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CHARACTERIZATION OF THE OLD RENDERING MORTAR OF A BRAZILIAN HISTORICAL BUILDING

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ABSTRACT

This paper studies the constitution and properties of lime-based mortars, collected in the *Casa Amarela* (Yellow House), a building of the end of the 19th century, located in Belo Horizonte (Brazil), object of this case study. An experimental study was developed to characterize the rendering mortar of this historic building, aiming to support future interventions/propositions of a recovery mortar compatible in physical, chemical and mechanical terms with the original mortar. The results of the investigation of this material are presented through its microstructural (scanning electron microscopy + energy dispersive spectroscopy and stereoscopic optical microscope), chemical and mineralogical (X-ray fluorescence, X-ray diffraction, scanning electron microscopy, and infrared spectrometry thermogravimetric) characterization. The detected phases were: quartz (SiO_2); calcite ($CaCO_3$) and microcline ($KAlSi_3O_8$). With the images obtained with SEM, it was possible to select microregions to target the EDS microanalyzer and perform semiquantitative chemical analysis. The microstructural and mineralogical characterization of the old mortar provided relevant data regard its composition, as well as its properties in the hardened state, which is closely related to the durability of the system. The results indicated a lime-based mortar without the presence of Portland cement or additions of organic materials.

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INTRODUCTION

In the last decades, studies of building materials and those dedicated to conservation and restoration have been emphasized, as well as those specifically concerned with the longest durability of edification (Santiago, 2007). There has also been a growing interest in the investigation of materials compatible with the conservation of historical buildings, in order to avoid radical interventions, losses and damages to valuable heritage structures (Kanan, 2008). Mortars should be adapted to each situation in view of the characteristics of the support, the type of building and its construction period, the climate of the region and the environmental conditions to which it is subjected, leading to the fulfillment of the functional and esthetic requirements (Veiga, 2003). Over hundreds of years, the lime was one of the most important materials in the construction and preservation of the traditional masonry (Santiago, 2007; Kanan, 2008).

Lime, sand and/or soil, organic additions and mineral admixtures were the most important materials used at that period (Kanan, 2008). On the other hand, the proportion lime: aggregate influences directly in the lime-based mortar's characteristics and performance, it must be compatible with historical building and sufficiently durable to insure the conservation and restoration of the building. The behavior of lime-based mortars also depends on physical (mortar's porosity, nature, shape and dimension of the aggregates) and chemical factors (type of lime, carbonation level, secondary compounds, dimension and shape of crystals) (Veiga, 2017). It is unnecessary for the replacement materials to be identical to the old ones, during repairs and replacement, partial or total, of lime-based mortars, plasters and finishing coats. However, it is needed to be compatible, it means, the old and new materials must conciliate physicochemical and esthetic properties (Kanan, 2008).

The compatibility principle states that the materials used for restauration must, whenever possible, show physical and mechanical strength equal or lower than the building materials adjacent to the restoration, because that ones with higher strength put in risk the integrity of the historical materials, turning them vulnerable to the deterioration, for their own nature and constituents(Veado, 2008). According to Veiga (2017), in the production of mortars for restauration and conservation of historic buildings, it must be adopted: a proportion, in volume, lime:aggregate between 1:2 e 1:3; an enough quantity of water, to insure a good workability, without excess; a good particle-size distribution of aggregates; a careful mixing of the materials until they achieve the homogeneity; an application of several coating layers; a protection of the mortar against cycles of wet/dry and freeze/thaw; the avoidance of excess of soluble salts. In order to define the recovering mortar of an historic building, besides the place and time in which it was executed, the origin of the materials used and its composition, it is necessary to take into account the characteristics of the constituent materials, such as binders, aggregates, additions, fibers and soluble salts. The knowledge of the characteristics of old mortars is possible through chemical and physical analysis of intact samples collected from old mortars and plaster (Kanan, 2008). This way, the main constituents of these materials are identified and new materials are prepared to be applied. It seeks to solve existing pathological manifestations without causing damage to the building, due to the undesirable chemical and physical reactions that could occur with the use of different and incompatible materials. The determination of the binder is very important to understand the behavior of this old mortar and its mechanism of wear. This determination can be easy and direct, but often in the case of lime-based mortars, it is difficult to identify the fractions of the binder and the aggregate if the latter has the same chemical nature (calcium carbonate). The same happens with lime-based mortars containing hydraulic compounds (pozzolana, cement or hydraulic lime), since all three have similar chemical characteristics, complementary microscopic examination is required (Kanan, 2008).

Mortars must meet certain requirements when used for the preservation of historic buildings: good water behavior (resistance to penetration and not hampering their drying); having some mechanical resistance, but do not transmit high stresses to the support; not introducing soluble salts into the carrier; being durable (and contributing to the overall durability); not impairing the visual presentation of the architecture, or de-characterizing the building; relatively low modulus of elasticity; reduced susceptibility to cracking; good behavior to ice (when applicable) and to soluble salts present in the support (Magalhães and Veiga, 2005). Water (liquid or solid) and soluble salts can cause damage to lime mortars, as well as organic compounds can also cause deterioration. The crystallization of the salts by evaporation near the surfaces of the masonry can cause tension in the walls of the pores of a mortar or plaster. Repeated cycles of dissolution and crystallization of salts, over time, lead to a successive failure of the pore structure, causing visible damage to the material (Kanan, 2008; Veiga, 2017). In addition, efflorescence and moisture spots may indicate presence of salts (Kanan, 2008).

Lime-based mortars contribute to the quality of the environment due to some physical and chemical characteristics of the lime, such as alkalinity (pH greater than 11.5), which makes the environment more aseptic; and the white color, which clarifies the mixtures, making them more reflective to the sun's rays, transmitting less heat and reducing the artificial illumination (Guimarães, 1998). On the other hand, some disadvantages have limited the use of lime in restoration: it is a material made available in the market presenting impurities, inadequate hydration and conditioning, reducing its quality; it requires considerable technical quality by the workers, who do not currently have experience in handling with lime; due to lack of knowledge of the techniques by the workers, there is a tendency to add excess water to improve the workability, which causes greater retraction during the drying phase and reduction of the mechanical strength; it requires a longer setting time than cement mortars, especially in situations of extreme moisture, requiring longer construction times (Veado, 2008). The aim of this work is to characterize an old rendering mortar, through analysis of samples obtained during the work of restauration of a building of the end of 19th century, evaluating the physical, chemical and mineralogical constitution of this material, through laboratorial tests. Besides, it was expected to subsidize future interventions/proportions of a recovering mortar compatible in physical, chemical and mineralogical terms with the original mortar, to assist professionals involved in works of restauration of historic buildings. As in the mentioned time the use of Portland cement was very restrict in Brazil yet, it was necessary to confirm if the binder material used in that old mortar was Portland cement or only lime.

MATERIALS AND METHODS

The methodology utilized in this work, to characterize old mortars, starts delimiting the aiming of the analysis in issue, it means, showing up the case study of the restauration of the *Casa Amarela* (Yellow House) – UMEI Timbiras (Belo Horizonte, Minas Gerais, Brazil) from where it was obtained the samples of old rendering mortars utilized in the laboratory tests.After that, the methods and instruments used to characterize these mortars are presented, which are based essentially on the application of physical, chemical-mineralogical, infrared and thermogravimetric analysis techniques. The microstructural and mineralogical characterization of the old mortar constituents provides relevant data in relation to its composition, as well as its properties in the hardened state, which is closely related to the durability of the system.

The building: The building of approximately 300 m², originally built to house the residence of the family of Judge Amadeu Alves da Silva, is located in a residential area of traditional and wealthy families. Its architectural design is authored by the architect and designer Edgard Nascentes Coelho and was approved by the City of Belo Horizonte on November 6th, 1899. It is made of solid brick masonry and has only one raised floor. Some modifications to the building were approved and made in 1935: a balcony and a garage, both designed by an Italian architect, Francisco Farinelli.

The cultural asset of the *Casa Amarela* (Yellow House) occurred on November 10th, 1994 and ratified on January 19th, 1995. From then on, the property was abandoned, with the house occupied by homeless people. On January 11th, 2000, the building caught fire. The fire destroyed part of the property, such as: part of the roof, wood floors, and windows. However, the walls were preserved. In June of 2008, the Municipal Prefecture of Belo Horizonte made an inspection to

evaluate the current situation of the property, as well as the necessary interventions for the elaboration of the architectural project and restoration of the building. During the retrofit and restoration works of the *Casa Amarela* (Yellow House) – UMEI Timbiras (Figure 1), located in Belo Horizonte (Minas Gerais, Brazil), which was inaugurated in 1987, it was necessary to repair the plaster mortar on the masonry of the facades. As it was a late-nineteenth-century construction, it was necessary to characterize the original mortar in order to identify the type of binder and aspects of sand used at the time of construction. From this practical necessity of reproduction of an old mortar, using current materials compatible with those of the original mortar, the search of knowledge that could elucidate the solution of the presented problem increased. It is also worth noting the importance of the knowledge and the in-depth analysis of the characteristics of old rendering mortars, when recovering, for the proposition of adequate solutions in each case, thus avoiding the appearance of new pathological manifestations of the incompatibility between the original material and the one to be applied.

The photos of the property, used in this section, prior to restoration, were all obtained from the archives of the Municipal Prefecture of Belo Horizonte (work place of one of the authors of this paper and of the master's dissertation that gave rise to it). As the restoration had already begun, when this study started, it was impossible to take pictures of the previous situation of the building. The first option to be considered in the interventions of historic buildings should be the conservation of the existing rendering mortars, using punctual repairs, if necessary, or even consolidation operations, depending on the value of the cultural property or coating. If there is a need for partial or total replacement of existing mortars, suitable mortars should be chosen for that specific use, and for this, certain functional and esthetic requirements must be verified. What has been seen so far in the building, however, is that it is often chosensolutions without scientific basis, without the knowledge of the characteristics of the selected mortar and without the clear control of the requirements to which the new mortar must comply (Veiga, 2003). Thus, this characterization aims to provide more real and qualifiable data to the recovery process of this coating.

In general, in the studied building, the internal rendering mortars were in poor condition. Most of the mortar of the surfaces was disintegrated, due to its direct exposure to weather conditions. In spite of this, small regions could be perceived with a special treatment in the painting, by the identification of the different pictorial layers and decorative treatment. In the kitchen, the ceramic coating was not complete, it had some broken parts, unsuitable filling with white cement mortar and some dirt, and the same thing happened in the bathroom, where there was a blue ceramic coating, certainly done in recent times. Despite the direct action of the weather, the facades adjacent to the public paths presented the surfaces in a reasonable state of conservation. However, it was observed humidity spots, dirt, missing parts of rendering mortar, structural cracks, superficial cracks, and still detachment of the painting in some regions. The facade facing the internal courtyard did not present the plastering mortar, having the masonry apparent.

It was noticed that the abandonment of the building intensified the problems and due to the characteristics of the mortar, a generalized detachment was generated in all internal and externalwalls of the edification. As the system is composed of solid brick, the recoating, with a mortar with characteristics similar to the time of construction, was an emergency.

Sample preparation: It was collected 12 samples from the old plaster mortar of the *Casa Amarela* (Yellow House) – UMEI Timbiras, presenting the following characteristics: irregular contour, with a flattened shape and some plan sides. The samples dimensions was around 15 cm x 8 cm x 5 cm and weight between 200 g and 815 g. In addition, theywere rough and heterogeneous, with grains that detached when touched, along with a fine powder. The plan faces presented a smoother texture, without grain detachment, besides thin layers of painting. In relation to the porosity, the samples presented many visible pores, with various diameters. From those samples, two of them (about 230 g) were put in the jaw crusher, followed by the homogenization and quartering, to realizer the characterization tests. The contents of these four portions had the following destination: the first and the second ones (1/4 + 1/4), totaling 113.52 g, were reserved for the tests; the third portion (1/4), weighing 55.77 g, was reserved to be powdered and used in the following tests: X-ray diffraction, X-ray fluorescence spectrometry, infrared spectrometry, thermal and chemical analysis; the fourth portion (1/4), weighing 55.33 g, was reserved for archiving. For powdering the third portion (55.77 g), a pan mill was used. The material was powdered for about one minute. The powdered content was divided into three portions, obtaining the following weight in each sample: Sample 1 – weighed 18.39 g; Sample 2 – weighed 18.69 g; Sample 3 – weighed 17.85 g. The sample to be tested in thescanning electron microscopy (SEM), previously chosen for having a flatter surface, was bondedto a brass base with a double-sided carbon conducting tape. Then, the sample was coated with carbon using the equipment: sputter coater type Spi-Module Carbon Coater. In order to make the surface of the old mortar sample as linear as possible, it was performed the vacuum inlay of two of its fragments in epoxy resin, in two round forms of silicone. After 24 h, with the hardening of the resin, the sample was cut with a low speed diamond saw, to obtain a flat surface, followed by the sanding of the two samples.

METHODOLOGY

To characterize the old mortar it was utilized the following methods and instruments:

- Semiquantitative or quantitative chemical analysis and determination of the elements present in the sample, as well as their concentrations: X-ray fluorescence spectrometry (XRF, variety WDS or Wave Length Dispersive X-ray Spectrometry) (instrument: Sequential X-ray fluorescence spectrometer, Philips brand (Panalytical), PW 2400 model);
- Qualitative analysis of crystalline phases (present minerals) of the sample and indication of the presence or absence of amorphous phase by X-ray diffraction (XRD) (instrument: X-ray diffractometer for powder samples, Philips brand (Panalytical), X'Pert-APD system, PW3710/31 controller, PW 1830/40 generator, PW 3020/00 goniometer);
- Investigation of the composition of the samples on the presence of organic and inorganic compounds through infrared spectrometry (IRS). A pulverized sample was

used with a tablet of KBr (Instrument: FTIR spectrometer, Shimadzu brand, IRAffinity-1 model);

- Obtainment of information of the thermal performance of the inorganic and/or organic phases composing the sample by thermogravimetric analysis(TGA) (instrument: Shimadzu brand, TGA-50model);
- Observation of three-dimensional samples, with greater or lesser relief by optical microscopy (instrument: Stereoscopic optical microscope, Leitz/Leica brand, model MZ6, with Canon digital camera, PowerShot S80 model);
- Scanning electron microscopy (SEM) with EDS microanalyzer to form images, being of two types: secondary electron image and backscattered electron image. In relation to the energy dispersive X-ray spectrometer (EDS), it performs chemical analysis on the microstructural scale, in points and microregions of the sample. In the microstructural analysis of the mortar sample, fractures and polished sections were used and in both cases, metallization with a very thin layer of gold was used (instrument 1: scanning electron microscope (SEM), JEOL brand, JSM-5410 model, with the energy dispersive X-ray spectroscopy (EDS) microanalyzer, Noran brand, TN-M3055 model; instrument 2: scanning electron microscope, FEI brand, Inspect S50 model and energy dispersive X-ray spectrometry (EDS) microanalyser, EDAX brand, Genesis model).

It was not possible to perform pull-off adhesion tests, because the collected plaster samples were no longer adhered to the substrate and the progress of the restoration work no longer allowed these tests.

RESULTS AND DISCUSSION

Physical characterization: The value found for surface area was 2.5 m²/g, which is relatively low. This value helps to eliminate the possibility of clay presence in the sample, which would imply much higher values. However, this value is consistent with the presence of sand and secondary calcium carbonate, as will be seen below, by the results of other methods. Through optical microscopy, it was possible to analyze the texture of the mortar, the presence of micro-cracks, particle-size distribution and the homogeneity or heterogeneity of the aggregates, as well as the petrographic identification of the sample. This method does not allow the identification of very fine fractions (Silva et al., 2011). The polished sections of the mortar that were previously prepared, as well as the three-dimensional fragments of the sample (Figure 2 and Figure 3) were observed with the stereoscopic optical microscope. In the Figure 2, white regions and dark sand particles are observed, both surrounded by a cream-colored matrix. According to Veiga (2017), these white regions may be related to: the particles of unhydrated quicklime; an inadequate blending of the hydrated powder or lime paste; or even the precipitation of calcium carbonate. These whitish nodules were also detected in the samples of old mortar studied by Silva et al. (2011), and the authors attribute this to a limited use of water during the lime hydration process. It was also noted a continuous matrix, with few details visible in the magnifying range, with a clear tone, almost white (Figure 3a and Figure3b), but also sometimes with a brown cream color (Figure 2). In the Figure 3a and Figure3b, it can be noted very angular particles, usually quartz (brown, sometimes darker). The clearer isolated areas are composed of calcite, while the darker particles are composed of iron (hematite) minerals. The clear matrix is composed of a fine mixture of quartz and calcite. Relatively thick particles in the range of 0.2 to 1.5 mm are observed, which are markedly angular (Figure 3a), while the rounded particles are clearly minority (Figure 3b).

Chemical and mineralogical characterization

Table 1 shows the chemical elements present in the old mortar sample, which were determined by XRF spectrometry. Moisture and loss on ignition (LOI), in turn, were analyzed by gravimetry. It is observed in Table 1 that the main chemical component present is silica (SiO_2), which is consistent with the great quantity of quartz mineral, the major component of the sand used in the mortar. Another silicate such as feldspar microcline also contributes to silica content; this mineral, plus part of the quartz, comes from the used sand with gneiss contamination. The second component is CaO, present in the sample as calcite ($CaCO_3$). The LOI value is also consistent with the presence of calcite, since this mineral alone presents 43.97% of LOI. X-ray diffraction is indicated to identify crystalline phases of the sample, especially when the size of the grains is very small. However, this method cannot be used to identify amorphous compounds. Moreover, neither the XRD nor the thermal analysis allow to accurately identify the trace of the mixture when there is a large amount of carbonate grains in the aggregate fraction Silva et al. (2011). From the diffractogram (Figure 4), the method allowed the identification of the following phases in the sample:

- quartz: SiO_2;
- calcite: $CaCO_3$, probably from the carbonation of lime;
- microcline (feldspar): $KAlSi_3O_8$.

The result is consistent with that observed by Silva et al. (2011) in their samples of old mortar. The authors suggested that the presence of this silicate phase indicates that the aggregate adopted in the mixture originates from a sedimentary source, it means, river sand, while the abundance of calcite is due to the use of lime as a binder. Thus, the calcite is the main responsible for the LOI of the sample; this fact is consistent with the results of XRD and TG. The low MgO content is related to the lime impurity. Iron oxides are associated with the iron minerals present in the sand, as well as other minor and low elements such as TiO_2 and MnO. The other elements, Al_2O_3, K_2O, and Na_2O, besides part of the silica, make up the feldspar microcline. The samples were analyzed by IRS (Figure 5) with Fourier transform infrared spectrometry (FTIR). The results obtained with the infrared spectrum corroborate with what was verified in the XRD, proving the presence of the following phases in the studied mortar: quartz, calcite and traces of kaolinite. In addition, no indicative ranges of organic compounds were detected, emphasizing that IRS is very sensitive to the presence of organic material. Thus, this is a strong evidence of the absence of organic compounds in the mortar sample.

Thermogravimetric Analysis (TGA): This method is very important for the study of phases that undergo thermal decomposition, such as many minerals and inorganic phases, as well as pyrolysis of organic polymers. Silva et al. (2011) complements that this test is very important in the mortar studies, to determine the nature and weight of the binder, as well as its hydraulic character. Thus, it was used to verify if in the sample of the old mortar there was the presence of organic material, besides minerals with loss of mass, due to the elevation of the temperature.

Table 1. Overall chemical composition of the old mortar (% by weight)

Moisture	LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	MnO	TiO_2	Cr_2O_3	Total
0.19	8.14	74.00	3.27	3.77	8.50	0.65	0.97	0.08	0.06	0.10	0.23	0.04	100.00

Table 2. Microanalysis referred to the regions marked in Figure 8

Area	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	Na_2O	TiO_2	CO_2	Mineral
1	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	N	Quartz
2	49.7	27.8	0.0	0.0	0.0	21.2	1.3	0.0	N	Microcline
3	23.4	24.2	39.2	0.5	9.9	1.7	0.0	1.0	M	Impurity with lime
4	2.8	2.9	24.4	0.0	12.4	0.0	0.0	57.5	M	Impurity with lime
5	0.0	0.0	0.0	0.0	100.0	0.0	0.0	0.0	A	Calcite
6	41.9	25.5	3.0	2.1	19.1	5.4	0.7	0.0	M	Impurity with lime
7	14.5	6.3	0.0	0.0	77.6	1.7	0.0	0.0	A	Calcite, quartz, microcline
8	7.6	5.2	0.0	0.0	86.3	0.0	0.0	0.0	A	Calcite, quartz

Table 3. Microanalysis referred to the regions marked in Figure 9

Area	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	Na_2O	TiO_2	CO_2	Mineral
1	97.9	0.8	0.4	0.0	0.5	0.4	0.0	0.0	N	Quartz
2	47.0	17.1	6.8	0.0	25.9	3.3	0.0	0.0	M	Quartz, microcline, calcite
3	7.5	3.3	1.0	0.0	87.3	1.0	0.0	0.0	A	Calcite, quartz

Table 4. Microanalysis referred to the regions marked in Figure 10

Area	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	Na_2O	TiO_2	CO_2	Mineral
1	6.3	1.3	0.0	0.0	92.4	0.0	0.0	0.0	A	Calcite, quartz
2	8.8	3.2	1.4	0.7	85.9	0.0	0.0	0.0	M	Calcite, quartz
3	100.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	N	Quartze
4	0.4	0.0	99.0	0.0	0.6	0.0	0.0	0.0	N	Hematite

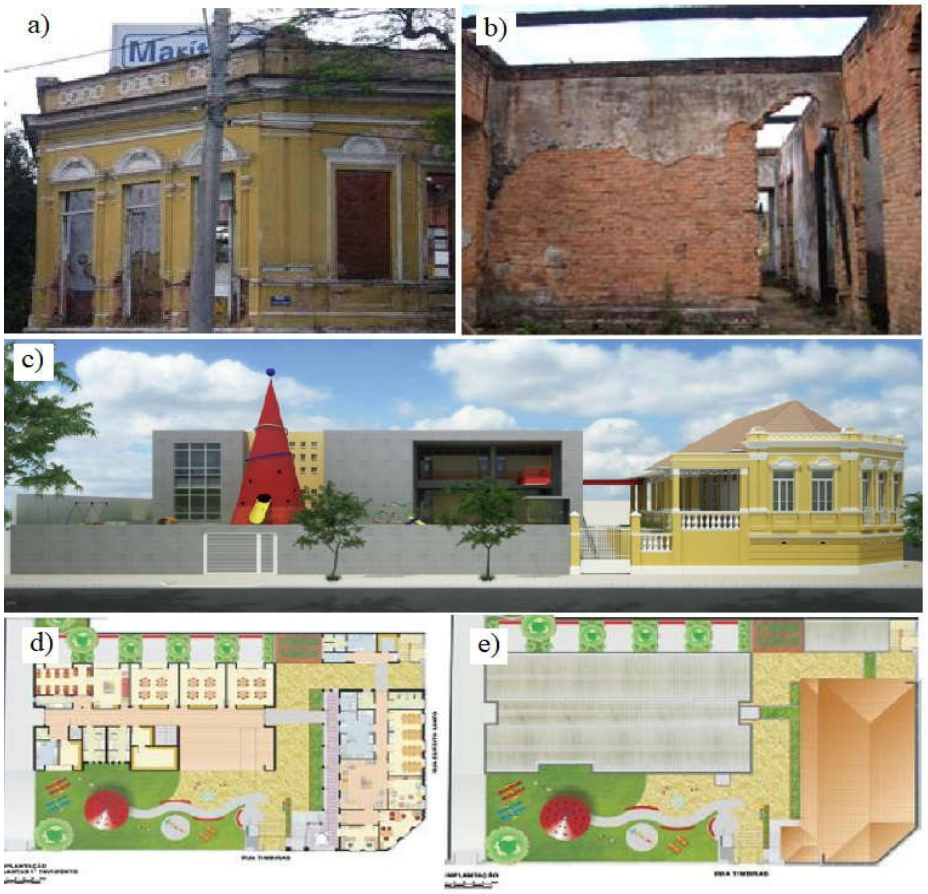
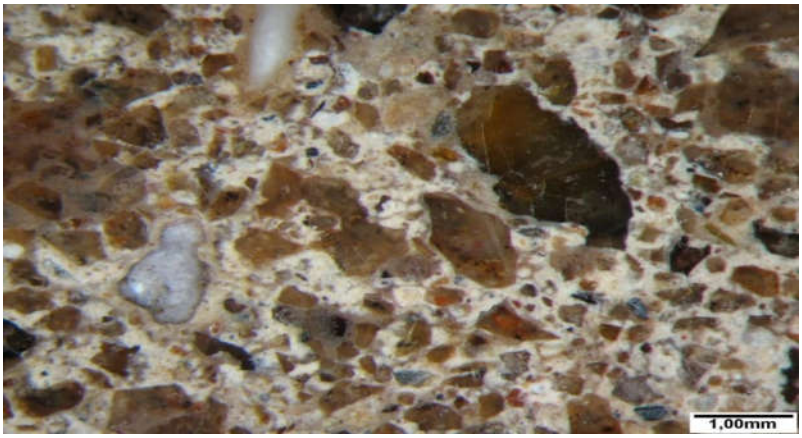
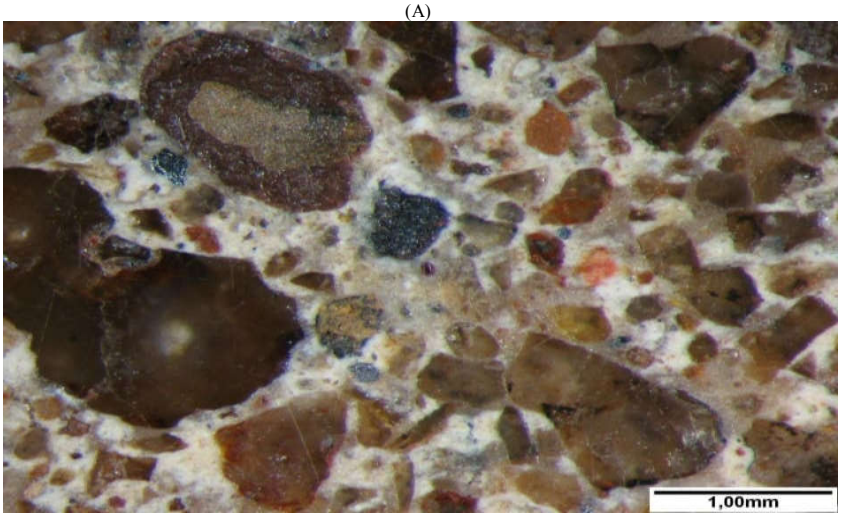


Figure 1. *Casa Amarela* (Yellow House) — UMEI Timbiras. View of the facade from Álvares Cabral avenue (a) Facade (b) Internal view (c) Design project of the recovery of the façade (d) Design Project of the 1st floor of the building and (e) Design Project of the 2nd floor of the building. Source: Rede Cidade (2008)

Figure 2. Three-dimensional fragment of the old mortar



(A)



(B)

Figure 3. Images ofstereoscopic optical microscope: (a) polished section 1and (b) polished section 2

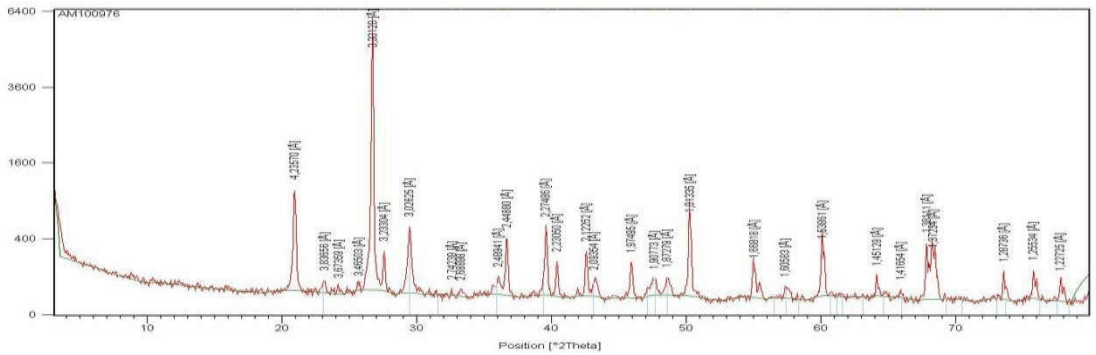


Figure 4. X-ray diffractogram of the old mortar

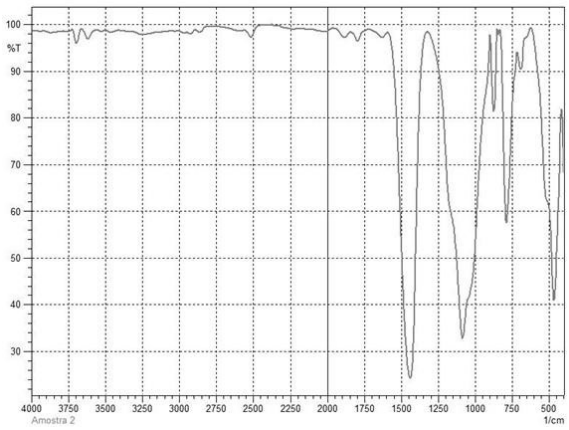


Figure 5. Infrared spectrum - old mortar sample. Transmission method with KBr tablet

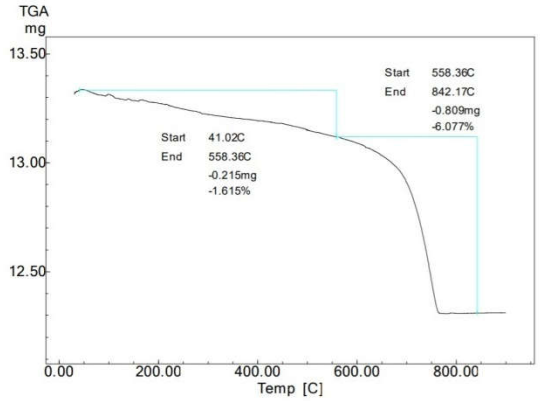


Figure 6. TGA of the samples of mortar, with the losses of mass indicated

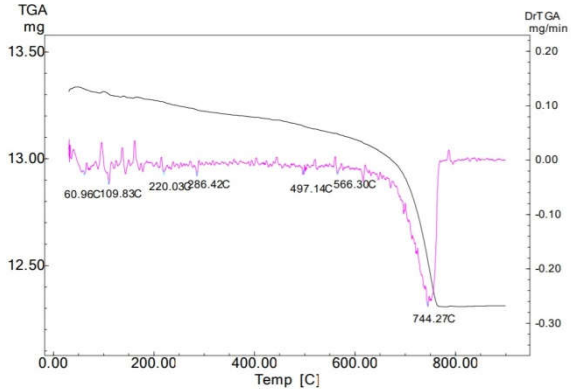


Figure 7. Differential Thermogravimetry (DTG) of the samples of mortar

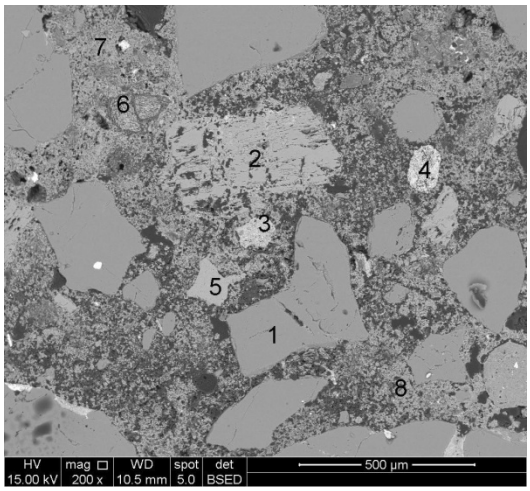


Figure 8. General microstructure of the mortar by SEM. Backscattered electron image (BEI) of the polished section

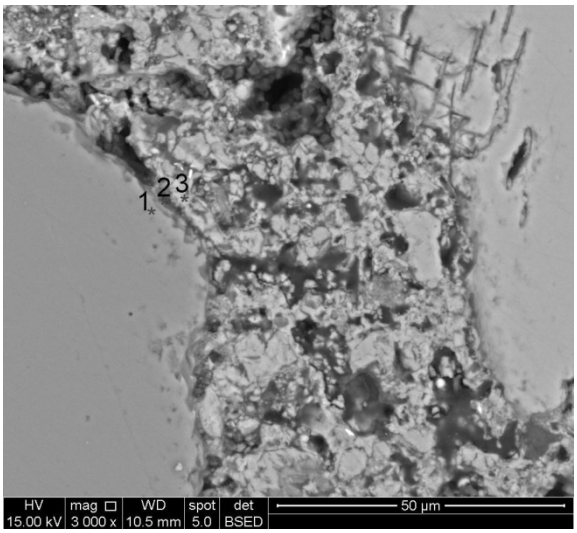


Figure 9. Detailed microstructure of the mortar by SEM. Backscattered electron image (BEI) of the polished section

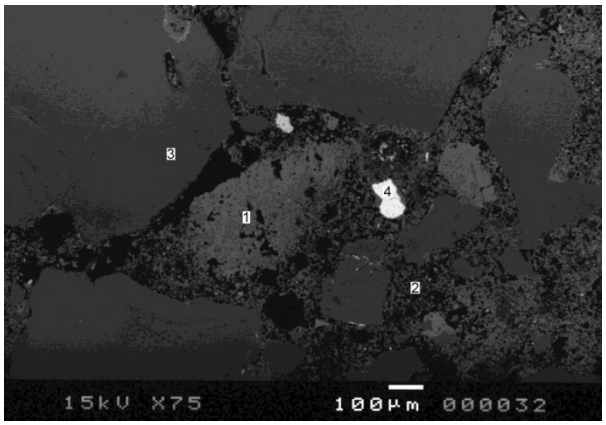


Figure 10. Mortar microstructure by SEM showing big particles of coarse quartz sand and the thin matrix. Backscattered electron image (BEI) of the polished section

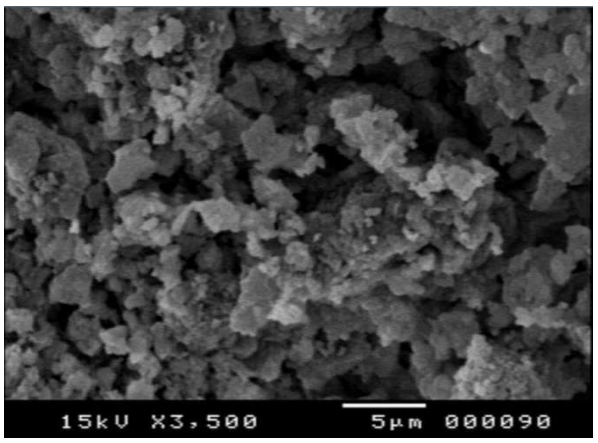


Figure 11. Detailed microstructure of the mortar by SEM, focusing on the thin matrix. Secondary electron image (SEI) of the fracture

It is a quantitative method, where it is possible to indirectly evaluate the hydrated or carbonated phases developed in the mortar, over time. In Figure 6and Figure7 it is verified a total loss of massof 7.7%, between the ambient temperature and the maximum temperature of 900ºC. Such loss can be divided into two parts or regimes:

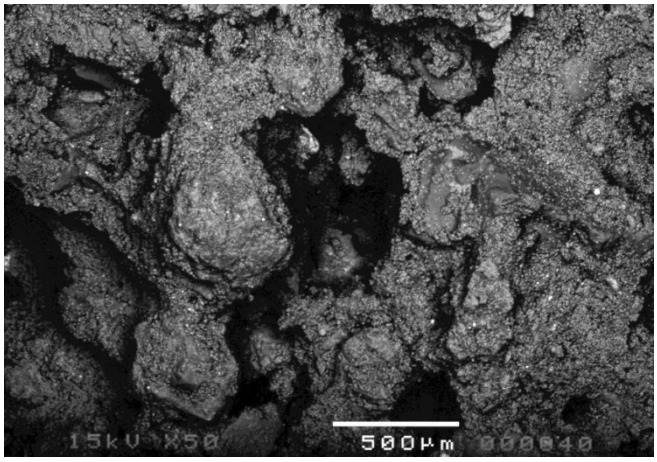


Figure 12. General microstructure of the mortar by SEM. Notably porous area. Secondary electron image (SEI) of the fracture

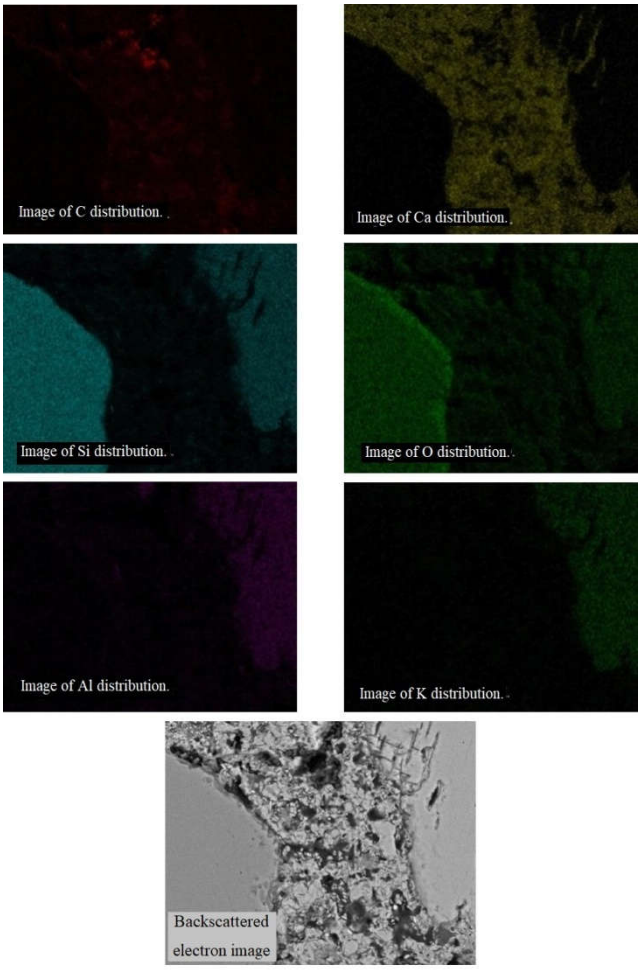


Figure 13. Area mapping of the matrix connecting two large particles (the same area of Figure9)

- A slight loss of 1.6% between ambient temperature and 558 °C, corresponding to the evaporation of moisture and adsorbed water. Similar to that observed by Silva et al. (2011);
- A significant loss of 6.1% between 558 °C and 842 °C, with a loss of mass peak at 744 °C; which corresponds to the decomposition of calcite ($CaCO_3$), with the emission of CO_2 (pyrolysis). Similar behavior was observed in the studies of Silva et al. (2011) at temperatures above 600ºC. In this case, this loss occurred in a temperature range lower than that of natural calcite, limestones and marbles, which shows this peak at a higher temperature (Brandão, 2010). This is evidence that the calcite present in the sample is not natural, it must be generated by the carbonation of the hydrated lime, which was present in the fresh state of the mortar.

Microstructural description: The microanalyses confirmed the identification of the minerals and phases present, already anticipated by XRD, global chemistry, IRS and TGA. In the EDS microanalysis, the carbon element was identified in the spectra, but is not quantified in the standardless analysis. Therefore, this element is correlated in the tables of microanalysis with letters that are estimates of its quantity: A = abundant; M = moderately abundant; L = low; N = null / unidentified. Microstructural and microchemical studies show that the mortar is composed of a coarse sand, with a composition mostly quartzous, it means, it is composed of high silica composed particles. This coarse sand also has a contribution of particles of gneiss origin, which is evidenced by the presence of microcline grains (feldspar) and traces of kaolinization. Iron ore particles were also observed, with hematite, goethite and impurities commonly associated with these minerals as manganese and titanium compounds. A notable aspect in the microstructure is the morphology of the coarse particles, which are highly angular (Figure 8, Figure 9 and Figure 10), while the rounded particles are clearly minority (Figure 9). This is an evidence that sand came from an eluvial deposit and not from an alluvial deposit.

Almost all of the sand particles are composed of quartz, that is, silicoses, for example, the region 1 in Figure 8. Microanalysis of the several regions in Figure8 are shown in Table 2. An intimate mixture of calcite and quartz (Figure 9 and Figure 11) currently forms the thin matrix, responsible for the adhesion of all group. The presence of calcite, restricted to the matrix (absent in the coarse particles) explains the clearer, even locally white, color of the matrix (Figure 2 and Figure 3). In the past, at the time of application of the fresh mortar, this matrix was composed of an intimate mixture of slaked lime (calcium hydroxide) and fine quartz sand, which was responsible for the setting of the material. Over the years, hydrated lime ($Ca(OH)_2$) has completely transformed into calcite (a phenomenon of carbonation, by reaction with atmospheric CO_2). This conclusion is reinforced by the absence of Portland cement and pozzolanic materials in the mortar. The particle size and crystallites of the matrix is actually very small, ranging from 3 µm to 15 µm (Figure 9 and Figure 11). Figure 9 shows the detailed microstructure of the mortar, focusing on the contact between coarse sand particles and the fine matrix. It consists on an intimate mixture of quartz (fine sand) and calcite (previously lime). Microanalyses of the various regionspresented in Figure 9 are shown in Table 3. The region 1 of the matrix (Figure 10) has a higher concentration of calcite (previously lime); region 2 is more typical of the matrix, composed of a mixture of calcite and quartz. Microanalysis of the various regionspresented in Figure 10 are shown in Table 4. Not only in the coarse particles, but also in the fine matrix, the degree of heterogeneity is very high (Figure 10) and the presence of mineral impurities is frequent. Figure 9 andFigure 11 illustrate these aspects, evidenced by microanalysis, indicating the occurrence of iron-rich impurities, with rarer phases rich in titanium, fragments of gneiss origin and areas richer in calcium. In its current condition, the mortar was quite friable, with fragments

detaching with little effort and showing porous regions; this is well evidenced in Figure 13. Figure 11 shows the intimate mixture of calcite and quartz, while Figure 12 shows the general microstructure of the mortar, showing coarse particles, generally angular and enveloped by the matrix. This area is notably porous, indicating the friable behavior of the mortar. The chemical and mineralogical compositions of the particles and the matrix are confirmed by the distribution maps of the chemical elements and by the backscattered electron image, as shown in Figure 13. These maps also show that the matrix, which forms the band between the two large particles, is rich in the elements Ca, Si, O and C, confirming that the matrix is composed of a mixture of calcite and quartz. Another interesting conclusion is that the particleon the right is composed of Si, O, Al and K, indicating that it is the mineral microcline, a feldspar of gneiss origin. The particle on the left consists only of quartz (Si and O).

Conclusion

The methodology used for the characterization of an old mortar, obtained from a building at the end of the 19th century, resorted to a diverse set of physicochemical techniques performed in laboratories, which were complemented, allowing a very reliable result. The use of these various laboratory techniques identified the compounds and elements present in the mortar sample of old plaster, besides its microstructural and morphological aspects. It is important to highlight that this paper focused specifically on the chemical and physical characterization of old mortars. It was not possible to perform mechanical tests due to the fact that the tests were performed on samples fragments and not on intact samples still adhered to the substrate. Through the mineralogical analysis performed by XRD it was possible to know the type of binder used in the old mortar. The XRF and SEM + EDS complemented the results, allowing characterizing, with reliability, the old mortar as a lime-based mortar. The XRD also permitted a mineralogical analysis, identifying the following crystalline phases present in the powder sample: quartz (SiO_2); calcite ($CaCO_3$); and microcline ($KAlSi_3O_8$). The well-defined chart peaks indicate that it is a crystalline material. The SEM verified as well, that the mortar is mainly composed of a coarse sand, whose composition is mainly quartz of gneiss origin. The IRS and the TGA discarded the presence of organic material in the sample. Based on the laboratory analysis carried out to characterize the rendering mortar of the *Casa Amarela* (Yellow House) – UMEI Timbiras, it was concluded that, when there is need for further intervention, a lime-based mortar would be the most advisable for restoration work in that historical building.

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