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# CHARACTERIZATION OF HISTORICAL MORTARS FROM ALENTEJO'S RELIGIOUS BUILDINGS

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Alentejo's religious buildings reflect undoubtedly the history and character of this southern Portugal region. Conservation of these buildings requires a deep knowledge of their masonry and renders' lime mortars to evaluate correctly their state of conservation, to avoid progression of pathologic situations, and to plan efficient interventions, with repair and substitution materials with similar characteristics. This article presents a synthesis of the main results obtained in the mortar characterization of religious buildings from Alentejo, which include Évora and Elvas Cathedrals, Mértola Mosque, and the Church of Amieira do Tejo. For each monument, several samples were collected from different sites and a set of tests was carried out, including chemical, mineralogical, and micro-structural tests, as well as physical and mechanical tests. The tested mortars correspond to different phases of construction and interventions on the buildings, comprising mainly origin periods from the twelfth to the eighteenth centuries; hence exhibited significant differences in composition and in application techniques. The obtained results of composition have given important information about the provenance of the materials used, including binder and sand types, and also about decay products and their correlation with the mortar's conservation state, which gave important clues on the repair strategy to adopt.

KEY WORDS: Historic mortars, characterization, conservation, mineralogy, microanalysis

# **1. INTRODUCTION**

The study of old mortars combines historical, conservation, and materials science research which enables the identification of the materials and available technologies during the several construction stages, the diagnosis of the state of conservation and, in some cases, the knowledge of the commercial routes in the society that built the monument. Underlined to any conservation intervention is the fact that all materials used must be compatible to the original ones and reversible.

Old buildings and particularly religious old buildings from each region are a part of their inhabitants' identity and memory and must be preserved as such, not only in their shape and colors but also in their structure, materials and functionality.

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Case study	Sample	Mortar function	Location
Évora Cathedral	SEV2	Render	Nave, north side
	SEV4	Render	Window arch of clock tower, inside
	SEV6	Filling mortar	Zimborium Tower, inner wall
	SEV7	Filling mortar	Zimborium Tower, inner wall
	SEV8	Render	High choir (behind wooden chair structure)
Elvas Cathedral	SEL1	Render	West terrace pinnacle
	SEL5	Render	Nave, west side
	SEL6	Filling mortar	Bell tower pinnacle
Mértola Mosque/Church	MT4	Render	Mihrab
	MT5	Filling mortar	Mihrab, bedding
Monumental Complex in	AM2	Render	Castle tower
Amieira do Tejo	AM3	render	Saint John the Baptist Chapel, exterior

Table 1. Description of the ancient mortar samples

This article presents the results obtained under the framework of the CATHEDRAL project (Characterization and Conservation of Traditional and Historical Mortars from Alentejo's Religious Buildings) cofinanced by Fundação para a Ciência e Tecnologia (FCT), with the reference POCI/HEC/57915/2004 in a joint collaboration between the National Laboratory of Civil Engineering (Lisbon, Portugal), the University of Évora (Évora, Portugal), the Regional Direction of Alentejo's Culture (Évora, Portugal), and the Sciences Faculty of Lisbon University (Lisbon) to study old mortars of historical buildings in the Alentejo region. The exhaustive analysis of their present composition, using several techniques modernly available must be crossed with the historical knowledge about their execution techniques and with the study of their evolution along the time, to preview their present and future behavior. The main objective of this interdisciplinary project was the development of an integrated conservation methodology for the preservation of important religious buildings from Southern Portugal.

# 2. CASE STUDIES AND METHODS

# 2.1. Samples

Several samples were taken from different sites of the selected monuments with the help of Regional Direction of Alentejo's Culture technicians. However, this work focuses only some samples of each monument, as described in Table 1.

The selected monuments were the Santa Maria Church of Évora (Évora Cathedral, twelfth to sixteenth centuries), Elvas Cathedral (sixteenth to eighteenth centuries), Mértola Mosque/Church (twelfth century) and the Monumental Complex in Amieira do Tejo (fourteenth to sixteenth centuries), all important classified monuments from Alentejo region, in Southern Portugal.

The Church of Saint Maria of Évora or Évora Cathedral (Figure 1a) is classified by UNESCO (Paris, France) as a *World Heritage Site* and is located in the historical town of Évora. The Cathedral has a Romanesque–Gothic or Gothic style with strong Cistercian and mendicant orders influence and its construction began in the twelfth to thirteenth centuries. The construction was inspired by Lisbon Cathedral and by foreign cathedrals, namely Spanish. The religious complex includes also a cloister.



**Figure 1.** Photographs of the monuments in this study: a) Évora Cathedral; b) Elvas Cathedral; c) Mértola Mosque/Church; and d) Monumental Complex in Amieira do Tejo (figure is provided in color online).

Like other buildings of this kind, after the construction period the Cathedral of Évora had restoration works and several increments that reflect the financial power of a particular period. The major intervention was produced in the sixteenth century with the substitution of several renders and the construction of the high choir wooden chair structure.

The Church of Our Lady of Assuncao in Elvas or Elvas Cathedral (Figure 1b) is located in the historical centre of the town and is classified as national monument, since 1910. The construction was initiated in 1517; even though the building had some further modifications, the actual church still preserves the original structure. Particularly important were the interventions during the seventeenth and eighteenth centuries where numerous decorative works were done inside the church including mural paintings and glazed tile panels and marble and wooden baroque altars. The Bishopric of Elvas was extinct in 1881, and afterwards no major interventions were performed.

The Church of Our Lady of Assuncao in Mértola or Mértola Mosque/Church (Figure 1c) dates from the days of Islamic rule and was a former mosque. Gothic in style, the mosque was converted in church in the twelfth century, and the mosque still retains a strong Islamic feel in its horseshoe arches and *mihrab* (prayer niche) facing east to Mecca, although further modifications were made in the sixteenth century, such as the Manueline and Renaissance entrance and the cylindrical towers. The building is a startling example of the adaptation of a place of worship to a different faith.

The monumental complex in Amieira do Tejo (Figure 1c) stands in the long and ancient defensive line of castles along Tagus River, in Portalegre district, approximately 14 km from the village of Nisa. Due to its characteristics and historical importance it was classified as National Monument in 1922 and as Military Monument in 1923. The construction of the castle was initiated between 1350–1360. In 1566, when military needs were already less demanding, a chapel was added in the east side of the *barbican*, dedicated to Saint John the Baptist and quite remarkable for its vaulted ceiling entirely decorated with rare *sgraffito* ornaments, depicting geometric, hybrid, and fantastic Mannerist motives.

During the sixteenth century, defensive functions of the castle were gradually abandoned, thus giving place to decay and neglect. Since its classification, the castle suffered several restoration campaigns. The major works took place in 1950, and responded to the theories of that period, which enhanced the medieval period as being the most representative of past glories.

### 2.2. Characterization Methodology

A great number of physical and chemical techniques can be applied in the characterization of old mortars. The characterization methodology developed by the authors, presented in Figure 2, comprises a wide range of techniques that complement each other. The mortars were thoroughly examined in the laboratory using a stereozoom microscope OLYMPUS model SZH and carefully disaggregated to avoid breaking the existing aggregates.



Figure 2. Schematic illustration of the sequence of tests to identify the composition of ancient mortars (adapted from Veiga et al. 2001).

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Scanning electron microscopy (SEM) observations were performed with an SEM JEOL JSM-6400 coupled with an OXFORD model Inca X-Sight energy dispersive spectrometer X-ray detector.

X-ray diffraction (XRD) was carried out with a Phillips diffractometer model X'Pert with Co K $\alpha$  radiation, speed of 0.05°/s, from 3° to 74°, 2 $\theta$ . Two types of fractions were analysed, the fraction corresponding to the mortar as collected, designated as *overall fraction* and obtained by grinding the disaggregated mortar to pass in a 106- $\mu$ m sieve and a fraction designated as *fine fraction*, which has a higher binder concentration and was obtained from the fine particles of the disaggregated material passing a 106- $\mu$ m sieve. The overall fraction of each sample was also used for thermogravimetric and differential thermal analysis [TGA-DTA] performed in a SETARAM TGA-DTA analyser model 92, under argon atmosphere, with heating rate of 10° C/min, from room temperature to 1000° C.

Thin sections and polished surfaces of the mortars were prepared by vacuum impregnation with an epoxy resin. These were observed with a Nikon petrographic microscope from NIKON, model Eclipse E600, in transmission, using crossed polarizers and images were recorded digitally.

For the chemical analysis of the binder components, small portions of the mortars were carefully disaggregated and all types of impurities and limestone grains were separated. Samples were afterwards attacked with warm diluted hydrochloric acid (1:3) to separate the siliceous aggregates from the lime paste. For the soluble fraction, the amounts of calcium, magnesium, aluminum, iron and sodium (expressed in terms of their oxides) were determined by atomic absorption spectrometry, chloride ion was determined by potentiometry and sulphate ion was determined by gravimetry. The insoluble residue was weighed and sieved to determine the particle size distribution of the aggregate fraction (i.e., the siliceous sand).

For the porosimetric tests, mercury intrusion porosimetry and nitrogen adsorption, was used approximately 1 g of sample disagregated with a rubber hammer. Nitrogen adsorption isotherms were determined in an automatic apparatus, Quantachrome NOVA. Before experiments, the mortar samples were outgassed under vacuum at  $120^{\circ}$  C for 2.5 hours. Mercury intrusion porosimetry experiments were determined in Quantachrome Autoscan60.

The water absorption by capillarity test was performed according to the method developped for irregular friable samples (Veiga et al. 2004; Magalhães et al. 2008) using non-manipulated small samples, which were placed in a wire basket with a wet geotextile in permanent contact with water, and thus avoiding the immersion in water and the loss of particles.

The compressive resistance was determined by the method of confinement mortar (Magalhães et al. 2008); Válek et al. 2005), consisting on the use of small samples extracted from the wall and regularized by means of a cement mortar stronger than the mortar in study. All mortars were dried at 40° C for at least 12 hours, with exception of the samples for chemical analysis, which were dried at 105° C.

# 3. RESULTS AND DISCUSSION

# 3.1. X-Ray Diffraction Analysis

Table 2 presents the mineralogical composition of the overall fraction of the mortars determined by XRD analysis. The results show that the mortars of Évora

<b>Crystalline phases</b>	SEV2	SEV4	SEV6	SEV7	SEV8	SEL1	SELS	SEL6	MT4	MT5	AM2	AM3
Quartz	+++++++++++++++++++++++++++++++++++++++	+++++	+++++	++++++	+++++++++++++++++++++++++++++++++++++++	+ + +	+++/++	+++++++++++++++++++++++++++++++++++++++	+	+++++++++++++++++++++++++++++++++++++++	+++	+++++
Feldspar	++	++	++	++/+	+++++++++++++++++++++++++++++++++++++++	+++++	++/+	++++	vtg	++	vtg	+++/++
Mica	+	++/+	++/+	+++++	+	+	vtg/+	++/+	vtg	+++/++	vtg	++++
Chlorite		+	vtg	+			ż		vtg	vtg/+		
Amphiboles	vtg	vtg/+			vtg/+	vtg/+	++	vtg/+		++/+		
Zeolitic mineral	vtg	vtg					vtg					
Halite	vtg	ċ			vtg							
Kaolinite		?/vtg				vtg/+	ż	vtg/+		ļ	vtg	vtg/+
Calcite	++/+	++	++	+	++/+	++	++	+++++	vtg	++	++	+++++++++++++++++++++++++++++++++++++++
Magnesite	vtg	+			vtg		vtg/+	I				
Dolomite	vtg	vtg			vtg		vtg/+					
Hydromagnesite	vtg	?/vtg			vtg		?/vtg	I				vtg/+
Aragonite	vtg/+									++/+		
Pyroxene	?/vtg											
Gypsum					vtg				++++/+++			
Brucite												



**Figure 3.** Graph of the thermogravimetric curves of: a) a calcitic mortar; and b) a dolomitic mortar (figure is provided in color online).

Cathedral are essentially composed of carbonates (calcite, aragonite, dolomite, hydromagnesite, and magnesite) and siliceous aggregates corroborating the stereozoom observations. Samples SEV6 and SEV7 have essentially calcitic lime, while the other samples possess calcium and magnesium carbonates, which indicate that dolomitic materials were used to produce the aerial lime for this mortars. The mortars containing dolomitic lime are thought to be of a more recent period.

XRD showed that the Elvas Cathedral, Mértola Church, and the Monumental Complex of Amieira do Tejo mortars are mainly calcitic, with some contribution of magnesium carbonates (dolomite and hydromagnesite) in case of samples SEL5 and AM3.

The results indicate that sample MT4 is a gypsum mortar, with a little content of siliceous aggregates and calcite. Little quantities of calcite were frequently added to gypsum during the mortar preparation. This material is probably one of the few examples of gypsum plaster from the Islamic period identified in Portugal.

# 3.2. Thermal Analysis

The thermograms of samples SEV6, SEV7, SEL1, SEL6 and MT5 are typical of aerial lime mortars (Figure 3a) with an important weight loss of temperatures ranging between  $550^{\circ}$  C–900° C. The other samples present a dolomitic/calcitic nature (Figure 3b) with weight losses corresponding to the hydromagnesite, magnesite, and dolomite compounds.

The thermogravimetric curve of sample MT4 presents an important weight loss of temperatures ranging  $100^{\circ}$  C– $200^{\circ}$ C due to the gypsum dehydration. These results corroborate the XRD information.

# 3.3. Chemical and Grain-Size Analysis

Table 3 shows the chemical analysis of the soluble fraction, which can give valuable information about the composition of the mortars and its environment. The chloride ion contents obtained in samples SEV2, SEV4, and SEV8 are higher than the other samples and even higher than in mortars found in high salinity environments (Alessandrini et al. 1996; Sabbioni et al. 2002). Considering that Évora is located far from the sea, thus in an environment with low salinity, and that chloride was found in mortars that were located inside the church at different levels, we can assume that salt was added during the manufacture of the mortar with the aim to accelerate the mortars hardening (Adriano et al. 2007).

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Weight %	SEV2	SEV4	SEV6	SEV7	SEV8	SEL1	SEL5	SEL6	MT4	MT5	AM3
CaO	15.78	9.14	8.86	8.75	8.46	5.77	2.97	7.85	32.46	13.30	6.93
MgO	5.45	7.08	1.39	1.79	5.69	0.29	2.44	0.24	0.56	0.75	1.83
K <sub>2</sub> O	2.36	0.51	0.34	0.45	0.37	0.05	0.19	0.17	0.04	1.03	0.47
Na <sub>2</sub> O	3.08	0.90	0.07	0.12	0.46	0.12	0.49	0.11	0.06	0.45	0.15
Cl	0.85	0.62	0.06	0.06	0.53	0.06	0.14	0.06	0.06	0.08	0.06
SO <sub>3</sub>	0.12	0.28	0.18	0.15	0.23	0.19	0.18	0.25	49.09	0.16	0.14

Table 3. Chemical composition of the soluble fraction of the mortars (wt %)



G1:<0.075 mm; G2: 0.075-0.160 mm; G3: 0.160- 0.315 mm; G4: 0.315-0.630 mm; G5: 0.630- 1.25 mm; G6: 1.25-2.5 mm; G7: 2.5-5.0 mm; G8:>5.0 mm size (mm)



Samples SEV2, SEV4, SEV8, and SEL5 present higher magnesium oxide contents than the other samples, in agreement with the XRD data, which showed that they are composed of calcium–magnesium carbonates.

The grain size distributions of the insoluble residues are presented in Figure 4. This analysis is important if one intends to produce compatible mortars for restoration (Bakolas et al. 1998; Maravelaki-Kalaitzaki et al. 2003; Benedetti et al. 2004). The grain size distribution revealed that most samples have aggregates with diameters between 0.315 and 2.5 mm. For all samples, the insoluble residue observation under stereomicroscope together with the XRD analysis allowed the confirmation that the aggregates used have uniform mineralogical composition, which can be correlated with the local geology for each case study.

# 3.4. Optical Microscopy

**3.4.1. Stereozoom observations** Polished surfaces observation under a stereomicroscope showed that all mortars contain round nodules of lime (Figures 5a and 5b), which may indicate that the lime was slaked with a minimum amount of water to convert



Figure 5. Stereozoom micrographs of polished sections mortars (figure is provided in color online).

CaO into  $Ca(OH)_2$  (Schouenborg et al. 1993; Elsen et al. 2004). In some cases, it was also possible to find ceramic fragments (Figure 5a). The presence of ceramics was also evident and has extreme importance since addition of this material was a technological way of improving the mechanical quality and the hydraulic properties of the material.

**3.4.2. Transmission microscopy** Under a petrographic microscope, some minerals that were not detected by XRD were identified, such as olivine (Figure 6b). The presence of this mineral is particularly important, since it allowed the establishment of the probable source of the aggregates, the S. Bento quarries in the vicinity of Évora Town, which held some olivinic gabbros outcrops.

The petrographic microscopy improved the capability to identify optically the aggregates and allowed the recognition of typical minerals from the geological igneous and metamorphic environment of Elvas, such as amphiboles, pyroxenes, sphene, and staurolite. This technique enabled also an evaluation of the spatial distribution of neoformation materials as result of pozzolanic reactions (Figure 7b). Particularly important was the possibility to distinguish carbonate aggregates from the binder (generally calcitic) (Figure 7c).

Regarding the Mértola Mosque thin sections, lime nodules (Figure 8a), quartzites, schist fragments, micas with high dimensions, and plagioclases were observed (Figure 8c). The clasts have an angular shape (Figure 8a), with a small quantity of biotite and chlorite. The aggregates can proceed from sedimentary origin and suffered a small transport.

As for the Monumental Complex of Amieira do Tejo thin sections, it was possible to observe lime nodules and neoformation compounds forming a reaction fringe. This



**Figure 6.** Photographs of thin-sections of the Évora Cathedral mortars: a) carbonated-siliceous matrix with neoformation calcosilicate agglomerates (magnification 12 X) in plane polarized light; b) olivine crystal (30 X) in crossed polars; and c) neoformation calcosilicate bands growing in fractures ad aggregates-binder interfaces (12 X) in plane polarized light (figure is provided in color online).



Figure 7. Photographs of thin-sections of the sample SEL1: a) granite grain in crossed polars; b) ceramic fragment in crossed polars; and c) plagioclase (1) and carbonate (2) in crossed polars (figure is provided in color online).



**Figure 8.** Photographs of thin-sections of the sample MT5: a) lime nodule (1) and an aggregate with angulous shape (2) in crossed polars; b) general aspect of the sample with lime nodules (1) in crossed polars; and c) plagioclase (3) in crossed polars (figure is provided in color online).



**Figure 9.** Photographs of thin-sections of the Monumental Complex of Amieira do Tejo: a) Quartz grain (1) with a neoformation compound (2) inside the fractures (sample AM2) in plane polarized light; b) quartzite of high dimensions with a mica in the center in crossed polars; and c) granitic aggregate with very altered feldspars (3) and quartz (4) in crossed polars (figure is provided in color online).

neoformation compounds are localized in the aggregate/binder contact and inside of the aggregates fractures (Figure 9a). The samples of the Castle are very similar to the samples of the Chapel. In the samples of the Chapel it was possible to observe granite clasts, quartzite (Figure 9b), feldspars in high quantity, and highly altered micas (Figure 9c), which is in agreement with the geologic characteristics of this region.

**3.4.3.** Scanning electron microscopy Figures 10–13 show the most important microstructural features of the studied samples. Analysis of the mortars showed that all mortars have a compact gelified microstructure, typical of old lime mortars, with

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**Figure 10.** Scanning electron microscope (SEM) micrographs of aggregates: a) quartz grain in sample SEL5; b) gypsum microstructure of the mortar MT4; c) ceramic fragment observed in the paste; d) altered mica grain (muscovite) in sample SEV4; e) energy dispersive spectrum of the gypsum microstructure in Figure 10b); and f) energy dispersive spectrum of the ceramic fragment in Figure 10c.



**Figure 11.** Scanning electron microscope (SEM) micrographs: a) carbon black particle typical of combustion emissions (sample SEV4) and different types of biological materials; b) sample SEV2; c) sample SEL5; d) sample SEL6; e) and f) sample SEL1 at different magnifications.

aggregates well embebed in the matrices. It was possible to identify the nature of the aggregates as mainly lithoclats of quartz (Figure 10a) and very altered micas (Figure 10d).

On sample MT4, it was possible to observe gypsum crystals (Figure 10b) and ceramic fragments in the paste (Figure 10c).

In all samples, countless biological colonizations were observed (Figure 11).

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Figure 12. Scanning electron microscope (SEM) micrographs of a) and b) halite crystals of sample SEV8; and c) the corresponding energy dispersive spectrometer spectrum.



Figure 13. Scanning electron microscope (SEM) micrographs of a) and b) hydromagnesite crystals in sample SEV8; and c) the corresponding energy dispersive spectrometer spectrum.

Besides the already mentioned features, on sample SEV2 it was possible to observe the presence of halite and hydromagnesite crystals. The presence of halite (Figure 12), which was also observed in other samples, namely, SEV4 and SEV8, was previously detected by XRD and indirectly by chemical analysis. The hydromagnesite crystals were located in porous and superficial areas (Figure 13).

The most stricking observation in Évora's Cathedral was the presence of carbon black particles in the mortars (Figure 11a) with magnesian binder which can be attributed to combustion of organic materials. The concomitant presence of abnormal amounts of chloride and the presence of these carbon black particles (in mortars from the interior of the church) may indicate that fire was produced inside the church and sodium chloride was added during the making of the mortars to accelerate the mortars carbonation and hardening. In fact, historical documents indicate that by the sixteenth century, the rocks from the inner walls of the Cathedral were very degraded and that a major intervention occurred, so we can assume that this was a technical solution that was envisaged (Adriano et al. 2007).

### 3.5 Mortar Composition

The simplified compositions of the mortars (Table 4) were calculated on the basis of the method designated as *Jedrzejewska* (Jedrzejewska 1960) referring to old lime mortars combining the percent calcium carbonate estimated by TGA/DTA with the residue analysis. As mentioned previously, all mortars, with exception of samples SEV6, SEV7, SEL1, SEL4, and SEL6, showed the presence of magnesium compounds, which indicates the use of dolomitic lime; therefore, the composition of each phase was calculated from the correspondent weight losses.

Samples	Siliceous Aggregate <sup>1</sup>	Calcitic Aggregate <sup>2</sup>	Calcite <sup>3</sup>	Magnesite <sup>4</sup>	Dolomite <sup>5</sup>	Hydro- magnesite <sup>6</sup>	Gypsum <sup>7</sup>	Soluble fraction <sup>8</sup>
SEV2	60	_	26	11	_	_	_	3
SEV4	63		13	10	7	2	_	5
SEV6	78		19				_	3
SEV7	78		17					5
SEV8	66	_	12	8	7	1		6
SEL1	66	1	29					4
SEL5	66	4	7	1	7	12		3
SEL6	53	1	35	_			_	11
MT4	13	_	3	_	_	_	84	0
MT5	71	_	23	_			_	6
AM2	55	_	30	8				7
AM3	81	—	15	—	—	—	_	0

Table 4. Simplified composition of the mortars (%)

<sup>1</sup>Siliceous aggregate=insoluble residue of contents; <sup>2</sup>calcitic aggregate=point counting by petrographic analysis; <sup>3</sup>calcite=CaCO<sub>3</sub> content determined by TGA-DTA;

<sup>4</sup>magnesite=MgCO<sub>3</sub> content determined by TGA-DTA;

<sup>5</sup>dolomite =  $CaMg(CO_3)_2$  content determined by TGA-DTA;

<sup>6</sup>hydromagnesite=hydromagnesite content determined by TGA-DTA and chemical analysis;

<sup>7</sup>gypsum=gypsum content determined by TGA-DTA; and

<sup>8</sup>insoluble fraction= $100 - \Sigma$  (aggregate + calcite + magnesite + dolomite + hydromagnesite).

### 3.6. Physical Analysis

Table 5 shows the physical and mechanical results of the studied samples. The magnesian mortars present higher values of compressive strength when compared with the calcitic mortars. In other studies (Mannoni 1988; Cucchiara et al. 1993; Atzeni et al. 1996) it was verified that the use of magnesium hydroxide as a binder in mortars yields products with mechanical properties superior to those obtained with the calcitic lime mortars. The enhanced mechanical strength observed may be due to the hydromagnesite formation in the binder.

	Capillarity coefficient l	by contact at 5 min (Ccc5)	Mercury	Compression		
Samples	$(kg/m^2/h^{1/2})$	$(kg/m^2/min^{1/2})$	porosimetry (%)	strength (N/mm <sup>2</sup> )		
SEV2	2.2	0.3	18.7	3.3		
SEV4	2.5	0.3	19.2	3.5		
SEV8	2.1	0.3	27.7	2.3		
SEL1	7.6	1.0	26	2.0		
SEL5	3.3	0.4	21	3.0		
SEL6	3.4	0.4	25	3.1		
MT4	25.3	3.3	_	3.5		
MT5	8.1	1.0	33	3.3		
AM2	5.5	0.7	_	3.6		
AM3	9.5	1.2	20	2.4		

**Table 5.** Physical and mechanical results of the several samples

-, not determined.

As a general rule, mortars with higher porosity show higher capillarity coefficients by contact and lower compressive strength. The gypsum mortar MT4 is an obvious exception, presenting a very high capillarity coefficient, as would be expected according to its composition, together with high mechanical resistance. The presence of ceramic fragments, for instance in SEL1 and SEL5 explains some increase of capillarity coefficient without the correspondent expected reduction of compressive strength. In all cases, the values obtained for the physical and mechanical characteristics are consistent with good state of conservation of the studied mortars.

# 3.7. Nitrogen Adsorption at 77 K

Figure 14 presents the nitrogen adsorption-desorption isotherms at 77 K. Mean pore radius and surface areas were estimated. The results obtained were compared to establish correlations between specific surface area estimated by Brunauer–Emmett–Teller (BET) model and the chemical and physical characteristics of old mortars. According to the obtained results (Figure 15) the water absorption and compressive strength increase with the mean pore radius. However, no clear correlation was observed between the surface area and the other properties. In future research the number of case studies will be extended to confirm these results, where the mortar disaggregation methodology and the studied fractions need to be more systematically controlled. Also, the analyzed sub-samples



**Figure 14.** Graph of the nitrogen adsorption-desorption isotherms at 77 K for the Évora Cathedral mortars (figure is provided in color online).

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Figure 15. Graph of the relation between the superficial area and the results obtained in the physicomechanical tests.

(approximately 1 g) need to be clearly representative of the all mortar, although this is an aspect that is common to the other analysis methods used in this work.

# 4. CONCLUSIONS

This work shows that in the four case studies of religious buildings of Alentejo's Region, which include Évora and Elvas Cathedrals, Mértola Mosque, and the monumental complex of Amieira do Tejo, the aggregates are sands from the region and can be correlated with the local geology. The relative abundance of each mineral component of the aggregates reflects its natural regional abundance and the resistance to weathering processes.

Besides the superficial apparent degradation mainly due to the presence of biological colonizations in all samples the mortars are in good conservation state. The results show a greater selectivity of raw materials and the use of predominately magnesium lime mortars that can be interpreted as a technological advance from medieval times. Other technological advanced features are the use of crushed or powder ceramics that was recognized in renascence as a useful roman technique or the incorporation of sodium chloride as a natural lime hardener. No hydraulic binders were found in those mortars, in particular no hydraulic lime or natural cement, confirming the idea that old Portuguese mortars were mainly based on air lime, both calcitic and dolomitic. The presence of gypsum as a significant component in one of the samples, confirms the use of gypsum plasters in Portugal during the Islamic period (seventh through twelfth centuries).

This work demonstrated the need to use a methodology that combines different physical, chemical and mineralogical characterization techniques for the study of old mortars enabling a deep insight on the mortars composition, historical and technical background and state of conservation. Although all those buildings were subjected to interventions throughout times and new mortars were seldom used, it was possible to verify that old mortars were kept, sometimes covered by new coats. The general good state of conservation of the old mortars studied and the testimony of ancient technologies and knowledge of the materials are strong reasons to orient future conservation interventions with resource to localized repair and consolidation, avoiding the removal of pre-existent mortars.

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