent Passing for Natural & Manufactured Sand				Weight Retained, g	Percent Retained	Cumulative Percent Retained	Percent Passing
US Sieve No.	mm	Natural Sand Lower Limit	Natural Sand Upper Limit	Manufactured Sand Lower Limit	Manufactured Sand Upper Limit	Mortar	Mortar	Mortar	Mortar
4	4.75	100	100	100	100	0.000	0.0	0.0	100.0
8	2.36	95	100	95	100	0.000	0.0	0.0	100.0
16	1.18	70	100	70	100	0.186	5.3	5.3	94.7
30	0.6	40	75	40	75	1.190	33.8	39.1	60.9
50	0.3	10	35	20	40	1.463	41.6	80.7	19.3
100	0.15	2	15	10	25	0.522	14.8	95.5	4.5
200	0.075	0	5	0	10	0.159	4.5	100.0	0.0



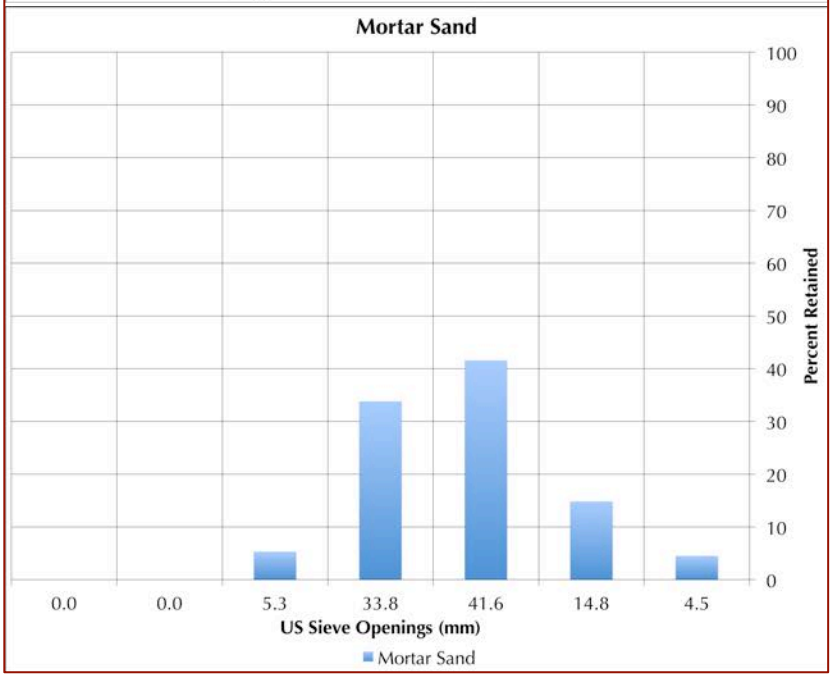


Figure 12: Size distribution Table (top) and a graph showing (cumulative percent passing versus particle size in mm) of sand extracted from the mortar. The bold red and blue lines in the graph indicate upper and lower limits of size distribution for natural sand according to ASTM C 144, whereas the dashed green line shows the size distribution for sand extracted from the present mortar. Bottom histogram shows percent retained on each sieve. Sand was extracted after 24 hours of digestion of bulk mortar in 1+3 hydrochloric acid without any major grinding to reduce breakdown of sand during processing.

MORTAR PASTE – BINDER TYPE, COMPOSITION, AND MICROSTRUCTURE

Cement-Lime Matrix

Figures 13 and 14 show the typical overall carbonated microstructure of paste in between sand particles, which show compositions and microstructures similar to many cement-lime mortars.

Fine-grained, Porous, Carbonated Matrix

The overall fine-grained, porous and carbonated nature of paste is evident from optical microscopy, severe absorption of blue dye-mixed epoxy, and typical interference color of carbonated paste at crossed polarized light mode in a petrographic microscope. This overwhelming carbonated paste indicates use of dominant lime component over cement in the binder.

Shrinkage Microcracks

The characteristic tale-tell microstructure of many historic lime mortars is short, discontinuous shrinkage microcracks, which are also present in this mortar. Figures 13 (top row) and 14 (bottom row) show these microcracks especially in plane-polarized light mode in a petrographic microscope.

Patchy-Textured Intermixed Lime and 'Cement'-Rich Paste Areas

The mortar is characterized by local patchy microstructure of the matrix consisting of cryptocrystalline to microcrystalline masses of carbonated lime (carbonated calcium hydroxide) as well as denser matrix especially around residual cement particles that is populated by hydration products and residual particles of Portland cement. Such patchy appearance of paste consisting of porous lime-rich (blue-toned) and denser cement hydration rich areas (beige-toned) are hallmark microstructural features of a cement-lime mortar.

Residual Portland Cement Particles

The boxed areas in Figure 14 show many residual Portland cement particles consisting of subhedral alite and spherical belite particles and interstitial dark brown ferrite phases in plane polarized light mode and dark near-isotropic mass in crossed polarized light mode. Paste immediately around such residual cement particles are denser due to *in situ* hydration of cement around residual cement particles than the paste elsewhere.

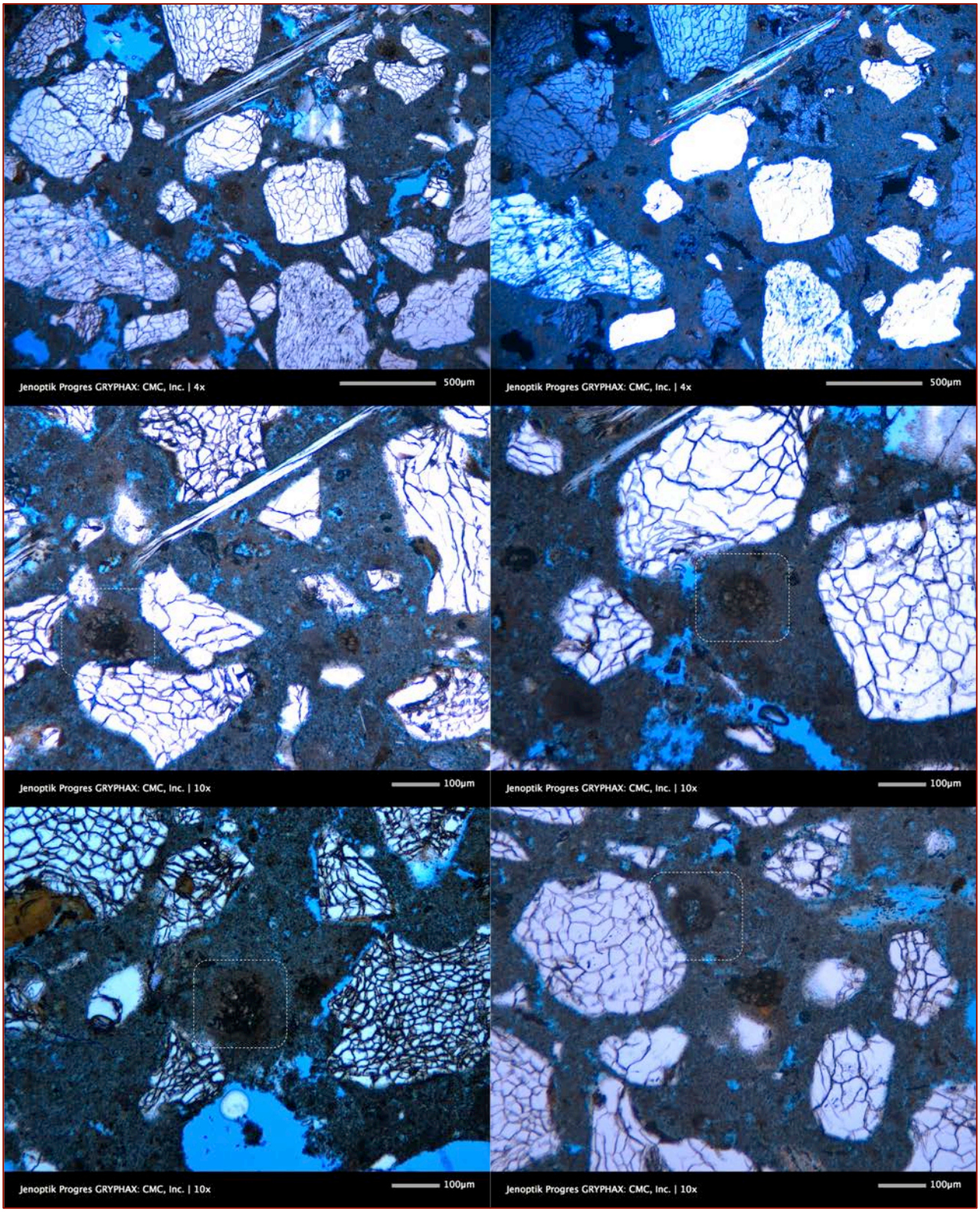


Figure 13: Photomicrographs of thin section of mortar showing overall fine-grained, porous and carbonated nature of the lime-rich matrix, distribution of siliceous (quartz) sand particles, absence of calcareous sand, and many residual Portland cement particles many of which are boxed. Left column photos were taken in PPL and right column photos in corresponding XPL modes.

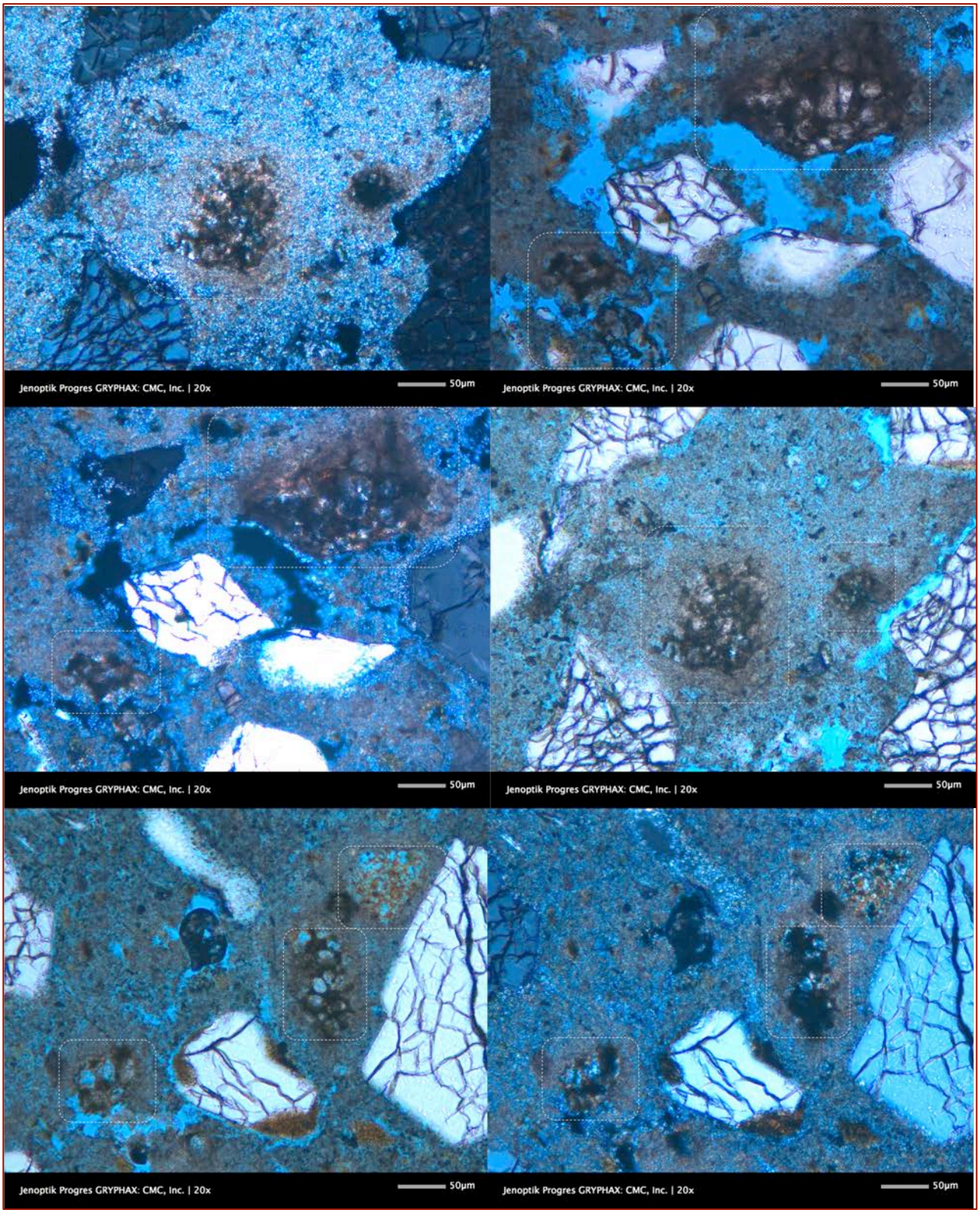


Figure 14: Photomicrographs of thin section of mortar showing overall fine-grained, porous and carbonated nature of the lime-rich matrix, distribution of siliceous (quartz) sand particles, absence of calcareous sand, and many residual Portland cement particles many of which are boxed. Left column photos were taken in PPL and right column photos in corresponding XPL modes.

Binder Compositions From SEM-EDS

Figure 15 shows backscatter electron (BSE) image and energy-dispersive X-ray elemental analyses of various areas of paste, to get a good representation of composition of the binder. Paste compositions thus determined are preferably free of any interference from minerals i.e. to obtain the true compositions of the paste. Point-mode analyses were done to get meaningful analyses of various areas of paste and reduce possible interference from fine sand particles.

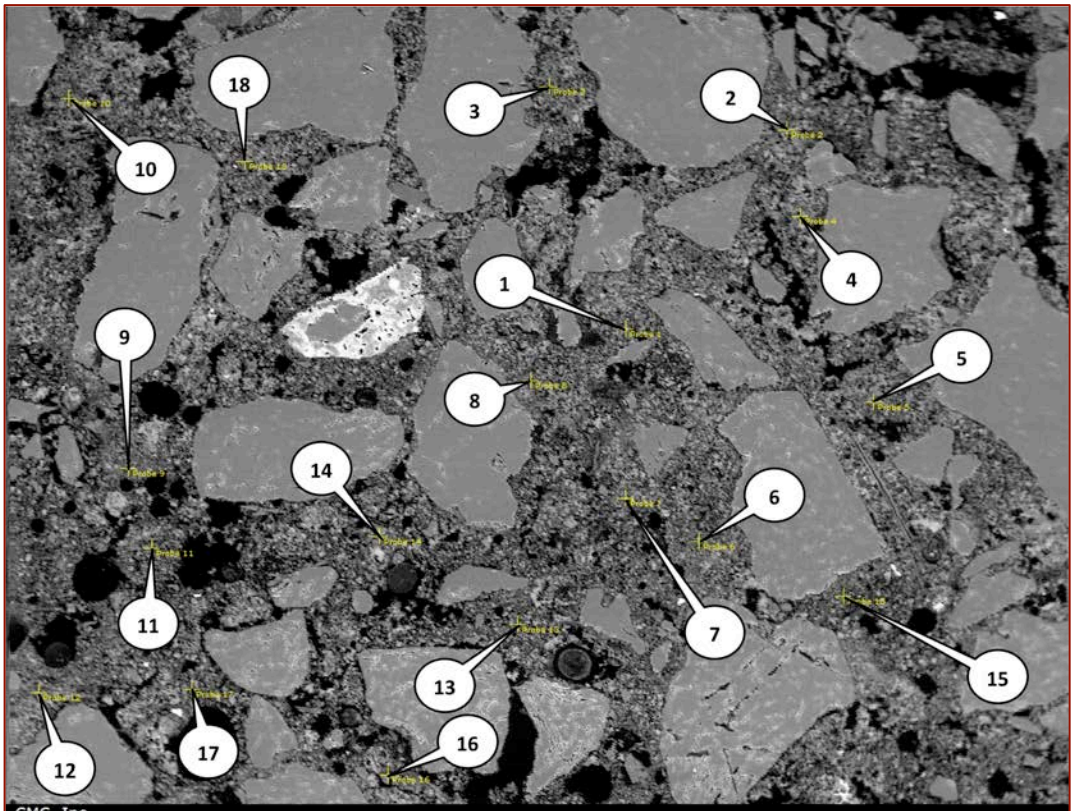
Last column in Figure 15 shows the cementation index after Eckel (1922, CI = [(2.8* SiO_2)+(1.1*Al_2O_3)+(0.7*Fe_2O_3)]/[(CaO)+(1.4*MgO)]), which measures relative hydraulicity of paste e.g., non-hydraulic lime pastes have very low CI (< 1) compared to Portland cement pastes (CI is >1).

SEM-EDS results are consistent with the optical microscopical observations of cement-lime binder composition of the mortar.

The very low to negligible magnesia contents of paste indicate lack of dolomitic lime and use of high-calcium lime binder. In the backscatter electron image, paste shows a more or less monotonous microstructure in between the sand particles that is often marred with a few residual Portland cement particles.

Compositional analyses of paste *per se* show lime-rich areas having very high calcium oxide and low silica and cement-rich areas rich in silica from hydration products of Portland cement. Paste thus shows predominant intermixed lime and silica-rich areas that are a testament of a cement-lime composition of binders in the mortar, which is also consistent with optical microscopical observations that found many residual cement particles and an overall carbonated microstructure of lime-cement matrix.

Compositional variations of paste are plotted in Figure 16 for variations in silica and calcium oxide compositions of paste against cementation index (CI). Paste shows a continuous linear variation in plots of silica versus CI or CaO versus CI – which is the main characteristic compositional feature of a paste made using cement-lime binders in a cement-lime mortar, but median CI around 0.50, which is consistent with a lime-rich proportion relative to cement in the binder, e.g., similar to an ASTM C 270 Type N cement-lime mortar.



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	82.04	11.65	3.39	0.00	0.00	0.00	0.55	0.13	2.25	100.01	Paste	0.46
2	82.05	7.82	7.74	0.00	0.00	0.00	0.23	0.00	2.16	100.00	Paste	0.39
3	81.13	9.75	7.09	0.00	0.02	0.00	0.00	0.10	1.91	100.00	Paste	0.45
4	79.87	11.33	5.53	0.03	0.08	0.00	0.49	0.26	2.42	100.01	Paste	0.49
5	79.52	13.86	3.86	0.00	0.00	0.00	0.38	0.73	1.64	99.99	Paste	0.56
6	77.02	11.17	7.87	0.00	0.07	0.00	0.44	0.52	2.91	100.00	Paste	0.54
7	78.29	12.97	3.58	0.27	0.20	0.00	0.61	1.07	3.02	100.01	Paste	0.54
8	46.39	25.11	15.48	0.00	0.00	0.00	0.89	1.66	10.46	99.99	Grain	2.04
9	82.09	10.96	4.36	0.00	0.03	0.00	0.91	0.00	1.65	100.00	Paste	0.45
10	80.12	11.62	5.03	0.00	0.00	0.00	0.47	0.37	2.38	99.99	Paste	0.50
11	75.72	11.47	7.52	0.04	0.22	0.00	1.04	0.61	3.37	99.99	Paste	0.56
12	81.93	11.60	3.77	0.00	0.00	0.00	0.52	0.00	2.18	100.00	Paste	0.47
13	80.77	12.05	5.08	0.00	0.00	0.00	0.45	0.09	1.56	100.00	Paste	0.50
14	66.35	23.02	7.61	0.00	0.00	0.00	0.30	0.00	2.72	100.00	Grain	1.13
15	76.71	14.04	5.24	0.01	0.00	0.00	0.83	0.00	3.16	99.99	Paste	0.62
16	80.35	13.07	4.16	0.00	0.00	0.00	0.19	0.00	2.22	99.99	Paste	0.53
17	78.74	12.63	4.93	0.00	0.00	0.00	0.35	0.67	2.68	100.00	Paste	0.54
18	54.58	16.13	22.66	0.00	0.03	0.00	0.59	0.60	5.40	99.99	Grain	1.35

Figure 15: Backscatter electron image (top) and energy-dispersive X-ray elemental analyses (bottom) of various areas of paste from different areas that were selected to be free of any mineral grains i.e. to obtain the true compositions of the paste. Last column shows the cementation index after Eckel (1922, CI = [(2.8* SiO_2)+(1.1*Al_2O_3)+(0.7*Fe_2O_3)]/[(CaO)+(1.4*MgO)]), which measures relative hydraulicity of paste e.g., non-hydraulic lime pastes have very low CI (< 1) compared to Portland cement pastes (CI is >1).

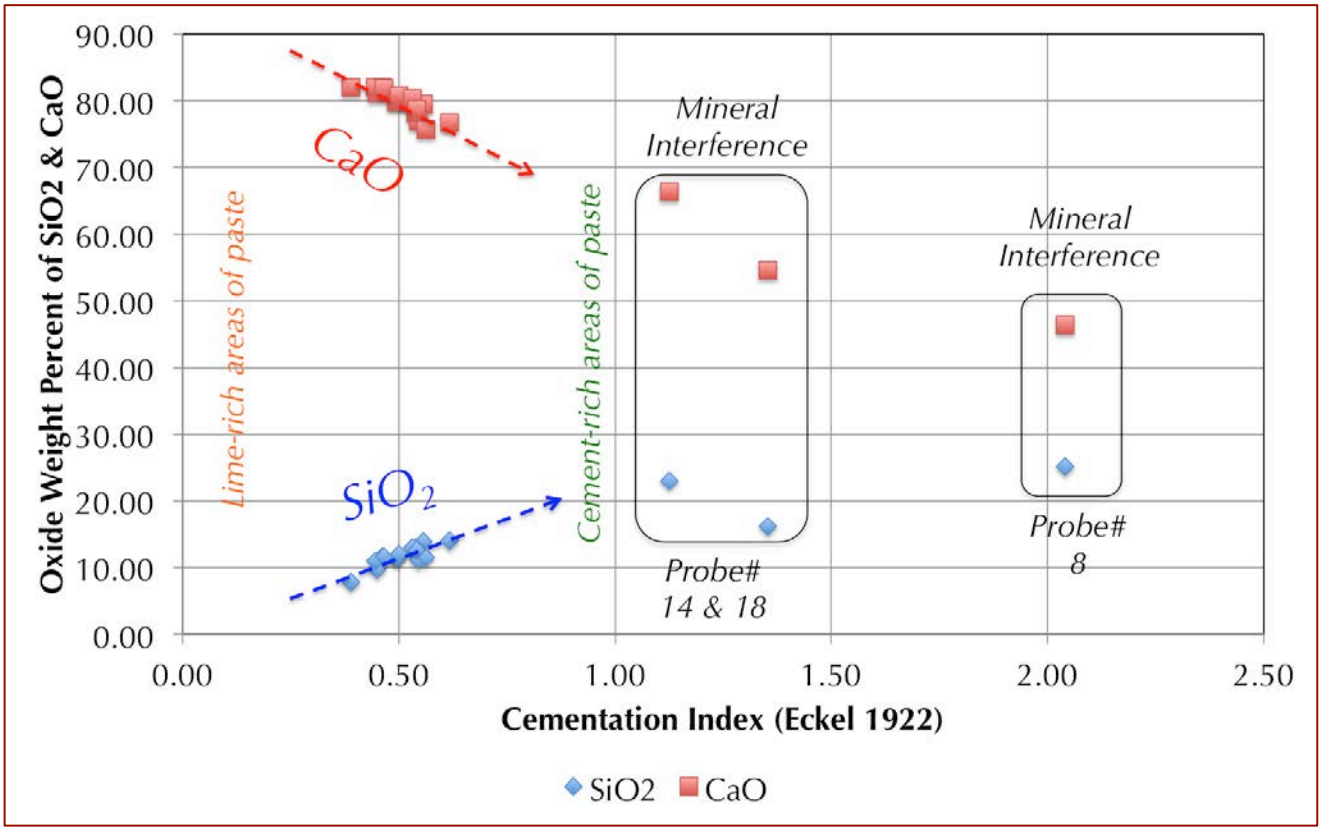


Figure 16: Variations in silica and calcium oxide compositions of paste from various areas shown in Figure 15 showing overall higher proportion of lime relative to cement thus concentrating the median CI around 0.50 (but still a linear trend on these oxide variation diagrams consistent with cement-lime composition of paste). The spread of the plots thus indicate a typical lime-cement composition of the binder. A few plots at higher CI areas from mineral interferences in the analyses.

AIR

The mortar is non-air-entrained (Figures 8, 13, and 14). The estimated air content is 6 to 8 percent, which are all interstitial void spaces between sand particles.

BULK MORTAR MINERALOGY FROM XRD

Table 3 and Figure 17 summarize results of semi-quantitative mineralogical compositions of bulk mortar from X-ray diffraction, and, sources of major minerals as suspected or determined from optical microscopy.

Mineralogical Components	Semi-Quantitative Amount (%)	Source
Quartz	94.5	Quartz sand particles in Sand
Calcite	5.5	Carbonated Paste

Table 3: Mineralogical composition of bulk mortar determined from XRD.

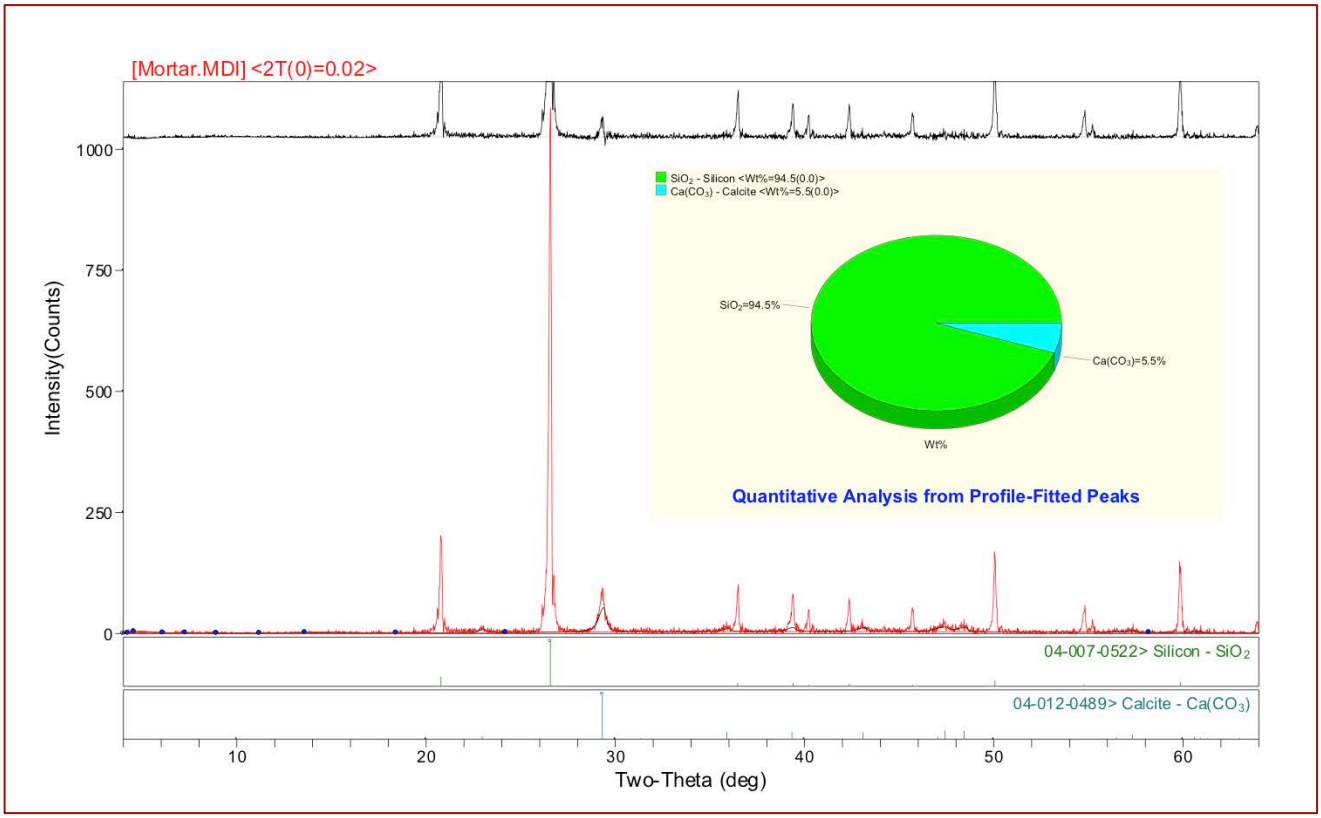


Figure 17: X-ray diffraction pattern of bulk mortar showing dominant quartz from quartz sand, and minor calcite from carbonated lime-cement matrix. Inset shows semi-quantitative estimate of phases, recalculated to 100% amongst the two detectable phases.

CHEMICAL ANALYSES

BULK OXIDE COMPOSITION OF MORTAR FROM XRF

Table 4 summarizes the bulk oxide composition of mortar as determined from energy-dispersive X-ray fluorescence spectroscopy (ED-XRF) of pressed pellet of finely ground mortar.

Oxide (wt.%)	Mortar
SiO_2	76.3
Al_2O_3	1.92
Fe_2O_3	1.47
CaO	12.5
MgO	0.427
Na_2O	ND
K_2O	0.115
TiO_2	0.102
P_2O_5	0.06
SO_3	0.181
Balance	6.97
Total	100.0

Table 4: Bulk oxide compositions of mortars as determined from ED-XRF. Balance corresponds to loss on ignition (e.g., H_2O, CO_2, etc.). ND = Not detected due to negligible amount.

Result shows overall SiO_2 rich composition of the bulk mortar that is consistent with dominant quartz mineralogy as determined from X-ray diffraction. Figure 18 shows X-ray elemental spectrum of mortar from XRF.

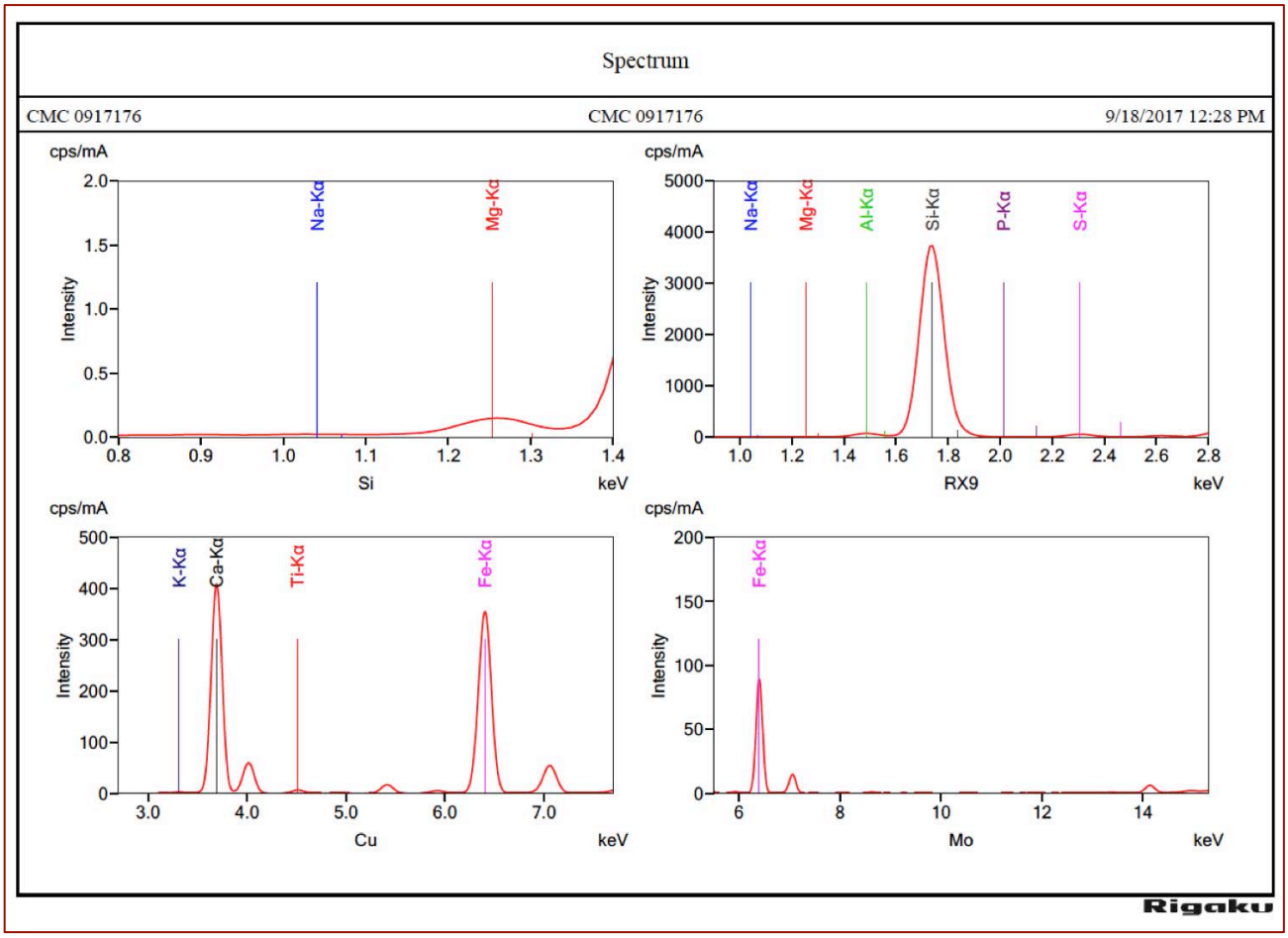


Figure 18: XRF peaks of elements (as oxides) detected in the bulk mortar (from Quant EZ software of Rigaku).

CEMENT CONTENT (FROM SOLUBLE SILICA CONTRIBUTED FROM PORTLAND CEMENT OR OTHER HYDRAULIC BINDER)

Table 5 shows results of determination of Portland cement content in the mortar from the soluble silica contents that are contributed from the Portland cement.

For soluble silica determination *a la* ASTM C 1324, the combined filtrate for analysis is obtained from double filtration of (i) an original 5.00 grams of pulverized mortar i.e. after digestion in 100-mL cold (at 3 to 5°C) HCL followed by filtration (filtrate#1), (ii) then digestion of the residue left along with the filter paper in 75-mL hot (below boiling) NaOH followed by filtration (filtrate#2) and then (iii) combination of two filtrates and re-filtering of the combined filtrate for the third time to remove any suspended silica. The combined filtrate is then used for determination of soluble silica and cement content in an XRF against empirical calibration of intensities of known soluble silica and cement contents of multiple mortars (5.00 grams) prepared by the same procedure of cold HCL-

digestion/filtration/hot NaOH-digestion/2nd filtration/combination of two filtrates/re-filtration. Cement contents from soluble silica contents are then calculated assuming 20.2 percent soluble silica in the Portland cement that was used for preparation of Portland cement mortar standards used in the calibrations. Cement content is calculated from the formula: Cement Content (%), CC = [ppm SiO_2 × 100] ÷ [5.00 (mortar weight used) × 0.202 (assuming a 20.2% SiO_2 content in the Portland cement used) × 4 (dilution factor of final 250 ml filtrate to 1L) × 1000 (conversation factor of g/L to mg/L or ppm)]. Cement content is calculated based on the determination of Portland cement component as a cementitious binder in the mortar, as confirmed from petrographic examinations.

Component	Mortar
Cement Content from Soluble Silica Content (%)	4.23

Table 5: Cement content calculated from the soluble silica content in the mortar. Cement content is calculated based on the determination of Portland cement component as a cement binder used in the mortar, and assumed 20.2 percent soluble silica in Portland cement.

SAND CONTENT FROM ACID-INSOLUBLE RESIDUE, FREE PLUS COMBINED WATER CONTENTS, AND CARBONATION FROM LOSS ON IGNITION

Table 6 summarizes results of acid-insoluble residue content of mortar, after digesting a pulverized (to pass US No. 50 sieve) portion of bulk mortar in hydrochloric acid, and, loss on ignition of a separate aliquot of pulverized mortar to 110°C, 550°C, and 950°C, which correspond to free water, combined (hydrated) water, and degree of carbonation, respectively. Due to the presence of siliceous components in the sand (as determined from petrography) and no calcareous components, the determined acid-insoluble residue content is considered to closely correspond to the sand content of the mortar. The loss on ignition at 550°C corresponds to combined water from cement hydration. The loss on ignition at 950°C corresponds to calcium carbonate and degree of carbonation in mortar.

Components	Mortar
Acid-Insoluble Residue (%)	66.56
Loss on Ignition: From 0°C to 110°C (Free Water) (%)	0.50
Loss on Ignition: From 110-550°C (Combined Water) (%)	1.80
Loss on Ignition: From 550-950°C (Carbonation, CO_2) (%)	8.90

Table 6: Hydrochloric acid-insoluble residue content and loss on ignition to 110°C, 550°C and 950°C.

Figure 19 shows photomicrographs of sand particles from acid-insoluble residue from mortar that contain siliceous components (e.g., quart, feldspar, sandstone) in sand.

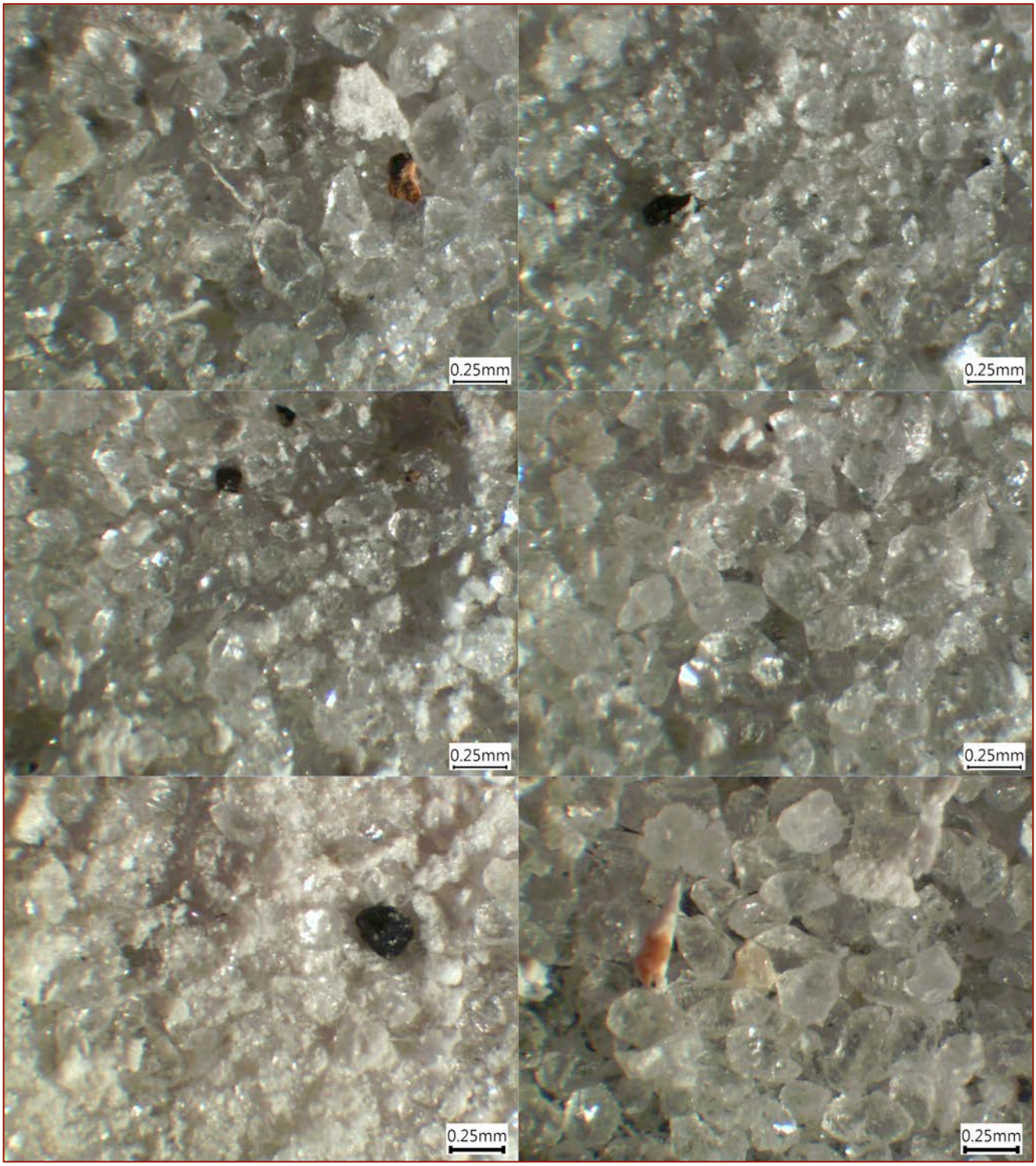


Figure 19: Acid-insoluble residue content of mortar, after digesting a pulverized (to pass US No. 50 sieve) portion of bulk mortar in hydrochloric acid showing pulverized siliceous sand residue consisting of clear (transparent) to off-white quartz sand.

CALCITIC VS. DOLOMITIC LIME FROM BRUCITE CONTENT IN BULK MORTAR & MAGNESIUM OXIDE CONTENT IN BINDER

The negligible magnesia contents of paste measured from SEM-EDS studies indicate use of a high-calcium lime rather than a dolomitic one in the binder along with Portland cement.

CALCULATIONS OF MIX PROPORTIONS OF MORTAR

Aided with the data obtained from petrography and chemical analyses of mortar, the following Table first summarizes all chemical data, followed by calculations of proportions of various ingredients in the mortar from a set of assumed compositions and bulk densities of the ingredients:

Mortar Composition & Mix Proportion	Mortar
Chemical Compositions	
Cement Content from Soluble Silica, SiO_2 (%)	4.23 (Portland cement)
Bulk Silicon dioxide, SiO_2 (%)	76.3
Bulk Calcium Oxide, CaO (%)	12.5
Magnesium Oxide, MgO (%)	0.427
Acid-Insoluble Residue (%)	66.56 (Siliceous sand)
Loss on Ignition: From 0°C to 110°C (Free Water) (%)	0.50
Loss on Ignition: From 110-550°C (Combined Water) (%)	1.80
Loss on Ignition: From 550-950°C (Carbonation, CO_2) (%)	8.90
Magnesium Hydroxide (Brucite) (%)	-
Assumed Compositions & Densities	
Portland Cement – From Soluble Silica (SiO_2) (%)	20.2 (Portland Cement)
Bulk Density of Portland Cement, (lbs./ft.^{3})	94
Hydrated Lime or Lime putty	From CaO content, after assigning CaO for Portland Cement and assuming 63.5% CaO in Portland Cement, and converting the residual CaO to lime $Ca(OH)_2$ by multiplying the residual CaO with the factor 1.322 (mol. wt. of lime to CaO = 74.03/56 = 1.322)
Bulk Density of Lime, (lbs./ft.^{3})	40
Bulk Density of Sand, (lbs./ft.^{3})	80
Calculated Volumetric Proportions	
Portland Cement Content (%)	Portland Cement (PC) Content from Soluble Silica Data = 4.23%
Lime Content (%), assuming 63.5% CaO in Portland cement	1.322 × [CaO content in mortar i.e. 12.5 – (cement content i.e. 4.23 × 0.635)] = 12.9%
Sand Content (%)	66.56 (from acid-insoluble residue content, entire sand is siliceous sand)
Portland Cement Volume	4.23/94 = 0.045
Hydrated Lime Volume	12.9/40 = 0.322
Sand Volume	66.56/80 = 0.832
Relative Volumes of Binder Phases: Sand	Portland Cement: Lime: Sand = 0.045: 0.322: 0.832 = 1: 7: 18.5 (2.3 times the sum of separate volumes of cement and lime)
Cement to Lime to Sand, by volume	**1 part cement to 7-part lime to 18.5-part sand**

Mortar Composition & Mix Proportion	Mortar
	(2.3 times sand of the sum of separate volumes of cement and lime) – under-sanded
Equivalent ASTM C 270 Mortar Type	**Type N cement-lime mortar**

Table 7: Calculations of mix proportions of mortar, by volume, from the determined chemical compositions, and, assumed compositions and bulk densities of mortar's ingredients.

The soluble silica content of the mortar has provided the corresponding Portland cement content, by assuming 20.2 percent soluble silica in Portland cement. Lime content is determined from the bulk calcium oxide content of mortar, after assigning CaO for Portland cement and assuming 63.5% calcium oxide in Portland cement, and converting the residual CaO to lime i.e. $Ca(OH)_2$ by multiplying the residual CaO with the conversion factor 1.322 [ratio of mol. wt. of lime i.e. $Ca(OH)_2$ to CaO is 74.03/56 = 1.322]. Since the sand is determined to be siliceous (quartz-quartzite) sand, and contains no calcareous component, the sand content is determined from the hydrochloric acid-insoluble residue content of mortar. Assuming bulk densities of Portland cement, lime, and sand as 94, 40, and 80 lbs./ft^3, respectively, the volumetric proportion of Portland cement to lime to sand is calculated to be: 1-part cement to 7-part lime to 18.5-part sand by volume, which is similar to an ASTM C 270 Type N cement-lime mortar.

CONCLUSIONS

AGGREGATE

Sand contains major amounts of quartz and quartzite, subordinate amounts of feldspar, granite, and ferruginous particles. No calcareous component is detected in the sand. Sand used was, clean, well-graded (compares within the upper and lower limits of size distribution of masonry sand specifications in ASTM C 144 for natural sand), well-distributed, and present in sound condition with no evidence of any deleterious alkali-aggregate reaction of sand.

An appropriate substitute of this original sand in a tuck-pointing mortar would be a siliceous natural or crushed sand conforming to the specifications of ASTM C 144 for modern masonry sand that is clear and free of any contaminants and does not contain excessive fines. The color of the sand should match with the existing sand.

ORIGINAL BINDER

The binder contains a Portland cement and hydrated lime (or lime putty) where the former is present as residual cement particles in a dominantly carbonated matrix of lime and hydration products of cement. The overall matrix is dominated by slaked lime of possible high-calcium origin as indicated by porous, fine-grained carbonated cryptocrystalline to microcrystalline calcite masses of carbonated lime matrix, nominal magnesia content of bulk mortar, and negligible magnesia content of paste. Residual Portland cement particles show well hydration with a few remnants and relicts of cement particles (mostly dark interstitial ferrite matrix of cement particles).

TYPE OF MORTAR

Based on detailed laboratory studies and calculated mix proportions, the mortar is determined to be a cement-lime mortar, equivalent to an ASTM C 270 Type N cement-lime mortar in having 1-part cement to 7-part lime to 18.5-part sand by volume.

SUGGESTED TUCK POINTING MORTAR

Based on:

a. The determined cement-lime composition of mortar,
b. Natural siliceous sand compositions of aggregate, and
c. The calculated volumetric proportions of 1-part cement to 7-part lime to 18.5-part sand by volume,

A possible tuck-pointing mortar could be:

a. A modern ASTM C 270 Type N cement-lime or a masonry cement mortar made using Portland cement in conformance to ASTM C 150, hydrated lime in conformance to ASTM C 207, masonry cement in conformance to ASTM C 91, and sand in conformance to ASTM C 144, to test the similarities in behavior and physical/mechanical properties of new tuck-pointing mortar to the existing mortar over a small test area;
b. The final choice of binder and sand ingredients would depend on the match in appearance, compositions, and properties with the original mortar. Design and formulation of an appropriate tuck-pointing mortar should be based on trial and error on small test areas by the project engineer/architect.
c. Appendix A2 provides various general suggestions for formulation of a tuck pointing mortar.

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✪ ✪ ✪ END OF TEXT ✪ ✪ ✪

APPENDIX A1 – METHODOLOGIES FOR LABORATORY TESTING OF MASONRY MORTARS

METHODOLOGIES

The mortar sample was tested by following the methods of ASTM C 1324 "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar," along with various analytical methods to test masonry mortars as described in various literatures, e.g., Erlin and Hime 1987, Doebley and Spitzer 1996, Chiari et al. 1996, Middendorf et al. 2005 a and b, Elsen 2006, Bartos et al. 2000, Valek et al. 2012, Jana 2005, 2006, and Goins 2001 and 2004.

For laboratory testing of masonry mortars, CMC provides two packages – a *basic package*, and, a *comprehensive package*. Tests followed in both packages are shown in Figure 5. Basic package is suitable for modern masonry mortars, formulated by following the specifications of ASTM C 270, whereas comprehensive package is more informative for historic mortars, or mortars that are not formulated according to the specifications of ASTM C 270, or have components outside the Portland cement, hydrated lime (or lime putty), masonry cement, and mortar cement components recommended in formulations of ASTM C 270 mortars. Both packages provide volumetric proportions of various binder and sand components that are useful for formulation of a tuck-pointing mortar to be used as a replacement of the examined mortar.

The present mortar was requested to be analyzed by the *basic package*. Therefore all tests including Test Nos. 1, 3, 4, 5, and 7 in Figure A1-1 were followed to determine the composition of the mortar and assess appropriate formulation for a tuck-pointing mortar that could be suitable for replacement of the examined mortar. Additionally, SEM-EDS and sand size distribution were done for better understanding of the mortar.

SAMPLE SELECTION & SAMPLE PREPARATION

From the mortar fragments received, 'representative' subset fragment(s) were selected for various laboratory techniques, e.g., visual examinations, digital and flatbed scanner photography, optical microscopy using a Stereozoom and a petrographic microscope both equipped with reflected, transmitted, polarizing-light facilities, scanning electron microscopy and energy-dispersive X-ray fluorescence spectroscopy (SEM-EDS), X-ray fluorescence of bulk mortar, X-ray diffraction (of bulk mortar, sand extract, or binder extract), various chemical analyses (e.g., gravimetric), and thermal analysis (e.g., DTA, TGA, DSC). As shown in the flowchart, original mortar was first examined visually and with the help of a Stereozoom microscope at low magnifications. Any piece that appeared 'unusual' in visual examinations from the rest i.e. in having markedly different appearance was not included, since the piece may represent a different mortar accidentally mixed up with the mortar to be analyzed during the retrieval process (e.g., in masonries that have received multiple episodes of renovations and repointing in the past). From the pieces representative of the mortar to be analyzed, two broad groups were separated, one for microscopy (optical microscopy and SEM-EDS), and, the other group for chemical analysis,

XRD-XRF, thermal analysis, sand extraction and sieve analyses, etc. Information obtained from microscopy is used to devise appropriate procedure to be followed in subsequent chemical analyses.

Laboratory Examinations of Masonry Mortar

Initial Mortar (50+ grams) [Photographed with digital camera & flat-bed scanner, As-received condition, total weight, and dimensions of largest piece are documented]

Intact Pieces (20+ g)

1. Optical Microscopy

i. Take digital and flat bed scanner photos of intact piece(s) as received,
ii. Encapsulate a representative piece in a mold with a low-viscosity colored dye-mixed epoxy to highlight voids, pores, cracks, etc.,
iii. Prepare thin section and polish the thin section for SEM-EDS analyses,
iv. Scan the thin section on a flat-bed scanner with the thin section residue,
v. Take transmitted light stereo-zoom photomicrographs of thin sections from different areas to determine volumes of pore spaces and sand by Image J,
vi. Take plane and crossed polarized-light photomicrographs of sand and binder fractions in thin section from a petrographic microscope and determine areas for further studies by SEM-EDS,
vii. Do detailed petrographic examinations in Stereozoom and Petrographic microscopes.

2. SEM-EDS

i. Gold-coat only the portion of polished thin section or a solid polished piece intended for SEM-EDS studies,
ii. Take backscatter and/or secondary electron images, and if needed x-ray elemental maps,
iii. Select multiple areas on paste by point and/or raster modes to determine elemental compositions and cementation indices
iv. Tabulate compositional variations of paste across the backscatter/secondary electron image.

Lightly hand-ground in a Mortar & Pestle (30+ g)

3. Chemical Analysis – Soluble Silica (5 g)

i. Grind 10 g of lightly ground fraction from mortar & pestle in a WC pulverizer for 30 sec.
ii. Sieve thru. No. 50 sieve, collect the fraction passing the sieve,
iii. Re-grind the residue retained on sieve for 15 sec. and mix thoroughly with the previous fraction;
iv. Use 5.00 g of thus prepared powder (passing No. 50 sieve) for digestion in 100 ml cold (3-5°C/ 38-41°F) HCl (1+4) in a 250 ml beaker for 15 min. on a magnetic stirrer,
v. Filter thru. two 2.5 micron filter paper and keep the filtrate# 1,
vi. Digest the residue with filter paper in 75 ml hot NaOH (below boiling) on hot plate for 15 min. on magnetic stirrer,
vii. Cool down to room temp. and filter thru. two 2.5 micron filter paper and collect filtrate# 2,
viii. Combine these two filtrates, filter the combined filtrates thru. two 2.5 micron filter paper to remove any suspended silica (especially for sand-rich mortars, or if mortar is grounded too long); then dilute to 250 ml in a volumetric flask with dist. water, an aliquot (about 10 ml) is then used soluble silica determination (e.g., in AAS/XRF against calibrations with standard PC mortars of known soluble silica and cement contents prepared in the exact same ways).

4. Chemical Analysis – Acid-Insoluble Residue (2 g)

i. Take 1-2 g of prepared mortar powder from Step 3 iii (passing No. 50 sieve) and digest in 50 ml HCl (1+3) in a 250 ml beaker (covered) on a hot pate rapidly near boiling, then 15 min. at a temp. below boiling, then cool down to room temperatures,
ii. Filter thru. two pre-weighed 2.5 micron filter papers, washing the beaker, paper, and residue thoroughly with hot water,
iii. Dry the filter paper at 110C for 10 min, cool in a desiccator to room temp. and measure the weight.
iv. Subtract from mass of dry filter paper to determine acid-insoluble residue content.
v. Take digital/optical photomicrographs of acid-insoluble residue (e.g., siliceous sand).

5. Chemical Analysis – Loss On Ignition (2 g)

i. Take 1-2 g (W_1) of prepared mortar powder from Step 3 iii (passing No. 50 sieve) in a tarred porcelain crucible (keep a record of mass of the empty crucible),
ii. Dry at 110°C for 15 min in a muffle furnace pre-set to 110°C, cool in a desiccator to room temp. and measure the mass (W_2) by subtracting the empty crucible mass from the total mass,
iii. Ignite at 550°C for 15 min. in the muffle furnace pre-set to 550°C, cool in a desiccator to room temp. and measure the mass (W_3) by subtracting the empty crucible mass from the total mass,
iv. Ignite at 950°C for 15 min. in the muffle furnace pre-set to 950°C, cool in a desiccator to room temp. and measure the mass (W_4) by subtracting the empty crucible mass from the total mass,
v. Calculate the losses on ignition at 110°C, 550°C, and 950°C for free water, combined water, and carbonation, respectively.

6. Sand Color & Size Distribution (10 g)

i. Take 10 g. of mortar lightly ground in mortar & pestle and digest in HCl (1+3) in a 250 ml beaker on a magnetic stirrer until all sand separates and settles at the bottom of beaker,
ii. Filter all through two 2.5 micron filter paper, wash the beaker, filter paper, and all sand residue with dist. water,
iii. Dry the residue at 110°C in an oven for 10 min., gently brush out from the filter paper and collect, then sieve the entire sand residue through No. 4 through 200 sieves,
iv. Determine the mass retained on each sieve, and on the pan (finer than No. 200 sieve),
v. Take photomicrographs of sand particles retained on each sieve for sand color variations.
vi. Provide plots of grain-size variations of sand against ASTM C 144 limits for masonry sands.

7. Bulk Mortar Composition from XRF (8 g)

i. Weigh 8.00 g of mortar lightly ground in mortar & pestle, add three grinding/pelletizing aid tablets and re-grind in a WC mill for 3 min. with anhydrous alcohol to get <45 micron size (passing No. 325 sieve),
ii. Take 6.8 to 7.0 g. of ground <45 micron prepared mass in an aluminum sample holder inside a die to prepare a 32 mm pellet with 25 ton pressure for 1 min,
iii. Use the prepared pellet for XRF and then use same the same pellet for XRD.

8. Bulk Mortar Mineralogy from XRD

i. Use the XRF pellet to run in Siemens D5000 theta/two-theta powder diffractometer (Cu Kα radiation, 40 kV, 30mA) with MDI's Datascan, Jade Search/Match, & Rietveld modules to determine mineralogical composition of bulk mortar.

9. Thermal Analyses (1 g), e.g., for dolomitic lime mortar to obtain the brucite content.

Figure A1-1: Flowchart of laboratory testing of masonry mortar followed in the CMC laboratories. For our 'comprehensive package' all above tests are done. For 'basic package', Nos. 1, 3, 4, 5, and 7 are done. Both packages provide volumetric proportions of various binder components and sand that are helpful for formulation of a suitable pointing mortar. Comprehensive package is more useful for historic mortars that are not similar to modern ASTM C 270 cement-lime or masonry/mortar cement mortars. Basic package is suitable for modern mortars that are formulated following the specification of ASTM C 270.

OPTICAL MICROSCOPY

Along with ASTM C 1324, procedures for optical microscopical examinations of construction materials are also described in ASTM C 856. Fragment(s) selected for microscopical examinations are examined and photographed with a digital camera, a flatbed scanner, and, a low-power stereomicroscope. After preliminary visual examinations and photographing, subsequent sample preparation steps are followed for optical microscopy. Figure A1-2 shows the equipments used during preparation of thin section and examination by using various optical microscopes.

For thin section preparation, representative fragments are placed in a flexible (molded silicone) sample holder, and encapsulated with a colored (blue or fluorescent) dye-mixed low-viscosity epoxy resin under vacuum to impregnate the pore spaces of mortar and improve the overall integrity by the cured epoxy. The epoxy-encapsulated cured solid block of sample is then de-molded and processed through coarse to fine grinding, lapping, attachment of the lapped surface to a frosted large-area (50 × 75 mm) glass slide, precision sectioning and precision grinding in a thin-sectioning machine (Figure A1-2), and final polishing steps to prepare a final polished thin section of 30 micron thickness suitable for examinations in a petrographic microscope and in SEM-EDS. Sample preparation steps are described in detail in Jana 2005, 2006.

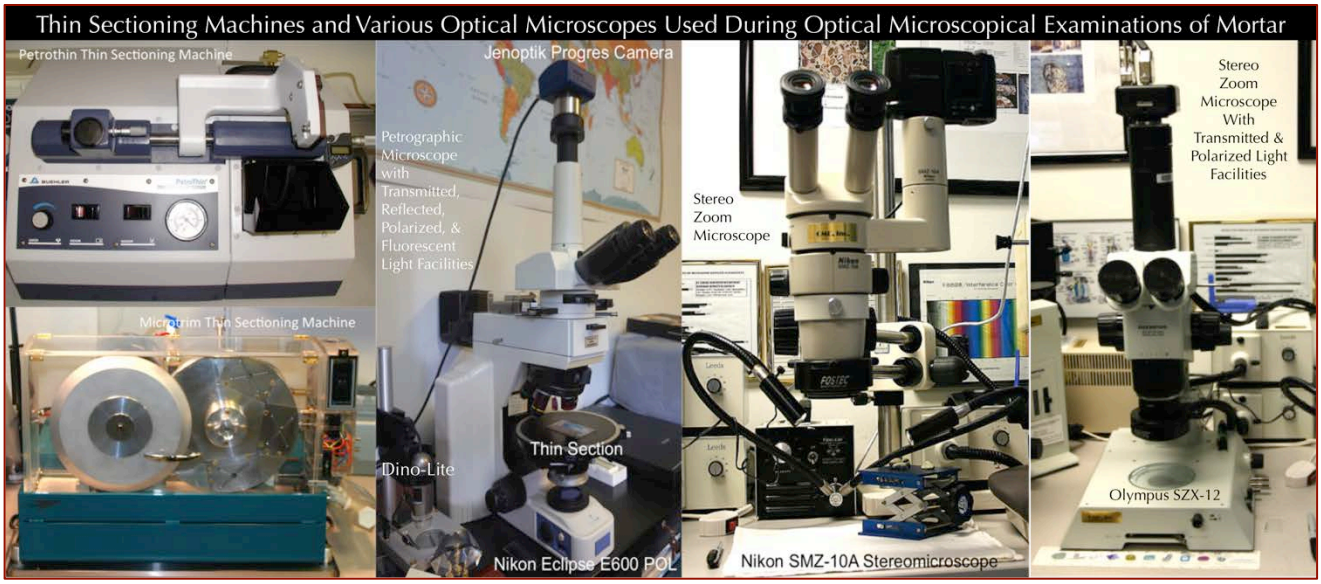

Figure A1-2: Buehler (top, left) and Microtrim (bottom left) thin-sectioning machines used for production of less than 30-micron thin sections of mortars. Four 50 × 75 mm size thin sections can be simultaneously prepared by the Microtrim unit. Nikon Eclipse E600 POL petrographic microscope with Jenoptik Progres Camera (left), Nikon SMZ-10A Stereozoom reflected-light microscope with Omax camera (middle), and Olympus SZX-12 Stereozoom microscope with reflected, transmitted, and polarized light facilities and Progres camera used for examinations of mortar

Steps followed during light optical microscopical examinations of a mortar sample include:

a) Visual examinations of mortar fragments, as received, to select fragments for detailed optical microscopy; initial digital and flatbed scanner photography of mortar as received;

b) Low-power stereomicroscopic (e.g., by using Nikon Stereozoom microscope shown in Figure A1-2) examinations of saw-cut and freshly fractured sections of mortar for evaluation of textures, compositions, and appearances;

c) Examinations of oil immersion mounts for special features and materials from mortar in a petrographic microscope (e.g., Nikon Eclipse E600 POL shown in Figure A1-2);

d) Examinations of colored (blue or fluorescent) dye-mixed epoxy-impregnated polished thin sections of mortar fragments in a transmitted-light Stereozoom microscope (e.g., Olympus SZX-12 microscope shown in Figure A1-2) for determination of size, shape, angularity, and distribution of sand, as well as abundance and distribution of void and pore spaces that are highlighted by the colored dye-mixed epoxy;

e) Image analyses of photomicrographs of blue dye-mixed epoxy-impregnated thin sections of mortar fragments for estimations of pores, voids, intergranular open spaces, and shrinkage microcracks (i.e. areas that were impregnated by blue dye-mixed epoxy and highlighted in image analysis) by using Image J, a Java-based image processing program developed by National Institutes of Health. Large-area thin section photomicrographs are collected in plane and crossed polarized light modes by using a high-resolution Stereozoom microscope equipped with transmitted and polarizing light facilities (e.g., Olympus SZX-12 microscope shown in Figure A1-2);

f) Examinations of colored (blue or fluorescent) dye-mixed epoxy-impregnated polished thin sections of mortar fragments in a petrographic microscope (Figure A1-2) for detailed compositional, mineralogical, textural, and microstructural analyses of aggregates and binders in mortars, along with diagnoses of evidence of any deleterious processes. The purpose of using a colored dye-mixed epoxy is to highlight the overall variations in density/porosity of mortars as well as highlighting any void spaces and cracks in the samples;

g) Examinations of any physical or chemical deterioration of mortar or signs of improper construction practices from microstructural evidences; and,

h) Optical microscopical examinations of size, shape, and color variations of sand extracted after hydrochloric acid digestion from determination of acid-insoluble residue content.

SCANNING ELECTRON MICROSCOPY AND ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (SEM-EDS)

For comprehensive package, a portion of the thin section used for optical microscopy is subsequently coated with a thin conductive gold film for detailed SEM-EDS studies.

Procedures for SEM examinations are described in ASTM C 1723. Polished and coated thin section (or polished solid encapsulated block) of mortar is examined in a CamScan SEM equipped with backscatter detector, secondary electron detector, and x-ray fluorescence spectrometer (Figure A1-3) to observe:

a) The morphology and microstructure of various phases; and,

b) Determine the chemical compositions of the binders, including the original components of the binders, and the hydration and/or carbonation/alteration products.

Figure A1-3: Cambridge CamScan Series II Scanning Electron Microscope and 4Pi Revolution software, backscatter detector, secondary electron detector, and energy-dispersive X-ray fluorescence spectrometer used for microstructural and microchemical analyses of mortar.

Due to characteristic difference in compositions of pastes made using various binders, e.g., non-hydraulic lime (CaO dominants over all other oxides), variably hydraulic lime (CaO with variable SiO_2 contents depending on hydraulicity), dolomitic lime (high CaO and MgO), natural cement (CaO, SiO_2, and MgO contents are high, high MgO and FeO contents are characteristic), and Portland cement (CaO and SiO_2 contents are higher than all other oxides), SEM-EDS analysis of paste is a powerful method for detection of the original binder components) in the mortar. Effects of chemical alterations and various chemical deteriorations of a mortar can also be detected by SEM-EDS.

X-RAY DIFFRACTION

X-ray diffraction is a powerful laboratory technique during investigation of masonry mortars, for various reasons, such as:

a) Determination of bulk mineralogical composition of mortar, including its aggregate and binder mineralogies; e.g., quartz in sand from major diffraction peaks at 26.65º, 20.85º, 50.14º 2θ, or calcite in sand or carbonated lime binder from major peaks at 29.41º, 39.40º, 43.15º 2θ, or Portlandite in binder from major peaks at 34.09º, 18.09º, 47.12º 2θ;
b) Individual primary mineralogy and alteration products of aggregate at various size fractions, and binder phases;
c) Detection of dolomitic lime binder from brucite in the mortar from major peaks at 38.02º, 18.59º, 50.86º 2θ;
d) Detection of use of lime (Portlandite), gypsum (11.59º, 20.72º, 29.11º 2θ), or cement binders from their characteristic mineralogies;
e) Detection of any potentially deleterious constituents, e.g., deleterious salts, or efflorescence deposits;
f) Detection of a mineral oxide-based pigmenting component in the mortar; and,
g) Detection of components that are difficult to detect by microscopical methods.

X-ray diffraction can be done on (see Figure A1-5): (i) pulverized (to finer than 45 micron) portion of bulk mortar, or (ii) on the sand extracted from the mortar by acid digestion, if sand has a complex mineralogy, or requested for additional examination, or also (iii) on the binder-fraction by separating the sand from the binder from a lightly ground mortar (in a mortar and pestle) and passing the ground mass through US 325 sieve (44 micron) to collect the fraction rich in binder.

Figure A1-4: Siemens D5000 X-ray diffractometer and MDI Jade search/match software used for determination of mineralogical composition of mortar.

X-ray diffraction is carried out in a Siemens D5000 Powder diffractometer (Figure A1-4) employing a long line focus Cu X-ray tube, divergent and anti-scatter slits fixed at 1 mm, a receiving slit (0.6 mm), diffracted and incident beam Soller slits (0.04 rad), a curved graphite diffracted beam monochromator, and a sealed proportional counter. Generator settings used are 45 kV and 30mA. A dry, finely ground sample pulverized to pass US 325 sieve (44-μm) is placed in a 1-in. diameter circular sample holder and excited with the copper radiation of 1.54 angstroms. Tests are performed at a 2-theta range from 4° to 64° with a step of 0.02° and a dwell time of one second.

The resulting diffraction patterns are collected by using DataScan 4 software of Materials Data, Inc. (MDI), analyzed by using Jade 9.0 software of MDI with ICDD PDF-4 (Minerals 2017) diffraction data, and, phase identification, and quantitative analyses were carried out with MDI's Search/Match and Easy Quant modules, respectively. Steps followed during

sample preparation for XRD are provided in Figure A1-5. For the present mortar, pulverized (to minus US 325 sieve) portions of representative fragments of bulk mortar sample (Step 2 in Figure A1-5) are analyzed.

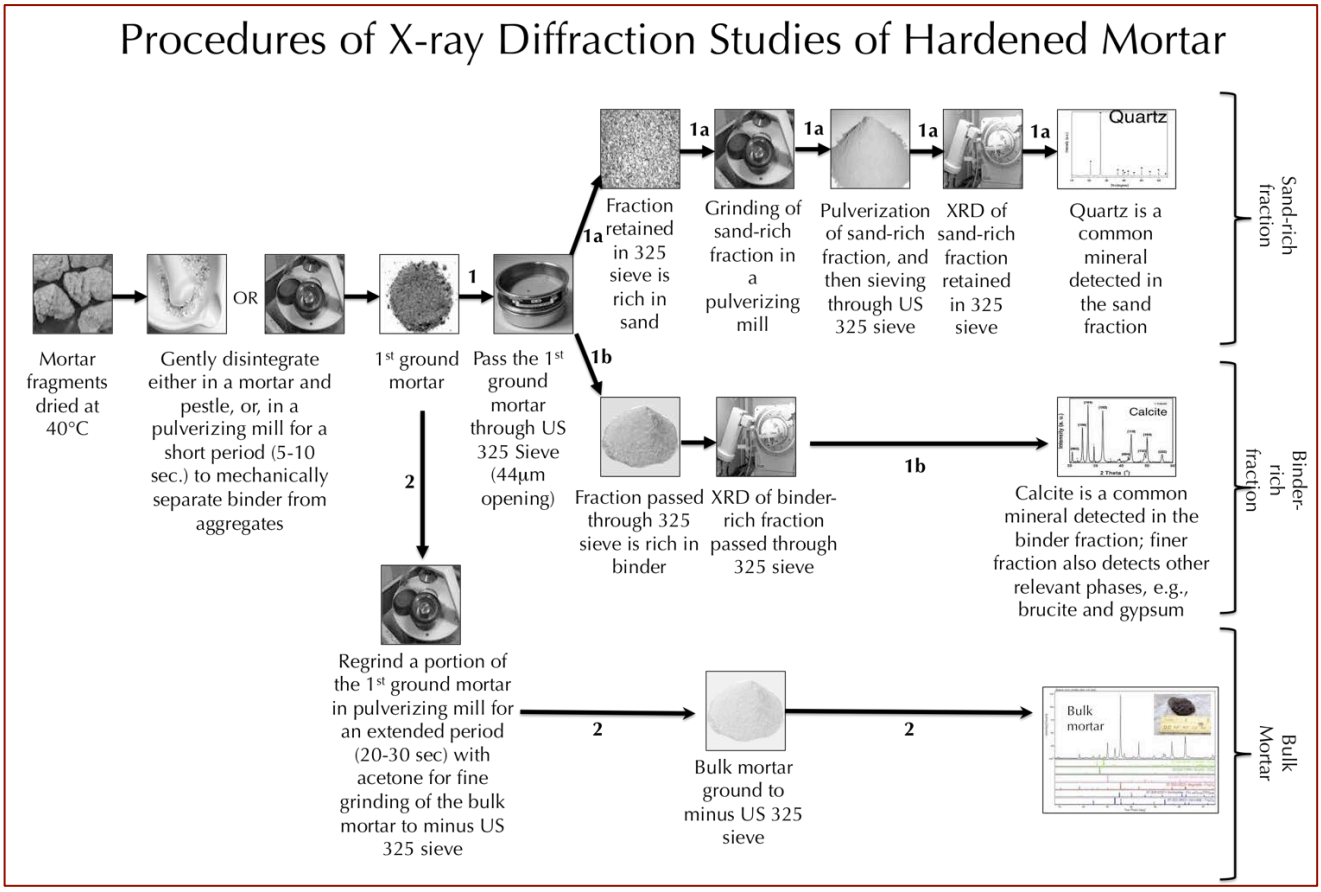

Figure A1-5: Steps followed during sample preparation for X-ray diffraction studies.

ENERGY-DISPERSIVE X-RAY FLUORESCENCE SPECTROSCOPY (ED-XRF)

An energy-dispersive bench-top x-ray fluorescence unit from Rigaku Americas Corporation (NEX-CG, Figure A1-6) is used for determination of bulk chemical (oxide) composition of mortar. The instrument is calibrated by using various certified reference standards of cements and rocks.

Figure A1-6: Rigaku NEX-CG bench-top ED-XRF unit used for bulk chemical composition of mortar.

A representative portion of mortar (about 8 grams) is pulverized down to minus US 325 sieve (finer than 45 microns size) in a Rocklab pulverizer with a grinding

aid/binder (7.5% binder by weight of sample), and then pelletized (approximately 7 grams) to a 31-mm diameter pellet in a 25-ton press.

The instrument is powerful enough to determine ppm-level silica in solutions suitable for determination of soluble silica content in an acid-digested mortar from calibration of laboratory-prepared standard mortar samples of known cement and soluble silica contents.

CHEMICAL ANALYSES (GRAVIMETRY & INSTRUMENTAL ANALYSES)

Following petrographic examinations, chemical analyses of the mortar are done to determine the:

a) Hydrochloric acid-insoluble residue content,
b) Loss on ignition,
c) Soluble silica content,
d) Calcium and magnesium oxide contents, and
e) The presence of magnesium hydroxide, if any (e.g. to determine if dolomitic lime was used).

Chemical analyses are done by using various methods outlined in ASTM C 1324 and Middendorf et al. 2005a, e.g., by wet chemistry, atomic absorption spectroscopy (AAS), inductively-coupled plasma atomic emission spectroscopy (ICP-AES), energy-dispersive X-ray fluorescence spectroscopy (EDS), thermal analysis (DTA, TGA, DSC), and X-ray diffraction (XRD). Steps followed during chemical analyses of mortars are described in ASTM C 1324, which are summarized in Figure A1-7.

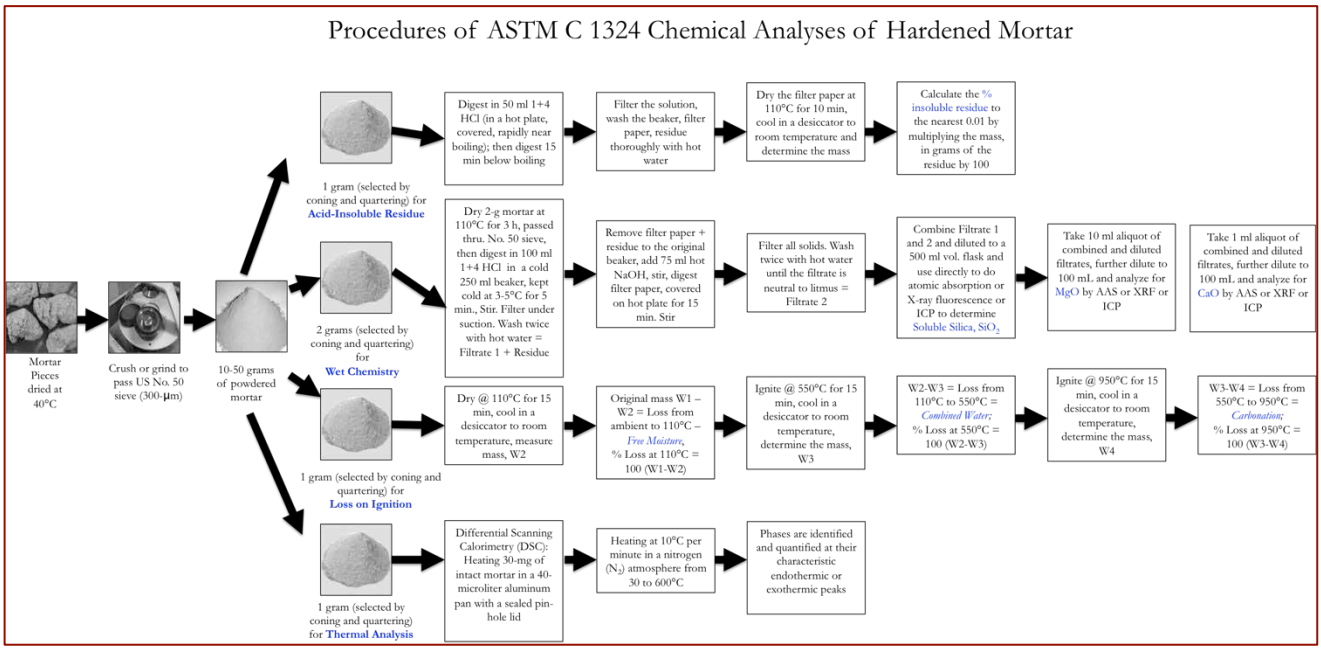

Figure A1-7: Steps followed during various chemical analyses of mortars according to ASTM C 1324.

The hydrochloric acid-insoluble residue content provides the siliceous (non-soluble) content of mortar, which corresponds to the siliceous components of sand. The soluble silica content corresponds to the silica mostly contributed from the binder components (and a minor amount from any soluble silica component in the aggregates). The loss in weight by ignition of a pulverized portion of bulk mortar in a muffle furnace from ambient temperature to 110°C corresponds to the free water content of mortar, whereas, further weight loss from 110°C to 550°C corresponds to the structurally bound hydrate water content, which is proportionate to the amount of hydrated component in the mortar. Finally, the loss of weight by calcination to 950°C corresponds to the degree of carbonation of lime binder of mortar. Oxide compositions determined from wet chemistry, or, other instrumental techniques (e.g., AAS, XRF, ICP) provide compositions of binder, and, bulk mortar.

THERMAL ANALYSES

Thermal analyses (DTA, TGA, and DSC) of mortar are done to determine the presence and amounts of:

a) Hydrates (e.g., detection of brucite by its decomposition at 300-400ºC to check the presence of dolomitic lime, or from soluble magnesium in the paste from use of natural cement),
b) Sulfates (gypsum from decompositions at 125ºC, and 185-200ºC, ettringite at 120-130ºC, thaumasite at 150ºC),
c) Hydrate water, e.g., calcium silicate hydrate from decomposition at 180-190ºC, Portlandite from decomposition at 400-600ºC,
d) High-temperature transformations of silica polymorphs (α to β form) at 573ºC,
e) Cryptocrystalline calcite in the carbonated lime matrix from decomposition at 620-690ºC, or
f) Coarsely crystalline calcite e.g., in limestone by decomposition at 680-800ºC or
g) Dolomite at 740-800ºC and 925ºC, etc.
h) Phases are determined from their characteristic decomposition temperatures occurring mostly as endothermic peaks.

Figure A1-8 shows the four main steps followed during laboratory investigation of masonry mortars, e.g.,

a) From preliminary visual examinations to petrographic examinations of mortars to determine the types of aggregates used and the binders present, based on which
b) Subsequent chemical analyses were done to determine the chemical compositions of binders and proportions of sand, water, and degree of carbonation. Information obtained from petrographic examinations is useful and form the very guidelines to devise the appropriate chemical methods to follow, and to properly interpret the results of chemical analyses.
c) For example, detection of siliceous versus calcareous versus argillaceous natures of aggregates in mortar, or the presence of any pozzolan in the binder (slag, fly ash, ceramic dusts, etc.) from petrography restricts which chemical method to follow, and how to interpret the results of such analyses, e.g., acid-insoluble residue contents.

d) Therefore, a direct chemical analysis e.g., acid digestion of a mortar without doing a prior petrographic examination to determine the types of aggregates and binder used could lead to highly erroneous results and interpretation.

e) Armed with petrographic and chemical data, and based on assumed compositions and bulk densities of the sand and the binder(s) similar to the ones detected from petrographic examinations volumetric proportions of sand and various binders present in the examined mortar can be calculated.

f) The estimated mix proportions from such calculations can provide at least a rough guideline to use as a starting mix during formulation of a tuck pointing mortar to match with the existing (examined) mortar.

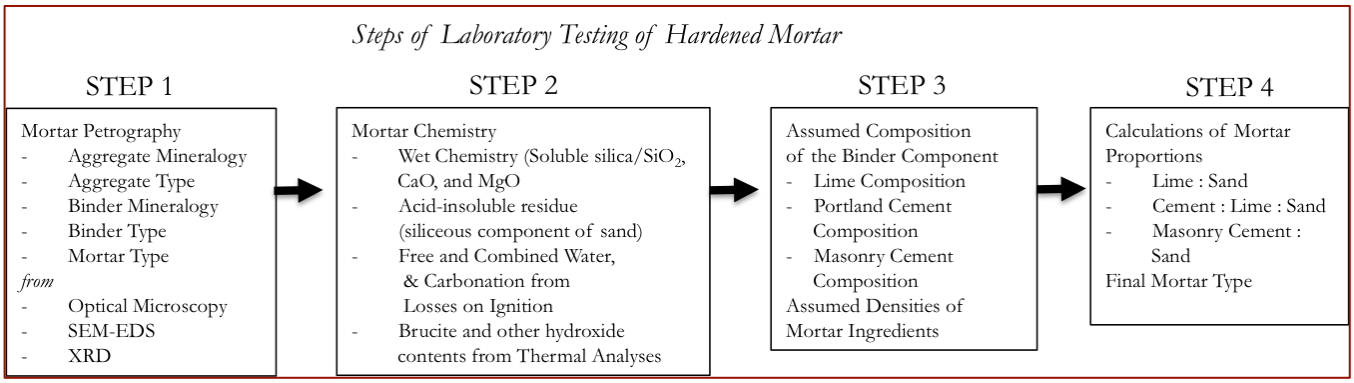

Figure A1-8: Steps followed during laboratory investigation of mortar.

APPENDIX A2 – SUGGESTIONS FOR TUCK-POINTING MORTAR

SUGGESTIONS ON FORMULATION OF TUCK-POINTING MORTARS

The following two Tables provide various tuck pointing mortar formulations many of which are commonly suggested for historic as well as modern masonry renovation projects where the choice depends on: (a) the type of the masonry units present, (b) the exposure condition during service, and (c) the type of the original mortar present. The following suggestions from various references are for general guideline purposes only and provide no guarantee to the overall match in appearance and properties to the existing mortars, which must be determined by trial and error by the project architect/engineer.

Masonry Units	Mortar Type		
	Sheltered	Moderate	Severe
Very hard and durable (e.g., granite, hard-cored brick, etc.)	O (1-2-9)	N (1-1-6)	S (1-0.5-4.5)
Moderately hard and durable (e.g., limestone, durable stone, molded brick)	K (1-3-11)	O (1-2-9)	N (1-1-6)
Minimally durable, soft (soft hand-made brick)	L (0-1-3)	K (1-3-11)	O (1-2-9)

Table A2-1: Various possibilities of tuck pointing mortars made using cement, lime, and sand for various masonry units and exposure conditions (Mack and Speweik, 1998), where the mix proportions by volume within parentheses indicate cement-to-lime-to-sand proportions for various formulations. Type 'L' is a straight lime mortar containing no cement.

Location	Mortar Type	
	Recommended	Alternative
Interior	O	K or N
Exterior - Above Grade, Exposed on one side, unlikely to be frozen when saturated, not subject to high wind or other significant lateral load	O	N or K
Exterior – Other than above	N	O

Table A2-2: ASTM C 270 Guide for selection of tuck-pointing mortar. Mix formulations for different suggestions are as follows: Type K: 1 part Portland cement and $2^1/_2$ to 4 parts hydrated lime; Type O: 1 part Portland cement and $2^1/_2$ parts hydrated lime or lime putty; Type N: 1 part Portland cement to over $1^1/_4$ to $2^1/_2$ parts hydrated lime or lime putty. Aggregate ratio of $2^1/_4$ to 3 times sum of volume of cement and lime for all formulations.

Finally, the following section provides some additional information to consider during selection of an appropriate tuck-pointing mortar for a renovation project (many of which may not be applicable for the present project):

a) It is more important for a tuck pointing mortar to be as close in physical, chemical, and mechanical properties to the existing mortar as possible than to conform to the ASTM C 270 specification for cement-lime or masonry/mortar cement mortars for unit masonry, which are for modern mortars to use for

modern structural applications, and not necessarily applicable to renovation of historic lime mortars. As a general rule, tuck-pointing mortar should be of same strength or softer than the original mortar.

b) Aggregate to use in the tuck-pointing mortar should be similar in color, gradation, appearance, mineralogy, and composition to the sand used in the existing mortar. Sand should be clean, free of any debris, unsound, or clay particles. Masonry sands should conform to the grading requirements of ASTM C 144. Avoid using sand that contains appreciable amounts of potentially alkali-silica reactive particles (e.g., strained quartz, quartzite, chert).

c) Binder for tuck-pointing mortar should be as close to the binder of the existing mortar in composition and properties as possible. For historic lime mortars, possible choices of binders are many:

(i) Non-hydraulic high-calcium lime, or magnesian lime, or dolomitic lime (ASTM C 51) either in dry hydrate (hydrated lime) form, or as slurry or putty form;

(ii) Hydraulic lime;

(iii) Natural hydraulic lime (i.e. NHL 2, NHL 3.5, and NHL 5 with increasing strengths; feebly, moderately and eminently hydraulic natural hydraulic limes with increasing hydraulicity and 28-day compressive strengths from >2 to <7 MPa, to >3.5 to <10 MPa, to >5 to <15 MPa, respectively, produced from calcination of impure limestones having up to 10% clay, 11-20% clay, and 21-30% clay, respectively);

(iv) Natural cements (conforming to specifications of ASTM C 10); or,

(v) A combination of these,

(vi) With or without a pozzolan (fly ash, slag, etc. if added strength and durability are needed).

(vii) Portland cement, if used must be added at lesser proportions than lime, having proportions tested to find the best match in properties to the existing mortar.

(viii) For breathability of the masonry wall, least stress to the exiting mortar, accommodation of building movements, and good bond to masonry units, the binder of choice should be durable and similar in properties and performance to the existing binder having a good service record.

d) During applications of modern masonry mortars: (i) a job-mixed cement-lime mortar is commonly preferred by the architects than a masonry cement mortar, due to the better quality control of the former mortar; (ii) a masonry cement mortar is characteristically air-entrained, which may interfere with the bond to the adjacent masonry units, whereas, a non-air-entrained cement-lime mortar provides a better bond to the adjacent masonry units than an air-entrained masonry cement mortar, (iii) air entrainment usually provides better workability and freeze-thaw durability to a mortar, however, as mentioned, it reduces the bond to the adjacent masonry units (depending on air content); (iv) for Portland cement-lime mortars, a Type M or S mortar (i.e. having a higher cement content than lime and hence a higher strength) is preferred for load-bearing applications than a Type N mortar (having a higher lime content than cement, hence provides better workability and water retention than a Type S or M mortar); (v) Portland cement to use in a mortar should conform to the specification of ASTM C 150; hydrated lime should conform to

ASTM C 207; masonry/mortar cement, if used, should conform to ASTM C 91/C 1329; blended hydraulic cement, if used, should conform to ASTM C 595; (vi) relative proportions of Portland cement and lime will control the overall strength, workability, and bond properties of the repointing mortar.

e) Mineral oxides or carbon-based pigments, if used and positively detected in an examined mortar, should be carefully replicated in the tuck pointing process to reproduce the color, texture, and appearance similar to the existing mortar (including the effects of atmospheric weathering on pigments). Dosage of pigment in the tuck-pointing mortars should be estimated from trial mixes of various dosages.

f) If the original mortar contains a polymer component as suspected from microscopy, characterization of polymer could be done by FTIR-spectroscopy.

g) A mortar strong in compressive strength might be desirable for a hard stone (such as granite), whereas a softer, more permeable lime mortar would be preferable for a historic wall of soft brick. Masonry deterioration caused by salt deposition results when the mortar is less permeable than the masonry unit. A strong mortar is still more permeable than hard, dense stone. However, in a wall constructed of soft bricks where the masonry unit itself has a relatively high permeability or vapor transmission rate, a soft, high lime mortar is necessary to retain sufficient permeability; using a strong mortar with a soft brick will result in spalling of bricks.

h) To have an optimum bond of a mortar to the adjacent masonry unit, relative proportions of cementitious materials and lime contents in the mortar should be carefully controlled. Lime provides the necessary workability and water retention, which are important in a mortar when used with a masonry unit of high suction). Therefore, the initial rate of absorption (or suction property) of the adjacent masonry units should also be carefully determined to match with the appropriate lime content in the mortar.

i) The final tuck pointing mortar should match in color and appearance to the existing mortars, and the closest match should be determined by trial and error on small test areas of the masonry wall to be tuck-pointed.

END OF REPORT[1]

[1] The CMC logo is made using a lapped polished section of a 1930's concrete from an underground tunnel in the U.S. Capitol.