Characterization of Renders and Plasters from a 16th Century Portuguese Military Structure: Chronology and Durability

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Abstract

In coastal areas of Portugal, many historical buildings and fortresses still exist that were constructed in masonry with aerial lime mortars. These mortars are often found to be in good condition, showing appropriate cohesion and adhesion to the background, although usually they show surface degradation as a result of the aggressive marine environment.

The aim of this paper is to present and characterize the mortars used for the renders and plasters of one of these fortresses, that of *Nossa Senhora da Luz* which was constructed and modified over several centuries. The study of the mortars permitted to identify a construction period chronology improving the knowledge of the changes in formulation of the aerial lime mortars thus allowing to select compatible repair solutions.

Keywords: Aggressive environment; Preservation; Conservation; Fortress; Aerial lime mortar.

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1 Introduction

In Portugal, a country having a relative extensive coast there are several historic buildings located near the sea. These are subjected to both physical and chemical deterioration processes which can accentuate the deterioration of their renders that in these buildings are base on traditional aerial lime mortars, often found in a very good state of conservation [1, 2, 3, 4, 5, 6].

Within the scope of an intervention on the Fortress of *N. Sra. da Luz (Our Lady of the Light)*, located on the Cascais coast, a study of the mortars, including their characterization and condition, was carried out by the Laboratório Nacional de Engenharia Civil (LNEC).

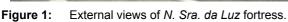
This Portuguese military structure, probably dating from the 15th century, is one of the oldest monuments in the municipality of Cascais. In its interior the old *Tower of St. António* can be found, also known as the *Joanina Tower* because it was built by order of D. João II, in 1488. In 1580, the tower was transformed into a fortress with three Bastions (Fig. 1) [7, 8].

The plan of this fortress is considered unusual in Portuguese military architecture (Fig. 2) [7, 8] and was constructed within the defensive coast-line program.

With the 1755 Lisbon earthquake part of the fortress was damaged, including the *Joanina Tower*. After this, the structure suffered major architectural transformations [7, 8] and was surrounded by walls.

Currently, the fortress belongs to the Municipality of Cascais, who developed a recovery and revitalization project for the monument with the objective of turning it into a museum.







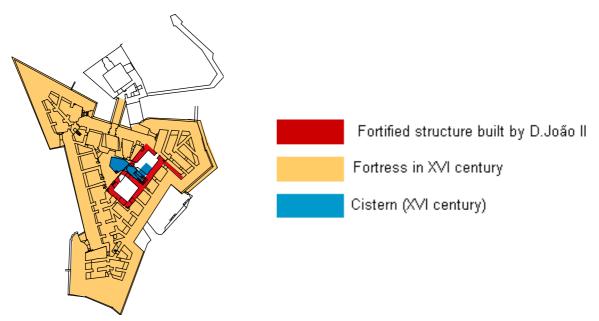


Figure 2: Plan of *N. Sra. da Luz* fortress, showing the main construction phases.

2 Experimental Work

2.1 Characterization of Render and Plaster Samples

Several render and plaster samples, together with some fragments of bedding mortars from various areas were collected to allow the compositional characterization and chronological identification of the material. The selection of samples was made according to the LNEC methodology of mortar characterization in ancient buildings [9, 10, 11], and taking into account the importance of the different areas in terms of archaeological survey, techniques and materials.

In the present paper we focus on the mortars from the renders and plasters of the *Joanina Tower*, the North and South Bastions and the Walls (Fig. 3), which, even in the harsh environment conditions, remain generally in good condition despite some superficial aesthetic degradation. The samples studied are identified and described in Table 1. A first approxima-

tion to their dating is made considering historical data about the Fortress construction phases and reported interventions [7, 8]. The laboratory test results will contribute to confirm or correct these dates.

2.2 Methodology

After some preliminary in situ analysis, additional physico-chemical tests were performed in order to characterize the mortars of these renders and plasters, identify the differences between them and assess their condition. The characterization methodology developed at LNEC [9, 10, 11] comprises a wide range of techniques that complement each order, including X-Ray powder diffraction (XRD), thermo gravimetric and differential thermal analysis (TGA-DTA), optical microscopy and petrography, scanning (electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS), grain size analysis, chemical analysis, water absorption capillarity coefficient and compressive strength.

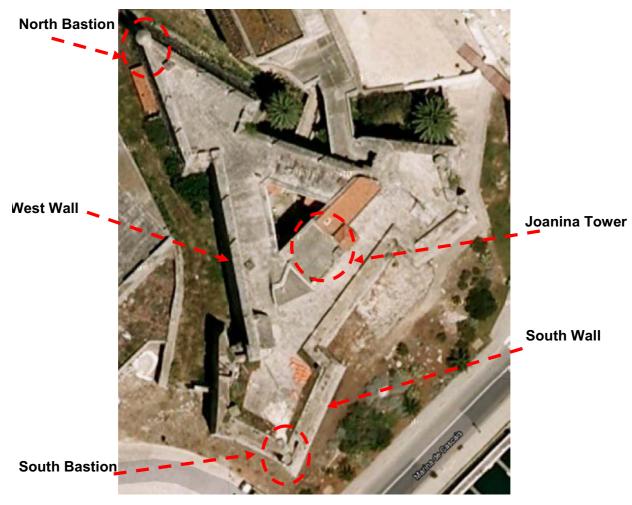


Figure 3: Plan of N. Sra. da Luz fortress, showing the areas where the samples were taken.

Table 1: Render and plaster samples identification and description.

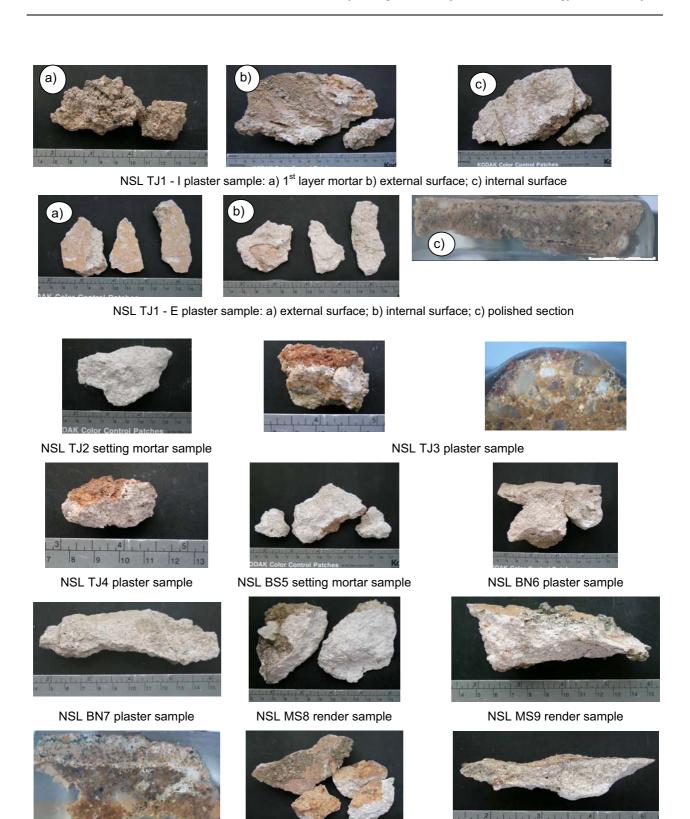
Samples	Localization ar	nd approximate dating	Description			
NSL TJ1 – I		Interior wall. 1 st coating systems	Plaster composed of three mortar layers covered by an ochre paint layer. The 1 st interior layer has brown colour, while the 2 nd and 3 rd layers are white, the last being a 1 mm thick stucco			
NSL TJ1 – E	Joanina Tower XV-XVI century	Interior wall. 2 nd coating systems	Plaster mortar composed of two mortar layers covered by an exterior ochre coloured paint. The 1 st and 2 nd layers have white colour while the 2 nd is a thin stucco.			
NSL TJ2		The wall region with a hole	Setting mortar composed of a single and uniform mortar layer of white colour.			
NSL TJ3		Entrance zone wall (old door)	Plaster composed of three mortar layers whith different colours: brown, yellow/ochre and white.			
NSL TJ4	Joanina Tower XV-XVI century	Entrance zone wall (previously buried)	Plaster composed of two mortar layers. The interior 1 st layer is brown and appears to be clay. The exterior 2 nd layer is white and rich in ceramic materials.			
NSL BS5	South Bastions XVI-XX century	Interior wall (with evident signs of high humidity levels)	Setting mortar composed of a single and uniform mortar layer of white colour.			
NSL BN6	North Bastions (NSL BN7	Interior wall (small room)	Plaster composed of a single and uniform mortar layer of white colour.			
NSL BN7	XVI-XX century)		Plaster mortar composed of a single mortar layer applied in three sublayers with white colour.			
NSL MS8			Render composed of a single mortar layer of white colour covered by an ochre colour paint			
NSL MS9	South Wall XVI-XVIII century	External walls	Multilayer finish system composed of four layers. Interior to exterior: 1. light colour mortar (5mm); 2. <i>ochre</i> colour paint; 3. light colour mortar; and 4. ochre colour paint.			
NSL MP10	West Wall XVI-XVIII century		Render composed of a single mortar layer of white colour and covered with lime paint of ochre colour.			

3 Results and Discussion

3.1 Chemical, Mineralogical and Microstructural Analysis

These analyses mainly aim to determine the current composition of the mortars, in order to define the original formulation and their chronology, and evaluate the compounds resulting from their deterioration. Physical and mechanical tests complement them to provide information about their conservation state.

XRD analysis was performed with a *Philips PW3710* X-ray diffractometer at 35 kV and 45 mA, using Fe-filtered Cobalt $K\alpha$ radiation (λ =1.7903 Å). Diffractograms were recorded between 3 and 74° 2 θ , at a speed of 0.05 °/s. Two fractions were analysed, both passing the 106 μ m sieve. The first one, fine fraction, was obtained by directly sieving the mortar as collected. The second one, bulk fraction, was obtained by first grinding the mortar. The bulk fraction of each sample was also used for thermal analysis (TGA-DTA) performed in a *SETARAM TGA-DTA* analyser, under argon atmosphere, with heating rate of 10 °C/min, from room temperature to 1000 °C.



Detail of NSL MS9 render sample

NSL MP10 render sample

Figure 4: Photographs of the render and plaster samples studied.

Thin and polished sections of the mortars were prepared by vacuum impregnation with an epoxy resin. These were observed with an *Olympus SZH* stereoscopic microscope, while petrographical observations were performed on an *Olympus BX60* polarizing microscope.

For the chemical analysis, small portions of the mortars were carefully disaggregated and all types of impurities and limestone aggregate grains were separated. Samples were then attacked with warm diluted hydrochloric acid (1:3) to separate the soluble fraction from the siliceous aggregate grains. In the soluble fraction, chloride ions were determined

by potentiometry, soluble silica and sulphate ions by gravimetry, sodium and potassium ions by atomic absorption spectroscopy. The insoluble residue was weighed and sieved to determine the content and particle size distribution of the aggregate fraction.

Scanning electron microscopy (SEM) was performed with a *Jeol JSM-6400* SEM coupled with an energy dispersive X-ray spectrometer (EDS, *Oxford-Inca X-Sight Si(Li)* X-ray detector).

Tables 2 and 3 present the mineralogical composition of the mortars by XRD analysis.

Table 2: Mineralogical composition of the mortars from Joanina Tower assessed by XRD.

	Samples									
Crystalline phases	NSL-TJ1-E		NSL-TJ1- I		NSL-TJ-2		NSL-TJ-3		NSL-TJ-4	
	В	F	В	F	В	F	В	F	В	F
Quartz	+++	+/++	+++	+/++	+++	++	+++	+/++	+++	+/++
Feldspars	++	+	+	?	++	Vtg	++/+++	+/++	++/+++	+
Mica	Vtg	Vtg/?	Vtg	?	-	-	Vtg	?	Vtg	Vtg
Kaolinite	Vtg	Vtg	-	-	?	-	-	-	-	-
Calcite	++/+++	+++	++	++	++	+++	++/+++	+++	+++	+++
Dolomite	-	-	-	-	-	-	?	-	?	Vtg
Halite	-	-	-	-	-	-	Vtg	Vtg/+	Vtg	Vtg
Hematite	Vtg	-	-	-	Vtg	Vtg	Vtg/+	Vtg	Vtg	Vtg
Ettringite	-	-	Vtg	Vtg/+	-	-	-	-	-	-
Monocarbo- aluminate	-	-	+	-	+	++	-	-	?	?
Chloroaluminate	Vtg/+	+	+/++	++	Vtg	Vtg	Vtg/+	Vtg/+	?	?
Sodalite	-	-	-	-	Vtg/+	+/++	-	-	-	-
Cristobalite	-	-	-	-	vtg	+	-	-	-	-
CSH	-	-	-	Vtg	-	-	-	-	-	-

peak intensity: +++ high, ++ medium, + low, T traces, ? doubtful presence, - undetected

B – Bulk sample; F – Binder rich fraction

Table 3: Mineralogical composition of the mortars from Bastions and Walls assessed by XRD.

	Samples											
Crystalline phases	NSL-BS5		NSL-BN6		NSL-BN7		NSL-MS8		NSL-MS9		NSL-MP10	
	В	F	В	F	В	F	В	F	В	F	В	F
Quartz	+++	+/++	+++	+	+++	+	+++	+	+++	+	+++	+
Feldspars	++	+	++	-	++/+++	+	Vtg/+	Vtg	+	+	+	Vtg/+
Mica	?	?	?	-	Vtg	-	?	?	?	?	Vtg	?
Calcite	+++	+++	+++	+++	+++	+++	+++	+++	+++	+++	++/+++	+++
Aragonite	Vtg/+	+	Vtg	?	Vtg	Vtg	+	+	Vtg	Vtg	?	?
Dolomite	+	+	vtg	vtg	Vtg	Vtg	-	-	-	-	-	-
Hydromagnesite	+	+/++	-	-	-	-	-	-	-	-	-	-
Halite	-	-	-	-	-	-	?	?	-	-	Vtg	Vtg/+
Goethite	Vtg/+	+	-	-	-	-	-	-	-	-	-	-
Hematite	-	-	Vtg	?	Vtg	Vtg	Vtg/+	Vtg	Vtg	Vtg	Vtg	?
Gypsum	-	-	-	-	-	-	-	ı	-	-	Vtg	Vtg
Ettringite	-	-	-	-	-	-	-	Vtg	-	?	-	Vtg
Monocarbo- aluminate	-	-	-	-	-	-	-	-	-	-	?	?
Chloroaluminate	-	-	-	-	-	-	Vtg	Vtg	Vtg	Vtg	Vtg	Vtg

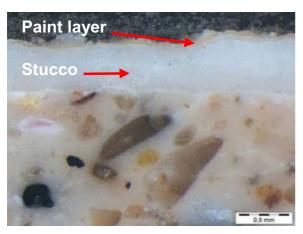
peak intensity: +++ high, ++ medium, + low, T traces, ? doubtful presence, - undetected

B - Bulk sample; F - Binder rich fraction

From the analysis of the mineralogical and microstructural results obtained it can be concluded that:

- The existing layers of paint and stucco (NSL TJ1-I, NSL TJ1-E, NSL MS8, NSL MS9, NSL MP10) were formulated with aerial lime. The pigment used in coloured layers is of mineral origin (Fig. 5), probably an ochre earth pigment.
- With the exception of NSL BS5 sample, that is a dolomitic aerial lime, all mortars have pure calcitic aerial lime binders.
- The sands are predominantly silicate minerals

 Fig. 6. Among the aggregates (mainly quartz and feldspars grains), with exception of the 2nd layer of sample NSL TJ4, the use of basaltic fragments was also identified. The occurrence of some fossiliferous carbonate fragments in all mortars suggests that the sands used are of marine origin, likely from close to the site. The use of ceramic fragments in samples (NSL TJ1-E, NSL TJ4 -1st layer and NSL BN7) is also to be noted.
- With the exception of NSL BN7 sample, all mortar samples show the occurrence of pozzolanic reactions between the lime and the silicate aggregates, resulting in the formation of hydraulic compounds such as calcium silicates (Fig. 7). The alteration of silicate sands, as well as the use of already altered sands, in particular quartz, feldspar and basalt grains, as well as the aggressiveness of the environment, and long reaction times enhanced these reactions.
- Taking into account the extent of the pozzolanic reactions observed in the mortars from the *Joanina Tower*, and as environmental conditions do not differ between the various sampling sites, it can be speculated that these are likely the oldest mortars in the fortress
- Some of the mortars show high levels of soluble salts, mainly chloride and sulphate ions.
 However, there was no evidence pointing out to microstructural phenomena of chemical deterioration.



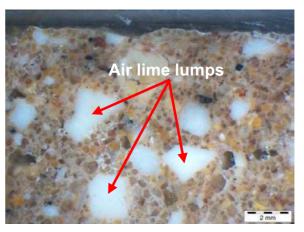


Figure 5: Optical observation of polished sections of the samples where the layers of the renders/plasters and white lime lumps in the binder are visible.

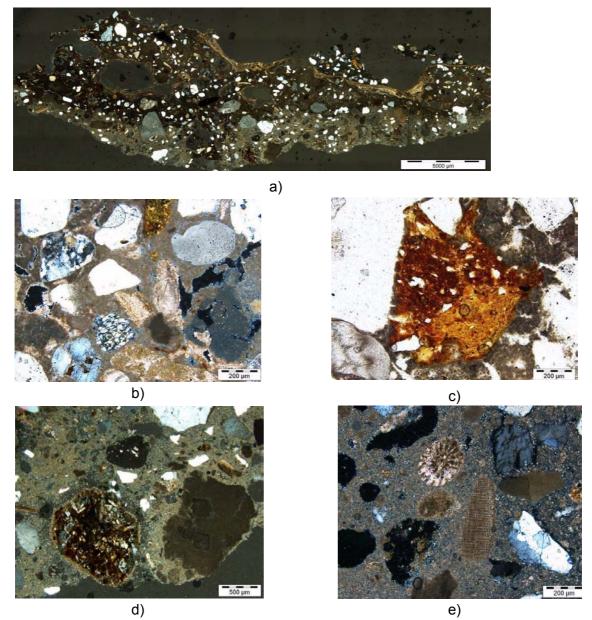


Figure 6: Polarized microscopy images: a) NSL-TJ1-I sample where the different plaster layers are visible; b) NSL-BN7 sample showing the presence of deformed polycrystalline quartz grains of the aggregate (arrows); c) NSL-BN7 sample showing a ceramic fragment with alteration rim; d) NSL-TJ1-I sample showing a basaltic fragment and a lime lump; e) Fossiliferous carbonate fragments in NSL-TJ1-E sample.

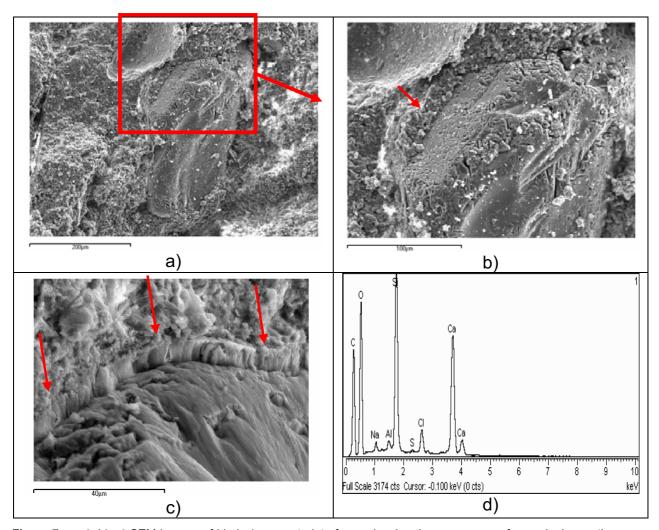


Figure 7: a), b), c) SEM images of binder/aggregate interfaces showing the occurrence of pozzolanic reaction compounds (arrows) in NSL-TJ1-E sample; d) EDS spectrum of the pozzolanic reaction compounds showing a calcium silicate composition.

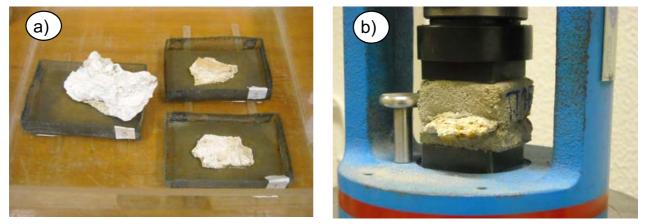


Figure 8: Physical and mechanical tests: a) Water absorption, b) Compressive strength.

3.2 Characterization of Mechanical Strength and Water Behaviour

In order to assess the conservation status of renders and plasters and their functional capacity, some mechanical strength and water behaviour tests were performed. These tests, developed and validated in previous works, are: capillary water absorption by contact test [12,13]; compressive strength [13,14] and ultrasonic test for determination of the dynamic elasticity modulus [15]. The techniques used for this characterization require some special test methods, as the samples are usually small, irregular and sometimes have low cohesion.

The capillary water absorption by contact test (Fig. 8a) consists on periodic weighing of samples which are put in a basket with a wet geotextile gauze placed in a transparent recipient with water. The specimen, which is in contact with water through the wet geotextile, is weighed together with the whole wire basket and wet gauze, every 5 minutes for the first 40 minutes and then within intervals of 60, 90, 180, 300, 480 and 1440 minutes. The absorbed water is determined through the difference between the weights measured periodically (basket + wet gauze + sample) and the initial weight. The capillarity coefficient obtained by this method is designed capillarity coefficient by contact (Ccc) and it is determined as the slope of the initial straight part of the absorption curve in function of the square root of time, which in the present mortars is from zero to 5 [12, 13].

For the compressive strength test (Fig. 8b), the mortar test samples are prepared by applying a fresh mortar (normally composed of cement and siliceous sand with weight proportions 1:3 (CEM II, 32.5: sand) on the surface of the mortar sample to be tested so as to obtain a regularly shaped mortar specimen (approx. 40 mm x 40 mm or a little more) as required for the equipment used in the standardised method EN 1015-11. An easily applied direct compression test is then carried out, giving compressive strength values (Sccm) by the division of the compressive force that produces rupture of the sample (old mortar) by the 40 mm x 40 mm area of force application [13, 14]. An electromechanical testing machine ETI HM-S was used, with a load cell of 200 kN.

Ultrasonic testing was used to evaluate the compactness and stiffness of the renders. This test is based on the Brazilian standard NBR 8802/94 [15], the propagation velocity of ultrasonic waves through the material under study is measured. An Ultrasonic Tester *Steinkamp BP-7* model was used, which emits waves, between two transducers positioned in different points of the sample (in this case, on the same face - indirect transmission) and registers the transmission times, in microseconds, between the two transducers. Samples with at least one dimension larger than 10 cm and a surface regular enough were used for these measurements.

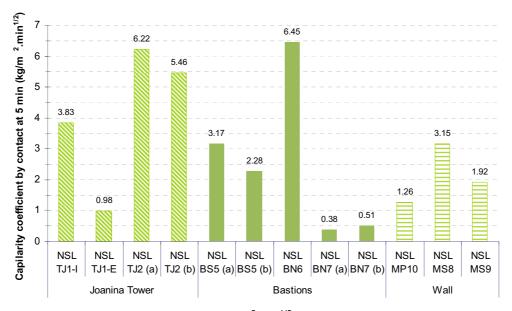


Figure 9: Results of water absorption coefficients (kg/m².min^{1/2}).

The main conclusions obtained from the physical-mechanical tests are:

- NSL TJ2 and NSL BN6 samples have the highest capillary absorption coefficients (> 5.46 kg/m².min^{1/2}) indicating larger capillary pore diameters of these samples, while NSL TJ1-E, NSL BN7 and NSL MP10 have the lowest values (< 1.26 kg/m².min^{1/2}), corresponding to smaller capillary pore diameters (Fig. 9).
- The compressive strength of NSL TJ2, NSL BS5 (a) and NSL BN6 samples is low ($< 1.7 \text{ N/mm}^2$) and their dynamic modulus of elasticity indicate high deformability (E $\approx 1500 \text{ N/mm}^2$) (Figs. 10 and 11). These data evidence a lower mechanical strength of

the same mortars that showed higher capillary absorption coefficients. On the other hand, samples NSL TJI-E, NSL BN7 and NSL MP10, have the highest compressive strength, corresponding to the lowest capillary absorption coefficients obtained.

In conclusion, these results indicate that the mortars from the *Joanina Tower*; external Walls and some from the North Bastions are cohesive, compact and in a good state of conservation. On the other hand, all the samples from the South Bastion and the remaining from the North Bastion, show high capillarity coefficients and low mechanical strengths, which may indicate a certain degree of degradation of these renders or may just be a result of their original composition.

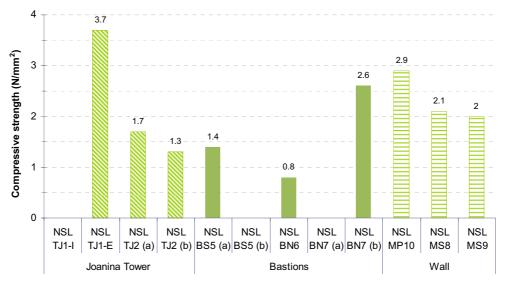


Figure 10: Compressive strength results (N/mm²).



Figure 11: Dynamic modulus of elasticity results (N/mm²).

4 Discussion and Conclusions

The *N. Sra. da Luz* fortress, built around 1580, was subjected to numerous construction phases over the centuries. The analyses carried out, both chemical, mineralogical and petrographical and physical (Table 4) confirmed that the mortars used for the renders of each of the three sections studied, in chronological order: the *Joanina Tower*, the walls and the Bastions are consistent within their group.

- The Joanina Tower mortars (TJ samples) are constituted of calcitic aerial lime and silicate aggregates, mainly composed of altered quartz and feldspar grains, with some altered basaltic grains as well. Pozzolanic reactions between lime and the altered aggregates were found in these samples. These reactions occur due to the existence of amorphous silica coming from the alteration of the aggregates in a very aggressive environment [1, 5]. Based on the composition and on the significant presence of the pozzolanic reactions, these mortars can be confirmed to be the oldest ones present in the fortress, consistent with historic records dating this to the 15th-16th century. In fact, as environmental conditions do not differ between the various sampling sites of the fortress and TJ samples having higher amounts of hydraulic silicate compounds, the only explanation is a longer period of reaction. These samples, especially TJ-1-E (the most exposed render) showed rather high mechanical characteristics and moderate capillarity, when compared with new aerial lime mortars and even with less exposed old lime mortars [13, 16], probably due to the hydraulic compounds formed.
- The mortars used for the Wall renders (M samples) are also mechanically resistant and, with the exception of MS-8 (with high development of biologic colonization), present moderate capillarity. They are composed of calcitic aerial lime and aggregates with a good grain size distribution, mostly altered quartzitic sands but also incorporating altered basaltic grains. Pozzolanic reactions between binder and aggregate were also detected but at a lower degree compared to those from the Joanina Tower suggesting that these mortars are not as old as those of the mentioned tower. They could date from 17th or 18th century, considering the similarity with mortars from other Fortresses of that period and area previously studied [1, 5, 6, 8].
- The Bastions mortars (B samples) can be considered as the most recent in the Fortress. The North Bastion mortar is constituted of calcitic aerial lime and silicate sand (without basaltic grains) and it doesn't show any neoformation products, meaning no alteration of the silicate aggregates and also a short period of reaction when compared to the J and M samples. As they are exposed to the same environment and they have mainly similar constituents, they are likely to be from the 20th century. The use of basaltic aggregates has been found in Lisbon important constructions in the 18th century and before [1, 5, 6, 9, 17], but not in more recent periods, which is also consistent with this probable date. On the other hand, the South Bastion mortar is constituted of dolomitic aerial lime, and is probably from 18th-19th century, based on its composition. In fact dolomitic lime is not known to have been used in Lisbon in the 20th century.

Table 4: Resume of physical and mechanical test results.

Localization	Samples	Ccc ₅	S _{Ccm}	E	
Localization	Cumpics	(kg/m ² .min ^{0,5})	(N/mm ²)	(N/mm ²)	
	NSL TJ1-I	3.83	-	2750	
Joanina Tower	NSL TJ1-E	0.98	3.7	2870	
	NSL TJ2	5.84	1.5	2250	
	NSL BS5	2.73	1.4	1380	
Bastions	NSL BN6	6.45	0.8	1250	
	NSL BN7	0.45	2.6	2895	
	NSL MP10	1.26	2.9	2960	
Wall	NSL MS8	3.15	2.1	2090	
	NSL MS9	1.92	2.0	3530	

The possibility of estimating the relative age of the mortars from the comparison of the thickness of the pozzolanic reaction rims around the aggregate rims was confirmed by the correlation with historic records. The antiquity, durability and diversity of these mortars evidence their historical and technical importance. These facts, along with their general good condition, were the main reasons used to recommend their preservation and conservation.

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She is the Portuguese representative in the European group of preparation of European Guides for Technical Approval of ETICS and she is a member of the RILEM Committee TC SGM "Specifications for non-structural grouting of historic masonries and architectural surfaces". She has supervised several PhD and Master thesis and is an author of many papers presented in Conferences or published in national and international scientific journals.



António Santos Silva

Institution: Laboratório Nacional de Engenharia Civil

Category: Research Officer

Main areas of technical and scientific work:

- Degradation of concrete by expansive chemical reactions. Study of the mitigation measures and repair systems.
- Study of new materials for a green concrete.
- Characterization of historical materials, including mortars/renders/mural paintings/tiles.



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PhD in Architecture, by the College of Architecture of Lisbon, Technical University, with the thesis: The conservation and restoration of external renderings of old buildings - a methodology of study and repair.

Masters in History of Art with the thesis: The Bom Jesus Street in Recife Quarter - a study on the use of colour in façades of historical buildings, Lusíada University, 1998, Lisbon. Specialization - Scientific principles of Conservation (SPC - ICCROM), carried out in Brazil, partnership with the CECOR/UFMG, March to June (360 hours).

Specialization in Conservation of Mobile Cultural Heritage, the Center of Conservation and Restoration - CECOR - School of Fine Arts - Federal University of Minas Gerais, Belo Horizonte.

Graduation in Artistic Education, by the Federal University of Pernambuco (UFPE), Recife.

I have worked as restorer since 1982, in Brazil I worked as a restorer in the Museum of Pernambuco state and for the Joaquin Nabuco Foundation. In 1995 I came to Lisbon and since then I developed activities in the area of conservation of external renderings in National Laboratory of Civil Engineering as a scholarship. Since 1996 I have worked as a freelancer for a company of restoration. Currently my research area is the conservation of old lime mortars and developing studies for their consolidation.

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Main areas of technical and scientific work:

- Conservation and rehabilitation of historical mortars;
- Techniques and materials for conservation and restoration of historic renders;
- Renderings pathology.



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Civil Engineer, master's in civil engineering since 2010 at Instituto Superior de Engenharia de Lisboa.

Main areas of technical and scientific work:

- Physical-mechanical performance and durability of ancient/historical/modern renders.
- Products and technologies for the restoration and conservation of historical renders.
- Efficacy and Sustainability of construction materials, e.g. through the incorporation of industrial waste materials in renders.

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