The production of protective earth-based mortars for earth constructions in southeastern Brazil during the 19th century coffee economy

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ABSTRACT: The study of materials used in ancient buildings provides a means to shed light on traditional building practices of past societies, but can serve as inspiration to tackle challenges faced by the current generation. Characterising earth-based structures and finishing elements is part of this mission, especially at a time when earthen architecture is the subject of renewed interest due to its many advantages, particularly in terms of sustainability. This paper considers a set of historical earthen houses built during the first phase of the coffee economy (1820-1880) in the middle *Paraíba do Sul* River valley, in southeastern Brazil. Physical (colour and texture) and chemical (FTIR, TGA, XRF, and XRD) analyses performed on a large set of mortar and local soil samples collected in the region formed the basis for discussing possible soil selection criteria as raw materials, texture solutions, and the stabilisation strategy of the final product. This last aspect implied the addition of small quantities of lime, in the case of external renders and more sporadically in plasters, without the use of fibres or organic additives. Chemical data and historical sources suggest that the raw materials used for this purpose were probably brought to the region from coastal areas.

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KEYWORDS: Mortar. Unfired earth. Earthen architecture. Vale do Paraíba. Brazil. Built heritage.

RESUMO: O estudo dos materiais utilizados nos edifícios antigos fornece um meio de lançar luz sobre as práticas tradicionais de construção das sociedades do passado, mas pode servir também de inspiração para responder aos desafios enfrentados pela atual geração. Caracterizar estruturas e elementos de acabamento das edificações construídas com terra crua faz parte dessa missão, especialmente numa altura em que a arquitetura com terra é objeto de interesse renovado devido às muitas vantagens, particularmente em termos de sustentabilidade. Este artigo considera um conjunto de construções históricas edificadas em terra crua durante a primeira fase da economia do café (1820-1880) no trecho paulista do vale do rio Paraíba do Sul. Análises físicas (cor e textura) e químicas (FTIR, TGA, XRF e XRD) realizadas sobre um grande conjunto de amostras de argamassas e de solos locais recolhidas na região formaram a base para discutir possíveis critérios de seleção de solo como matéria-prima, soluções de textura e a estratégia de estabilização do produto final. Esse último aspecto implicava a adição de pequenas quantidades de cal, no caso de rebocos externos e, mais esporadicamente, nos revestimentos internos, sem a utilização de fibras ou aditivos orgânicos. Dados químicos e fontes históricas sugerem que a matéria-prima utilizada para esse fim foi provavelmente trazida para a região a partir de zonas costeiras.

PALAVRAS-CHAVE: Argamassa. Terra crua. Arquitetura com terra. Vale do Paraíba. Brasil. Patrimônio construído.

INTRODUCTION

For millennia, humankind has used mortars for structural, protective and aesthetical purposes, but mainly as masonry elements between bricks and stones or as finishing materials on internal and external wall surfaces.[3] Although during the history of construction the most common binders in mortar manufacturing were essentially limited to mud, gypsum and lime,[4] the differences in the solutions adopted in different locations and at different times are inevitably significant.[5] As such, it is scientifically relevant to investigate the composition of historic mortars, especially when other sources of information about mortar production practices are scarce.

This paper focuses on the production of finishing mortars in earthen buildings located in a geographic area of special cultural interest due to its history, traditions and architectural heritage. The region, today known as the *Vale Histórico Paulista* (São Paulo's Historical Valley), corresponds to a segment of the middle basin of the *Paraíba do Sul* river, situated in northeastern São Paulo.[6] Its hilly 2300 km^2 territory—distributed along the old route between Rio de Janeiro and São Paulo—was the stage, in the early 19th century, for the introduction of coffee plantation in Brazil, whose expansion was responsible for the country's economic growth between 1820 and 1880.[7] This is why an exceptionally large number of rich country houses and elegant city manors, as well as churches, chapels, and vernacular buildings can be found in both rural and urban settings of the region.[8]

An important aspect of this group of buildings is that they were built using traditional techniques based on unfired earth (adobe, wattle anddaub and rammed earth) that were quickly abandoned thereafter due to the spread of industrialised materials and modern construction practices. These buildings represent, therefore, some of the last examples of a craft whose principles and characteristics may undergo a serious degree of oblivion.[9] This clearly includes mortar-making practices, since in traditional earthen architecture mortars were also made using unfired earth.

Importantly, in recent years, sustainable building practices, pursuant to Agenda 21, have received increasing attention and the revival of techniques based on local unprocessed raw materials is becoming an interesting prospect in many contexts.[10] This constitutes an additional reason why traditional earthen construction practices should be revalued and investigated.

The following sections present and discuss results on the composition of a wide set of mortars and local soil samples collected in the *Vale Histórico Paulista* in order to enlighten the process of raw material selection and its original processing for manufacturing final plasters and renders in earthen historical buildings. First and

3. Cf. Moropoulou, Bakolas, and Anagnostopoulou (2005).

4. Cf. Beas (1991), Callebaut *et al.* (2001), Moropoulou, Bakolas, and Anagnostopoulou, *op. cit.*, Rodrigues (2004), and Sabbioni, Bonazza, and Zappia (2002).

5. Cf. Elsen (2006), Hamard et al. (2013), and Moropoulou, Bakolas, and Anagnostopoulou, *op. cit.*

6. Cf. Cavicchioli, Sant'Anna, and Perroni (2018), Cavicchioli *et al.* (2019), and Fazio *et al* (2015).

7. Cf. Marquese (2008).

8. Cf. Benincasa (2007), Carrilho (2006), and Sá, Santos, and Cavicchioli (2018).

9. Cf. Sá, Santos, and Cavicchioli, *op. cit.*

10. Cf. Avrami (2011), and Gomes, Faria, and Gonçalves (2018).

11. Cf. Average values of September 2017 at the meteorological station of Resende (RJ) according to CETESB (https://bit.ly/3IX412m).

12. Cf. Cavalcanti, Cavicchioli, and Rolon (2018).

foremost, this study seeks to shed light on the technical skills of the original builders who operated in the area and other socio-economic aspects (i.e., raw material trade). We expect that the results obtained will provide scientifically robust elements to anticipate or explain vulnerabilities and pathologies, plan conservation and restoration strategies, and stimulate the expansion of using earth as a material in sustainable construction practice.

EXPERIMENTAL

Sites and sampling

This paper presents and discusses the results of the analyses performed on mortar samples obtained from historical buildings located in the municipalities of Areias, São José do Barreiro, and Bananal in northeastern São Paulo.

A set of 47 mortar samples was collected in this area from sixteen urban and rural locations—27 samples from external walls and 20 from indoor walls.

Sampling was carried out between September 24 and September 28, 2017, period in which the average regional temperature and relative humidity were 22.4 °C and 57%, respectively, with no precipitation (the weather had been completely dry throughout the month).[11] Table 1 summarises the locations where sampling took place with a description of each wall surface (including solar orientation), as well as the conditions under which sampling was performed.

Samples were collected from walls designated by the owners or the person in charge of each property. As a rule, plasters and renders that had an earthy aspect were chosen and, whenever possible, preference was given to walls that were unlikely to have undergone frequent alterations, if any, over time (storerooms, basements, attics, backyard walls). Sampling was performed from intact or already damaged mortar layers. Overlapping mortar layers were collected separately when found. The samples (usually irregular pieces 4-6 centimetres on its largest dimension) were either extracted manually, using protective gloves to avoid contamination, or with the help of hammer and chisel and then conditioned in plastic bags before arriving at the laboratory. Throughout the process, sample removal was limited to minimal amounts as to avoid substantial damage to the historical buildings. In the case of intact mortar layers, after sampling, the wall was repaired using a previously developed technique.[12]

The study also included a set of 31 soil samples for comparative purposes. These soil samples were the subject of a previous research whose results, location and sampling criteria were described in an earlier paper.[13] Importantly, the soil sampling sites were randomly selected along the roads connecting the cities and rural locations in the study area. We assumed that ancient builders would likely collect the raw materials they used for earth structures at locations from which they could easily transport them to the desired destination within the valley. The precise sampling points were among those where the soils were already exposed due to erosion processes.

Prior to analysis, the mortar samples were treated following the same technique used for the soil samples analyzed in the previous paper. In the laboratory, after all samples were oven-dried at 40 °C for 48h, fragments with the macroscopic characteristics (colour and texture) of the predominant plaster or render from each sampling site were selected for analysis. Whole samples were characterized by colour and texture. Colours were described by visually comparing dry samples with Munsell soil colour charts,[14] as done in the previous study[15] on local soils and masonry materials. The intention was to approach the colour perception that original builders might have had of the soils under dry weather conditions, when raw material collection was likely to take place. Fine soil texture (particles <2mm) was determined using a Microtrac Bluewave particle size analyser and the results were classified according to the Soil Science Division Staff and EMBRAPA.[16] Then, an aliquot of approximately 5 g was finely ground to obtain particles smaller than 75 μm in diameter. This powder was used to perform attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR), thermogravimetric analysis (TGA and TGA/DSC/MS), X-ray fluorescence elemental analysis (XRF) and X-ray diffraction analysis (XRD) for identifying minerals.

Soil analytical data were partially either recovered from previous work (texture)[17] or obtained anew (XRF and XRD).

13. Cf. Cavicchioli, Sant'Anna, and Perroni, *op. cit.*

14. Cf. Munsell Soil Colour Charts (2000).

15. Cf. Cavicchioli, Sant'Anna, and Perroni, *op. cit.*

16. Empresa Brasileira de Pesquisa Agropecuária (1979), and United States Department of Agriculture (2017).

17. Cf. Cavicchioli, Sant'Anna, and Perroni, *op. cit.*

Table 1 – List of mortar samples analysed in this work and the main characteristics of sampling sites and surfaces. To identify the buildings, the presumed original name was adopted to avoid confusion arising from the use of different names over time. The assumed foundation date informed by local sources (mainly owners, local population, government bodies) are given in parentheses when available. Geographical coordinates are in decimal degrees using the WGS84 datum.

Sample code	Description of sampling surfaces
Location 1: Fazenda São Miguel (1857), latitude: -22.681093, longitude -44.599712. Municipality of São José do Barreiro	
00	Indoor adobe wall (wall 1, facing NW) of a ground-floor storage room of the main building. Fragments were collected at a 1.5 m-height from an area of intact plaster (40 × 40 cm, 4-5 cm thick) exhibiting an earthy aspect
01	Like sample 00, but fragments were collected within the same area from a patch with a brighter colour and a harder texture
02	Like sample 00, but fragments were collected within the same area from a patch with a slightly brighter colour
05	Indoor adobe wall (wall 2, facing NE) of a secondground-floor warehouse of the main building. Fragments were collected at a 1.2 m-height from an area of intact plaster (40 × 40 cm, 4-5 cm thick) exhibiting an earthy aspect
06	Like sample 05, but fragments were collected within the same area from a patch with a brighter colour
07	Like sample 05, but fragments were collected from a layer with similar features as 05 but underlying sample 06
09	Outdoor render (facing SE) covering a wooden structural column (eaves partially reduce exposure to weather factors). Fragments were collected at a 1.0 m-height from the edge of the remaining layer of a partially collapsed render (3 cm thick) exhibiting either an earthy or cement aspect. Sample 09 corresponded to an earthy fragment, but with an unusual hardness
10	Like sample 09, but sample 10 corresponded to a cement fragment (probably Portland cement)
11	Outdoor render (facing SW) of a stone column (column 1) from a disappeared farm building (the column is fully exposed to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (0.5-1.0 cm thick) exhibiting an earthy aspect and hard texture
12	Outdoor render (facing NW) of a second stone column (column 2) of a disappeared farm building (the column is fully exposed to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (0.5-1.0 cm thick) exhibiting an earthy aspect and hard texture
13	Outdoor render (facing NW) of a third stone column (column 3) of a disappeared farm building (the column is fully exposed to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (1-2 cm thick) exhibiting an earthy aspect and hard texture
Location 2: Fazenda dos Coqueiros (1855), latitude: -22.681093, longitude: -44.599712. Municipality of Bananal	
14	Indoor adobe wall (facing SW) of an underground storage room of the main building. Fragments were collected at a 0.8 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
15	Indoor stone column (facing SW) of an underground storage room of the main building. Fragments were collected at a 0.8 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
19	External adobe wall (facing S) in the access alley (the wall is fully exposed to weather factors). Fragments were collected at a 0.8 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
Location 3: Urban house in Rua Ernani Graça (1860), latitude: -22.683339, longitude: -44.323535. Municipality of Bananal	
20	Outdoor render (facing NW) of the façade adobe wall (eaves partially reduce exposure to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (1-2 cm thick) exhibiting an earthy aspect

Sample code	Description of sampling surfaces
Location 4: Urban house in Rua Antonio Valipte (possibly second half of the 19th century), latitude: -22.683166, longitude: -44.323387. Municipality of Bananal	
21	Outdoor render (facing W) of a side rammed-earth wall of the house (the wall is fully exposed to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (1-2 cm thick) exhibiting an earthy aspect
Location 5: Urban house in Rua Manoel de Aguiar (possibly second half of the 19th century), latitude: -22.683930, longitude: -44.323717. Municipality of Bananal	
22	Outdoor render (facing NE) of a side wattle and daub wall of the house (the wall is protected from weather factors by a canopy). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (1-2 cm thick) exhibiting an earthy aspect
Location 6: Santa Casa de Misericórdia (1871), latitude: -22.681386, longitude: -44.320479. Municipality of Bananal	
24	Indoor rammed-earth wall (wall 1, facing NE) of a ground-floor storage room of the main building (first rammed-earth wall). Fragments were collected at a 0.5 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
25	Indoor rammed-earth wall (wall 2, facing NW) of a second ground-floor storage room of the main building (second rammed-earth wall). Fragments were collected at a 0.5 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
Location 7: Church of Bom Jesus do Livramento (1811), latitude: -22.684005, longitude: -44.322934. Municipality of Bananal	
26	Indoor rammed-earth wall (facing W) of the upper part of the bell tower. Fragments were collected at a 1.5 m-height (from floor) from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
Location 8: Solar Luciano de Almeida (1847), latitude: -22.683160, longitude: -44.322965. Municipality of Bananal	
29	Exterior ground floor rammed-earth wall (wall 1, facing N) of the main building (eaves partially reduce exposure to weather factors). Fragments were collected at a 1.8 m-height from a damaged render that was excavated and revealed several overlapping layers. Sample 29 (earthy aspect) was the innermost layer
28	Like sample 29, but a layer above it
34	Like sample 29, but collected from another site
32	Like sample 29, but a layer above sample 34
31	Like sample 29, but a layer above sample 32
30	Like sample 29, but a layer beneath sample 31 (innermost)
35	A second exterior ground floor rammed-earth wall (wall 2, facing E) of the main building (eaves partially reduce exposure to weather factors, second rammed-earth wall). Fragments were collected at a 1.8 m-height from a damaged render that was excavated and revealed several overlapping layers. Sample 35 (earthy aspect) was the innermost layer
36	Like sample 35, but a layer above it
37	Like sample 35, but a layer above sample 36
Location 9: Fazenda Catadupa (1827), latitude: -22.665186, longitude: -44.531744. Municipality of São José do Barreiro	
39	Indoor adobe wall (wall 1, facing NE) of a ground-floor storage room of the main building (first adobe wall). Fragments were collected at a 1.8 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
40	Like sample 39, but a layer above it (innermost)

Sample code	Description of sampling surfaces
44	Indoor adobe wall (wall 2, facing NW) of a ground-floor storage room of the main building (second adobe wall). Fragments were collected at a 1.8 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
45	Like sample 44, but fragments were collected within the same area from a patch with a brighter colour
48	Indoor stone column (facing NE) of a underground storage room of the main building. Fragments were collected at a 1.8 m-height from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
49	Indoor wattle and daub wall (facing NE) of a first-floor room of the main building. Fragments were collected at a 0.5 m-height from the edge of the remaining layer of a partially collapsed plaster (2-3 cm thick) exhibiting an earthy aspect
Location 10: Fazenda da Barra (1851), latitude: -22.681924, longitude: -44.541362. Municipality of São José do Barreiro	
50	Exterior ground floor wattle and daub wall (wall 1, facing NW) of an annex building in ruins (the wall is fully exposed to weather factors). Fragments were collected at a 1.0 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
51	Like sample 50, but a layer beneath it
52	A second exterior ground floor wattle and daub wall (wall 2, facing NW) of an annex building in ruins (the wall is fully exposed to weather factors). Fragments were collected at a 1.0 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
53	A third exterior ground floor wattle and daub wall (wall 3, facing SE) of an annex building in ruins (the wall is fully exposed to weather factors). Fragments were collected at a 1.0 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
55	Indoor ground floor wattle and daub wall (facing SE) of an annex building in ruins. Fragments were collected at a 1.0 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
Location 11: Urban house in Rua Comendandor Luiz Ferreira (possibly second half of the 19th century), latitude: -22.645575, longitude: -44.576240. Municipality of São José do Barreiro	
60	Outdoor render (facing N) of the façade wattle and daub wall of the house (eaves partially reduce exposure to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (2-3 cm thick) exhibiting an earthy aspect
61	Like sample 61, but fragments were collected within the same area from a patch with a brighter colour
Location 12: Solar Capitão-mor Gabriel Serafim da Silva (1798), latitude: -22.580545, longitude: -44.696666. Municipality of Areias	
62	Indoor wattle and daub wall (facing NE) of the attic of the main building. Fragments were collected at a 1.5 m-height (from the floor) from the edge of the remaining layer of a partially collapsed plaster (2-3 cm thick) exhibiting an earthy aspect
Location 13: Solar Aguiar Valim (1855), latitude: -22.682914, longitude: -44.322629. Municipality of Bananal	
63	Indoor wattle and daub wall (facing N) of a first-floor room of the main building. Fragments were collected at a 1.5 m-height (from the floor) from the edge of the remaining layer of a partially collapsed plaster (3-4 cm thick) exhibiting an earthy aspect
Location 14: Urban house in Rua Manoel de Aguiar (possibly second half of the 19th century), latitude: latitude: -22.684025, longitude: -44.324622. Municipality of Bananal	
64	Outdoor render (facing S) of the façade adobe wall of the house (eaves partially reduce exposure to weather factors). Fragments were collected at a 1.8m-height from the edge of the remaining layer of a partially collapsed render (1-2 cm thick) exhibiting an earthy aspect

Sample code	Description of sampling surfaces
Location 15: Fazenda Morada do Sol (possibly second half of the 19th century), latitude: -22.621427, longitude: -44.708864. Municipality of Areias	
65	Outdoor render (facing SW) covering the ruins of an old construction of the current farm (fully exposed to weather factors). Fragments were collected at a 1.5 m-height from the edge of the remaining layer of a partially collapsed render (3 cm thick) exhibiting an earthy aspect
Location 16: Fazenda Santana (possibly second half of the 19th century), latitude: -22.658617, longitude: -44.683297. Municipality of Areias	
66	Fragments, exhibiting an earthy aspect, were scattered on the ground at the site of the abandoned ruins of a disappeared farm. It was assumed they belonged to an outdoor render of a rammed-earth wall. They were fully exposed to weather factors

Fourier Transform Infrared Spectroscopy and Thermogravimetric Analysis

ATR-FTIR spectra were obtained using a Bruker Optics ALPHA FTIR spectrometer equipped with KBr optics and DTGS detector in the ATR module (diamond crystal, single reflection mode). Spectra were recorded at 4 cm^{-1} resolution. TGA was performed using a TA Instruments Q500 thermogravimetric analyser, typically with 10 mg samples, from room temperature up to 950 °C, at a heating speed of 10 °C/min. A limited group of samples was also analysed using simultaneous TGA/DSC coupled with a MS detector for the released gases (STA 409 PC Luxx, Netzsch) under the same operating conditions.

X-ray Fluorescence Analysis

Elemental analysis for Si, Al, Ca, Fe Ti, Na, K, Co, Ni, Zn, V, Cr, Mn, Cu and S was performed by X-Ray Fluorescence, in triplicate, using a portable Tracer III Spectrometer (Bruker). The equipment was operated in bench mode at tube voltage and current of 15 kV and 25 μA and with an acquisition time of 120 s (under internal vacuum and without any excitation filter). Quantitative results were obtained based on the soil, ceramic and mud rock calibration provided by Bruker. The results discussed correspond to the average of the triplicate analysis of each sample.

18. *Ibid*.

19. Cf. Santiago (2007).

X-ray Diffraction Analysis

Measurements were made using a Bruker D8 Advance diffractometer with theta-theta system (CuKα radiation, 40 kV, 25 mA), automatic Air-scatter and LYNXEYE XE detector at the Petroleum, Natural Gas and Bioenergy Division, Institute of Energy and Environment from the University of São Paulo (IEE-USP), Brazil. The scanning range of bulk material was from 2° to 70° 2 θ with a step size of 0.02° 2 θ and measuring time of 10 s per step, using primary and secondary slits of 0.5 mm and 1 mm, respectively. Mineralogical identifications and graphic processing of the diffractograms were performed using DiffracEva software version 4.2.2 (Bruker ASX GmbH), based on the International Centre for Diffraction Data (ICDD) database.

Scanning Electron Microscopy – Energy Dispersive Spectroscopy

Whole fragments of selected samples had their surfaces covered with a thin platinum film and were analysed on a FEI model Quanta 650 FEG Scanning Electron Microscope (SEM) equipped with Energy-Dispersive X-ray Spectroscopy (EDX) operated at low vacuum and 20 kV.

RESULTS AND DISCUSSION

Carbonate content

A previous study showed that lime had not been employed for soil stabilisation in the manufacture of rammed earth, adobe or wattle and daub walls of the traditional earth structures in the study area.[18] But it was particularly relevant to verify to what extent lime had been an ingredient in the making of plasters and renders, since its use was well established in the Portuguese tradition and in previous colonial building practice (16th-18th centuries).[19]

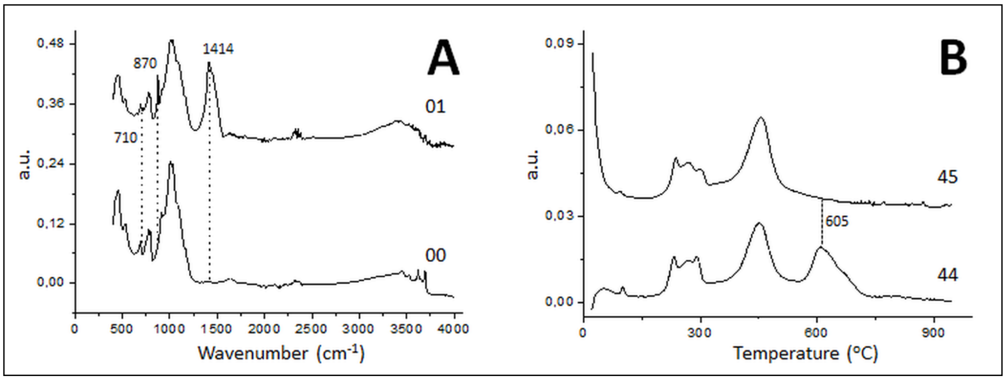

Figure 1 – Typical examples of FTIR spectra (A) and differential TGA curves (B) of mortars exhibiting (01 and 44) and not exhibiting (00 and 45) carbonate characteristics, that is, bands at 1400, 870 and 710 cm^{-1} (FTIR) and mass losses at T>600 °C (TG).

20. Cf. Cavicchioli, Sant'Anna, and Perroni, *op. cit.*

Since local soils have an insignificant presence of carbonates,[20] the use of lime could be inferred from the calcium carbonate ($CaCO_3$) content that is formed from lime (CaO) upon reaction with CO_2. This was performed in two steps: all mortar samples were analysed using FTIR (see Figure 1A) and those revealed to have typical CO_3^{2-} bands at 1400 cm^{-1} (asymmetric stretching), 870 cm^{-1} (out-of-plane bend) and 710 cm^{-1} (in-plane bend) had the carbonate content accurately assessed by TG (see examples in Figure 1B) as a function of CO_2 formation.

The combined approach to assessing the use of lime in mortar manufacturing resulted in the bar graph in Figure 2.

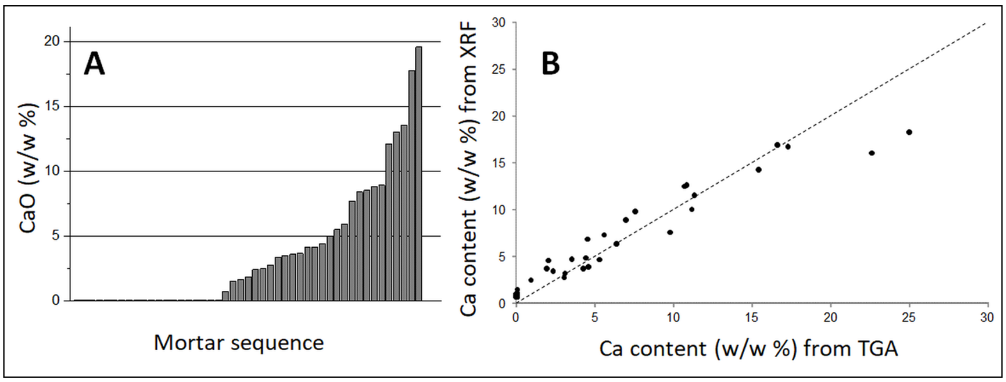

Figure 2 – A: Distribution of theoretical CaO amount used in mortar manufacturing calculated from CO_2 loss by TGA; B: correlation between Ca amount measured from TGA data (assuming the only Ca compound is $CaCO_3$) and from XRF spectra.

Calculation of the CaO used in mortar formulation surmises total transformation of calcium oxide into calcium carbonate upon reaction with CO_2, an assumption that, to a good extent, is confirmed by the results in Figure 2B, which shows the

21. Cf. Cardiano *et al*. (2008), Moropoulou, Bakolas, and Bisbikou (1995), Sauman (1971), and Shoval *et al*. (1993).

correspondence between the Ca concentration calculated from TGA data and its content obtained from XRF measurements of elemental Ca. The linear correlation between the two data sets with unit slope is notable, within a reasonable margin of error, except for two observations in which the presence of carbonates other than $CaCO_3$ is likely (although $MgCO_3$ alone does not account for the difference).

In all cases, the carbonate peak in the differential TGA curves occurs at an early stage, that is, between 600 and 700 °C. This reflects the presence of crystal imperfections in the carbonate structure typical of the rapid transformation undergone by lime when exposed to atmospheric carbon dioxide, which further helps exclude the mineral origin of $CaCO_3$.[21]

Four groups of mortars can be distinguished based on the $CaCO_3$ data: mortars with undetected $CaCO_3$ (lime-free mortars, 20 samples) corresponding to wall finishes in which no lime was used as a ligand, and mortars prepared with CaO amounts of less than 6% (17 mortars) or in the range of 6-10% (five mortars) or 10-20% lime (five mortars, including sample 10 that had the appearance of Portland cement and exhibited approximately 20% w/w CaO).

Figure 3 presents the distribution of lime-containing versus lime-free mortars among the different sites and their respective sampling surfaces. The amount of lime used as ligand in the manufacture of each mortar sample is highlighted in red, while lime-free mortars are indicated with the letter U (undetected $CaCO_3$), with the sample number shown in parentheses. The underlying support is also shown (adobe, rammed earth, wattle and daub or stone, and, in one case, wood) and the outer walls are discriminated by an orange background.

Looking at the schema, it can be observed that lime-free and lime-containing mortars are found both on internal and external walls. In lime-containing plasters, the CaO content ranges from 0.1% (sample 39) to 17.7% (sample 15), with an average of 4.8%. In renders, the concentration ranges from 2.7% (sample 28) to 19.6% (sample 10), with an average of 7.8%. But there are also some clear anomalies: the 0.1% amount (sample 39) seems rather unlikely and might be the result of contamination from the overlapping mortar (40); sample 15, although an indoor plaster, was applied over a stone column where normally the amount of ligand is understandably higher (see below); and sample 10 is considered a much later addition, as it resembles a cement mortar. Yet, even disregarding the values of these materials from the calculated averages, the means become 3.7% (plasters) and 6.9% (renders), suggesting that a larger amount of lime was generally preferred for external coatings. Which is perfectly in line with the need for a marked resistance of renders against weather factors, especially the incidence of rain.

Another interesting aspect that can be drawn from analysing Figure 3 is the lack of clear correlations between the use of lime in the mortars and the geographical distribution of samples, and the type of underlying substrate, except for the ubiquitous use of CaO in all stone structure coatings. This may be explained by the lower adhesion of earth-based mortars on a less porous support, and thus the need to improve this property by adding a strong binder.

Moreover, the data shown in Figure 3 can point to one more notable observation that might help in understanding the manufacture of earth-based mortars in the study area. In all situations where more than one sample could be extracted from the same wall, both lime-free and lime-containing mortars were found (locations 1, 8, 9, 10 and 11 – sample 39 allegedly being a contaminated lime-free mortar). At location 1 (Fazenda São Miguel, São José do Barreiro), both indoor walls had predominantly lime-free plaster with patches of a lime-containing material (samples 1 and 7), which can be attributed to later repairs. This may suggest the evolution towards a construction practice where the use of lime was increasing and becoming more regular. As for the overlapping layers, the use of lime-free renders always in the lower (innermost) layer can be interpreted as the sign of a specific construction practice, involving the use of a preparatory (lime-free) layer and a finishing lime-containing layer on top. This reading is supported by the absence of a paint film between them. The four overlapping layers of render on wall 1 of location 8 (Solar Luciano de Almeida, Bananal) present an even more complex case where the assumed oldest layer (sample 34, lime-free) is progressively covered with mortars with an increasing amount of lime (3.6, 8.8 and 12.1%, samples 32, 31 and 30, respectively), again with no layers of paint in between. This may reveal the need for a specific adjustment during construction, or the demand for an even stronger outer coating for the building.

22. Medina (2017).

23. According to Souto (2016), local newspapers in the 19th century used to advertise lime manufactured from shell deposits (*cal de marisco*) as a high quality product.

24. Cf. Santiago, *op. cit.*

25. Cf. Tucker, and Wright (1990), and Vita, Luna, and Teixeira (2007).

26. Santiago, *op. cit.*

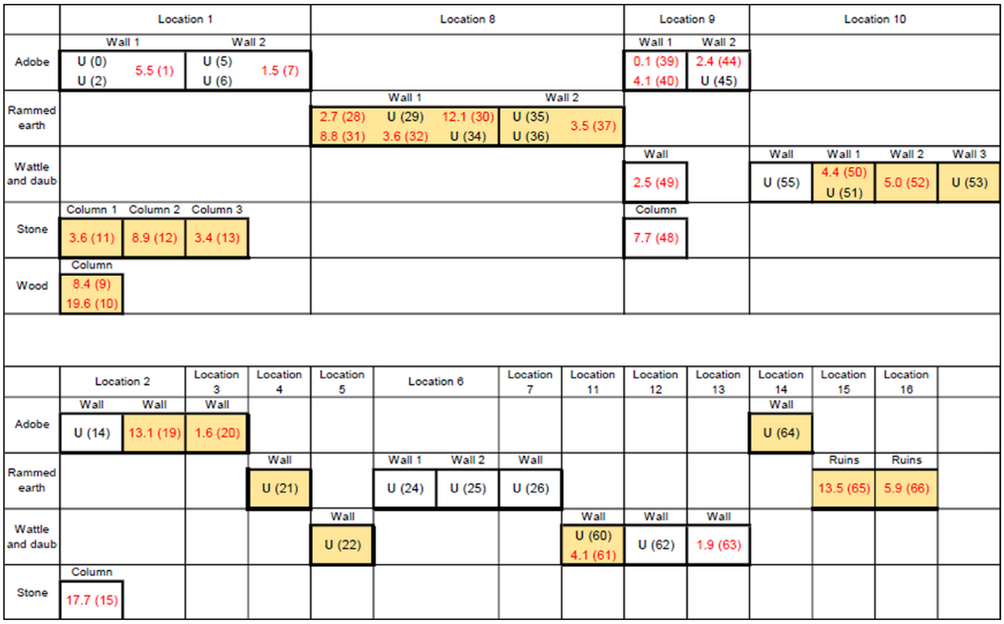

Figure 3 – Schema showing the CaO content (% in mass) theoretically used for manufacturing mortars (according to the sample analysis) distributed by location, sampling point, and technique of the underlying structure. Locations and wall numbers are listed in Table 1. Lime-free mortars (identified by **U**) are also reported. Sample numbers are reported in parentheses. The orange background identifies exterior mortars.

One feature that was clearly evaluated with the elemental composition of the mortars is the absence of a correlation between Ca and Mg content, in most cases (but not always) with very low levels of Mg (less than 0.5%). This means that the variation in Mg concentration is generally not associated with the Ca variation and therefore does not depend on the amount of CaO that was added to the earth mixture as a binder. As a result, the use of extremely pure calcitic carbonate as a raw material can be inferred for the production of most mortars. From a contemporary perspective, this outcome corroborates the established notion of the worst performance of lime as a binder for mortars when prepared from calcareous material exhibiting significant amount of $MgCO_3$ (according to Medina,[22] this element should not exceed a 15% concentration). In the 19th century, however, chemical information was clearly yet to be assessed, and consequently the choice of suitable (i.e., low-magnesium) raw materials was associated with the selection of corroborated sources[23] or other visual characteristics (such as colour and hardness, which, however, did not necessarily guarantee low levels of this element).[24] This suggests that the source of the raw material for lime manufacturing was probably coastal shell deposits (of recent formation,[25] hence poor in Mg, as shown by the analyses reported in Santiago, 2007)[26] and that lime was manufactured in seaside locations, which concentrated the production of this component in the 19th century.[27] If this hypothesis is accepted as the most likely, then the area around the city of Rio de Janeiro (approximately 200 km away) would probably be the most feasible source. At that time, the circulation of goods between the coast and the inland was quite intense due to the coffee route,[28] and thus the import of lime from distant production areas should not be surprising, even if the use of animal transportation (the first railway system was inaugurated in the late 1870s) might have limited the actual availability of lime and should explain why it was used somewhat sparingly, that is, in rather low concentrations.

On the other hand, lime production in the 19th century from carbonate rocks in the inland is mentioned in literature,[29] and therefore the supply of lime from such a source should not be ruled out (and possibly accepted for the samples that showed higher Mg concentration, such as samples 9, 11-13, all from location 1). As such, the raw material could be any marble occurrence in southeastern Brazil where strips and lenses of marble and dolomitic marble, intercalated in the Precambrian basement (e.g., in Campos do Jordão or in the western part of Rio de Janeiro, between the municipalities of Valença and Barra Mansa) were described.[30] If this were the case, however, one must assume that pure calcitic limestone was used systematically throughout the 19th century across the region,[31] and this seems unlikely as the surprising stability in Mg content in lime-containing mortars would clash with the natural fluctuation of Mg in carbonatic rocks.

Colour of mortars

All but one mortar colour belong to the 10 YR and 7.5 YR hue groups (sample 10 was classified as 5 Y 7/1, light grey). Figure 4 shows their distribution in terms of chroma and values, with carbonate-containing mortars highlighted in red.

27. Cf. Souto, *op. cit.*

28. Cf. Hollanda (2010).

29. Souto, *op. cit.*, for example, reports the existence of a lime production site in Barra do Piraí, near the study area.

30. Cf. Heilbron *et al.* (2007), and Perrotta *et al.* (2005). The dolomitic character of the rocks in larger geological limestone reserves within the municipalities of Cruzeiro, Pindamonhangaba and Taubaté, between 50 to 200 km upstream the *Paraíba do Sul* River (GUIMARÃES, 1952), and Barroso, 30 km east of São João del Rei, on the old road towards the gold mines of Minas Gerais, about 300 km north (GUIMARÃES, *op. cit.*, NOCE, 1988, and RIBEIRO *et al.*, 2013, and references cited therein) seems to be in disagreement with the low Mg content found in the samples analysed here.

31. Low magnesium content was consistent in the samples, within the narrow range of 0.3-0.5% in 19 (73%) of the 26 lime-containing earth-based mortars.

32. Cavicchioli, Sant'Anna, and Perroni, *op. cit*.

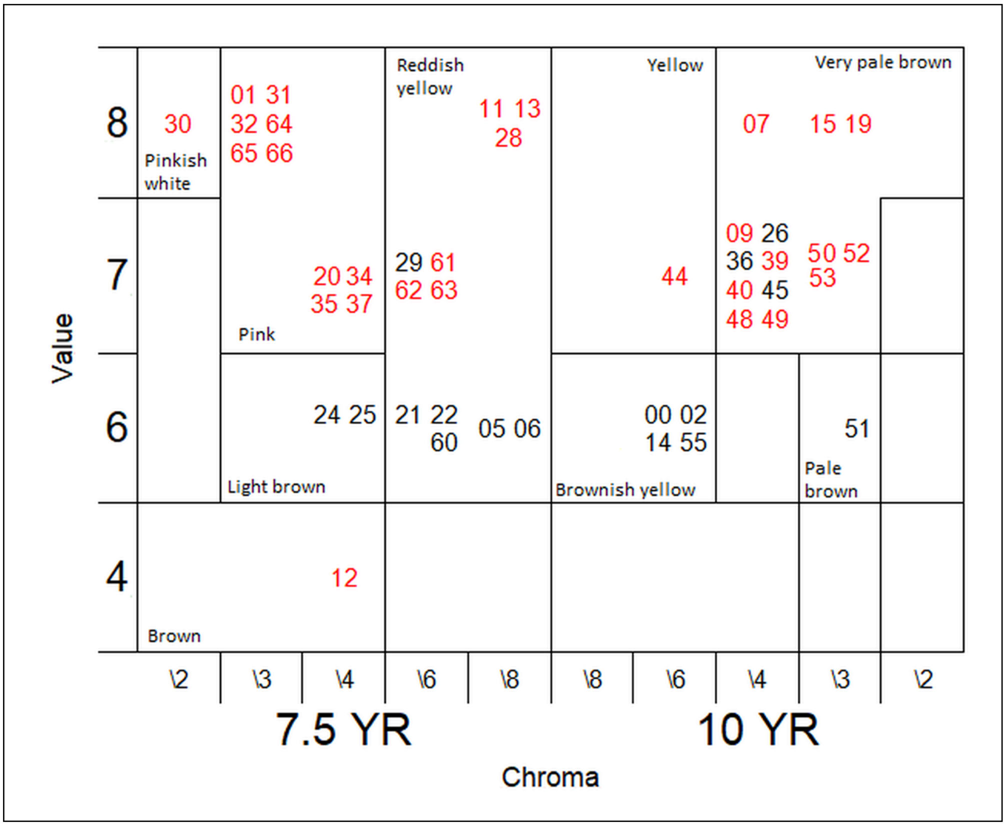

Figure 4 – Colour distribution of mortars (red numbers indicate carbonate-rich materials).

Two aspects should be highlighted in the interpretation of colours. First, all samples (except sample 10, which is a Portland cement-based mortar) belong to yellow-red domains, and were therefore all produced using soil as raw material. Although only part of them could be confirmed as original from when the historical earth structures were built, the general presence of an earthen substrate suggests that the practice of using soil in mortar formulation has been customary in the region for quite some time. The transition to more modern solutions has probably been gradual and may have continued for some time after the introduction of burned clay-based building technology.

A second aspect to be highlighted is that the range of hues covered by the mortars is considerably narrower than the general range of soils found in the historical valley and described by Cavicchioli, Sant'Anna, and Perroni (2018).[32] While in soils the colours range from 2.5 YR to 10 YR hues, with a predominance of 5 YR (therefore with a stronger reddish character), mortars are concentrated in a more yellowish area (between 7.5 YR and 10 YR). Consequently, colour might have been a factor in soil selection in the practice of vernacular mortar manufacturing in the 19th century (as has been suggested for the production of adobes[33] and mortars).[34] But whether this actually occurred and whether it effectively corresponds to a criterion for choosing soils with the best properties is a hypothesis that requires further investigation.

33. *Ibid*.

34. Cf. Vasconcelos *et al*. (2020).

Interestingly, carbonate-rich mortars still belong to the same chroma categories, but with higher values, possibly because of the white contribution of $CaCO_3$. This can be considered an early indication that the same range of soils was used with or without the introduction of lime as an aggregating agent.

Organic matter

To assess the presence of organic matter, FTIR spectra and TG curves were used. FTIR did not show the set of bands that would be characteristic of organic molecules, that is, 2960/2925 cm^{-1} (C-H stretching of $-CH_3$ and $-CH_2$ groups), 1730 cm^{-1} (C-O stretching of COOH groups), 1580-1620 cm^{-1} (C=C stretching of aromatic bonds and C-O stretching in COO^-), 1460/1378 cm^{-1} (C-H bending of CH_3 and CH_2), 1285/1270 cm^{-1} (C-O stretching and OH bending), 950-1125 cm^{-1} (C-O stretching of carbohydrates and polysaccharides) and 900 cm^{-1} (aromatic ring breathing). Interpreting the TGA spectra is less straightforward, since the T interval in which organic matter normally decomposes corresponds to the interval in which various minerals undergo thermal processes associated with mass H_2O loss. For this reason and to obtain quantitative information on the presence of organic molecules in the mortars, a selected group of samples was analysed using a simultaneous TGA/DSC instrument coupled to a MS detector for the released gases, set to record the emission of molecules with $m/z=18$ (thus mainly H_2O) and $m/z=44$ (CO_2). All analysed samples showed very reduced CO_2 emission in the 200-400 °C interval, as observed in the MS *versus* temperature graph (Figure 5), which is representative of the behaviour of all other samples. Importantly, the ion current associated with the CO_2 peak at 313 °C is at least one order of magnitude smaller that the H_2O signal in the same range, thus indicating a very reduced CO_2 emission compared with water and thus a hardly noticeable presence of organic matter.

35. Cf. Gleize *et al.* (2009), and Moropoulou, Bakolas, Bisbikou, *op. cit.*

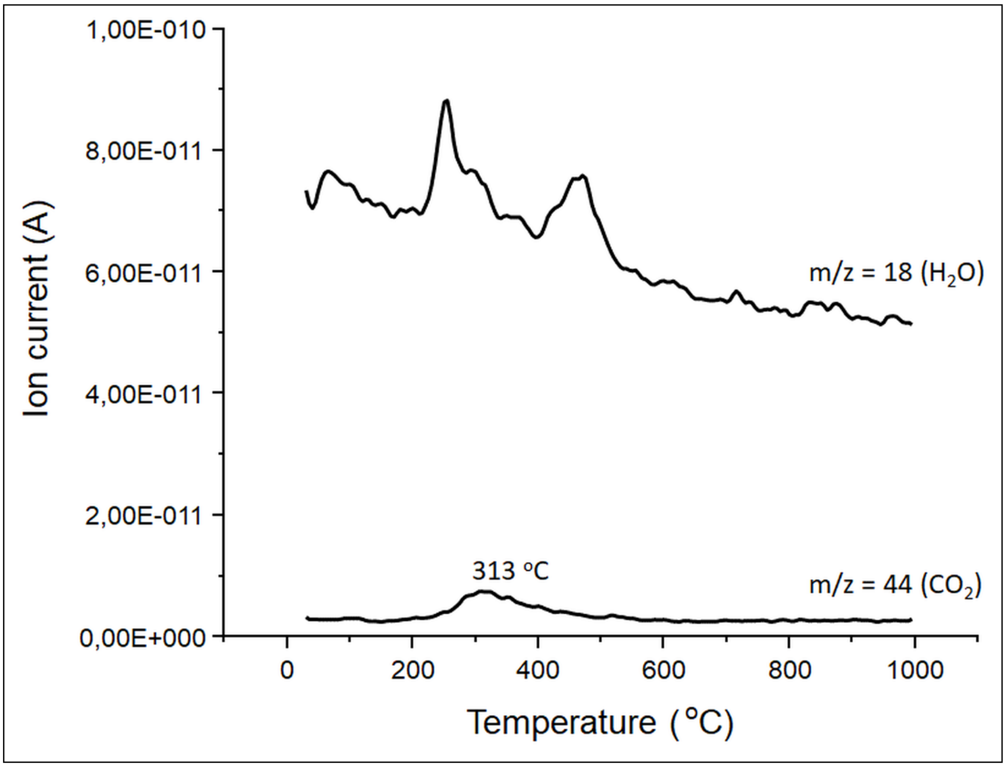

Figure 5 – Evolution of gases during TGA/DSC analysis of mortar no. 00: H_2O (m/z=18) and CO_2 (m/z=44).

Texture and grain size distribution

Texture assessment, performed according to the procedure described in section 2.3, showed that all mortars had a maximum percentage of coarse particles (>2mm) of 3%, with only one sample (no. 26) exhibiting a substantial amount of plant fibres.

This result, together with the information in section 3.3, shows that the strategies adopted by traditional builders in mortar manufacturing did not include the use of plant fibres nor organic additives, that is, solutions that appear in the range of possibilities in some historical sources.[35]

Grain size distribution, on the other hand, shows a completely different scenario. Both mortars and soils were characterised in terms of grain size distribution and the proportion of clay, silt and sand found in the set of samples is illustrated in Figure 6.

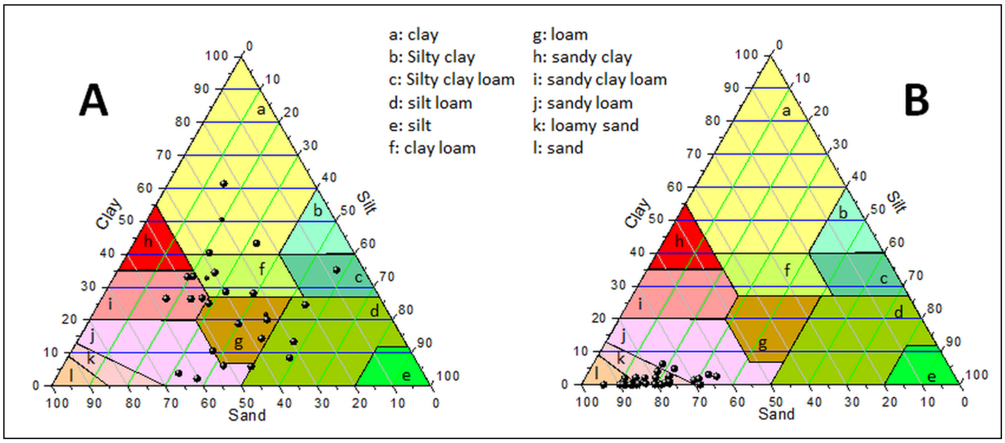

Figure 6 – Tertiary graphs indicating the volume % of clay, silt and sand content in soils (A) and mortars (B) and classification of each sample in USDA-NRCS texture classes.[36]

Soils are distributed across seven texture classes, concentrated mainly in the middle section of the graph, with most showing concentrations of three textures that are quite similar. Mortars, on the other hand, are markedly concentrated in the sand-rich corner of the graph, with very low clay concentration (<10%) and sand concentration ranging from 60 to 100% (although most are concentrated in the 75-90% range).

Clearly, only a minor proportion of soils reflects the grain size distribution of mortars. Better insight into the process behind adjusting the soils to the required grain size distribution could be obtained by elemental analysis data.

Soils used in mortar manufacture

Figures 7 and 8A indicate the existence of a good anti-correlation between the proportion of sandy fraction and water loss in the 120-550 °C temperature range obtained by TGA (Figure 7), and the amount of elemental aluminium (Figure 8A). Both parameters are associated with the content of clay minerals that are mainly alumino-silicates, with a predominance of kaolinite ($Al_2Si_2O_5(OH)_4$) in the case of the study area (Figure 10). As expected, therefore, the search for and use of coarser raw materials correspond to mortars relatively poor in clay minerals. On the other hand, Figure 8B indicates that the correlation between the proportion of sandy mortars and Si content is practically non-existent, thus suggesting that, to obtain the desired texture, the common practice was not to add sand, which is largely composed by quartz grains (SiO_2). At least, this is not expected to be the

36. United States Department of Agriculture, *op. cit.*

main approach to the aggregate adjustment, as is the case in current mortar manufacturing. Rather, the thesis of a practice based on looking for coarser, less weathered (rather than clayey) soils is corroborated.

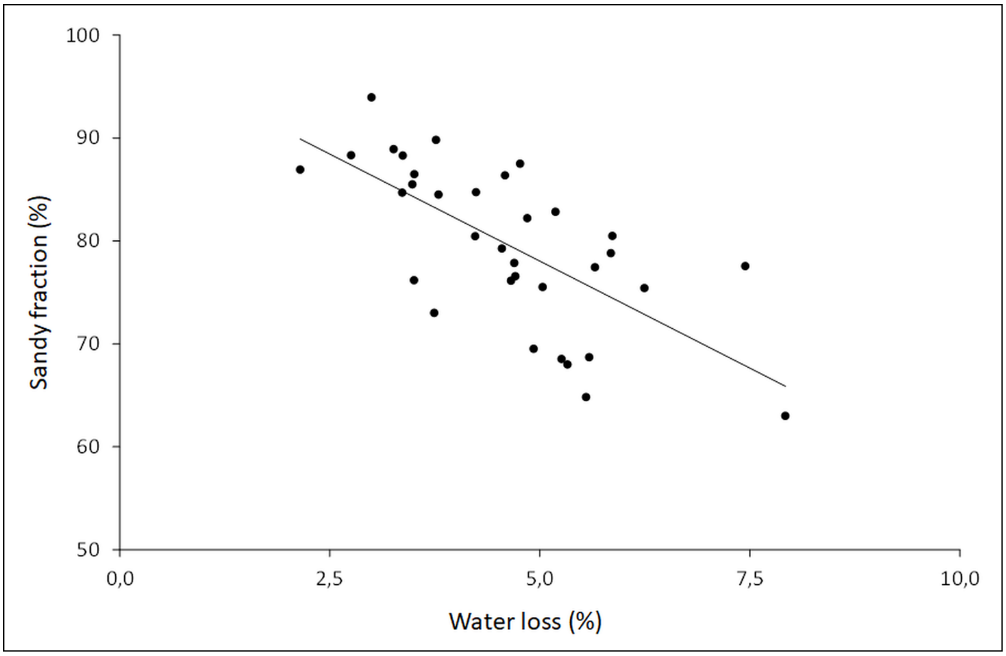

Figure 7 – Correlation between the proportion of sandy fraction of mortars and water loss in the 120-550°C range obtained by TG curves.

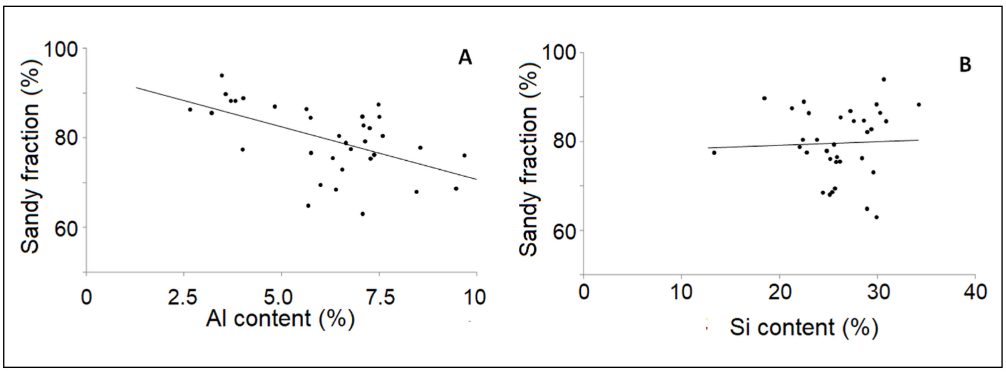

Figure 8 – Correlation between the proportion of sandy fraction of mortars and (A) Al and (B) Si.

Two main arguments support this assumption. First, considering the chemical distribution map of the soils and mortars based on the bidimensional plot of the first and second principal component scores (Figure 9) obtained by Principal Component Analysis (PCA) of the original elemental composition data, there is a distinct separation between most soils and the group of mortars. Although lime-free mortars are understandably chemically closer to the soils, such overlap occurs only with a limited group of soils, which are among those exhibiting the highest coarse particle content, as highlighted in the graph. Moreover, the mineralogical data are also pointing in this direction. Figure 10 highlights the minerals identified by XRD diffractometry and the relative abundance of each in the set of soils (red bars) and mortars (black bars). It can be observed that, in general, soils and mortars exhibit the same characteristic minerals, with a few exceptions and some differences in relative abundance. First, calcite appears only in mortars, thus confirming that the presence of this compound is exclusively associated with the addition of lime to the mortar formulation. As expected, $MgCO_3$ (dolomite and magnesite) is only occasionally found, thus corroborating the use of pure calcitic lime in the production of such materials, with rare appearance of unreacted CaO.

Among the higher abundance minerals, it is significant that the overall abundance of quartz between soils and mortars remains similar, thus indicating that mortars are not particularly richer in river sand. On the other hand, both groups show important differences regarding the frequency of feldspars (Pl and FK), mica (M) and amphibole (Am)—all more abundant in mortars—and clay minerals, mainly kaolinite (K), but also gibbsite (Gi), illite/chlorite (I/C), chlorite (Clo) and illite/smectite (I/S)—all less abundant.

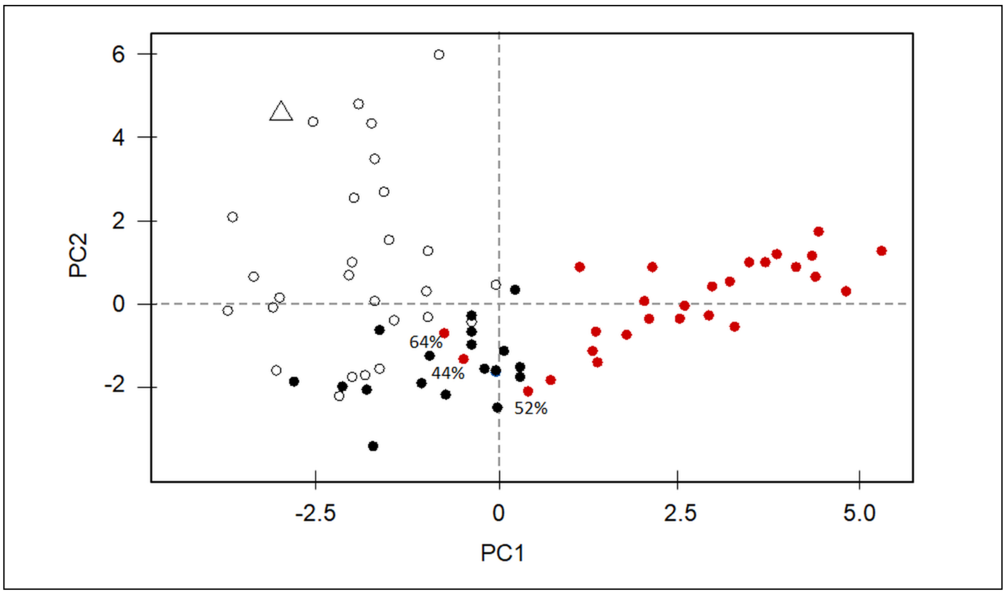

Figure 9 – Bidimensional score plot of each PCA1 and PCA2 (all variables except Ca) analysis. Soils are marked as red circles, mortars without carbonate in their composition are marked as black circles, mortars with carbonate in their composition are marked as void circles and triangles (triangle indicates a mortar manufactured with Portland cement). Separation between the two groups of mortars (with and without lime) occurs along the PC2 axis, which is overwhelmingly ruled by Ca content.

37. Cf. Vasconcelos *et al.*, *op. cit.*

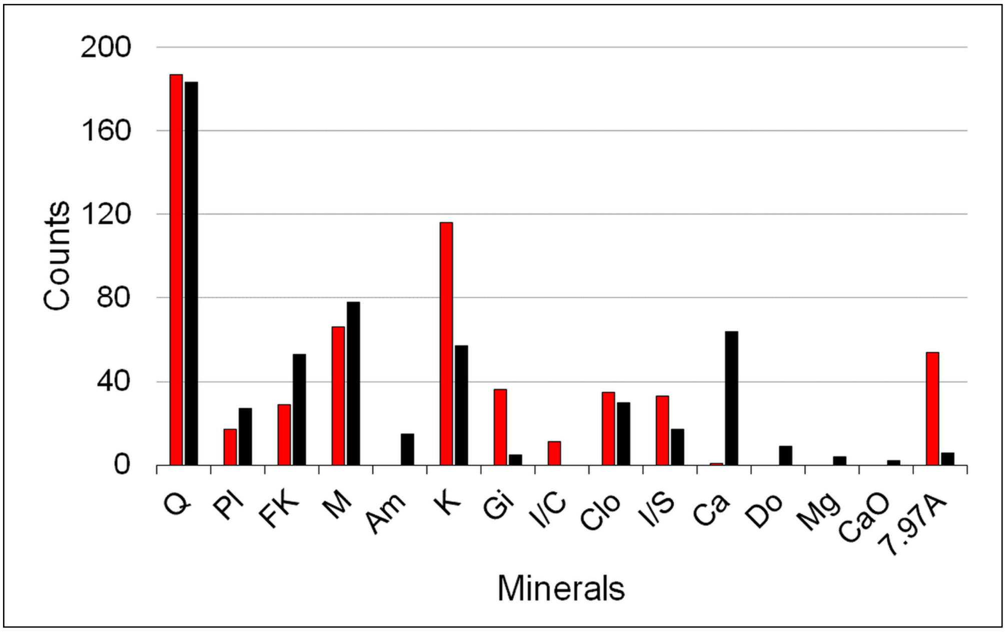

Figure 10 – Estimated relative abundance of bulk minerals identified in the general group of soils (red) and mortars (black). Q: quartz; Pl: alkaline plagioclase; FK: potassium feldspar; M: mica; Am: amphibole; K: kaolinite; Gi: gibbsite; I/C: illite/chlorite; Clo: chlorite; I/S: illite/smectite; Ca: calcite; Do: dolomite; Mg: magnesite; CaO: calcium oxide (peak 7.97 A was not identified).

All these mineralogical aspects (similar Q content, increased abundance of Pl, FK, M and Am and reduced clay components such as K, Gi, Clo, I/C and I/S) support the hypothesis of a mortar production practice in which less weathered, coarser soils with reduced clay content are preferred and where correction of grain size distribution by river sand is not as common as in current masonry. This form of soil selection may be due to: i) a reduced labour activity required by the extraction and treatment of river sand, and ii) the presence of coarse particles with better mechanical properties in less weathered soils. In fact, quartz particles from river sand are expected to have rounded shapes, whereas rock fragments are usually irregular, angular particles with a larger surface area.[37] This characteristic was confirmed in the mortars that were analysed by SEM-EDS (Figure 11). The images show that, in general, coarser particles do not have the typical spherical shape of river sand and are often rich in K, Ca or Na, as expected from alkali or potassium feldspars.

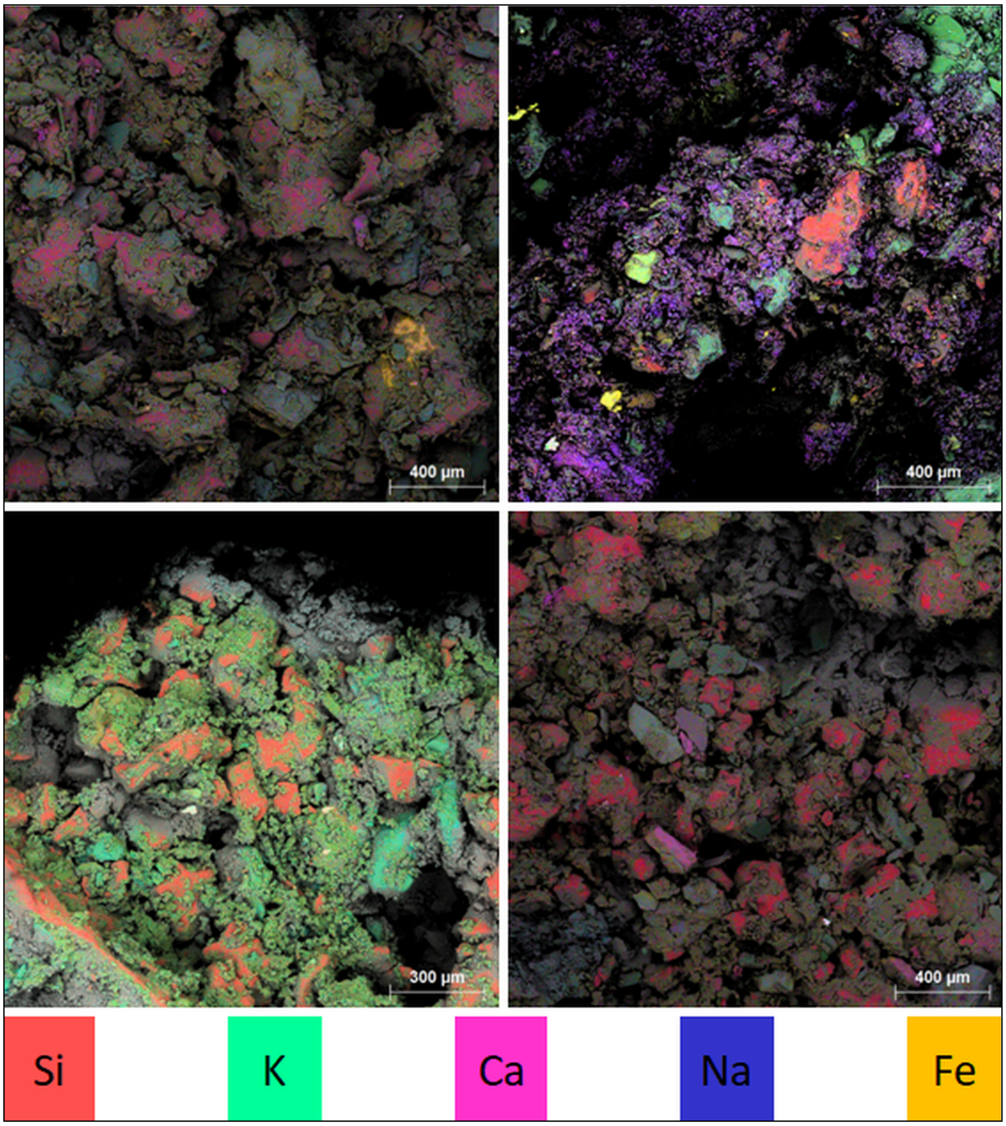

Figure 11 – SEM-EDS images of the following mortar samples: no. 25 (upper-left), 12 (upper-right), 34 (bottom left) and 00 (bottom-right).

CONCLUSION

The 47 mortar samples from the 19th century buildings in the *Vale Histórico Paulista* investigated in this study were all prepared using soil as the main component (except for a single mortar prepared using Portland cement).

Results show that the renders contain lime as binder at least in the outmost layer and its content tends to exceed that of plasters. Most lime-containing mortars (65%) were prepared with no more than 6% CaO in weight. This solution is in line with a strategy of sparing the use of this raw material, most likely due to its scarcity at the time of coffee production in the region. In particular, the present study revealed that pure calcitc lime was mainly employed in mortar manufacturing, a finding that was interpreted as a strong indication of the use of seashore calcium carbonate sources and thus of lime being imported from coastal areas. But the possibility of local production cannot be ruled out and is certainly a topic for further investigation. It was also found that unreacted CaO is rarely found in mortar samples, thus indicating competent ability in the manufacture of these protective coatings.

Although the use of additives such as organic substances or plant fibres in mortar manufacturing was ruled out, at the time of their production the adjustment of the texture was of concern. All mortars show a proportion of at least 70% coarse particles (up to more than 90%), with less than 10% of clay fraction. This is completely at odds with the usual texture of local soils, which are usually characterised by a more even distribution of sand, silt and clay.

This observation was corroborated by elemental data and statistical multivariate analysis, which revealed a separation of soils and mortars into quite distinct groups. Moreover, mineralogical screening of mortars and soils indicated that although almost the same minerals are found in both sets, the relative proportions are diverse. Mortars show an increased abundance of feldspars, amphibole and mica, and reduced contents of clay minerals such as kaolinite, gibbsite and others, while quartz tends to maintain similar levels. These results were interpreted as indicative of the use of a less common, less weathered and less clayish (thus coarser) source of soil to manufacture mortars, that is, soils compatible with the mineralogical profile found by the XRD analysis and confirmed by some SEM-EDS images, as discussed in section 3. This explanation implies excluding the process of soil texture adjustment with river sand (confirmed by the absence of polished spherical particles) and suggests that local traditional masons might have preferred to search for less weathered soils due to better mechanical performance associated with an aggregate phase made of irregular and angular particles with greater surface area.

These conclusions are an average overview based on the results obtained for the totality of mortars and may not reflect individual situations that deviate from the general trend. An investigation of specific cases, as well as the intercomparison of the overlapping layers of mortars found in the same walls or of plasters and renders of the same building, will be the subject of forthcoming papers.

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