



Appraisal of non-destructive in situ techniques to determine moisture- and salt crystallization-induced damage in dolostones

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ABSTRACT

The characterisation of both surface and subsurface pathologies (position, depth, width, ...) that affects the porous materials used in building constructions, once in service, is important to establish the most suitable intervention strategy. In this sense, the use of non-destructive techniques allows the analysis of different properties without affecting the material. The present study shows the accuracy of different non-destructive in situ techniques, such as: electrical conductivity and capacitance, infrared thermography, ultrasonic pulse velocity, sound absorption, and electrical resistivity tomography, applied on dolostone ashlar stones outer façade of a sixteenth-century belltower, affected by moisture and salt induced decay. The joint analysis of the results obtained with different techniques substantially improves the interpretation and characterisation of the detected pathologies, as they complement each other perfectly. Electrical resistivity tomography, which delivers resistivity cross-sections, yields very good results in detecting subsurface pathologies, and sound absorption is particularly useful for stone surfaces. In both cases, the frequency of the electric field and that of the acoustic emission to detect the extent of damage must be established in advance. The joint study of electrical conductivity and capacitance determines the degree of moisture/salts, both at the surface and subsurface, in the materials tested, one of the main causes of scaling and flaking in stony materials. However, the petrological characteristics of the materials used and the identification of the saline phases present must be known in advance to make a correct interpretation of the results.

1. Introduction

In the stone widely used in historic structures the presence of salts and water is one of the causes of the most severe decay patterns such as scaling, granular disintegration and fissuring [1]. Such damage, which originate inside the material, are often associated with air or water pollution [2,3] or the effect of the components of the underlying soil or terrain [4] and may be due to the presence of cements [5,6] or other chemically incompatible materials [7].

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Accurately diagnosing the problems, based on a correct identification of damage and decay causes, is imperative to ensure the suitability of the action to be undertaken. That, in turn, calls for a working knowledge of both the damage per se and the prevailing climate and history of the building itself. Damage mapping favours readier visualisation of the damage and its specific position on the building, a prerequisite to planning and establishing intervention criteria. Digital imaging, photogrammetric surveying and the use of LIDAR [8–14] are among the techniques most widely deployed. Other non-destructive techniques (NDTs) have also been successively introduced in recent years as a result of the significant improvement in the technology required for in situ assessment [15–19]. Such techniques are applied to determine the distribution of the physical and/or chemical parameters denoting the most severely decayed areas on the surface of buildings or other structures of cultural interest [20]. Of the techniques in place, those involving contact with the surface, including ultrasonic velocity, hardness testing, spectrophotometry, electrical resistivity or conductivity and georadar, are the most popular. Several must be combined to compensate for the shortcomings of each and to complement and improve the accuracy of results [21,22].

Contact techniques entail the formulation of analytical grids or webs, a laborious task when large areas are involved. They are also quite costly, given the need to process and run statistical calculations on immense amounts of data. That has prompted the use in recent years of techniques that minimise analysis and consequently cost, despite the loss of accuracy involved. However, it is true that such techniques may be specific to determining certain parameters such as types of moisture, saline efflorescence and cryptoefflorescence, granular disintegration, degree of soiling, etc [23–29]. Electrical resistivity tomography, ultrasonic velocity and georadar, among others, return information on the internal structure of materials, detecting voids, fissures, discontinuities, moisture and unevenness, unlike others such as spectrophotometry, hardness testing and moisture measurement that deliver information about the surface only. It is therefore essential to identify the decay processes present and select the techniques best suited to each case to gather the optimal amount of highly accurate information from a holistic perspective and determine the causes and degree of decay [30,31]. As noted earlier, diagnosis informs the decisions on the intervention to be undertaken and reduces the potential margin of error.

In addition, the impact of the presence of salts on the results of non-destructive techniques such as electrical conductivity and resistivity must be borne in mind. Combining several techniques to fine-tune the results [30–32] is likewise essential for subsequent data entry into building information models (BIM) to generate a broad database from which to draw in heritage intervention decision-making [33]. Sampling with minimally invasive techniques is often a necessary complementary measure [34]. In light of the foregoing, heritage conservation has borrowed non-destructive techniques from other disciplines. Impedance testing may furnish additional data, contributing to material characterisation and decay process identification [35–39], although this procedure has been insufficiently explored in heritage restoration.

This study aims to validate the use of certain novel non-destructive techniques (NDTs) to identify moisture- and salt crystallization-mediated damage in the built heritage. That aim is pursued with a case study chosen for the degree of deterioration present in which non-destructive in situ techniques were applied to assess various types of surface and subsurface damage in some ashlar.

2. Materials and methods

The study was conducted on San Juan Bautista Church at Guadalix de la Sierra, a town in the province of Madrid, Spain. More specifically, it involved the east-facing ashlar stone outer façade of its sixteenth-century belltower, affected by substantial moisture- and salt-induced damage. This carbonate ashlar stone wall springs from a flat roof 15 m off the ground. The dolostone ashlar measure from 50 cm to 55 cm in length, although with maximum lengths of up to 80 cm, and from 30 cm to 35 cm in height, with elements just 22 cm and others up to 40 cm in height. Five ashlar, with different types and degrees of decay, representative of the tower overall were selected for the case exercise (Fig. 1).

The stone is affected by a number of decay processes, including scaling, flaking, crumbling, alveolisation and fissuring, with significant loss of volume on ashlar edges and corners as well as on part of their surface, where quarry finishes have all but vanished (Fig. 2).

A preliminary inspection also detected surface and subsurface saline efflorescence on all the façades that had induced scaling, flaking and crumbling. The presence of these salts as well as the concentration and shape of their crystals varied during the observation period and crystallised salts were occasionally found inside ashlar cavities, denoting incipient alveolus formation.

The ashlar are skirted with very hard and stiff cement mortar characterised by significant shrinkage fissuring at the interface with the stone. Surface and subsurface efflorescence generated by the cement mortar [6] accumulating at the mortar-stone interface causes scaling and material detachment around the perimeters of the ashlar.

In the first phase of this two-phase study, the stone ashlar and mortars, along with the decay-inducing salts, were fully

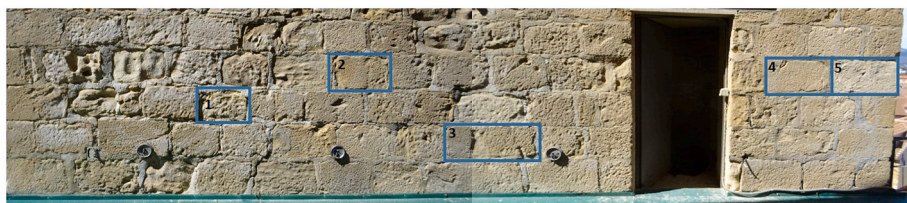


Fig. 1. East façade of the Guadalix de la Sierra Church belltower.

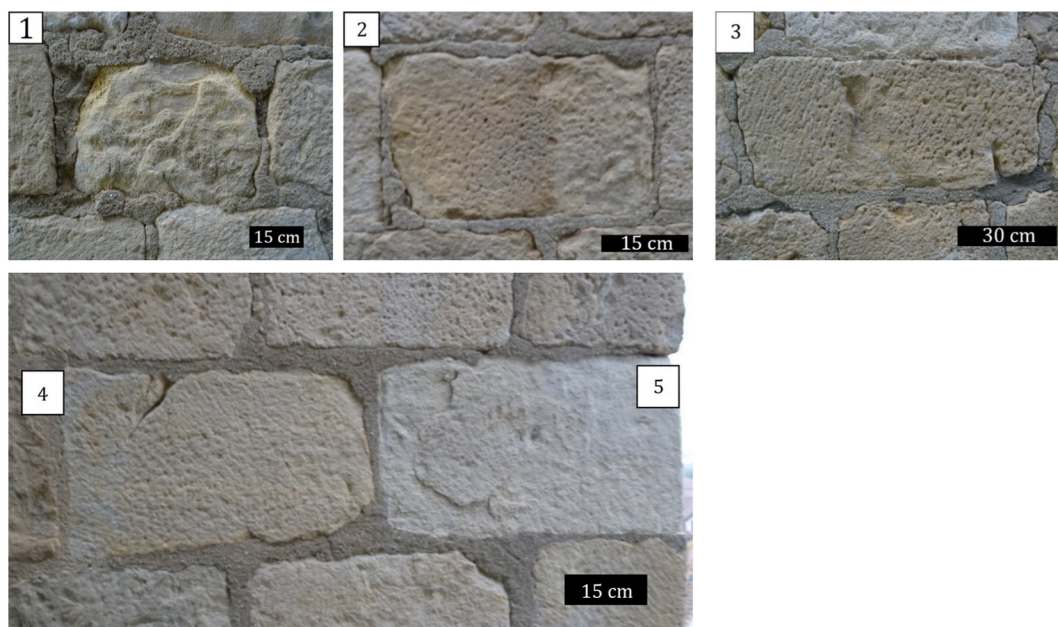


Fig. 2. Pathologies in ashlar studied 1. Alveolisation and scaling with hard crusts and saline efflorescence 2. Alveolisation 3. Scaling and pick marks with fissures 4. Cracks and saline efflorescence 5. Scaling, crumbling and saline efflorescence.

characterised. In a second phase, a number of non-destructive techniques were used and their findings were compared.

2.1. Characterisation of materials

In the first phase, as noted, stone and mortar composition and texture were characterised as stipulated in the respective standards [40–42].

2.1.1. Stone

The findings of a mineralogical study based on X-ray diffraction (XRD) were complemented by petrographic observations of 30 μm thin section made using a Janapol polarised light optical microscope (PM) fitted with a Canon 650 digital camera. To quantify petrographic components, the qualitative method based on visual estimation of percentages was used. X-ray diffraction readings were taken on 2 g of powder sample with a particle size of $<53 \mu\text{m}$. The Philips Panalytical X'Pert MPD diffractometer used, operating at 45 kV and 40 mA, was fitted with a $\text{CuK}\alpha$ anode tube, a flat graphite monochromator and a vertically aligned theta-two theta (θ - 2θ) dual goniometer using Bragg-Brentano geometry.

2.1.2. Salts

X-ray diffraction was also used in the mineralogical characterisation of the salts. Complementarily, the ionic concentration of salts (specifically chlorides, sulphates, nitrates and oxalates), in both the ashlar and mortar samples, was determined by ion chromatography (IC), using a Metrohm Compact IC model 761 ion chromatograph. In addition, the distribution of anions at 1 cm, 3 cm and 5 cm below the surface was determined. For this purpose, powder samples were extracted by drilling with an 8 mm drill bit, in which the three depths considered were pre-marked. On the surface, the salt crusts were scraped off. The samples were grinded to $<0.25 \mu\text{m}$. 0.15 g of the grinded powder extracted at each drilled interval was stirred by ultrasonic bath in 15 mL of ultrapure water during 45 min, and subsequently filtered through 0.45- μm nitrocellulose filters. The anion content was measured by IC in each of the aqueous extractions obtained.

2.2. Non-destructive techniques (NDTs)

Physical inspection was conducted using a number of non-destructive techniques on five specifically selected ashlar to compare decay mechanisms and causes and their relationship with the forms of decay identified in each of them. The techniques used were electrical conductivity, ultrasonic pulse velocity, infrared thermography, acoustic impedance and electrical resistivity tomography. Data were collected at $T = 12.1 \pm 1.3 \text{ }^\circ\text{C}$ and $\text{RH} = 83.2 \pm 8.6\%$. The surface values or physical parameters found with NDT on the five ashlar were modelled using the mathematical software Surfer and using the kriging process as statistical interpolation method.

2.2.1. Electrical conductivity and capacitance

This technique was used for contact and contactless measurement of surface and subsurface moisture content of ashlar. As conductive materials present in the stone and mortar, salts induce an increase in the values detected by the instruments [43].

The Protimeter Surveymaster dual-function moisture meter used, with two fixed points spaced at 13.5 mm, measured the surface

moisture as wood moisture equivalent (%WME) values across a range of 6%–90%, while subsurface moisture content was found with radiofrequency (capacitance) over a range of 70–999 at 19 mm below the surface [43].

The 50 × 50 mm sampling grid prepared for each ashlar was also used in the other techniques to ensure inter-technique comparability. Measurements were made by placing the detectors in the middle of each grid cell.

2.2.2. Ultrasonic pulse velocity (UPV)

Another technique used to identify the damage was ultrasonic pulse velocity. The data gathered with this technique can be related to the presence of voids, such as pores or fissures, and to the degree of stone decay, since these structures attenuate the transmission velocity of the waves [44]. The CNS Electronics LTS Pundit UPV tester with 1.2 cm diameter 1 MHz transducers used was fastened to the stone with putty. The measurement mode used was the indirect mode, placing the transducers in parallel. The distance between transmitting and receiving transducers was 50 mm, using the same grid as in the electrical conductivity and capacitance technique. The measurements were made by maintaining the distance between the transducers and moving them horizontally between each of the measurements made, from one corner of the grid to the end point. The procedure was repeated in each of the rows that made up the grid according to the size of the ashlar.

2.2.3. Infrared thermography (IRT)

This technique delivers surface temperatures in real time with no need for physical contact [29,45–48]. The infrared thermographic study was performed with a FLIR ThermoCamTM B4 camera featuring a spectral range of 7.5 μm–13 μm, a temperature detection ranges of −20 °C to +130 °C, thermal sensitivity of 0.08 °C and a resolution of 640x480 pixels. Given the importance of accurately determining material emissivity, the values were referred to a high quality contrasting adhesive tape of known emissivity (0.95) [49].

Inasmuch as the samples were expected to exhibit high salt concentration, active IRT was used to elude the shortcomings of the passive technique in high salt media [50–52]. The surface was thermally activated with two 500 W flashlights positioned symmetrically around the camera centreline to heat the surface for 10 min at a distance of 1 m to ensure uniform and sufficiently intense heating to observe inter-area differences. The images were processed and analysed with ThermoCam Researcher 2.10 (FLIR) software. The estimated emissivity assumed for the dolostone and the cement mortar was for both 0.96 [52,53].

2.2.4. Impedance testing

A Microflown Technologies impedance gun was used to measure the value of the sound absorption coefficient and generate a color representation of the material surface. Sound pressure, particle velocity, sound intensity or acoustic impedance are other magnitudes that can also be calculated with this technique. Among all these acoustic parameters, the absorption coefficient has been found to be the most useful for establishing the surface condition of the material [35].

The gun was fitted with a 15 cm diameter spherical loudspeaker as the source of sound, a combination sound pressure MEMS microphone/particle velocity sensor and a handheld body that ensures ease of shooting and a constant 27 cm distance between sensor and loudspeaker. Its Microflown MFSC-2 scanning probe was connected to a laptop computer where all the signals were logged and the data were processed using Velo software.

Here the data were collected with the Scan & Paint tool [54] for more convenient comparison with the other techniques deployed. Working frequencies ranged from 500 Hz to 5000 Hz. The results are displayed in octave bands.

Although this technique has been applied to detect damage in construction materials [55], to date it has been consistently used under laboratory conditions only, making this a first-time in situ experience.

2.2.5. Electrical resistivity tomography

This technique is used to determine the resistivity of a material, i.e., its resistance to the flow of electrical current across its length [56,57]. Resistivity is related to a number of parameters, including moisture content, the presence of soluble salts or other ionic compounds transporting electric current, permeability or porosity, and the presence of other materials [58–62]. Broadly speaking, a high moisture content in combination with a high ionic load and considerable porosity favours current flow and decreases the electrical resistivity of a material. The Geolog2000 GeoTom ERT analyser deployed was used in conjunction with the Wenner four-point method, consisting in applying an electric current between external electrodes and recording the drop of voltage in the internal electrodes [63] as per the equation:

$$\rho = 2 \cdot \pi \cdot a \cdot R$$

where ρ is resistivity in Ω·m, a the spacing between electrodes in metres and R resistance in Ω.

Due to the difficulty for ensuring a good contact between the electrodes and the ashlar, especially in the case of cultural heritage, where is not possible to drive the electrodes into the material and the surface usually has a high roughness as a result of alteration processes, the Geolog2000 GeoTom ERT was attached to each surface by a device designed for that purpose. This device consists of a bar with a series of aligned stainless steel electrodes 0.50 cm in diameter and 0.25 mm apart. Absorbent sponges are housed in the electrode heads. All sponges were wetted, prior to measurement, in a 0.5 M NaCl saline solution to ensure a proper current flow between both materials. Inside the bar, each electrode has a compressive spring that allows, once the bar is supported on the wall and maintaining a certain pressure by the operator, to transmit that pressure and guarantee a correctly couple between the electrodes and the surface. In addition, before performing the measurement, the equipment used for the measurement (Geolog 2000) performs a coupling test to validate that the coupling between the electrode and the stone is correct and ensure that the electrode-stone resistivity is low enough to guarantee good results (in this study, 200 mV was established as the maximum acceptable potential value). Measurements were taken at a frequency of 8.33 Hz. Three horizontal measuring profiles were performed in each ashlar: one each at

approximately 50 mm from the upper and lower rims of the stone and a third across the middle. The number of active electrodes in each measurement was adjusted to the length of each ashlar. Under these conditions, the equipment makes a progressive scan of the entire line of connected electrodes, selecting them 4 by 4, which allows to obtain a resistivity profile throughout the ashlar and at different depth levels (as the distance “a” between the 4 active electrodes is increasing). The data were used to map the subsurface resistivity profiles in the measuring areas from the data inversion performed with RES2DINV software.

3. Results and discussion

3.1. Characterisation of the materials

The ashlars were originally hewn from beige-coloured, massive, tightly cohesive dolostone and with a microcrystalline texture (Fig. 3A). The internal microstructure of the rock was observed to comprise 75% anhedral dolomite microcrystals (30–60 μm) forming fossil remains (bioclasts) and massive grains (intraclasts), and larger (125–500 μm) euhedral crystals that cemented the fossil grains and intraclasts (Fig. 3B). Monocrystalline quartz grains (62–125 μm ; <5%) and crystalline calcite cements were identified as secondary components. Intercrystalline porosity was found to range from 5 to 10%. Given its characteristics and location near historic quarries, this stone must have been taken from Upper Cretaceous formations in the vicinity [64].

The inter-ashlar joint mortar proved to be a massively textured, tightly packed, granular, very hard and mechanically strong grey modern cement with a binder:aggregate ratio of 1:4 to 1:5 (Fig. 4A). The siliceous aggregate was observed to be angular, poorly sorted, have a particle size ranging from 0.5 mm to 4 mm and contain quartz, feldspars (microcline and albite), micas (biotite and muscovite), and metamorphic (schists and slate) and plutonic (granite) rock fragments (Fig. 4B). The scant binder present consisted in a dark-toned, microcrystalline (<4 μm), partially carbonated (calcite) mass with small aggregates of crystals consisting in calcium silicates (XRD: alite $3\text{CaO}\cdot\text{SiO}_2$ and belite $2\text{CaO}\cdot\text{SiO}_2$) and fibrous masses of calcium aluminates (XRD: mayenite $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$), all characteristic of hydraulic binders (Fig. 4C). The binder and aggregate were observed to be tightly bonded, with porosity on the order of 5%–10%.

Sulfates and primarily nitrates were found to be clearly present on both the mortar and ashlar surfaces and subsurfaces, although the mortars had a higher anionic load than the stone (Table 1).

The generally well crystallised salts present in the stone ashlars formed white powdery masses filling the subsurface and surface (subsurface and surface efflorescence) pores or brownish hard surface crusts. The salts identified by XRD were primarily: potassium nitrates (nitre - KNO_3) and various types of magnesium (hexahydrate - $\text{MgSO}_4\cdot 6\text{H}_2\text{O}$), sodium (thenardite - Na_2SO_4) and to a lesser extent calcium (gypsum - $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$) sulfates. The white powdery efflorescence that prevailed in the mortars was observed to primarily affect the binder. The salts present, and identified by XRD were: ettringite $[\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}]$, a sulfate typical of artificial cement, along with calcium sulfates (gypsum), sodium nitrates (nitratine - NaNO_3) and magnesium carbonates (hydro-magnesite- $\text{Mg}_5(\text{CO}_3)$), products of interaction with the environment.

3.2. Non-destructive technique inspection

3.2.1. Electrical conductivity and capacitance

Ashlar surface moisture, as measured via electrical conductivity, ranged widely from 41% to 90% WME (Table 2). Ashlars 1 and 5, which exhibited alveoli and scaling respectively, were the two with the highest surface moisture content (Fig. 5, left). In this latter case, in particular, the percentage was 90%. The lowest value, 41%, was recorded for ashlar 4, the best conserved and located adjacent to stone 5. The other two ashlars had similar mean values and an uneven distribution with a higher moisture or salt content around the edges (Fig. 5, left).

In contrast, subsurface moisture in the ashlars, measured by electrical capacitance, exhibited medium-high values (715–932). Ashlars 4 and 5, in particular, had the highest values and most uniform distribution, while ashlars 2 and 3 also exhibited high but more variable values (Table 2 and Fig. 5, right). The capacitance values, while lower (715) in ashlar 1, also ranged widely. Capacitance was lowest around the edges in ashlars 1, 2 and 3 (Fig. 5, right).

Surface moisture was highest in the ashlars with greatest decay and salt content (ashlars 1, 2 and 5), although that was not the case in the subsurface, where ashlars 1 and 2 had a lower moisture content than ashlar 5 (Table 2). Ashlar 1, whose surface was more severely decayed than any other, was the least moist below the surface (715). On the contrary, the ashlars with least severe decay (stones 3 and 4) had a lower surface (41%–68% WME) but a higher subsurface moisture (capacitance values): 932 in ashlar 4 (Table 2).

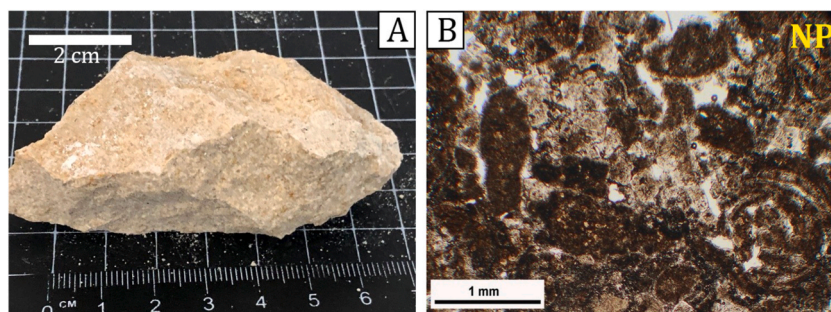


Fig. 3. Dolostone (A) and its PM image (B) in parallel polarised light mode (NP), showing intraclasts and bioclasts.

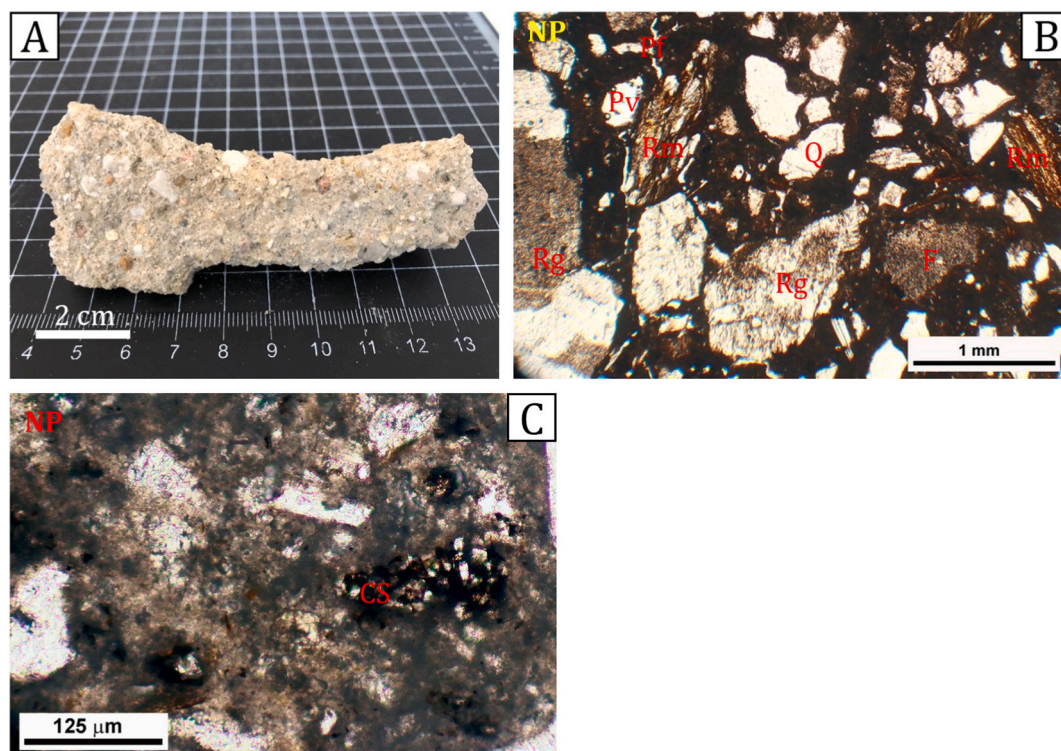


Fig. 4. (A) Joint cement mortar and their PM images in parallel polarised light mode (NP) of: (B) angular siliceous aggregate containing quartz (Q); feldspars (F); granite fragments (Rg); schist and slate fragments (Rm); and areas with scant fissure (Pf) or vacuole (Pv) porosity; and (C) hydraulic binder bearing calcium silicates and aluminates (SC).

Table 1

Surface and subsurface anion content in ashlar and mortars.

Sample	Depth	Chlorides (mg/L)	Nitrates (mg/L)	Sulfates (mg/L)	Oxalates (mg/L)
Mortar	0 cm	85	484	111	1.6
Stone	0 cm	5.8	82	29	BD
	1 cm	16.2	114	78	2.8
	3 cm	12.2	74	19	7.1
	5 cm	13.9	74	30.4	1.2

BD: below instrument detection limit.

Table 2

Surface and subsurface conductivity and capacitance in ashlar and mortar joints.

	Ashlar	Stone				Mortar			
		Mean	std	Max	Min	Mean	std	Max	Min
Surface moisture (contact, %WME)	1	72	25	95	12	92	9	95	58
Conductivity	2	79	14	95	48	95	0	95	95
	3	68	8	93	45	95	0	95	93
	4	41	32	94	8	84	15	95	32
	5	90	11	95	49	94	5	95	68
Subsurface moisture (contactless)	1	715	131	900	450	671	220	860	230
Capacitance	2	895	148	990	250	826	81	980	630
	3	905	126	960	250	809	113	960	360
	4	932	63	990	770	892	69	990	700
	5	908	67	990	710	895	77	990	720

Mean - average value; std - standard deviation; Max - maximum; Min - minimum.

The presence of hygroscopic salts on the surface of these ashlar, such as nitrates and chlorides, impacted electrical conductivity readings, which were driven upward by the effect of the ionic conductivity of their components. In areas characterised by fissures, cracks, cavities and mass loss (ashlar 2, 3 and 4) surface moisture was locally higher than below and in the rest of the surface (Fig. 5).

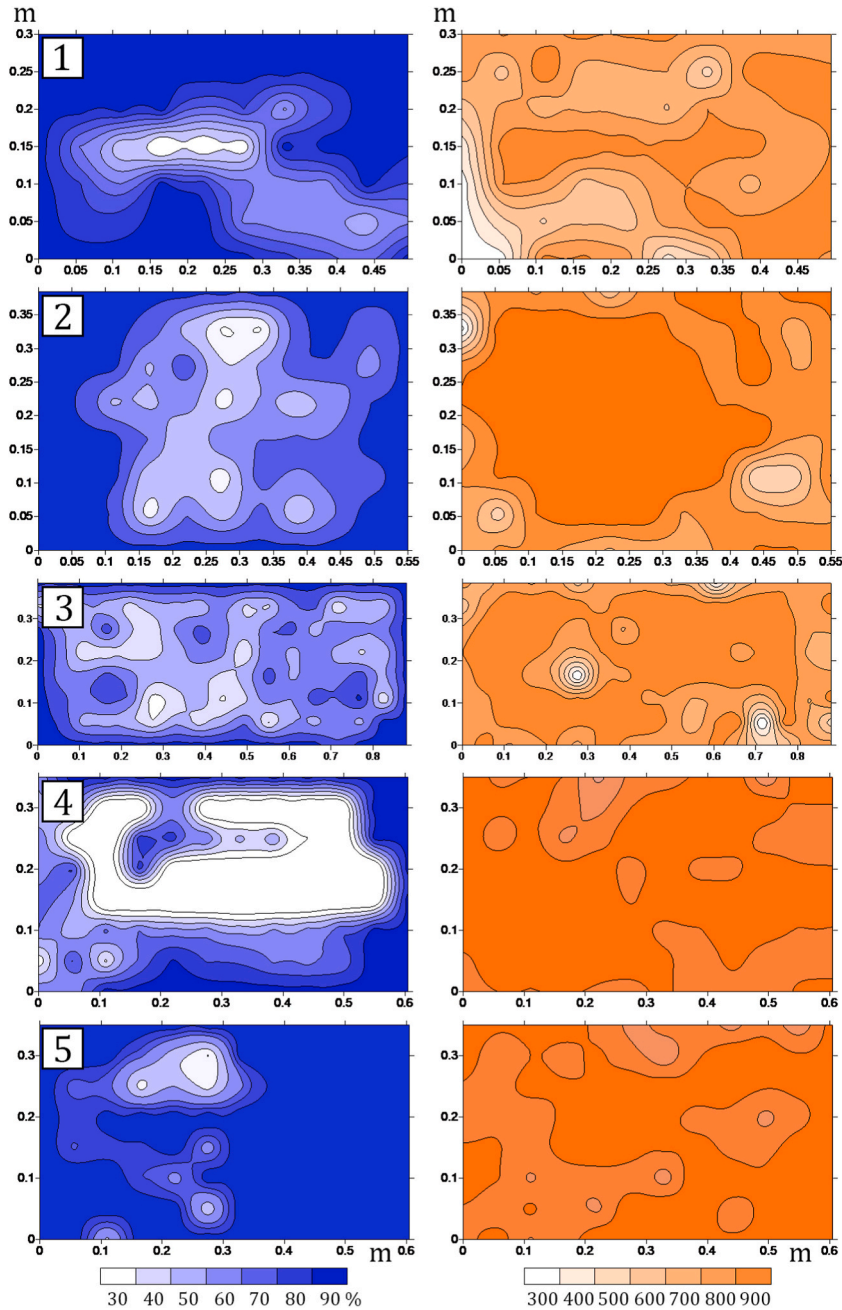


Fig. 5. Electrical conductivity (left) and capacitance (right) distribution in the five ashlars studied.

The presence of moisture and its effects inside stone ashlar cavities or alveoli has also been detected in geological outcrops exhibiting tafoni (honeycombing) [30]. Ashlar alveolisation due to differential evaporation-driven drying may be favoured by subsurface salts.

The surface of the joint mortars was moister than the surface of the stone and locally saturated (>90% WME) except in ashlar 4 (Table 2), possibly due to its smaller thickness [65]. In contrast, subsurface moisture was slightly lower in the mortar (671–895) than in the ashlars (Table 2). The surface moisture in the mortar around ashlar 4 was, as in the adjacent stone, the lowest recorded in mortar (84% WME), and subsurface moisture in this mortar was also the lowest, as in ashlar 1 (Table 2).

Moisture, and with it salts in the joints, migrated from the subsurface of the mortar to its surface up to the edges of the ashlars, where the highest capacitance values were observed in cavitated areas. That may mean that given the lower mortar than stone porosity, the moisture accumulating in the former flowed toward the stone, mobilising ashlar salts and favouring their decay. That hypothesis would correlate well with the effect of incipient alveolisation visible along the right side of ashlar 2 (Fig. 2). The ashlar edges where material has detached exhibited lower subsurface moisture due to the evaporation taking place there. The same could be said of the

wider fissures found in ashlar 3 and 4 (Fig. 5).

Earlier studies [66] showed that saline solutions may be very effective for sealing stone pore networks, affecting subsequent moisture permeability and evaporation patterns. The processes involved are not uniform on ashlar surfaces but impacted by the direction of the saline solution and water vapour flows [29,66,67]. That is relevant to scaling- and flaking-mediated decay, where surface and subsurface moisture is high. Lower values of both types of moisture are associated with the areas where some materials have broken away, such as in the centre of ashlar 5 where salts were observed below the flaking (subsurface saline efflorescence, Fig. 5). This explains why exists an inversely correlation ($R^2: 1.0$), in ashlar 1 and 2, between the values of conductivity and electrical capacitance. In these ashlar, surface moisture is located at the edges of the ashlar, with a lower degree in the centre. On the contrary, in depth (up to about 2 cm) the edges present a lower value of capacitance and higher in the centre, which is due to a distribution or accumulation of salts in the central and right lateral zone of the ashlar, as occurs in ashlar 1. Meanwhile, in the other ashlar the values are similar with a correlation close to zero.

Lastly, the findings showed capacitance readings to be practically unaffected by the presence of soluble salts, except in the presence of deliquescence salts [68]. Inasmuch as these measurements were made in winter with a relative humidity of $83.2 \pm 8.6\%$, the salts in the materials may have dissolved, which would explain the higher subsurface capacitance values in the ashlar studied.

3.2.2. Ultrasonic pulse velocity (UPV)

Interpreting the ultrasonic velocity readings from measurements made in situ is less straightforward than interpreting laboratory results because relative humidity varies with environmental conditions at any given time. Variations in in-stone moisture content and saline phases also impact ultrasonic wave propagation.

The average ultrasonic pulse velocities in stone and joint mortars were 1387 ± 781 m/s, and 1419 ± 639 m/s respectively (Table 3). As the data in the table show, the results for the ashlar studied were widely scattered. The quarry stone has a UPV, measured indirectly, of 2042 ± 349 m/s. The difference between the values measured in the ashlar and that recorded in the quarry stone indicates the degree of deterioration of the stone used on the monument.

Further to the data in the table, the UPV values were lowest around the edges in most of the ashlar, an indication that decay was most severe in those areas (Fig. 6). The highest values, over 2000 m/s, were observed in areas with hardened saline crusts (ashlar 1, Fig. 2) or traces of the original stonework on their surface (ashlar 2, 3 and 4, Fig. 2). The most severe surface decay (alveolisation, scaling, crumbling, ...), mirrored in the lowest values (923–1170 m/s) across most of the surface, was found in ashlar 1 and 5 (Fig. 6).

Ashlar 1, with the greatest decay and a mean UPV of 923 m/s, had high values (up to 3300 m/s) in the centre only, where the most protuberant hardened salt crusts were also present (Fig. 6). The same development was visible in ashlar 2, where areas with incipient alveolisation (right side) exhibited the lowest values (<1000 m/s) (Fig. 6), while the highest values (>3200 m/s) were observed in the mid-space between the less damaged area and the decayed surfaces (Fig. 6).

The highest values in ashlar 3 (>2000 m/s) were recorded in the centre of the stone, where most of the original surface was in good condition, despite incipient alveolisation and precipitated salts not only on but also possibly below the surface, perhaps associated with scaling (Fig. 6). Lower values were logged in deeply cracked or cavities areas. That was observed in ashlar 5, where the lowest values (<600 m/s) were observed in the severely flaked centre, denoting area weakness due to detachment of some of the flakes from the rest of the stone (Fig. 6). The densest areas, where values of 1500 m/s to 2500 m/s were registered, the flakes had detached altogether, leaving a more tightly packed stone.

Lastly, ashlar 4, with fissures and cracks of different sizes and at varying depths, exhibited a more irregular ultrasonic velocity distribution (Fig. 6). This ashlar also had the highest capacitance values (subsurface moisture) in both stone (932) and mortar (892), which affected ultrasonic wave behaviour. In walls with no inner moisture, the UPV is lower than when the pores are water-saturated, for velocity is faster in water than in air (water: 1450 m/s; air 330 m/s). If the water contains dissolved ions, as is usually the case, velocity may be even higher, up to 1550 m/s, depending on the nature of salt and concentration of the ions [69].

In addition, evaporation-mediated salt crystallization from inside the stone and/or mortar tends to fill the pores and consequently raise UPV [70,71]. According to the temperature vs. relative humidity phase diagrams of the salts, the sulfate salts in the ashlar studied, under the temperature conditions that affect the monument, primarily hexahydrate ($\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$) and thenardite (Na_2SO_4), can hydrate to epsomite ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) and mirabilite ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) if the relative humidity exceeds 86.6% or the humidity inside the materials is over 76.4% [72]. At such values hydration entails a rise in salt volume, which may close the pore network and raise its UPV. If relative humidity rises above the stability values for these hydrated phases (92.7% for epsomite and 83.3%–95.1% for mirabilite), they may deliquesce, generating an aqueous solution [73].

Table 3

Mean ultrasonic pulse velocity (mean), standard deviation (std) and maximum (Max) and minimum (Min) values for stone and mortar.

	Ashlar	Stone				Mortar			
		Mean	Std	Max	Min	Mean	Std	Max	Min
Ultrasonic pulse velocity (m/s)	1	923	671	3333	123	1454	593	2414	526
	2	1504	693	2800	170	1510	431	2121	761
	3	1683	714	3333	326	1316	810	2593	376
	4	1260	632	2333	122	1505	737	2800	226
	5	1178	743	3333	270	1198	553	2244	574

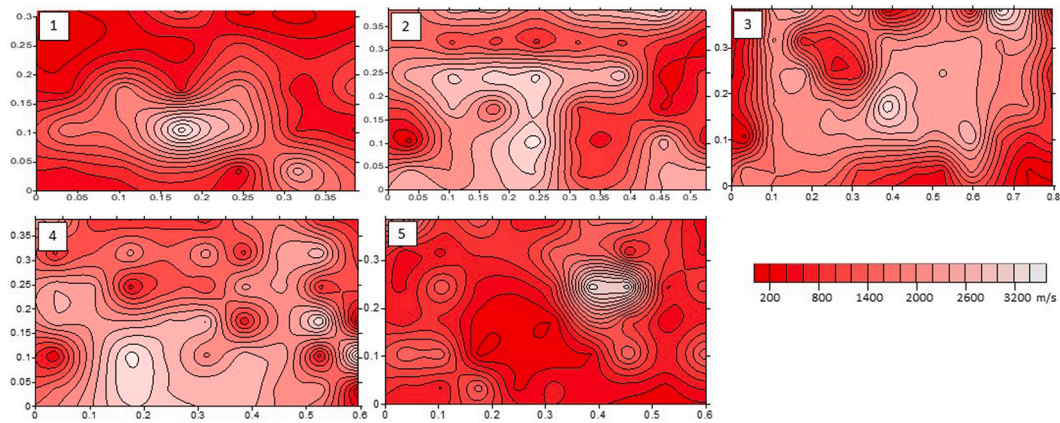


Fig. 6. Ultrasonic pulse velocity distribution in the five ashlar studied.

3.2.3. Infrared thermography

According to the infrared thermography findings, in general the mortars had higher apparent temperatures than the ashlar surfaces (Fig. 7). That may be the result of two effects. The first is the difference in the thermal response deriving from the distinct mineralogy of the two materials. The dolostone would foreseeably exhibit thermal effusivity of approximately $1887 \text{ kJ}/(\text{K}\cdot\text{m}^2\cdot\text{s}^{1/2})$ (assuming stone thermal conductivity equal to $1.70 \text{ W}/\text{m}\cdot\text{K}$ and density $2000 \text{ kg}/\text{m}^3$ to $2190 \text{ kg}/\text{m}^3$), whereas mortar effusivity would be $1944 \text{ kJ}/(\text{K}\cdot\text{m}^2\cdot\text{s}^{1/2})$ (estimating thermal conductivity equal to $1.80 \text{ W}/\text{m}\cdot\text{K}$ and density to $1.800 \text{ kg}/\text{m}^3$) [74]. The second possible explanation would be the presence in the mortar of hygroscopic salts such as ettringite whose high thermal inertia would retain its temperature for longer during both cooling and the liquid-to-solid phase change, with the concomitant release of thermal energy [75].

Temperature was seen to vary across ashlar 1 depending on if measure is taking inside of the alveoli or outside. Those formations act as conduction thermal insulation, lowering conduction-mediated heat transfer and temperatures relative to undecayed areas [46, 49,52,76]. An example was visible in ashlar 2, where a clearly distinguishable line separated the alveolised area on the right from the healthy stone on the left (Fig. 7).

The presence of scaling or saline crusts slightly detached from the substrate, visible primarily in ashlars 2 and 5, generated a subsurface air chamber where a number of phases could co-exist (air, water and/or salts). That would explain the higher temperatures in those areas than in the rest of the surface (Fig. 7) [52,77], even as the air at the back of the stones resisted heat flow [49].

However, the presence of deep cracks on ashlar 3 and 4 has no notable effect on the thermography as opposite to what happens in surface damages [78] which denotes a better conservation state than the previous ones.

3.2.4. Impedance testing: sound absorption

Stone is a rigid material with a sound absorption coefficient ranging from 0.1 to 0.4, depending on its porosity [78, 79]. Local variations of these values of the absorption coefficient on its surface will be indicative of the presence of alterations or defects [35, 80] on it. This fact should be taken into account in the tests and in the interpretation of the images obtained. Fig. 8 shows for each ashlar

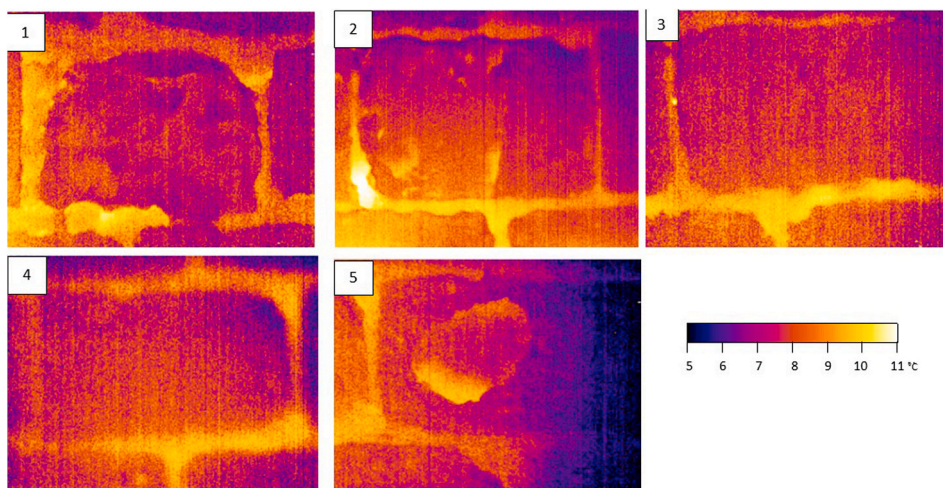


Fig. 7. Active thermography-determined ashlar surface temperature distribution.

two images measured for two different octave bands. The selected frequencies of the images have been chosen because they are the frequencies at which, for each ashlar, the different types of lesions on its surface and their size are best visualized.

At 3150 Hz (Fig. 8) areas with an absorption coefficient close to 0.6 were visible across ashlar 4 in its entirety, denoting a rise in porosity due to surface decay as well as decay at the more granular stone-mortar interface. The bright and dark blue areas in the images, indicative of very low absorption coefficients, may confirm the presence of salts forming a hard crust that would fill pores and disguise surface decay. At that frequency, with a wavelength of around 10 cm, diffraction around ashlar 4 edges provided obvious evidence of a fissure. In this case, the frequency at which it first became visible afforded an approximate estimate of its length, which would have to be under 10 cm. In the 5000 Hz image (Fig. 8), the fissure was clearly visible as an area of maximum absorption. Given its depth, its behaviour as a simple resonator [36] might also explain why it could be visualized at this higher frequency. At a shorter (~ 6 cm) wavelength and a resolution of 5000 Hz, the more porous areas were readily distinguishable from the ones with lower porosity, whilst the red areas denoted the presence of edges or arris < 6 cm [78].

At 1000 Hz the presence of surface grooves and salts on ashlar 3 raised stone porosity; its unsmooth surface exhibited an absorption coefficient of 0.6–0.7. Here also, the blue tones in the centre were possibly related to hard-crusting salt concentrations that would fill the pores in these areas. At 4000 Hz the absorption coefficient rose in the most porous areas, whilst the red areas would be indicative of misalignment between ashlar and joint mortar. At that frequency the unevenness induced by central scaling was visible on the image as an area of maximum absorption due to the effect of diffraction [79].

At 1600 Hz and 2500 Hz the ashlar 5 surface exhibited a higher absorption coefficient in the centre, surrounded by lower, blue-coded, low coefficient areas. The blue areas would denote a stiff, smooth, barely decayed surface with a salt crust filling its pores. The edges of the scaled central area were more clearly visible at the higher resolution (2500 Hz) [78,79] as diffraction indicating that the areas where surface material had detached was 10 cm–12 cm wide. The images obtained for ashlar 1 can be explained in much the same way. The highest absorption coefficient values would denote increased porosity as a result of surface decay. The coefficients for these areas rose with frequency and the blue areas would identify the regions with the least porous surfaces or hard salt crusts. A higher frequency, 1250 Hz, delivered a more detailed image of surface area types in this ashlar.

At 1600 Hz, alveolisation and the presence of salt on the ashlar 2 surface translated into an image where alternating reflective and absorptive behaviour denoted an unsmooth, irregular surface with different types of damage in areas with different tonalities. Although determining the specific origin of the type of decay would entail inspecting the surface, blue (low absorption coefficient) areas can be interpreted to have a smooth texture (perhaps the original stone) or a salt crust, whilst higher absorption coefficient (red) areas would denote surface decay (alveoli, cracks ...) that would be more intense with rising ashlar porosity.

3.2.5. Electrical resistivity tomography

Fig. 9 shows the subsurface electrical resistivity profiles obtained by inverting the data recorded in the three areas of each ashlar measured, together with the number of interactions performed and the error made in the inversion process.

The low resistivity values logged for ashlars 1 and 2 indicated that, at the depth closest to the surface (up to 2.54 cm), and at the different heights at which the measurements were taken, the ashlars had areas with some decay, including alveolisation (Fig. 9). As discussed earlier when analysing ashlar moisture, that was attributable to: the higher moisture content on the inner side of cavities and alveoli; possibly to accumulated salts; and also to the greater porosity in decayed areas, as corroborated by the lower ultrasonic velocity values observed.

Resistivity rose at deeper depths (due to lower moisture, salt content, and porosity), where intensification of the inter-area differences enhanced perception of the pathologies present. From 3.73 cm inward, ashlar 1 exhibited greater resistivity in the centre (at 0.15 m height) and lower right side (at 0.05 m height), which was consistent with the hardened salt crust detected with the techniques discussed above (UPV and impedance testing). Regarding ashlar 2 and at a depth of 3.73 cm, higher resistivity was logged in the centre-left side of the entire ashlar, where the stone was in the best condition and hence less porous, as attested to by the UPV values.

The ashlar 3 resistivity profiles were also correlated to some extent with the pathologies detected by the other techniques studied. Scaling was detected up to a depth of 5 cm at 0.31 m from the ashlar base (upper horizontal line), and at a distance of about 30 cm from

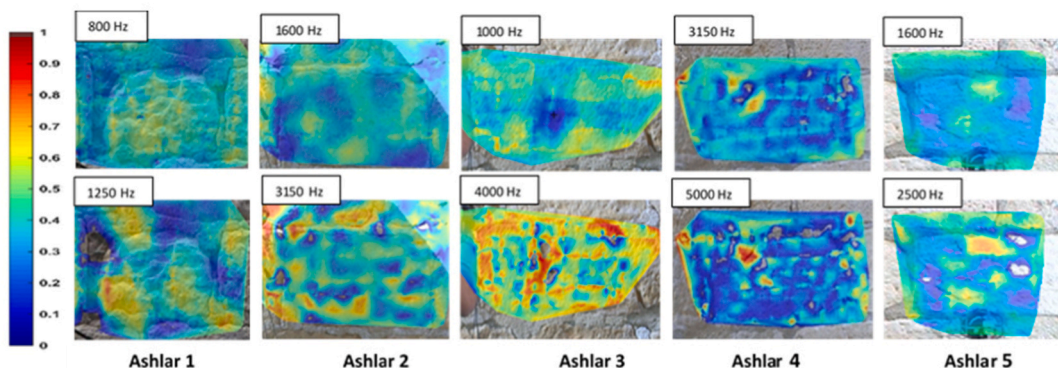


Fig. 8. Sound absorption distribution at different frequencies in the five ashlars studied.

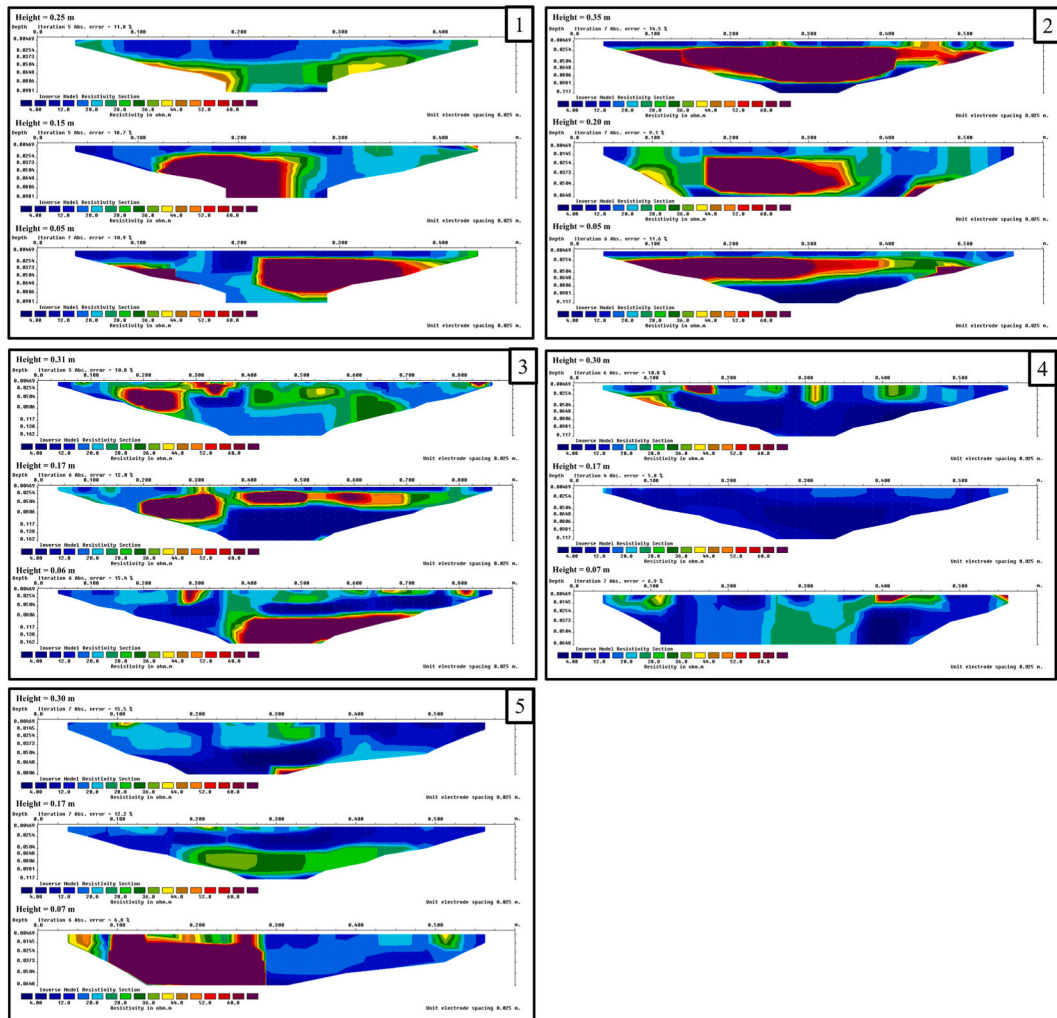


Fig. 9. Subsurface electrical resistivity profiles for the five ashlars studied.

the left-hand corner. Smaller areas with lower resistivities were also observed, very likely identifiable with the ones bearing pick marks (surface finish) of sufficient depth to be recorded by the tomograph at the low frequency used in this study (8.33 Hz). Resistivity was higher in subsurface areas lying below the pick marks where the material was less intensely decayed. Further to the profiles obtained near the bottom (0.06 m) and at mid-height (0.17 m) in this ashlar, scaling continued to the base of the stone, with a concomitant reduction in its width (from about 8 cm at 0.31 m height to 1 cm at 0.06 m height). The rise in electrical resistivity with depth, observed in the profile obtained at a height of 0.17 m through a layer approximately 2 cm thick in the central area (from 40 cm horizontal distance), extending to both sides of the profile, could be interpreted as variation in moisture content with depth or a decline or obstruction in material porosity. The lower moisture is consistent with the capacitance readings at a depth of 1.9 cm graphed in Fig. 5. It is also consistent with the UPV and impedance testing results that indicate that this zone has a greater consistency related to a lower porosity or to the presence of a salt crust (Figs. 6 and 8). Below that depth resistivity declined, possibly inferring the presence of a) greater porosity that would favor current-driven dissolved ion mobilisation; b) saline phases; or c) a higher moisture content. Those two factors, salts and moisture, may be the precursors of scaling in the sounder upper layer of the ashlar, an argument consistent with the findings of the other tests conducted. This technique can also detect some of the cracks located in ashlar 3, such as the one at the lower right zone, detected as a rise in electrical resistivity at a height of 0.06 m, which has a depth of approximately 2 cm.

Ashlar 4, with less severe surface decay, exhibited uniformly low resistivity values at the deepest depths. Such findings may be attributable to greater ashlar porosity, a higher dissolved ion content or greater subsurface moisture content (which is compatible with the conductivity data and UPV). That result was also visible because unlike the others, ashlar 4 exhibited a decline in resistivity with depth, an indication that one of the aforementioned parameters rose with depth. Ashlar fissures were perceptible and could be characterised from the subsurface profiles obtained at different heights. The profile obtained across the upper horizontal line (at 0.3 m from the ashlar base) records the fissure observed in the ashlar, which is fairly wide and has a depth of 2.5 cm. The presence of a fissure in the central zone can also be seen at this same height. The crack near the base was not recorded because it was positioned below the

bottom horizontal line (at 0.07 m off the base). In contrast, the lower profile did show two high resistivity areas at the ends and with a depth of less than 1.5 cm. One of them corresponds with the presence of a smaller fissure, such as the one observed on the right border (Fig. 2), while the other may denote the presence of a hardened saline crust.

The upper (0.30 m) and mid-height (0.17 m) subsurface profiles generated for ashlar 5, in turn, exhibited an area of high electrical resistivity at around 1.5 cm below the surface, coincident with the lower edge of the scaling layer. Below that area resistivity values declined, very likely as a result of the moisture and salts accumulating underneath the scales. Such accumulation of salts was confirmed by the respective thermogram (Fig. 7). Scaling was thickest at the bottom of the ashlar where very high resistivity values were found to a depth of 6.5 cm, affording evidence that the area affected must be detached from the rest of the stone. That separation, which is consistent with the thermographic camera results, could obstruct the electrical field penetration into the deepest areas of the ashlar or hinder their flow. The ultrasonic velocity values were directly correlated with the electrical resistivity values in this ashlar. Both higher resistivity and lower ultrasonic pulse velocity were associated with detachment of the central layer.

The resistivity values recorded on the scantily decayed left side of the ashlar were low as a rule, arguably due to the high moisture content deduced from the capacitance data and the thermographic findings (lower temperatures would be due to a higher moisture content).

4. Conclusions

Combining different non-destructive techniques delivers effective and reliable results on the degree of decay and its effects on material surfaces and subsurfaces. Such combined use also yields fairly accurate data on the distribution of the various forms of decay and the presence or absence of moisture and dissolved, crystallised or hard-crusts salts. That is particularly important since interpreting the data obtained directly from outer-facing ashlar stone in monuments, exposed to uncontrolled changes in environmental temperature and humidity, is less straightforward than interpreting laboratory results.

The crystallization, rehydration and deliquescence dynamics associated with different salts in such media complicates matters further. Crystallization and hydration phase changes in the existing salts close the pore network wholly or partially depending on the material (mortar or stone) and raise salt volumes. The surface formation of powdery or hard-crusts saline efflorescence is favoured by moisture evaporation in the ashlars. That induces a rise in electrical conductivity, infrared thermographic, electrical resistivity and ultrasonic velocity values, and declines in sound absorption. Salt deliquescence, favoured by high relative humidity and low ambient temperatures, increases the content of dissolved ions on the surface of the ashlars (hard-crusts saline) and their interior, which affects instrumental readings: ultrasonic velocity and capacitance are higher and electrical resistivity values are lower than in salt-free zones.

Salt hydration involving subsurface efflorescence favours ashlar surface scaling and flaking. That, in turn, translates into rising thermographic, sound absorption, electrical conductivity and electrical resistivity values and declining ultrasonic wave propagation. Efflorescence in cavities induces alveolus formation. The presence of fissures and cracks favours evaporation with declines in electrical conductivity and ultrasonic velocity values and rises in sound absorption and electrical resistivity.

More severe decay around ashlar edges is associated with cement mortar stiffness and bonding and the moisture and salts migrating from that material to the adjacent stone. The findings for all the ashlars were obviously impacted by the joint mortars, source of the moisture that afforded salt mobility. Salt concentration was highest and electrical resistivity lowest in the areas closest to the mortars. The effects on test results include a rise in infrared thermographic, ultrasonic velocity and electrical conductivity values and a decline in electrical resistivity at the interface between the two materials.

The non-destructive techniques used are wholly complementary, confirming the validity of all five for characterising and interpreting damage and its impact on construction materials. Ultrasonic pulse velocity, known for its accuracy and widely deployed in many fields of science, here confirmed the findings delivered by other techniques, electrical resistivity tomography in particular, for both have sufficient penetration power to provide reliable data on the internal structure of materials.

Surface electrical conductivity and subsurface electrical capacitance, although normally used for organic materials such as timber, proved here to be quite versatile and deliver highly reliable results on moisture content in natural (stone) and artificial (mortar) inorganic materials. The data afforded by these techniques correlated closely with the findings of other related procedures for detecting damage such as ultrasonic velocity and electrical resistivity.

Active infrared thermography corroborated and complemented the other non-destructive techniques deployed. Given that specific heat is greater in water than in air, high salt reflections associated with phase change identify areas of higher temperature in both mortar and stone, as well as in areas affected by scaling and flaking in the presence of salt. In contrast, lower surface temperatures are found in areas with alveoli, which act as thermal insulation in moist areas.

Pathologies are effectively detected with the interpolation data processed from electrical tomographic readings. Subsurface data in particular are useful for characterising pathologies observed *in situ*. Measurements should nonetheless be made with much higher frequencies than used here to raise the resolution and enhance perception of more superficial pathologies.

Impedance testing, based on the sound absorption coefficient, is a novel non-destructive technique that is a valuable complement to other more routine methods. It is a convenient imaging technique that supports qualitative analysis of the condition of some surfaces. Changes in the surface absorption coefficient provide for a quick and qualitative estimate of both the degree of ashlar decay relative to absorption in undamaged material and of variations in the state of conservation in different areas of the stone.

As summarize, it can be observed that it is necessary to know the causes and degree of deterioration of the materials in the facades to be restored. The techniques based on acoustic impedance and infrared thermography allow to detect the areas with different deteriorations in a fast and economic way to be able to inspect large surfaces such as building facades. The other techniques are effective to determine the degree of deterioration and the distribution of humidity and salts on the surface and inside the stone ashlars.

However, the use of non-destructive techniques does not avoid having to carry out analysis with the extraction of samples to know the products of alteration, such as the existence of salts, which are generated both on the surface and inside the ashlar. These analyses are essential to know the environmental conditions that favor the deliquescence and hydration processes of the saline phases.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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