# Fernandina Wall of Lisbon: Mineralogical and Chemical Characterization of Rammed Earth and Masonry Mortars

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**Abstract:** This paper aims to provide information on rammed earth and masonry mortars of the Fernandina defensive Wall of Lisbon, Portugal, which was built in the second half of the 14th century. No previous information exists concerning the materials and construction techniques used in this defensive structure, which is essential to increase knowledge and to define requirements for a reliable conservation. An experimental campaign was carried out on samples extracted in nine different sections of this structure, which were analyzed by X-ray diffraction, thermogravimetry and wet chemical analysis. Calcitic lime was employed as a binder in the stone masonry bedding mortars and was also identified in the rammed earth samples. The results obtained allowed us to verify the use of different binders and aggregates, with heterogeneous binder/aggregate ratios, in different sections of the Wall. To reduce time for lime preparation and to achieve a durable lime matrix, most probably quicklime was used and hydrated together with the moistened earth to produce rammed earth and masonry bedding mortars.

**Keywords:** defensive structure; stone masonry bedding mortar; rammed earth; air lime; architectural heritage

## 1. Introduction

The construction of the Fernandina Wall in Lisbon dates back to the second half of the 14th century [1,2]. It was built in response to the need to ensure the city's protection in a period of continuous and intense growth, but also in response to enemies' attacks. There was a previous defensive wall in the city, called the Moorish Wall of Lisbon, that was no longer able to accomplish the desired protection of the city due to city enlargement (Figure 1) [3,4]. Therefore, king Fernando I of Portugal (1367–1383) decided that a new defensive wall was needed and ordered its urgent construction. In addition to the main structure of walls, this rampart included several towers, turrets (*cubelos*), entrances and small entrances (*postigos*).

The layout of the Fernandina Wall developed in line with the dense growth seen in various surrounding areas of Lisbon, in the 14th century. The Wall can, therefore, be divided into four main sections: the Eastern Section, the Western Section and two Marginal or Fluvial Sections, having a total extension of 4.69 km (Figure 1). These main sections delineated two large pockets which enclosed the city's existing old walls—namely, the Moorish Wall of Lisbon (which formed the central pocket) and the walls of Saint Jorge Castle (Figure 1). The Fernandina Wall is, thus, defined to the East by the Eastern section, the first to be built, with an extension of about 1.38 km, and to the West by the Western section, extending 2.05 km. To the North, the layout of the Wall is contained between the

Eastern and Western sections and, to the South, the Eastern and Western Marginal and Fluvial sections extend around 0.72 km and 0.54 km, respectively [2] (Figure 1).
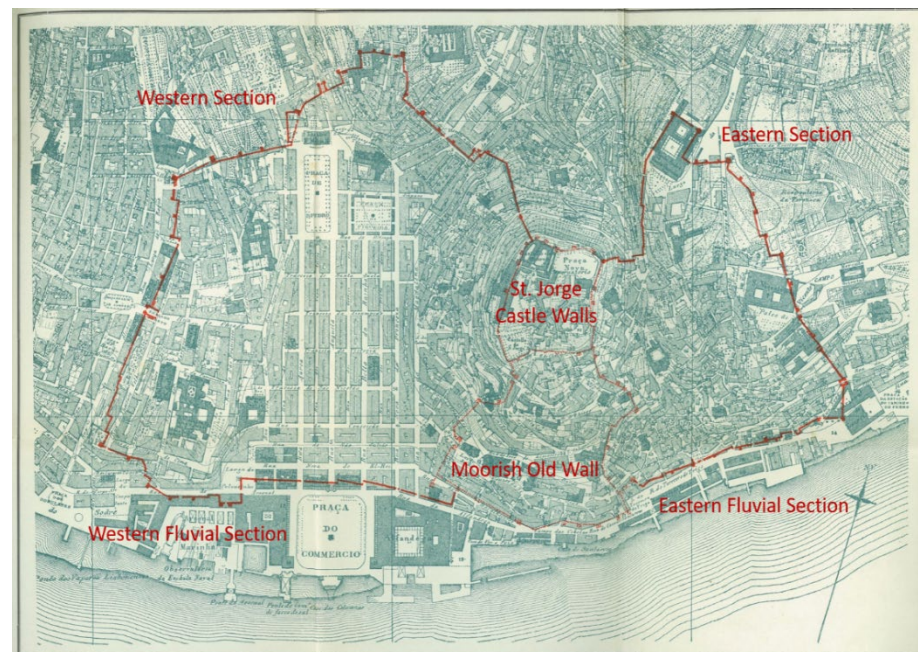


**Figure 1.** Surveying of St. Jorge Castle Walls, the Moorish Old Wall and the Fernandina Wall of Lisbon layouts carried out in 1956/58 (adapted from Vieira da Silva, 1987 [2]), with the location of its sections.

Regarding the type of construction and materials used to build the Fernandina Wall, the existing information is very scarce. According to Vieira da Silva [2], rubble stone masonry and rammed earth are the main materials. Complementary sections were composed by three-leaves, consisting of two parallel walls, each with a thickness of about 0.5 m, filled with a compacted earth core, presenting a total thickness of between 1.75 and 2.20 m.

Rammed earth (RE) consists of moistened earth compacted in consecutive layers within temporary frameworks, forming a monolithic wall. The chemically unstabilized RE technique functions with clay acting as the only binder; in stabilized rammed earth, other binders can be added to the earth mixture [5], generally to improve durability to weathering. With that aim, lime was frequently used to stabilize rammed earth in old structures with military and defensive purposes [6,7]. Therefore, the technique is called military RE. This technique was employed in several structures around the World, and in particular in the Iberian Peninsula, with still-existing structures such as the Castle of Paderne [6], the Juromenha Fortress [8] and the Silves Castle [9] in Portugal, and the Alcázar of King Don Pedro I [10], the Alcazaba or the ramparts of Málaga and the ramparts of Sevilla [11], the Castle of Atalaya [12] and the Monumental Complex of the Alhambra [13] in Spain. The compacted earth core of the three-leaf walls was similar to RE, but the exterior stone walls acted as permanent frameworks.

Geometrically, the Wall was composed of several elements that coordinated with the various sections of the walled structure, with prominence given to the imposing towers, turrets and gates. The Wall height varied depending on the type of architectural element used, 8 m being the most common, reaching a maximum of 15 m where the towers extend upwards [2].

According to Vieira da Silva [2], the Wall included 76 towers, one of the main constituent elements that played a key role in the city's defense, representing robust elements that, in most cases, exceeded the height and width of the adjacent walls. These towers were generally massive, except for those along the Eastern section flanking the river, some

of which are hollow inside, and each covered a base area of 8 m × 8 m. Most are now destroyed, but some were incorporated in buildings. In contrast, the smaller turrets (*cubelos*) covered a base area of 5 m × 5 m and, although of varying lengths, they never extended above the height of the Wall [14]. The turrets present three visible vertical surfaces projecting outwards and are attached to the Wall by their fourth side [15,16]. Some can still be found, mainly in the Eastern section, often integrated in residential buildings.

Being the main connecting axis between the entrances and exits of the city of Lisbon, the Fernandina Wall would have contained around 35 gates, in the form of robust, fortified large-scale entrances [2]. Many consisted of a square or rectangular walled enclosure with one or two openings, in the front and the back, the front-facing gate being embellished on both sides by defense towers. Many of these entrances were later demolished and turned into arches, often to overcome difficulties related to circulation and accessibility of the city [2]. The smaller gates (*postigos*) consisted of entrances located near one or two towers or between two turrets. They offered access to the city, facilitating security control in comparison with the larger gates. Not all were built when the walled structure was first constructed. Many were only opened after the construction was completed and are still visible today.

In 1910, the Fernandina Wall of Lisbon was recognized and classified as a national monument of public interest (Portuguese register: IPA PT031106120023). At present, remnants of the Fernandina Wall are still evident in several parts of Lisbon given the Wall's large-scale geometric characteristics, making it an important component of the city's architectural heritage.

The rapid growth in the rehabilitation of Lisbon's historical and urban center, and of its built heritage, is becoming increasingly noticeable. Faced with this trend, more rigorous and technical knowledge is required to ensure the conservation of archaeological heritage, namely that the materials and techniques used nowadays do not comprise the identity and originality of the old structures. It is, thus, of fundamental importance to consider the conservation interventions carried out to this day when proceeding with the characterization of the Fernandina Wall, in terms of its original materials and those used in subsequent interventions. Therefore, this article intends to collect and describe the interventions that were performed in some sections of the Wall over the past years, the description of some sections of the Wall, the collection of samples and the mineralogical characteristics that the materials employed in the construction of the Fernandina Wall of Lisbon present nowadays. That data is of fundamental importance in the conservation and maintenance of this type of architectural heritage.

## 2. Interventions in the Wall over the Last Years

The Wall has undergone several interventions in recent years, many of which have also targeted the old buildings confining it. Many of them are not (or not yet) documented. As a result, some sections of the Wall are now seen with new renders and plasters.

To the authors' knowledge, the interventions that were documented were carried out in the last 20 years. The first documented intervention took place in 1998, to the Western section of the Fernandina Wall, namely in the *Jogo da Péla* tower (Figure 2), near the *Martim Moniz* square. It was carried out by geologist Rui Brito in the context of his dissertation, in straight collaboration with the Lisbon City Council. It was intended to provide information on plastering and jointing mortars used between stone masonry blocks, as well as on the rocky substrate at the site of the tower. Laboratory tests revealed that the original mortars were mainly composed of air lime ($CaCO_3$) and silica sand. Based on a macroscopic examination, the stones used for the masonry were classified into four different types: two sandstones, one fine-grained and the other very fine-grained, and marl and shell limestones [17].

**Figure 2.** Aerial mapping showing the locations where interventions were carried out (identified by capital letters) and of the section's location on the Wall (identified by numbers). Notation: A—*Jogo da Péla* tower; B—*António Maria Cardoso* street; C—*São Vicente de Fora* parish; D—*Graça* parish; E—*Chafariz de Dentro* square; F—*Terreiro do Trigo* street; 1—Independency Palace; 2—*Calçada de Santana*; 3—*Calçada do Jogo da Péla*; 4—Rose Palace; 5—*Bragança* Terraces complex; 6—*Corpo Santo* Hotel; 7—*Gil Vicente* school; 8—*Graça* square; 9—*Terreiro do Trigo* street.

The 2014 intervention on *António Maria Cardoso* street, in the historic center of Lisbon (Figure 2), focused on identifying the conservation state of the Western section of the Wall. Recommendations were made in order to preserve and restore the original characteristics of the walled structure. Inside one building, rammed earth main walls were identified in a reasonable state of preservation, including the concavities of the wooden needles inherent to its construction. Extending outside the building, one of the sections of the Wall presented a severe state of degradation, mainly owing to the impact of atmospheric agents. Air lime mortar and small pieces of bricks were, respectively, used to fill the existing smaller and larger gaps. After filling these gaps, the walls were rendered with two layers of natural hydraulic lime (NHL) mortar, the first layer with a proportion of 1:3 (NHL: sand) in mass, and the second of 1:4. In addition, the cement mortar render found in the interior of the building was removed and replaced with NHL mortars [18].

In 2016, several interventions were carried out to the Eastern section of the Wall, particularly in the *São Vicente de Fora* parish (Figure 2). The first intervention arose due to the construction of sanitary facilities in a tourist accommodation that was in direct contact with the Wall. Rammed earth was proven to be the construction technique used in this section. Although a visual analysis of the wall did not provide information concerning the composition of the plasters, which were possibly the original ones, the notorious exposure to which the rammed earth walls were subjected was very evident. It was also observed that there were gaps at the base of the RE walls which, due to the lack of protection, accentuated the material degradation of the structure. Therefore, it was proposed to reinforce the stone masonry basement of the rammed earth walls with a bedding NHL mortar. The application of an NHL mortar of a color similar to that of the existing ones was additionally recommended, aiming to fill and repair cracks, holes and joints. Once the RE showed signs of moisture, it was suggested that a hollow brick masonry wall (coated from the inside with tiles or other similar coat) should be constructed between the Wall and the compartments in contact with it, to prevent future moisture problems [19]. However, if the air layer between

the RE wall and the indoor masonry wall is not ventilated, this intervention can introduce high levels of relative humidity in that layer and damage the RE wall.

In another intervention, carried out in 2016 in the same Eastern section of the Wall, a preliminary visit was conducted to the site located in *Graça* parish, currently occupied by a car park (adjacent to the *Gil Vicente* School North Wall, not far from *Graça* square), which allowed for the performance of a visual analysis and several archaeological surveys (Figure 2). Some RE sections were found to be coated with non-original cement-based renders and, at the very top of the wall, brick capstones applied long after its construction were observed. The archaeological surveys found traces of a small entrance (postigo) and, through the analysis of a west facing Wall sector, it was possible to establish the settlement of the first layer of RE in the site's geological substrate [20].

The archaeological interventions to the Fernandina Wall involved four surveys. In the first, carried out in the west wall of this Eastern sector, the rocky substrate that constitutes the Wall was visibly altered. A lengthier survey of the same location revealed that it was a turret, the third present in this section of the Wall. The second survey confirmed the heterogeneity of the material composition of the Wall, namely in terms of the rendering mortars, with different constituent elements. Beneath the render, the original structure of the Wall revealed an RE construction, which presented four distinct layers of varying composition and thickness. The Wall's constructive system was, thus, visualized and identified, noting the upper and lower limits of the formworks, the joints and some voids left by the formworks [8]. In the third and fourth surveys carried out in the northern section of this Eastern sector and inside a small opening, respectively, the existence of RE was once again confirmed, this time in a turret, and in a small entrance made of rigged limestone masonry.

During the archaeological and urbanistic works carried out between 2007 and 2008 in the *Chafariz de Dentro* square, excavations allowed access to the Eastern fluvial section of the Fernandina Wall. One of the two towers in this section was identified by observation of a stairway path along the top of the Wall. The Wall's foundation ditches were also uncovered by these excavation works. It was found that wooden planks had been used as formworks and that timber had also been used to build other elements, such as scaffolding and shores (props) [21]. Hence, it was possible to gain greater knowledge on the methods employed in the construction of the Wall.

In 2016, main walls of the Eastern fluvial section were discovered during rehabilitation works on buildings located on *Terreiro do Trigo* street (Figure 2). Concerning the RE walls, chipping was performed, which permitted identification of the formworks used, as well as the voids left by the formworks' pieces at the time of the Wall's construction. Several mortars were identified, mostly lime mortars with ceramic fragments, siliceous sand and calcareous aggregates. Nevertheless, some of the identified materials are non-original, being applied over the years, sometimes without the necessary compatibility with the original materials. In the north-facing section of this Eastern fluvial section, the internal part of the Wall was noted as being composed of irregular stone masonry with mortars with ceramic and calcareous aggregates, pebbles and bone fragments [22].

Many of the interventions carried out were archaeological, to document the site and structures, and did not involve the characterization of materials. Therefore, it was intended to proceed to a material characterization of the employed mortars and the RE. In this paper, a chemical and mineralogical characterization is presented. In a forthcoming paper, it is foreseen to present a physical-mechanical characterization.

## 3. Samples Collection and Localization

Sections of the Wall are scattered throughout the city of Lisbon, with the authorization and support of the Directorate General for Cultural Heritage (DGPC) and Municipal Council of Lisbon. The intervention works described on Section 2 allowed for a preliminary assessment of the visual features of the Fernandina Wall in different sites.

Nine of these (Figure 2) were analyzed in the present work and are referred to in the following sub-sections.

### 3.1. Palácio da Independência (Independency Palace)—Masonry Wall

Built in 1467, the Independency Palace, classified as a national monument, is located in *São Domingos* square, just off *Rossio* square, at the very heart of the city of Lisbon (identified by "1" in Figure 2). This national monument is of great historical and political importance and houses several institutions and museums.

At the north of the palace, there is a garden, where a section of the Wall is located, of about 20 m length, and a staircase (most probably from a former round path) at its top. This remnant belongs to the Western section of the Wall, composed mainly by a rubble stone masonry intensely exposed to atmospheric action.

This may explain the degradation of the surface of the southern exposed Wall (Figure 3a), which lacks protective render and joint mortars. The various interventions carried out to this section, including the inclusion of the staircase and repair works using, eventually, inappropriate mortars, may also have contributed to the deteriorated conservation state of this original Wall section.



**Figure 3.** Case studies in Wall Western section: Independency Palace (**a**), *Calçada de Santana* (**b**), *Jogo da Péla* (**c**) and Rose Palace (**d**).

### 3.2. Calçada de Santana—Turret

This turret is integrated in a private building located on *Calçada de Santana* (Figure 2). The creation of this *Calçada* (name meaning sloping street) involved the destruction, in 1676, of the *Postigo de Santana*—an opening in the Wall, located between the Santana turret and the entrance.

The turret in limestone masonry, with an implementation area of 5.5 m × 9.0 m and a height of 11 m (Figure 3b) [23], contains, at its top, a 36 m$^2$ terrace with walls facing east and west. In the past, elements such as chicken coops and flower beds diminished the authenticity of the structure. As previously mentioned, the Fernandina turrets were as high as the walls, i.e., about 8 m tall. The fact that the *Santana* turret is taller suggests than an extension was added at a later date.

### 3.3. Jogo da Péla—Tower

The tower of *Jogo da Péla*, placed in the sidewalk with the same name, is located near *Martim Moniz* square, an important landmark of Lisbon located between *Arco da Graça* street and *Calçada do Jogo da Péla* (Figure 2). It is one of the better-preserved elements of the Wall, found in the Western section. Nowadays, it holds no visible continuity with other sections, either to the west or to the east. This tower, made with limestone masonry and air lime mortar, is the only completely isolated example that still preserves some of its original features (Figure 3c).

Several works have been carried out on it over recent years, such as the demolition of nearby old buildings and the renovation of infrastructures. However, some new buildings were also built nearby, causing a major impact on the surrounding area.

Regarding the tower geometry, its height varies between 8 m to the east and 17.5 m to the west [17]. At the top of the tower, there is an excavated area of approximately 2.5 m × 3.0 m and about 4 m high, executed during the archaeological interventions mentioned by Leitão, in 2014 [24].

A variety of projects involving the integration and musealization of the tower, which is currently property of the Municipal Council of Lisbon, are underway.

### 3.4. Palácio da Rosa (Rose Palace)—Masonry Wall

Rose Palace, classified as a monument of public interest, is located on the Western section of the Fernandina Wall, near Lisbon´s *São Jorge* Castle (Figure 2). With its irregular L-shaped layout, the palace was architecturally modified and rehabilitated several times during the XVIII century [25].

A section of the Fernandina Wall in rubble stone masonry is present in the palace gardens (Figure 3d). Archaeological works being carried out in the palace exposed the foundations of the structure and discovered a tunnel dug inside the wall.

### 3.5. Terraços de Bragança Complex—Stone Masonry and Rammed Earth Walls

The *Terraços de Bragança* residential and commercial complex, located on Alecrim street (Figure 2), is composed of eight five-story buildings. Designed by the renowned Portuguese architect Siza Vieira, its construction began in 2003. During the construction works, a section of the Fernandina Wall was discovered and remains one of the most representative existing examples of the Western fluvial section of the Wall made in limestone masonry and RE (Figure 4a).



**Figure 4.** Case studies in Wall Western fluvial section: *Terraços de Bragança* Complex (**a**) and *Corpo Santo* Hotel (**b**).

### 3.6. Corpo Santo Hotel—Limestone Masonry

Located in the historic city center, close to the Tagus River (Figure 2), the *Corpo Santo* Hotel was built in 2015. During the archaeological works conducted previously to its

construction, a large number of archaeological remains, structures of the Fernandina Wall and surrounding structures were identified (Figure 4b) [26]. The most impressive is the *João Bretão* tower, built with limestone ashlar masonry. The concern with the preservation of Portuguese historical heritage by *Corpo Santo* Hotel has already been recognized with the achievement of several international awards.

### 3.7. Graças Square—Rammed Earth Wall

Located in the neighborhood of *Graça*, the square is one of the most visited touristic sites in the city of Lisbon. The square was outside the boundaries of the Old Moorish Wall; however, with the construction of the Fernandina Wall, it became part of the city and, thus, became protected. Remnants of the walled structure in RE are visible in the fire station, located close to the square, and in the square itself (Figure 5a).



**Figure 5.** Case studies in Eastern section: *Graça* square (**a**) and *Gil Vicente* school (**b**).

### 3.8. Gil Vicente School—Masonry and Rammed Earth Wall

The *Gil Vicente* School is located in *Graça* parish, one of the traditional neighborhoods of Lisbon, as previously mentioned. On the school north limit area, there is a 50 m long sector of the Eastern section of the Wall, well preserved and relatively protected (Figure 5b).

Several characteristic elements of the Wall can be found in this section, mainly walls and two turrets in RE, with the corners and base in regular limestone masonry [23].

### 3.9. Buildings Number 6–26 on Terreiro do Trigo Street—Masonry Wall

During the rehabilitation works on the buildings numbered 6 to 26 on *Terreiro do Trigo* street (Figure 2), several traces of the Fernandina Wall in rubble stone masonry were found, namely main walls, one tower and one small entrance. These buildings are located along the Eastern section of the Wall, close to Tagus River.

## 4. Materials and Experimental Methods

### 4.1. Materials

The samples were extracted manually or mechanically by core drilling, depending on the materials compactness. In five sites presented in Section 3, a total of 13 cylindrical cores were collected, namely in *Jogo da Péla* tower (3 cores), *Calçada de Santana* turret (1 core), *Gil Vicente* School (3 cores), *Bragança Terraces* complex (4 cores) and in the *Corpo Santo* Hotel (2 cores).

The terminology used for the samples' identification (Table 1) is based on the initials of the local designation: Independency Palace (IP), *Calçada de Santana* (CS), *Jogo da Péla* tower (JP), *Bragança Terraces* Complex (BT), *Corpo Santo* Hotel (CSH), *Gil Vicente* School (GVS), *Graça* square (GS) and *Terreiro do Trigo* street (TTS). The samples nomenclature also includes the identification of the Wall's element: M for main wall, T for tower, P for small gate (*postigo*) and C for turret (*cubelo*). Additionally, each sample was identified by a sequential number, which can include another number indicating the number of the specimen. For example, JP-T1-1 corresponds to the specimen 1 from sample JP-T1 collected in *Jogo da Péla* tower.

**Table 1.** Location of the samples collection and their identification.

| Wall's Section | Location | Samples Identification |
|---|---|---|
| Western | Independency Palace | IP-M1 |
| | *Calçada de Santana* | CS-C1 |
| | *Jogo da Péla* tower | JP-T1-1 |
| | | JP-T1-3 |
| | | JP-T3-3 |
| | | JP-T4 |
| | Rose Palace | RP-M3 |
| | | RP-M6 |
| Western Fluvial | *Bragança Terraces* | BT-T1-2 |
| | | BT-M1-3 |
| | | BT-M2-4 |
| | *Corpo Santo* Hotel | CSH-T2-1 |
| Eastern | *Graça* Square | GS-M1 |
| | *Gil Vicente* School | GVS-M1-2 |
| | | GVS-M2-2 |
| Eastern Fluvial | *Terreiro do Trigo* Street | TTS-T1 |
| | | TTS-M1 |
| | | TTS-P1 |

After collection, the samples identified in Table 1 were conditioned in a climatic room at a temperature of $20 \pm 2$ °C and 65% relative humidity. After, the samples were prepared analysis by X-ray diffraction, thermogravimetry and wet chemical analysis.

*4.2. Methods*

For analysis, two fractions were prepared as described by Santos Silva et al. [21]. A binder rich fraction, designated as BF, was obtained by disaggregating the sample, removing the aggregate particles without breaking them. The disaggregated portion of the paste was then milled and sieved through a 106 μm mesh sieve [22], and then stored. Additionally, to obtain the overall fraction (OF), a representative portion of the sample (about 20 g of the whole sample, without aggregate removal) was milled/grinded using a ball mill until all material passed a 106 μm sieve. After this process, the fraction obtained was homogenized using a spatula and was also stored.

A Philips PW3710 X-ray diffractometer was used to perform the mineralogical analysis. The XRD was operated at 35 kV and 45 mA with a scanning rate of 0.05° 2θ/s. The two fractions of the sample were used: the fine fraction, to determine the mineralogical composition of the binder, and the overall one, which includes all mortar constituents.

The thermogravimetric analysis and differential thermal analysis (TGA/DTA) were carried out using simultaneous DTA-TGA thermal analyzer Setaram TGA92. The samples of the overall fraction were placed in a platinum crucible and heated from room temperature to 1000 °C at a uniform rate of 10 °C/min under argon atmosphere (3 L/h). By this analysis, it is possible to evaluate the mass variations associated with dehydration, dihydroxylation and decarbonation processes, occurring at specific temperature intervals. Decarbonation occurred in the temperature range of 550–900 °C, which enables one to obtain the carbonates content, expressed as a percentage of $CaCO_3$, and which can be attributed, in the absence of limestone aggregates, to the carbonated lime content.

The siliceous aggregate content was obtained from the determination of the acid insoluble residue, which was performed by the attack of representative mortar sample with a hydrochloric acid solution with a ratio of 1:10 ($H_2O$:HCl). The sample used was previously disaggregated, in an amount of 10–15 g of disaggregated fragments, taking care not to break the aggregate particles; the existence of limestone aggregates and/or shells was verified, and removed manually whenever present. Furthermore, a visual analysis was carried out to record the consistency and presence of lime lumps, coal, ceramics and fibers,

among other materials. Previously to the acid attack, the samples were dried in a ventilated oven at a temperature of 105 °C, for a period of time not less than 12 h. The material obtained in the insoluble residue, plus the coarse limestone grains manually separated, were then placed in a RETSCH electromagnetic sieve shaker equipment, for 10 min, with a series of ASTM sieves (4.5 mm to 0.063 mm), to obtain the granulometric curves of the aggregates [27].

## 5. Results and Discussion

### 5.1. XRD Analysis

A total of 18 samples were analyzed; the results are presented in Tables 2–4. The mineralogical qualitative composition of the mortars (Tables 2 and 3) is rich in quartz, feldspars and mica, therefore, indicative of the use of siliceous sands [23]. The quartz and feldspar ratio is quite variable among the different case studies analyzed, which is an indication of the use of different sand pits. The diffractograms are presented in Figure 6 and Figures S1–S17 in the Supplementary Materials.

**Table 2.** XRD composition of mortars from *Jogo da Péla* and Rose Palace.

| Samples | | Quartz | Feldspars | Mica | Calcite | Dolomite | Gypsum | Calcium Aluminate Hydrate | Ettringite | Tobermorite |
|---|---|---|---|---|---|---|---|---|---|---|
| JP-T1-1 | OF | ++/+++ | tr | ? | +++ | - | - | - | - | - |
| | BF | +/++ | tr | - | +++ | - | - | - | - | - |
| JP-T1-3 | OF | ++/+++ | tr | tr | +++ | tr | - | - | - | - |
| | BF | +/++ | + | tr/+ | +++ | - | - | - | - | - |
| JP-T3-3 | OF | ++/++ | tr/+ | tr | ++ | tr | - | + | ? | - |
| | BF | +/++ | + | tr | +++ | - | - | + | + | - |
| JP-T4 | OF | +++ | +/++ | + | ++ | tr | - | - | - | - |
| | BF | ++ | ? | - | +++ | - | - | - | - | - |
| RP-M3 | OF | +++ | ++ | + | ++/+++ | - | tr/+ | - | - | - |
| | BF | +/++ | + | ? | +++ | - | tr/+ | - | - | - |
| RP-M6 | OF | +++ | ++ | + | ++/+++ | - | - | - | - | - |
| | BF | +/++ | + | ? | +++ | - | tr | - | - | tr |

Notation: OF—Overall Fraction; BF—Binder rich Fraction; +++ high proportion (predominant compound); ++ medium proportion; + low proportion; tr—traces; - undetected; ? doubts of the presence.

**Table 3.** XRD composition of mortars from Independency Palace, *Calçada de Santana*, *Terraços de Bragança* Complex, *Corpo Santo* Hotel and *Terreiro do Trigo* Street.

| Samples | | Quartz | Feldspars | Mica | Talco | Kaolinite | Calcite | Gypsum | Calcium Aluminate Hydrate | Ettringite | Weddellite |
|---|---|---|---|---|---|---|---|---|---|---|---|
| IP-M1 | OF | +++ | + | tr/+ | - | - | +++ | - | - | - | - |
| | BF | +/++ | tr | tr | - | - | +++ | ? | - | - | |
| CS-C1 | OF | ++/+++ | +/++ | + | - | - | +++ | - | - | - | - |
| | BF | +/++ | tr | - | - | - | +++ | ? | - | - | - |
| BT-T1-2 | OF | ++ | tr/+ | tr/+ | - | ? | +++ | - | tr/+ | tr | - |
| | BF | +/++ | tr | - | - | ? | +++ | - | tr/+ | + | + |
| CSH-T2 | OF | +++ | tr | tr | - | - | ++/+++ | - | - | - | - |
| | BF | +/++ | ? | ? | tr | - | +++ | - | - | - | - |
| TTS-T1 | OF | ++/+++ | ++ | + | - | tr | ++/+++ | ? | - | - | - |
| | BF | + | tr | tr | - | - | +++ | - | - | - | - |
| TTS-M1 | OF | +++ | tr | tr | - | - | ++/+++ | - | - | - | - |
| | BF | ++ | + | tr | - | - | +++ | tr | - | - | - |
| TTS-P1 | OF | +++ | +/++ | tr | - | - | ++/+++ | - | - | - | - |
| | BF | +/++ | + | tr | - | - | +++ | - | - | - | - |

Notation: OF—Overall Fraction; BF—Binder rich Fraction; +++ high proportion (predominant compound); ++ medium proportion; + low proportion; tr—traces; - undetected; ? doubts of the presence.

**Table 4.** XRD composition of the rammed earths from *Terraços de Bragança* Complex, *Graça* Square and *Gil Vicente* School.

| Samples | | Quartz | Feldspars | Mica | Kaolinite | Calcite | Dolomite | Anhydrous Calcium Silicates |
|---|---|---|---|---|---|---|---|---|
| BT-M1-3 | OF | +++ | +/++ | + | tr | ++/+++ | - | - |
| | BF | ++ | + | + | tr | ++/+++ | - | - |
| BT-M2-4 | OF | +++ | ++ | +/++ | tr | ++/+++ | - | - |
| | BF | ++ | +/++ | + | tr | ++/+++ | - | - |
| GS-M1 | OF | +++ | +/++ | +/++ | - | +++ | - | - |
| | BF | ++ | tr | +/++ | - | +++ | - | tr |
| GVS-M1-2 | OF | +++ | ++ | + | - | +++ | tr | - |
| | BF | +++ | +/++ | + | - | ++ | tr | - |
| GVS-M2-2 | OF | +++ | + | + | - | +++ | ? | - |
| | BF | +++ | + | + | - | +++ | - | - |

Notation: OF—Overall Fraction; BF—Binder rich Fraction; +++ high proportion (predominant compound); ++ medium proportion; + low proportion; tr—traces; - undetected; ? doubts in presence.
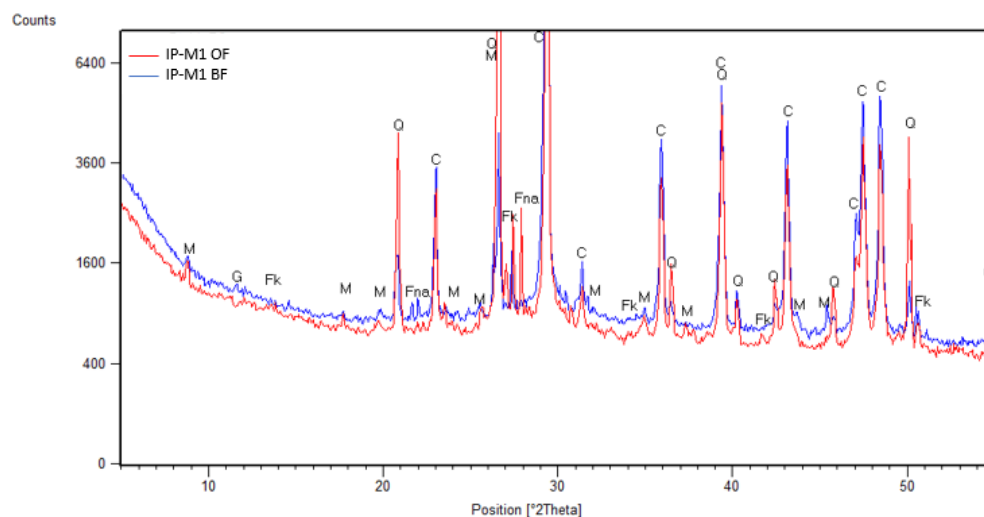


**Figure 6.** Diffractograms of sample IP-M1 (OF—overall fraction; BF—binder fraction). Notation: M—Mica, G—Gypsum, Fk—Potassium feldspar, C—Calcite, Q—Quartz.

Concerning the paste compounds of the mortars, the main compound detected was calcite (Figure 6), thus, confirming the use of air lime as a binder. Nevertheless, in some mortars, such as JP-T3-3, RP-M6 and BT-T1-2, some hydrated calcium silicates and calcium aluminates were detected, thus, suggesting the presence of a hydraulic binder. The origin of these hydraulic compounds is not possible to explain at this stage, but they could be attributed to recent interventions or due to the development of pozzolanic reactions between the binder and some siliceous constituents of the aggregates.

Mortars RP-M3, RP-M6 and TTS-M1 showed traces of gypsum, while BT-T1-2 present traces of weddellite; thus, both compounds indicated degradation processes [24].

Regarding RE samples (Table 4), the main compounds present are quartz, feldspars, mica and calcite. Hence, it can be confirmed that the use of siliceous aggregates also in the RE samples, most probably as earth component. The identification of calcite in the binder fraction confirms that the constructive technique used was the military RE [25], as the earth was stabilized with air lime. However, in terms of clayish minerals, such as mica and kaolinite, it can be seen the high variability of its proportion, thus confirming the employment of different earths in the areas analyzed, most probably picked in different

pits due to the urgent and fast building of the Wall (accomplished in very few years) and high raw materials consumption.

It is also worth mentioning the presence of anhydrous calcium silicates in the GS-M1 sample, compounds usually found in cement mortars. These silicates may be due to cement mortars that would have been applied in recent repairs, but that have presently been removed.

### 5.2. TGA/DTA Analysis

From the TGA/DTA charts obtained, presented in Figure 7 and Figures S18–S34 (Supplementary Materials), and according to the mineralogical compositions of the mortars, three mass losses ranges were considered [26] (Table 5):

i.    25 to ~200 °C—mass loss due to dehydration of free or absorbed water, and hydration and zeolitic water;

ii.    ~200 to 500 °C—mass loss due essentially to the dihydroxylation of clay minerals and of calcium silicate hydrates;

iii.    500 to 900 °C—mass loss due to the decarbonation of carbonates.
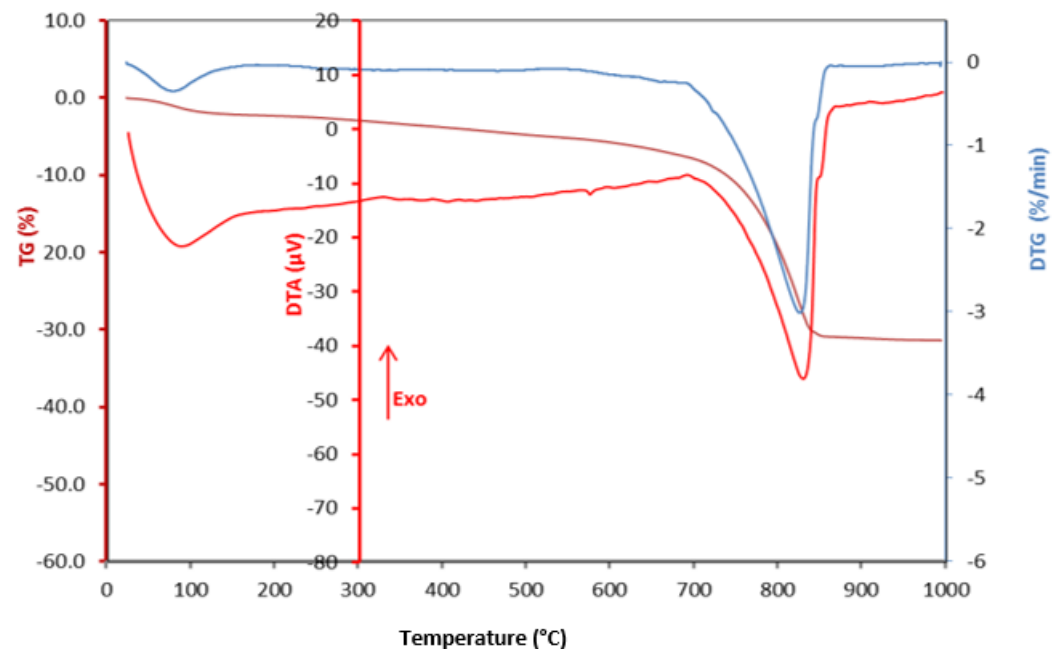


**Figure 7.** TGA/DTG/DTA charts of *sample* TTS-T1.

The TGA/DTA charts present a behavior in accordance with the mineralogical compositions previously obtained, with the main mass losses attributed to the dehydration of hydrated calcium silicates and calcium aluminates (e.g., samples JP-T3-3, BT-T1-2), and to the decarbonation of calcite and dolomite. The TTS_T1 sample showed the highest mass loss, which is essentially related to its highest value in the decarbonation zone (500 to 900 °C)—Figure 7.

Regarding the RE samples, the same three temperatures ranges were considered for TGA analysis (Table 6). The higher mass losses were obtained in the decarbonation zone, as expected. The mass losses regarding the range covering ~200 to 500 °C, and according to XRD data obtained, are essentially due to the dihydroxylation of clay minerals [27]. Therefore, it is confirmed that main losses are due to the presence of hydrated and carbonated minerals.

**Table 5.** Sample mass losses obtained by TGA for mortar samples.

| Samples | Mass Loss [%] per Temperature Range [°C] | | | Loss of Ignition * [%] |
|---|---|---|---|---|
| | 25 to ~200 | ~200 to 500 | 500 to 900 | |
| IP-M1 | 1.16 | 1.53 | 24.05 | 26.91 |
| CS-C1 | 2.35 | 1.91 | 18.85 | 23.15 |
| JP-T1-1 | 1.36 | 1.28 | 25.23 | 27.96 |
| JP-T1-3 | 1.09 | 1.09 | 24.50 | 26.79 |
| JP-T3-3 | 5.29 | 2.13 | 20.31 | 27.90 |
| JP-T4 | 0.72 | 1.23 | 20.51 | 22.50 |
| RP-M3 | 0.61 | 1.29 | 16.28 | 18.47 |
| RP-M6 | 4.33 | 4.20 | 6.95 | 16.59 |
| BT-T1-2 | 5.87 | 1.63 | 17.18 | 24.87 |
| CSH-T2-1 | 1.98 | 0.94 | 21.13 | 24.08 |
| TTS-T1 | 2.40 | 2.44 | 26.32 | 31.38 |
| TTS-M1 | 1.34 | 1.60 | 22.26 | 25.17 |
| TTS-P1 | 0.45 | 0.78 | 21.04 | 22.32 |

* mass loss obtained between 25 and 1000 °C.

**Table 6.** Sample mass losses obtained by TGA for rammed earth samples.

| Samples | Mass Loss [%] per Temperature Range [°C] | | | Loss of Ignition * [%] |
|---|---|---|---|---|
| | 25 to ~200 | ~200 to 500 | 500 to 900 | |
| BT-M1-3 | 8.21 | 2.12 | 7.22 | 17.84 |
| BT-M2-4 | 3.09 | 2.18 | 7.03 | 12.74 |
| GS-M1 | 0.69 | 0.54 | 9.17 | 10.38 |
| GVS-M1-2 | 3.71 | 3.27 | 20.32 | 27.68 |
| GVS-M2-2 | 7.27 | 4.91 | 18.46 | 29.64 |

* mass loss obtained between 25 and 1000 °C.

### 5.3. Aggregate Content and Grain-Size Distribution

Tables 7 and 8 present the results of the insoluble residue (IR), corresponding to the siliceous and the total aggregate contents, respectively. It appears that the values of IR are quite heterogeneous, ranging from 30–80% for mortars and 42–78% for rammed earth samples. The total aggregate content is quite high and is mainly due to the siliceous sand. The grain-size distribution of the total aggregate of mortar and RE samples is presented in Figures 8 and 9, respectively.

By analyzing Figure 6, it is verified that mortar sample RP-M6 is the one with the highest percentage of coarse particles (4.75 mm), while samples JP-T1-3 and IP-M1 are those with the lowest percentages. The JP-T4 sample presents the highest percentage of constituent material between 150 µm and 1.18 mm. Of the remaining samples, the grain-size distributions of the aggregates are very similar.

Concerning the grain-size distribution of the RE samples (Figure 9), sample BT-M2-4 presents the highest percentage of coarse particles among all others. Samples GVS-M1-2 and GVS-M2-2, although extracted in the same location, present different aggregate grain-size distributions, namely up to 300 µm, being similar in the coarse dimension. As each earth is composed by different types and contents of clay, silt and sand (apart from coarser aggregates), this suggests, as before, the use of different earths when building the RE wall. In turn, the GS-M1 sample contained the lowest percentages of finer and coarser particles.

**Table 7.** Insoluble residue and total aggregate content for mortar samples.

| Sample | Insoluble Residue Content [%] | Total Aggregate Content (IR + Limestone Aggregate) [%] |
|---|---|---|
| IP-M1 | 37.9 | 77.2 |
| CS-C1 | 49.6 | 59.0 |
| JP-T1-1 | 46.7 | 54.3 |
| JP-T1-3 | 30.5 | 56.8 |
| JP-T3-3 | 73.6 | 74.7 |
| JP-T4 | 56.3 | 57.7 |
| RP-M3 | 46.8 | 56.3 |
| RP-M6 | 79.0 | 82.0 |
| BT-T1-2 | 42.7 | 65.5 |
| CSH-T2-1 | 63.3 | 68.6 |
| TTS-T1 | 35.1 | 45.5 |
| TTS-M1 | 44.2 | 52.6 |
| TTS-P1 | 65.7 | 79.3 |

**Table 8.** Insoluble residue and total aggregate content for rammed earth samples.

| Sample | Insoluble Residue Content [%] | Total Aggregate Content [%] |
|---|---|---|
| BT-M1-3 | 59.2 | 65.6 |
| BT-M2-4 | 77.8 | 81.8 |
| GS-M1 | 56.7 | 74.7 |
| GVS-M1-2 | 54.7 | 59.0 |
| GVS-M2-2 | 42.9 | 61.5 |
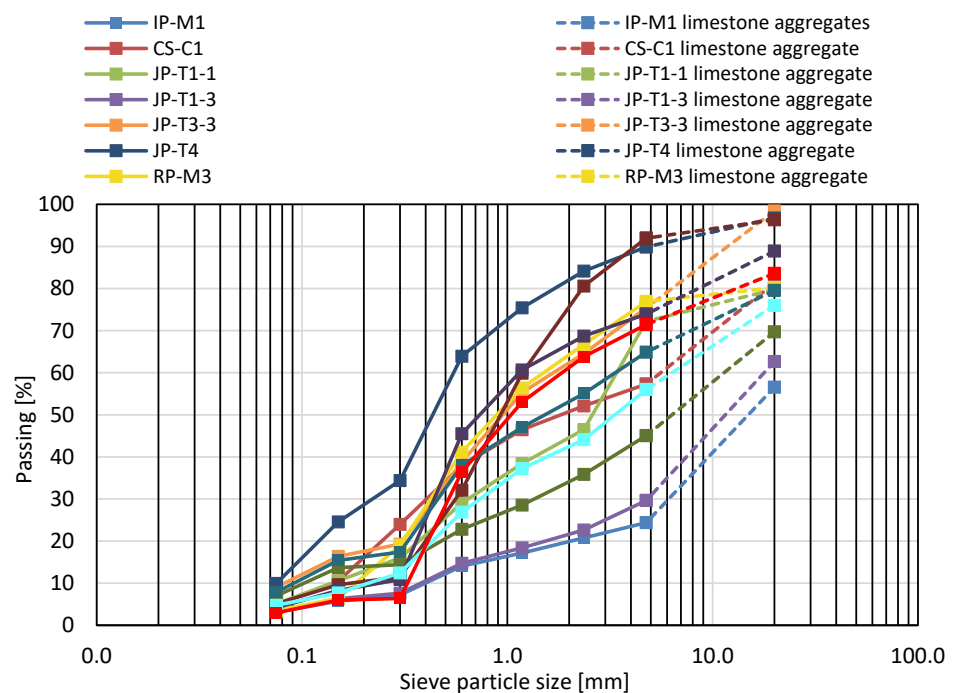


**Figure 8.** Particle size distribution of the aggregates in mortar samples.
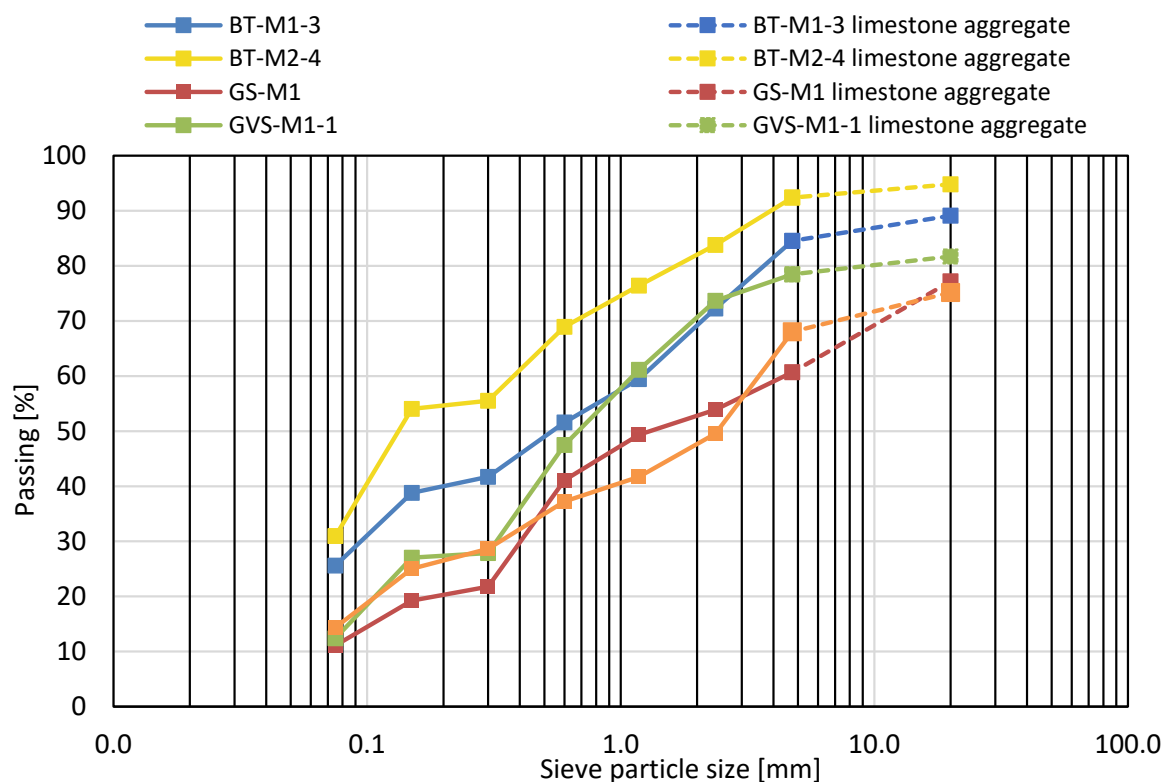
**Figure 9.** Particle size distribution of the aggregates in rammed earth samples.

## 5.4. Binder/Aggregate Ratio of Mortar and Rammed Earth Samples

Tables 9 and 10 present the mass contents of the different constituents and the binder/aggregate ratios [21] obtained for lime and RE samples, respectively. From these values, a great heterogeneity and diversity in constituents' contents are visible. Even within the same case study, important differences are present, thus, implying the employment of heterogeneous materials in the Wall's construction.

**Table 9.** Mortars constituents content and Lime:Aggregate (calcareous and siliceous separated or together) ratios.

| Sample | Content [Mass%] | | | Lime:Calc. Aggr.:Silic. Sand (in Mass) | Lime:Aggregate (in Mass) |
| --- | --- | --- | --- | --- | --- |
| | Siliceous Sand [1] | Calcareous Aggregate [2] | Hydrated Calcic Lime [3] | | |
| IP-M1 | 38 | 39 | 12 | 1:3.3:3.2 | 1:6.5 |
| CS-C1 | 50 | 9 | 25 | 1:0.4:2 | 1:2.4 |
| JP-T1-2 | 47 | 8 | 36 | 1:0.2:1.3 | 1:1.5 |
| JP-T1-3 | 30 | 26 | 22 | 1:1.2:1.4 | 1:2.6 |
| JP-T3-3 | 74 | 1 | 33 | 1:0.03:2.2 | 1:2.23 |
| JP-T4 | 56 | 1 | 34 | 1:0.03:1.6 | 1:1.63 |
| RP-M3 | 47 | 10 | 20 | 1:0.5:2.4 | 1:2.9 |
| RP-M6 | 79 | 3 | 10 | 1:0.3:8.2 | 1:8.5 |
| BT-T1-2 | 43 | 23 | 12 | 1:1.9:3.6 | 1:5.5 |
| CSH-T2-1 | 63 | 5 | 32 | 1:0.2:2 | 1:2.2 |
| TTS-T1 | 35 | 10 | 37 | 1:0.3:0.9 | 1:1.2 |
| TTS-M1 | 44 | 8 | 32 | 1:0.3:1.4 | 1:1.7 |
| TTS-P1 | 66 | 14 | 25 | 1:0.6:2.6 | 1:3.2 |

[1] Value corresponding to the insoluble residue content; [2] Value corresponding to the limestone grains manually separated during samples preparation; [3] Value obtained by TGA analysis.

**Table 10.** Rammed earth constituents content and Lime or Lime + Earth:Aggregate ratios.

| Sample | Content [%] | | | | Lime: Earth: Calc. Aggr: Silic. Sand (in Mass) | Lime + Earth: Aggr. (in Mass) |
| --- | --- | --- | --- | --- | --- | --- |
| | Siliceous Sand [1] | Calcareous Aggregate [2] | Earth Fines (Clay + Silt) [3] | Hydrated Calcic Lime [4] | | |
| BT-M1-3 | 59 | 6 | 17 | 7 | 1:2.3:0.8:8 | 1:2.7 |
| BT-M2-4 | 78 | 9 | 26 | 9 | 1:2.9:0.5:8.8 | 1:2.4 |
| GS-M1 | 57 | 18 | 8 | 2 | 1:3.7:8.1:25.7 | 1:7.2 |
| GVS-M1-2 | 55 | 4 | 8 | 31 | 1:0.3:0.1:1.8 | 1:1.5 |
| GVS-M2-2 | 43 | 4 | 8 | 31 | 1:0.6:1.14:3.2 | 1:2.7 |

[1] Value corresponding to the insoluble residue content; [2] Value corresponding to the limestone grains manually separated during samples preparation; [3] Value corresponding to the size fraction < 0.063 mm; [4] Value obtained by TGA analysis.

Furthermore, the visual observation has shown some lime lumps, thus indicating that to the moistened earth was added quick lime and then mixed, for hydration, resulting in RE stabilized with lime. Therefore, this mixture would allow for the aggregates surface to be attacked by the causticity of calcium oxide, leading to a better connection between the binder's matrix, earth and lime, and the coarser aggregates.

According to the results obtained, the lime content in the RE of the Fernandina Wall is very low when compared to the content of the aggregates and earth fines. This composition is also different from the composition employed in other RE structures, as is the case for the Castle of Paderne [6] in Portugal or the Walls of Cáceres [7] in Spain, where the lime content is much higher. This behavior may indicate that the use of lime was restricted in the sections of the Wall in RE, since there were other sections in rubble stone masonry more exposed to the action of water, or where there was a need for the construction to have more important defensive functions, in which lime was more necessary. On the other hand, it should be noted that the construction of the Wall took only two years, which required a great effort in terms of the construction materials to be used, which certainly also limited the use of lime in those sections of RE construction.

## 6. Conclusions

The existent rammed earth (RE) buildings and, particularly those in military RE, are an integral part of Lisbon's military, cultural, social and architectonic heritage. Hence, for their effective conservation and preservation, knowledge of the original techniques and materials employed is imperative. Nevertheless, and due to a neglect on these structures, it is also fundamental to have adequate information of the conservation interventions held over the years, namely to learn about the original characteristics, to ensure that previous conservation mistakes are not made again and that good practices can be disseminated.

The Fernandina Wall of Lisbon is an impressive defensive structure that was constructed in the second half of the 14th century. Its construction was carried out in a very short period of time, between 1373 and 1375, using limestone from the Lisbon region, air lime and earth. Several elements are visible in the 21st century around the old quarters of Lisbon, attesting to its greatness and durability, as its main walls, towers, turrets or former gates.

This work was focused on collecting and analyzing information about past interventions, its conservation state and the general composition of some Wall's sectors. Masonry mortars and RE samples were extracted from nine different sites of the Fernandina Wall, and mainly its chemical and mineralogical characterization assessed.

The sites analyzed in the Western Wall section are manly constituted by rubble stone masonry, with the exception of the site closer to river Tagus in Bragança Terraces Complex, where both RE and limestone ashlar masonry were used. The limestone ashlars were also used in the eastern fluvial section of the Wall, where stone blocks were layered with a lime mortar. In opposition, in the Western section of the Wall, the RE was the main material used, while the rubble stone masonry was the most used material in the Western fluvial section.

Calcitic air lime was the binder used in masonry mortars. In some sites, traces of hydraulic binders were also found, their presence being attributed to 20th century interventions. With few exceptions, the grain size distribution of the aggregates is similar. However, their compositions vary according the wall sections analyzed.

RE samples shows also the use of air lime as additive, confirming that the construction technique used was the military rammed earth. Although, these RE are poorer in lime content when compared with others RE used in Iberian Peninsula in the same period.

As in the case of sands used for masonry mortars, the use of earths of different composition was also found in RE. Besides, siliceous sands and coarse limestone aggregates were also found in RE sections of the Wall. The heterogeneous grain size distribution of the aggregates confirms the use of different earth sources in the RE manufacture. One RE sample also contains traces of the use of a hydraulic binder, an indicator of 20th century repair works.

This work shows that materials with different composition and binder:aggregate ratios were employed in the construction of the Fernandina Wall of Lisbon. This variety and heterogeneity is observed not only in the different elements or Wall sections, as well as in the same site analyzed. That may be due to the limited construction period and the need of large contents of raw materials. The use of both RE and rubble stone sectors in different sections of the Wall can be due to the availability of materials, but also with the professional labor skills that were built the defensive Wall.

It is expected that the information obtained in this work about the composition and formulation of bedding mortars and RE used in the construction of the Fernandina Wall of Lisbon will be a contribution to carry out future conservation interventions properly supported and guarantying the compatibility with the original materials.

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