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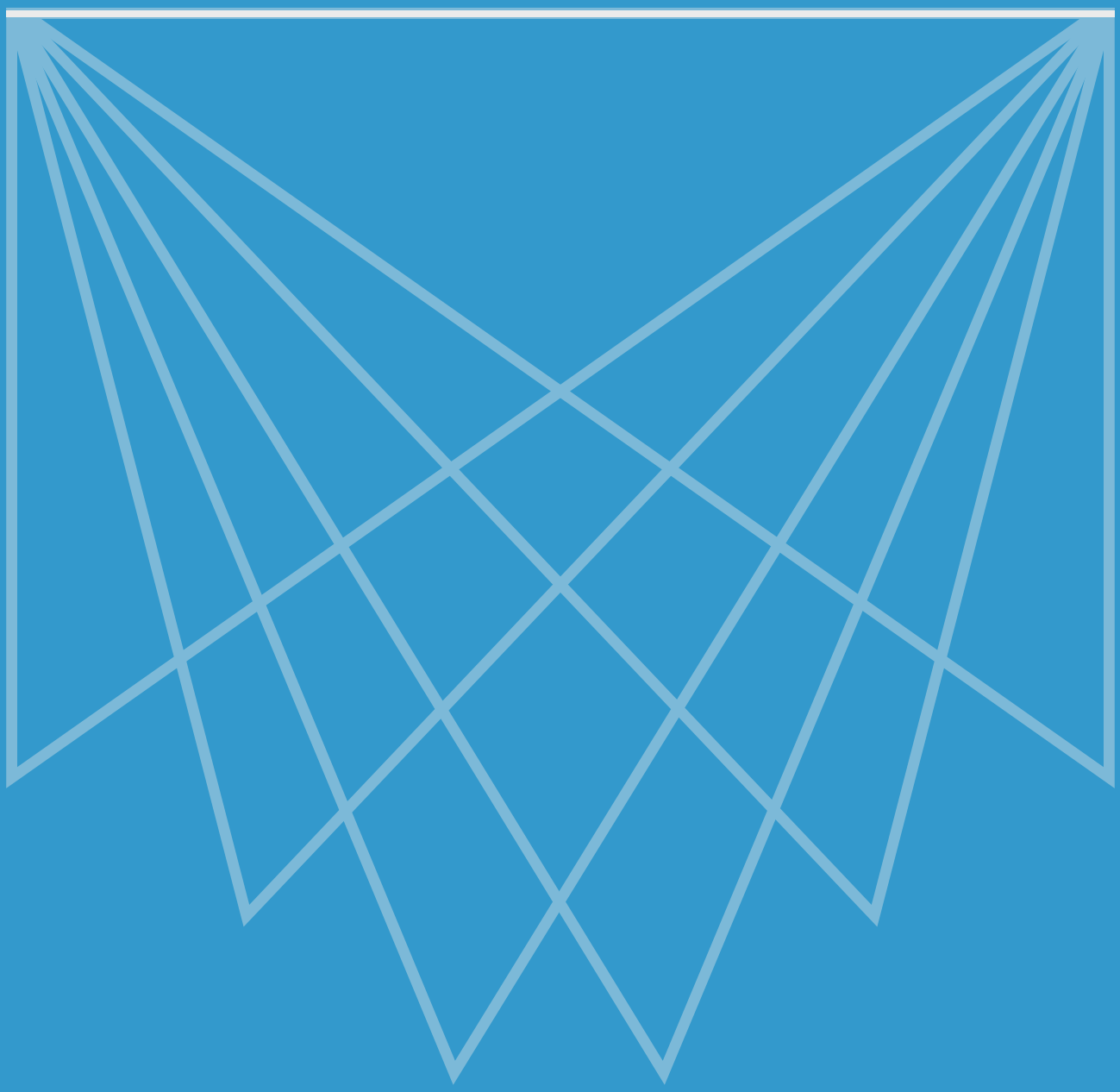

DATAZIONE ASSOLUTA E RELATIVA

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Fission Track Dating of obsidian samples from Lipari Neolithic settlements

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The present work is part of the project "Lipari obsidian and Neolithic human communities in the Aeolian islands" in the frame of a collaboration between the department of Physics of the University of Milan and Parco Archeologico delle Isole Eolie, Museo Luigi Bernabò Brea. It aims at studying the connection between obsidian flows on the island of Lipari and Neolithic populations on the Aeolian archipelago, in Italy. Obsidian is a natural volcanic glass used to produce cipped tools; in the Neolithic period it was the sharpest known material and its trade played an important role in the Mediterranean area. It is thus of particular interest for tracing prehistoric trading patterns. Indeed, Lipari obsidian has a wide distribution and has been found even in southern France, Dalmazia, Sicily and mainland Italy. The project outputs will give a general vision of both archaeological and volcanological aspects through the stratigraphic and radiometric dating of eruptions which produced obsidian, in relationship with the first phases of the human settlements and row material exploitation.
To reach this goal, we considered both raw materials from different obsidian flows (geological samples) and artefacts from Neolithic settlements on the Aeolian islands, and performed fission track dating. Fission-track dating is a radiometric technique that can be used for uranium-bearing minerals and glasses. The spontaneous nuclear fission of 238U creates damage trails, the fission tracks, which density represents a measure of the time over which tracks accumulate. To this end, obsidian samples were divided in two fractions, one of which was irradiated at the TRIGA Mark II Research Nuclear Reactor of the LENA Laboratory (University of Pavia, Italy). Standard glasses, together with mica foils acting as external detectors, were also irradiated for thermal neutron fluence determination. The irradiated and non-irradiated fraction of obsidian samples were then prepared for etching and microscope observation, applying the standard technique consisting of mounting samples in epoxy resin. Etching was performed with HF in monitored conditions and samples were observed with total 500x magnification for spontaneous and induced fission track identification. This procedure allowed to get the density of fossil and neutron induced tracks and therefore the cooling age of obsidian source and artefacts.
Preliminary results allowed the dating of geological samples, which we could relate the different eruption phases. Archaeological samples have also been measured and dated: some of them are clearly linked to one of the studied lava flows, while for others the fading effect has to be evaluated to correct the obtained apparent age.
Final results are expected to shed some new light on the raw material procurement and on the ability of the Neolithic populations to move from their locations, with particular attention to the consequences of environmental features on the first human settlements on the Aeolian islands.

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Optimization of the sample selection procedure of historical mortars for radiocarbon dating

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The use of radiocarbon (14C) for dating ancient mortars was proposed as early as in the 1960s, applying the method to the inorganic binder. In mortars, the radiocarbon datable component is the so-called anthropogenic calcite, which is formed by the reaction of calcium hydroxide with atmospheric CO2 during the setting of the material. However, since mortars are complex and heterogeneous materials, other C sources that can contaminate 14C concentrations may be present in mortar samples.
A multidisciplinary approach is essential to ensure accurate dating of mortars. Complete characterization of historic mortars prior to radiocarbon dating is an essential step in obtaining accurate and meaningful results. An effective sample selection procedure (Fig. 1) has been developed in the laboratories of DST-UNIFI, ISPC-CNR and INFN-LABEC in Florence, ranging from sample collection to Accelerator Mass Spectrometer (AMS) measurements. Samples are selected based on the results of minero-petrographic and chemical characterization. In particular, the origin of the calcite is investigated by non-destructive methods (Calandra et al. 2022; Calandra et al. 2023). Proactive identification of the origin of calcite present in the sample powder allows selective measurement of only anthropogenic calcite, reducing the time and cost of AMS measurements. 14C-AMS measurements are performed either on bulk samples or on individual lumps, with the CO2 extraction procedure adjusted according to the type of samples.
Our procedure has been applied to Florentine mortars, which are characterized by a natural hydraulic binder. This allowed us to select samples for dating that would otherwise have been excluded.

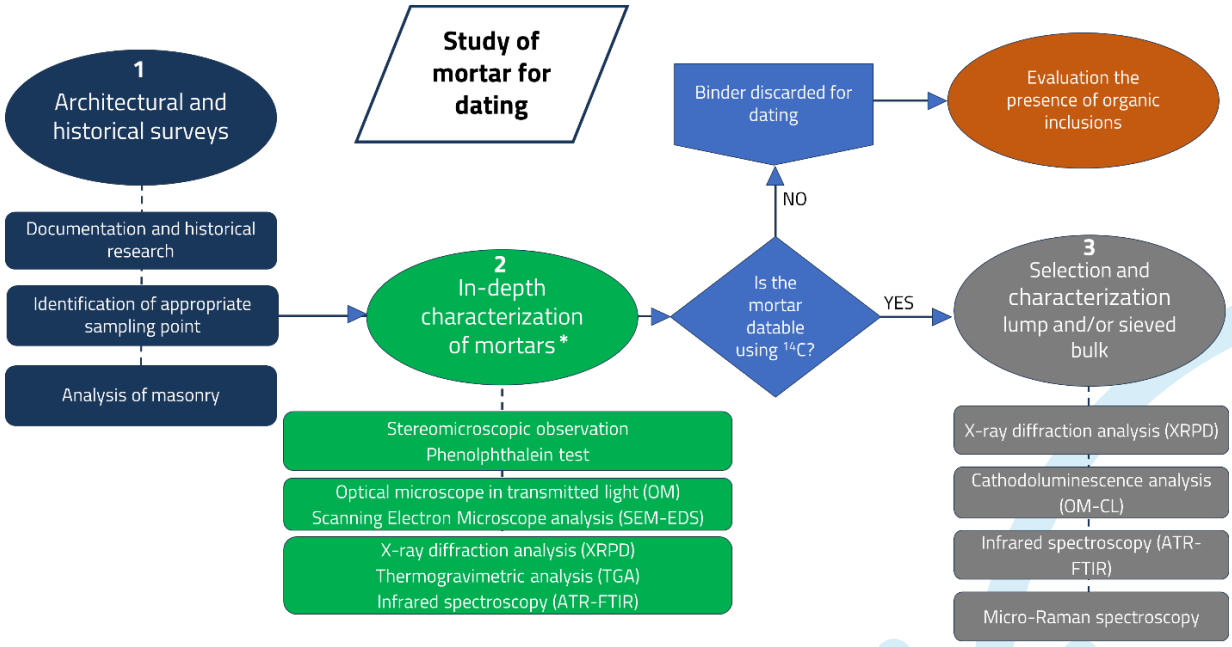

Fig.1. Our analysis procedure of sample for radiocarbon dating of historical mortars.

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Advance in radiocarbon dating of mortar: non-destructive identification of calcite origin by micro-Raman spectroscopy and machine learning

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Accurate 14C dating of anthropogenic calcite from binder or lumps in aerial mortars is closely related to the removal of contaminants (Urbanova et al., 2020; Lindroos et al., 2020; Toffolo et al., 2020). Identifying remnants of geogenic calcite from carbonate aggregates or underburned rock fragments for lime is a major challenge.
The possibility of distinguishing anthropogenic and geogenic calcite with an effective, fast, and efficient method has led us to combine high-resolution micro-Raman spectroscopy with data mining and machine learning methods (Calandra et al., 2023). This approach provides an effective way to obtain robust and representative Raman datasets from which the origin of samples can be effectively inferred.
A wide range of different carbonate rocks (geogenic calcite) and lime binder for mortar (anthropogenic calcite) were selected to create the dataset, from a variety of geological and archaeological/historical contexts.
We collected Raman spectra with a Renishaw InVia Raman spectrometer, which features high resolution and is used in combination with a Leica DMLM microscope. Spectragryph v 1.2.15 software was used to process the wavenumbers, intensities, and areas of the characteristic vibrations of the carbonate groups in calcite (L, librational mode; v4, in-plane bending mode; and v1, symmetric stretching mode). In the preliminary study, the position of the L, v4, and v1 bands and full width at half-maximums (FWHMs) were recorded to investigate the results of the Raman techniques.
Samples of anthropogenic calcite show systematic and reliable differences in Raman peak positions, band shape, and intensity. The observed systematic Raman shifts in the L and v1 bands of the anthropogenic calcites prompt us to apply methods of data analysis and integrated machine learning. The successful parameters (among position, intensity, area subtended of bands and FWHMs of L, v4, v1) for discriminating calcite origin were determined using KIF, PCA, K-means Clustering, and the relationship between the target and predictor variables using Logistic Regression and Random Forest models.
Structural order-disorder, degree of crystallinity (Toffolo et al., 2023), and polarization effect are the main factors affecting the Raman spectral signature of calcite.
The proposed method is effective in discriminating anthropogenic calcite in pyrotechnological materials (i.e., mortar and plaster) to select the most appropriate carbonate fraction for radiocarbon dating in a non-destructive manner.

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Challenges in radiocarbon dating of Pompeian pozzolanic mortars

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In this work, the feasibility of 14C dating of Pompeian pozzolanic mortars has been evaluated. Roman mortars were usually made by mixing an aerial lime with materials with pozzolanic behavior (i.e. pozzolana or cocciopesto), obtaining a strong hydraulic feature of the material. Indeed, many characterizations of pozzolanic mortars have been carried out, revealing their complexity (Jackson et al., 2013; Seymour et al., 2021). The possibility of using radiocarbon to date such mortars is still under debate (Lindroos et al., 2011; Michalska and Mrozek-Wysocka, 2020); published data even suggest that radiocarbon dating might not be feasible. However, the literature lacks a study that relates the characterization results to the reasons why radiocarbon dating may be unreliable.
Public monuments in the Forum of Pompeii were studied (Dobbins, 1994). It is worth noticing that the preservation context of these mortars is extremely complicated (Morra et al., 2010). Samples from the Eumachia Building, the Temple of Genius Augusti, the Tabularium and the Temple of Apollo were selected to test radiocarbon, after a comprehensive mineralogical, petrographic and geochemical characterization. Powders of binder or lumps were analyzed using non-destructive techniques, such as XRPD, OM-CL, ATR-FTIR, micro-Raman, SEM-EDS.
CO2 from the selected inorganic fractions of the mortar samples was extracted through dissolution by orthophosphoric acid and then converted to graphite for the following Accelerator Mass Spectrometry (AMS) measurements using the so-called Lilliput graphitization line at INFN-LABEC in Florence.
As the characterization procedure itself suggested, AMS measurements resulted in samples apparently older than expected, except for two of them whose age is compatible with the archaeological phase. These data pave the way to understanding the nature of the material and its influence on the dating results.

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Twenty years of 14C-AMS dating at INFN-LABEC, Florence

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LABEC (Laboratorio di tecniche nucleari per l'Ambiente e i BEni Culturali) has been in operation since 2004, when a new tandem accelerator was installed in the Florence unit of INFN, taking advantage of the well-established expertise in applied nuclear physics. The mission of the laboratory has always been indeed the development of technologies and tools to be applied on archaeometry, on environment and material science in general. At present, LABEC, as far as Cultural Heritage studies are concerned, is the reference structure of INFN-CHNet.
Radiocarbon measurements by Accelerator Mass Spectrometry (AMS) has been one of the key activities of LABEC since its beginning. Here, we will give a review of the developments in 14C-AMS measurements in our laboratory, showing improvements both on hardware and on sample preparation procedures. Radiocarbon applications will also be discussed, highlighting the importance of this rare isotope for the authentication of artworks, as well as for "more traditional" studies in archaeology and history of art.

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Exploring Luminescence Dating of carbonate fractions in historic mortars

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In past years, the indirect dating of historical buildings has shifted from the dating of bricks by Thermoluminescence (TL) to the dating of mortars by Optically Stimulated Luminescence (OSL) to overcome issues related to brick reuse and the potential temporal gap between manufacture and placement.
Despite the promising outcomes observed in mortar dating using OSL, the challenge of partial signal bleaching during the zeroing event remains an open issue. Multiple authors have addressed into this problem. Some of them have noted that larger grain sizes enhance the likelihood of achieving complete signal bleaching (Goedicke, 2003), while others have obtained favorable results employing finer grain sizes (Gueli et al., 2010; Stella et al., 2013, 2018). Discrepancies in these findings may be attributed to a different geographic origin of the quartz studied by the authors and thus attributed to variations in the sedimentary history and sand aggregate processing employed in mortar preparation (Urbanová et al., 2020). The adoption of the Single Grain technique (SG-OSL) represents a potential solution, although, at the same time, the limit of this method to achieve accurate and precise results were already pointed out (Panzeri et al., 2019).
An alternative approach involves dating the carbonate fraction, particularly in the case of hydraulic mortars. For the carbonate fraction, the zeroing event is ensured through the firing process, where calcite used in mortar fabrication is subjected to temperatures exceeding 850°C, resulting in the formation of crystalline calcium carbonate, $CaCO_3$, during the carbonation process of the lime putty $Ca(OH)_2$.
The main objective of the present study is to assess the feasibility of luminescence dating employing the carbonate fraction of mortars. The examination encompasses the TL signals of calcite, quartz, and a mixed phase by varying the range of useful emission to obtain (a) the temperature range of the relevant signal, (b) discrimination between the total quartz and calcite contributions to the signal, and (c) key parameters for dating procedures, including the maximum reading temperature, optimal heating rate, and pre-heating temperature. To filter luminescent signals across distinct wavelength ranges, three filters (Hoya U340, BG-39, and Schott OG590) were employed, with their transmittance assessed via the Perkin-Elmer Lambda 1050 UV-Vis-NIR spectrophotometer. During measurements, BG-39 and OG590 filters were combined with a Schott BG-12 filter to cut infrared emission. Luminescence signals were acquired using the semi-automatic Risø TL-DA-15 reader equipped with an EMI 923QA photomultiplier. Artificial irradiation was administered through a 90Sr-90Y beta source, delivering a dose rate of 4.14 Gy/min.
Results were derived through signal analysis of the studied samples. Experimental parameters in terms of heating rate, preheating and maximum temperature were identified. The signal of interest detected from calcite has a peak emission around 350 °C when using detection with BG39 filter. This signal is not detected with the Hoya U340 and OG590 filters, so it is a luminescence emission with wavelength in the 400-550 nm band. However, under the same set up conditions a weak luminescence of quartz is also detected. Measurements on the mixed phase confirm the overlap of the two components but with dominance of the signal from carbonate. The preliminary results point to a potential use of carbonate for mortar dating. Research related to the discrimination of the two components in a mixed phase is still in progress. With the obtained results, an algorithm could be developed to separate carbonate emissions by deconvoluting the signals from the other luminescent crystals.

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Preparation and characterization of archaeological mortars for radiocarbon dating: the case study of decorated plasters from Pollena Trocchia (Campania region)

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The determination of the age of the ancient structures is one of the most important issues in the study of historical buildings and their evolution. One of the most effective building materials that provide important dating clues is represented by the mortars, mixtures of binders and aggregate of different nature. In particular, lime-based mortars are largely used for dating procedures since carbon dioxide absorbed during the setting of the mortar on the archaeological structures reflects the content of 14C existing in the atmosphere at that time, although some factors may influence the reliability and accuracy of radiocarbon measurements (i.e., presence of unburned limestones, carbonate-bearing sand as aggregate, and/or secondary calcite forming during the burial for the interaction with current water).
Taking into account the complexity of the problem, during the last decades lime mortars were prepared for radiocarbon dating using several chemical and mechanical procedures that affected both the fine binder fraction and pure lime lumps. However, the procedures may not eliminate completely the contamination agents (carbonate in aggregate, calcination relics or secondary calcite) from the carbon belonging to the original binder, leading to an incorrect dating of samples, despite the efforts in the improvement of separation procedures.
To reduce as low as possible the effects of contamination agents on dating procedures, the selection of the material must be accurate, namely by sampling mortars in which unburned limestones, carbonate-bearing aggregate and secondary calcite should be ideally absent, to give, consequently, a preliminary feasibility of radiocarbon dating of mortar. Thus, an appropriate characterization of mortar appears fundamental to obtain such a preliminary feasibility of radiocarbon dating of mortar.
The present research deals with the preparation, characterization and dating of decorated plasters collected in the archaeological site Masseria de Carolis in Pollena Trocchia (Campania region) representing the “ideal” materials for reliable dating procedure, because: a) the remains have strict chrono-stratigraphic constrains, marked by the deposits of 79 CE and 472 CE Vesuvius’ eruptions that sealed the site; b) they show a multi-layer technology and at least one layer is constituted by lime-based mortars containing volcanic aggregate.
To collect clues about the presence of possible dating contamination sources, in fact, observations via Polarized Light Microscopy (PLM) on thin sections permitted the identification of mineralogical phases constituting the binder and aggregate of each layer, to correctly discriminate the different carbon dioxide sources. PLM observations revealed that which had volcanic nature in rinzaffo (when present) and arriccio layers, whereas calcite-bearing aggregate was used for intonachino
For this reason, the arriccio layers of 3 samples were mechanically separated and prepared by using the Cryo2SoniC preparation protocol (Lubritto et al. 2018). Samples obtained by sieving the fine fraction at different, decreasing meshes were then characterized by means of Fourier Transform Infrared spectroscopy and simultaneous thermal analyses (termogravimetry and differential scanning calorimetry -TG-DSC-), to monitor the efficacy of method in the removal of possible sources of geogenic calcite, and to define the more effective preparation strategy for the selection of samples do be dated.
Radiocarbon dating have been performed by Accelerator Mass Spectrometry technique (AMS), to identify the main characterization parameters that can signal the presence of geogenic carbon and therefore give an indication of the reliability of the measurement. Results obtained at different step of the whole procedure will be presented.

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Late Antiquity Florence: a case study on necropolis outside the ancient roman walls

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Some years ago, in the centre of Florence, an amazing finding was recovered in the basement area underneath the Biblioteca Magliabechiana, Uffizi area: a necropolis with many multiple tombs was found. About seventy individuals were identified and excavated. Since the very beginning, according to the characteristics of the tombs and to how the individuals had been laid to rest, archaeologists hypothesized that the burials were the result of a sudden and catastrophic event. In addition, the area itself where the necropolis was found represents a sort of enigma: it was outside the walls of the original Roman town, in a zone often subjected to floods and also used to deposit wastes.
Such an important finding started a mutidisciplinary research, coordinated by Soprintendenza dei Beni Archeologici della Toscana, spanning from anthropology and archaeology to paleopathology. In this context, an absolute dating method such as 14C-AMS can definitely give us important information. We dated nine individuals from the cemetery, also checking whether the possible ancient floods had introduced contaminations. Radiocarbon data were calibrated using a bayesian approach, also considering chronological constraints given by archaeological evidences. Final results point to a period corresponding to the second half of the III century CE.

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CASTLES Project: a multidisciplinary study on the chronological transformation of the Medieval Castles in Italy

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In the present paper we present the PRIN 2020 CASTLES Project, dedicated to the study of castles in medieval Italy. It focuses primarily on the chronology of their formation and of their transformations, a key theme within broader European historiographical research. The archaeometric analysis concerned specifically the mortars used in the building of these sites and charcoal founded in this samples.
The study of medieval castles is a key theme within European historical research, one that has been tackled extensively, but which still retains numerous open questions. In Italy, recent work on the subject has greatly contributed to a sharpening of these questions, in particular those pertaining to the chronology of the transformations of castles. Between the second half of the 11th century and the end of the 12th, in fact, castles underwent a significant material shift, transforming from largely wooden palisades to durable stone fortifications featuring aristocratic residences and enclosed castral settlements; a shift connected with a transformation of practices of power in the countryside, but also with a reconfiguration of rural social fabric. The precise timings and the rhythms of these transformations, however, remain difficult to discern within the broader period in question. The aim of this project is thus sharpening these chronologies, through the development and the application of a novel research strategy, not previously applied to an Italian context in a systematic manner until now: this is a crucial chronological datum because it can change the way we understand the dynamics of transformation of social power linked to the rise of seigneurie in Italian countryside.
The uniqueness and originality of this project rests on four key aspects: its multidisciplinary approach; the high number of mortar samples that will be analysed, selected from a vast geographical area; the linking of these analyses to historiographical themes of crucial significance; and the creation of a dating protocol which can be exported and applied to other contexts, thanks to the elaboration of models of advanced statistical analysis to process the multidisciplinary data acquired.
The focus of the multidisciplinary research will be on Tuscany, as one of the best-studied regions of Italy. The acquired data and the new investigative protocol will be then compared and tested against a sample of selected castles from Piedmont and Liguria.
The apparent simplicity of the project's goals disguises a significant degree of complexity. There are a series of methodological challenges that need to be faced and conditions that need to be met to ensure the feasibility and the success of the project. On one hand, the samples need to be selected from areas that have previously been subject of ample and high-quality research and which can in turn provide a solid foundation of knowledge to build on, in both historical and archaeo-architectural terms. A preliminary mortar characterization provides crucial information about the nature of the materials, including the type of binder, the presence of lumps and charcoal, the nature of the aggregate, and any additives. This information is mandatory for the selection of a datable fraction.
On the other hand, the archaeometric analyses must be based on a significant level of experience in this field of research, as they will require the testing of new analytic protocols (radiocarbon mortar dating and mortar characterization) to counter the problem of the calibration curve for 14C dating (which, at present, does not allow for narrow calendar age ranges for the twelfth century).

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The complex link between the successful dendrochronological dating of a violin and the properties of its front plate

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Supplementing the study of musical stringed instruments with dendrochronological analysis has become a common practice, as it plays a crucial role in the dating process and provides valuable information useful in defining the attribution of the instrument to its maker (Grissino-Mayer et al. 2004; Čufar et al. 2017). Indeed, dendrochronological analysis makes it possible to identify the year of the most recent tree ring present in the violin front plate, corresponding to the last year of wood production by tree used to make the violin. However, it is impossible to determine the year in which the tree was felled, how long the wood was seasoned before being used, and how long it took the luthier to finish the instrument. Therefore, dendrochronological dating provides a terminus post-quem, i.e. the instrument cannot be attributed to a date preceding that of the most recent ring identified in the front plate.
The chance of successful dendrochronological dating depends on several elements, including: characteristics and conservation status of the front plate, correct identification of a sufficient number of tree rings (usually 60), chronologies with well-defined signatures, availability of reference chronologies suitable for the geographic area where the tree grew (even if its provenance is usually unknown).
In the present work, we analyzed by dendrochronological approach two violins of the collection “Istituto Santa Maria della Pietà”, in Venice, with very different bellies characteristics.
The first one (VEN 3) had a worn front plate, with some cracks, irregular grain inclined with respect to the central joint and the number of rings increasing from the bottom to top. Finally, a thick layer of varnish covered the surface, making the identification of tree rings more difficult, such that it was doubtful that dating was achievable. On the contrary, the second violin (VEN 8) has a well-preserved front plate, with regular tree rings along the table on both side, and it seemed, a priori, to present the ideal conditions for successful dating.
Dendrochronological analysis was performed on high-resolution images of the bellies, by measuring the annual tree ring widths in different position. As each front plate was composed by two different pieces of wood, finally we obtained a chronology for each piece (bass and treble chronologies) in each violin. Cross-dating was initially performed comparing chronologies within the same violin. Then the series were compared with several reference chronologies, partly from the International Tree Ring Data Bank and partly built in the LabDendro of University of Pavia.
Contrary to expectations, Violin VEN3, allowed us to build good quality long chronologies (> 100 years), well synchronized each other, suggesting that the two parts that make up the front plate belong to the same tree. The violin chronology showed good correlation with several reference chronologies of both spruce and larch, with particularly high values in comparison with two violins of Mittenwald Neuner School, suggesting 1760 as the date of the most recent measured ring. As the central joint was covered by the handle in the highest part of the table and the number of rings increases from the bottom to the top, 5 additional years was added, fixing the terminus post-quem in 1765.
In contrast, despite the well conserved wood and the easy identification of rings, measures on Violin VEN8 provided bass and tremble chronologies without any synchronization each other, suggesting a different origin of the wood. Moreover, good correlation with reference chronologies was found only for the bass at 1761, while no dating was obtained for the treble side. That why it was not possible to establish a terminus post-quem for this instrument. The causes of this unsuccessful dating may be due to the small number of rings identified on the front plate (55 for bass and 56 on treble), or to the lack of adequate reference chronologies, or both.
These study cases emphasize that tree growth conditions and tree-ring features may be crucial in violin dating process, even more than the conservation status and characteristics of front plate.

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Chronological insights: unraveling the History of Tumshukayko, a Monumental Andean Site, by Optically Stimulated Luminescence

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Tumshukayko is an ancient, monumental site located in the north-central Andes of Peru, specifically in Caraz, Ancash. Its historical significance dates back to approximately 3500 B.C., and remarkably, it has seen continuous occupation up to the present day. The site is composed of a principal mound called Tumshukayko A and several adjacent mounds.
The exploration of Tumshukayko began in 2021 with a topographic survey that included the collection of a terrain sample for Optically Stimulated Luminescence (OSL) dating analysis [1]. This initial phase was focused on an open gallery situated in the southern sector of the principal mound. In 2022 and 2023 a more extensive investigation took place. Researchers excavated seven test pits with the purpose of uncovering the site's morphology, establishing its chronological development, and deciphering its construction sequence. Most of these tests were conducted on the principal mound, but one test pit was strategically placed in an ancillary mound known as Tumshukayko B, situated south of Tumshukayko A. In this case, both terrains and rocks were collected and analyzed. In 2023, a further four pits were excavated at Tumshukayko A with the aim of determining the continuity of the retaining walls of the upper platforms of the mound and thus its shape. On this occasion, ten duplicate samples (soil mortar and building stones) were taken from as many walls and one soil-only sample from an alluvial deposit. This sampling, together with the excavation data, should contribute to the final objective of establishing an accurate and absolute chronology for the monumental site. This involves determining the age of the buried sediments and rocks (in the latter case using surface dating technique [2,3,4,5]). For sediment dating, the team utilized the multigrain sand-sized quartz dating method, while fine-grain techniques were employed for rock surface dating. The Equivalent Dose was determined using the Single Aliquot Regenerative (SAR) protocol [6]. To assess the rock surface dating, luminescence profiles were studied from the surface of the rocks down to a depth of about 1 centimeter to ascertain whether a single or multiple burial events had occurred.
The resulting ages obtained from the terrains and rock surfaces were rigorously compared and statistically analyzed. This analysis aims to reconstruct the various building phases of the monumental site, offering valuable insights into its historical construction and development.

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Reconstructing the past geomagnetic field's variation in Asia as a tool for archeomagnetic dating

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The large number of archeomagnetic studies, which were conducted since the early 60s on archeological artifacts from Asia, can provide valuable insights into the past behavior of Earth's magnetic field within this continent. Taking advantage of this comprehensive and well-distributed dataset, this study presents a first regional reconstruction of the ancient geomagnetic field variation in Asia, covering the past two millennia.
This paleoreconstruction incorporates both data from well-dated archeological baked clay structures and artifacts, as well as data from volcanic eruptions of known date, included within the target geographical area and chronological timeframe. Prior to modeling, a meticulous data selection process has been applied to establish a robust weighting scheme, in order to enhance the quality of the data and thus the accuracy of the modeling approach. Furthermore, historical data from the HISTMAG compilation have been integrated to further refine our comprehension of the geomagnetic field's behavior over the last few centuries.
The regional Asian paleoreconstruction employs R-SCHA2D functions in spatial dimensions and cubic splines in the temporal domain. The study region is defined within a 50° spherical cap centered at the coordinates 35.0ºN, 102.5ºE. This model not only advances our capacity to better characterize the past behavior of the Earth's magnetic field in Asia but also enables the generation of new paleosecular variation curves that can be used as reference curves for archeomagnetic dating. Using these curves, dating precision has been assessed for the past two millennia within the Asian continent.

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Rediscovering A Long-Forgotten Fresco by Giotto

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Numerous accounts from travelers and artists, including Lorenzo Ghiberti and Giorgio Vasari, documented the existence of a vast fresco attributed to Giotto and his school in the Sala del Capitolo of the Sant'Antonio complex in Padova, Italy, during the 14th and 16th centuries. Records indicate that the fresco was last sighted in 1537 before being concealed during extensive renovations of the room [1], [2]. Although small sections of the original fresco were accidentally revealed during conservation efforts in the mid-1800s, surprisingly, no comprehensive modern investigation has been undertaken to confirm the presence of the entire artwork [3]. Based on the results of a two-year project conducted as part of the IPERION CH initiative, which employed cutting-edge non-invasive diagnostic techniques, we propose that the majority of Giotto's original creation remains concealed beneath the 16th Century plastering of the wall. This hidden masterpiece eagerly awaits proper restoration and recognition.
The study employed a wide array of techniques combined with state-of-the-art portable probes available today for non-invasive investigation of cultural heritage structures and objects. Specifically, the selected techniques included: portable X-ray fluorescence (p-XRF) and fiber optic reflectance spectroscopy (FORS), Raman spectroscopy, ground-penetrating radar (GPR) investigations with high-frequency antennas, multispectral reflectometry in the visible range, stimulated InfraRed thermography (SIRT), TeraHertz (THz) imaging and spectroscopy, digital holographic speckle pattern interferometry (DHSPI), nuclear magnetic resonance (NMR-MOUSE) [4], [5].
Moreover, in order to verify the actual presence of the Giotto fresco in currently non-visible portions and potentially confirm its age, plaster samples were collected, characterized and radiocarbon dated through 14C AMS. Despite the limited quantity of material, the binding fraction was extracted through wet gravimetric separation to eliminate the aggregate component present in the plaster mixture and avoid potential carbonate contaminants for dating purposes [6], [7].
The obtained results were promising and show that the dated samples from the layer under the 16th Century fresco yield calibrated dates that precede the last reported observation of the Giotto fresco and are statistically compatible with the 14th Century.
Furthermore, this study highlights the potential for dating frescoes, despite the limited availability of samples and the complexity of these types of artefacts.

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Radiocarbon Dating of Magnesian Mortars: The case of San Salvatore in Massino Visconti (NO), Italy

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The presented study involves the comprehensive analysis and radiocarbon dating of historical mortar and plaster samples from the San Salvatore - Massino Visconti complex, in Piedmont, Northern Italy. 13 mortar samples and one charcoal sample were collected from various areas within the complex's lower chapels. The samples underwent a thorough multi-analytical approach, revealing their compositional, mineralogical, and microstructural characteristics. Hydromagnesite and magnesite, were identified in the mortar samples and indicate the use of a magnesian binder mortar. These mineral phases in the mortar mixing might slightly shift the dating towards more recent dates due to the slower carbonation reaction of magnesian mortars when compared to usual calcitic ones. Magnesium oxide (MgO) from the calcination of magnesian carbonates is less reactive with water than calcium oxide (CaO) and has a lower carbonation kinetic. Typically, the carbonation of magnesian lime does not exceed 60% (compared to 95% for calcium lime), so calcium hydroxide transforms into calcite while most of the magnesium hydroxide either remains uncarbonated or carbonatates slowly and partially to form compounds like hydromagnesite [1]–[3]. Furthermore, the identification of LDH phases like hydrotalcite, also found in the binder fractions, is crucial for radiocarbon dating. LDH minerals form as a result of the pozzolanic reaction and have a high capacity to fix carbonate anions (CO_3^{2-}) into their crystalline structure, introducing contamination from younger carbon into the system, which would postpone the radiocarbon dates [4]–[7].
To address these issues, a method was developed to purify the mortar binders and the thermal treatment at 550°C (a temperature at which LDH phases decompose but not the calcium carbonate in the binding fraction, which decomposes at higher temperatures, approximately 800-850°C) was adopted [8], [9]. Therefore, the employed purification methods allowed to obtain datable binding fractions and the results indicate reliable radiocarbon ages, aligning with historical context. These findings provide valuable insights into the history and materials used in the complex's construction, ranging from the 12th to 16th centuries. Furthermore, consideration is given to radiocarbon dating of magnesian mortars, opening new perspectives for dating this type of mortar which was previously considered unsuitable for radiocarbon dating [10].

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More on Mortar: the study of Capiate site with statistical and deconvolution analysis

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Capiate, situated just 10 kilometers from Lecco, has maintained its significance since Roman times, owing to its strategic location along the Adda River. Over the centuries, this settlement has played a central role in numerous historical events [1].
The mortar samples under examination were extracted from the tower that stands as a welcoming symbol for the village and its church. Each of these mortars, when viewed as a palimpsest of the town's history, is assumed to represent distinct phases of construction.
However, even in the present day, the precision and accuracy of mortar dating continue to be topics of ongoing debate. This dating method relies on the unique characteristics of mortar, a masonry material that cannot be recycled. What sets mortar apart is its irreversible hardening process, which unfolds in the same time with the construction process.[2]
The primary aim of this study is to shed a light on the contributions of Optically Stimulated Luminescence (OSL) in mortar dating and highlight the central role that statistical analysis plays in refining the results obtained through this method.
Is known as, OSL dating of mortars depends on the phenomenon of quartz grains in the sandy aggregate being bleached during the mixing and laying of the mortar under natural light [3]. As a result, the ages deduced via OSL are intricately linked to the degree of quartz bleaching [2]. Exposure of the sand to light often fails to completely erase the luminescence signal of the quartz grains, resulting in an overestimation of the age determined by OSL.
A comprehensive data analysis was conducted on the Capiate mortars, which involved distributing the equivalent doses (ED), identifying exclusion criteria using various statistical indicators such as skewness and kurtosis, and, in addition, performing a statistical analysis, which included selecting an appropriate age model, including descriptive statistics, central age model, and minimum age model [4-6]. This is done with the purpose of refining the ages obtained with the SAR protocol alone that discorded from the a posteriori information obtained. [4–6].
Furthermore, the analysis delved into deconvolution of the raw OSL curve, examining multiple aspects such as kinetic parameters and the percentage contribution of each OSL component to the overall signal [7,8]
Our thanks go to Associazione Capiate-Radici nel futuro ONLUS for the samples and related information provided.

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Another brick in a Vitrified Walls

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On the slopes of Monte Serravuda, in the territory of Acri, Calabria, one encounters a puzzling phenomenon: a vitrified wall. This is a singular case of a vitrified fortification within Italian territory. Knowledge of these fortifications was widespread throughout Europe until the 18th century. Interestingly, these architectural enigmas are mainly present in island Europe and are rarely visible on the mainland. With the exception of a few isolated cases on the Iberian Peninsula, the countries of Mediterranean Europe have remained unaware of the presence of these structures [1,2].
Ongoing efforts have been undertaken to reveal the enigma surrounding these vitrified structures. Early investigations have ruled out associations with forest fires, smelting furnaces or lightning strikes, speculating instead on potential impact-shock events triggered by celestial bodies or, alternatively, the influence of as yet unidentified anthropogenic activities, perhaps rooted in prehistoric or protohistoric rituals [3,4].
The Italian vitrified forts is an amalgam of rocks of different natures [1]. More than twelve samples, with different geological compositions, including heated or partially vitrified rock fragments and assorted glass cements, have been meticulously dated. Thermoluminescence (TL) dating emerges as a potential tool to reveal whether the vitrified rocks discovered at Serravuda actually have the imprint of anthropogenic activities. Due to the glassy composition of these materials, some difficulties have emerged in applying a conventional protocol. Consequently, a different approach, known as "Pre-bleach with blue LEDs," was explored [5]. This procedure involves an initial signal bleaching phase that includes 500 seconds of blue LED illumination, followed by TL measurements with a known incremental dose, followed by subsequent cycles of bleaching and TL measurements. By adapting and testing this protocol on historical materials, such as mosaic tiles [6], it was found that the tested samples showed proportional signal growth as the dose increased, making them suitable for this protocol. A comparative analysis with the traditional measurement protocol was also performed, with a focus on quartz-rich samples.
Subsequent investigations included fading studies to correct for age data that had previously neglected this phenomenon. Curve fit parameters provided the means to reconstruct the fading effect and thus derive accurate ages. In addition, the reflections extended to the impact of iron oxides and the presence of moisture in the samples.
The results of these dating studies, together with technological insights gained from recent geological investigations [1], suggest that the vitrified forts located on Serravuda Hill underwent heating between the Late Bronze Age and the Iron Age, a period in which the hill was certainly populated. In conclusion, these new revelations provide valuable insights into the civilizations of the time, showing technological achievements of the time and causing a revaluation of the artefactual levels achieved.

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New radiocarbon dates reveal Brînzeni I cave's oldest past

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Brînzeni I is a cave site located in NW Moldova, in the Edinet district, a region characterized by its richness in resources, such as high-quality raw materials for the lithic industry, access to water, and food resources. The area, distinguished by adjacent river valleys with an abundance of caves on karstic limestone ridges, may have served as an ideal refuge for Homo sapiens during the colonization of Europe, even under reduced animal and plant productivity. This provided respite from the harsh climatic conditions that alternated throughout Eurasia during the Marine Isotopic Stage 3 (MIS 3, 60-25 kyr), as evidenced by the wealth of archaeological records in the region.
The oldest phase of Brînzeni I archaeological deposit, known as Cultural Layer III, were initially attributed to the Early Upper Palaeolithic, i.e. Aurignacian, based on flaked industry analysis (Noiret 2009; Allsworth-Jones et al. 2018; and literatures cited within). From this layer, 114 putative Ground Stone Tools (GSTs) were recovered, establishing this site as one of the richest in GSTs in Eurasia for the Upper Palaeolithic.
Our ongoing study of these artifacts has already examined a sample of 36 tools, considering morphometric, traceological, and residue aspects (Longo et al. 2021, 2022; Birarda et al 2023; Sorrentino 2023). The research has revealed the multifunctional nature of these tools, which were used for grinding, pounding, and crushing various resources, with a notable emphasis on plant storage organs. Furthermore, the traceological analysis indicates their prevailing involvement in vertical motions, such as pounding and crushing, which are typically associated with the processing of Underground Storage Organs (USOs), rather than the grinding gestures required for seeds and achenes. This observation is supported by the analysis of residues, showing the presence of starch granules attributed mainly to USOs.
In consideration of the high number of GSTs yielded by the Cultural Layer III of Brînzeni I, the largest collection attributed to the Palaeolithic to best of the author's knowledge, it is crucial to chronologically frame this evidence in a consistent setting. Until present 12 radiocarbon dates have been published bracketing between 14,700-26,600 uncal BP, among which those from 1993 are obtained according to AMS preparation but without ultrafiltration (Allsworth-Jones et al. 2018). This shift in dating attributes the site to a later phase, specifically the Gravettian or to an intermediate stage between the Aurignacian and the Gravettian (Noiret 2009). In this framework the GSTs collection appears to indicate a technological lag, which stands in contrast to the environmental carrying capacity of the Edinet basin evidenced by the abundance of archaeological sites. This disconnect was particularly surprising given the numerous opportunities for cultural exchange and development in the region.
The broad chronological range was then addressed during the present study by obtaining AMS-Ultrafiltrate dating on three anthropically modified bone samples from Cultural Layer III. The new dates are confirming the older estimates and will be discussed in the framework of the 'archaic' characteristics of the Brînzeni I GSTs collection. Our results confirm a long-lasting occupation but reattribute to the site its central role in the early stage of the Upper Palaeolithic.

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Archaeomagnetic dating: Does archaeointensity determination improve the dating precision? Insights from an ancient kiln excavated at Ceva (Northern Italy)

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Dating plays an important role in archaeological research and in the cases where age diagnostic artefacts and chronological evidence are missing, the use of laboratory scientific dating techniques is necessary. Archaeomagnetic dating is a promising dating technique, based on the ability of baked clay archaeological structures and artefacts (e.g., kilns, bricks, ceramics) to acquire a thermal remanent magnetization (TRM) when heated at high temperatures and cooled in the presence of the Earth's magnetic field. The precision of archaeomagnetic dating depends on several factors, among others the determination of the geomagnetic field vector and the availability of a detailed reference secular variation (SV) curve for the given territory.
We present here the results of an archaeomagnetic investigation carried out on baked clay samples collected from a rescue excavation kiln found near Ceva (Cuneo, Northern Italy). A total of 20 samples have been collected from different parts of the combustion chamber, all of them oriented in situ. Magnetic mineralogy experiments show the presence of both low coercivity and high coercivity minerals. Stepwise alternating field demagnetization was applied for the determination of the direction of the characteristic remanent magnetization. The obtained results were used for the archaeomagnetic dating of the kiln's last use after comparison with both the Italian directional reference curve and the predictions of regional and global geomagnetic field models.
To further investigate the improvement of the obtained dating interval by using the full geomagnetic field vector, archaeointensity experiments were carried out using the Thellier modified by Coe protocol, including partial thermoremanent magnetization (pTRM) checks and pTRM tail-checks. Our results show that the use of archaeointensity in archaeomagnetic dating can only be advantageous for the periods characterized by fast archaeointensity changes and only if a well-constrained intensity reference curve is available. Differently, archaeointensity does not significantly improve dating, taking into consideration that such analysis is very laborious and time-consuming.
The archaeomagnetic dating results obtained for Ceva kiln are in very good agreement with the archaeological evidence and with the radiocarbon dating obtained on a charcoal sample. Such results suggest that the kiln belongs to roman times, and it was abandoned before the first century AD, confirming the potential o

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When Absolute dating and indirect dating methods converge to magnify the archaeometric results: the case of the Eleousa icon from the Cefalù Cathedral

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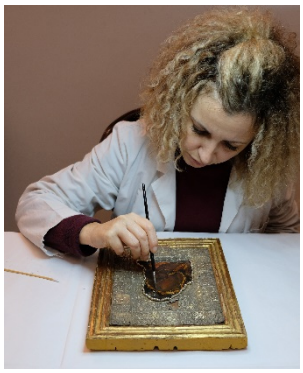
The restoration of the Eleousa, a composite artwork, made up of heterogeneous materials and different execution techniques, allowed the synergy of different professional skills to converge towards material and immaterial knowledge of a very valuable masterpiece of sacred art belonging to the treasure of the Cathedral of Cefalù (Palermo). The icon depicting the Virgin with Child is a small panel painting (h. 20.2 cm, l. 16.2 cm, max. thickness 1.6), enriched with a precious riza and a refined silk brocade fabric.
The technical-scientific study, starting from the needs of knowledge for the planning of the restoration project and from the poor information available in the literature about contemporary artistic production, involved the integration of the following methodologies for carrying out the investigations: Radiography, multispectral investigations, XRF and FORS analysis and in-depth analysis on the polished section analysed by SEM-EDS, and on a micro-fragment of support Carbon 14 dating and identification of the wooden species.
The purposes of the diagnostic and archaeometric study were: to identify the chronological context and contemporary references of the same production, to evaluate and to document the state of conservation of the wooden support, of the metal sheet constituting the riza, of the pictorial layers, restoration materials overlapped on the original ones over time (retouching or integration, traces of protective or consolidating treatments); to understand the methods of assembling the riza to the original support and, of the latter, to the framed table on which it was placed during a past intervention. Moreover, the scientific study was aimed to provide a deepen knowledge of the artistic technique and modus pingendi, starting from the observation of the underdrawings and revealing the subsequent phases of construction of the figurative system up to the final pictorial layer. Finally, the study allowed to characterize the materials constituting the different parts of this complex artwork: the pictorial palette through the identification of the pure or mixed pigments, the preparation layer and the primer, the chromophores in the fabric on the back of the panel; the precious alloy of the embossed sheet and the organic matric which was found at the pictorial surface - riza interface.


The integration of the data from the different methodologies which have provided evidence from indirect dating, through the identification of materials and techniques, and the data from the absolute dating, as well as the cross-referencing with the historical-stylistic data and the considerations deduced during the intervention conservative, have allowed a full chronological and artistic contextualization of the analysed artwork.

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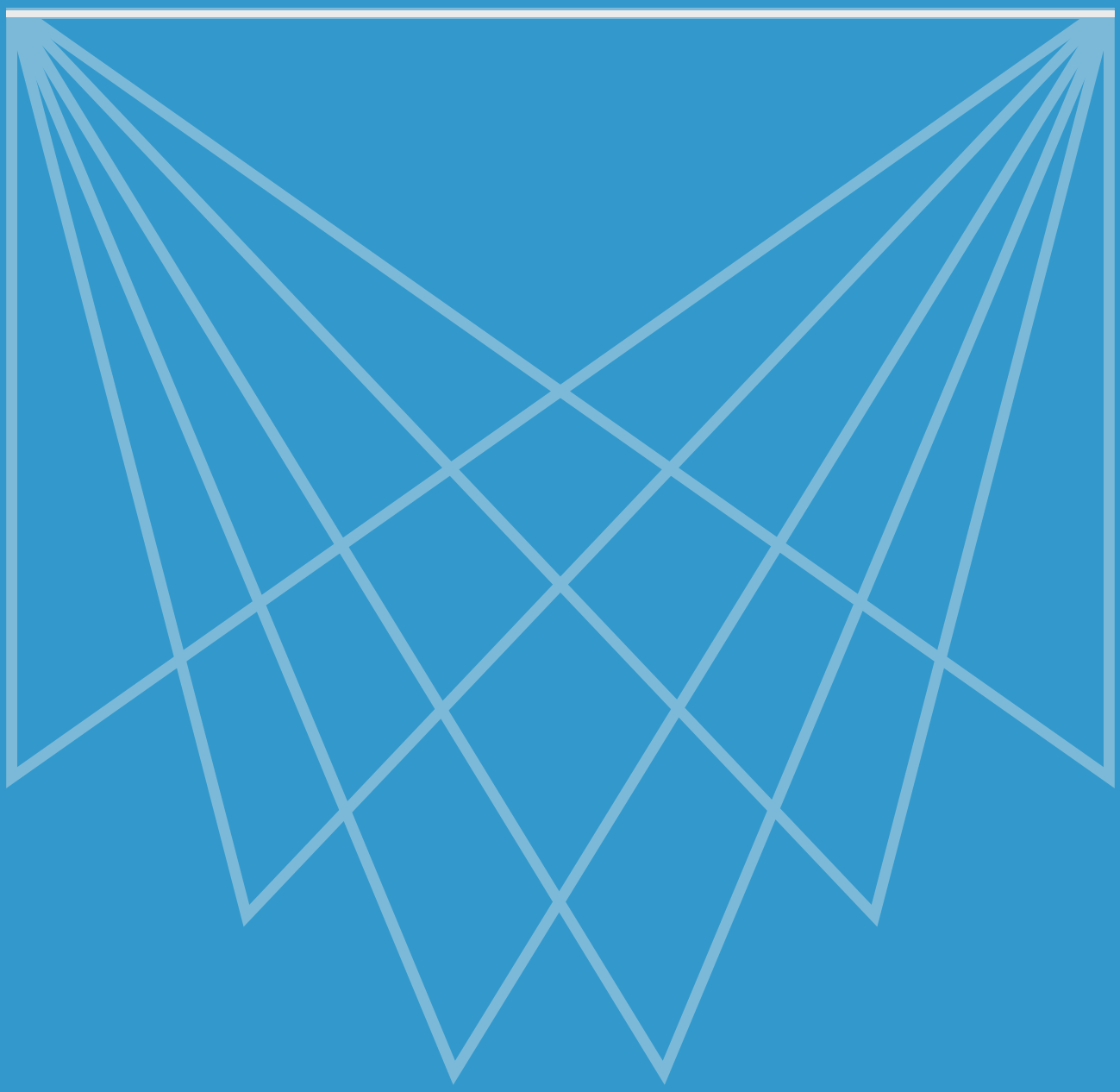

DATAZIONE INDIRETTA E AUTENTICAZIONE

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Combining RX results and historical-artistic features to recognize the attribution of XVII century wooden crucifixes: materials and techniques of the Fra' Umile production

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The Digital Radiography investigation carried out before the restoration works allow to collect useful data to define the state of conservation but increasingly representing an important moment of deepening of knowledge of the artistic technique. Nowadays the Digital Radiography has indeed great potential in solving archaeometry issues, such as dating studies, authenticity and studies on techniques typical of an artist or a workshop.
In the case of polychrome statuary, generally, Radiography provides an evaluation of the connections between the original materials and the parts added during the past restoration works. Number and typology of assembled parts, fractures, joints, nails, screws or grafts can be localized; and original or restoration materials can be distinguished thanks to their different radiopacity level. In order to illustrate the importance of the information provided by Radiography also for historical and artistic studies for supporting the attribution studies, three case studies of statuary depicting Christ Crucified will be shown. The analysed wooden sculptures belonging to: 1) the Sanctuary of Saint Umile from Bisignano (Bisignano, Cosenza); 2) the SS Crocifisso Church at Cutro (Crotone); 3) the Saint Salvatore Church at Gangi (Palermo). These sculptures are attributable to Fra' Umile from Petralia (Giovanni Francesco Pintorno) born in Petralia Soprana (PA) between 1600 and 1601 and died on 9 February 1639, belonging to the Order of Friars Minor.

The diagnostic results have been overlapped whit the available historical information. In particular, the Bisignano Crucifix is dated to the 17th century based on the following inscription on the back of the original cross: "1637. P.F. GREGORIO A BISIN. CUSTOM F. HUMILIS IN PETRALIA REFOR. SCULP."
The RX investigations have revealed for the three analysed sculptures the same construction technique and similar pictorial materials, supporting the coincident chronology and the attribution at the same workshop.
As far as the authors know, a systematic search of the materials and constructive solutions of these works is not yet available to the scientific community, which would allow the correct identification and differentiation of the workshops and active artists and consequently a scientific attribution with respect to that now based on artistic-stylistic criteria. This traditional approach indeed results often influenced by the difficult reading of the original models and surfaces altered by the numerous overlays and remakes to which these works have generally been subjected, limits that can be easily overcome thanks to a systematic radiographic investigation.

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The scan of relative chronologies of ancient buildings through the analysis of mortars processed by multivariate statistics. The case of The Sarno Baths in Pompeii.

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The analysis of mortars has become a research method that is increasingly applied for the scanning and verification of building phases in ancient monuments (Crisci et al. 2001), and it has frequently adopted for the study of Vesuvian contexts (Bonazza, Santoro, Mastrobattista 2010; Miriello et al. 2010). For this purpose, archaeometric results of mortars' characterization are often processed by multivariate statistical methods to facilitate the recognition of coherent groups of samples sharing common compositional features.
This presentation reports the results of the research on the structural mortars from the Sarno Baths complex in Pompeii, collected since 2016 by the team of Department of Cultural Heritage and Department of Geosciences of the University of Padova. Around 100 wall mortar samples were analyzed from already exposed structures (Secco et al. 2019, Dilaria et al. 2022) or during recent stratigraphical excavations in the building for a detailed in-laboratory compositional characterization. This was performed by integrating Polarizing Light Optical Microscopy, Quantitative Phase Analysis – X-Ray Powder Diffraction (QPA-XRPD) and Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS), allowing the determination of coherent groups of mortars. On the basis of known chronological markers, it was possible to scan the building interventions the complex was object of over time, both related to Roman times and modern restoration which were not mapped in the nineteenth- and twentieth-century cartographic documentation.
In particular, the contribution aims to present the analytical protocol we adopted, delving into the potential offered by multivariate statistics (PCA and Discriminant analysis) applied on QPA-XRPD data for the recognition and scanning of building phases and its possible application to the entire Pompeian palimpsest.

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Advanced analytical techniques to unravel the origin and dating of a presumed Caucasian floral carpet

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In the study of ancient carpets, the problem of determining the origin and the dating is fundamental to recognize the historical-artistic relevance of the artwork. Our collaboration with an international carpet dealer has allowed us to analyze interesting pieces from the past, in particular the carpet presented here. A first qualitative observation would seem to indicate that the carpet belongs to the group from the Caucasus region, with field floral decorations. The two main typologies of the 18th century Caucasus carpets were "Dragon carpet" and "Floral carpet". The carpet is woven with symmetrical (Turkish) knots on a cotton foundation (warp and weft), but the proportion between the sides is different from that shown in the reference examples. These last two characteristics raise doubts about the results of the qualitative observation; therefore, it was decided to undertake an analysis of the materials.
In the first part of this analysis, the Caucasian carpet was studied using non-invasive techniques: optical stereo microscopy and Fourier transform infrared spectroscopy (FTIR) in external reflection mode were applied to identify the nature of the textile fibres, while spectrofluorimetry combined with multivariate analysis for data interpretation was used to study the colouring materials. This information is, in fact, crucial to establish the dating and origin of the carpet since, in the past, dyes were extracted from natural sources, such as madder to obtain red hues, weld and indigo for the yellow and blue ones, while synthetic colorants were introduced starting in the 1850s.
Subsequently, as cross-validation, micro-samples were taken from the coloured areas already investigated by spectrofluorimetry and were subjected to dye extraction procedures with suitable solvents. The extracts were analysed by high-performance liquid chromatography coupled with a photodiode array detector (HPLC-PDA), liquid chromatography-mass spectrometry (LC-MS), surface-enhanced Raman spectroscopy (SERS) and micro-FTIR. The results obtained by these techniques supported the spectroscopic data, allowing us to formulate some preliminary hypotheses about the origin and dating of the carpet.

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Phoenician head pendants: Contextualizing archaeological and collection pieces

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The present study focuses on the archaeological and archaeometric characterization of two glass head pendants. The first pendant (fig. 1, a) was excavated from tomb nr. 9 of Nora cemetery (CA, Sardinia; Bonetto et al. 2022). It is set in a Phoenician-Punic cultural period, and this is also supported by the elements of the grave goods of the tomb nr. 9. The second one (fig. 1, b) is part of the Marchetti Collection, a private assemblage donated to the University of Padova in 2015 (Salvadori et al. 2018). It shows a Phoenicio-Punic style, but no information about the origin is available. Stylistic, typo-chronological and archaeometric analyses are combined to reconstruct the manufacturing techniques, identify the raw materials used, and potentially determine the origin of the pieces. The analytical techniques applied include stereomicroscopy (SM), OM, SEM-EDS, LA-ICP-MS and μ-Raman. In the case of the pendant from Nora, the sampled colours were dark blue-green and white. The results indicate that they are all soda-lime-silica glasses, characterized by high Na and low Mg content. Notably, the dark blue-green coloration of the body is attributed to a high concentration of Fe (5.5 wt%). The white glass was found to contain Ca antimonates in the crystalline form $CaSb_2O_6$. Traces of Sr and Zr recorded by LA-ICP-MS analysis, suggest the use of Levantine sands. The head pendant from the Marchetti Collection evokes a Phoenicio-Punic specimen and the closest comparisons for the piece are Seefried's types B III and C VI (Seefried 1982). The stylistic analysis seems to suggest that this pendant is not a philological reproduction of a specific type, but rather a pastiche inspired from two different models. The analysis was performed on 3 micro-samples of white, purple and blue glass. All results showed that these glasses are soda-lime-silica glasses, with Na_2O content ranging from 12 to 7 wt%. Interestingly, μ-Raman analyses on the white sample revealed the presence of cassiterite crystals. The use of cassiterite as an opacifier is documented within a limited chronological range, including the 2nd-1st centuries BC in Central European Celtic glass, from the 4th century AD onwards, and in modern glass. These findings contrast with the chronological range of the head pendants, which exhibit typological affinities with the Marchetti Collection's specimen. Ultimately, the analyses suggest that the pendant is most likely a forgery. The results obtained from the two head pendants will be compared with the limited analytical data available in the literature for similar objects (e.g., Arletti et al. 2012) and with the analyses of beads of different typologies discovered in the Nora cemetery (Bettineschi et al. 2020).

Fig. 1. a) The head pendant from Nora; b) The head pendant from the Marchetti Collection.

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Bridging the Gap: multidisciplinary insights into long-term climate variability using shallow-water sediments in the Magra river delta, Liguria, Northern Italy

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In our pursuit of comprehensive insights into long-term climate variability, a ground-breaking multidisciplinary approach is undertaken, highlighting the symbiotic relationship between radiocarbon dating, geological records, and historical data. This research is focused on the delta of the Magra River in Liguria, Northern Italy, a region marked by a convergence of flood-prone terrains and a rich history of human settlements.
Radiocarbon dating, a pivotal tool in this project, allows us to precisely date organic materials and decipher their chronological significance. Geological records, comprising sedimentary rocks and deep-sea sediments, offer a second layer to this intricate puzzle. They capture climatic oscillations over millennia and high-resolution data on glacial-interglacial cycles.
The essential third dimension is historical data, spanning centuries and often overlooked in conventional geological studies. These historical archives bridge the gap between instrumental data and geological records. These repositories provide crucial context, offering a unique window into climatic conditions and human interactions over the past 1,000 years.
The study capitalizes on the Magra River delta's unique attributes, characterized by high sedimentation rates (~1 cm/year). This natural phenomenon enables us to record short-term climate events with unparalleled precision, accentuating the need for a synergy of radiocarbon dating, geological records, and historical data.
This holistic approach recognizes the intricate nature of long-term environmental processes and underscores the unparalleled potential of combining radiocarbon dating, geological records, and historical data to unravel the complex tapestry of past climate change. By forging these interconnections, this research aims to provide vital insights into long-term climate variability, thus empowering us to address pressing issues like rainfall patterns, infrastructure planning, and sustainable water resource management. In a world facing ever-escalating environmental challenges, this multidisciplinary endeavour offers an innovative roadmap to holistic understanding and informed action.

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A Bridge between the Pillars of Hercules: Dating an Ancient Quranic Manuscript

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Quran volumes are a product of the Muslim East, involving the work of multiple professionals and sometimes incorporating reclaimed elements.
We present the research employed and the results obtained for the dating of an ancient Quran volume that has been the subject of an in-depth study that proceeded gradually and consisted of an initial extensive indirect dating investigation, followed by radiocarbon dating for only some of the pages.
It is important to consider that, like many other documents and art objects, this book is composed of multiple elements: the cover and its inserts, the paper and its watermark, the binding, the layout, the internal and external decorations within the frames, the content of the colophon, its language and the calligraphy used to write it. A proper assessment of the artifact must take all of these factors into account, both for indirect dating and to evaluate its state of preservation.
The diagnostic campaign presented mainly involved non-invasive diagnostic imaging such as geometric surveys, transillumination and UV photos, and non-invasive XRF testing of some spots of the decorations. Through these analyses, a wealth of valuable information was collected making it possible for" invasive" analysis with the C14 method to be limited to a small number of fragments.
Among the elements useful for indirect dating, alongside e.g. the use of certain pigments, one specific example is the use of watermarked paper, which was found to be of European production, as opposed to non-watermarked paper, which had been long requested for Muslim sacred texts. This allowed for situating the production of this specific manuscript within a broad yet specific time frame.
This paper aims to illustrate all the phases of the analytical study that led to the dating of the manuscript, considering material, technical, historical, and socio-cultural aspects.




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Stylistic dating and compositional analysis of the two understudied works by Benedetto Buglioni (1459/1460–1521) in Polish Collections

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Partly inspired by ceramic imports from Asia, North Africa and Spain, tin-glazed terracotta sculptures constituted a distinctive Italian Renaissance project. Previous studies have focused on the understanding of the technique of the Della Robbia artists (Vaccari 1996; Gianoncelli et al. 2006; Bouquillion et al. 2011; Kupiec 2016; Hykin 2016; Walker and Riccardelli 2019), the inventors of the glazing method according to Italian contemporary written sources. By contrast, this study addresses the stylistic dating and compositional analysis of Italian Renaissance glazed terracotta sculpture created by Benedetto Buglioni (1459/1460–1521). Two artworks, now in the National Museum in Warsaw and in the Czartoryski Collection in Cracow, dated on stylistic grounds to the most prolific period of his artistic career namely, c. 1490–1510, have been examined using X-ray fluorescence spectroscopy (XRF). Notwithstanding the limitation of the technique, really for its portability and non-destructiveness, XRF has been used in several cases of glazed ceramic studies (Bajnóczi 2015; Zumbulyadis 2020). In the paper by Pappalardo and co-authors (Pappalardo 2004), XRF has been coupled with PIXE for the evaluation of both light matrix and trace medium-heavy elements.
The results we obtained are interpreted against the details of the technique, as reconstructed by previous published examination of the Della Robbia glazes and written sources, including an early sixteenth-century recipe book from Montelupo, a Tuscan centre of tin-glazed earthenware production. The study sought to assess whether the dating of those artefacts can be verified through the non-invasive method of analysis, conducted in situ. The physical examination of the two considered objects shows that within a relatively short time span the composition of Benedetto Buglioni's glazes changed, which can point to a high degree of experimentation during those two decades.
The paper argues that though around 1490s Benedetto Buglioni might have struggled to achieve the same results as the Della Robbia artists, towards the 1510s he moved beyond the faithful repetition towards the experimentation and development of his own composition of the glaze. This hypothesis seems to be supported by the differences in the composition of the glaze, observable using the non-destructive method of analysis, as well as through observable change in the quality of the glaze from lumpy and uneven surfaces of the Tondo with Christ the Redeemer from the National Museum in Warsaw dated to c. 1490, to merging and interactive effects of the glazes in the Virgin and Child from the Czartoryski Collection, Cracow dated to c. 1510.

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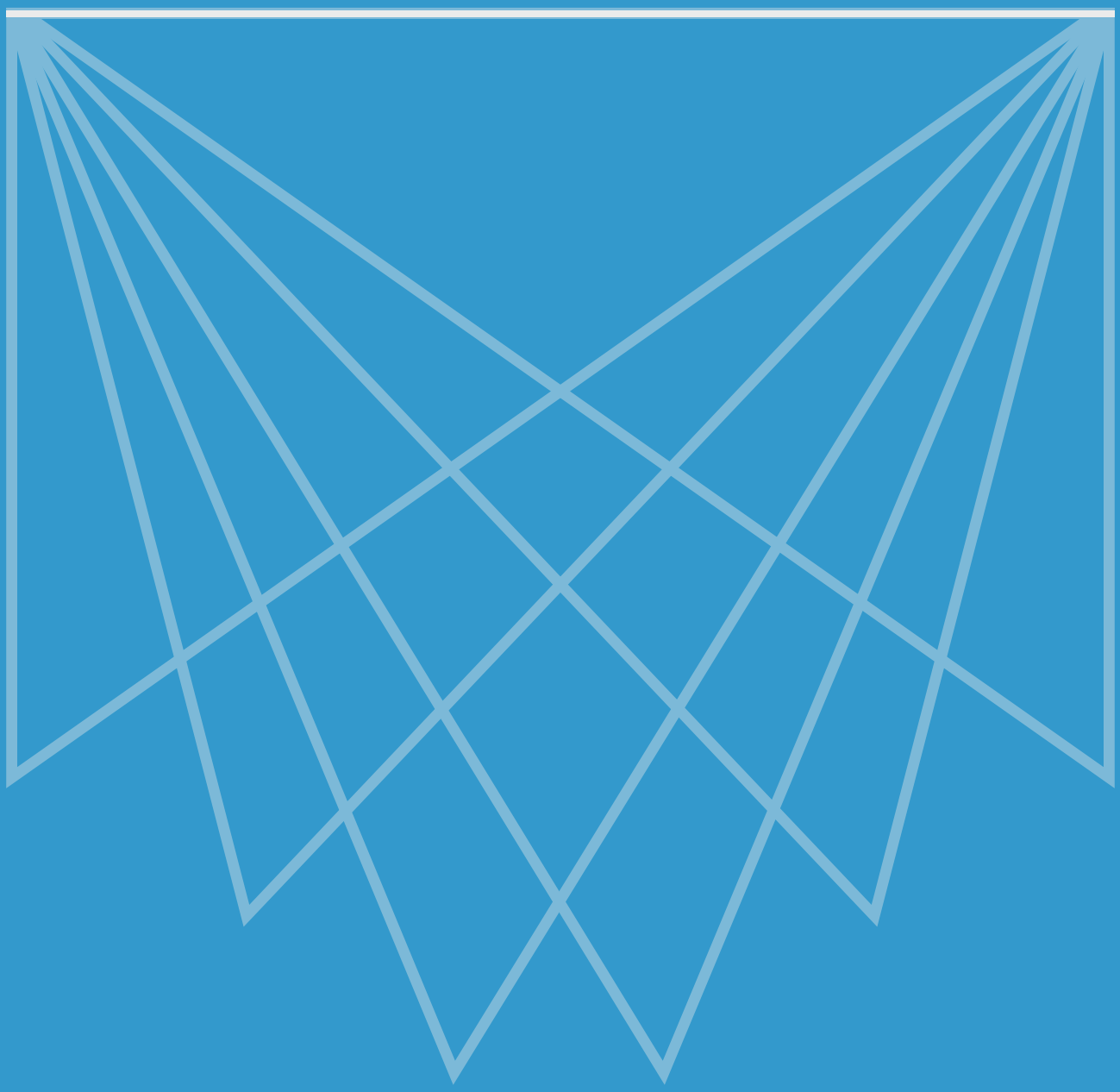

I MATERIALI NEL TEMPO: TECNOLOGIA, DEGRADO E CONSERVAZIONE

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Evaluation of possible conservation methods for the stone façade of St. Stephen Cathedral (Vienna) on the model of the St. Mark Basilica in Venice

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Although there exist various scientific and internationally accepted guidelines describing valuable procedures for the restoration of monuments, the implementation of these recommendations differs from region to region. To represent two different conservation approaches for historically significant buildings, two prominent religious monuments, central to their respective cities, were selected: St. Mark Basilica in Venice and St. Stephen Cathedral in Vienna.
Since their erection onwards, these two cathedrals have been preserved and studied by their own institutions devoted to protection, maintenance and repairs of the cathedrals and their pertinences, i.e. the Procuratoria di San Marco and the Dombauhütte von St. Stephan. Until today, both institutions have developed and documented distinct conservation approaches for the façades, creating a huge archive and numerous publications. On one hand, the preservation of historical stones, marbles and glasses employed in the building has been the priority in Venice, whereas traditional techniques and craftsmanship have been used to frequently replace deteriorated stonework in Vienna. The main reason for the development of distinct approaches to monument preservation could be the differences in construction and materials, which could have influenced the perception and treatment of the monumental façades. St. Mark Basilica is covered with precious marbles often bearing symbolic values; in contrast, the façade of the St. Stephen Cathedral is built of a local calcareous sandstone named Leithakalk. In particular, for replacing deteriorated stones, it has been used the St. Margarethen cal- carenite from the ancient roman quarry declared World Heritage Site by UNESCO in 2001. However, the substitution of historic elements implies the loss of the original ones and the use of a lithotype similar but not equal as the original one or the one in place. For this reason, this work aims to elaborate an alternative approach to preserve the historical stonework of the St. Stephen Cathedral façade by applying the conservation procedure implemented on St. Mark Basilica.
Starting from the nineteenth century, the most significant restorations from each church were studied and placed within the context of the ongoing debate on restoration principles.
In order to gain profound knowledge on the characteristics of the building stones, a series of mineralogical-petrographic and chemical-physic analyses were carried out on samples from the quarry and from a highly deteriorated stone ornament covered by black crust. Four different consolidation mixture were tested in an attempt to extend the resistance of the stone material over time. The selected strengthening agents are (i) KSE 100 (silicic acid ester) by Remmers, currently adopted for restoring specific statues and ancient stonework of St. Stephen's Cathedral; (ii) Nano Estel purchased by CTS, a nanosilica commercial product chosen in reference to the European project called Nano-Cathedral; (iii) the “Bologna cocktail”, an acryl-siliconic mixture widely and successfully used for restoring marbles of St. Mark basilica in Venice. The latter was tested by preparing a Paraloid B72 (CTS) and siloxane resin W224 (Siliconi Italia) fresh solution, as well as by using Acrisil 201 O.N. (CTS), a commercial “Bologna cocktail” ready to use.
From the comparison between the results obtained, a proposal for the maintenance plan of scheduled conservative interventions of St. Stephen Cathedral was developed in order to reduce replacements and to slow down the deterioration of the historic stone façade.

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Cultural Heritage Preservation and Sustainability: Conservation Strategies for "The Sassi and the Park of the Rupestrian Churches of Matera"

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Preserving cultural heritage and ensuring its sustainability have become crucial responsibilities for humanity in relation to unprecedented challenges deriving from climate change and socio-economic shifts.
The vulnerability of cultural heritage has escalated over time and new strategies and approaches for adaptation are requested for safeguarding heritage against ongoing stresses and decay. This requires not only effective policies and management systems, but also in-depth research to address knowledge gaps and to facilitate practical implementation.
In this context, this study focuses on the conservation of Gravina calcarenite, a weakly cemented carbonate sandstone with a yellowish-white color, high porosity, and low mechanical strength, which hosts the UNESCO site The Sassi and the Park of the Rupestrian Churches of Matera (Southern Italy). The Sassi di Matera exemplify the harmonious integration of ancient architecture with geological and geomorphological features of the area, resulting in a unique and culturally significant form of long-standing habitation, dug into the rock, partially constructed using the same excavated materials or made from rock sourced from nearby quarries. Gravina calcarenite formation exhibits a significant degree of heterogeneity. This implies the formation of various decay morphologies and alteration products due to weathering, which is important to recognize and contextualize for developing the most suitable conservation strategy. Consequently, the possibility to identify effective methods of monitoring and preserving the UNESCO site enhancing the historical and landscape context, through highly sustainable and eco-compatible approaches is of paramount importance.
A multi-analytical intervention protocol has been implemented for establishing a practical procedure based on efficient diagnostic methods and technologies to characterize Gravina calcarenite and the related deterioration processes. On the other hand, an easy, low-cost, monitoring practice after restoration has been proposed to assess the stability of consolidation actions using innovative and eco-compatible products. For this purpose, the effectiveness of some nanoparticles (calcium hydroxide and silica) and traditional products (ethyl silicate-TEOS) has been tested comparing commercial mixtures present on the market.
The preliminary results obtained encourage further research and support the development of a protocol for assessing the durability of Gravina Calcarenite.
This work is part of the current common interest in the built heritage subject to climate change, complementing and supporting conservation and restoration efforts in areas of particular originality and cultural distinctiveness.

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Elemental analysis of black crusts using Laser Induced Breakdown Spectroscopy for the determination of pollutant sources

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Black crusts represent one of the most widespread degradation phenomena on stone surfaces exposed to the outdoor environment and protected from washout. The metal-rich carbonaceous particles which compose particulate matter (one of the major air pollutants) play a key role in the formation of these dark patinas by catalyzing the sulphation of the calcareous substrate leading to the formation of gypsum. Numerous source apportionment studies carried out on particulate matter highlight the possibility of determining the pollutant sources based on the elemental composition of the particles [1]. For this reason, elemental analysis on black crusts has been carried out throughout the years to determine the main pollutant sources of the surrounding areas and put forward targeted mitigation strategies for the protection of cultural heritage.
However, the most employed analytical technique for these studies is Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) [2], which requires extensive sample preparation, including the need to retrieve part of the black crust from the monument. In this context, Laser-Induced Breakdown Spectroscopy (LIBS) may represent a valid alternative, due to its micro-destructive nature and the potential for in-situ applications. Another advantage of LIBS is the possibility of performing stratigraphic analyses to determine the in-depth distribution of metals and other elements. To date, few studies have been performed on black crusts using LIBS [3,4] and none of them assess the ability of this technique in recognizing the main pollutant sources by evaluating the presence of tracer elements.
In this study, black crust samples were collected from the Monumental Cemetery of Milan (Italy) in order to assess the ability of LIBS in recognizing the main pollutant sources of the surrounding area (surface analysis) and their variation throughout the years (stratigraphic analysis). Indeed, Milan is one of the most polluted European cities and the sampling area is located less than 1 km way from the second main railroad of the city, as well as being at the center of numerous heavily trafficked arterial roads. Therefore, railroad and vehicular traffic represent two significant pollutant sources, which are characterized by several tracer elements (markers). Only previously detached samples were taken for analysis from funerary monuments, enabling precise dating of the crust and of the years of pollutant accumulation.
LIBS surface analysis proved successful in identifying the main tracer elements of railroad traffic (Fe, Mn) and vehicular traffic (Ti, Zn, Cu, Cr, Ba). Also, using CaF molecular bands it was possible to identify the presence of fluorine for the first time in black crust sample analysis. This element is used as a flux in the production of steel, which is the main component of train wheels and rails. Moreover, stratigraphic analysis of the four elements found in all samples (Fe, Mn, Ti, Ba) showed a decrease in concentration from the surface towards the bulk of the crust, giving interesting insight into the variation of the impact of pollutant sources throughout the years.

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The white illusion: archaeometric study and authentication of white decorations on bronze fibulae from Parre (BG)

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he archaeological site of Parre, Bergamo province, is known for the discovery of a conspicuous bronze hoard at the end of the 19th century. Further excavations performed by the Superintendence of Lombardy uncovered an important Iron Age settlement (Poggiani Keller-Rondini, 2020).
Among the numerous bronze artefacts from this archaeological site, 13 bronze fibulae with white decorations were selected for analyses. These fibulae belong to different types and display various linear and/or geometric decorations, marked by an unusually bright white colour. The presence of whitish decorations on bronze artefacts dating back to the Iron Age in Northern Italy is well documented, as in the case of leech fibulae with circular inlays (De Marinis, 2000). These latter fibulae, however, are generally filled with denatured red coral, nowadays bleached and displaying a dull whitish hue. The decorations of the archaeological finds from Parre, instead, show a much brighter hue with respect to the other artefacts of comparable chronology – even quite similar to the white ink often used in the marking of archaeological finds (white Indian ink).
The 13 fibulae were thus analysed with a completely non-destructive, multi-analytical archaeometric approach – involving optical microscopy, μ-Raman spectroscopy, μ-X-ray diffraction and scanning electron microscopy with EDS – the same one used to study the bronze artefacts decorated with coral inlays (Berruto et al., 2023). This approach brought to an exhaustive characterisation of the raw materials used to produce these 'brighter' white decorations, allowing a check for what concerns their authenticity and chronological compatibility within the period in which these artefacts were made. In most cases presence of titanium dioxide (TiO2: white Indian ink) was unequivocally acknowledged: this material accounts for a modern intervention, presumably carried out with the aim of highlighting the decorations for a presumed public and scholarly use.

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Chemical degradation induced by the photo-aging of beeswax found as additive in multi-component archaeological adhesives

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The adoption of adhesives marked a significant milestone in the behavioral evolution of prehistoric people [1]; in fact, the possibility of joining together two different parts to produce a composite tool was a significant advance in the evolution of stone tools, paving the way for further development of hafting technologies [2], [3]. Resinous substances secreted by trees were widely used as adhesives, either in their natural form or as tar and pitch, but, over the years, the base material required improvements. For that reason, additives, such as ochre and beeswax, were added to manipulate the physical and chemical properties of the adhesive, especially malleability, plasticity, and adhesion performance [1], [4]. In this optic, it is fundamental to study how the simultaneous presence of multiple components could impact the aging process of each material. As already known in the literature, beeswax is susceptible to UV radiation [5]–[7]; starting from this point, we studied the
modifications provided by photo-aging on the beeswax and if the presence of a resinous material could affect this process.
For that aim, evolved gas analysis coupled with mass spectrometry (EGA-MS) and double-shot pyrolysis gas chromatography/mass spectrometry (Py-GC/MS) were performed. All experiment was performed using both raw beeswax and beeswax mixed with pine resin, either fresh or naturally aged by exposing them to solar radiation for one month. In addition, the mixtures were also heated to simulate the most common ancient processes used in adhesive manufacturing.
EGA-MS was used to study the thermal stability of the samples to obtain information on the generation of a macromolecular network ascribable to the aging process.
For the double-shot Py-GC/MS, the experiments started with the thermal desorption of the more volatile components of the samples and their gas chromatographic analysis. The non-volatile fractions of the samples were then subjected to pyrolysis in the second step. In addition, the experiment was performed also using an online UV irradiator, to directly analyze the compounds generated during the exposure of the sample to UV radiation [8].
The gas chromatographic analysis highlighted that the pine resin did not act as an antioxidant on the beeswax, which underwent chemical degradation after exposure to UV radiation to produce smaller oxidized compounds.

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"A warning from Mars": effect of microclimate changes at the Specola Museum of Palermo

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The Specola Museum is located at the Palermo Astronomical Observatory and preserves most of its material heritage. It is part of the Museum System of the University of Palermo (SiMuA) and its management and conservation are entrusted to the INAF - Astronomical Observatory of Palermo. Among the historical instruments on display, there is a 19th-century painted wooden globe reproducing the surface of Mars. It has been on display in a showcase, inside the Merz Telescope Hall, for about 20 years without undergoing any type of intervention or evident deterioration sign. From 2021, in less than two years, damages of its pictorial layers occurred at a progressive rate. The European Standard EN 15757 explains that hygroscopic materials are highly vulnerable to microclimate changes. The wooden globe was in good conditions after two decades of permanence at the museum, but the recent control and management of the indoor climate was not compatible with the past climate history of the globe to which it has adapted.
The speed of the degradation made it evident that the microclimatic conditions of the room changed abruptly. The warning deduced from the Mars globe deterioration was hence easy to decode: there is something wrong with this climate!
It is well known that museum microclimate plays a key role in the conservation of the objects on display. It may trigger or aggravate degradation processes of the materials constituting the object exhibited and affect their "life expectancy". Moreover, each object responses peculiarly to environment variability, depending on its composition and conservation history. When an artifact is composed of different materials, unexpected synergistic effects may develop. Finding appropriate values range of temperature, relative humidity and illuminance and managing them to guarantee the conservation of the entire collection, and at the same time improving the thermal comfort for people working there, is a difficult task. The issue becomes even more challenging in buildings not originally designed for conservation purposes, as the Specola Museum, located in the ancient observatory rooms, built in 1790 on the top of the 12nd-century Royal Palace.
An accurate restauration has been made, and conservation measures have been adopted, to stop the serious deteriorating processes affecting the model of Mars but the risk of further deterioration phenomena involving other objects is expected to increase substantially if no actions are taken.
This contribution intends to present the results of the preliminary study concerning the thermo-hygrometric records taken in the Merz Telescope Hall over the last two years and propose specific actions to improve its microclimate conditions.

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Stones of Comiso, Modica and Ragusa: Study of Characteristics and Durability of Calcarenites for the Conservation of the Iblean Heritage

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Many cities in the Iblean area of eastern Sicily (Southern Italy) are characterized by the presence of majestic and imposing Baroque monuments constructed with different types of calcarenite (Belfiore et al., 2010), among which Ragusa Stone, Comiso Stone and Modica Stone. These stones, although their relevance and diffusion in the field of architecture, are subject to intensive processes of degradation (Barone et al., 2008), making their conservation an urgent issue.
The present research aims to conduct a detailed investigation of the characteristics of these three types of calcarenite, which, although they may appear similar in terms of colour and macroscopic features, exhibit differing responses to degradation and conservation efforts. These differences stem from variations in the petrographic and physical features of the materials (Anania et al., 2012; La Russa et al., 2015). Factors such as mineralogical composition, capillary rise, pore size, and differential mechanical properties are elements conditioning the type of degradation observed (Belfiore et al., 2021). For this reason, minero-petrographic and physico-mechanical analyses will be carried out on representative samples of each calcarenite type (Belfiore et al., 2021; La Russa et al., 2011). Specifically, the analyses consist of optical microscopy, x-ray diffraction (XRD), mercury intrusion porosimetry (MIP), salt crystallization test, determination of ultrasound speed, uniaxial compressive strength, flexural strength, and elastic modulus. Samples of Comiso Stone will be directly extracted from quarries, while those of Modica and Ragusa stone will be obtained from natural outcrops (Barone et al., 2008).
Thus, the definition of the physical and chemical properties and the related criticalities of these stones will provide a substantial contribution to the development of strategies for the preservation and protection of the built heritage in the Sicilian region.

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A 3D Diagnostic Imaging Model (DIM) approach for the preservation of cultural heritage: the M3DEA vision

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M3DEA proposes a novel methodological approach (Diagnostic Imaging Model, DIM) for the quantification of surface damage of cultural heritage in relation to the climate change effects, by comparing high-resolution 3D reconstructions obtained from selected monuments and those obtained from historical replicas (with known date of production).
Besides its historical importance (part of UNESCO World Heritage Sites of Rome), the Trajan's Column (113 A.D) represents a very significant on-site monument in order to optimize this novel DIM approach because of many reasons: the Trajan's Column counts with many historical plaster casts/replicas (1667-1670, 1861-1862, 1939-1943) and photographic reports realized during a major last century restauration work (1980-1990); the environmental parameters of the city of Rome are largely known with complete annual climate historical series available since 1862 (meteorological observatory Collegio Romano); the bas-reliefs of the Trajan's Column are characterized by a complex decoration and a large number of details, hence its meaningfulness in defining standards for a morphometrical/geometrical analysis in relation with the deterioration process; the bas-reliefs are made with Carrara marble, a primary choice for historical monuments in Mediterranean basin and modern buildings all over the world.
M3DEA proposes a damage assessment approach that i) combines laboratory tests, on-site surveys, and 3D modelling to provide quantitative indicators highly representative of potential climate change effects on long-time scale, based on multi-scale phenomena from the current state of decay and historical climate data; ii) identifies the main environmental stressors and refine the dose-response functions (DRFs) in order to foresee future deterioration patterns with reliable simulations according to the Intergovernmental Panel on Climate Change (IPCC) scenarios (e.g., to years 2050, 2100); iii) considers the loss of surface material in relation to a hierarchical classification of geometrical features and converts stone recession in terms of Loss of Details (LoD) as readability reduction, which will be further elaborated to decline of Future Cultural Value (FCV), the capacity of a cultural heritage to transmit its cultural message in its future appearance; iv) develops a DIM, a suitable tool to be implemented on a larger scale for the preservation of historical built heritage.
M3DEA vision adheres to the Sustainable Development Goals of the Agenda 2030, in particular to that dedicated to the protection and safeguarding of cultural heritage (SDG 11). It holds the ambitious goal to support local authorities in historical urban planning by defining quantitative indicators of the climate potential impacts, employing an innovative approach for evaluating the permanent loss of cultural value beyond material damage.

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From macro- to micro-scale study and characterization of the 15th century painting Coronation of the Virgin

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This research study is focused on the characterization of an altarpiece from a private collection, namely the Coronation of the Virgin by Michele di Matteo (1410-1469), a painter of the late Gothic period who actively worked in Bologna. A diagnostic campaign on this artwork was carried out to identify the constituent materials (pigments and binders), the execution technique, the presence of restoration interventions, and to assess the state of conservation to identify the appropriate restoration intervention to be performed.
Non-invasive and micro-destructive analyses were carried out both before and after the restoration to monitor the quality of the cleaning work and highlight any differences. The non-invasive investigations, such as multispectral imaging (MDI), colorimetric and Raman analyses, were carried out to obtain useful information on the surface composition of the panel [1-2]. The first technique has recently shown great potential when applied to ancient paintings [3]; in fact, it provides information for the study of pictorial materials and their mapping on the painting's surface. Moreover, being able to choose the appropriate wavelength with which to illuminate the painting enables the effective observation of details which would otherwise not be easily visible to the naked eye. The second was used on different areas of the painting, obtaining the colorimetric coordinates of the points analyzed before and after restoration and their reflectance spectra in the visible range. The third technique is useful for identifying pigments. The micro-destructive investigations involved taking micro-samples before the cleaning phase in order to try identify the pigments and eventually the binders used in the painting. In addition, investigations were carried out on the swabs used during the cleaning operation to map the presence of varnishes and resins applied to the surface of the painting over time.
Investigations conducted using visible luminescence induced by ultraviolet light (UVL at 365 nm) showed an inhomogeneous fluorescence generated by the superimposition of several materials of different nature [4]; some areas were affected by blue/green fluorescence, sometimes associated with protein-based varnishes, and others more yellow associated with oil-resin materials. This layering, together with the observation of numerous retouches, made it possible to assess that the work has probably undergone several restorations over the years. Infrared reflectography (IRR; 950 nm high-pass IR filter) revealed the presence of a double signature at the bottom, below the currently visible one [5], and a light preparation on which the preparatory drawing was executed, outlining the figures and rendering the details in a special way. This technique made it possible to advance hypotheses on the use of certain pigments, such as azurite for the Virgin's mantle and the use of cinnabar for the Virgin's robe. Infrared Reflectance False Colour (IRRFC) analysis revealed the presence of a repainting on the blue mantle resting on the Virgin's head that appears dark red, probably belonging to a blue pigment of different nature. The robe and background, on the other hand, which appear red in the visible and transparent in the IRR, here appear orange/yellow, indicating probably the use of the pigment cinnabar [6]. Analyses on the micro-samples and swabs performed using SEM-EDX and FT-IR/ATR techniques made it possible to hypothesize the binder used and recognize the paint applied during the restoration, validating the observations obtained through the non-invasive study. Analyses using the Raman technique are still in progress and will be used to validate the hypotheses made about the pigments.

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Rediscovering pigments and stratigraphy of the 15th century panel painting depicting the Coronation of the Virgin by Michele di Matteo

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In the field of diagnostics and conservation of cultural heritage, the study of a wide variety of artworks may be conducted with a scientific multi-analytical approach. A combination of non- and micro-invasive spectroscopies may be essential to increase knowledge of all the heterogeneous materials in a complex and multi-layered panel painting stratigraphy [1,2].
This diagnostic study focused on the panel painting depicting the Coronation of the Virgin, dating from 1426 and belonging to a private collection. It is attributed to Michele di Matteo, a late Gothic Italian painter active in Bologna in the 15th century [3]. The main aims of this project were the characterization of the original painting materials (e.g. pigments, lakes, binders, gilding) and the identification of materials used in the preparation of the wooden support.
A combined multi-analytical approach based on non- and micro-invasive spectroscopic techniques was employed to study the panel painting: the X-Ray Fluorescence (XRF) and External Reflection Fourier Transform Infrared (ER-FTIR) spectroscopies allowed the chemical identification of pigments, binders, and varnishes. Considering the historical and artistic importance of the work and the painter, a micro-invasive approach, limited to the areas of interest, was conducted: in particular, Attenuated Total Reflection Infrared (ATR-FTIR) spectroscopy, Optical Microscopy (OM), and Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS) were selected to visualize and characterize the materials of the pictorial layering. Visible light (VIS) and Ultraviolet Induced Fluorescence (UVIF) photography were used to select the areas of interest for the spectroscopic analyses, as well as X-ray radiography (RX) was necessary to assess the state of conservation of the support.
The wooden support was prepared with a canvas in the upper part, and the preparation was made with the use of chalk and lead white. The multi-analytical approach highlighted the presence of a selection of pigments consistent with the period of production of the painting, as well as restoration pigments used during the most recent restorations. Cinnabar, red ochre, and smalt were identified in the red and blue areas, possibly applied with an egg binder. Modern pigments such as Prussian blue have been revealed. As for the gilding areas, the use of red bolus has been confirmed as a preparation for the gold leaf.

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Monitoring and assessment of the state of preservation of bronze sculptures exposed outdoor

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The preservation of cultural heritage is a critical issue, and the study of atmospheric corrosion of metallic works of art exposed outdoors is an essential step toward effective preservation strategies that can avoid their degradation over time. Preventive conservation strategies play a crucial role in the long-term protection of Cultural Heritage, and the ultimate goal is to define the most suitable conditions to guarantee the safeguarding of artworks as long as possible.
To achieve this goal, an interdisciplinary multi-analytical approach can help evaluating the current state of preservation of cultural heritage artifacts and the effects of their interaction with the environment. In particular, non-invasive diagnostic techniques and in-situ measurements are indispensable tools to obtain valuable data and information in long-term monitoring campaigns [1-3].
From this perspective, this paper presents a long-term in-situ diagnostic campaign carried out on several bronze artworks from the Collezione Gori, a private collection of metallic sculptures and installation located in the Fattoria di Celle, Santomato (Pistoia, Italy). The campaign, employing multiple portable analytical techniques, began in 2018 and is still ongoing [2-3]. Electrochemical Impedance Spectroscopy (EIS) is employed to study the corrosion processes affecting metallic artworks, while X-ray fluorescence (XRF) and Raman Spectroscopy are used for chemical and microstructural characterizations. Additionally, a 3D photogrammetry survey is conducted to create a comprehensive documentation of the artworks. Eventually, the measurement campaign was recently integrated with a series of sensors for temperature and relative humidity monitoring.
The developed multi-analytical approach has been applied in different measuring campaigns since 2018 and it has enabled the identification of the composition of corrosion products present on the metallic surface, the characterization of their chemical and microstructural features, and the correlation of the microclimatic parameters with the formation of the various corrosion products, based on the different orientation and exposure to atmospheric agents. All the gathered information on the state of preservation of the artworks has been integrated into a virtual 3D model that can be stored and shared with curators and conservators. By establishing monitoring campaigns at different periods, every 6 months, dangerous situations and the need for restoration can be highlighted.
In particular, the measuring campaign allowed the identification of the surfaces more exposed to the aggressive agents and to the washing action of the rain, which induced the formation of reactive corrosion products and selective degradation of the protective coating previously applied on the metallic surface. The collected data were used by conservators to tailor their actions, paying particular attention to the areas identified to be at risk.
The paper will present and discuss the findings of the ongoing in-situ monitoring campaign, emphasizing the benefits of the proposed approach, and addressing the challenges that need to be tackled for the development of customized preservation strategies for outdoor bronze artifacts.

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Microclimate Analysis of the King Apartment in Royal Palace of Turin

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The Royal Palace in Turin was originally built in the XV century and it was the residence of the Savoy family until 1946 when, with the birth of the Italian Republic, it became a State property and was turned into a museum.
Inside the Palace there are several apartments which over the centuries have hosted prominent members of the House of Savoy. The King Apartment is located at the ground floor in the main body of the Palace and it is composed by five rooms and some communication and service spaces.
The apartment is currently closed to the public and has no heating and air conditioning system. With a view to a future use of these rooms for exhibition purposes a microclimate study has been planned in the framework of the CHANGES project, funded by the PNRR. The aim is the identification of the microclimate conditions inside the apartment at its current state of disuse, which can pave the way for guidelines to follow in the event of a future public fruition.
The monitoring activity involves the measurements of the main microclimate quantities, temperature and relative humidity, for at least thirteen months, at 3 vertical levels inside the apartment, with a total of 23 sites. The outdoor meteorological conditions are provided by the near meteorological station. Moreover, some intensive measurement activities are planned during specific days during the year (Camuffo 2019, Ferrarese et al., 2022).
The results will be fundamental to plan the future use of the King Apartment.

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Underwater decay of archaeological stone in the Mediterranean Sea

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The Mediterranean region boasts an extremely rich and diverse archaeological heritage, which includes many historical sites and landscapes now underwater. The protection and preservation of these cultural assets need as much care and planning as their land counterpart, and therefore require the knowledge of ancient materials and their interaction with the submarine environment and decay. One of the goals of the EU-funded project WATERISKULT (https://wateriskult.geoscienze.unipd.it) is to explore the causes and effects of material deterioration in underwater archaeological sites, also in view of climate change. This research addresses submerged structures and artifacts made of stone, which is used as building and decorative material in many ancient cities and port structures whose ruins rest on the Mediterranean seabed. We selected three archaeological pilot sites in western, central, and eastern Mediterranean Sea, namely Anse des Laurons in France, Baia in Italy, and Amathus in Cyprus, evidences of the Roman and Hellenistic eras. We organized diving and sampling campaigns (Fig. 1), followed by laboratory analyses aimed at investigating the state of conservation of different stone materials. We applied microscopic techniques for characterizing the material composition, biofouling, and chemical alteration, examining their surface and stratigraphic features (Fig. 2); and 3D morphometric techniques for quantifying the physical damage of the archaeological surfaces. The analytical results, combined with site-specific topographic information collected during the dives and the environmental data provided by seawater monitoring agencies, allow discussing the relationship between decay and changing stone and seawater properties in a semi-enclosed basin like the Mediterranean Sea.

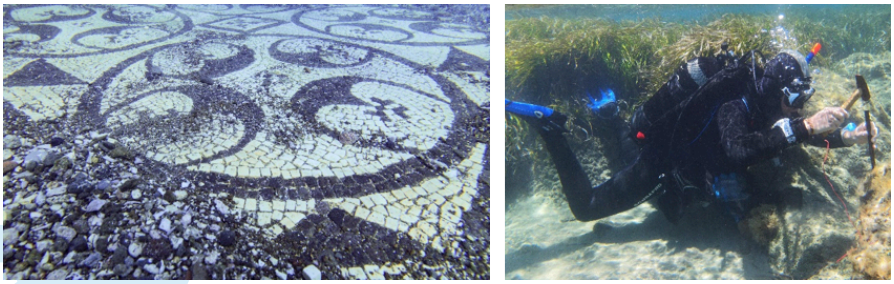

Fig. 1. Photos from the research dives arranged in the first half of 2023: stone mosaic in Baia (left) and sampling in Amathus (right).

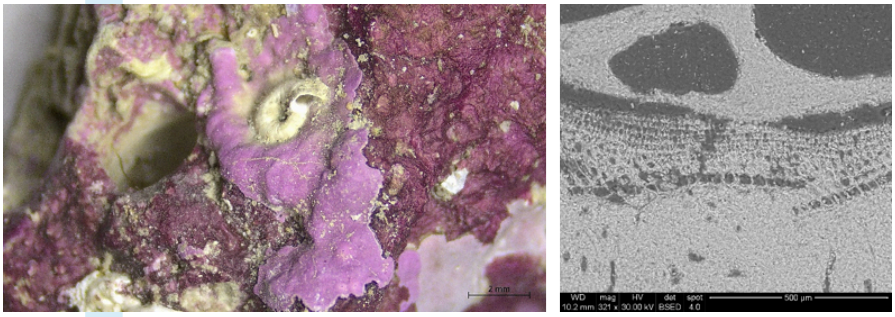

Fig. 2. Biofouling of a limestone sample from Anse des Laurons observed with the stereomicroscope (left) and SEM (right).

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The Study of Bone Remains and Fragments of Clothing from the Archaeological Monuments of the Ryazan-Oka Culture by Using a multi-analytical approach

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During the study of the monuments of the Ryazan-Oka culture (5th-6th centuries AD), along with the bone remains, a large number of fragments of clothing, in which the buried individuals were dressed, have been found.
The findings from the Undrich and Borok-2 burial grounds (Ryazan Region – Central Russia) – both bone materials and fragments of clothing – have been studied by the authors in the laboratories of the Center of Visual and Physical Anthropology of the Institute of Ethnology and Anthropology of the Russian Academy of Sciences, using long-wave ultraviolet radiation and spectrometry and chromatography methods.
The same materials have then been studied in Italy by scanning electron microscopy coupled with EDS microprobe and vibrational spectroscopic methods, such as ATR-FTIR and Raman spectroscopies.
The research made it possible to obtain new anthropological and historical information about the findings, as for example to determine the dyes, as well as to the technology used for dyeing fabrics in the 5th-6th centuries AD in Central Russia regions. About the latter, it is worth mentioning that there are no written witnesses, and this makes the work a really important step toward the understanding of uses and traditions of the ancient populations of this particular area.

Figure 1 – a) Traditional Ryazan Region coloured dress dating to the 15th-16th century AD; b) The coloured findings while being extracted with solvents; c) A row of test tubes with different colored liquids.

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Monitoring the conservation state and decay processes of works of art by means of CT reconstruction

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X-ray Computed Tomography (CT) has been borrowed from the medical field since the 1980s for the investigation and diagnostics of Cultural Heritage. Its advantages include non-invasiveness, non-destructiveness and the possibility of exploring the inner parts of an object without modifying it in any way. CT allows to create digital models to support the analysis, intervention, monitoring and enhancement processes of artworks, as proposed in the Spoke 6 guidelines in the framework of the CHANGES project (PNRR: PE5). Moreover, the analysis can be repeated endless times at different stages in the life cycle of the object, in order to monitor the evolution of the conservation state and identify an eventual decay of the constitutive material or of the structure.
Several case studies will be presented, in which the CT technique has been decisive to identify the effects of time and events occurred during the object life and influencing its state of conservation. Some examples will be offered from the study of wooden-based multi-material object, such as historical woodwind instruments where ion migration occurs inside the wood from the brass key [1]. In some cases, the CT tridimensional reconstruction can instead shed light on the damages caused by the continuative use of the object in time, such as ancient ceramic tintinnabula where the constant shaking can induce internal breaking [2].
Also, the conservation conditions and environment can influence the preservation of the artifact and CT scanning can be used to monitor the occurrence of features caused by burial soil characteristics or environmental conditions in museums: a case study on this topic has been recently investigated for ancient Roman glass found during excavations in Aquileia (Udine, Northern Italy) [3]. In this regard, CT can also be exploited for analyzing and extracting the original form and scope of artifacts embedded in soil blocks and heavily damaged from the time spent under burial, supporting restoration interventions [4].
The activity of biological factors can also be surveyed by means of CT, which can for example reveal xylophagous insects strikes in wooden objects [5,6]; repeating the analysis some time after an anoxic treatment can be an easy way to confirm the success of the procedure, verifying that no new holes have been formed inside the artwork.

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Development of Polyphenol-containing Konjac Glucomannan Hydrogels for Cleaning Historical Wood Surfaces

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This study, in collaboration with the Arvedi Laboratory of Non-Invasive Diagnostics (University of Pavia) at the Museo del Violino of Cremona, aimed at developing a new hydrogel system for the cleaning of historical wooden surfaces, with a particular focus on string musical instruments. The cleaning of historical wooden instrument is traditionally performed using free water-based solvent, which are able to remove pollutants like dirt, soil and human sweat. However, due to the hydrophilic character of wooden surfaces, the loading of water should be localized in specific areas, avoiding excessive solvent leaching and the consequent damage risks. Konjac glucomannan (KGM), a biopolymer of natural origin, was chosen for our study because of its ability to absorb and retain significant amounts of water through its high content of hydroxyl groups. Hydrogel formulation as proposed in previous studies [1], consisting of KGM crosslinked with borax (sodium tetraborate decahydrate), was optimized, and an improved crosslinking method involving boric acid and sodium hydroxide (NaOH) was introduced, resulting in uniformly crosslinked gels with enhanced moisture retention and elasticity. KGM polymer matrix was also subsequently modified with natural polyphenols (PPs), in order to provide an alternative sustainable crosslinking option alongside the introduction of antioxidant properties and protection against UV radiation [2]. Three PPs have been selected to implement the modification: softwood kraft lignin (SKL), Vitis Vinifera tannin (Vv) and tannic acid (TA). PPs have additionally been functionalized with epichlorohydrin (ECH) to allow for a covalent bonding between the PP and KGM and to induce self-polymerization of the PP for reducing leaching especially of smaller PPs from the gel to the treated surface. Various analytical techniques were employed to characterize the starting PPs and their derivatives, and to analyze the morphology and moisture properties of the hydrogels comprising the PP-modification. Based on the overall analysis results, two representative gels of the newly developed systems, i.e., the KGM gel modified with the ECH-modified Vv derivative crosslinked with borax and the KGM gel modified with Vv crosslinked with boric acid and NaOH, were chosen for cleaning trials on mock-ups replicating both Western and East Asian string instruments' surfaces, contaminated with synthetic soil and sweat mixtures. [3] The cleaning efficacy was assessed through non-invasive analyses, i.e., stereomicroscopy, X-ray fluorescence and external reflection Fourier Transform IR spectroscopy, which were performed before and after cleaning trials. Results were compared with the non-modified KGM gel crosslinked with borax and the gold standard in conservation, i.e., agar-agar-based hydrogel. The results showed that generally, KGM-based hydrogels, which were characterized by softer texture, showed better cleaning efficiency than the rigid agar-agar gels. Lastly, a direct relation between the moisture properties of the gel and its cleaning efficacy was found, with hydrogels that maintained better retention capabilities performing more effectively, like the simple KGM gel. In conclusion, in this study it was possible to develop hydrogels with tuned moisture properties from sustainable materials, suggesting future exploration of mechanical, antioxidant, and UV-protection features.

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Characterization of cosmetic pigments found in shells from Iron Age graves (SE Arabia)

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Marine and terrestrial shell remains found in archaeological excavations is considered a cultural heritage to be preserved and studied. Such findings can be analyzed applying methods of different fields, such as Earth Sciences and chemistry. In these cases, the fruitful collaboration of different areas of research also provides useful information for historians and archaeologists. Archaeology employs malacology to understand for example the evolution of the climate or to interpret functional aspect of a site.
The present study focuses on marine shells found in burial contexts from Arabia as part of the grave goods and containing greenish or blackish pigments, interpreted as eye make-up containers. Such archaeological contexts date to the Iron Age, (ca. 300-400 BC). Such artefacts are commonly found in pre-Islamic archaeological contexts from south-eastern Arabia, but in the current literature [1,2] there are indeed some, though not many, studies of their excavation and characterization.
The set of samples consists of 37 shells, both bivalves (Figure) and gastropods, the latter being much less abundant; some of them contains abundant pigments, others show only a residual patina of pigment on the surface. The pigments observed have a predominantly green color, other shells, although fewer in number, show a black-brown color.
The aim of the investigation is to study pigments found inside shells to characterize their mineralogical composition and to identify the occurrence of organic binders. The samples are investigated using non-invasive and micro-invasive techniques to establish the origin of pigments. All samples have been observed using a digital microscope (DinoLite); then SEM-EDX analyses were carried out on samples (18 specimen) as is to observe the morphology of the shell fills and to obtain information on their elemental composition. ATR/FTIR analysis was performed on micro-samples taken from each specimen, providing information on the molecular composition of pigments. Finally, Raman spectroscopy was applied, especially for those samples that did not respond satisfactorily to other diagnostic techniques.

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Calibration of neutron techniques for the analysis of conservation state and manufacturing processes of ancient bronze artifacts

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Ancient metallurgy is a research topic of strong importance in the study of artistic and technological evolution of different civilizations. Archeometallurgical studies are generally carried out by means of invasive techniques, such as metallography and scanning electron microscope observation of cross sections of the artifacts [1]. On the contrary, neutron-based techniques can provide alloy and phase composition of ancient artifacts in a totally non-destructive way. Several studies based on neutron diffraction, imaging and transmission are present in literature [2][3][4] giving interesting information on technological, degradation and conservation issues of ancient metallic artifacts. A deeper comprehension of these techniques will allow to monitor the crystal structure and the microstructural alterations over time, providing knowledge on degradation mechanisms of the objects and manufacturing techniques of past societies.
The work here presented is part of the CHNet_BRONZE project, financially supported by the Italian National Institute of Nuclear Physics (INFN). The aim of this project is to calibrate non-invasive neutron techniques for the analysis of Cu-based archaeological artifacts. Three complementary analytical methods are employed: Time-of-Flight Neutron Diffraction (ToF-ND), Neutron Resonant capture Transmission Imaging (NRTI) and Bragg-Edge Neutron Transmission imaging (BENT). These techniques are used to first analyze a reference set of samples with known structure and composition in order to optimize the experimental parameters for the analysis of historical artifacts and help in the interpretation of the collected data.
The production of the reference set and the preliminary result of the measurements carried out by means of the BENT technique will be discussed. Powder samples of pure copper, pure lead and calibrated mixtures of the two with relative concentration close to the typical values of historical alloys are realized. Additionally, solid blocks of cast copper-lead alloys with the same relative concentrations are created in order to verify possible solidification effects on isotropy and on multiphase distribution analysis. The samples are prepared with different thicknesses to verify multiple scattering and extinction effects.
BENT is a non-destructive method that provides information on composition, texture and microstructure of materials. Exploiting a 2D imaging detector, the position-dependent Bragg-edge transmission spectra can be acquired and the spatial distribution of the phases can be reconstructed. Thanks to these characteristics this technique has gained interest also in cultural heritage applications [5][6].

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Strontium isotope analysis for pottery provenance studies along the Nile: does it work?

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Provenance of raw materials is extremely important in archaeological studies because it helps to reconstruct possible trades, exchange activities and social organization within ancient societies. Specific petrographic markers and geochemical signatures of reference groups and clay deposits may provide constraints on the possible provenance of raw materials and ceramic products for coarse and fine paste pottery, respectively. Unlike human remains and other materials used in antiquity (such as obsidian, marble and glass), the provenance of pottery has been only recently investigated by isotope analysis and limited to few cases, since the isotopic signature of a potsherd depends on both that of the clay and of the possible added temper. This research explores the possibility of combining petrographic, geochemical and strontium isotope analyses on the study pottery from central-northern Sudan, to understand the social organization, the material culture and their changes occurring from the Mesolithic to the Neolithic period at regional level. Previous archaeometric analyses indicate that the pottery is here characterised by a variety of decorative motives, and produced by river clays tempered with quartz or K-feldspar. For the first ceramic type paste (quartz-tempered), the monotony of the Nubian desert sand did not allow to univocally constrain the provenance of the pottery tempered with quartz sand. On the basis of the Sr isotope ratio and for comparison with sediments collected along Nile and its tributaries, characterised by different isotope signatures in relation to the different rock types outcropping in the respective hydrographic basins, the provenance of pottery was defined. Possible cases of Sr contamination related to the use of the pottery or to post-depositional alteration were also identified and critically analysed in terms also of Sr isotope ratio.

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CIBA: the interdepartmental research center for cultural heritage at the University of Padova, where humanities and technical science meet

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The Interdepartmental Center for Research, Study, and Conservation of Archaeological, Architectural, and Historical-Artistic Heritage (Cultural Heritage) - CIBA operates in Italy within the University of Padua to bring together the resources and expertise to conduct multidisciplinary research and studies finalized to the protection and conservation of cultural heritage [1].
CIBA gathers and coordinates its research activities at the University of Padua through laboratories and scholars from various departments, both in the humanities and technical-scientific fields. This multidisciplinary cooperation aims to provide a comprehensive and integrated approach to issues related to cultural heritage, addressing both general and specific challenges.
The main areas of interest and activities of CIBA include archaeological contexts and artefacts, ancient monuments, and historic buildings, from their geometrical relief, non-invasive characterization, and structural analysis up to the characterization of raw materials and production techniques, with imaging techniques, spectroscopic portable measurements up to sample preparation and radiocarbon (14C) dating. Additionally, CIBA is committed to promoting interdisciplinary research activities, knowledge communication and integration, and resource collection to support research in specific areas of interest.
CIBA is also now the reference centre for the world-class research infrastructure (WCRI) SYCURI (Synergic strategies for cultural heritage at risk), co-founded by the University of Padua, devoted to the multidisciplinary studies related to the cultural heritage exposed to different risks.

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Tracing the evolution of street art materials: A study of contemporary mural paintings stratigraphies from Graffity Alley (Ghent, Belgium) and Galeria del Arte Urbana (Lisbon, Portugal)

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Visual art in urban contexts consists of both commissioned and often non-commissioned street art. The ephemeral character, free access, and exposure to the environment and anthropic actions, make public paintings vulnerable to removal, vandalism, and degradation. Urban art is at present widely recognized by art history and by the wide society as an important part of contemporary cultural heritage, and a historical perspective on the evolution of materials and techniques is highly needed to gain a better knowledge of this form of art and to contribute to sustainable preservation strategies.
The synthetic paint materials used by street artists in the last decades, such as spray paints, have undergone a fast evolution in their compositions. This aspect is related both to the implementation of new health regulations, leading to substances being banned from commercial use, and to the continuous improvement and evolution of commercial formulations. Targeted analytical experiments have been carried out to contribute to a comprehensive understanding of the chemical composition of paint materials used by street artists over the past decades, by studying samples which contain stratigraphies representing over 20 years of paint materials.
Two case studies of great importance are presented: the Graffiti Alley (Ghent, Belgium) and Galeria del Arte Urbana (Lisbon, Portugal). These two locations are characterized by up to 25 years of repaintings on the same paint supports, giving a unique point of view on the evolution of the materials in a specific and well-defined time segment. The use of traditional analytical approaches on these samples did not provide an accurate evaluation of the distribution of the materials due to the thinness of the paint layers.
In the presented study carried out at IPANEMA, a state-of-the-art European facility renowned for its expertise in multiscale and multispectral approaches in heritage science, we conducted Multi-spectral Luminescence Microscope (MSLM), Mid-infrared Hyperspectral Imaging (MIRHSI), and combined Micro-XRF/XRD analysis to obtain an exhaustive spatial resolution apt to study the stratigraphy of both the organic and inorganic materials. Moreover, SEM-EDX was carried out in Pisa to provide the elemental composition of the different paint layers. Subsection of the stratigraphies were also analysed by pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS) to obtain a more detailed molecular composition of the samples.
By applying this multiscale and multispectral approach combined with the micro destructive Py-GC-MS, we were able to obtain detailed chemical information about the different paint layers in the cross-sections. This allowed us to gain insight into the paint binder stratigraphy, differentiating between various classes of organic polymers, such as alkyds, acrylics, and nitrocellulose resins, present in each layer, and the evolution of their use in formulation during time. Additionally, the analysis provided valuable information about the inorganic content of the paint layers, revealing not only the presence of pigments and inorganic fillers. While some elements such as calcium and titanium were homogenously present in all the layers, specific metal were detectable only in the oldest paint layers. In particular, the presence of lead was highlighted only in the oldest layers.
A detailed microscale element distribution can be crucial in the comparison with relevant literature (patents, European health regulations, peer-reviewed journals, etc.) to tentatively date the different paint layers.
The research has been carried out in the framework of the Italian PRIN2020 project SuPerStAr - Sustainable Preservation Strategies for Street Art.

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Painted architectural terracottas at the Northern Sanctuary of Cuma: preliminary results on the production technology from the Archaic to the Hellenistic period

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Research carried out by the Centre Jean Bérard (CJB) next to the northern fortifications of the ancient city of Cuma unveiled a complex of structures associated with a consecrated zone, exhibiting varying historical phases. Excavations unearthed votive pits, likely marking the formal closing and abandonment rituals of the site, in which painted architectural terracottas, dated back from the 6th to the 4th century BCE, were discovered.
The presence of both archaic and Hellenistic elements within the pits suggests an intentional effort to safeguard a collection of elements of edifices to which they belonged, effectively preserving the memory of this place of worship throughout the historical evolution of the site.
If a clear typological evolution is discernible in the female-headed antefixes, it becomes of particular interest to investigate the potential technological evolution in their production process; the researches, in fact, are looking for deepening this aspects, investigating the raw materials composition (clayey matrix and temper), firing technologies, and decorative techniques.
In this perspective, a meticulous selection of 15 well-crafted specimens was carried out.
A multi-analytical, preliminary study was focuses on pictorial layer, in terms of type of pigments and application technique. Fiber Optic Reflectance Spectroscopy (FORS), Fourier Transform Infrared (FTIR) and X-ray Fluorescence (XRF) permitted the identification of pigments, consisting in red ochre, carbon black and white clays. The in-depth assessment and investigation of application technique will be carried out by observations and micro-analyses via Scanning Electron Microscopy (SEM-EDS).
Regarding the terracotta pastes, preliminarily observation via Digital Microscope (DM) highlighted the structural and textural features, defined in more detail through a minero-petrographic study on thin sections via Polarized Light Microscopy (PLM). Interestingly, the ceramic pastes of both chronological phases showed similar minero-petrographic features.
Except for some sample showing finer ceramic pastes, the analysed specimens generally have coarse texture, given by the presence of volcanic temper scattered in the clayey matrix, composed of scoriae, pumices, and crystals of feldspars, clinopyroxene and biotite, compositionally consistent with mineralogical assemblage of Phlegraean volcanic products, suggesting the local production of terracotta samples in the investigated time span.
Further analyses (XRD, SEM-EDS, TGA) will elucidate on the ceramic process they experienced, allowing to assess the level of technological refinement.

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Discovering the history of the wooden scene of the Teatro Olimpico in Vicenza: a multidisciplinary approach based on scientific analyses and HBIM

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The wooden perspective scenes conceived and realised by Vincenzo Scamozzi in 1585 to complete the Teatro Olimpico, i.e., the oldest stable and indoor theatre of the modern era designed by Andrea Palladio in 1580, are part of the artistic heritage of the city of Vicenza and the Italian Renaissance. However, their history is not yet fully revealed. Over the past decades various campaigns of investigation have dealt with this masterpiece, mostly in order to verify its state of conservation, although the sequence of the interventions that the theatre has undergone, the nature of the original material used and eventual retouching and repainting suffered by restoration interventions, still remains unclear. In this research with a strong interdisciplinary connotation, the photogrammetric survey, the study of archival resources, the investigation of materials and execution techniques, and the assessment of the state of conservation were closely interconnected tools with the aim of answering these questions.
More in detail, in order to fill these gaps and to understand the conservation status of the theatre for the design of an architectural restoration project, a broad analytical approach was applied on micro-samples (light microscopy, XRPD, SEM-EDX, μFTIR and μRaman). The selection of the samples was carefully guided by the HBIM model, that provided a critical evaluation of archival knowledge, which not only indicated the less investigated areas but also provided a cognitive starting point on which the new investigation could be structured. The primary steps of the research were therefore to obtain a characterisation of the pigments, binders and microstructures of the pictorial layers, as well as to assess their microstratigraphic sequence, the state of conservation of the scenes and the deterioration processes involved.
The results obtained revealed a certain variability in the production techniques of the paintings, which often present a microstratigraphic sequence that deviates from the classic sequence corresponding to the traditional painting technique on 16th-century wooden panels (Lalli and Innocenti 2015). Furthermore, the identification of certain pigments and binders that can be used as temporal markers made it possible to unequivocally establish, for the first time, that a significant part of the scenes designed by Scamozzi was heavily repainted after World War II.
All these analytical results together with architectonical survey, archival sources and reports of investigations carried out on the cultural heritage were related through the HBIM model. This approach proved to be a powerful analytical method to establish relationships through all the linked information for the benefit of the Teatro Olimpico and a better consciousness in its future maintenance and conservation.

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The importance of microclimate monitoring as a basis for preventive conservation: a case study over time

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Microclimate monitoring is a fundamental practice for the preventive conservation of cultural heritage preserved in indoor spaces, such as museums, libraries, archives, etc. It consists in the continuous measurement and recording of environmental parameters which influence the deterioration of materials, such as temperature, relative humidity, illuminance, atmospheric pollutants concentration and so on. Among the materials constituting artefacts and the surrounding environment ("microclimate") close balance relationships are established depending on intrinsic (artefacts composition, chemical-physical properties of their constituent materials, exhibition space characteristics) and extrinsic factors (conservation history, presence of visitors, methods of use/consultation, accidental mechanical actions, influence of the external climate) making the control of the microclimate environment complex.
Environmental parameters should therefore be maintained within an appropriate range according to the characteristics and needs of the artefacts, also taking into account the historic climate to which they have been acclimatized. Microclimate monitoring allows to identify risk conditions and to plan preventive conservation strategy to guarantee the long-term conservation of cultural heritage in indoor spaces [1].
Over the last 30 years, the practice of monitoring the environmental parameters indoors has become increasingly frequent and conservation protocols adopted by museums, libraries and archives are based on it. Contrary to a practice in force until about ten years ago, which aimed to establish for each object, based on the material that constitutes it, absolute and optimal values of environmental parameters, the approach has now changed. In order to achieve the objective of conservation of the artefacts, it is necessary to try to keep the values of the parameters stable. This may be considered the best compromise between the different factors that come into play, such as the conservation of the objects, public use and the comfort of both visitors and of the occupants.
A case study of microclimate monitoring was performed in the Alessandrina Public Library, housed in the Rectorate Building in the Campus of the Sapienza University of Rome. In this site the monitoring was carried out in different periods 20 years apart, and here is presented to draw attention to the importance of microclimate monitoring as a basis for preventive conservation, evaluation of the state of conservation and expected lifetime of artefacts.
The two experiences conducted, due to a general need of specific technical and scientific knowledge aimed at guaranteeing adequate display conditions for books, made it possible to assess the indoor conservation condition in spaces intended to the custody and the consultation of the books and to the staff working activities, and to develop the control procedures for environmental parameters aimed at the correct conservation of the artefacts and management of the library [2-5].

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A liquid-anode X-ray source multi-technique setup for the non-invasive compositional and conservation state characterization of materials

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In the last decades, a novel technology for X-ray sources based on the use of a liquid anode has been developed, in order to increase the maximum achievable brilliance by at least one order of magnitude compared to conventional microfocus sources [Hemberg et al., 2003].
With this innovative equipment, a High-Brilliance X-ray laboratory (HiBriX Lab) is presently under development at the University of Torino, hosted at the NIS inter-departmental Centre. It was designed by integrating different detectors and focusing optics to represent a unique laboratory in Italy and with a handful of comparable examples in the world. The aim is to cover several applications such as:
- material characterization via μXRD and μXRF maps;
- investigation of detector performances in terms of charge collection efficiency or as a function of damage effects;
- single cell level radiobiology;
- X-ray imaging (2D radiography and 3D computed tomography - CT) of objects having a wide size range.
Procurement of the different components has been almost completed and their integration is underway, also by means of the development of specifically dedicated software for system control. To date, concerning the microfocused branch of the lab, a minimum spot size of about 10 microns has been achieved by means of a set of twin paraboloidal mirrors, and a maximum flux density of 2.7×1010 ph s-1 mm-2 has been obtained with a polycapillary optics system specifically delivered by INFN X-lab in Frascati. On the other side, where a 30° cone beam is available, a versatile X-ray imaging setup is installed, which allows the acquisition of radiographs and tomographic scans of very different kinds of samples: objects of dimensions in the sub-mm to few tenths of cm range, with wide variability in atomic number and density values.
Using a combination of these techniques, it will be possible to study both the composition, the conservation state and the internal structure of different samples of archaeometric interest.

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In situ test for evaluation of protective film degradation, a study using thermographic spilling drop test

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Degradation of stone materials is a significant and ongoing issue affecting cultural heritage. Paraloid B72 is an artificial and acrylic resin that has found wide application in past decades both in the protection of stone and artistic surfaces such as wall paintings because of its property to be transparent and chromatically stable. A study was conducted in the DIART laboratory of the Physics Department of the University of Milan to study the potential use of the Spilling Drop Test (SDT) to investigate modifications in water repelling properties following photochemical degradation. The approach to study the degradation of Paraloid B72 is investigated using SDT, an innovative and non-invasive thermographic technique that allows to investigate the hygroscopic behavior of sample; it evaluates the absorption of a drop of water with a constant volume.
In the experimental trial, limestone specimens were partially treated with Paraloid B72 and subsequently exposed to photodegradation using a solar box, a chamber that simulates prolonged and continuous exposure, equipped with sources that simulate the spectrum of sunlight through the use LED panels and two UV lamps.

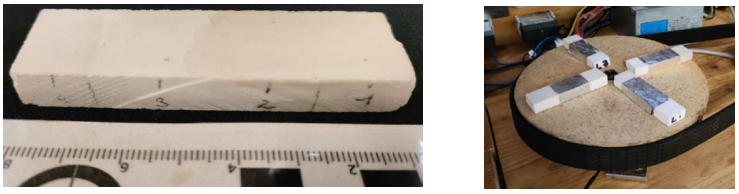

Figure 1: Example of sample division (a) and four limestone in a solar box (b)

All samples are divided into four parts and half of them are covered by Paraloid B72 (points 1 and 2, figure 1a) and the others instead are in a natural state (figure 1a).
Spilling drop tests were carried out at 7 and 14 days and for comparison on untreated points of the stones and not exposed to radiation (figure 1b). Interesting in these preliminary results is that after a total of 21 days of degradation Paraloid seems to lose the properly to protect and therefore to be not hydrophobic.
To have a broad overview of protective film degradation, this study is also compared with a recent project in which limestone specimens are covered by cyanobacteria biofilms in a laboratory experiment. Aged biofilms may play a protective role with regard to the stone substrate by decreasing capillary absorption and drying rates while enhancing the surface's hydrophobicity.

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The challenge of 19th century iron gall inks: from their detection to the disclosing of ageing mechanisms by an ultrasensitive HPLC-DAD-HRMS method

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Several manuscripts and drawings of our historical and artistic heritage have been produced with iron gall inks. Inks are liquid, semi liquid or solid materials obtained in the past from many different colored extracts of plant and animal origin. From an analytical point of view, the identification of inks in drawings or manuscripts is highly challenging. Many conservation issues arise from the several empirical formulations used over the centuries showing variations in the ink composition, and from the interactions between the ink and the paper support. To date, most research has been aimed at paper preservation, due to the degradation state and the fading of the text detected in artifacts, performed by non-destructive analytical methods that provide only limited information on ageing process trends and minor components representative of iron gall ink recipes. Thus, analytical investigation in heritage science requires novel sensitive protocols for the analysis of writing inks.
In this study, we developed an analytical procedure based on High Performance Liquid Chromatography coupled with Diode Array Detector and High Resolution Mass Spectrometry (HPLC-DAD-HRMS) for the analysis of iron gall inks. Three historical recipes for iron gall inks (modern gall ink, alizarine ink, Reid ink), reproduced in the laboratory according to 19th-20th century historical recipes, and differing for the preparation method and additives, were characterized in terms of major and minor compounds. Several molecular markers of iron gall inks have been identified (e.g. gallic acid, ellagic acid, digallic acid and poly-galloyl glucose species) widening the databases available for their identification in documents and artworks. Different chromatographic profiles were revealed depending on the historical ink recipes adopted. Furthermore, by performing ageing tests on reference materials in different conditions (natural and artificial ageing), two different degradation mechanisms have been observed: hydrolysis of poly-galloyl glucose species and auto-oxidation of gallic acid. The comparison of the results highlighted different ageing effects in relation to the ink recipe. The role played by relative humidity on ageing processes was also evaluated, allowing us to determine different intermediates depending on the environmental conditions. Finally, the analytical method developed was successfully applied for the study of samples from historical manuscripts, granting access to the complete molecular profile of an iron gall ink with just 15 µg of sample, improving the approaches reported so far in the literature. This study demonstrates as ultra-sensitive liquid chromatographic-mass spectrometric methods can be valuable options to be introduced in the array of analytical tools for the analysis of manuscripts and drawings on micro samples.

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Microclimatic and entomological monitoring for paper and parchment documents conservation: the case study of Diocesan Archives, Brixen/Bressanone

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In the realm of preventive conservation, the examination of temperature (T) and relative humidity (RH) data, as well as entomological monitoring, enables a comprehensive evaluation of the potential risks to the preservation of valuable heritage collections in dedicated spaces.
The present study aims to investigate the degradation of paper and parchment induced by the microclimate and biological pests. This study has been undertaken to support the Diocesan Museum and Archives of Brixen/Bressanone in the preliminary assessment of the change of use of a 60-square-meters-wide ground- floor storage space for paper and parchment documents into an exhibition room, and the transfer of the materials to the attic.
The Diocesan Museum and Archives are located within the Episcopal Palace Hofburg of Brixen/Bressanone (Lat. 46.4 N and Long. 11.4 E, 559 m a.m.s.l.). Originally constructed in the 13th century as a fortified castle with a moat, it underwent modifications and expansions, culminating in its current Baroque appearance in 1701.
To investigate the preservation of paper and parchment assets stored within the archives and the potential degradation they might undergo following relocation to the attic, two types of monitoring have been implemented: microclimatic monitoring using thermo-hygrometers to measure T and RH, and entomological monitoring using traps without attractants. Both microclimatological and entomological monitoring started in 2022 in both spaces (ground-floor storage and attic) and are still ongoing. Furthermore, data from the Brixen/Bressanone weather station, provided by Eurac Research's Meteo Browser, have been employed to evaluate potential environmental influences, with a particular focus on conditions in the attic.
The latest literature has highlighted the importance of maintaining paper and parchment supports within specific safe temperature and relative humidity ranges for proper conservation. The national and international standards for the conservation of cultural heritage, applied worldwide, often do not observe these ranges [1]. Moreover, the ranges proposed by various associations often diverge and occasionally conflict with one another. Disregarding these up-to-date safety ranges could lead to chemical-physical deterioration in paper, structural and mechanical distortion in parchment [2,3], and making both supports susceptible to pest attacks. Through the analysis of microclimate parameters, namely temperature and relative humidity, as well as biochemical factors, notably pH and cellulose degree of polymerisation of the paper, it has been possible to determine the Expected Lifetime (EF) [1] of the documents stored in the archive and what would transpire after the relocation of these assets to the attic.
Entomological monitoring has led to the discovery of insects such as Psocoptera, Dermestidae, and Ctenolepisma longicaudatum. The latter is a subject of study as it infests numerous museums in Germany and Austria and is associated with the transportation of fungal spores [4,5].
The results of the preliminary study conducted within the Diocesan Archive of Brixen/Bressanone have revealed the noticeable difference in microclimatic characteristics between the current archival space and the attic. These findings offer the potential to formulate a preventive conservation plan aimed at mitigating the risks posed by infesting pests and preventing chemical-physical and mechanical-structural degradation of paper and parchment supports.

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Diagnostic analysis of the three paintings by Artemisia Gentileschi created for the Cathedral of San Procolo in Pozzuoli (Naples)

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During her stay in Naples in the 1630s, Artemisia Gentileschi was commissioned to paint three paintings for the choir of the Cathedral of St. Procolo in Pozzuoli (Naples, Italy) [1]. She created the Adoration of the Magi, St. Gennaro in the Pozzuoli amphitheater, and St. Procolo and St. Nicea, which the bishop mentioned as evidence of their completion in the Relatio ad limina of 1640. The three paintings have undergone various vicissitudes over the centuries, such as a poor state of conservation, restorations before and after the fire that broke out in the Cathedral in 1964. This event damaged the pictorial film, causing alterations and the loss of parts of the preparation and the color.
Diagnostic analysis of the three paintings was carried out using multispectral imaging at different wavelengths (VIS, IR and UV) [2] and X-ray fluorescence (XRF) [3]. The results are integrated and compared with the artistic-scientific literature on Artemisia's activity, especially in Naples after 1630, in order to better understand the pictorial technique, the color palette and the materials used in the restoration, and also to highlight the possible influence of some of the artists working in Naples at that time.
UVF (ultraviolet-induced visible fluorescence) images highlight retouches, while IR images show dark outlines and pictorial alterations (pentimenti).
The XRF technique, together with IRFC (infrared false color) imaging, suggested the pigments and materials used for the paint and preparatory layers. Preliminary results point to pigments such as smalt, Naples yellow, vermilion and ultramarine, as well as copper or lead white for the various shades of blue. The study carried out provides useful information for the study of Artemisia's paintings, which will also be useful for future research.

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Establishing an archaeological experimental collection: Exploring Ground Stone Tools analysis through different settings

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Replicative experiments are a well-established procedure in archaeological methods. However, the setup and the aspects to emphasize continue to divide the archaeological community. In the study of ground stone tools (GSTs), replicative experiments can be employed to explore the formation and evolution of damage patterns during their use in transformative tasks, such as processing plant organs. These experiments also allow us to observe the motion and force that contribute to the formation of use-wear.
When designing an experimental ground stone collection, various approaches can be utilized. In this research, three main approaches were employed, each with a different degree of control over the variables that can occur. Initially, we leveraged the finality and settings of actualistic experiments to assess the tools' applicability, efficiency, and durability in specific tasks by attempting to replicate ancient technology and gestures, all conducted in a natural environment. In a separate instance, a more stringent control over possible contamination was applied by relocating the experiment to a laboratory setting (Sorrentino et al. 2023a). Subsequently, the third approach involved the use of mathematical and mechanical models to simulate the tasks and damage patterns resulting from the use of these instruments (Marulli et al. 2023). A highly accurate digital twin (DT) model of the lithic tools has been developed based on precise 3D photogrammetric data collection, for which a purpose-built environmental setting for data collection was created (Sorrentino et al. 2023b). In general terms a DT model can be defined as a dynamic computer representation of a physical object that incorporates its geometric, mechanical, and structural attributes. This model simulates how the virtual representation responds to external influences and stimuli. The results from the various approaches are compared to highlight the differing degrees of variable control and their deviation from potential real scenarios and possible factors, while also considering the pros and cons of the methods.

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Ultrasonic seismic waves to characterize calcarenitic stones

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The Southern part of the Apulia region (Southern Italy) shows a dense cultural built heritage. The building materials consist of soft calcarenitic stones. These materials present significant conservation problems related to their low durability. Knowledge of the intrinsic physical-mechanical characteristics of these materials is crucial to evaluate their performance properties and susceptibility to degradation. Methodologies for evaluating the structure of historic masonry Cultural Heritage sites have been developed and improved. Geophysical methodologies have been implemented, tested and validated as diagnostic and monitoring tools for works of art or historical monuments (Calia et al., 20013; Dunham, 1962; Leucci and De Giorgi, 2006; Leucci et al. 2011; Leucci et al., 2012).
Firstly, they are non-destructive methodologies and furthermore provide an image of the internal structure of the investigated medium in relation to a particular physical parameter.
In this paper, non-destructive testing (NDT) was performed in laboratory and in situ using high-frequency seismic waves (figures 2 and 1 respectively).
A brief description of the methodology used is presented and the main processing steps are illustrated. The relationship between i) frequency and P and S wave propagation velocity; ii) density and P and S wave propagation velocity; iii) volumetric water content and P and S wave propagation velocity; iv) compressive strength and P and S wave propagation velocity, has been studied on some porous Apulian calcarenites known as Pietra Leccese, Pietra di Ostuni and calcareous Tufo delle Murge. P- and S-wave velocity measurements have been performed on cubic samples, under natural conditions (e.g., without applying external pressure to the samples), using the transmission method.

Figure 1. NDT performed in laboratory.

Figure 2. NDT performed in situ.

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Non invasive investigation of frescos of the complex St. Mary of the Cave in Marsala (Trapani, Italy)

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The rock-cut complex of Mary of the Cave in Marsala (TP), inside the Punic necropolis of Lilybaeum, was used until the IV century A.D. The stratigraphic reading suggests an original extent of frescoes on several walls, especially in relation to the use as an italo-greek coenobium (12th-13th century); the frescoes of the rooms 1 and 2 are partially still visible today [1-4].
The frescoes in fact are in a very poor conservation state. A diffuse inconsistent and coherent surface deposit is present. The chromatic alteration is mainly due to a whitening of the surface (salt efflorescence) and to a moisture stains. The most commonly degradation is the presence of concreted salt efflorescence. These are a consequence of the crystallization pressure of salts and accentuated by rising capillary moisture, condensation. The presence of sub-efflorescence has caused uplifts and swelling resulting in the detachment and loss of constituent material and pictorial film, for this reason, the paintings are extremely patchy. The causes of the degradation can be related primarily to the presence of water in various forms: capillary rise, infiltration, and condensation. Cause of the detachment, on some frescoes, the superimposition of different pictorial layers, which can be associated with different historical phases, can be clearly distinguished.
In order to find a correlation of the conservation state with the micro-environmental conditions, N. 2 dataloggers were placed in the room 1 to monitor in real time the values of temperature and humidity. In addition, the wall paintings of room 1 and 2, depicting "The Theory of Saints" (identified as fresco F), "The Martyrdom of St. Stephen" (identified as fresco G), the niche depicting " The Madonna Odigitria" (identified as fresco H) and a fragment of a fresco in room 2 with unknown subject (identified as fresco I) were investigated trough the non-invasive techniques X-ray Fluorescence (XRF) and Fiber optical Reflectance Spectroscopy (FORS) and, Infrared and Raman Spectroscopy in order to identify the pigments.
First results showed not only the nature of pigments and the executive techniques but also the alteration of same pigments and the presence of superimposed fresco.
These findings get light on the evolution of the site and in details of frescoes in the time, as consequence of the different use of the complex and of the changes in the environmental conditions.

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Diagnostic investigation for the characterization of artificial stone materials (mortars, plasters, bricks) from the "Teatro di Marcello" (Rome)

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The study illustrates the results of the minero-petrographic and chemical investigations of artificial stone materials (mortars, plasters, bricks) taken from the Teatro di Marcello (Rome).
Optical microscopy (OM) was aimed at collecting information relating to the textural and mineralogical characteristics of the samples (identification of the main minerals constituting the aggregate, its grain size and shape, and the evaluation of the binder/aggregate ratio). The data will also support technological assessments through the definition of the used raw materials for the manufacture of the mortars/plasters. Furthermore, through SEM/EDS investigations, data relating to the chemical composition of the samples were acquired, useful for estimating their hydraulicity index, HI (Boynton, 1980). Secondary phenomena of alteration and degradation were also evaluated through the documentation of fractures and primary/secondary porosity. The diagnostic plan allowed us to obtain interesting information on the mortars used in the Teatro di Marcello together with their probable production technology. In particular, the raw materials are quite homogeneous, thus confirming the typical "recipe" used in Roman times to create natural hydraulic mortars by the addition of pozzolanic/volcanic material to the aerial lime. The volcanic component of the aggregate is compatible with that of ultrapotassic products of the Roman Magmatic Province, likely with the volcanic district of the Alban Hills (Peccerillo, 2005).

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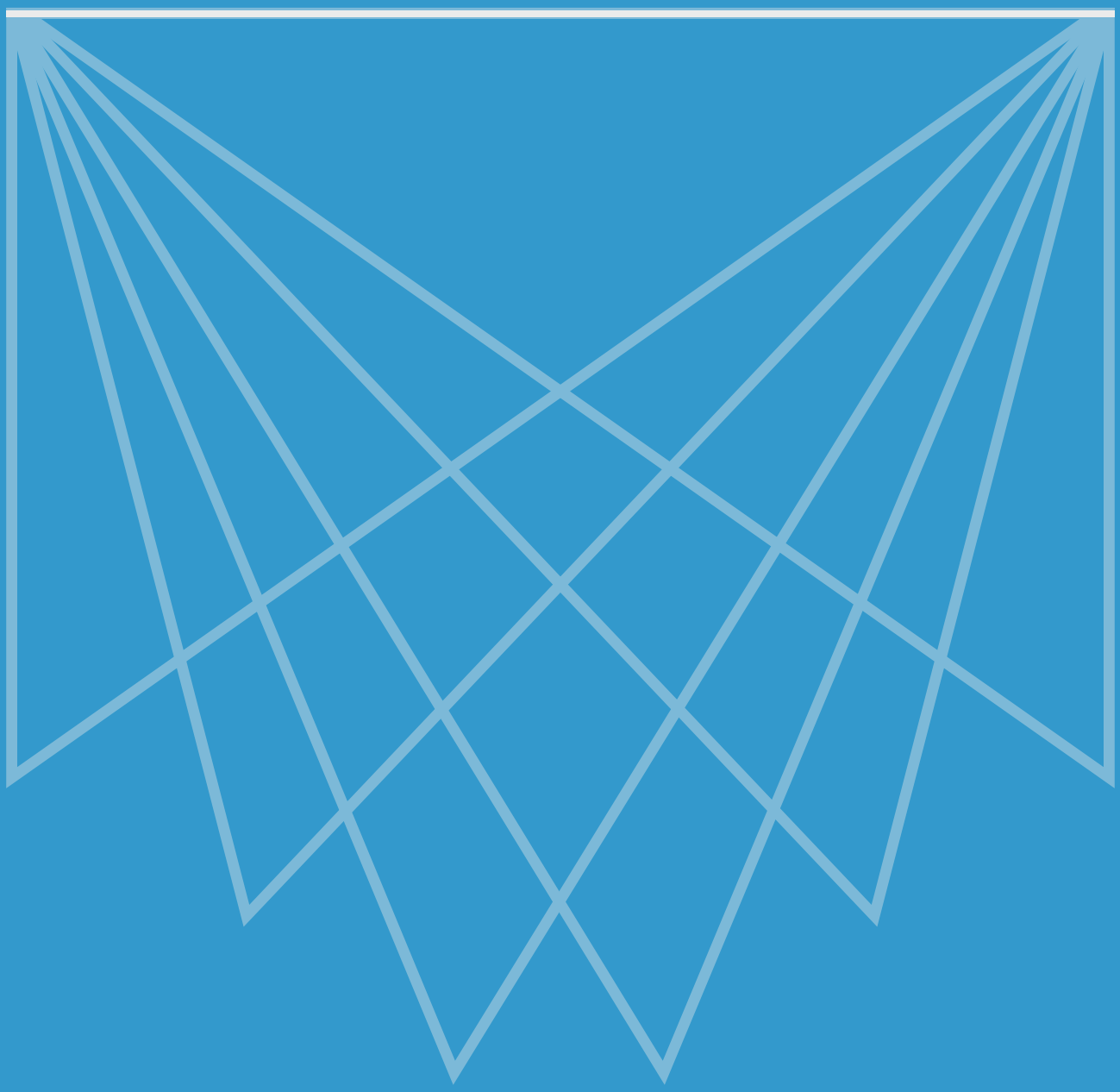

RICOSTRUZIONI STORICHE E STRATIGRAFIA

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The Thousand Arrows of Motya, a reference point for the Bronze Age in the Mediterranean

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Mozia Island, located in the central-western Mediterranean basin, is an intriguing archaeological site offering a picture on a well defined time, the 342 BC when Dionysius I of Syracuse attacked the island destroying everything and Motya disappears from documented history. The war event provides a huge number of finds stuck under the rubble of structural collapses, including a large number of copper based arrows of 4 different types [1,2].
This study focuses on a comprehensive investigation of these artifacts through a chemico-physical analysis and the implementation of chemometric approaches to discern similarities and differences based on their composition and metallurgy defining the characteristic of metal for weapon used by the two civilities. The research methodology involves a multi-step process, starting with the acquisition of a sizable number of Greek and Punic arrows from Mozia. Subsequently, X-ray fluorescence (XRF) as non-destructive and non invasive technique is employed to obtain detailed information about the elemental composition of each arrow. Chemometric techniques, including principal component analysis (PCA) and hierarchical clustering, are then applied to the analytical data. These methods assist in the classification and grouping of the arrows based on similarities and differences in their composition and metallurgical characteristics. By identifying clusters of arrows with shared traits, it becomes possible to draw conclusions regarding their origin, production techniques, and potential cultural influences.
This approach serves as a valuable framework for future archaeological studies involving large and diverse sets of metal artifacts, enabling a more comprehensive analysis of their historical context and significance. Moreover, the significance of this research extends beyond the mere classification and historical understanding of the Greek and Punic arrows from Motya. As these arrows are believed to originate from the historical event of the war in 342 BC, the accuracy in dating and attributing similar objects to this specific period becomes paramount. The wealth of data and insights gained from this study serves as a crucial reference point for evaluating the age of other bronze arrowheads and artifacts of similar composition. This research thus not only enriches our understanding of the past but also provides an essential resource for future investigations seeking to date and link objects to this historically significant period in the ancient Mediterranean.

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New insights on the Western production of Ionian cups type B2

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This contribution focuses on the minero-petrographic and chemical characterization of Ionian cups type B2 found in Gela (southern Sicily). The Ionian cups are a ceramic class widely spread in the Mediterranean basin from the second half of the VII century BC and even more throughout the VI century and a little beyond. All types (A1, A2, B1, B2 and B3) were initially considered to be imported from Eastern Greece. However, Vallet and Villard, already in 1955, doubted about their exclusive Greek origin due to numerous attestations of B2 cups in the West. Afterwards, the restitution of waste from kilns related to B2 cups from excavations in southern Italy and Sicily (Van Compernolle, 2000) confirmed the early intuition of Vallet and Villard that the type was widely produced also in the West, in the colonies and in the indigenous hinterland. The “area of the Strait” had already been strongly suggested for the production of B2 cups, on the basis of quantitative considerations (Vallet & Villard, 1955). In addition, during an intervention of urban archaeology in Messina an ancient dump was identified, from which several forms of fine band-decorated ceramics dating to the archaic/ late-archaic period came out. Among these, the Ionian cups of type B2, with some variations, were attested with numerical relevance (Tigano, 1999). Three overfired fragments were also found, probably waste from the kiln, which most likely were ascribed to local production (Barone et al., 2002). Other investigations were carried out later on specimens of B2 cups coming, instead, from archaeological sites of central-eastern Sicily (Belfiore et al., 2010; Barone et al., 2011). They have provided scientific evidence of different productions, although most of the samples examined seemed to belong to a unique group whose precise provenance attribution was not possible, but whose characteristics were compatible with those attested in the samples from Messina. To verify the importance and commercial potential of this group, it was decided to ascertain its diffusion starting from Gela, an archaeological site located along the southern coast of Sicily, where cups of type B2 and their variants exceed 600 specimens.
For this study, 30 samples have been selected for the mineralogical-petrographic and geochemical investigations. The aims of archaeometric analysis was threefold: i) verifying whether the macroscopic resemblance of the highest number of cups attested in Gela could actually be attributed to a single site or production area; ii) assessing if the examined cups could be assigned to a local production, through comparison with a fairly substantial database on the geloa ceramic production, even fine (Aquilia et al., 2012; Ingoglia et al., 2018); iii) verifying whether they, instead, can be considered imports in Gela from the “area of the Strait”, since to the naked eye it appears evident the affinity of Ionian cups from Gela with those from the Peloritani area.

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Study and radiocarbon dating of the Romanesque architecture in the church of Santa Maria Assunta in Sorengo (Ticino, Switzerland)

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The church of Santa Maria Assunta in Sorengo preserves one of the oldest examples of Romanesque mural painting in the Canton Ticino. The present building is the result of several transformations. The original plan, which probably dates back to the beginning of the 11th century, consists of a single nave church with an oriented apse and a gabled roof, whose interior was decorated with at least two cycles of paintings, of which large fragments from the 11th and 13th centuries remains. The arrival of the Friars Minor of St. Francis in the 16th century created the conditions for a radical transformation: while the new convent was being built, the church was enlarged and being transformed from a simple hall into a monastic church with new cross vaults. The two Romanesque cycles were partly destroyed, partly hidden by the vaults, and then forgotten. They were rediscovered in 1938 by the restorer Emilio Ferrazzini. In 1979 the church was the subject of an archaeological campaign, thanks to which it was possible to reconstruct the fundamental phases of its architectural evolution. The research project "Wall painting cycles of the 11th-13th centuries in the Canton of Ticino" - funded by the Beate und Hans Peter Autenrieth Stiftung - aims to study the architecture of the original church and the Romanesque paintings through the analysis of archival documentation, direct observation and the use of instrumental techniques. A study site, carried out by the second-year students of the Conservation and Restoration course, focused on the two Romanesque painting cycles in order to analyse their execution technique and to assess their condition.
With regard to the architecture, the archaeological records were analysed in parallel with a detailed study of the material evidence. The surviving wall textures of the Romanesque church were identified, their construction characteristics and materials defined, and their relative chronology clarified through stratigraphic analysis. The analysis of the masonry allowed the identification of a wooden corbel belonging to the first construction of the church, the dating of which would therefore allow the dating of the church itself. Two stratigraphic opening on the ground floor made it possible to identify the remains of the thick walls of the ancient bell tower, developing a previous hypothesis that ignored its precise location.
Samples of mortar and plaster were collected, prepared and analysed with a polarised light microscope and a scanning electron microscope. By combining the analysis of the wall textures with the study of the mortar and plaster, it was possible to distinguish the phases of construction according to the materials and their use. In the first phase, in the 11th century, a lime plaster was applied shortly after the construction of the masonry. Both the mortar and the plaster were prepared with a Mg-lime, from a dolomitic raw material with well calibrated firing procedures and uniform temperatures. The painted plaster of the first cycle, found on the former triumphal arch (now the counter-façade) and in the attic, may date from the same period. The painting represents the Annunciation and Saints and is of incredible quality: simple, with thick strokes rich in pigments, that creates refined volumes inscribed in architecture of Byzantine and Oriental flavour. The second cycle, from the 13th century, is in the attic of the nave and probably represents the Last Supper. Here the plaster is different, with a high percentage of binder and washed, well-sorted sand. The painting layer is very thin and applied directly to the plaster. Further research is underway to verify the dating of this early Romanesque phase, using absolute radiocarbon dating of the mortars and of wooden corbels inserted into the older masonry.

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OSL dating of alluvial sediments at Cahuachi (Southern Peru) not reconciling the paradigm of catastrophic mega-floods for the decline and end of the Nasca Theocracy

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The Nasca Theocracy had in Cahuachi (Southern Peru) and its various temple areas the most important ceremonial site (Lasaponara et al., 2016) whose continuity of use as religious centre over the millennia was highlighted by the discovery of much earlier architectural structures (1700 BCE) than those in the urban context dated in the Paracas-Nasca period (400 BCE-550 CE). The mainstream explanation for the decline and demise of Cahuachi implies the damage and burial of such a ceremonial centre as a consequence of two catastrophic mega-floods, which occurred, respectively, around 600 CE and 1000 CE (Grodzicki, 1990) probably linked to the El Niño-Southern Oscillation (ENSO) events. However, recent geological results achieved at Cahuachi (Delle Rose et al 2016; Delle Rose et al. 2019) hypothesized that the conglomerate at the top of the succession at Piramide Sur and Gran Piramide was not deposited by catastrophic ENSO floods as hypothesized by Grodzicki (1990). During April 2019 a geological survey was carried out at Cahuachi mainly addressed to sample and date by Optical Stimulated Luminescence (OSL) the alluvial sediments supposed to be those of the first millennium CE, thought to be responsible for the decline of the ceremonial site. The OSL analyses were performed using the SAR protocol (Wintle and Murray, 2006) applied on quartz grains (180-250 μm) extracted from the samples. The range of OSL dating were set from different hypothesized weather humidity condition of the samples (dry up to 70% wet). One sandy sample (NZ1a) adjacent to a wall of the Gran Piramide temple has a minimum OSL age of 23-25 ky BP whereas other sandy levels (NZ11) sampled close to the adobe construction along the same type-section studied and considered of paramount importance by Grodzicki (1990) to prove the catastrophic river floods of the first millenium CE give two possible age (due to bimodal distribution of Equivalent Dose obtained) of 30-35 and 23-27 ky. NZ16 sands underlying a conglomerate with imbricated pebbles at Estaqueria (a site 4 km to the west of the main temples) which assumed a dominant role after the abandonment of Cahuachi, gave an OSL age between 18 and 21 ky. All these alluvial sediments adjacent to the adobe ceremonial temples or walls do not match with the first millennium CE. Also, a natural colluvium (NZ3) upon anthropic waste in the eastern sector of Tempio Sur is around 2 ky BP (OSL). This dating closely matches the calibrated radiocarbon age of 1960± 93 years before 1950 CE (conventional radiocarbon age: 2018 ± 35 years BP) of charcoal samples at the bottom of the colluvium itself. Searching more recent alluvial sediments only produced modern ages ≤150-180 years ago (NZ7, NZ8).
The OSL survey does not therefore reconcile with any ENSO-related mega-floods in the first millennium CE emphasized by Grodzicki (1990). This author probably made a geomorphological over-interpretation (through aerial photo) which led him to consider the sandy levels and sandy conglomerates as the alluvial deposit reaching the adobe constructions instead of pre-existing geological layers the Cahuachi temples were built upon. It is worth to note that archaeological evidence in the Nasca Phase IV period emphasizes debris flows and at least two heavy rain/alluvial events (around 450 CE, also documented in northern Peru) and intense periods of earthquake activity which are consistent with the progressive weakening of resiliency of the population and abandonment of large areas of Cahuachi (Orefici, 2016). It is thus time to overcome the paradigm of the ENSO catastrophic mega-floods for the decline of Cahuachi and the end of the Nasca Theocracy.

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Preliminary petrographic, mineralogical and geochemical characterization of mortars from public buildings in Pompeii aiming at relative chronology

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An archaeometric approach on bedding mortars from important public buildings in Pompeii (i.e. Eumachia Building, Sanctuary of Public Lares, Macellum, Temple of Jupiter, Sanctuary of Apollo, Basilica, Temple of Fortuna Augusta) has been performed to i) identify raw materials and technologies used in different construction phases; ii) provide information on construction strategy and procedures before and after the earthquake which struck the city in 62 CE (Mau, 1908; Maiuri, 1942; Dobbins, 1994; Wallat, 1997; Ruggieri, 2019; Dessales, 2022).
The sampling strategy was based on a systematic analysis of the public buildings in the Forum and for comparison, other public structures, with a specific focus on the post-seismic restoration phase and on the methods and technical procedures by which these restorations were carried out. In this perspective, the archaeometric analyses of mortars contribute to enrich the knowledge on ancient technologies.
A multi-disciplinary protocol was adopted including archaeological and architectural analysis combined with a minero-petrographic and geochemical best practices. All sampled mortars were analysed by means of optical and electronic microscopy: OM highlights the main morphometric and morphological parameters of aggregates and the B/A ratio; in the meanwhile, SEM-EDS was performed for geochemical characterization of binders and magmatic aggregates. Bulk X-ray powder diffraction (XRPD) and thermogravimetric analyses coupled with differential scanning calorimetry of enriched portion of binders and lumps better illuminated the mineralogical composition of mortars.
Samples were grouped on the basis of texture of the binder (micritic/sparitic/ heterogeneous), binder/ aggregate ratio, shape of the aggregate and the particle size distribution. The presence of under burnt fragments of stone for lime and the re-use of ancient mortars as aggregate were also evaluated.
The results suggested that the binders were obtained by burning Mg-rich limestones likely outcropping on the Monti Lattari, on the other hand, the aggregates have geochemical features fully compatibles with the Somma-Vesuvius products (Morra et al., 2010; Santacroce et al., 2008). It is evident that technologies employed in mortar production have differences in the time span before and after 62 CE mortars: a less care in raw materials selection and mortar production was observed, probably due to the urgency of restoration and reconstruction of damaged buildings.

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The use of stone resources in the Roman architecture in Oderzo (Treviso, Italy)

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In the framework of a broader research project on the Roman architecture of Regio X - Venetia et Histria conducted over the past few years by the Department of Cultural Heritage in collaboration with the Department of Geosciences of the University of Padova (Previato 2015; Previato 2018; Zara 2018; Previato, Zara 2018; Germinario et al. 2018; Boschetti et al. 2021, Dilaria et al. in press), this study aims to determine the provenance of the stones used in the city of Oderzo (Opitergium) for architectural purposes during the Roman Age.
In detail, we analyzed 47 stone samples collected from archaeological sites in Oderzo (29 samples) and architectural artifacts preserved at the local Museo Archeologico "Eno Bellis" (18 samples). The materials were petrographically, mineralogically and chemically characterized adopting a multi-analytical approach involving Polarized Light Optical Microscopy (PLM), X-Ray Fluorescence (XRF) and Quantitative Phase Analysis X-Ray Powder Diffraction (QPA-XRPD).
The results have enabled us to determine the quarrying areas that Opitergium mostly relied on in antiquity for architectural purposes and the stone trade networks in which the city was inserted. Furthermore, we have been able to determine how the utilization of stone resources evolved over time and how different lithotypes were selected and employed in the architecture of the Roman town. Preliminary findings suggest a higher frequency of stones from outcrops distributed along the Prealpine Arc of North-Eastern Italy and Istria, such as the Aurisina Limestone (Triestine Karst), and micritic limestones possibly quarried in the Istrian peninsula for the production of architectural artifacts, while lithotypes from North-Western Prealps appear to be less frequently used.

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Archaeometric investigations on bronze artefacts from Acerra, Campania (Southern Italy): early results

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In recent decades, the number of archaeometric studies of metal artefacts dated to Copper Age - Early Bronze Age increased considerably. often the research involves both chemical analyses and lead isotopic data (Artioli et al., 2020). However, a comprehensive research program aimed at establishing the origin and circulation of metals in this period has never been conducted, particularly in southern Italy. To address this gap, the present project analysed twenty bronze objects, primarily daggers and pins that were excavated from two funerary areas near Acerra (Naples, Campania). These objects are dated between 2400 and 1800 BCE (Aurino et al., 2021).
Chemical and microstructural characterizations were achieved through a multianalytical approach, including OM-RL, SEM-EDS, EPMA, and metallographic analyses. Lead isotope analysis (LIA) was employed to determine the origin of the metal. The findings indicate that all the objects are made of tin bronze, with tin content in the range 6.41% - 15.42% for daggers and 1.07% - 9.19% for pins. Some of the bronze objects exhibit rare metallic segregations or mineral inclusions in the alpha phase, while others display sulphides, delta phase, and small lead particles dispersed in the metallic matrix.
An internal database containing chemical and isotopic data of copper mineralizations (Artioli et al., 2016, 2020) is used to interpret the results of the lead isotope analysis (LIA). Not surprisingly, approximately half of the objects were produced using copper sourced from Southern Tuscany. The remaining finds exhibit various and complex signals of the copper sources, which will discussed during the presentation.

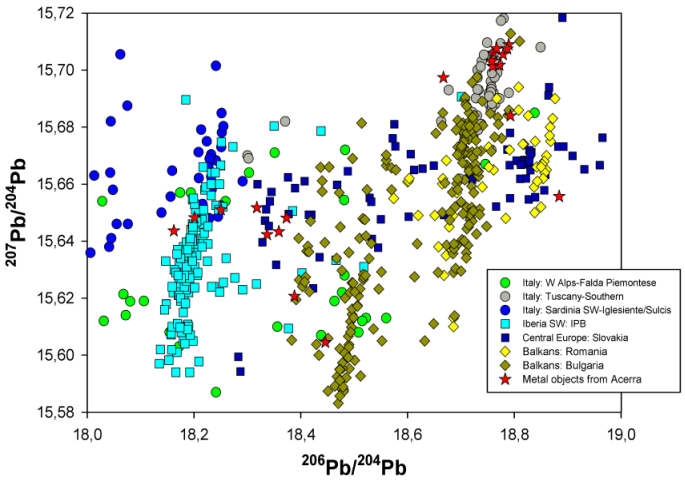

Figure 1: Lead isotope ratios diagram (207Pb/204Pb vs. 206Pb/204Pb) of the analysed metal object from Acerra compared with some cu mineralizations

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An innovative application of laser micro-cleaning on pyrite crystals in lapis lazuli to allow provenance study

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Lapis lazuli is a semi-precious blue stone widely used since the antiquity (VII millennium BCE) for the manufacture of precious carved artifacts. Although the Badakhshan quarries in Afghanistan are widely considered as the only sources of lapis lazuli in ancient times, others have been debated throughout history, so the origin of this rock remains an open question to this day. In the past years, our group have developed and successfully applied to lapis lazuli objects a protocol to identify the provenance of the raw material by means of a non-invasive methodology based on Ion Beam Analysis [1]. The use of μ-PIXE (Proton Induced X-ray Emission) and μ-IBIL (Ion Beam Induced Luminescence) has proved effective in finding physicochemical markers within reference geological rocks able to distinguish among five provenances: Afghanistan, Tajikistan, Siberia, Chile and Myanmar [2]. The realized protocol has been so far applied to different lapis lazuli archeological artifacts such as carved artifacts of the Egyptian Museum of Florence [1] successfully obtaining results regarding the provenance of the material.

However, samples from archaeological excavation contexts frequently show superficial weathering processes affecting the crystals of the mineral phases useful for provenance attribution. There are different weathering processes that were observed and studied [3]; for example, the pyrite phase partially transforms into iron oxide (hematite or goethite) from the surface or fractures towards the inner part of the crystal. In the last years we analysed a consistent number of lapis lazuli findings coming directly from archaeological excavations of sites of the II-III millennium BCE and located in different areas of the Middle-East, but the presence of these alterations limited the possibility to achieve a final provenance determination. Altered phases generally led to high scattering of data on trace element concentrations and thus to an ineffective evaluation of markers or, in extreme cases, to the impossibility to analyse the crystals of interest. To overcome this problem, we have recently started to test micro-cleaning treatments based on the use of micro-pulsed laser source on very small areas (about 100x100 μm2), strictly targeting pyrite crystals. The aim of this procedure is to create a small breach in the surface layer, allowing the proton beam to reach the preserved crystal beneath. The issue of pyrite alteration and the preliminary results of this cleaning approach, innovative in the case of lapis lazuli material, will be presented.

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Mortar characterization for radiocarbon dating within the PRIN 2020 CASTLE Project

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Within the PRIN 2020 CASTLES Project, dedicated to the study of mediaeval castles in central and north-eastern Italy and aimed at determining their chronology, the archaeometric analyses, mainly focused on the mortars used in the buildings of the various archaeological sites, constitute an important part.
In radiocarbon dating studies of mortars, characterization is fundamental to determine the nature of binder and aggregate, the technology of preparation of the mixture, and any contaminations. Moreover, only the complementarity of several analytical techniques can ensure a correct and comprehensive knowledge of the material.
In this first phase of the study, a mortar characterization procedure has been developed with the aim of identifying the most suitable samples to date.
It is important to emphasize that the raw materials used are usually associated with the place where the mortar was used. Therefore, the first step is to study the geology of the place. The next phase of sampling mortar fragments is particularly important and is carried out together with a team of historians and archaeologists who are able to read the stratigraphy of the masonry and indicate the correct point of extraction so as not to alter the final dating results.
After sampling, laboratory tests are performed: they start with a macroscopic observation of the sample and continue with a- phenolphthalein test (also carried out in situ); b- minero-petrographic characterization of the mortar (PLM); c- mineralogical analysis of the sample (XRD); d- microchemical analysis by scanning electron microscope (SEM-EDS). The samples are also subjected, where possible, to physical-mechanical tests that can provide further information on the properties of the mortar.
It is important to emphasize that the criteria for the selection of the samples are the presence of, i) fully calcic lime binder, ii) binder without recrystallization, iii) predominantly silicate aggregate, iv) presence of lime lumps. Only the samples with these characteristics are selected for the next step, dating. For this purpose, the samples were subjected to the CRYO2SONIC preparation method and accelerator mass spectrometry (AMS) measurement, for the evaluation of the radiocarbon content.

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Archaeometry of bricks of the UNESCO World Heritage Site of Urbino (Marche, Italy)

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The work is focused on mineralogical and chemical characterization of representative bricks from the historical centre of Urbino (UNESCO site), coupled with thermoluminescence dating (Martini & Sibilia, 2001). Fifteen samples from buildings dated to different historical times such as remnants of the Roman period, Medieval and Renaissance constructions (Mazzini, 1982; Luni, 1985; Agnati, 1999; Luni & Ermeti, 2001; Negroni, 2005) were collected and analyzed for a comprehensive study.
Thin section observations highlighted an improvement of brick production techniques from Roman times to Renaissance, marked by a reduction of temper coupled with a finer grain size and a better sorting of the aplastic components. In all samples the temper consists of quartz, K-feldspar, plagioclase and micas, and at least five groups of sedimentary fragments, namely from pelitic/marly, siliciclastic and micritic rocks, and by two strongly oxidized lithotypes. Only the bricks used in Roman times contain all the above-mentioned types of rock fragments, the latter being less represented in the other samples. XRD results suggest increasing firing temperatures from Roman to Renaissance times, probably due to changes in pyrotechnology. Major-trace elements analyses (ICP-OES-MS) allowed compositional comparisons with local raw materials, which are present in the surrounding of Urbino, and already indicated by Busdraghi et al. (1992). Finally, the thermoluminescence (TL) dating was used as a powerful method to date the original firing of bricks to constrain the chronology of building periods of the town. The oldest TL datings, which straddle the end of the Roman Empire, are those related to the external structure of a little furnace located just outside the historical centre. Bricks sampled from the Late Medieval structures of Santa Maria della Torre and Albornoz Fortress indicate a time interval extending from the second half of XIV century up to the XV century. The multi-stage construction of the Urbino Cathedral, documented by historical data (Negroni, 1993), and mainly due to damages by some earthquakes at different times (e.g. 1741, 1781 and 1789 A.D.), is documented by the obtained TL datings, from the mid-XV to the beginning of XIX century.

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Multianalytical investigations to understand the changes of a wall which layer by layer becomes pictorial support: the case of Bicci di Lorenzo found in Cerbaia (Florence)

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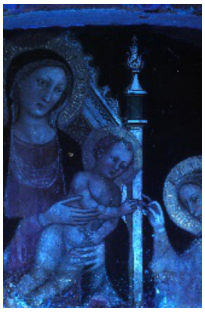
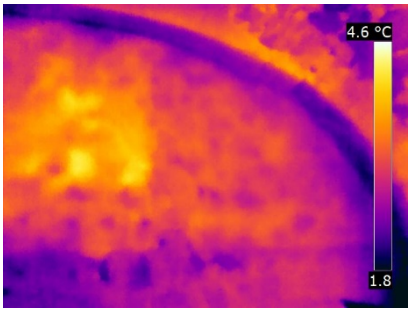
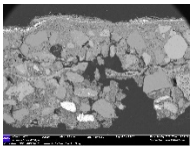
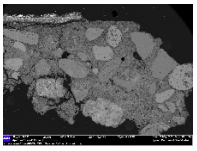
A complete set of diagnostic investigation has been carried out during the restoration work on the wall painting depicting the "Mystical Marriage of Saint Catherine" attributed to Bicci di Lorenzo (Florence, c. 1373 - 1452) found at the end of the 19th century in a privately owned building in Cerbaia (San Casciano Val di Pesa - Florence). The scientific analyses have been aimed to the identification of pigments and of the executive technique, characterization of the alteration products and of the restoration materials applied on the pictorial surface, in particular on the lower part which appears most degraded and incomplete. Furthermore, an important question posed by the art historians has been concerned the timing of the creation of the left part separated by a geometric frame from the main scene of the fresco. The UV fluorescence images allowed to locate the areas of pictorial integration and gave first evidence of the presence of organic materials and their distribution. The IR reflectography provided a clearer reading of the iconographic details, supporting the hypotheses about artistic technique. No compositional and technical differences emerged regarding the pictorial layers between the two figurative parts separated by the geometrically decorated frame. The thermographic investigation highlighted a different thermal behaviour between the central and right part of the scene compared to the left one in which San Lorenzo is depicted and which is separated by the fractures that run longitudinally to the scene. The greater accumulation of heat in the central and right part suggests a different stratigraphy as confirmed by the SEM-EDS analyses which show additional layers (arriccio and intonachino) compared to the sequence found in the left one. It is hypothesized that this consists of an infill made when the fresco was created and that, after the manufacture of the wall texture, the entire surface of interest was homogeneously prepared to the fresco.









The SEM-EDS spectra showed for both areas the presence of chlorine in the blue grains, suggesting that the alteration of the background layer with a greenish colour is due to the formation of copper chlorides. This colour change is more widely present in the background of San Lorenzo and could be explained by the more direct contact with the underlying masonry. The FT-IR investigation verified the presence of oxalates attributable to the mineralization of organic compounds, applied in the past as protective agents, which led to the degradation of the lower part of the fresco.
The multianalytical approach, planned starting from the art -historical question, has provided new data for the diachronic reconstruction of the changes in the architectural structure on which the fresco was created.

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The glass mosaic of S. Martino ai Monti: new data in the complex scenario of early Medieval Rome

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In the context of a wider inter-disciplinary research project on the Medieval Rome funded by the Swiss National Science Foundation, which allowed the archaeometric analyses on the glass mosaics of S. Agnese fuori le mura and S. Sabina, already discussed in previous AIAr Congresses, the present study, which focuses on the glass mosaic of S. Martino ai Monti, provides new data in the complex scenario of Early Medieval Rome.
The mosaic, dated to 5th-6th cent. AD, is located within a small niche in an underground space, which originally served as antechamber to the early Christian cult building placed beneath the actual church. It is, now, in poor state of conservation, because most of tesserae of the upper part were lost, and this prevents the exact readability of the iconography [1]. The lower part, showing the dress of a supposed bishop, is the best preserved one, and the majority of the analysed tesserae come from there. All the colours identified by the visual analysis were sampled for archaeometric analyses, and they are composed of various hues of purple/brownish, blue, and white; one orange tessera and one colourless, probably a gold tessera without gold foil and cartellina, were also selected, for a total of 11 samples.
The glass tesserae from San Martino ai monti were investigated by means of a laboratory analytical protocol, already applied on other glass mosaics from Rome investigated in the same research project, such as those from S. Agnese fuori le Mura, still in situ [2], and from S. Sabina, and which included optical microscopy (OM) for preliminary morphological observations, scanning electron microscopy, coupled with energy-dispersive spectrometry (SEM-EDS) for high-resolution morphologic inspection of glass and qualitative chemical analyses, electron microprobe (EMPA) to determine quantitative chemical composition of glassy matrix, and X-ray powder diffraction (XRPD) to define the crystalline phases of opacifiers/pigments used in the tesserae.
Results of the archaeometric analyses reveal how the tesserae from S. Martino ai Monti are characterised by glassy matrices having chemical compositions comparable to main compositional groups identified during both Roman and early Medieval periods, and by opacifiers/pigments mostly composed of antimony-based compounds, typical of the Roman tradition. This suggests the propension of recurring to the practice of re-using and/or recycling previous tesserae by the S. Martino ai monti mosaic-makers.
Further comparisons with other mosaics from Rome [3] are in progress, to broaden the understanding of the production and circulation processes of glass tesserae, emphasising the potentiality of this material of uncovering the technical and socio-cultural knowledge that underpins its manufacturing, use, re-use, and recycling in the early Medieval Rome.

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Characterization of the stone tesserae from the "A grandi fiori" Mosaic in the archaeological site of Casignana (Reggio Calabria, Italy)

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This study focused on the archaeometric investigation carried out on the stone tesserae constituting the "A grandi fiori" Mosaic in the room "N" of the Residential area of the Roman Villa of Palazzi of Casignana. The archeological site of Casignana is known to be, for its dimensions and for the value of the mosaic floors, the most significant historical and artistic heritage of Roman times within the Calabria region. The mosaic under analysis is a bichrome tassellatum mosaic with white marble and green micritic tesserae dating back to the 3rd century AD, characterized by decorative motifs of bell-shaped flowers arranged within circles and enclosed within a border that extends along the perimeter.
In particular, minero-petrographic examination by means of polarized optical microscope, X-ray diffractometry on powdered samples and scanning electron microscope equipped with energy dispersive spectrometer have been used to fully characterize the green micritic tesserae.
At the same time, provenance studies have been conducted on the white marble tesserae constituting the mosaic. The determination of the provenance of ancient marble artefacts represents one of the most debated issues among the art historians and archeologists. The reason for this such increasing interest is explained considering the possibility to trace the ancient commercial connections, obtain fundamental information about the attribution to a specific artist or workshop, as well as select the most appropriate material for replacement interventions. A multi analytical approach is essential to solve provenance characterization and requires the application of different methodologies coming from geoscience, chemistry and physic disciplines. It has been demonstrated that the employment of a single analysis is not efficient in equivocally establishing the marbles provenance. Following this line, twenty-four white marble tesserae have been collected and investigated under complementary techniques. In particular, the results obtained from detailed mineralogical-petrographic examinations on thin sections, XRD characterizations, isotopic measurements and SEM-EDS analysis were combined. In more detail, the fabric type, the presence of significant accessory minerals, shape of the boundaries (GBS) and the maximum size (MGS) of the calcite and/or dolomite grains were determined by means of a Polarized optical Microscope. Furthermore, the X-ray diffraction investigation carried out on samples grounded to powder (XRDP) allowed to identify the mineral phases, and therefore made possible to distinguish calcite grains from the dolomite ones. In addition, oxygen and carbon stable isotope compositions (δ13C and δ18O) were established through mass spectrometry. Finally, the scanning electron microscope equipped with an energy dispersive spectrometer was performed to analyze the chemical composition and the presence of accessory minerals. The results from the different investigations were then compared with most recent uploaded databases for the main Mediterranean marbles used in antiquity in order to assess the most probable origin of the investigated marbles.
The preliminary results obtained showed a great variability and a significant heterogeneity among the marble tesserae, indicating the use of different lithotypes coming from diverse ancient Mediterranean quarries. This conclusion demonstrates and confirms the richness and the great archeological importance of the Roman Villa.

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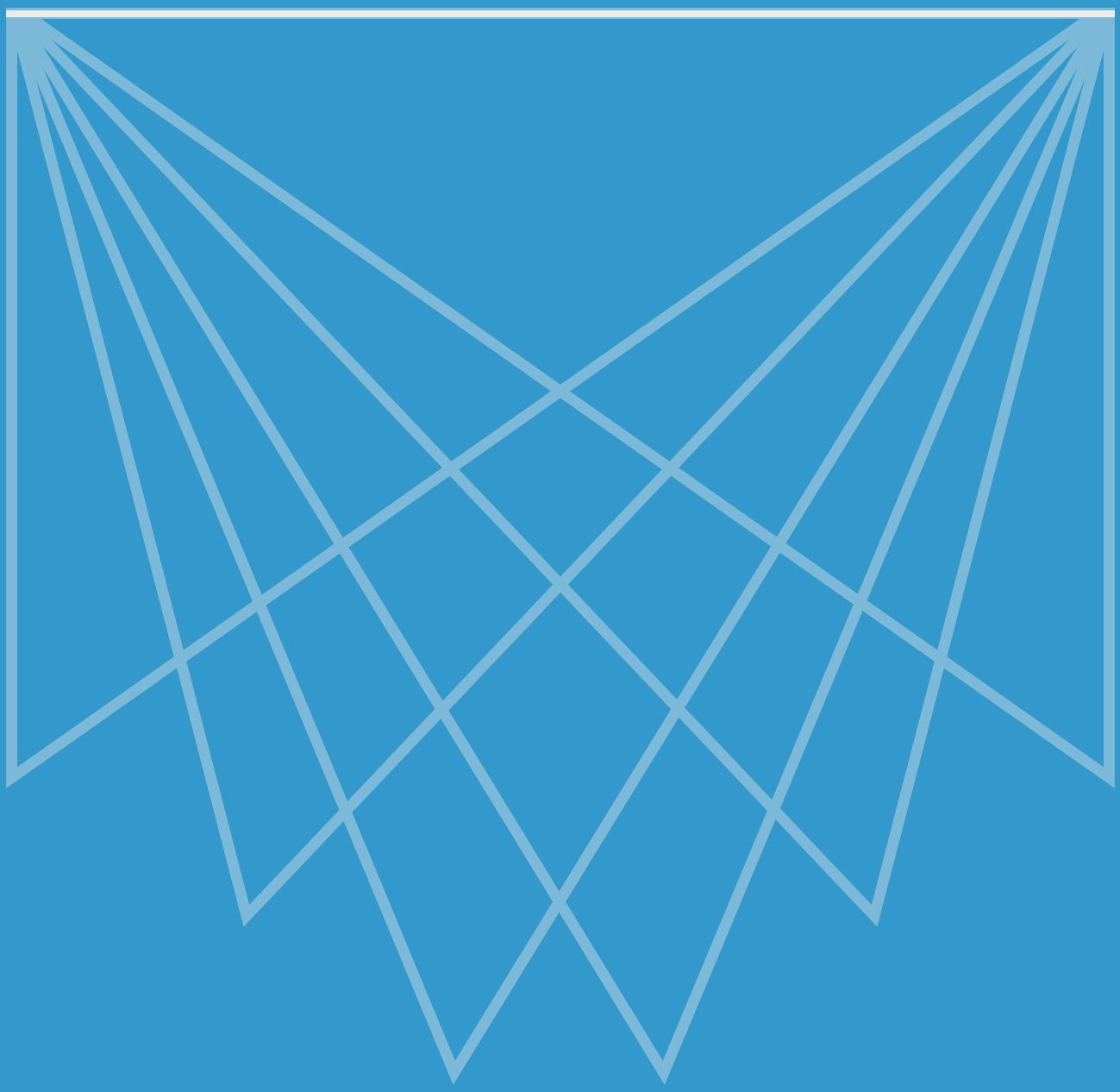

EVOLUZIONE DELLE TECNICHE DIAGNOSTICHE NEL TEMPO

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Comparative analysis of three-dimensional digitalization techniques for the exhibition of an Egyptian mummy of the Museo Egizio di Torino

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Digitalization techniques are attracting the interest of experts in the field of cultural heritage. Several instruments allow the creation of three-dimensional virtual replicas of historical artifacts acquiring data about the overall appearance of an item, its geometry, and texture [1]. Indeed, digitalization is a useful tool to document the current state of preservation of an artwork and to simplify the detection of potential damage progression as a support for conservators and conservation scientists.
Photogrammetry and laser scanning are among the most widely used three-dimensional acquisition techniques in the field of cultural heritage, characterized by distinct features. Photogrammetry is a technique that allows the reconstruction of a virtual replica of an object through the use of a camera: several acquisitions of the subject at different angles are taken, and then processed within specific software that recognizes the homologous points between the different images, leading to the creation of a point cloud, a mesh and an extremely detailed 3D model with a photo-realistic texture [2].
Laser scanners are more expensive than photogrammetry but they are able to reconstruct the geometry of an object with greater accuracy with respect to photogrammetry, reaching 0.1 mm accuracy for small objects [3]. However, laser scanning allows a texture to be acquired at medium or low resolution, and if the instrument used is working in structured light, it cannot be reconstructed.
With the goal of setting up a low-cost system that is free to access by the use of open-source software, this research is focused on the dimensional comparison between the meshes obtained through these two techniques, using the model obtained by laser scanning as “ground truth”.
In this paper, a case study regarding an artifact from the Museo Egizio di Torino is presented. In particular, the research regards the digitalization of a Roman period Egyptian mummy consisting of a painted shroud and cartonnage restored at the laboratories of the Centro Conservazione e Restauro dei Beni Culturali “La Venaria Reale”.
During the restoration work, the shroud and the cartonnage were temporarily separated: this revealed the presence of the mummy’s skull, lacking the usual bandage, probably from a previous theft. Therefore, the creation of virtual replicas of the skull and the cartonnage was useful for the creation of a custom-made support structure, required both to enable the restoration and for the correct relocation of the elements, and for its exhibition in the museum.

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Multi analytical approach for in situ non-invasive analyses on orichalcum ingots from Mediterranean Sea

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In February 2016, 47 ingots were found in the seabed of Contrada Bulala (Gela, CL, Italy) near the site where in 2014 40 ingots had previously been recovered. The ingots composition was determined to be a Cu - Zn alloy [1], dated by the archaeologist to the VI century B.C. This specific alloy was then identified as orichalcum. From an archaeological point of view, the interest was to define the relation between the two discoveries and the origin of primary sources of metals. In previous studies, fundamental information was achieved by applying chemometric treatment of the analytical results obtained on microsamples from ingots from first and second discovery [2,3]. Starting from this database and from availability of microsamples taken from the ingots, we discuss the feasibility to obtain elemental and spectroscopic information from the investigated area by using a non-invasive approach.
A recently commercialized portable instrumentation dedicated to synchronous and co-registered XRF and Vis-SWIR hyperspectral measurement, with non-destructive and contactless approach to the samples, has been used to analyze three microsamples taken from three different ingots of the 2016 group.
With the purpose to build a non-invasive in situ protocol, the XRF obtained results are compared with the literature data [1-3] and they match. Moreover, the hyperspectral measurements are cross-checked with diffuse reflectance spectra acquired with a spectrophotometer in the UV-vis region, where the response related to the Cu transition in the alloy can be detected. The spectra of the three samples look very similar, with a broad minimum from about 250 nm up to 450 nm and a shoulder at about 600 nm; both these features, typical of brass, are known to vary as a function of Cu and Zn concentrations [4], which are indeed very close for the three samples (Cu ≥70% and Zn ≤20%). In addition, the spectra of the samples with the same Pb-isotope ratio are nearly identical.

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From waste to resource: fabrication of flexible and low-cost SERS sensors for Cultural Heritage materials

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Within the framework of the SAMOTHRACE project, the IPCF-CNR Messina is developing a protocol for the creation of groundbreaking, flexible and eco-friendly SERS (Surface-Enhanced Raman Spectroscopy) sensors [1] specifically designed for the characterization and diagnostics of materials integral to Cultural Heritage.
Their fabrication is based on the use of Pulsed Laser Deposition (PLD), a physical technique employed to deposit nanoparticles (NPs) of noble metals (Au or Ag) [2], onto substrates made of cellulose fibers reclaimed from waste. Deposition conditions (as laser fluence, gas pressure, target-substrate distance, ablation time, etc.) and paper roughness play a crucial role in determining SERS effect. The prepared substrates have been meticulously studied using UV-Vis absorption spectroscopy to determine optical resonance peaks, SEM to evaluate NPs shape and aggregation, and AFM to map surface topology and measure NPs dimensions. Through rigorous testing and analysis, we have evaluated the performance, sensitivity, and reliability of the sensors involving several test molecules [3-4] up to real-world scenarios. In a series of case studies, results of which will be shared during the presentation, we have validated our devices on archaeological materials from pigments and dyes to complex degradation products, all associated with artifacts preserved in museums and archaeological sites. The results of this study highlight the potential of using eco-friendly SERS sensors in the field of archaeology and cultural heritage preservation, offering a sustainable and effective tool for non-invasive and non-destructive analysis and conservation of valuable artifacts.
In conclusion, the SERS sensors developed in the SAMOTHRACE project mark a significant stride in heritage science, integrating sustainable practices with advanced technology (Fig. 1). Our findings not only demonstrate the practical applicability of these sensors in analyzing cultural artifacts but also highlight the potential of sustainable technologies in future research and preservation efforts.

Fig. 1 – From waste to resource: sustainable production cycle of SERS sensors for Cultural Heritage

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Advanced isotopic techniques to investigate Cultural Heritage: The research activities at the iCONa laboratory.

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Bulk stable isotope (δ13C, δ15N, δ18O, δ34S, 86Sr/87Sr) analyses have become nowadays an essential tool in archaeological research, providing valuable insights into the investigation of ancient populations lifeways. In this presentation, we will showcase the current research projects and the advanced isotopic techniques currently in use at the "iCONa" (isotopic Carbon, Oxygen and Nitrogen analysis) laboratory of the University of Campania "Luigi Vanvitelli", in collaboration with the Sapienza University of Rome, together involved in the inter-university laboratory MAReA Centre (Mediterranean bioArchaeological Research Advances).
The laboratory is equipped with high-precision analytical instrumentation (all branded Thermo Scientific), to measure isotopic ratios: a Delta V Advantage isotopic ratio mass spectrometer, with dual inlet system for gaseous samples. For solid/liquid samples, the spectrometer is connected in continuous flow, via Conflo IV, to a Flash EA 1112 Series elemental analyzer, for δ 13C and δ 15N analyses, to a Thermo Chemical Elemental Analyzer TC/EA, for δ 18O analysis, and to a gas chromatograph Trace 1310 – GC Isolink, for compound-specific one.
Our research activities are centred around investigating the relationship between ancient societies and the environment using advanced biomolecular techniques, encompassing different time periods and geographic areas. We use innovative scientific protocols to pretreat samples of zooarchaeological, archaeobotanical, and osteological human remains to carry out stable isotope measurements and radiocarbon analyses. We specifically focus on investigating paleodiet (δ13C, δ15N) and spatial mobility (δ18O,86Sr/87Sr) in ancient populations. Moreover, the field of bioarchaeology has witnessed a significant evolution in stable isotope analysis techniques over the last years. Advancements in mass spectrometry, analytical instruments, and biomolecular methods, have improved the resolution and accuracy of stable isotope measurements with the emergence of novel methodologies. Specifically, the employment of advanced techniques such as compound-specific stable isotope analysis of amino-acids (CSIA-AA) and incremental dentine analysis is allowing us to gain valuable insights into past human subsistence practices at a high-resolution level. Furthermore, the increased number of published bioarchaeological databases and their meta-analyses has played a crucial role in addressing archaeological questions at different spatial and temporal scales. In collaboration with the Pandora and IsoMemo Initiative, supported by the Max Planck Institute for the Science of Human History, we employ R-based modelling tools to perform meta-analyses on isotopic data. We put a specific focus on estimating the macronutrient contribution of food sources in past populations to increase the resolution of isotope-based dietary reconstructions.
With our contribution, we aim to address the rapid evolution in the last decades of isotopic analyses and their applications in the archaeological field through the overview of the research activities and methodologies performed by the MAReA Centre research group at the "iCONa" laboratory.

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Recent advances in neutron resonance transmission imaging and heritage science application

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In recent years, Neutron Imaging (NI) established its strengths and extraordinary versatility for non-destructive investigation of many kinds of morphological and microstructural properties of materials expanding its availability at neutron sources worldwide and providing fully complementary information with respect to conventional imaging techniques based on other probes such as X-rays. The rapid development of NI methods and detection systems has led to a striking improvement of resolution and efficiency, as well as of techniques based on a broad range of contrast mechanisms, making imaging with neutron beams indispensable in modern research.
The current state-of-art of neutron imaging techniques is particularly advanced. This work, in particular, aims to provide a glimpse into the advancement of a neutron imaging method named Neutron Resonance Transmission Imaging (NRTI) [1-6] and performed at the INES beamline of the ISIS spallation neutron source [7]. Conventional NI often uses cold or thermal neutron beams. However, there is another rather interesting and underutilized energy range for imaging, the epithermal range, which is exploited to perform the NRTI experiments. High epithermal neutron fluxes are made available by neutron spallation sources, enabling the isotopic and elemental characterisation of materials thanks to the presence of intense resonance structures in the neutron-induced reaction cross-sections. The NRTI technique is based on the resonant absorption of the incident epithermal neutrons, resulting in a transmitted neutron beam containing characteristic dips univocally related to the material's elemental composition.
What sets NRTI apart from standard neutron radiography/tomography is the possibility to conduct simultaneously spectroscopic and imaging analysis, and potentially quantitative imaging. In fact, for phenomena based on resonant neutron capture, the interaction of neutrons can be exactly calculated, therefore they can be used for precise quantitative analysis.
NRTI allows to enhance the contrast of specific elements compared to others (in particular those with similar neutron attenuation coefficients) within the bulk of an object, thanks to the full unintegrated transmitted spectrum stored in each detector pixel. Furthermore, this technique is one of the first imaging methods that can be used for isotopic imaging.
Recent advances in NRTI have been achieved with the development of a reliable protocol of measurements, data normalization and analysis, and will be presented with a particular focus on a Cultural Heritage application and on a first approach for a quantitative calibration of this imaging technique.
The striking features of NRTI make it suitable for the characterization of inhomogeneous samples [5,6,8], in particular for Cultural Heritage studies. Potential applications of NRTI will be presented with special examples of characterization of archaeological samples.

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Macroscopic FT-IR, UV and XRF mapping for the in situ characterization of pictorial materials: Technological advances and applications

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The in-situ characterization of pictorial materials represents a fundamental aspect for the authentication, restoration, and conservation of investigated artworks. In recent years, the trend in the development of analytical instruments dedicated to the cultural heritage is to convert point-by-point techniques into imaging devices with the aim of simultaneously obtaining the spectral characteristics of the materials and their localization on the investigated areas.
In this perspective, FT-IR-UV and XRF macroscopic mapping represents a unique tool for detecting the distribution of inorganic (sulphates, carbonates, silicates, etc.) and organic (binders, paints, degradation products, etc.) materials on painted surfaces.
We present here a macroscopic FT-IR-UV-XRF scanner for in-situ analysis developed during the ARTEMISIA (ARTificial intelligence Extended-Multispectral Imaging Scanner for In-situ Artwork analysis) project funded by the Lazio Region (Italy) within the DTC Excellence Center for Cultural Heritage.
The integrated scanner has been applied to selected paintings with the aim of supporting restoration treatments and investigating the artists' palette. In this work we present the application of MA-Scanner to an oil painting dated at XIX sec. and attributed to Giacomo Favretto.

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Application and testing of coatings for the conservation of Underwater Cultural Heritage: new challenges within the TECTONIC project

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Underwater cultural heritage (UCH) concerns all traces of human existence also found in the seabed, such as shipwrecks, submerged structures and various historical/archaeological sites and objects. In recent years there has been a growing interest in the study of the finds and degradation phenomena that occur underwater. Numerous researches have focused on examining the deterioration of stone materials in underwater environments and developing innovative strategies and challenges to safeguard them. Natural processes play a significant role in the degradation of UCH. Underwater sites are subject to relentless natural erosion, which occurs as a result of water currents, wave action, and the abrasive force of sediments carried by the sea, as well as to the biological colonization [Ricci et al. 2009; La Russa et al 2015; Davidde B. et al. 2010; Relini G. 2003]. Therefore, the conservation of UCH presents numerous challenges due to the particular environmental conditions, the difficulties associated with accessing and working in underwater environments, and the potential risks posed by natural forces and human activities. The TECTONIC project aims to foster collaboration between academic and non-academic professionals, facilitating the exchange of skills and expertise. The project's objective is to implement, enhance, and evaluate innovative materials, techniques, tools, and methodologies for the protection of UCH [Ruffolo S. et al. 2017; Ricca M. et al 2020; Donato A. et al 2023]. This work shows the first results obtained from the experimentation conducted between the University of Calabria (Italy) and the company Synpo akciova spolecnost (Czech Republic), aimed at developing an underwater coating with antifouling properties suitable for safeguarding the UCH in situ.

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Toward the monitoring of cleaning treatments on painted surfaces: the potential of macroscopic FT-IR mapping

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Over the centuries, several types of coatings were applied as final pictorial layer by artists and/or restorers to reach the color saturation of paintings and gloss properties and to preserve the pictorial layers from the environment in which the artworks are exposed.
Since the exposure to the pollutants, to the light, and to the dust could leads to chromatic alterations and physical and optical degradations of coatings, mainly due to oxidation phenomena [1,2], cleaning processes are required to remove and replace these layers.
In order to evaluate the effectiveness of cleaning treatments on painted surfaces, several scientific research have been addressed to the development of new analytical methods or approaches involving both point-by-point techniques and imaging devices [3].
From this perspective, FT-IR spectroscopy represents a good technique to detect the presence of varnishes, degradation products and residues of the cleaning products, since it is very sensitive to the organic matter [4]. Although point-by-point analyses in reflection mode is a successful approach to monitor the removal of patinas., therefore we propose the use of a Macroscopic FT-IR scanner for in-situ analyses to map the distribution of the compounds on the surfaces [5, 6]. This technique was applied on several wooden paintings during the restauration treatments, especially the removal of the varnish layers and oxalates have been monitored.

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To be or not to be? Open questions when the conventional approach is not working

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Today, the non–invasive approach for the investigation of painted surfaces is a consolidated methodology. The spectroscopic techniques, applied with the portable instrumentation, are quite spread and used in Museum and Archaeological Parks. In the common knowledge, the multi-technique approach and the chemiometric data analysis are able to give the most of answer. However, not all the questions have an answer and sometimes, more dubts appear after the first step of the investigation. Obviously, the answer could be obtained by a “simple” microsampling, but not it can be done for the most of the artefact. In addition, the permission for the microsampling can be not given.
So, is then impossible to find the answer?
In this presentation, some “difficult” case studies are introduced, mainly about painted ceramics. It wants to be a moment to share the difficulties found in the conventional approach and to open a discussion about the possible strategies to use and the challenges of the incoming era of the non-invasive approach.

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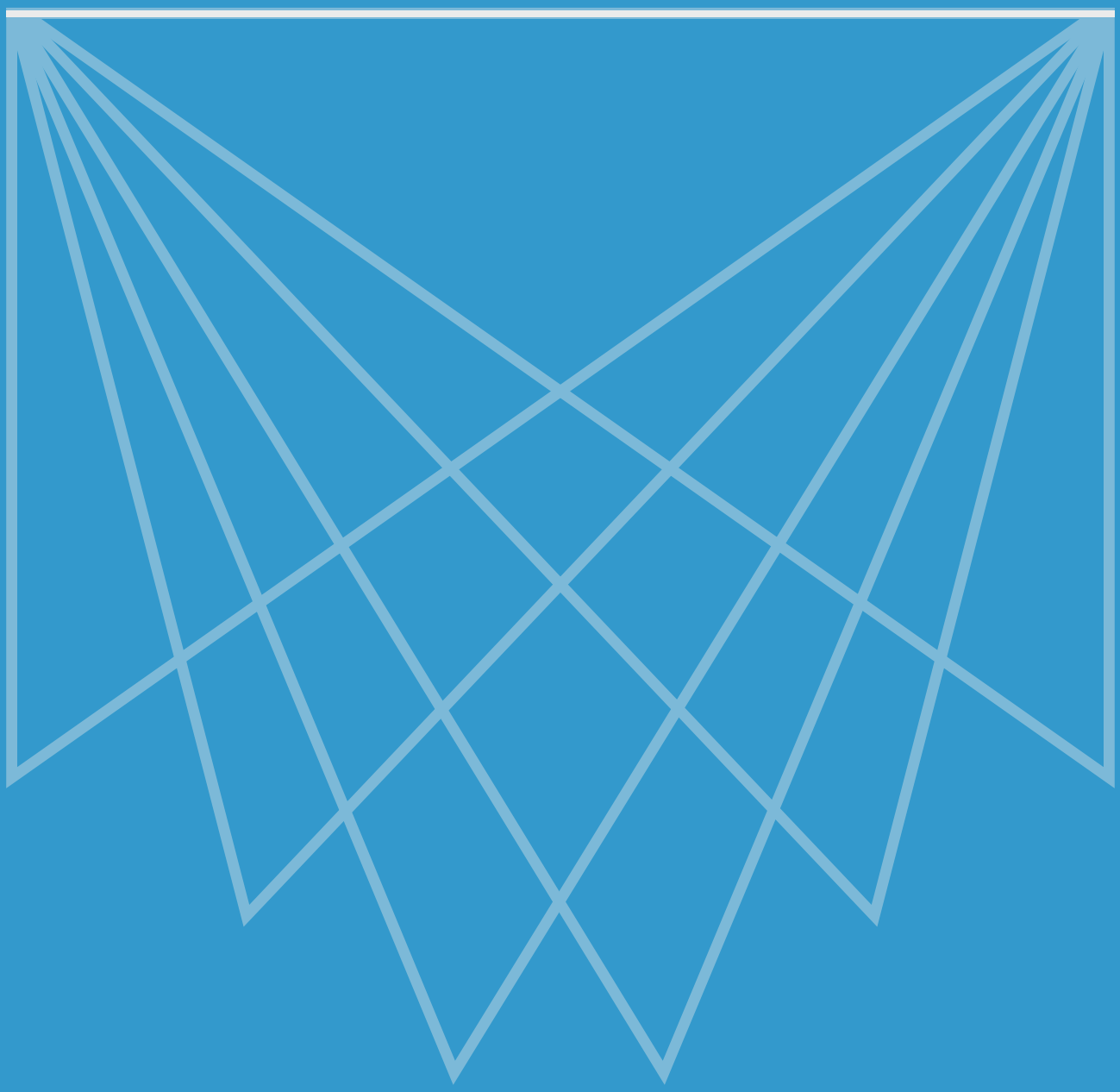

TECNICHE DI ELABORAZIONE E ANALISI DEI DATI TEMPORALI

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Climate-based damage functions for geoheritage preservation: data governance and analysis perspective

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A climate-based damage function is a mathematical equation that models the potential changes and exposures of cultural heritage to damaging events over time in response to changing climate and environmental conditions [1]. Its primary purpose is to predict and anticipate the impact of climate-related stressors on heritage materials, providing a quantitative means to evaluate and proactively manage potential deterioration or damage. These functions typically incorporate various climatological and microclimatological parameters such as air temperature, relative humidity, precipitation, and other relevant variables. The initial stage involves gathering climate data from various sources, including satellite observations, reanalysis models, ground-based weather stations, and in-situ data loggers. The integration of these data sources requires multiple preprocessing steps and rigorous data management. Additionally, ensuring accuracy and reliability involves applying statistical analysis and evaluation metrics to consolidate the results.
The primary focus of this work is to develop the methodology for processing climate data as input for computing damage functions, using the soapstone trench quarry in Parco Archeologico Botanico del Paradiso (Chiavenna, Italy) as an illustrative geoheritage case study [2]. Two validated damage functions, widely utilized in the literature for stone materials, are employed: freeze-thaw cycles [3] and thermal disintegration [4]. Repeated exposure to these temperature-driven cycles heightens the risk, impacting the stability of geological features and historic sites, particularly open-air geoheritage locations. Climate-based damage functions serve as tools for estimating the potential for material degradation. The climatic parameters used include air temperature series near the rock in different outcrop areas, precipitation, and relative humidity. The methodology outlined in this study involves a comprehensive process of quality assessment, correction, and validation of climate and microclimate data. This process includes addressing missing data and rectifying inaccurate readings through a series of quality assessment tests, encompassing range, step, consistency, and persistency checks [5]. In the homogenization phase, potential reference series are identified and subjected to a secondary quality assessment [6]. The selection of reference series is facilitated by hierarchical clustering analysis based on time series derivatives. To assess variations and similarities across different spatial and temporal scales, a combination of parametric and nonparametric tests (e.g., Mann-Kendall Test, Petit test, Standard Normal Homogeneity Test) with autoregressive models (SARIMA) is utilized [7]. Finally, the outputs generated by the damage functions are subjected to comparative analysis, involving statistical tests and frequency decompositions. This analysis aims to assess disparities across various geographical areas and temporal patterns, while also evaluating distinctions between processed and unprocessed climate data in computing damage functions.

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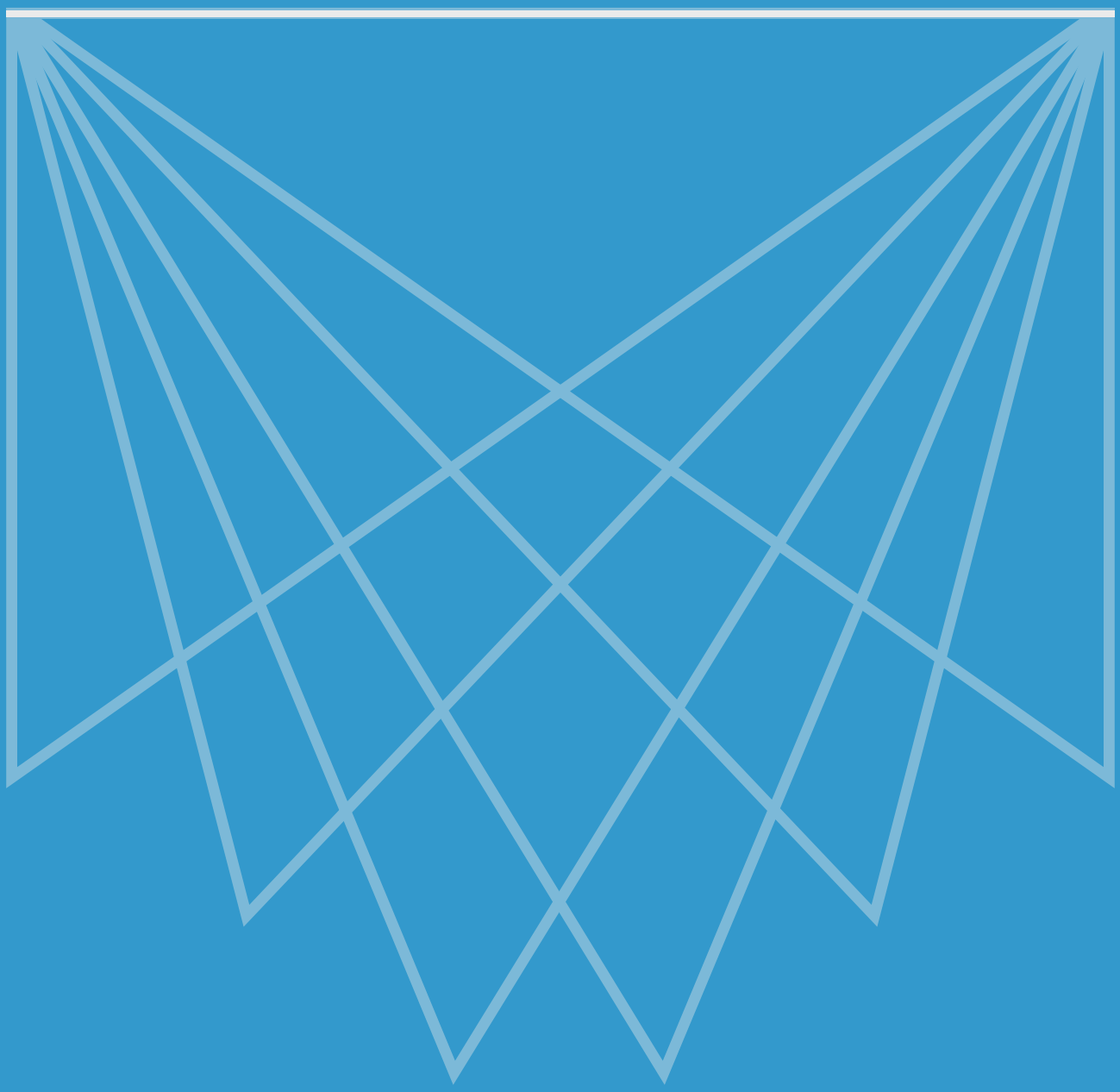

COMUNICAZIONE E FRUIZIONE DEI BENI CULTURALI

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A trustable multispectral photogrammetry pipeline for the reproduction of non-collaborative materials

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Many non-invasive techniques are now widely available to experts for the study and understanding of real objects, encouraging the development of new low-cost survey tools. The opportunity to create digital twins opens up many possibilities for in-depth study of art objects. In particular, the increasing adoption of photogrammetry and multispectral reflectography in the field of cultural heritage has provided art historians, conservators and conservation scientists with precise tools for the study of pigments and artistic techniques, facilitating the attribution and dating of art-historical artifacts [1].
Photogrammetry has established itself over time as one of the most common modern survey tools, distinguished by its ease of implementation and ability to obtain metrologically reliable and photo-realistic three-dimensional models in a quick and cost-effective way [2]. Although its use is currently quite popular, certain limits prevent its proper application in the field of cultural heritage, especially in the presence of objects made of shiny, reflective materials such as metals. In recent times, the use of polarized light in the 3D field exploiting the Physically Based Rendering has also provided encouraging insights into the use of photogrammetry even in the presence of non-cooperative materials, expanding the application range and improving the resemblance to the real object [3].
Furthermore, the possibility of producing three-dimensional models integrating data from other spectral bands (UV/IR luminescence and reflectography) and other diagnostic techniques (XRF, RAMAN etc.) opens up new perspectives in the study of constituting materials, allowing information to be combined in a three-dimensional model of more immediate consultation.
This work presents a workflow that addresses the various issues and combines them into a single three-dimensional model that is correct in size and color, light and easy to consult thanks to the use of free, cross platform visualization tools.The chosen case study, a Chinese gold statuette depicting Buddha, general chronology 6th century BCE, holds many interesting enigmas by addressing the limitations imposed by photogrammetry and integrating data from different techniques into a single three-dimensional model that can be navigated online.

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The Aragonese Castle in Reggio Calabria Reconstructed: Laser Scanning, 3D Modeling, and Virtual Public Access

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The work introduces a comprehensive 3D modeling and interactive online access for cultural heritage sites. In the frame of the project “Circuito delle Aree Archeologiche Urbane di Reggio Calabria” led by the Municipality of Reggio Calabria, the IPCF Messina digitally documented the historic Aragonese Castle of Reggio Calabria (Italy) alongside other archaeological sites [1-2]. This iconic southern Italian fortification has medieval Byzantine origins, though only a portion of the original complex survives today. The castle has undergone transformations across centuries, leaving an intricate history that digital modeling can help reconstruct. Our cutting-edge techniques captured highly detailed 3D data of the remaining architecture [3] for preservation and public dissemination. By making these virtual models widely accessible online, our project advances community connections with cultural heritage.
The use of terrestrial laser scanning (Faro Focus 3DS 120) provided ultra-high density point clouds to digitally model the architecture in meticulous detail. The instrument's phase-shift technology and built-in RGB camera enabled highly precise and information-rich 3D data capture suited to heritage preservation. The survey pipeline included 157 scanning stations for complete coverage alongside data processing protocols to generate meshed models. These comprehensive digital datasets, unprecedented for a southern Italy municipality, enabled the creation of an interactive website where diverse users can explore and learn about the site virtually (Fig. 1). This online 3D museum represents an innovation in participatory heritage visualization, empowering public knowledge and engagement with interactive models, historical images, and multimedia content. Moving forward, our agreement with Reggio Calabria prioritizes disseminating these virtual models and educational resources to citizens and students. The website's accessible and engaging platform advances digital archaeology's meaningful societal impacts, bringing heritage sites directly to people's homes for learning and enjoyment. Our collaboration also enables continued model enrichment to visualize the castle's evolution across centuries. Overall, the project showcases the immense potential of digital archaeology to study, preserve, and communicate cultural heritage.

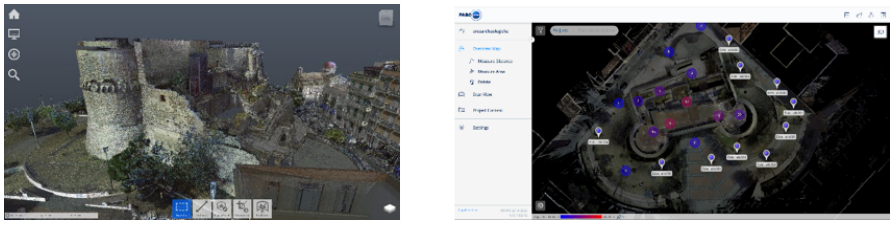

Fig. 1 – (a) 3D dense cloud visualisation and (b) web-application with scan locations.

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INDICI

INDICE DEI CORRESPONDING AUTHORS

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