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Technological affinities between qinghui and carbonaceous mortars from Baocheng and Yaotai walls at Xianling Mausoleum

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Abstract

The Baocheng and Yaotai walls of Xianling Mausoleum (AD 1527–1541) preserve dark-grey pointing and finishing mortars and pale bedding mortars of uncertain formulation. Microscopic, mineralogical, spectroscopic and thermal analyses compared mortars from Qian Baocheng, Yaotai and Hou Baocheng. All contain a calcitic lime binder; bedding mortars contain more broadly graded aggregate, whereas the darker mortars contain poorly ordered carbonaceous matter. The latter is enclosed within the mortar as dispersed fines, local enrichments and 2–4 mm composite agglomerates, but corresponding carbon signatures were absent from bedding mortars. These observations indicate incorporation before hardening followed by mixing without complete disaggregation, consistent with material differentiation among bedding, pointing and finishing applications. The lime–carbonaceous combination and its selective use in exposed joints and wall surfaces show strong technological affinity with traditional Chinese qinghui-type materials, providing material evidence for Ming-period applications and a basis for compatible repair-material selection.

Keywords: carbon-bearing lime mortar; qinghui; Raman spectroscopy; Ming dynasty; historic masonry; mortar technology

Introduction

Xianling Mausoleum is located in Zhongxiang, Hubei Province, and is the joint mausoleum of the parents of the Jiajing Emperor (Emperor Shizong of the Ming dynasty). Construction began in 1519 and was completed in 1566. The mausoleum comprises more than thirty monumental architectural groups and is the largest imperial mausoleum of the Ming dynasty in China. In 2000, Xianling Mausoleum was inscribed on the World Heritage List as an important component of the Imperial Tombs of the Ming and Qing Dynasties.^{1,2} The construction of the mausoleum walls was closely associated with the site's transformation from a princely tomb into an imperial mausoleum and with later expansion for the joint burial. After several phases of alteration and enlargement, the complex developed into a layout with one mausoleum and two burial mounds, with Qian Baocheng and Hou Baocheng connected by Yaotai.^{3,4} The brick masonry preserves not only the formal characteristics of the mausoleum's construction but also mortars that record information on material selection, joint treatment and surface finishing, providing a material basis for understanding the building technology of Xianling Mausoleum.

The walls of the core burial precinct comprise three sections: Qian Baocheng, Hou Baocheng and Yaotai. Field investigation showed that grey to dark-grey pointing mortars survive in some brick joints, while local areas of Hou Baocheng retain a dark-grey finishing mortar; both contrast markedly with the pale bedding mortars in the three wall sections. This contrast

suggests that mortars used in different locations may have involved different material selections or preparation methods. Because the darker pointing and finishing mortars were applied directly to wall joints and surfaces, their composition and colour provide important clues to the construction characteristics of exposed mortars. Accordingly, building on macroscopic observations, this study further examines the nature and internal occurrence of the dark components and discusses the relationships among material composition, structure and architectural function.

The appearance and wall application of the darker Xianling mortars closely resemble *qinghui* (青灰) in traditional Chinese construction practice. Volume 13 of the Song-dynasty *Yingzao Fashi*, in the section on mud work, records *he qinghui* (合青灰), a mixture of lime and *ruan shitan* (软石炭, literally “soft coal”); when *ruan shitan* was unavailable, *cu mo* (粗墨, literally “coarse ink”) or *mo mei* (墨煤) could be used instead (*Yingzao Fashi*, Vol. 1, p. 61).⁵ These records indicate that *qinghui* referred not only to colour but also to a specific combination of raw materials and a particular construction context. Recent studies of traditional Chinese lime craftsmanship have further treated raw-material selection, preparation and application as interconnected technological processes, providing a framework for understanding traditional mortars in terms of both materials and operations.^{6,7} Documentary and material studies also show that traditional Chinese lime mortars included diverse organic–inorganic composite formulations used in city walls, tombs, surface finishes and other architectural contexts, while a range of chemical and instrumental methods has been developed to identify additives in such mortars.^{8–11} For the darker Xianling mortars, evaluating their technological relationship with *qinghui* therefore requires comparison between documentary formulation records and the dark components, lime matrix and particle organisation observed in the material itself, interpreted in relation to specific application locations.

This study focuses on the darker pointing mortars from Qian Baocheng, Hou Baocheng and Yaotai and the darker finishing mortar from Hou Baocheng, with pale bedding mortars from the same wall sections used as comparative materials. Microscopic and mineralogical analyses, Raman spectroscopy and mapping, and thermal analysis were combined to identify the principal components of the darker mortars and characterise the structural features and spatial distribution of their carbonaceous component. On this basis, evidence across multiple material scales was integrated with architectural-use information to discuss the formulation characteristics of the darker mortars and their technological significance in pointing and finishing applications. These results were further compared with historical records of the raw materials, preparation and uses of traditional *qinghui*, establishing an evidential link from material identification to technological interpretation and providing material evidence for understanding specific applications of traditional *qinghui* and for future research on conservation materials.

Methods

Sample collection and preparation

Samples were collected from three wall sections of Xianling Mausoleum: Qian Baocheng, Yaotai and Hou Baocheng (Fig. 1). Samples were coded according to wall section and function on the basis of their structural position and field appearance: Q denotes Qian Baocheng, Y denotes Yaotai, H denotes Hou Baocheng, G denotes pointing mortar, M denotes finishing mortar and Z denotes bedding mortar. Sampling preferentially targeted apparently original material that was well preserved and showed no obvious evidence of later repair, while areas with visible salt efflorescence or biological growth were avoided. Detailed sampling information is provided in Table 1.

Before laboratory analysis, loose surface accretions were removed mechanically. The samples were then vacuum-dried at 50 °C to constant mass and stored in a desiccator. Depending on the analytical requirement, samples were prepared as bulk fragments, polished cross-sections, thin sections, powders or acid-insoluble residues. The analytical methods applied to each of the seven mortar samples are summarised in Supplementary Table S5. During sample preparation, 2–4 mm black agglomerates were observed in the pointing and finishing mortars; these were isolated and sectioned as auxiliary specimens for the carbon-bearing mortars.

For acid treatment, three independent replicate experiments were performed for each sample. In each replicate, 0.500 g of dry mortar was treated with excess 1 mol L⁻¹ HCl until visible effervescence ceased. The insoluble fraction was repeatedly rinsed with deionised water to neutrality, vacuum-dried at 50 °C to constant mass and weighed. After drying and weighing, the residue was further separated into four particle-size fractions (>230, 150–230, 75–150 and <75 µm), each of which was weighed separately. The acid-insoluble residue yield was calculated relative to the initial dry mortar mass, whereas the mass fraction of each particle-size class was calculated relative to the total acid-insoluble residue mass. Results are reported as mean ± SD from three independent replicates. Representative stages of acid treatment and the morphology of the recovered acid-insoluble residues are shown in Supplementary Figs. S1 and S2.

Fig. 1. Study area and sampling context of the Xianling wall mortars.

(a) Location of the core mausoleum walls within Xianling Mausoleum; (b) spatial relationship among Qian Baocheng, Yaotai and Hou Baocheng; (c) field occurrence of mortars with different functions in the three wall sections; and (d) schematic positions of bedding, pointing and finishing mortars within the brick masonry. Q, Y and H denote Qian Baocheng, Yaotai and Hou Baocheng, respectively; G, M and Z denote pointing, finishing and bedding mortars, respectively.

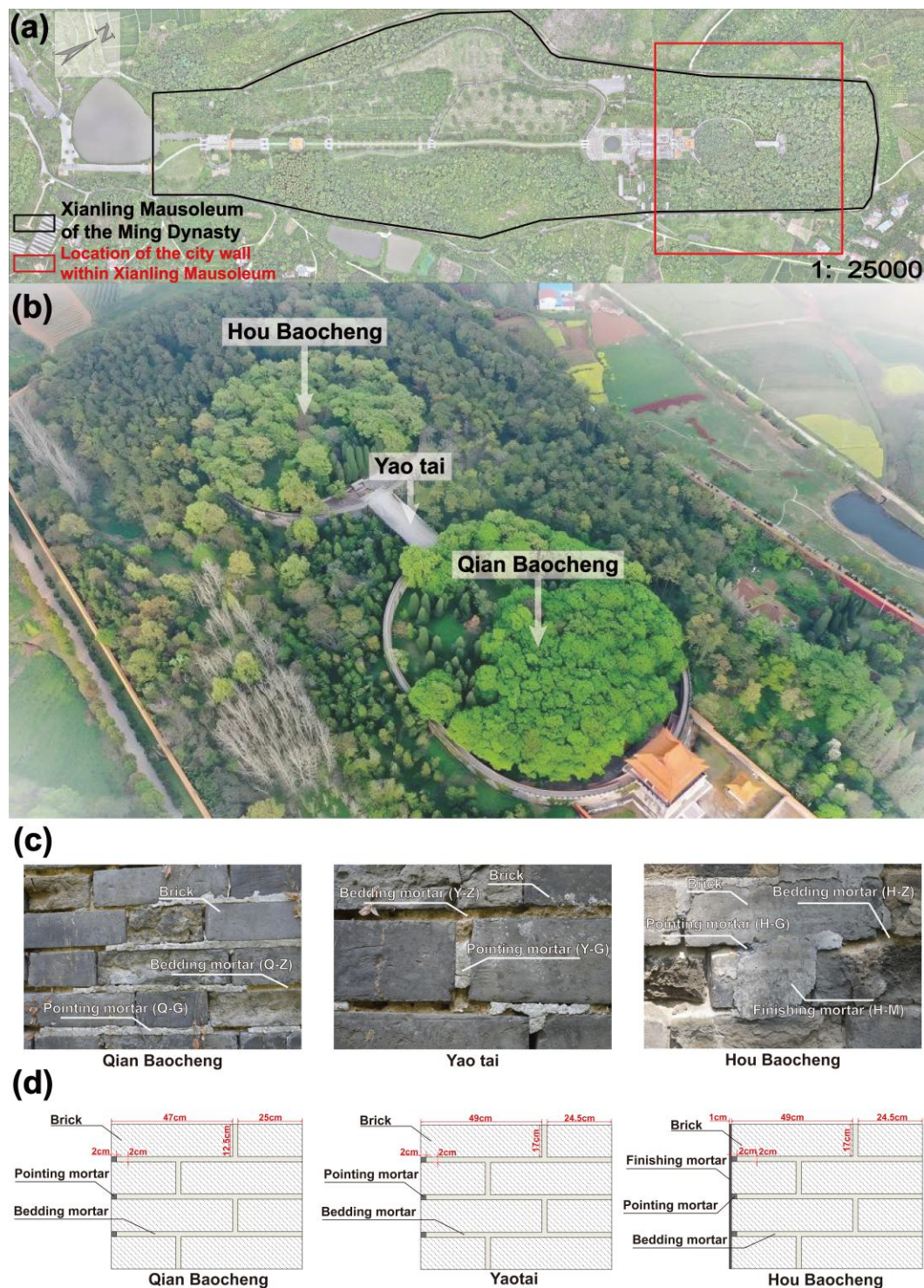


Table 1. Sampling information and macroscopic characteristics of the Xianling wall mortar samples.

Sample	Wall section	Function	Sampling context	Construction date	Colour
Q – G	Qian Baocheng	Pointing	1.3 m above ground; approximately 3.2 cm from exposed face	AD 1527 – 1529	Dark grey
Y – G	Yaotai	Pointing	1.7 m above ground; approximately 3.5 cm from exposed face	AD 1539 – 1541	Grey – black
H – G	Hou Baocheng	Pointing	1.4 m above ground; approximately 3.5 cm from exposed face	AD 1539 – 1541	Grey – black
H – M	Hou Baocheng	Finishing	1.3 m above ground	AD 1539 – 1541	Grey – black
Q – Z	Qian Baocheng	Bedding	1.3 m above ground; approximately 1.5 cm from exposed face	AD 1527 – 1529	Pale yellow – white
Y – Z	Yaotai	Bedding	1.5 m above ground; approximately 2.0 cm from exposed face	AD 1539 – 1541	Pale yellow – white
H – Z	Hou Baocheng	Bedding	1.6 m above ground; approximately 1.5 cm from exposed face	AD 1539 – 1541	Pale yellow – white

Note: Q, Y and H denote Qian Baocheng, Yaotai and Hou Baocheng, respectively; G, M and Z denote pointing, finishing and bedding mortars, respectively.

Optical microscopy and petrography

Polished mortar cross-sections were examined using a Zeiss SmartZoom 5 digital microscope. One thin section was prepared from each mortar sample and examined petrographically using a Zeiss Axio Scope.A1 polarising microscope. Observations focused on the lime matrix, aggregate types and distributions, pores and cracks, and the positions of dark particles and agglomerates relative to the exposed surface and surrounding mortar matrix.

X-ray diffraction and Fourier-transform infrared spectroscopy analysis (XRD and FTIR)

Before X-ray diffraction analysis, bulk samples were ground in an agate mortar and passed through a 75 µm sieve. XRD was performed using a Rigaku SmartLab diffractometer with Cu K α radiation over 5–55° 2 θ at 20 kV and 20 mA, with a step size of 0.02° and a scan rate of 4° min⁻¹. Crystalline phases were identified using Jade 9.0 software and the ICDD PDF-4 2009 database.

For Fourier-transform infrared spectroscopy, approximately 1 mg of sample powder was mixed with 100 mg of dry KBr and pressed into a pellet. Spectra were acquired using a Bruker ALPHA II spectrometer over 500–4000 cm⁻¹ at a resolution of 4 cm⁻¹ with 64 scans. After subtraction of a blank KBr spectrum, baseline correction was performed in OMNIC.

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDS)

Acid-insoluble residues were examined by backscattered-electron imaging and elemental mapping using a TESCAN VEGA3 scanning electron microscope. Samples were mounted on copper double-sided adhesive without carbon tape or other carbon-bearing mounting materials and were gold-coated for 3 s. Analyses were performed at an accelerating voltage of 15 kV, a beam current of 2 nA and a working distance of approximately 17–18 mm. Background correction for elemental maps was performed automatically in AZtecFlex.

Micro-Raman spectroscopy and spectral processing

Micro-Raman spectra were acquired using a Renishaw inVia Qontor Raman microscope with a 785 nm laser and a 50× objective. The laser output power was 50 mW, and the instrument power setting was 1%. Single-point spectra were acquired with a 10 s integration time and one accumulation. Where required, wide-range spectra extending to approximately 3200 cm^{-1} were collected for screening and mineral identification; first-order carbon spectra were evaluated primarily over 1000–2000 cm^{-1} . The Raman shift was calibrated against the silicon band at 520.7 cm^{-1} , and cosmic-ray spikes were removed and baselines corrected in WiRE.

Five representative carbon-positive spectra were selected from each darker pointing or finishing mortar for sample-level descriptive statistics. After cosmic-ray removal and baseline correction, the experimental-spectrum maxima within 1200–1500 cm^{-1} and 1500–1700 cm^{-1} were recorded as the D-band maximum and G-band maximum, respectively. The D- and G-region integrated areas were also calculated over the same fixed windows, and their ratio was expressed as A_D/A_G . These descriptors were obtained directly from the baseline-corrected experimental spectra and were not calculated from fitted peak parameters.

To resolve overlapping bands in the first-order carbon envelope, Gaussian D+G or D+D3+G models were fitted according to spectral complexity. Fitting parameters were not retained when model parameters reached imposed boundaries or when the fit was unstable. Peak fitting was used only for band resolution; the D-band maximum, G-band maximum and fixed-window A_D/A_G were all obtained directly from the baseline-corrected experimental spectra and were independent of the fitting model.

Raman mapping and data processing

Raman mapping was performed on multiple independent cross-sectional regions of Q–G, Y–G, H–G and H–M using the same 785 nm laser, 50× objective and 1% power setting. For each mortar, one 10 × 10 grid was mapped at a 25 μm step size together with several smaller auxiliary regions. Each mapping spectrum was acquired with a 10 s integration time and one accumulation. For each valid spectrum, the median intensities over 1000–1100 and 1900–2000 cm^{-1} were calculated and used to define a linear baseline. Trapezoidal integration was

then used to calculate the integrated areas of the D region (1200–1500 cm⁻¹), G region (1500–1700 cm⁻¹) and combined D+G region (1200–1700 cm⁻¹).

To distinguish weak carbon-related signals from random noise, a dual-band D/G signal-to-noise ratio (SNR) criterion was introduced. The standard deviations of the baseline-corrected signals over 1000–1100 and 1900–2000 cm⁻¹ were calculated, and the larger value was used as the noise estimate, σ_{noise} . The D- and G-region signal intensities were defined as the maximum baseline-corrected intensities within 1280–1450 and 1560–1650 cm⁻¹, respectively:

$$SNR_D = \frac{I_{D,\text{max}}}{\sigma_{\text{noise}}}$$

$$SNR_G = \frac{I_{G,\text{max}}}{\sigma_{\text{noise}}}$$

A mapped pixel was classified as carbon-positive only when $SNR_D \geq 3$ and $SNR_G \geq 3$. All data were reconstructed into spatial matrices using the original acquisition coordinates. Grid dimensions, step sizes, numbers of acquired spectra, quality-control results and dual-band SNR-positive fractions for each mapped region are provided in Supplementary Table S3.

Thermogravimetric analysis–differential scanning calorimetry

TG–DSC measurements were performed using a NETZSCH STA 449 F3 Jupiter simultaneous thermal analyser. Nitrogen and air measurements were carried out independently on separate small aliquots of homogenised powder from the same mortar sample. Approximately 10 mg of homogenised mortar powder with a particle size <75 μm was used for each measurement. Samples were placed in alumina crucibles and heated from room temperature to 1000 °C at 10 °C min⁻¹ under a gas flow of 50 mL min⁻¹. Mass changes were calculated over 25–200, 200–500, 500–800 and 800–1000 °C, together with the total mass loss over 25–1000 °C.

Results

Microstructural characteristics

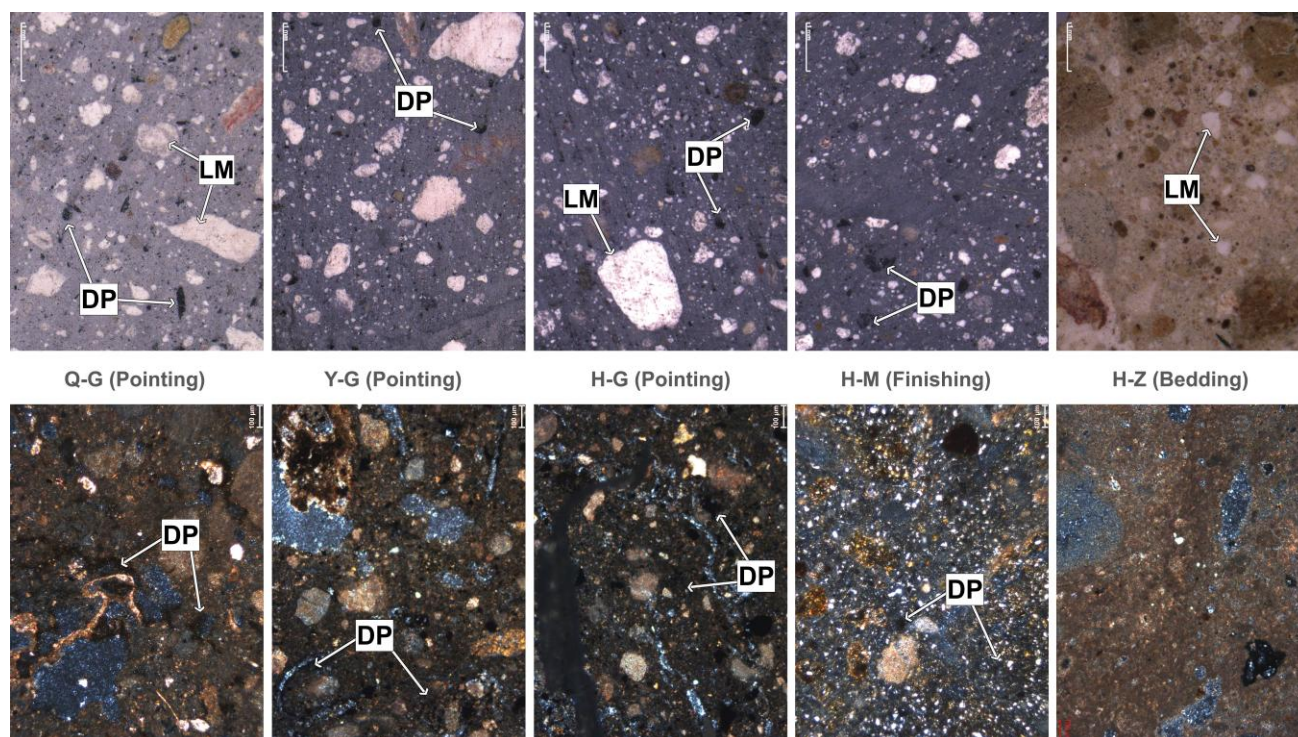
Optical microscopy of polished cross-sections showed that the pointing and finishing mortars were generally dark grey and contained conspicuous white and black particles (Fig. 2). Petrographic thin-section observations showed that these mortars were dominated by a calcitic lime matrix containing small amounts of quartz, feldspar, mica, lithic fragments and irregular pores. The detrital grains were mainly subangular to subrounded and approximately 0.2–1.5 mm in size. The matrix consisted chiefly of randomly distributed microcrystalline

calcite ($\sim 0.003\text{--}0.01$ mm) and micritic calcite (<0.003 mm), with predominantly subangular forms. Abundant dark opaque particles were also present in the thin sections as fine particles, irregular grains, short flakes and local agglomerates, all fully enclosed within the mortar matrix.

The bedding mortars appeared pale yellow under the microscope. Thin-section observations likewise showed a matrix dominated by calcite, with aggregates consisting mainly of $0.03\text{--}0.1$ mm quartz grains that were predominantly subangular to subrounded; no conspicuous dark particles were observed. Complete polished cross-sections, acid-insoluble residues and representative black agglomerates, together with petrographic thin-section images of all seven samples, are provided in Supplementary Figs. S2 and S3.

Fig. 2. Representative polished cross-sections and thin-section micrographs of the mortar samples.

The upper row shows polished cross-sections and the lower row the corresponding thin sections; from left to right: Q–G, Y–G, H–G, H–M and H–Z. Dispersed dark particles are visible in the darker pointing and finishing mortars, whereas H–Z represents a pale bedding-mortar comparison. DP, dark particle; LM, lime matrix.



Acid-insoluble residue yield and particle-size distribution

The acid-insoluble residue yields of the pointing and finishing mortars were relatively similar: $18.14 \pm 1.45\%$ for Q–G, $11.67 \pm 0.59\%$ for Y–G, $10.90 \pm 0.91\%$ for H–G and $11.15 \pm 0.60\%$ for H–M; Q–G nevertheless differed to some extent from the other three darker mortars. Abundant black particles and smaller amounts of orange-red particles were visible in the acid-insoluble residues of all pointing and finishing mortars. Particle-size separation showed that

the <75 μm fraction accounted for 56.50–80.05% of the acid-insoluble residues in these mortars, whereas the >230 μm fraction accounted for only 0.99–7.78%; black particles occurred mainly in the <75 μm fraction. Weighing data from the three independent replicates and the corresponding residue-yield statistics are provided in Supplementary Table S1.

The bedding mortars had higher acid-insoluble residue yields than the darker pointing and finishing mortars, with some differences among Qian Baocheng, Hou Baocheng and Yaotai. Their acid-insoluble residues were pale yellow, with yields of 37.05–44.17%, and their particle-size distributions extended across several size classes and were broader than those of the pointing and finishing mortars. Acid-insoluble residue yields and particle-size fractions for all samples are listed in Table 2.

Table 2. Acid-insoluble residue yields and particle-size distributions of the mortar samples.

Acid-insoluble residue yields were calculated relative to the initial dry mortar mass, whereas particle-size fractions were calculated relative to the total mass of the acid-insoluble residue. Values are presented as mean $\pm$ SD from three independent acid-treatment replicates ($n = 3$).

Sample	Acid-insoluble residue yield (%)	Particle-size distribution of acid-insoluble residue (%)			
		>230 μm	150–230 μm	75–150 μm	<75 μm
Q – G	18.14 $\pm$ 1.45	7.78 $\pm$ 0.45	17.95 $\pm$ 1.78	17.76 $\pm$ 1.08	56.50 $\pm$ 2.99
Y – G	11.67 $\pm$ 0.59	2.39 $\pm$ 0.18	9.27 $\pm$ 0.52	8.28 $\pm$ 3.00	80.05 $\pm$ 3.47
H – G	10.90 $\pm$ 0.91	2.55 $\pm$ 0.89	9.22 $\pm$ 0.86	8.34 $\pm$ 0.85	79.90 $\pm$ 0.48
H – M	11.15 $\pm$ 0.60	0.99 $\pm$ 0.90	13.35 $\pm$ 1.13	8.78 $\pm$ 3.87	76.84 $\pm$ 3.70
Q – Z	37.05 $\pm$ 0.97	4.07 $\pm$ 0.92	54.77 $\pm$ 0.44	13.33 $\pm$ 0.70	27.83 $\pm$ 0.99
Y – Z	41.29 $\pm$ 0.95	3.34 $\pm$ 0.86	26.42 $\pm$ 1.56	34.22 $\pm$ 1.36	36.02 $\pm$ 1.39
H – Z	44.17 $\pm$ 0.55	3.54 $\pm$ 0.87	28.54 $\pm$ 2.50	34.50 $\pm$ 2.68	33.42 $\pm$ 1.13

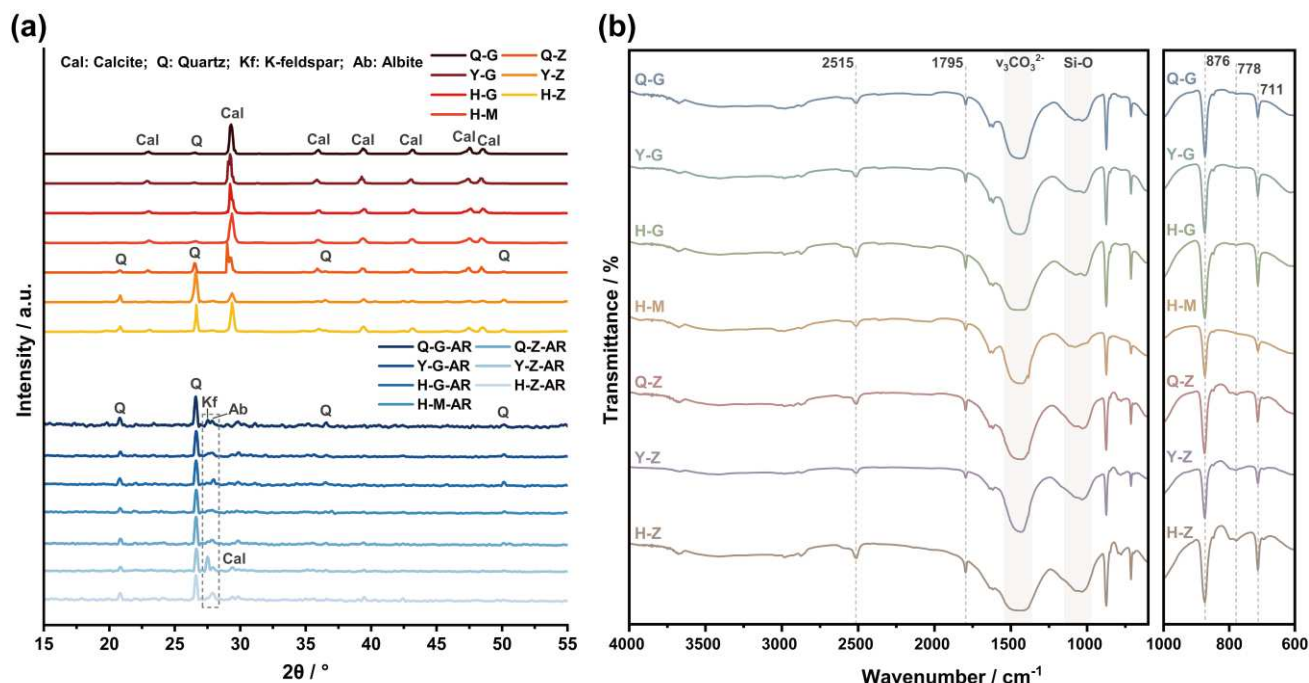
Mineralogical composition

XRD showed that all Xianling wall mortars were dominated by carbonate phases (Fig. 3). Calcite was the principal crystalline phase, with quartz and minor silicate minerals as secondary components; quartz diffraction peaks were more prominent in the bedding mortars. The acid-insoluble residues were dominated by quartz and feldspar.

In the FTIR spectra, carbonate ν_3 , ν_2 and ν_4 vibrations occurred at 1433–1453, 874–876 and 711–713 cm^{-1} , respectively. Absorptions at 1016–1080 cm^{-1} were associated with silicate components, and a weak quartz-related feature occurred near 775–779 cm^{-1} . Broad bands near 3414–3420 and 1616–1620 cm^{-1} were attributed to adsorbed water or moisture uptake by KBr. No infrared features attributable to organic matter were observed.

Fig. 3. XRD and FTIR characterisation of the Xianling mortar samples.

(a) XRD patterns of the bulk mortars and corresponding acid-insoluble residues; (b) FTIR spectra of the bulk mortar samples. The inset in (b) enlarges the 1000–600 cm^{-1} region. Spectra are vertically offset for clarity.



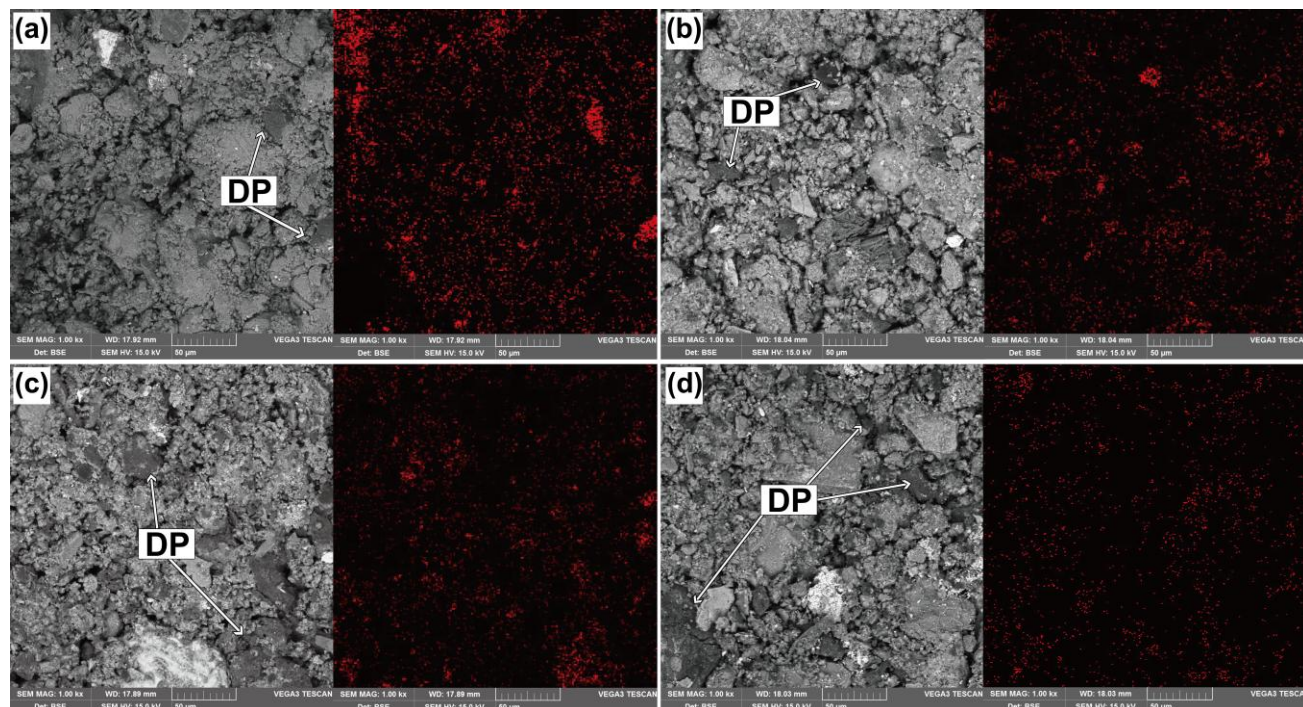
SEM–EDS characterisation of acid-insoluble residues

The acid-insoluble residues of the pointing and finishing mortars were distinctly heterogeneous and consisted of irregular particles of varying size, including local platy and fibrous particles and fine grains with contrasting backscattered-electron intensity. Some fine particles adhered to the surfaces of larger grains or formed loose aggregates. Differences in BSE intensity among particles indicate components with different mean atomic numbers; both high- and low-BSE-intensity components were heterogeneously distributed.

EDS mapping showed a heterogeneous spatial distribution of C in the acid-insoluble residues, with local enrichment in low-BSE-intensity regions (Fig. 4), indicating carbon-bearing microregions within the solids remaining after acid treatment.

Fig. 4. BSE images and corresponding EDS carbon maps of acid-insoluble residues from the four darker mortars.

(a) Q–G; (b) Y–G; (c) H–G; and (d) H–M. In each panel, the BSE image is shown on the left and the corresponding carbon elemental map on the right.



Raman identification and spatial distribution of carbonaceous matter

Broad D- and G-band responses were consistently detected from visually discernible black particles in the pointing and finishing mortars. D- and G-band features were also detected in regions without obvious black particles and in black particles isolated from the corresponding acid-insoluble residues (Fig. 5a). Across these measurement types, the D and G bands were broadened and variably overlapped, consistent with Raman responses of poorly ordered carbonaceous matter.^{12,13} These results show that the carbonaceous component was not confined to macroscopically visible black particles or agglomerates but also occurred as fine or dispersed material within the mortar matrix; corresponding D- and G-band carbon signatures were not observed in the bedding mortars (Supplementary Fig. S4).

Descriptors were extracted from the baseline-corrected experimental spectra. The D-band maxima ranged from 1316.49 to 1345.97 cm^{-1} and the G-band maxima from 1595.63 to 1601.69 cm^{-1} . Although the positions varied among samples, the overall ranges largely overlapped (Table 3). Fixed-window integration of the D and G regions yielded A_D/A_G values of 1.44–2.82, with Q–G showing the relatively lowest ratio. This ratio represents only the relative integrated signal contributions of the D and G regions under the present acquisition and processing conditions and was used to compare first-order carbon band-shape characteristics among samples. Peak fitting of representative spectra showed that the first-

order carbon envelope comprised overlapping broadened components (Fig. 5b). Individual fits and residuals for 20 representative carbon-positive spectra are shown in Supplementary Fig. S5; corresponding experimental D- and G-band maxima, fixed-window A_D/A_G ratios and retained peak-fitting parameters are listed in Supplementary Table S2.

Millimetre-scale black agglomerates were observed in all pointing and finishing mortars. These agglomerates had black matrices containing small amounts of red and white particles (Fig. 5c). Multiple measurements from the black matrices showed broadened D and G bands and were identified as poorly ordered carbonaceous matter; the red particles showed Raman features consistent with hematite, and the white particles with calcite.¹⁴ The co-occurrence of carbonaceous matter, hematite and calcite within individual agglomerates indicates that these millimetre-scale features were not compositionally uniform carbon particles but composite constituents comprising a carbonaceous matrix and mineral particles. In addition to H–G-A1 shown in Fig. 5, cross-sectional morphologies, Raman measurement positions and representative spectra for Q–G-A1, Y–G-A1 and H–M-A1 are provided in Supplementary Fig. S6; complete point identifications for carbonaceous matter, calcite and hematite in all four representative black agglomerates are summarised in Supplementary Table S4.

Raman mapping was performed over multiple regions of the pointing and finishing mortar cross-sections. Every mapped region contained carbon-positive pixels satisfying $SNR \geq 3$ for both the D and G bands, demonstrating reproducible carbonaceous responses across the four mortars and among independent mapped regions (Fig. 6). The carbon-related signals were spatially heterogeneous: some regions contained locally high-response particles or enriched microdomains, whereas others showed lower-intensity dispersed responses, and areas that did not meet the carbon-detection criterion were also present within the same mapped field. Differences in the number and spatial distribution of carbon-positive pixels among mapped regions show that the carbonaceous component was not uniformly distributed at the micrometre scale. Complete multi-field D+G integrated-area maps and fixed-window A_D/A_G ratio maps for Q–G, Y–G, H–G and H–M are provided in Supplementary Figs. S7–S10; dual-band SNR classifications and carbon-positive fractions for each mapped field are reported in Supplementary Table S3.

Fig. 5. Raman characterisation of carbonaceous matter and black agglomerates in the darker mortars.

(a) Representative first-order Raman spectra of black particles from Q-G, Y-G, H-G and H-M; (b) D-D3-G peak fitting and residual for a representative first-order carbon Raman spectrum; (c) cross-section of a representative black agglomerate showing Raman measurement regions; and (d) representative Raman spectra of white calcite and orange-red hematite inclusions within the agglomerate.

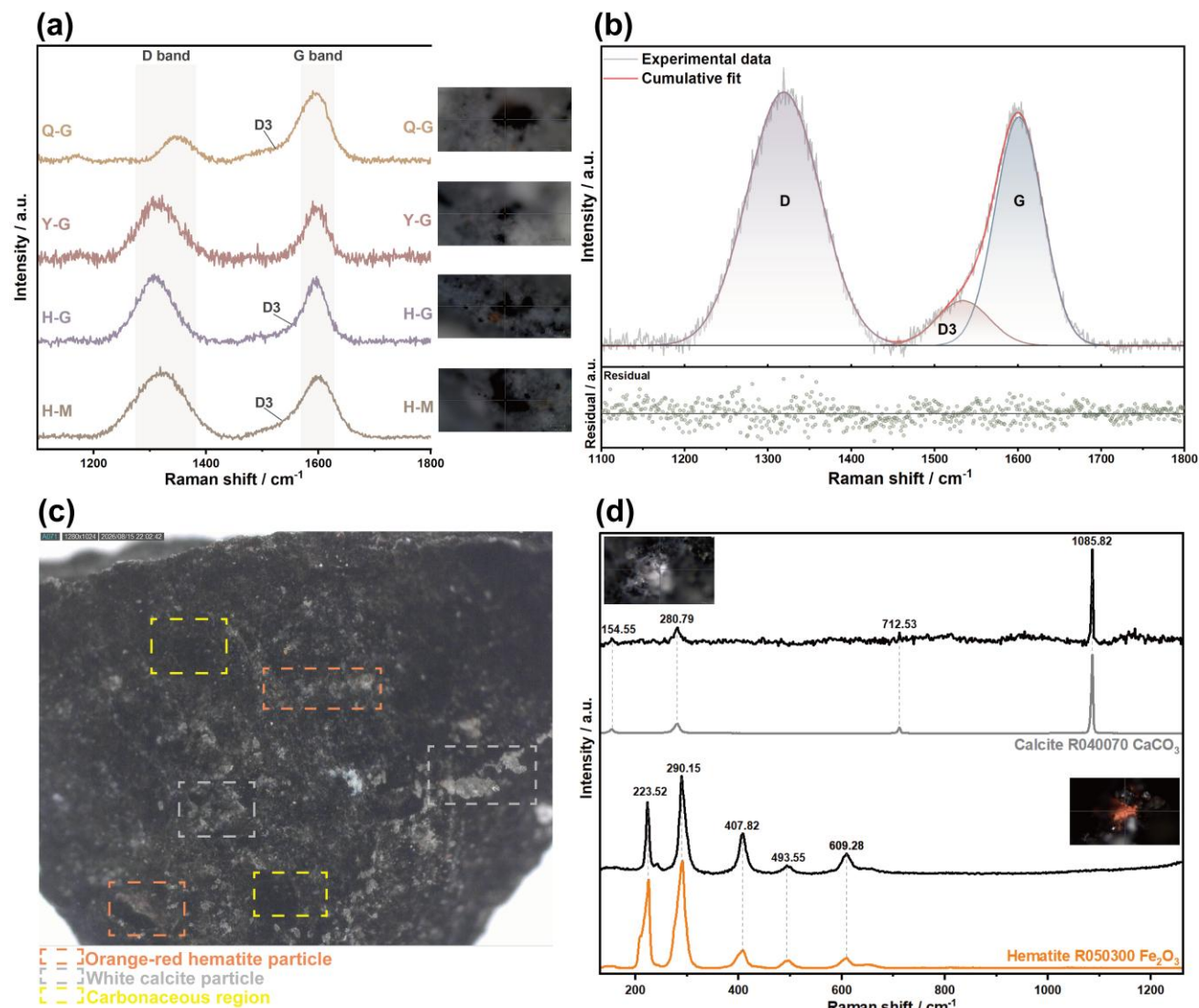
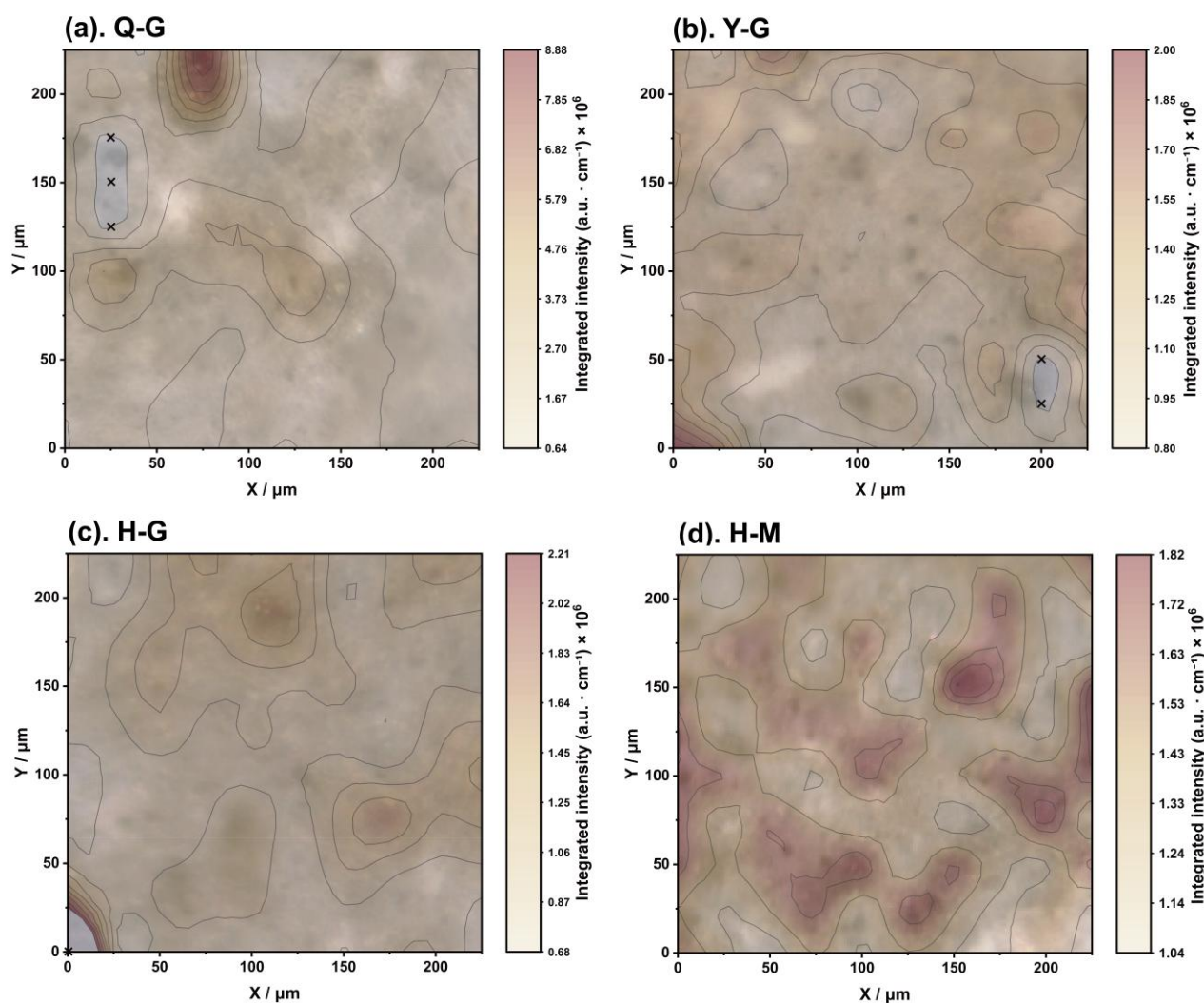


Table 3. First-order Raman spectral descriptors of carbonaceous matter in the darker mortars.
Values are presented as mean $\pm$ SD (n = 5 representative carbon-positive spectra per sample).

Sample	n	D maximum / cm^{-1}	G maximum / cm^{-1}	Fixed-window A_D/A_G
Q - G	5	1345.97 $\pm$ 8.57	1595.63 $\pm$ 5.61	1.44 $\pm$ 0.20
Y - G	5	1322.76 $\pm$ 12.56	1596.07 $\pm$ 3.30	2.82 $\pm$ 0.27
H - G	5	1316.49 $\pm$ 15.05	1597.06 $\pm$ 4.30	2.44 $\pm$ 0.80
H - M	5	1319.48 $\pm$ 2.83	1601.69 $\pm$ 1.78	2.70 $\pm$ 0.71

Fig. 6. Representative Raman maps of carbon-related D+G integrated areas in the four darker mortars.

One representative cross-sectional field is shown for each of Q-G, Y-G, H-G and H-M. D+G integrated areas were calculated over the baseline-corrected 1200–1700 cm^{-1} region and overlaid on the corresponding optical images; missing or invalid pixels are marked with $\times$.



Thermal behaviour under nitrogen and air

TG–DTG–DSC results for the bedding, pointing and finishing mortars, together with mass changes over individual temperature intervals, residual mass at 1000 °C and characteristic temperatures, are summarised in Table 4. All samples showed only minor mass changes from the initial temperature to 200 °C, with most mass change occurring between 200 and 800 °C.

Under nitrogen, the pointing and finishing mortars showed total mass losses of 14.15–44.20% and residual masses at 1000 °C of 55.80–85.85%. The principal mass-change interval was 500–800 °C, with losses of 13.09–27.22% and corresponding main DTG minima at 515.44–577.43 °C. Under air, total mass loss increased to 47.16–66.13%, while the residual mass at 1000 °C decreased to 33.87–52.84%. Mass change under air was concentrated mainly between 200 and 800 °C; losses over 500–800 °C were 34.29–61.75%, with main DTG minima at 525.47–686.22 °C. Differences between the two atmospheres were most pronounced for the Yaotai and Hou Baocheng pointing mortars and the Hou Baocheng finishing mortar, whereas Q–G showed a smaller change. Under the same test conditions, the bedding mortars showed no marked difference in total mass loss between nitrogen and air. Complete TG–DTG–DSC curves for all seven mortars under nitrogen and air are provided in Supplementary Figs. S11 and S12.

Table 4. Temperature-resolved mass changes, total mass loss, and characteristic DTG and DSC temperatures of the Xianling mortar samples under nitrogen and air.

Δm denotes the mass change within the corresponding temperature interval relative to the initial sample mass; positive values indicate mass loss and negative values indicate slight mass gain. Total loss is the net mass loss over 25–1000 °C, R_{1000} is the residual mass percentage at 1000 °C, $T_{DTG,max}$ is the temperature corresponding to the maximum mass-loss rate during the principal mass-loss stage, and $T_{DSC,min}$ is the characteristic DSC minimum temperature.

Sample	Atmosphere	Δm_{25-200} (%)	$\Delta m_{200-500}$ (%)	$\Delta m_{500-800}$ (%)	$\Delta m_{800-1000}$ (%)	Total loss (%)	R_{1000} (%)	$T_{DTG,max}$ / °C	$T_{DSC,min}$ / °C
Q – G	N ₂	–0.02	16.83	27.22	0.17	44.20	55.80	515.44	519.97
Q – G	Air	0.67	1.79	44.73	–0.04	47.16	52.84	604.27	588.27
Y – G	N ₂	1.22	8.75	24.54	0.55	35.07	64.93	541.74	548.69
Y – G	Air	1.45	19.84	34.29	0.04	55.62	44.38	525.47	524.91
H – G	N ₂	0.21	1.26	26.74	–0.05	28.16	71.84	577.43	594.31
H – G	Air	1.96	2.12	52.29	1.73	58.10	41.90	686.22	594.31
H – M	N ₂	0.17	0.90	13.09	0.00	14.15	85.85	564.36	553.77
H – M	Air	0.77	1.31	61.75	2.29	66.13	33.87	618.42	687.91
Q – Z	N ₂	2.05	22.98	42.99	0.00	68.02	31.98	524.09	533.13
Q – Z	Air	0.40	52.36	16.31	–0.61	68.46	31.54	485.62	476.60
Y – Z	N ₂	0.89	3.32	55.27	0.26	59.74	40.26	596.16	566.49
Y – Z	Air	1.01	12.85	46.11	–0.03	59.94	40.06	580.32	533.80
H – Z	N ₂	0.42	0.93	49.94	1.46	52.75	47.25	619.57	683.87
H – Z	Air	1.44	2.01	48.66	1.55	53.67	46.33	680.97	593.08

Discussion

Taken together, the petrographic, mineralogical, acid-insoluble-residue, particle-size and Raman results reveal material differences among the Xianling wall mortars that correspond to their construction locations. The darker pointing and finishing mortars are composite lime mortars with a calcitic lime binder, small amounts of fine mineral particles and poorly ordered carbonaceous matter occurring as fine particles, dispersed microdomains and local agglomerates. Their acid-insoluble residues are concentrated mainly in the $<75\ \mu\text{m}$ fraction. By contrast, although the bedding mortars likewise have calcite as the principal binder phase, their acid-insoluble residue yields reach 37.05–44.17% and span a broader particle-size range. They therefore differ markedly from the pointing and finishing mortars in both material composition and carbonaceous components.

These material differences correspond to mortar function. Bedding mortars occur between brick courses and serve to bed, level and connect bricks during masonry construction, whereas pointing and finishing mortars occur in brick joints and on wall surfaces and directly form exposed joints and surface finishes. Against a common lime-based binding system, the exposed mortars selectively incorporate carbonaceous matter, indicating that the Xianling walls were not constructed using a single mortar formulation but instead employed different material configurations for bedding, pointing and finishing applications. Examples from other high-status Ming-period buildings likewise show differentiated mortar use among architectural locations, indicating a contemporary technological context in which mortar formulations were adjusted to building function.^{15,17}

Within this broader functional differentiation, internal variation also occurs among wall sections. The Yaotai and Hou Baocheng mortars show relatively similar compositional characteristics, whereas the Qian Baocheng pointing mortar, although also a dark carbon-bearing lime mortar, differs from the Yaotai–Hou Baocheng samples in its carbonaceous characteristics. The aggregate size and proportion in the Qian Baocheng bedding mortar also differ consistently from those in the Yaotai–Hou Baocheng bedding mortars. Thus, the Xianling wall mortars show not only functional differentiation among bedding, pointing and finishing mortars but also variation among wall sections. In light of the site's historical background and construction sequence, these differences may relate to raw-material sources, construction batches, local practices or construction history.

Poorly ordered carbonaceous matter is the principal material feature distinguishing the darker pointing and finishing mortars from the bedding mortars. Micro-Raman spectroscopy showed broadened D and G bands in all visually recognisable black particles, and corresponding carbonaceous features were also detected in mortar regions without obvious black particles and in black particles from the acid-insoluble residues. SEM–EDS further showed locally

enriched C signals in low-BSE-intensity regions of the acid-insoluble residues. The carbonaceous component therefore occurs within the mortar at several scales, as discernible particles, fine dispersed phases and local enrichments rather than only as a small number of macroscopic black particles.

Raman mapping further reveals the spatial heterogeneity of this carbonaceous component. Multiple independent cross-sectional regions of the darker pointing and finishing mortars contained carbon-positive pixels satisfying both $\text{SNR}_D \geq 3$ and $\text{SNR}_G \geq 3$. Their distributions included locally high-response particles and enriched microdomains, as well as lower-intensity dispersed responses, while areas below the detection criterion also occurred within the same mapped field. This distribution indicates that carbonaceous matter had entered the lime-mortar matrix but did not form a continuous, homogeneous carbonaceous phase.

The internal position of the carbonaceous component further constrains its mode of formation. Microscopy showed dark particles enclosed within the calcitic matrix, while Raman mapping repeatedly detected carbonaceous responses in multiple internal mortar regions, demonstrating that the signals were not confined to exposed surfaces, cracks or connected pores. Their repeated occurrence in different wall sections and samples is also difficult to explain solely by local soot deposition, short-term fire smoke or accidental inclusion. At the same time, 2–4 mm black agglomerates were preserved in all pointing and finishing mortars; their black matrices consist of poorly ordered carbonaceous matter and also contain calcite and minor hematite particles. These composite agglomerates enclosed by the lime matrix indicate that carbonaceous material had entered the mortar system before hardening and was not completely disaggregated during mixing.

The co-occurrence of hematite and carbonaceous matter within the agglomerates also reflects compositional heterogeneity in the associated raw materials. The hematite may derive from iron-bearing minerals carried by the carbonaceous material itself or from incompletely combusted fuel ash; it may also derive from fired clay fines, brick debris or kiln-cleaning residues generated during brick and tile production. Alternatively, iron oxides from sandy soil or other mineral fines may have entered independently during mortar preparation. Because hematite can occur in coal-bearing materials and their combustion residues and can also form or become enriched when iron-bearing clay is fired under oxidising conditions, the present evidence cannot distinguish among these mechanisms. It does, however, indicate that the carbonaceous material used in the Xianling pointing and finishing mortars was compositionally and granulometrically complex rather than a highly purified carbon material of uniform particle size.

This heterogeneity is also expressed in the particle size and spatial distribution of the carbonaceous material. Particle-size separation of the acid-insoluble residues showed that

black particles were concentrated mainly in the $<75\ \mu\text{m}$ fraction, while mortar regions without visually obvious black particles still yielded carbonaceous Raman responses; conversely, millimetre-scale composite agglomerates persisted in different darker mortars. The multiscale organisation—from dispersed fines and local enrichments to millimetre-scale agglomerates—indicates that the carbonaceous raw material underwent crushing, comminution and dispersion in the slurry before or during incorporation into the mortar but was not completely disaggregated. Its distribution therefore cannot be attributed solely to a few coarse particles, but instead reflects relatively extensive mixing between the carbonaceous material and the lime matrix, consistent with incorporation of the carbonaceous material during mortar preparation.

The carbonaceous matter in the different darker mortars shows broadly similar poorly ordered Raman characteristics. The D and G bands are broadened and overlap, and their peak-position ranges largely overlap; fixed-window A_D/A_G ratios range from 1.44 to 2.82, with the Qian Baocheng pointing mortar showing a relatively lower value, indicating some variation in the Raman characteristics of the carbonaceous matter among samples. Thermal analysis provides supplementary information for this interpretation. The pointing and finishing mortars show greater total mass loss in air than under nitrogen, and the direction of the thermal response is consistent with oxidative mass loss of carbonaceous matter in air. However, the temperature ranges of carbonaceous-matter oxidation and CaCO_3 decomposition overlap, and the exothermic effect of carbon oxidation may overlap with the endothermic effect of carbonate decomposition. Accordingly, the absence of an independent exothermic DSC peak is not inconsistent with the presence of carbonaceous matter. Likewise, differences in mass change between air and nitrogen cannot be converted directly into carbon or CaCO_3 contents, although the atmosphere-dependent thermal behaviour of the pointing and finishing mortars provides supporting evidence for the presence of carbonaceous components.¹⁸

Taken together, the evidence indicates that the carbonaceous material in the darker Xianling mortars entered the mortar system before hardening and underwent crushing, mixing and dispersion in the slurry. Because the raw material was not completely disaggregated, the hardened mortar retained a heterogeneous structure in which dispersed fines, locally enriched regions and millimetre-scale composite agglomerates coexist. The present evidence cannot determine whether the carbonaceous material was added during lime slaking, raw-material premixing or final mortar mixing, nor whether it was introduced as a discrete ingredient or as part of a carbon-bearing composite raw material. Its specific identity as a coal-derived, char-derived or soot-like material likewise cannot be established from the present D- and G-band characteristics alone.

In traditional Chinese construction, carbonaceous materials such as charcoal ash, coal, ink and soot were widely combined with lime under different names and with different carbon

sources and construction uses; such materials were often referred to as *qinghui*. The *chuihui* (捶灰) preserved at the Leshan Giant Buddha is one of the earlier material examples of carbon-bearing lime. Some infill bricks retain adhering *chuihui*, and thermoluminescence dating of associated bricks yielded approximately AD 800–900. Traditional *chuihui* was prepared by mixing lime, charcoal ash, chopped hemp fibres and water, repeatedly pounding the mixture, and applying it to the Buddha surface by pounding and plastering. This evidence shows that by around the Tang dynasty carbonaceous material could serve as an important component of lime-based materials used on exposed surfaces.^{19,20}

The Northern Song *Yingzao Fashi* provides a more explicit institutional record. Volume 13, in the section on mud work, specifies *he qinghui* made from lime and *ruan shitan*; when *ruan shitan* was unavailable, *cu mo* or *mo mei* could be substituted. Volume 27 further lists the corresponding material allowances. These records show that combining lime with coal- or ink-type carbonaceous materials had become a construction method with specified raw-material choices and quantities. *He qinghui* was incorporated into systems for wall mud work and lime finishing, indicating that it was not a universal formulation for all lime mortars but was associated with particular surface locations and construction requirements. These characteristics correspond to several features of the Xianling pointing and finishing mortars (*Yingzao Fashi*, Vol. 3, p. 80).⁵

From the Ming dynasty onward, *qinghui* increasingly appears in written sources as the name of a building material listed alongside lime and used in construction and repair works. Texts including the *Da Ming Hui Dian* (大明会典) and *Gongbu Changku Xuzhi* (工部厂库须知) record *qinghui*, but mainly in relation to material procurement, quantities and prices, without specifying the carbonaceous raw materials or preparation procedures involved.^{21,22} Consequently, the actual material composition of Ming-period *qinghui* has long lacked material evidence that can be directly compared with documentary records.

Traditional Chinese building manuals from the Qing dynasty and modern period further indicate that *qinghui*-type materials did not follow a single fixed raw-material formulation. Terms such as *qinghui*, *qinghui* backing layers, *qingjiang* (青浆, a filtered suspension prepared by adding water to *qinghui*, stirring and filtering) and *yanzi jiang* (烟子浆, a soot-containing lime slurry) were used in different construction systems; *yanzi jiang* explicitly used black soot-like material, while modern investigations also record dark lime materials prepared with carbonaceous shale, pine soot or low-rank coal.²³⁻²⁵ These different carbon sources could all be combined with lime to produce grey to dark-grey mortars, suggesting that *qinghui* was better understood as a traditional material category defined jointly by appearance, use and craft practice rather than as a standard formulation with a fixed mineralogical composition. In this period, the traditional Chinese mortar system was summarised as *jiujiang shibahui* (九浆十八灰, literally “nine slurries and eighteen limes”), in which the preparation of several

composite mortars involved *qinghui* or *qingjiang* and was applied across multiple building components. Representative examples include *qinghui* backing layers in roofing systems and *zhuojie hui* (捉节灰) and *jialong hui* (夹垄灰), mortars used to fill joints between roof tiles. These applications illustrate the use of *qinghui*-type materials in roof backing and tile joints, respectively. Similarly, the pointing and finishing mortars of the Xianling brick walls show selective use of dark lime materials in exposed surfaces and joints, reflecting the close relationship between *qinghui*-type materials and specific construction functions.²⁶⁻²⁷

Against this technological background, the darker pointing and finishing mortars of the Xianling walls provide a Ming-period example with clear chronological and architectural-location contexts. Their material and technological characteristics identify them as composite lime mortars containing poorly ordered carbonaceous matter and show strong technological affinity with *he qinghui* recorded in the *Yingzao Fashi*: both use lime as the binder and incorporate carbonaceous components, and both apply carbon-bearing materials to exposed locations such as wall surfaces or joints, in contrast to ordinary lime mortar used within brick masonry. The affinity therefore extends beyond a grey-black appearance to the lime–carbonaceous formulation and the construction logic of differentiating materials according to application location.

A distinctive feature of the Xianling mortars is that carbon-bearing composite lime material was used not only in local finishing on Hou Baocheng but systematically in the pointing of brick walls at Qian Baocheng, Yaotai and Hou Baocheng, showing that similar lime–carbonaceous material systems were applied to pointing and local finishing in Ming-period imperial-mausoleum walls. The Xianling carbon-bearing mortars therefore do not represent a direct replication of Qing-period roofing practices involving *qinghui*, but rather a distinctive manifestation of traditional *qinghui*-type technology in the walls of a Ming imperial mausoleum. They provide a Ming-period material example with explicit chronological and architectural-location information for considering technological developments between Song-period formulation records and the more diverse *qinghui* practices documented in the Qing dynasty, and they reveal how carbon-bearing lime materials were configured across different architectural locations and construction functions.

This study is limited by the number of samples and the restricted sampling area, while the intrinsic heterogeneity of ancient mortars also increases uncertainty in quantitative comparisons among wall sections. The Raman spectroscopic results confirm the presence of poorly ordered carbonaceous matter but cannot uniquely determine its specific source, original formulation or stage of addition. Moreover, because the mortars' workability, mechanical properties and moisture-transport behaviour could not be tested directly, it remains unclear whether the carbonaceous material also affected workability, shrinkage, pore structure, water absorption or durability. Future research should compare regional candidate

raw materials, expand sampling, employ micro-area compositional analyses and conduct experimental reconstructions to further test raw-material sources, preparation processes and performance effects. For the repair of historic masonry, the material composition and technological characteristics of the original mortar are important for selecting replacement materials, and compatibility assessment should also consider physical, chemical and moisture-transport properties.²⁸

This study identified poorly ordered carbonaceous matter in the exposed pointing and finishing mortars of Xianling Mausoleum and revealed systematic material differences between these mortars and the bedding mortars. The multiscale occurrence of the carbonaceous component is consistent with its incorporation before mortar hardening, followed by mixing without complete disaggregation. This example provides material evidence, with a clear chronological and construction-location context, for discussing lime–carbonaceous material systems in Ming-period imperial-mausoleum walls and their relationship with traditional *qinghui*-type technology. These material differences also indicate that future compatibility assessments for repair mortars should extend beyond colour matching to consider binder systems, aggregates and carbonaceous components.

Data Availability

The data supporting the findings of this study, including optical microscopy and petrographic images, XRD and FTIR data, SEM–EDS data, Raman spectral data, and TG–DSC data, are available from the corresponding author upon request. Data presented in the Supplementary Information are provided with this article. Certain heritage-management records, including detailed sampling-location information and high-resolution site-condition images, are not publicly available because their disclosure may be subject to conservation and site-management restrictions. Access to these restricted materials may be considered by the corresponding author in consultation with the relevant heritage-management authority and, where required, subject to an appropriate data-use agreement.

Author Contributions

W.Q.Z., J.Y.Z. and Y.Z.L. contributed to Conceptualization, Methodology, Formal analysis, Supervision, Visualization, and Writing – original draft. X.S.L., J.Y.W., M.P., J.W., H.X. and Y.N.C. contributed to Investigation. Y.Z.L., X.S.L., J.Y.W., M.P., J.W., H.X. and Y.N.C. contributed to Resources and Data curation. W.Q.Z., J.Y.Z., Y.Z.L., X.S.L., J.Y.W., M.P., J.W., H.X. and Y.N.C. contributed to Project administration and Writing – review & editing. All authors reviewed and approved the final manuscript.

Competing Interests

The authors declare no competing financial or non-financial interests.

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