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Chemical and mineralogical characterization as a reliable approach for assessing the mechanical properties of ancient mortars and masonry: A case study of the bell tower of Melfi Cathedral (Basilicata, Italy)

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ABSTRACT
Construction techniques employed in historical buildings reflect the diversity of historical and cultural eras, constituting tangible evidence that must be preserved and transmitted. Their preservation requires a complete and detailed understanding of the materials used. Therefore, several investigation methodologies are addressed, carefully chosen in order to provide useful data for specific and effective restoration interventions, using minimal invasiveness.
In Italy, diagnostic services in the construction and cultural heritage sectors are regulated by NTC 2018 (Technical Standard for Construction). These technical standards, along with UNI (Italian Standards Body) norms, describe several investigation techniques for the characterization of construction materials. However, the described methodologies are not always applicable or suitable for specific purposes, particularly for the determination of the mechanical strength class of the masonry in historic and protected buildings.
In the present article, we present an alternative and valid methodology, focusing on the strength classification of the masonry in the medieval bell tower of the Cathedral of Melfi (Basilicata, Italy). The study includes chemical and physical analyses of mortars and stone bricks using laboratory micro-destructive techniques such as optical microscopy, X-ray diffraction and thermogravimetric analyses. The results evidence the validity of the micro-invasive scientific method, providing reliable data that is useful for specific and effective restoration and maintenance interventions, without the need for highly invasive and destructive approaches.

Section: RESEARCH PAPER

Keywords: diagnostics; masonry strength test; mortars; seismic vulnerability assessment; standards application; norms; optical microscopy; petrography; X-ray diffraction analysis; thermogravimetry; cultural heritage

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1. INTRODUCTION

Nowadays, the field of conservation of cultural heritage is widely known and studied. From a social point of view, the concept of heritage conservation contributes to strengthening collective memory and identity. At the same time, valorisation and fruition of heritage significantly contributes to a community's economy and development, especially in contexts where tourism represents a major industry. For these reasons, the safety and preservation of historic sites are fundamental to ensuring the stability of communities and the quality of life of their inhabitants.

As it is well known, conservation purposes require a deep consciousness of the material composition of the heritage, in order to have complete information aimed at planning restoring and maintaining operations. Materials testing is usually carried out by using several analytical techniques, to contribute to the understanding of material degradation causes, aimed at developing appropriate compounds for restoration and maintenance activities.

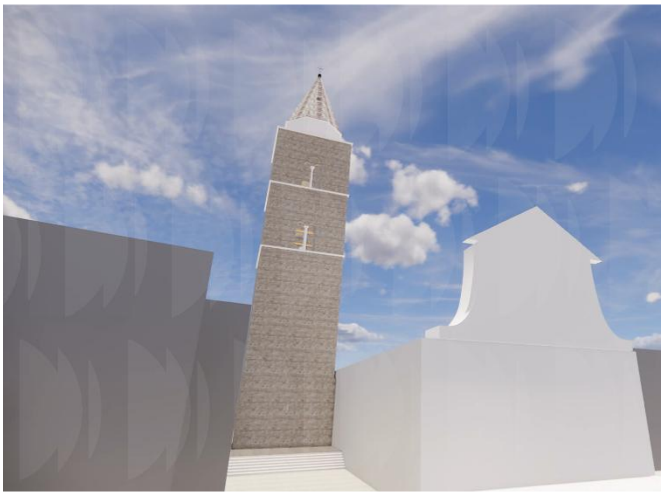

Figure 1. The medieval bell tower of the Cathedral of Melfi (Basilicata, Italy), as rendered by the HBIM modelling from the integrated survey [27] (ISTEMI© s.r.l.).

Besides the involvement of research institutes and universities, which basically work on innovative scientific experimental tests (not always feasible due to economic restraints), in Italy, the public and private sector also request diagnostic investigation services from companies specialized in the construction field and historical heritage.

Diagnostic investigations, even those performed on cultural heritage, are regulated by the Italian standard of the MIT (Ministry of Infrastructure and Transport) in the NTC 2018 [1], [2]: D.M. del Ministero delle Infrastrutture e dei Trasporti del 17/01/2018. Aggiornamento delle Norme Tecniche per le Costruzioni (in Italian), update of the former NTC 2008 [3], [4] first published as a Ministerial Decree on November 20, 1987 [5]. It explicitly refers to the Italian *Guidelines for the assessment and mitigation of the seismic risk of cultural heritage* [6] as a reliable source of guidance that can be employed for the seismic vulnerability assessment of buildings.

Among the different types of historic structures, masonry towers are recognized as one of the most vulnerable to seismic events [7], [8]. For this reason, and due to the variety and complexity of materials used, it is particularly challenging to adopt procedures reported for the mechanical characterization of the masonry [1], [2], [9] (specifically addressed to new constructions), especially without knowing the chemical-physical properties of the on-site material components. It is important to consider that historic materials have been subjected to several degradation processes, in addition to the fact that it is mandatory to respect the principle of non-invasiveness of operations, mainly adopting non-destructive (or, at least, micro-invasive) methods.

In accordance with NTC 2018, the determination of mechanical strength, aimed at characterizing pre-existing mortars, depends on the level of knowledge achieved during on-site investigations. Until today, the indicated regulation has only concerned the wall structure in terms of mortar placement [1], without considering the chemical and physical characteristics of the mortars used, often leading to an incorrect estimation of the strength parameters [10].

Table 1. Summary of samples collected and analysed.

Sample	Description	Analyses
C01, C06, C07	Lime mortar	OM (SL)
C02, C03, C04, C05	Cementitious mortar	XRD, TGA
P2, P4, P7, P10, P11, P12	Limestone (litho-bioclastic)	
P1, P6	Yellow tuff	OM (SS)
P3, P5	Igneous rock (haüynophyre [13])	

Furthermore, the *Resistance classes of mortars* (as reported in Table 1; 1.10.3.1.2 – *Estimation of compressive strength* in [1]) is exclusively referred to newly manufactured mortars, as parameters to be respected for new constructions. On the contrary, no defined requirements on the investigations of pre-existing materials, belonging to existing structures, are reported, for which only a brief mention is provided (§ 8.5.3. [1]).

Although nowadays experimental methods for the determination of the mechanical strength of masonry are proposed [11]–[14], it is always preferable to employ non-invasive methods in the case of cultural heritage [15].

Previous studies have demonstrated the applicability of micro-invasive methods and physical-chemical analyses on structures of historical and cultural interest. These approaches have been employed for characterizing the nature of mortars and masonry [16], [17], and finishing surfaces [18]-[20]. Generally, such studies aim to characterize materials to deduce manufacturing techniques, production technologies, and the origin of mortars and building stones [21], [22].

In the engineering field, physical-chemical analysis methods are notably utilized for evaluating the composition of mortars and concrete, as well as their degradation products [23], [24], and to evaluate the durability and compliance of construction materials [25].

Therefore, a valid and micro-invasive alternative methodology, appropriate for cultural heritage structures, is reported in the present work. The case study investigated is the medieval bell tower of the Cathedral of Melfi (Basilicata, Italy) (Figure 1), on which micro-invasive operations were executed, in compliance with the principles of non-invasive investigations. Chemical and physical laboratory tests were done on samples collected on-site, adopting non-invasive methods, to obtain the Resistance classes of the mortars [1], useful for determining an estimation of the masonry's mechanical characterization.

The investigations reported in the present paper are a part of the complex and rich diagnostic plan carried out on the historic site, and aimed at its restoration, including integrated survey, digitization, and HBIM modelling [26], as well as non-invasive structural investigations.

2. MATERIALS AND METHODS

The materials collected and analysed for this study consist of mortar fragments sampled from various points of the bell tower, considering representative types of mortars found (both, presumably, original mortars and modern, mixed or cementitious, mortars).

A total of 7 mortar samples and 13 stone brick samples were collected from the bell tower. All the samples were divided into 5 main groups, depending on their similarities (they are summarized in Table 1), and were analysed with the analytical techniques as follows.

2.1. Stereomicroscopy and microscopy observations

Samples were preliminarily observed by using a Canon EOS 760D camera, for the purpose of defining the morphological characteristics of each fragment.

Subsequently, a small portion (< 15 × 15 mm) of each sample collected was embedded in a bicomponent epoxy resin (Hardrock 554), then cut in cross (polished, SL) and thin sections

(SS), for stratigraphic and petrographic observations, respectively. Optical microscopy observations (hereafter named as OM) were carried out in both reflected light (for SL) and transmitted polarized (for SS) light, by using a Zeiss Axioscope 5 microscope, equipped with an Axiocam 208 colour camera for the image acquisition. By means of microscopy observations of the polished and thin sections, characteristics on the texture, the binder, and the inert fraction of the samples were identified, as well as any alteration phenomena (if present).

2.2. Powder sample preparation

Samples were preliminarily pulverized and sieved (< 2 mm), with the aim of excluding most of the inert materials, and allowing the characterization of the binder in particular. Then, the thinnest fraction was further pulverized and finally quartered, obtaining a representative fraction useful for the following analyses.

2.3. X-ray diffraction analysis

Mineralogical X-Ray diffraction (hereafter named as XRD) analyses were performed with nickel-filtered Cu-Kα radiation (1.540 Å), and data were obtained in reflection mode, with a GNR Europe Theta/Theta diffractometer. Patterns were recorded in the diffraction angle range $5° < 2\,\theta < 60°$ with an acquisition of 0.2 step/s.

2.4. Thermogravimetric analysis

Thermal analysis (hereafter named as TGA) was aimed at investigating the features of the samples, by characterizing binder behaviour under thermal treatment, and revealing some thermal transformations, such as dehydration, dihydroxylation, and decomposition of the material constituents. These features are useful for distinguishing between binders, even by giving quantitative information on compounds analysed. Thermogravimetric tests were carried out on ≈ 10 mg of sample, with a PerkinElmer TGA 4000 instrument, under 20 mL/min flowing air atmosphere, at a scan rate of 10 °C/min, from 30 to 900 °C temperature range. TGA scans are reported in temperature (°C) versus weight loss (wt %).

2.5. Uniaxial compression strength tests

Uniaxial compression strength (hereafter named as UCS) tests were done on small, cylindrical (45 × 90 mm) stone samples, collected by sampling the main stone employed for the realization of the tower masonry (i.e., biocalcarenite stone and yellow tuff), on the basis of the chemical and physical properties identified by laboratory tests. Compression tests were executed according to UNI EN 196-1:2016, by using a Controls 65-L38Z10 compression test machine, with a maximum capacity of 300 kN, and with a loading rate of 0.6 MPa/s.

3. RESULTS AND DISCUSSION

3.1. Mortar sample analyses

The samples' macroscopic photographs, together with their relative stereomicroscopy images, are shown in Figure 2, while the optical polarized-light microscopy (SL) images of the samples are presented in Figure 3. Sample C07 (Figure 2 and Figure 3, C07) is an ochre-coloured, lime-based bedding mortar fragment with friable cohesion. The inert fraction is mainly composed of volcanic aggregates and minerals immersed in the carbonate-based binder.

Volcanic fragments consist of grey or reddish matrix in which sialic and mafic minerals are immersed. Mafic minerals appear to be the most abundant, presumably pyroxenes, amphiboles, micas (i.e., biotite, phlogopite), melilites, and accessory oxides; while, among the sialic phases, feldspars, feldspathoids (i.e., leucite, analcime), and sodalite mineral (i.e., haüyna [27]) are observable [28]. A high amount of coarse lime lumps is also visible [29]. The grain size of the inert fraction is arenaceous-like (up to 2 mm). The inert has low sphericity, a sub-round shape, and poorly selected grading. There is no preferential distribution of the inert

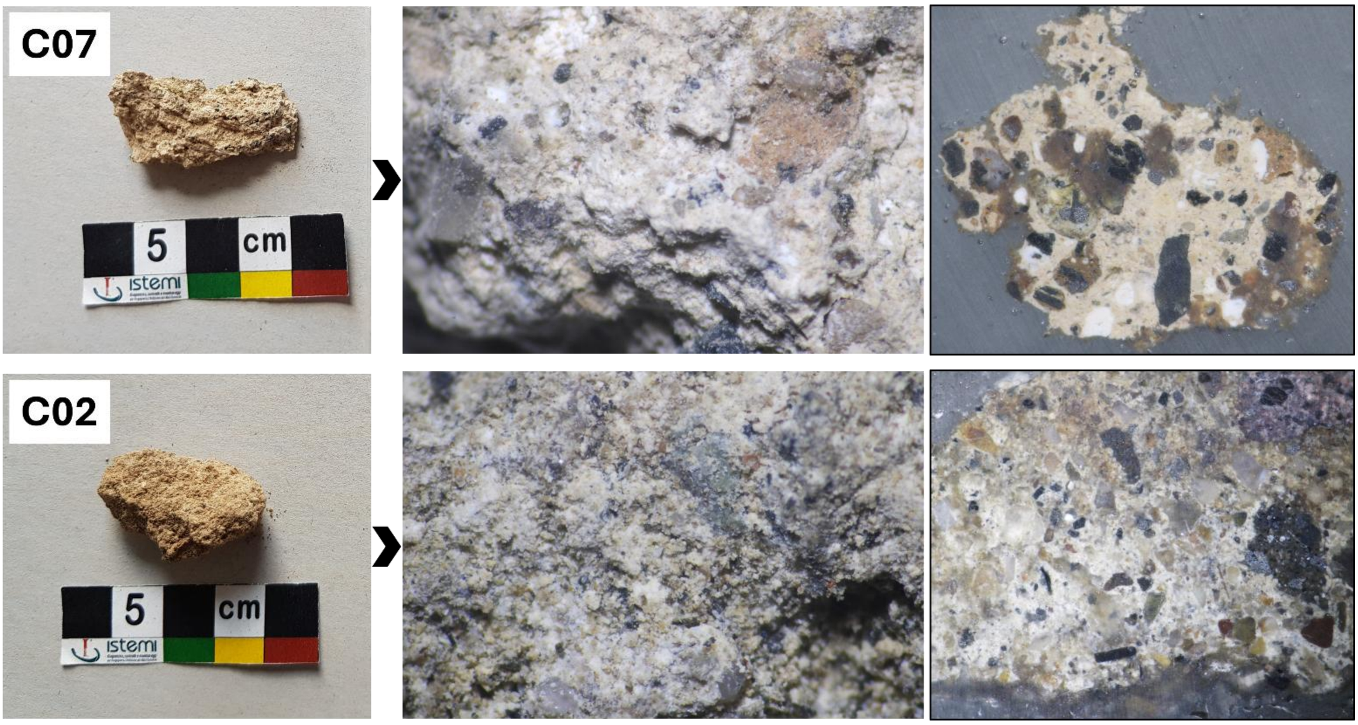

Figure 2. Macroscopic, stereomicroscopic, and cross-section photographs of mortar samples C07 (upper line) and C02 (bottom line), revealing the morphology and the texture of the investigated samples.

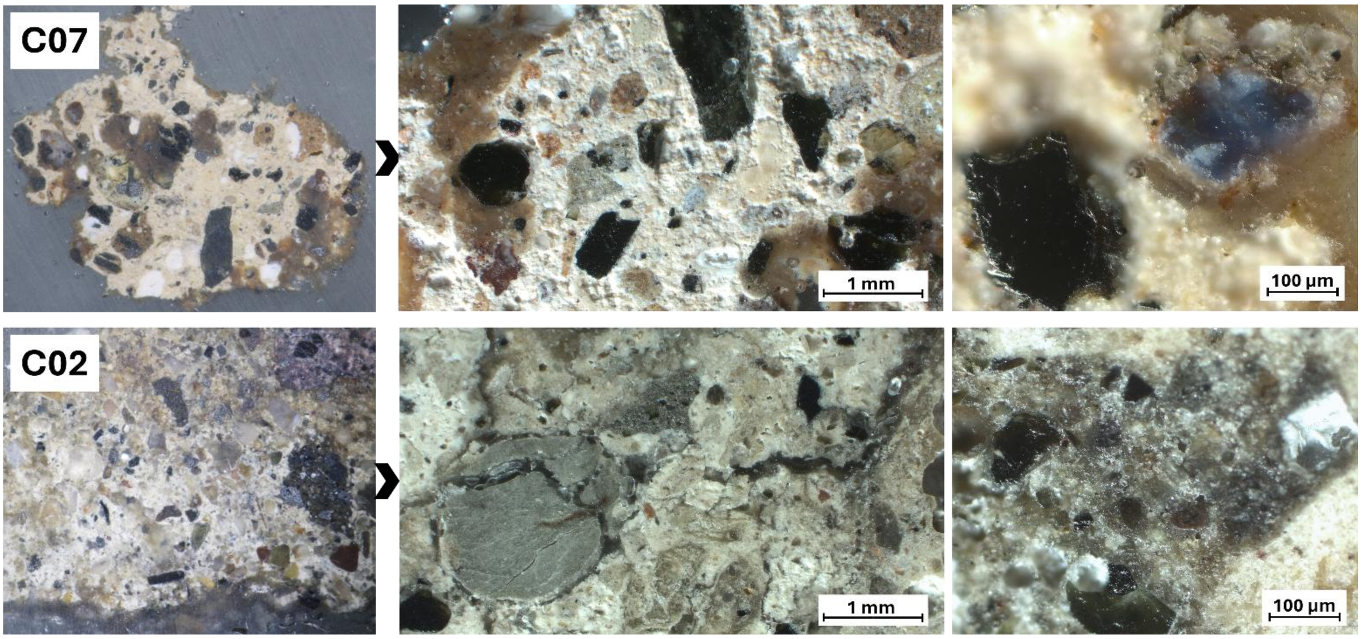

Figure 3. Cross-section photographs of mortar samples C07 (upper line) and C02 (bottom line), and optical microscopy images in reflected light mode. The images were collected in different zoom (from 25x to 100x).

fraction, which is arranged homogeneously and randomly within the matrix. The estimated binder/inert ratio is 1/3.

On the contrary, sample C02 (Figure 2 and Figure 3, C02) is a greyish-white carbonate-based bedding mortar fragment with medium-friable cohesion. The inert fraction is mainly composed of volcanic aggregates and minerals, as well as carbonate grains, immersed within the carbonate-based binder.

The volcanic fragments consist of a grey-blackish or amber-coloured matrix, in which mafic (pyroxenes, micas, i.e., biotite), and sialic (feldspars, feldspathoids, i.e., leucite, analcime) minerals are immersed. The grain size of the inert fraction is arenaceous-like (up to 1 mm). The inert has high sphericity, a sub-rounded shape, and medium-selected grading. There is no preferential distribution of the inert fraction, which is arranged homogeneously and randomly within the matrix. The estimated binder/inert ratio is 1/3.

XRD patterns and TGA scans for samples C07 and C02 are shown in Figure 4, aiming at the definition of the nature of the mortar binder and the inert fraction. For reference, a cake-graph is shown alongside the spectra, to easily understand the semi-quantitative estimation of the mineralogical phases.

Sample C07 (Figure 4, C07) contains a high amount of calcite (up to 94 %), while the rest of the content (close to 6 %) is composed of an inert fraction (mainly clinopyroxene, analcime, and quartz traces).

TGA analysis shows weight loss ($T < 100$ °C) due to the water content naturally present in the matrix (less than 3 wt%). Then, a weight loss occurs at a temperature of ≈ 130 °C, related to a small amount of dihydrate calcium sulphate (gypsum), with a content which is close to 5 wt%, which may be a product of the sulphation process [30]. At a temperature of 238 °C, a small weight loss could be observed, which might be related to the loss of the coordination water from reactive aggregates. In fact, in the case of pozzolanic fragments used as an inert fraction, weight loss at temperatures between 200–600 °C is mainly due to the loss of chemically bound water of hydraulic products such as calcium silicate hydrates and calcium aluminate hydrates present in the binder [31]. Finally, a loss close to 20 wt% occurs at a temperature of 736 °C, which is related to the volatilization of CO_2 of calcium carbonate, resuting in a lime content of 42 % in the binder. The remaining fraction is mainly composed of aluminosilicate fractions coming from the pozzolanic inert, which needs a temperature above 1200 °C for degradation.

On the contrary, sample C02 (Figure 4, C02) shows a calcite amount of nearly 43 %, while a content close to 36 % is composed of an inert fraction (from volcanic fragments, mainly analcime, biotite, and clinopyroxene). A high amount of gypsum was also detected (≈ 19 %), together with a small amount of portlandite (≈ 2 %).

TGA analysis shows a small weight loss in a low temperature range ($T < 100$ °C) due to the water content naturally present in the matrix (less than 4 wt%). Then, a weight loss occurs at a temperature of ≈ 135 °C, related to the gypsum content (nearly 12 wt%), which is usually added to cement as a retarding agent [32]. Moreover, a weight loss occurs at a temperature of 422 °C, typically related to the de-hydroxylation of portlandite ($Ca(OH)_2$) in cement mortars [33]. The presence of such a high content of gypsum and portlandite indicates the cementitious-like nature of the binder (specifically, hydraulic-lime binder) and confirms the mixed nature of the mortar (i.e., "bastard mortar"). Finally, a significant weight loss at 713 °C is related to the volatilization of CO_2 from calcium carbonate, leading to a lime content close to 14 % in the binder.

3.2. Stone brick sample analyses

The collection of the stone samples, together with their relative macroscopic photographs and optical polarized-light microscopy (SS) petrographic analysis, are shown in Figure 5.

Sample P12 (Figure 5, P12) is a carbonate sedimentary stone with tenacious cohesion. As seen from the petrographic examination, the stone has a carbonate-limestone composition, classifiable as lito-bioclastic calcirudite—i.e., Folk's (1959, 1962) non-assorted bio-sparite; Dunham's (1962) grainstone-rudstone—composed of re-sedimented grains of various origins, predominantly extraclasts and benthic foraminifera (Nummulites and Orbitoididae, which characterize Oligo-Miocene geological formations outcropping in the Apennine areas of the provinces

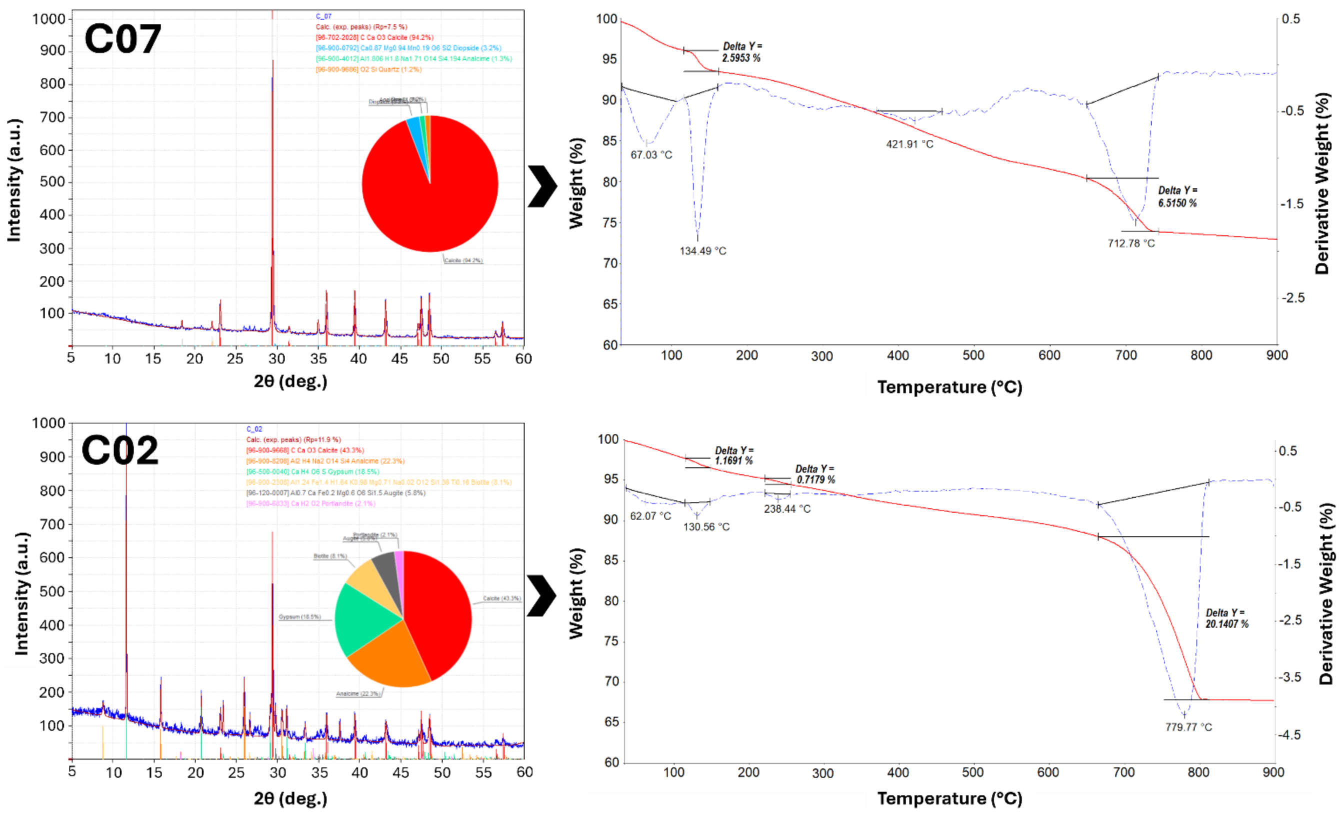

Figure 4. XRD patterns and TGA scans performed on lime mortar sample C07 (upper line) and cementitious mortar C02 (bottom line).

of Avellino and Potenza). These characteristics allow the rock to be classified as one of the many fossil-rich calcareous breccias, intercalated with other types of rock, such as argillites and marlstones, relating to the Frigento Unit, which is traditionally designated as *Flysch Rosso* [34], a lito-bioclastic calcirudite from the Oligocene–Miocene period.

UCS tests performed on sample P12 and similar samples (see Table 1) have allowed an average value of 39.4 MPa to be obtained (according to what was reported by De Gennaro et al., 2013 [34]).

Sample P6 (Figure 5, P6) is a pyroclastic igneous stone fragment with medium-friable cohesion. From the petrographic examination of the thin section, it was possible to recognize the typical *Neapolitan Yellow Tuff* matrix, showing a texture characterized by fine (millimetre-sized) particle aggregates of ash-coated pumice and lapilli. Millimetre-size obsidian fragments and phenocrysts of alkali-feldspar (i.e., sanidine), plagioclase, biotite, and rare black phenocrysts (likely clinopyroxenes, i.e., augite) are embedded in the ash matrix. A diffuse zeolitization process—leading to the crystallization of phillipsite and, subordinately, chabazite and analcime—is easily recognizable [35], [36].

UCS tests performed for sample P6 and similar samples (see Table 1) have allowed an average value of 12.4 MPa to be obtained (according to Colella et al., 2017 [36]), relating such low strength values to the tuff texture (pumice, matrix, porosity, etc.) and even considering the weathering to which they are subjected.

3.3. Mechanical characterization of the masonry

The indirect evaluation of the compressive strength of the masonry was obtained starting from the procedure reported in NTC 2018 [1] (*Estimation of the compressive resistance*, §11.10.3.1.2, Tab. 11.10.VI – f_k values for masonry in solid and semi-solid artificial elements [values in N/mm²]). In accordance with the values reported in the section *Resistance classes of mortars* (Tab. 11.10.V – *Correspondence between resistance classes and volume composition of mortars*) and considering the overall results, it was possible to classify the mortars and the masonry of the bell tower of Melfi Cathedral (results are shown in Table 2).

Following the results obtained from the lime mortar samples (i.e., C07), for which the binder/inert ratio (lime/pozzolanic fragment) is 1/3, the estimation of the mortar class is M2.5. On the contrary, for the cementitious mortar samples (i.e., C02), for which the binder/inert ratio (cement or hydraulic-lime binder/pozzolanic fragment) is 1/3, the estimation of the mortar class is, at least, in the range of M2.5–M8.

From the overall results, and by considering UCS tests carried out on the natural stone bricks (samples P12 and P6), the compressive strength of the masonry with lime mortar samples (i.e., C07) can be assumed to be a maximum of 8.2 MPa (for masonry with lito-bioclastic calcirudite stones) and 4.6 MPa (for masonry with yellow tuff stones).

For the cementitious mortar samples (i.e., C02), for which the class is from M2.5 to M8, the compressive strength of the masonry can be assumed to be a maximum of 10.4 MPa (for masonry with lito-bioclastic calcirudite stones) and ≈ 4.5 to 6.0 MPa (for masonry with yellow tuff stones).

Table 2. Resistance classes of the mortars (RCM) as determined with chemical-physical laboratory analyses, and mechanical characterization of the masonry (f_k), according to NTC 2018 [1].

Sample	RCM	Description	Analyses
C07	M2.5	**8.2**	**4.6**
C02	M2.5 – M5	**- 10.4**	**≈ 4.5 – 6.0**

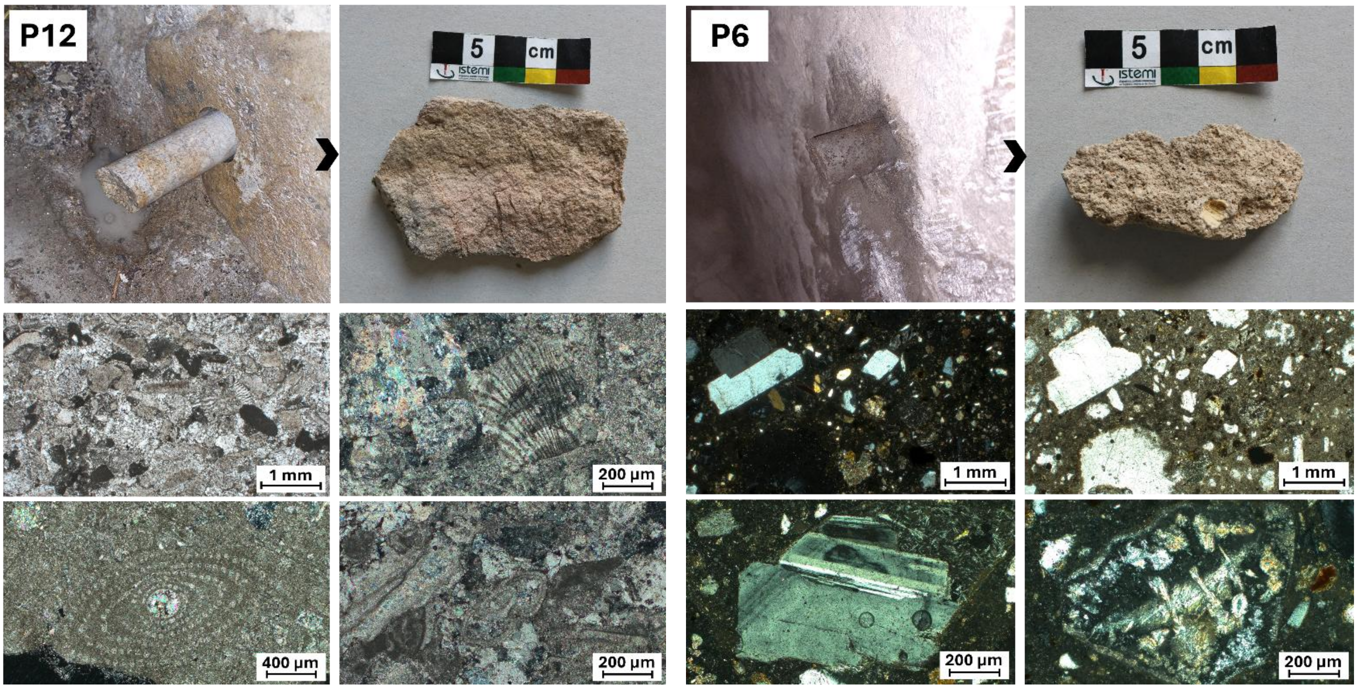

Figure 5. Sampling operation of P12 (left image) and P6 (right image) stone bricks, together with their macroscopic images, and optical microscopy polarized-light images. Images were collected in different zoom (from 25x to 200x).

Above all, it is worth noting that, since the tables provided by the NTC 2018 [1] are valid for newly built structures, the above assessments are indicative.

4. CONCLUSIONS

In this contribution, a valid and micro-invasive alternative methodology, appropriate for cultural heritage structures, was reported. The case study investigated is the medieval bell tower of the Cathedral of Melfi (Basilicata, Italy), for which non-destructive or, at least, micro-invasive operations were executed, respecting the principle of non-invasive investigations. Laboratory tests, by means of chemical and physical techniques, were carried out on samples collected on-site, aimed at obtaining the resistance classes of the mortars, useful for determining an estimation of the mechanical characterization of the masonry (according to the Guidelines of NTC 2018).

In detail, the multidisciplinary approach adopted, by means of microscopic, petrographic, mineralogical, and thermal analyses, as well as mechanical compressive strength tests performed on small samples collected, has allowed important information on the materials used for the construction of the bell tower to be obtained. Firstly, the possibility to determine the mechanical characteristics of the masonry by using micro-invasive methods, instead of complete destructive tests, highlights the sustainability of the approach, focusing on the micro-invasiveness of the operations.

Moreover, it demonstrates the importance of consciousness regarding the construction materials employed in historic cultural heritage, which is continuously subjected to degradation processes and is important to be preserved, with the main aim of contributing to supporting collective memory and identity, and, at the same time, ensuring valorisation and fruition of heritage as a source of economy and development of the entire community.

AUTHORS' CONTRIBUTION

Conceptualization: C.G. and E.C.; methodology, investigation: C.G., G.M.C.G., S.C.; validation: C.G.; resources: M.S., Y.C., N.M., C.N.; original draft preparation: C.G.; writing, review and editing: C.G., G.M.C.G., S.C.; supervision: C.G., E.C.; project administration: C.G. and Y.C.

All authors have read and agreed to the published version of the manuscript.

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