



# BOOK OF ABSTRACTS

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# PLENARY LECTURE

# Safeguarding Cultural Heritage in a changing environment: sustainable solutions for preparedness and risk management

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The risk to cultural heritage as a consequence of the related hazards and impacts of climate change, particularly extreme events, is globally recognized. However, this is not sufficiently addressed by research-based measures dedicated to its safeguarding, nor is it properly set out in national disaster risk-reduction and management plans and measures (Bonazza et al, 2018, Cacciotti et al, 2021).

This contribution intends to highlight the key steps in the state of progress of the research activities at European level addressed to the protection of Cultural Heritage at risk, focusing on the still existing challenges and methodologies currently under development for their overcoming.

Starting for the results produced in the EU FP6 Project Noah's Ark on quantification of the impact of slow cumulative climate changes on heritage materials outdoors exposed (surface recession, thermal stress, biodeterioration, blackening), discussion will then be dedicated to the methods and tools for risk mapping imposed on cultural heritage and landscape by extreme hydrometeorological hazards developed in the framework of the Interreg Central EU Projects ProteCHt2save and STRENCH. The methodological approach therein adopted involves: i) the development of hazard maps at territorial level, based on outputs from climate models and earth observation (Copernicus programme), and ii) the vulnerability assessment at building scale, underlying on the determination of physical and managerial criticalities at local level (Bonazza et al., 2021).

Research results clearly demonstrate that in dealing with the impact evaluation of climate change on monumental complexes and historic buildings, downscaling in spatial resolution of outputs from global and regional models still remains an open issue in order to allow the implementation of adequate future projections of deterioration phenomena occurring at local-building scale. In addition still resources need to be addressed to the identification of risk indicators and damage functions purposely tailored at local level based on a user-driven approach, by meeting the requirements of the targeted stakeholders (Public Authorities, decision and policy makers, rescue bodies).

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## Valuation as valorization towards sustainability: holistic impact assessment of cultural initiatives

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While the relevance of Cultural Heritage for the well-being of individuals and communities is generally acknowledged, policy makers, cultural managers, and professionals still lack reliable tools to ascertain whether resources are used effectively and efficiently, and to give a full account of outputs, results, and outcomes that go beyond the narrow limits of formal accountability. To address this gap, the SoPHIA project was initiated to draw consensus towards a holistic impact assessment model to evaluate interventions on cultural heritage. In particular, the SoPHIA approach aims at ensuring that interventions in cultural heritage (at local, national, and European level) have positive impacts on all dimensions of society. In line with the crucial switch from a logic of spending (*“it is important to allocate funds for culture”*) to one of impact (*“it is important to give evidence of the impacts obtained from the interventions”*) proposed by the European Commission, SoPHIA moves forward by presenting an innovative approach. First of all, it highlights the link between the quality of interventions and the impacts. In fact, it is crucial to define a precise quality goal, direction, and benefits for people and communities to measure the desired impacts of policies and actions. The SoPHIA model adopts a multi-dimensional approach based on three axes (domain, people, and time) to:

- analyze all aspects (domains) of society in which a CH intervention may create an effect;
- advocate for all people engaged in the intervention and that may benefit from the intervention itself;
- present a longitudinal perspective to measure the intervention’s legacy over time.

Referring to the second aim, SoPHIA promotes a participatory model that involves a wide and diverse number of stakeholders (academics, experts, practitioners, and policy makers working in the CH field, as well as active citizens) within a new digital social platform. The SoPHIA community has been actively engaged throughout the project by working with the Consortium in implementing the holistic impact assessment model and the policy recommendations.

Finally, the SoPHIA project aims at providing suggestions and recommendations on relevant issues in the field of impact assessment to support Europe’s future political and research agenda on CH intervention.

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**SESSIONE 1:**  
**SOSTENIBILITA' NELLE PRODUZIONI**  
**ANTICHE**

# Once upon a glass

## Cycles, recycles and reuses of a never-ending material

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Glass can be considered a locus of meaning, a material which has been the repository of traditional knowledge and technological expertise for at least three millennia. The history of glass speaks of know-how, technological transitions, and contaminations of recipes for its manufacture, that have changed across the world over the centuries.

As the amount of recovered glass from archaeological contexts is much lower compared to ceramic and metal finds, research has often considered glass as a rare material. Furthermore, glass production, in ancient times as in the present days, requires the use of selected raw materials and noticeable amounts of fuel, making reuse and recycling practices necessary to foster sustainability, from both an economical and an environmental perspective [1].

Latin authors like Juvenal and Martial, reported buyers of broken glass in the Imperial Rome [2], presumably destined for recycling. Archaeometry has also provided data that allow, today, to clarify different aspects related to production cycles, uses and reuses of a material that, starting from the Roman age, became as common as modern plastics [3, 4].

From beakers and goblets reused with different purposes, to mosaic tesserae detached for making new mosaics or to be refused and employed as "pigments" for colouring glass, this paper aims to provide an overview on reuse and recycling practices of ancient glass, through a disamina of selected case studies from Roman to Middle Ages, showing how the cycle of this material can be framed as an actual example of sustainable circular economy in the past.

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## The use of lime over the centuries: the complexity of the Apulian built heritage

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From Subappennino dauno to Salento, the carbonate geological substratum characterizes the landscape and the material culture of Apulia. In the field of historical buildings, the wide use of lime as a binder in various contexts of application emerges from a series of case studies covering a chronological span from the 4th century BCE to the 18th century CE.

The petrographic, mineralogical and chemical study of lime mortars and paints brings to light technological complexity, almost completely cancelled by the widespread presence of industrial products, that starts from the choice of the stone to be calcined and of the aggregates and passes through the modalities of lime hydration and of preparation of the mixture up to the laying.

Through examples of use from antiquity to the modern age in central-northern Apulia, technological solutions indicating an ecological dimension of production will be presented and discussed, bearing witness to technologies on a human scale and sustainability.

# Pyroclastic rocks in the structural mortars of Roman Nora (Sardinia). A green material for the production of sustainable concretes in antiquity

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It has been estimated that about the 8% of the total CO<sub>2</sub> emissions into the atmosphere of our planet depends on the production of cement. A large part of these emissions derives from the *clinker* manufacturing method, whose production, even nowadays, mainly involves coal fossil sources necessary to reach the very high temperatures (~1500 °C) at which the formation of *clinker* occurs. In order to reduce the global CO<sub>2</sub> emissions, many companies are studying traditional building techniques to re-discover alternative materials suitable for the production of cementitious compounds for modern construction without lowering the physical-mechanical capabilities offered by traditional cements. The Romans did not know cement but were able to produce highly cohesive concretes based on "aerial" lime, produced by calcination at relatively low temperatures (~850 °C) of carbonate rocks in wood-fired limekilns. This happened because the ancient builders empirically knew the properties of certain elements (pozzolanic materials, i.e. volcanic tephra and *terracotta*), which, by chemically reacting with lime, can improve the properties of the final concrete once it had set, thus obtaining structural properties sometimes comparable to modern cements. The most famous pozzolanic material, cited even by Vitruvius and Pliny, are the pyroclastic rocks outcropping in loose deposits in the Phlegrean Fields around Pozzuoli. This *pulvis* gave excellent structural characteristics and waterproofing properties to ancient concretes, and it was recommended by authors for the construction of *opus caementicium* maritime piers. In fact, Brandon *et al.* 2014 demonstrated the use of Phlegrean tephra in the construction of maritime *pilae* of Roman Mediterranean ports, preserved in excellent conditions even nowadays.

In this paper, we discuss the presence of pyroclastic rocks in the concretes and renders of some Roman buildings at Nora, Sardinia. Preliminary archaeometric investigations (OM, XRPD, SEM-EDS, EMPA, XRF), carried out in collaboration with the Department of Biology, Ecology and Earth Sciences of the University of Calabria, highlight the provenance of most of these materials from the Phlegrean area. These are mainly pumices and tuffs, which were used in large quantities in the *opus caementicium* structures of large public buildings of Nora dated to the Imperial Age. In some circumstances, the materials were also used to improve the waterproofing of the mortar linings of some cisterns, where they represent a component subordinate to prevalent *terracotta* fragments (Secco *et al.* 2020). The import in Nora of such volcanic materials seems primarily targeted at strengthening masonries. This evidence aligns perfectly with the functional destination of the *pulvis* as intended by Vitruvius. Moreover, whereas the presence of locally sourced Phlegrean pyroclastic rocks in mortars appears common at sites near the Gulf of Naples (i.e. Rispoli *et al.* 2019), their use for the construction of above-ground masonries appears less frequent elsewhere. This opens new questions about the constructive reasons the demand for these products was intended for.

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## Food traces from high medieval village of nogara- archaeometric analysis on soapstone fragments

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Archaeometry studies on ancient objects of common use give us insights on the ordinary life in the past. As the preparation and consumption of food represent one of the central aspects of all cultures, the vessels used to prepare and to cook foods are their self objects of investigation not only on the archaeological point of view, but also in archaeometric consideration.

During time different types of vessels have been used for the preparation and storage of food, mainly ceramic and metallic made, but in the Early Middle Ages in the northern part of Italy also soapstonemade vessels became very common. Cooking vessels of soapstone – or steatite, have been used since the late Bronze Age thanks to their strength and their excellent heating properties: they are, in fact, heated quickly, lead warmth evenly, and retain it for a long time. Soapstone is also supposed to be especially suited for cooking organic food ingredients.

In this work, framed in the P.R.I.N. grant “food & S.T.O.N.E.S.”, we focused the attention on 31 of soapstone vessels fragments that presented baked encrustations presumably related with the presence of food remains.

Samples have been collected in the High Medieval village of Nogara (Verona) a centre collocated in the low plain south of Verona that in the centuries 8<sup>th</sup>, 9<sup>th</sup> and 10<sup>th</sup> knows important development and structuration that culminates with the construction of a castle in the year 906 after a Diploma by King Berengario, main objective of the castle was the defence from the incursions of the Hungarians, but certainly there was also the will to give strategic importance to a centre in strong rise.

Stereomicroscopic observations have been carried out on the samples and on a selection of them have been performed SEM-EDS analysis. In parallel to the SEM-EDS observations and analysis we also performed pollen analysis consisting in various chemical treatments of encrustations samples, followed by observations using a transmitted light microscope.

The SEM-EDS shown that in many fragments were present possible traces of bones constituents, an important find, that probably links these vessels to meat preparation. The pollen analysis resulted in finding a low number of palynomorphs, probably due to diagenetic factors, however this did not prevent the finding of single interesting pollens with possible implications in the ordinary usage of these vessels.

In conclusion, the archaeometric approach has paid off by showing us its usefulness in opening windows into the daily lives of people who preceded us in the past, in particular in this case possibly clearing our vision on a part of the food habits of the medieval inhabitants of Nogara.

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**SESSIONE 2:**  
**CAMBIAMENTI CLIMATICI E PATRIMONIO  
CULTURALE**

# Assessing climate change risk to cultural assets by monitoring and quantifying the decay of heritage materials and its environmental constraints

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The EU-funded project Hyperion aims at developing a “decision support system for improved resilience and sustainable reconstruction of historical areas” facing the impact of climate change and extreme weather events, with the aid of sensor-based and modeling tools. An essential part of the project involves the quantification of the current decay of cultural heritage, which may shed light on how heritage materials interact with the environment, react to microclimate stresses, and resist to future climate change in weathering models and simulations.

To that purpose, a novel experimental method based on the long-term monitoring of historical building materials, their deterioration, and the relevant environmental parameters in different geographic contexts, was designed. A selection of stone and wood materials historically used in different European countries are exposed outdoors to natural weathering, in the urban environment of Italy (Padova and Venice) and Norway (Tønsberg). In every location, the same set of samples are exposed at different orientations (North, South, and horizontal plane) and their surface microclimate parameters are being monitored with temperature and moisture sensors. The microclimate data series are compared with the climate data provided by both the complementary weather stations installed in each monitoring site and the stations of the official regional agencies of environmental monitoring. At the same time, the material decay is being measured by monitoring the changes in surface topography/recession, chemical composition, and color of each building material, that is, comparing the maps acquired before and during the exposure tests by 3D optical profilometry, micro X-ray fluorescence, and colorimetry. This investigation is expected to provide a solid base for developing models of future deterioration of cultural heritage and prediction of its endurance in a changing climate.

## Climate risk assessment for strengthening resilience of cultural and natural heritage in Europe

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The risk to cultural and natural and heritage (CNH) as a consequence of natural hazards and impact of climate change is globally recognized. The assessment and monitoring of these effects impose new and continuously changing conservation activities and urgently needs for innovative preservation and safeguarding approach, particularly during extreme climate conditions [1].

The present contribution aims at illustrating the “Risk mapping tool for cultural heritage protection” specifically dedicated to the safeguarding of CNH exposed to hydrometeorological extreme events due to climate change, developed within the Interreg Central Europe STRENCH (2020 - 2022), which development is strongly based on a user-driven approach and the multidisciplinary collaboration among the scientific community, public authorities and the private sector ([www.protecht2save-wgt.eu/](http://www.protecht2save-wgt.eu/)) [2]. The “risk mapping tool” provides climate hazard maps in Europe and Mediterranean Basin where CNH is exposed to heavy rain, flash and large basin flooding and prolonged drought. Risk level is assessed by the elaboration of extreme changes of precipitation and temperature performed using climate extreme indices defined by the Expert Team on Climate Change Detection Indices (ETCCDI) and by integrating data from: i) Copernicus C3S products ERA5 Land (~9 km resolution, from 1981 at monthly/seasonal/yearly time scale) and ERA5 (~31 km – 0.25° resolution, from 1981 at seasonal time scale); ii) NASA GPM IMERG product (10 Km resolution, from 2000 at seasonal time scale); iii) Regional Climate Models from the Euro-CORDEX experiment under RCP4.5 and RCP8.5 scenarios (12 Km resolution, 2021–2050 and 2071-2100) and iv) State-of-the-art observational dataset E-OBS (25 Km resolution, 1951-2016).

The tool allows users to rank the vulnerability of the heritage categories under investigation at local scale taking into account 3 main requirements: susceptibility, exposure and resilience. The functionalities of the “risk mapping tool” are currently under testing at European case studies representative of cultural landscape, ruined hamlets and historic gardens and parks. The application of Copernicus C3S, Earth Observation-based and products and their integration with climate projections from regional climate models constitutes a notable innovation that will deliver a direct impact to the management of CNH, with high potentiality to be scalable to new sectors under threat by climate change. By the achievement of the planned objectives, STRENCH is expected to proactively target the needs and requirements of stakeholders and policymakers responsible for disaster mitigation and safeguarding of CNH assets and to foster the active involvement of citizens and local communities in the decision-making process.

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## Degradation of venetian stone buildings threatened by climate change and land subsidence

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"Acqua alta" (high water) is the term used for the exceptional tide peaks that periodically occur in northern Adriatic Sea reaching their maximum in the Venetian lagoon. This natural phenomenon takes place in autumn and springtime due to the combination of astronomical tide, strong south wind and seiche. The consequence of these events is the flooding of the city of Venice caused by the overflowing of the canals. This represents a critical issue because of the interaction between water and buildings leads to severe deterioration effects over time (i.e. mechanical damages, cracks, erosions, efflorescences, chromatic alterations and biological colonizations). Moreover, high tides frequency has been increasing in Venice in the last 60 years. Since 2010, the situation became dramatic, as the number of high tides events exponentially increased. Particularly, twenty-six high tides ( $\geq 1.10$  m) were recorded between November and December 2019. On 12 November 2019, the city was hit by the highest tide in more than 50 years, with an intense high water peaked at 1.87 meters. Therefore, the climate changes of the recent decades and the last exceptional high water events, urgently require the implementation of new strategies for the preservation of monuments (Trincardi et al., 2016; Cavaleri et al., 2000; Cornerlati et al., 2004).

This study aims to better understand the effects of high water on Venetian historical buildings and to develop strategies for their preservation. For this purpose, six different building sites in Venice and Torcello island were selected to place five of the most common carbonate rocks of the Venetian architecture in order to evaluate the weathering effects caused by high tide. These sites were chosen according to both their different altimetry with respect to the mareographic zero and their different exposure to marine aerosol, winds and wave motion. The selected dimension stones are five more or less pure limestones, with different porosity and texture (Ammonitico Rosso from Verona, Istrian Stone, Vicenza Stone, Aurisina Fiorita Stone) and one crystalline marble (White Carrara Marble). An integrated multi-analytical approach was established to identify and map the chemical-physical deterioration and their related macro- and micro-morphologies due to high tide events. The stones' decay was evaluated during ten monitoring periods by means of different tests and analyses, such as dry weight loss, ultrasound P-waves velocity, colorimetric measures, X-Ray Fluorescence, stereomicroscope observations and recognitions of biological patinas.

Preliminary results show how, even if in slight entity, the main deterioration patterns (e.g. bleaching and discoloration, efflorescences, increase of porosity, loss of material) are already recognizable after a short time of exposure to exogenous agents. The elaboration of the collected data tries to highlight how and to what extent the microclimate, the intrinsic characteristics of the building stones and above all the trends of the high tides influence the development of the deterioration phenomena and the timing of their development, suggesting in some cases a hypothesis of long-term prediction of the evolution of the decay.

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## **SESSIONE 3:**

# **PROGETTO AIAr: STUDIO ARCHEOMETRICO DEL CICLO PITTORICO DI SATURNINO GATTI E TONIMPARTE**

## Results of the II National Research project of AIAr: archaeometric study of the frescoes by Saturnino Gatti and workshop at the church of San Panfilo in Tornimparte (AQ)

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The archaeometric study of the frescoes by the painter Saturnino Gatti (1463-1518) in the apse of the Church of San Panfilo in Villagrande di Tornimparte (L'Aquila) is the subject of the II National Research Project conducted by members of the Italian Association of Archaeometry (AIAr). The research activities were carried out as part of a scientific agreement signed in 2020 among AIAr, Abruzzo Regional Secretariat of the Ministry for Culture and Superintendence of Archaeology, Fine Arts and Landscape for the provinces of L'Aquila and Teramo. Several *in situ* non-destructive investigations and in laboratory analyses on micro-fragments sampled from the different levels of pictorial cycle were carried out thanks to the co-working of 21 Research Groups with more than 60 AIAr researchers involved in the different stages of scientific studies of pictorial materials. Also the environmental conditions in which the frescoes are now preserved were investigated.



The Special Session proposed during the AIAr Padua 2022 Conference represents an important opportunity to illustrate for the first time the results of the pre-restoration diagnostic study. The macro-objectives followed by the working groups coordinated by the Governing Council AIAr members were:

- ✓ to document the state of conservation of the architectural building and the painted surfaces;
- ✓ to analyse microclimate and inertia level of the church with respect to outdoor conditions;
- ✓ to understand the degradation phenomena of the pictorial surfaces and the mural structures;
- ✓ to identify and map non-original materials overlapped to pictorial surfaces during previous restoration works;
- ✓ to characterize the original materials of the frescoes and “a secco” mural paintings;
- ✓ to identify the artistic technique and typical features of Saturnino Gatti and the others painters who worked at the frescoes in San Panfilo.

The results of the multi-disciplinary research, clustered in the 9 oral contributions of the Special Session, allowed to improve the knowledge both from a historical-stylistic and conservation point of view, for supporting the methodological choices in the planning of the next restoration project.

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## Multi-technique characterization of painting drawings of the pictorial cycle at the San Panfilo Church in Tornimparte (AQ)

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In the framework of the A.I.Ar. Research project "*Studio archeometrico del ciclo pittorico di Saturnino Gatti e bottega presso la chiesa di San Panfilo in Villagrande di Tornimparte (AQ)*", we present here some results, obtained by a multi-scale approach based on the employment of different and complementary techniques, i.e. Optical Microscopy (OM), Scanning Electron Microscopy (SEM),  $\mu$ -Raman spectroscopy and Surface Enhanced Raman Spectroscopy (SERS),  $\mu$ -FT-IR, Fourier transform infrared (FT-IR) spectroscopy equipped by Attenuated Total Reflectance (ATR) analyses, X-ray diffraction (XRD) and Thermal Ionization Mass Spectrometry (TIMS). This integrated activity was focused on the characterization of micro-fragments of painting drawings (original and restoration) of the pictorial cycle at the San Panfilo Church in Tornimparte, sampled from specific areas of interest. The study was aimed, on one side, at the identification of the overlapping restoration materials during previous conservation interventions (documented and not), and, on the other side, at the understanding of the degradation phenomena (current or previous) of the painted surfaces and the architectural structures on which they insist. As main results, the identification of blue, black, yellow and red pigments (both ancient and modern) was achieved, other than of binders and protective/consolidating agents eventually present. In particular, in the case of blue pigments, original (azurite pigment) and retouching (Prussian blue) materials were recognized, together with alteration products (malachite and atacamite). Yellow chromium, together with titanium dioxide, was found in the yellow/orange and yellow/blue points, and ochre in this latter. The study of the stratigraphy allowed us to evaluate the number of layers, and the materials (pigments, minerals and varnishes) present in each layer. The presence of a modern resin, probably used during a previous restoration work, and of oxalates due to a biological attack, was also identified. The obtained results are crucial to support the methodological choices during the restoration intervention of the aforementioned site, helping to ensure the compatibility principles of the materials on which a correct conservative approach is based [1,2].

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# Microclimate analysis of the San Panfilo Church in Tornimparte, Italy

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Works of art can be conserved over time if the surrounding environment does not favour the processes of degradation. Microclimatic monitoring is therefore essential to know the environmental situation and possibly to propose solutions or improvements in order to promote conservation (Camuffo, 2019, Bernardi, 2008)

With the aim to investigate the microclimate in the church of San Panfilo in Tornimparte, a measurement campaign was organized from February 2021 to April 2022. The microclimatic quantities (relative humidity and temperature) were monitored in various sites inside and outside the church.

In this work we present the collected data during the campaign: a) temperature and relative humidity measured on a regular spatial grid in 4 days on February 2021, July 2021 (figure 1), November 2021 and April 2022, b) temperature and relative humidity measured during the whole period at 12 positions inside the church and at one outside under the external portico. Additional meteorological data were provided by the meteorological station at Colle San Vito managed by Centro Funzionale e Ufficio Idrografico Regione Abruzzo.

Some quantities were computed from measured data: dew point temperature, dew point spread and specific humidity. Measured and computed data permitted to evaluate the daily and monthly values, their evolution during the year, the daily fluctuations and delay times of the structure. The result discussion allowed to identify the potentially dangerous events for the conservation of the frescoes.

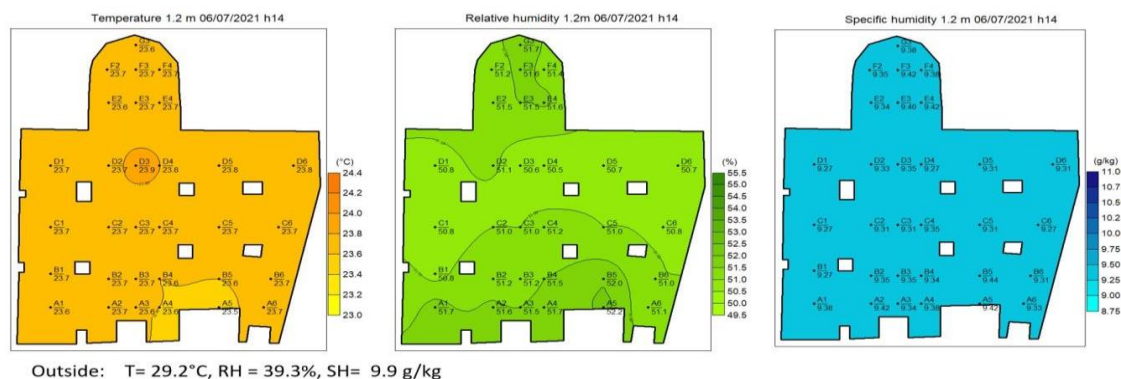


Figure 1: Temperature, relative and specific humidity at 1.2 m from ground level on 6<sup>th</sup> July 2021 at 14.00 CEST.

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## Materials and technique: the first look at Saturnino Gatti

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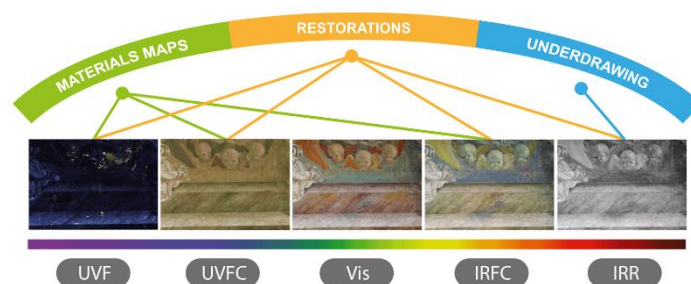
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As part of the study project of the pictorial cycle attributed to Saturnino Gatti at the church of San Panfilo in Villagrande di Tornimparte (AQ), image analyses were performed to document the conservation conditions of surfaces and to map the use of the different painting materials to be subsequently examine by spectroscopic techniques. For images acquisition, radiation sources ranging from ultraviolet to near infrared were used; ultraviolet fluorescence (UVF), ultraviolet false colour (UVFC), infrared reflectography (IRR), infrared false colours (IRFC) and optical microscopy (OM) were carried out on all the panels of the mural painting of the apsidal basin. The Hypercolorimetric Multispectral Imaging (HMI) technique was also applied.



The integrated analysis of the images made it possible to distinguish the portions affected by retouching or remaking and to recover the legibility of some figures that showed chromatic alterations. The IR reflectography, besides highlighting the portions with loss of materials and restorations, has emphasised the presence of the underdrawing reported with the *spolvero* technique. The UV fluorescence led to a preliminary mapping of organic and inorganic materials that had a characteristic induced fluorescence, indicating the presence of a binder in correspondence with the original azurite coating, and the massive use of zinc white for painting retouching.

All the collected data have made it possible to make a better iconographic interpretation, as well as to allow an accurate selection of the areas to be subjected to spectroscopic analysis both *in situ* and on sampling.

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## Identifying original and restoration materials through spectroscopic analyses on Saturnino Gatti mural paintings: how far a non-invasive approach can go.

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In the frame of the AIAr project on Saturnino Gatti, the mural paintings in the church of San Panfilo in Villagrande di Tornimparte (AQ) were investigated through spectroscopic non-invasive techniques, namely Optical Digital Microscopy (OM), X-Ray Fluorescence spectroscopy (XRF), Fibre Optics Reflectance Spectroscopy in the UV-Vis-NIR range (FORS), Fourier transform infrared spectroscopy in External Reflection mode (ER-FTIR), and Raman spectroscopy. For all the applied techniques, points for investigations were selected on the bases of image analysis results and the few available documentation on previous intervention, with the aim of characterizing both original and restoration organic and inorganic materials.

The synergic application of analytical techniques exploiting different radiation frequency allowed to obtain complementary information for a complete picture of the palette, main alteration products and organic substances, consisting in rather ubiquitous lipid materials (oils and/or waxes) and less widespread resins. Moreover, spectral evidences of proteinaceous materials were observed in specific points. The identification of modern compounds permitted the individuation of restoration areas, confirmed by the comparison with imaging results. Particular attention was paid to white pigments, golden details and to the different hues of greens and blues, especially for the vault background. It's worth noting that some green and blue pigments were strictly related to the presence of high signals of zinc, permitting to discriminate them from the original ones.

This analytical protocol left only very few ambiguities, clarified by sample laboratory analyses, thus minimizing the number of taken samples to the dubious points.

## Photogrammetric 3D restitution, raking light documentation and mapping of degradation phenomena of the wall painting in St. Panfilo Church at Tornimparte (AQ, Italy)

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The apsidal conch of San Panfilo Church (Tornimparte, Italy) is a semicylindrical chamber decorated by Saturnino Gatti in 1495. Wall paintings in the lower part consist of five panels displaying some moments of the Passion of Jesus, whereas on the vault are depicted God the Father along with Angels and Blesseds in Paradise.

Documentation of this National Monument is a fundamental step to correctly perform the restoration. Different modalities are possible and in the case of large surfaces, 3D photogrammetry could be a valid and useful approach to obtain the 3D model that can be used as a working basis for inserting points of analysis, cleaning test areas, restoration interventions, mapping of any previous interventions and, finally, for viewing the paintings also on platforms for sharing three-dimensional models, such as Sketchfab. In the case of Tornimparte project, 3D photogrammetry was performed through a Nikon D5300 digital camera on a photographic stand, led lamps on tripods and Total Station Topcon GPT 7005 to gather the GCP coordinates. The 156 acquired images were then processed by Agisoft Metashape for obtaining the 3D model that could be interactively seen online<sup>1</sup>. The five panels and the vault of the apsidal conch were exported from the aforementioned 3D model as orthomosaics and then imported into a Computer-Aided Drafting (CAD) software for a damaging diagnosis based on a monument mapping procedure of materials, weathering forms and damage categories.

Moreover, raking light photography documentation is a simple tool used to study the painting surface, to document retouching and losses in paintings. The raking light image was performed through a Nikon D7000, illuminating the painted surface through a LED light from the left to the right side, at different angles depending on specific requirements. The whole surface of the apsidal conch was examined. The raking light images obtained showed conservation problems (surface warping, colour detachment and retouching) and provided indications about the painting techniques (incision marks and also "giornata" boundaries, markers of the fresco technique).

The examined surface was almost entirely covered by mural painting (ca. 90%) and restoring mortars (ca. 10%), the latter mainly located in the lower part of the apsidal conch. Several degradation phenomena, attributable both to the executive technique of wall painting and microclimate conditions, can be observed. In particular, the mural painting was interested by the intense microfissuring, lacune and discolouration (bleaching, moisture trap and deposit), as well as convex deformation due to water uptakes with consequent efflorescence and sub-efflorescence phenomena. Graffiti (scratching) represents the most important anthropogenic damage.

According to the overlapping of weathering forms and their intensities, most of the examined surface are interested by a moderate to very severe damage categories with a progressive damage index ranging from 3.0 to 3.7 (medium value 3.2).

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<sup>1</sup> <https://sketchfab.com/models/88de7bbf64944408ad5fdda1d6eda141>

# Infrared thermography and sonic investigation for the analysis of decaying frescoes of apsidal conch of San Panfilo Church (Tornimparte, Italy)

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Historic masonry are complex systems, often characterized by different constituent materials and construction techniques. The quality assessment of these structures requires considering many aspects, from the historical analysis to the study of materials, including the mechanical characterization of the walls. Non-Destructive Testing (NDT), such as Sonic Pulse Velocity (SPV), hygrometric tests (HT), and Infrared Thermography (IRT) are particularly suitable for inspecting Cultural Heritage items due to their non-invasive feature, speed of execution, and meaningful results. The SPV and HT tests were performed on the frescoed walls of the apsidal conch of San Panfilo Church, selecting on 4 areas, characterized by fractures, infill of the underlying masonry, and near openings (doors, windows). SPV exploits the propagation in the sample under test of elastic compression waves generated by a short elasto-mechanical impact on the surface, to investigate in-depth the materials. The SPV tests were performed through the Novasonic U5200 CSD of IMG Ultrasuoni S.r.l., in indirect/surface transmission. The HT were carried out using the Protimeter instrument (MMS2), set on the "pin mode", to measure the surface moisture of the painting layer. The instrument measures the moisture content in WME%, in non-conductive solid materials. Both SPV and HT were performed according to a specific survey scheme on the same regular grid of points. Collected data were processed through GIS software, for the graphical representation of velocity and humidity distribution. Images in false colour allow emphasizing the different properties of the masonry. In SPV maps, low speeds correspond to detachments, voids, and discontinuities of the plaster, while high speeds correspond to a good coherence of the painting layer and masonry. The use of the same grid allowed comparing the humidity distribution with the sonic velocity. The maps in false colour showed conservation problems (surface warping, painting layer detachments, humidity distribution) and provided indications about different properties of the infill wall.

The IRT tests were performed in both passive and active approaches (see [1]) by using a 480x640 thermal camera (7.5 – 13  $\mu\text{m}$ , T660 FLIR System). Passive IRT was used to image frescoes' support wall and detect potential anomalies. The active IRT was applied in some specific area to acquire information at different depths. An 8-LED system driven by a pseudo-noise voltage signal was used as heating source [2]. Potential detachments, part of the wall structure, a presumably humid area below the wooden roof, and clues on the nature of some pigments were retraced.

The integration of SPV, HT, and IRT results demonstrated being a smart way to inspect artistic and architectural heritage and this test represents an important starting point to deepen the knowledge of the structures and direct further and targeted investigations and restoration interventions.

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## Characterization of soluble salts on the frescoes by Saturnino Gatti in the church of San Panfilo in Villagrande di Tornimparte (L'Aquila)

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During the archaeometric study of the frescoes by Saturnino Gatti in the church of San Panfilo in Villagrande di Tornimparte (L'Aquila), 11 samples of powder and fragments were taken from different panels of the fresco in order to characterize any degradation produced by the presence of salts.

The samples were taken in areas where there were evident detachments, exfoliation and saline crystallization (efflorescences). Precisely, 9 samples were taken from panels A, C, D and E and two samples taken from the top part of the vault.

The characterization of the samples was performed using two analytical methods such as Ion Chromatography (IC) and FT-IR / ATR infrared spectroscopy. Ion chromatography was used for the quantification of the main ions and to evaluate the presence of soluble salts. Measurements of cationic ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{NH}_4^+$ ) and anionic ( $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ) species were performed using an ICS-1000 HPLC system equipped with a conductivity detector. The anion analysis was performed with a column of Ion Pac AS14A using  $\text{Na}_2\text{CO}_3$  8 mM/ $\text{NaHCO}_3$  1mM, flow rate = 1.5 mL/min and for the detection a conductivity system equipped with anionic self-healing suppression ULTRA (ASRS-ULTRA). While, the determination of the cations was carried out using a CS12A column (Dionex) and 20mM of methanesulfonic acid (MSA) as eluent at a flow rate = 1.5 mL/min and for the detection a conductivity system equipped with an ULTRA self-healing cationic suppressor (CSRS-ULTRA).

Infrared spectroscopy has been used to characterize the mineralogical phases. The apparatus used is a Nicolet 380 (Thermo Electron Corporation) instrument coupled with ATR accessory Smart Orbit equipped with a diamond crystal. The spectra have been acquired in the range  $500\text{-}4000\text{cm}^{-1}$  at a resolution of  $4\text{cm}^{-1}$ .

The results indicate that some, but not all, samples show high concentrations of sulfate and calcium, attributable to probable efflorescence consisting of newly formed gypsum. Furthermore, a good correlation between sodium and chloride was observed, indicating the presence of an efflorescence composed of newly formed sodium chloride. The calcite and silicate bands attributable to the substrate were observed by FT-IR and also gypsum bands were highlighted confirming what observed by the IC technique.

## Minero-petrographic characterization of the frescoes by Saturnino Gatti: micro-stratigraphic analyses on thin sections

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The petrographic characterization of the frescoes by Saturnino Gatti within the church of San Panfilo in Tornimparte (AQ), was carried out on a total of 16 cross-section samples, taken from different areas: the original phase; attributed to subsequent periods (XVII century); past restoration works (localized also thanks the preliminary in situ analyses). The sections were taken in such a way as to highlight the entire sequence of materials: from the support mortar to the painted surface. The petrographic analysis on a thin section highlighted the presence of at least three layers: a preparation layer consisting of lime plaster and sand, a layer of pigmented lime, probably applied with the fresco technique and an additional pigmented layer. The colour palette used includes black, red, yellow, and blue. In most of the samples the black, red, and yellow pigments appear linked with the fresco technique, while the blue pigment is applied *a secco*. In the sections where a blue layer appears, it appears that this was applied at a time when the surface was already dry or semi-dry. In one sample, called SG40, we note the presence of two phases of painting, probably the result of a rethinking or a makeover / restoration.

In addition, the 16 thin-sectioned specimens were examined using cathodoluminescence microscopy,  $\mu$ -Raman, SEM-EDS. Two more samples, (SG 35 and 36), were also analyzed through EPR (Electron Paramagnetic Resonance).

All the samples were characterized by a base layer of plaster (layer A), and up to 5 pictorial layers (B, C, D, E, F). The layers containing ochre, raw sienna and carbon black are related to the original fresco.

Red and yellow ochres were found as the main pigments in the deeper pictorial layers in most samples (layers B, C: samples SG9-SG15, SG16, SG17, SG25, SG39B, SG46 and SG47). These layers were likely applied *a fresco*, as indicated by the analysis on the SG16 sample. Carbon black was also detected in many of these inner layers. External pictorial layers (C-D) presented often more recent pigments of different colour, sometimes applied *a secco*. E.g., in SG16 malachite was detected, and maybe lead chromate, in SG25 ultramarine and titanium white, in SG39B ultramarine and azurite, in SG40, ultramarine and lead white. In many samples, the occurrence of calcium sulphate, as likely alteration product, was inferred by the presence of sulphur and calcium in SEM-EDS analysis. Also, the presence of polymeric organic compounds was supposed by Raman analysis in two samples (SG40, SG25). Worth to note that, SG9 and SG10 have a layer containing calcium phosphate (bone black?), zinc oxide and probable K-alum is present as probable material from later interventions.

In SG35, the EPR analysis detects signals due to carbon black radicals and copper alteration products; in SG36, the EPR signal due to Mn (II) in calcium carbonate was detected.

## The study of the organic materials

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This work relates to the analysis of samples collected from the painting with the aim of characterizing original binders in order to define the executive technique and to identifying paint materials used for pictorial reintegration/restoration of the surfaces. Sampling areas were carefully chosen based on multiband imaging and single spot techniques results.

To this aim a multi-analytical approach has been used, which entailed the use of Optical Microscopy (OM), Fourier Transform Infrared Spectroscopy (FTIR), Gas- Chromatography Mass Spectrometry (GC-MS), and analytical Pyrolysis coupled with Gas- Chromatography Mass Spectrometry (Py-GC-MS).

Often the samples analysed present a complex stratigraphy, with several layers superimposed to one another. As an example Fig 1a shows the OM image of a sample collected from Panel E, showing a blue layer over a white background. When possible, paint layers were mechanically detached and analysed separately.

The analytical approach implemented revealed a variety of organic materials, both of natural and synthetic origin. As an example Figure 1b shows the chromatogram relative to the presence of a terpenoid plant resin found in a sample collected from the vault, as it can be inferred by the detection of dehydroabietic acid (DHA) and 7-oxo-dehydroabietic acid (7-oxo-DHA).

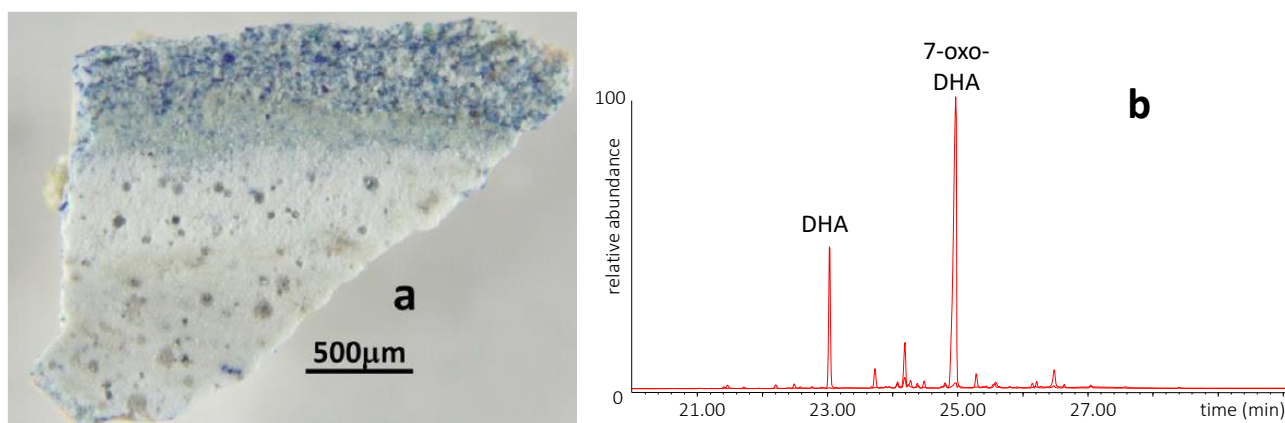


Fig 1. A) OM image of a sample collected from Panel E. b) chromatographic profile of the molecular markers of a plant resin.

The main results obtained will be presented and commented in relation to other investigations carried out with non-invasive and complementary techniques

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## **SESSIONE 4:**

# **ARCHEOMETRIA E QUESITI ARCHEOLOGICI**

## An interdisciplinary approach to the study of graphite-painted pottery from the Prehistoric Balkans.

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Graphite decoration on ceramic vessels is a distinctive phenomenon spread over most of the Balkans during the 5th millennium, becoming one of the dominant forms of decoration for Neolithic and Chalcolithic material cultures in the region for over 1000 years. This technique consists of applying a slip containing graphite to vessel surfaces either before or after the firing. Traditionally, archaeologists have closely related graphite decoration with the rise of metallurgy. However, the emergence and development of this decorative technique on pottery remain unclear, mostly due to the lack of systematic technological investigations of this technique.

An interdisciplinary approach employing thin section petrography, XRPD,  $\mu$ -XRD<sup>2</sup>,  $\mu$ -Raman, SEM and carbon isotope analysis was applied to a selection of ceramic samples decorated with graphite from different areas of the Balkans, including Serbia, Romania and Bulgaria. The results give important information about the procurement and processing of raw materials as well as essential insights into the pyrotechnology related to graphite painted pottery. Very importantly, the combination of  $\mu$ -XRD<sup>2</sup> and  $\mu$ -Raman analysis revealed that both well crystalline natural graphite and the application of pigments containing poorly crystalline carbon were used, pointing to the possible existence of at least two different manufacturing recipes to produce this type of decoration. The initial results of our research illustrate well the advanced pyrotechnology knowledge reached by these populations at that time.

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## Implementation of an integrated methodology to assess provenance of ancient ceramic raw materials

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Chemical and mineralogical composition, provenance and production process of ceramics are the main investigation topics in archaeometry. Clay-dominant deposits are a common source of ceramic raw materials. Each deposit is characterized by a fingerprint. However, comparing the chemical-mineralogical composition of the ceramic fragment with that one of a clay, without considering the chemical and physical transformations during firing process is extremely misleading. For this reason, we implemented a methodology that could become a best practice in the identification of raw materials used for ceramic productions. Several clay-dominant deposits outcropping in Basilicata and belonging to different geological formations were sampled and analyzed to identify the types of raw materials most suitable for the vast production of ancient ceramics, to locate the most likely sources of raw materials, to formulate hypotheses on the circulation of them and/or artifacts, and to discriminate local production from possible "imports". With the help of a Territorial Informative System in a GIS environment and a relational database of raw materials, it was possible to identify the outcrops of clay deposits to sample and compositionally characterize. Illitic clays are very abundant in Basilicata and constitute the prevalent clay deposits. Experimental firing tests on clay samples were performed at different temperature steps. The final color of the firing test specimen was coded using Munsell Tables. TG-DSC analysis were carried out on clay samples and correlated to firing tests. The comparison with the composition of medieval ceramic fragments selected for the study of provenance from the archaeological sites of *Satrianum*, Castello di Moliterno and Santa Maria d'Anglona (Basilicata, southern Italy) was carried out taking into account the trend of the mineralogical composition of clay deposits as temperature increases, as deduced from the firing tests and TG-DSC curves, and contextualized in the landscape scenario within which the production centers operated. This methodological approach has allowed to return an articulated picture of the presence of productions in the region, that during the Middle Ages circulated on a regional as well as extra-regional scale thanks to a network of land and river communication. The integration of the results of the archaeological analysis, the firing tests, the geolocalization of the sampled outcrops and the identification of the geographical relationships with the archaeological sites have made it possible to circumscribe any local productions, to define the circulation of raw materials and know-how in the diachrony for a territory such as the Basilicata region, rich in archaeological evidence and densely populated from prehistory to the Middle Ages.

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## Improved chemometric approach for the analysis of XRF data. The case of the reverse glass painting from Lipari collection

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The Aeolian cultural heritage preserves countless testimonies of the past that have passed through six millennia of history in a wide variety of forms and cultural expressions. Alongside its more distinctive archaeological asset, the Aeolian Archeologic Park of Lipari (Italy) preserves a valuable set of artworks, which are related to a less-known 'popular' figurative heritage [1]. It is an assemblage of small glass foils decorated with the technique of reverse painting, datable between the end of the 17<sup>th</sup> and the 18<sup>th</sup> century. Reverse painting on glass is an old decorative technique used since the Roman time consisting in applying a cold paint layer on the reverse side of a glass support. The compositional study of glass support can provide useful information about the origin of the painting [2].

Here, an X-ray fluorescence spectroscopy (XRF) study combined with a multivariate approach that allow us to define the best way to detect compositional differences and similarities among the glass supports is reported. The Principal Component Analysis and Cluster Analysis were applied both on normalized spectra and on normalized peaks area in order to establish the chemometric approach with the highest grouping ability. The collection shows a large spreading of provenience and dating of the items. In consideration of the current classification based solely on stylistic criteria, we applied a multivariate analysis on the XRF measurements data set to find a more objective classification criterion based on the elemental composition of the glass support.

Results showed that the analysis of normalized area provides the most reliable grouping based on the different elemental composition, without troubles coming from back ground or peak shape distortions. Thanks to the proposed approach the glass supports can be divided into different typologies but the assignment of these typologies to the current assignment to an artistic style or a given period is not straightforward, due to reuse or original erroneous attribution.

This study was carried out in the framework of the Scientific Agreement between IPCF-CNR, the Department STEBICEF of University of Palermo and the Aeolian Archeologic Park of Lipari.

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# The production of opaque red glass during the 2<sup>nd</sup> century AD in Rome. Archaeometric study of the red glass *sectilia* from the imperial villa of Lucius Verus

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Through a multi-analytical approach, this work aims to shed light on the production technology of opaque red glass and its different hues (from orange to brown), during the 2<sup>nd</sup> century AD in Rome. Although a vast literature and several chemical analyses are devoted to opaque red glass, the technological aspects concerning its manufacturing are still not fully understood. In particular, the Roman period is represented by only a few depth archaeometric investigations, while the lack of glassmaking written source makes it difficult to have a clear vision on the production of this complex colour.

The present research studied the opaque red glass *sectilia* from the *opus sectile* decoration of the villa of the Emperor Lucius Verus. Due to the presence of numerous red hues, the abundance of pieces of opaque red glass and their secure date, these samples represent a rare opportunity to explore the Roman glassmaking technology during the 2<sup>nd</sup> century AD<sup>1</sup>.

40 samples were grouped by hues and characterised through the colourimetric measurements and UV-VIS spectroscopy; EPMA and LA-ICP-MS were performed to define the chemical composition of the different red hues, identifying the probable raw materials used;  $\mu$ -Raman, XRD and FEG-SEM showed the nature and the main characteristic of the colouring agents, responsible for the different red shades.

The analyses identified two main colouring techniques to make opaque red glass during the 2<sup>nd</sup> century AD. Furthermore, it was established the presence of specific recipes employed to make five different red hues, which included different raw materials, distinct chemical compositions, well-engineered thermal treatments, and two different colouring particles (metallic copper particles, and cuprite crystals). These results provide new information on the sophisticated production technology of opaque red glass during the 2<sup>nd</sup> century AD and underline the extraordinary skills of Roman glassmakers to control the formation of this demanding colour. Moreover, this work reveals that a multi-analytical approach affords a clearer and more complete understanding of the production technology of opaque red glass during the Roman age.



Figure 1: Glass *sectilia* of different red shades collected from the opus sectile decoration of Lucius Verus villa.

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## Plaster radiocarbon dating of St. Philip Church, Hierapolis (Denizli, Turkey)

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The possibility to date an aerial mortar by radiocarbon relies on calcite that results from the reaction of calcium hydroxide with the atmospheric CO<sub>2</sub> during the hardening of the material. Considering the critical aspects encountered in <sup>14</sup>C dating of ancient mortars and the importance of sample selection, great attention is paid to characterization of mortar samples to estimate whether the samples can be good candidates for dating. Usually, the presence of organic inclusions in the mortar mixture is used to have more reliability of the radiocarbon measurement. Such components are straw, chaff, horsehair, dung, coal, iron filings, which discovery in the mixture cannot be due to intentional additions, e.g. the residues of the burning; they may have been added to the paste to give specific properties to the mortar, e.g. addition of straw.

Some plasters used for the decorations and the covering of the walls of St. Philip Church, in the archaeological site of Hierapolis (Denizli, Turkey), are characterized by addition of straw. Mineralogical characterization of plasters allows the identification of raw materials and production techniques. The suitable archaeological and stratigraphic studies of different ancient structures allowed to evaluate the datable component of these materials [1, 2].

Moreover, the knowledge of archaeological context and the raw materials and techniques for production of mortars/plasters has highlighted more contamination sources and lime mortars not datable.

<sup>14</sup>C experimental dating was carried out by collecting organic residues (straw fragments) and measuring the their <sup>14</sup>C concentration.

After the mechanical selection of straw fragments, the treatment was carried out at INFN-LABEC laboratory in Florence, where a new experimental set-up, aiming at reducing the necessary mass of graphite for the dating was build.

Thanks to the Lilliput graphitization line [3], it is possible to deal with samples with few tens of µg of carbon and proceed to <sup>14</sup>C measurements.

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## Going back to Pompeii in 79 AD: a leap in time to uncover sustainable craft tradition by studying the ancient ceramic productions

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Pompeii is one of the most important archaeological sites of the world and represents an invaluable source of archaeological data. Destroyed by the 79 AD Vesuvius eruption, the site preserves a well-constrained time-frame of ancient Roman habits, culture, and technology. A useful tool for dating events, revealing production technology, and tracking commercial pathways is represented by pottery. Recent discoveries in Pompeii proved that, at the moment of the eruption, pottery was being made in two workshops located near the two main gates of the city. Close to *Porta di Nocera* oil lamps and little pots were produced, while in *Via dei Sepolcri*, next to *Porta Ercolano*, a workshop was specialized in the production of thin-walled pottery (Grifa et al., 2021). In these contexts, shaping facilities and kiln structures were found along with ready-to-use raw materials, unfired, fired, and overfired shaped ceramic vessels.

A multi-analytical approach including chemical, mineralogical, petrographic, and spectroscopic techniques was applied to study the materials from these two workshops, revealing peculiar aspects of the ancient ceramic craft. Data provided a complete overview of the entire production cycle, from the preparation of the ceramic vessels to the firing operative conditions, as well as on the economic aspects of raw material procurement. Interestingly, archaeometric results revealed a shared ceramic practises. Analyses of raw materials and unfired vessels precisely revealed an exploitation of Apennine clays from the surroundings of *Salernum*. In fact, based on current data, there are no deposits of clays in the vicinity of Pompeii such as to justify these productions. It is easy to imagine that clay was supplied from the port at *Salernum* to Pompeii via well-established maritime routes inside and outside the Bay of Naples. As a matter of fact, at that time, transport by ship was much more sustainable and economic than land transport, which would have required more resources and time for the same amount of material.

This raw material is a CaO-rich (10-18 wt%) marine sediment with a clay fraction composed of illite-smectite mixed-layers, kaolinite, and chlorite, along with calcite, quartz and minor feldspars. Apennine clays were mixed with volcanic temper represented by sands from the Vesuvian coastland. The vessels were slipped by immersion in a mixture of red ochre and water.

If unfired fragments well reflect the original mixture of marine clay and volcanic sand, the fired ones show evident pyrometamorphic changes with the lack of hydrous phyllosilicates and presence of newly-formed high-temperature phases, which suggest firing temperatures ranging from 800 to about 1000 °C. This difference was likely due to a wide thermal gradient in the furnace during firing or poor control of firing conditions in different production cycles.

Surprisingly, Pompeian ceramics contain clinopyroxene and Ca-plagioclase, but they did not show the occurrence of detectable gehlenite. Analyses on experimental replicas are suggesting us that such a phase is likely absent because of post-depositional alteration phenomena experienced by the vessels.

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## Cold cases from MIS 3 (60-25 ka). Why detailing archaeometric investigations enhances research transparency and sustainability

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In the last 10-15 years archaeometric investigations became common ground in Functional Analysis – FA, the methodology that studies stone tool use – pushing FA towards developing analytical pathways that improve both imaging and physical-chemical characterization, in an attempt to promote standardization and reproducibility of the analytical processes. We report on the analytical refinements applied to the study of the biogenic residues retrieved on the used surfaces of macrolithic tools – non-flaked industry – dated to the MIS 3 (60-25 ka) from a selection of sites across the Pontic steppe. Our main research question is whether simple stones (often just pebbles) were involved in the processing of plants either to extract starch or to obtain fibers or even dyes.

Our experience shows that the relevance of the objects' selection, their biography, and the sampling strategy(es), all influence the further applicability of sound analytical techniques and the different experiment set-ups. The careful and informed approach to these steps and to the contingencies that can be encountered – from the excavation and lithic industry management, to the museum conservation strategies and to the laboratory/equipment constraints, just to list a few – is relevant to enhance the sustainability in archaeometric investigations and the research transparency according to the Singapore Statement for Research Integrity.

In order to achieve a balance in the attempt to attribute equal importance to qualitative (more humanistic) and quantitative aspects (more STEM) in our reasoning, we develop a multidimensional approach that provides us with congruent results that drives us towards proposing the best explanation that answers the research question in different “cold cases”.

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## Red and black surfaces on Apulian red figure pottery. Raw materials and manufacturing practice by Raman and SEM-EDS investigations

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Black gloss and red surfaces on 4th century BC Apulian red figure vases, from sites among the most relevant in Apulia (Italy), are studied by means of Raman spectroscopy and Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy. The aim is to highlight manufacturing procedures and raw materials. The ultimate archaeometric goal is to recognize the interconnections between Apulian and Attic production so as to validate the hypothesis of the Apulian production being independent from the Attic one in terms of raw materials, but also of manufacturing and technology.

Results show that, unlike Attic production, two different clays have been employed for black gloss and ceramic body. The black gloss proves composed of the finest fraction of terre rosse – very common all over Apulia, – with no organic material added to the clayey suspension. Traces of carbon found on the surfaces can be actually ascribed to fly ash deposited during the firing process.

Regarding red surfaces, the results highlight that two more methods, besides red engobe, were used by Apulian potters to color red the whitish/grayish surface of vases realized with local raw materials, namely the addition of miltos and a clayey layer enriched in Fe oxides.

A multi-step firing, reaching the maximum temperature of about 900 °C, can be deduced from the different mineralogical phases identified through Raman.

The possibility of gaining information on the hematite particles size from the position of its characteristic Raman bands is successfully investigated, allowing to get clues on the presence of a surface layer in a nondestructive manner by comparing the particle size on the surface and in the ceramic body.

## Man and territory. The contribution of petrographic characterization to the study of mobility in Mahal Teglinos, Eastern Sudan

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The site of Mahal Teglinos (K1) lies in a secluded valley located to the North-East of Jebel Taka, a granite inselberg near Kassala, in Eastern Sudan. The archaeological area is investigated by the Italian Archaeological Expedition to the Eastern Sudan (IAEES) directed by Professor Andrea Manzo.

The artefacts considered here are the Gash funerary stelae and the Jebel Mokram macro-lithic tools, both made out of granite, that are particularly abundant on the site during the Kassala Phase (IV - I millennium BC) (Fattovich, 1993).

During the 2019 field season, the IAEES began collecting granite geological samples with the aim of identifying the specific sources exploited during this phase. The extraction points were chosen on the basis of their proximity to the excavation trenches to understand whether the granite was locally sourced or not. The samples were taken from two different rock boulders in Mahal Teglinos (GEO1; GEO2) and from a modern quarry known as Jebel Tareg (GEO3; GEO4; GEO5; GEO6), the granite outcrop closer to the site (Manzo, 2020).

The criteria used to compare archaeological artefacts with the geological samples are petrographic ones: the observation of the colour and grain with the naked eye, and microscopic observation via polarizing optical microscopy (POM) of the polished cross-thin sections to observe the grains and recognize the minor minerals (muscovite and/or sericite, garnets, zircons and biotite) and the myrmekite - because their presence or absence is helpful in linking finished products to their sources (Mackenzie et al., 1982).

The analysis of the Mahal Teglinos area granite sources highlights two main points:

- the heterogeneity of the granite sources exploited during the Kassala Phase. The results suggest proximity occasionally influenced the choice of the source to be exploited. However, for some unknown reason, the granite from the Jebel Tareg quarry was sometimes preferred. Finally, for some artefacts, the source of the raw material may have been much further from the site, as their samples are different from both the Mahal Teglinos and Jebel Tareg samples;
- despite the chronological and cultural differences between the Gash Group and Jebel Mokram Group, these communities exploited the same granite sources at different times, consequently the memory of their location was not lost across the centuries.

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## Archaeometric characterization of Roman *terra sigillata* pottery from Arezzo, Italy

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A full characterization of 25 selected samples of roman *terra sigillata* pottery fragments from Arezzo is here reported. Analysed potsherds date back to a period between the middle of the 1<sup>st</sup> century BC and the middle of the 1<sup>st</sup> century AD. The samples (Fig. 1) come from workshops and drains found during the excavations carried out in the second half of the 19th century in the complex of Santa Maria in Gradi and surrounding areas of the city of Arezzo. Most of the analysed fragments belong to the famous ceramic workshop of *Marcus Perennius*, while others are signed by other important producers such as *Cornelius* and *Ateius*. The pastes characterization was carried out by integrating macroscopic observations with petrographic, mineralogical and chemical analyses. Optical microscopy on thin sections was used for petrographic characterization. XRPD was employed for qualitative and semiquantitative mineralogical analyses. SEM-EDS and Raman spectroscopy have been used to characterize the chemical composition of pottery coatings. All the examined pottery samples showed a well purified pastes with very similar mineralogical compositions, both in the bodies and in the coatings. From the set of mineralogical phases detected, the firing temperatures have been estimated between 900°C and 1000°C.

The results of the Raman and SEM analyses on coatings, showed a similitude in composition between Italic and south Gallic sigillata slips as well as the use of calcareous clay for the bodies. In both cases, a K-rich illitic clay with a Fe content around 10-15% (in wt%) was used to elaborate the slips. However, the slip composition between these two production areas shows a systematic difference in magnesium content, which modifies the vitrification temperature of the slip and leads to an increase of the firing temperature by about 50-70°C for the Gallic production.



Figure 1: Two selected samples of *terra sigillata* fragments from Arezzo.

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## Production technology and conservation state of ancient Roman coins from *Cumae* (southern Italy)

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The present research was performed on a selection of ancient Roman coins (*antoniniani*) coming from a treasure found in a well dug in a volcanic tuff and pertinent to the Roman necropolis area of ancient *Cumae* (modern Cuma, southern Italy) during archaeological campaigns carried out by the Centre Jean Bérard. The selection of twenty samples was based on cognitive criteria of the coins chosen from a limited chronological period between 261 and 271 AD, with the aim of verifying their composition and the processing technology of the metal alloy in this historical period. In addition, the mineralogical characterization of the corrosion and alteration products enabled to increase the knowledge of the state of decay to implement adequate conservation strategies.

A multi-analytical approach, consisting of non-destructive techniques, such as video microscopy, SEM-EDS, pXRF, Raman spectroscopy,  $\mu$ -CT 3D imaging was implemented.

While considering the surface enrichment of silver, a semi-quantitative chemical characterization of metal alloy was carried out. The pXRF analysis showed that the Roman mint prepared the metal with argentiferous bronze alloy. A group of coins presents a low concentration of the precious metal, in agreement with the financial crisis of the Roman Empire of the third century. Another group of two coins shows a higher silver content, in agreement with their lower chronology and, in one case, with the Milanese mint standards. More in-depth micro-destructive analyses will allow to determine the chemical standards of coinage.

The compactness of the coin observed with  $\mu$ -CT is consistent with the minting technique by beating, which eliminates the bubbles formed during the solidification process. The latter technique has proved to be particularly useful for identifying legends and coin types no longer legible due to wear. As far as the conservation state is concerned, some typical corrosion patinas in post-depositional conditions were identified (such as malachite, cuprite). Worth to note that, the particular conditions of the earthen condition into the well caused in some cases more aggressive patinas with rich in Cl and S. These results are consistent with the alteration products of ceramic materials found in the same context. Finally, the presence of carbon black, often attributed to burnt organic substance, suggests that the coins came in contact with material of organic origin.

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## Technological evolution of hydraulic binders in Padova from the Roman period to the Renaissance

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Located in north-eastern Italy, the city of Padova gained importance in the Roman Republican age, becoming one of the wealthiest cities of the region during the Imperial period. After the fall of the Roman Empire, the city flourished in the Middle Ages, also thanks to the establishment in 1222 of one of the first universities in Europe, becoming a centre of crucial artistic importance during the 14<sup>th</sup> Century, as testified by the occurrence of several cycles of mural paintings by the most renowned artists of the time, recently recognised as UNESCO World Heritage. After the conquest by the Venice Republic in the 15<sup>th</sup> Century, the city experienced several phases of building expansion during the Renaissance, testified by the construction of one of the better-preserved systems of city walls in Europe.

Due to the unfavourable geomorphological characteristics of the territory, characterized by unstable, heavily water-saturated soils, ancient building technology always pushed for optimizing the composition of structural materials and the layout of architectural structures, to guarantee proper structural stability over time for public buildings and infrastructures.

In this contribution, a comprehensive characterization study was performed on the structural binders employed for the construction of the most important structures of the city and its surroundings, including public Roman buildings (theatres, amphitheatres, temples, thermal baths) and several Medieval and Renaissance defensive systems and religious complexes. Furthermore, Roman maritime concretes were analysed, employed to build marine infrastructures in areas of the Venice Lagoon within the influence of the city.

The materials were studied through an integrated multi-analytical approach developed by the research group, including X-ray powder diffraction (XRPD), magic angle spinning nuclear magnetic resonance spectroscopy (MAS-NMR), and scanning electron microscopy coupled to energy-dispersive microanalysis (SEM-EDS), correlating the compositional and microstructural information with the results obtained from mechanical tests. Furthermore, the reaction kinetics of the binding systems were parametrized through dedicated studies on experimental replicas of the determined mix designs.

The results obtained demonstrated constant research by the ancient crafts of the best hydraulic performance of the structural binding composites, to deliver adequate static behaviour to the structures, without relevant loss of strength due to water percolation and deterioration. This was obtained during Roman times by employing a local, highly reactive volcanic pozzolan, conferring excellent mechanical performances to the manufactured concretes, widely surpassing mean compressive strength values determined so far in materials of similar nature. Then, after the end of Roman domination, a natural hydraulic lime obtained by calcination of local marly limestones started to be employed, with a period of crossover with the previous technology in early Medieval times, and became the key material for the construction of the Renaissance city walls during Venetian domination.

The detailed studies on the crystal-chemical nature of the hydraulic phases, complemented by a parametrization of the degree of polymerization of silica and alumina in the binding systems, clearly demonstrated from a structural standpoint the advanced degree of optimization of the binding materials, giving novel insights on pozzolanic and hydraulic systems useful to optimize modern formulations of sustainable binders.

## The site-specific geochemical fingerprint of fine pottery from *Cales* (South Italy)

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The study focuses on 79 ceramic fragments of fine pottery from different ceramic classes (black glazed pottery, terra sigillata, fine common ware) recovered at the archaeological site of *Cales* (the modern Calvi Risorta, near Caserta) and subjected to a multi-analytical investigation via mineralogical-petrographic and mass spectrometry techniques (Polarized light microscopy, X-Ray Powder Diffraction, X-Ray Fluorescence, Thermal Ionization Mass Spectrometry). Finds of important production indicators, represented by welded pieces of black glazed pottery and a significant number of spacers, were also analyzed to attest a possible local production.

Polarized light microscopy showed that almost all samples have an optically inactive matrix with a bimodal distribution of grains and an extremely compact matrix. In some cases, diffuse birefringence is visible in the clay paste due to re-carbonated microcrystalline calcite (Fabbri et al. 2014). The inclusions are characterized by feldspar, quartz, sporadic brown mica, tiny calcite crystals and lithic fragments of both sedimentary (carbonate, occasionally >200 µm) and volcanic origin (trachyte >200 µm), along with microfossils of planktonic foraminifers. Additional information about the mineralogical assemblage comes from the XRPD analysis that revealed the presence of neo-formed Ca-silicates in most all samples in a firing temperature range from 850 to 1050 °C. Exceptions were noticed in very few samples showing lower temperatures estimated between 750 and 850 °C. All pottery samples show a high-CaO content and an extreme chemical homogeneity that matches that of the composition of the production indicators.

Geochemical comparison with some Campanian clayey raw materials high in CaO show a greater affinity with the Mio-Pliocene marine clay sediments of the Apennine sector (De Bonis et al., 2013), which include clays from the area of *Cales*. This affinity is clearly demonstrated using the Sr–Nd isotopic fingerprint of the ceramic samples, the production indicators (wastes of black glazed pottery and spacers), and a sample of local raw material, and attests of a strictly local and homogeneous production. This approach highlights the importance of combining mineralogical, petrographic and geochemical data to better constrain the potential geological sources of clay raw material (Renson et al., 2013) and more precisely define the provenance of pottery.

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## Archaeometric study of the white marbles used in the basilica of Santa Giustina (Padova): new data on the import of Proconnesian marble in Late Antique Adriatic

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This paper deals with the archaeometric study of white crystalline marbles used for the liturgical furniture of the ecclesiastical complex of Santa Giustina in Padova (Italy), founded by the prefect of the praetorium Venantius Opilio before 524 A.D., as attested by the dedicatory inscription now preserved in the chapel of San Prosdocimo (Brogiolo 2021).

Eight marble samples collected from some representative elements of the sixth-century liturgical furnishings today survived, were analyzed. They comprise one lintel with inscription, one fragmentary slab with inscription, three chancel pillars and three chancel screen slabs (Vedovetto 2021). Provenance determination of the marbles was carried out by means of a multi-analytical approach combining PXRD analyses, mineralogical–petrographic investigations, performed by microscopic observations of thin sections and the measurements of the ratios of stable carbon and oxygen isotopes ( $\delta^{13}\text{C}$  and Ratios of stable carbon and oxygen isotopes ( $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$ ) were 180).

The results obtained were compared with the currently most up-to-date petrographic and isotopic databases (Antonelli and Lazzarini 2015).

The laboratory data show that all the analyzed marbles come from the quarries of the micro-Asiatic island of Proconnesus, present day Marmara Adası, in the western region of the sea of the same name, little more than 100 km from Istanbul (Turkey). The archaeometric evidences, together with the stylistic and functional analysis of the pieces, authorize to suppose the direct importation of an homogeneous set of sculptures, arrived in different stages of workmanship from the quarries of Proconnesus, expressly ordered at Constantinople by an individual patron, identifiable with Opilio himself, founder of the basilica of Santa Giustina at the beginning of the sixth-century.

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**SESSIONE 5:**  
**DIAGNOSTICA, CARATTERIZZAZIONE E**  
**TECNICHE ANALITICHE**



## OpenAIAR: a new sustainable initiative for young members of our association

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OpenAIAR is the new sustainable initiative dedicated to young members of the association to have free access to the laboratories of AIAR researchers [1]. It involves the entire scientific AIAR community and is divided into two calls: the first is for all the members who, thanks to their collaboration, will make available time and instrumentations to carry out measurements using the methodology normally employed in their laboratories. A list of available facilities has been created to which young members, who is reserved the second call, can have free access and assistance for their research. Twelve laboratories are available until now and they are spread on the whole national territory. They allow for the analysis of almost all the materials of interest in the archaeometric field: paintings, glasses, potteries, stones, metals and alloys, organic materials, patinas, etc. The applications of the obtained results are manifold: characterisation (both chemical and structural), provenance, authentication, dating, monitoring, documentation, etc. OpenAIAR is therefore an activity aimed at supporting the research of young members based on an effective collaboration with the research groups of AIAR members. The available laboratories will be presented and the first two accesses of young members to OpenAIAR will be shown.



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## A library for the Apulian (Italy) wall paintings: a non-invasive approach for the study of mineral pigments and their mixing

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Mineral pigments such as ochres and earths have been always searched and used in human history alone or in combination with other colouring materials for the preparation of red, yellow, blue, green and black powders for paintings. At times, artists used to mix two or more pigments in different ratio, to enrich the chromatic palette, to limit the use of more expensive pigments and to obtain different desired colour hues. This practice has been found recurrently in Apulian paintings, both from rupestrian contexts and historical masonry buildings, in which precious pigments were replaced by well pondered mixes of ochres, earths and other common materials.

This research aims to create a library, as reference tool for the study of mural paintings, by collecting the results of non-invasive analytical methods carried out on specifically created pictorial layers composed of pure pigments and locally frequent recipes.

For this purpose, laboratory replicas of different colours, by using 18 pigments by both *fresco* and lime painting techniques and 37 locally recurrent colour mixes, composed of two or more pigments, have been prepared. Pigments were kindly offered by Kremer Pigmente GmbH & Co. KG (Aichstetten, Germany).

Before to be applied, pigments were characterised by X-ray Powder Diffraction analysis (XRPD). For the characterisation of replicas a non-invasive approach has been adopted. Portable digital microscopy equipped by polarized light filter, colorimetry test (in CIEL\*a\*b\* coordinate system), Fiber Optic Reflectance Spectroscopy (FORS) and X-ray Fluorescence Spectroscopy (XRF) have been used as diagnostic techniques for the identification of pigments in replicas.

A remarkable database on mixes widespread in the Apulian wall paintings has been produced. This approach has proven to be sustainable in terms of measurement speed and effectiveness, but above all in terms of data comparison with wall paintings of other and farer contexts.

## Interdisciplinary study of the Medieval glass mosaic of “S. Agnese fuori le Mura”, Rome. From art history to archaeometry

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The present contribution, which is part of a wider research project on early Medieval wall paintings and mosaics in Rome, aims to share the interdisciplinary protocol applied for characterising the apsidal glass mosaic of *S. Agnese fuori le Mura* (625-38). This mosaic is unique in the artistic panorama of the early Medieval Rome and requires further studies to be completely understood. However, its characterisation is hindered by two main factors: the presence of Byzantine features within a composition faithful to the urban artistic tradition, and the extensive restorations which have modified its surface [1].

To obtain new data for a better contextualisation of the mosaic, its material profile needs to be investigated, by means of archaeometric analyses on glass tesserae, which were never undertaken before on the *S. Agnese* mosaic. The dataset obtained can be compared with outcomes on coeval mosaics published until now [2], providing evidence able to clarify the processes of production and circulation of glass materials in the early Middle Ages.

Due to the complexity and heterogeneity of the mosaic surface of *S. Agnese*, an interdisciplinary approach, already carried out on other Italian Medieval mosaics, was here applied. This approach combines art-historical data with a campaign of multispectral investigations preliminary to the sampling of materials destined to laboratory analyses. Multispectral investigations revealed an articulated and complex conservative situation, with areas of discontinuity in the texture of the mosaic imputable to renovations invisible to the naked eye and not recorded by the historical sources or in other studies published so far [3]. Thus, the potential of multispectral investigations, as a preliminary tool to guide sampling hypotheses toward precise areas, the alleged original ones in this case, was confirmed.

Preliminary results, obtained by laboratory analyses – carried out by using optical and scanning electron microscopy, electron microprobe and X-ray powder diffraction, show that the sampled glass tesserae are compatible with the compositions and technologies used during the 7th century. This analytical evidence confirms the value of the selection, made by combining art-historical data and multispectral investigations. Further studies on data obtained from the laboratory analyses will allow to implement the interdisciplinary research conducted so far on Late Antique and Medieval glass. They will also provide an opportunity to promote the preservation of the *S. Agnese* mosaic, to highlight its value and clarify its role in the development of the Early Medieval artistic production.

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# First scientific studies on the handwritten manuscripts of “I Promessi Sposi” and other rare writings of Alessandro Manzoni

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“I Promessi Sposi” were written by Alessandro Manzoni between 1821 and 1842, when the final version called “Quarantana” was published. During this twenty-year period, Manzoni returned to the original manuscripts several times to add, remove, and modify parts of the text. Nowadays the large part of the original manuscripts are preserved in the Biblioteca Braidense of Milan and, for the first time, were investigated through spectroscopic and imaging techniques by two groups of University of Pavia and University of Milan. The project, at its preliminary stage, is aimed at investigating and characterizing the composition of the inks, as well as studying the erasures that Manzoni made on his handwritten manuscripts of the introduction (1821, Manz.V.S.XI.1) and chapter 7 (1823-25, Manz.B.III) (Figure 1a) of “I Promessi Sposi” novel. In addition, other rare writings, named “Modi di dire irregolari” (Manz.B.IX.4), and two annotated books (Manz.11.0085 and Manz.11.0090) (Figure 1b) were studied to compare the inks and possibly read notes under erasures. To these purposes, X-Ray Fluorescence (XRF) and FTIR in reflection geometry (FTIR-R) spectroscopies [1] were performed *in-situ* to obtain molecular and elemental information together with UV-Vis-IR multiband high-resolution imaging [2] to highlight specific manuscript features related to writing media, erasures and paper conservation. By combining results of the non-invasive, multi-analytical and portable techniques, relevant information about the composition of the different inks used by Manzoni in the manuscripts were achieved, shedding light on the overwritings that will help scholars in reading the text hidden by Alessandro Manzoni (Figure 1b).

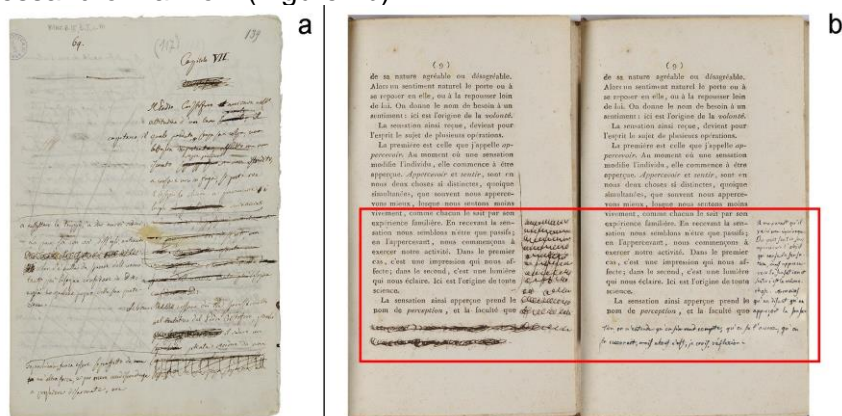


Figure 1. (a) Visible light image of the Chapter 7 manuscript and (b) comparison between visible light image (left) and IR reflectographic image (right) of the annotated book Manz.11.0090.

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## From materials to technique: a complete non-invasive characterization of a group of six *ukiyo-e* Japanese woodblock prints of the Oriental Art Museum E. Chiossone

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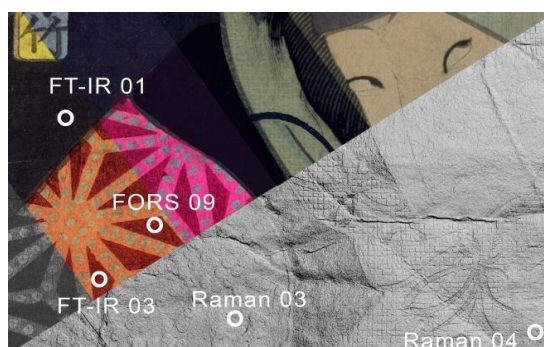
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As part of the conservation project of six woodblock prints recently donated by prof. Madeleine Cavalier, widow of the Genoese Archeologist Luigi Bernabò Brea, to the Oriental Art Museum E. Chiossone, a complete non-invasive scientific investigation was performed in collaboration of several Italian and international institutions.

The six Utagawa Kunisada's woodblock prints (*nishiki-e*) and the entire donation that today constitutes the L. Bernabò Brea collection is to be considered the

result of the vivid scientific interest and admiration that the professor had for Japanese art, especially for theatrical prints. The project was aimed to guide the conservation intervention and to characterize the materials and the technique of the *ukiyo-e* woodblock prints as well as their conservation conditions. The campaign was carried out *in situ* in the Museum venue and started with high resolution multiband imaging (visible, multiband fluorescence, near infrared) followed by reflectance transformation imaging (RTI) to characterize and highlight the peculiar printing technique and the conditions of the support. Then FORS (Fiber Optics Reflectance Spectroscopy), spectrofluorimetric, Raman and Fourier-transform infrared (FTIR) spectroscopies were successfully applied in synergy for the characterization of the painting materials (pigments, binders, support).

The results obtained represent a set of very important information for these never studied work of art useful to different skills: historians, conservators and curators in the museum. The materials identified are completely in agreement with those traditionally used in the Edo period in the 19th century. The computational imaging technique of RTI gave an additional amount of information in terms of surface characterization that could not be overlooked when studying these works of art. RTI data were further processed for a 3D rendering of the surface.



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## Provenancing beads: challenges of non-invasive analysis by means of ion beams

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Ion beam analysis (IBA) is widely used in the characterization of materials [1,2], thanks to its noninvasiveness, to the availability of microbeams for the study of small features and to the possibility to acquire simultaneously different signals produced in the ion-matter interaction, that can carry many valuable information on the target. However, the investigation of non-perfectly flat objects can be problematic: the focus of the beam can change within just a few micrometres, dips and cavities can reabsorb the emitted signal (e.g. elemental characteristics X rays), hence remaining blind to the detectors, and the acquired maps might present artefacts.

Such a situation can be frequently encountered in the analysis of Cultural Heritage objects, that are very often small, curved, carved, decorated with inserts and generally tri-dimensional. In addition, superficial alterations can increase the problems introducing different compositions and structures.

In the provenance study of lapis lazuli, started almost 20 years ago by the Solid State Physics group of the University of Torino in collaboration with the National Institute of Nuclear Physics (INFN) [3], this issue was faced many times, especially for very old archaeological artefacts made with this stone, that in most of the cases are really small beads. Several of those beads were analysed at the New AGLAE facility (ANR-10-EQPX-22, C2RMF, Paris, France) with an extracted 3 MeV proton microbeam, in particular two samples from Umm An-Nar site in Oman (III millennium BCE) and four cylindrical beads from Loralai in Pakistan (III-II millennium BCE). Specifically,  $\mu$ -PIXE (Particle Induced X-ray Emission) and  $\mu$ -IBIL (Ion Beam Induced Luminescence) maps were collected. Obtained results related to the provenance investigations will be presented, focussing on the challenges arisen from the morphology of the objects, both in the IBA analysis and in the precharacterisation phase.

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## Hydraulicity of plasters from the archaeological site of Teotihuacán, Mexico

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The study of the hydraulic properties of the pre-hispanic plasters of Teotihuacan and their correlation with the petrographic and mechanical characteristics has begun to be explored (Miriello *et al.* 2021). There is still a long way to go before achieving complete knowledge of the Mesoamerican plasters and the technological choices that have been made in each site and in the different types of plasters present, which often were dependent on the availability of raw materials (Barca *et al.* 2019; Pecci *et al.* 2018). Among the different aspects of plasters that will be considered, we will focus on one of them, which is very important, that of hydraulicity. This phenomenon has been studied for European plasters, however, there have been few studies for Mesoamerican cultures. We are interested in understanding how these people could developed methods of preparation and application of plasters to achieve certain characteristics. Teotihuacan plasters are generally made of two layers, with the outermost layer (*Enlucido*), composed of a thin mixture of lime and volcanic glass shards, and the underlying layer (*Firme*), consisting of crushed volcanic scoria (*tezontle*) mixed with a mud-based binder (Miriello *et al.* 2015). The hypothesis that the presence of glass shards would produce hydraulicity in the plasters was rejected. It has been observed that there is a dependence of the mechanical resistance of the samples on the presence of *tezontle*. As can be deduced from the studies carried out, the contact between the two layers gives hydraulic properties to the lime binder and therefore gives it greater toughness and resistance. What has been noted is that as the distance from the *Firme* layer varies, the hydraulicity index varies. More precisely, a decrease in the calcium content is observed in the part closest to the volcanic slag layer and therefore an increase in the hydraulic index. The results obtained appear significant, but the study concerning the hydraulicity of the Teotihuacan plasters and how this is related to petrography, requires more in-depth analysis. However, the combination of microchemical, microphysical and petrographic measurements made it possible to definitively clarify that the significant external lime plaster layer hardness was due to the reactivity of the small fragments of *tezontle*, which makes it hydraulic.

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## Selected ion flow tube-mass spectrometry (SIFT-MS) in heritage science: characterization of organic materials and archaeological samples

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The identification at molecular level of organic materials in heritage objects as paintings requires in most cases the collection of micro-samples followed by micro-destructive analysis. In this study, we explore for the first time the possibility to characterize different types of organic materials by means of non-invasive analysis of released volatile organic compounds (VOCs) through selected ion flow tube-mass spectrometry (SIFT-MS). SIFT-MS is a transportable direct mass spectrometric technique that achieves the analysis of VOCs at trace levels in real time, by controlled ultra-soft chemical ionization using eight different chemical ionization agents.

We initially applied SIFT-MS analyses on different reference synthetic resins, natural resins, and paint binders to evaluate the possibility to obtain qualitative data for the identification of these materials in heritage objects avoiding any sampling. The instrumentation was then tested in laboratory on small archaeological objects to evaluate the feasibility of applying this transportable approach directly in situ in museums.

Finally, an in-situ analysis campaign was designed for the study of the burial assemblage of Kha and Merit, discovered in 1906, and now preserved at the Museo Egizio in Turin. The discoverer of the tomb and director of Museo Egizio, Ernesto Schiaparelli, limited investigations to a few non-unique items, leaving most of the assemblage untouched. His decision has enabled contemporary scholars to study such exceptional archaeological findings with non-invasive approaches that could not be foreseen at the time. The interpretation of the mass spectrometric data, supported by statistical data analysis, shed new light on the chemical nature of the organic materials preserved in 50 vessels of the Museo Egizio in a completely non-destructive way.

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# Magnetic Resonance Imaging clinical scanner as a novel tool for archaeological waterlogged wood analysis and 3D digitalization

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Moisture is the main factor that causes deterioration in cultural heritage objects made of porous materials. It is essential to determine moisture content (MC) to understand the state of preservation and choose the best conservation treatment [1]. At the same time, it is essential to choose nondestructive and non-invasive approaches for more sustainable investigations, make them safe for the environment and the sample. In this study, Nuclear Magnetic Resonance Imaging (MRI), the most important non-invasive medical imaging technique for human tissues analysis, was applied to study archaeological waterlogged wood samples. This type of archaeological material has a very high moisture content (400%–800%), thus proving to be an ideal investigative subject for Nuclear Magnetic Resonance imaging which detect water molecules inside matter. However, despite MRI being a very promising and attractive method for providing detailed, non-invasive, and quantitative information in living materials containing water, MRI in wood research is still far from being a routine tool [2]. By this methodology it is possible to obtain information about water content and conservation status through  $T_1$ ,  $T_2$  and  $T_2^*$  weighted images analysis [3]. Furthermore, it permits to processing 3D reconstruction that could be an innovative tool for marine archaeological collections digitalization. Moreover, samples are directly scanned in the water where they are stored, without any sampling or handling and any water artefact on images. In this study, an MRI protocol analysis is shown, in order to obtain useful information about moisture content, conservation status and acquire 3D reconstruction in an all-in-one methodology.

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## Archaeological waterlogged wood from two archaeological sites: investigations and comparison

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The conservation and protection of fragile and non-renewable archaeological finds, especially in situ conservation, emerged as extremely relevant to the UNESCO Convention. The Biskupin site in Poland, representative of the Lusatian culture settlement, is one of those sites where, since first archaeological excavations in 1933, the decision was made to preserve the archaeological wooden finds in-situ, without removing them from the environment in which they were found. Recently, various monitoring programs have been conducted, to investigate the in-situ burial environmental conditions and their effect on the remains (Babiński 2007, Lucejko 2020, Tamburini 2015). A second archaeological Polish site with some similar characteristics for what concerns the environment and the wooden remains has been object of investigation: Izdebno. The physicochemical parameters of the deposition environment and the physicochemical properties of wooden remains have been compared.

This work is part of the JPI StAr project (Development of Storage and Assessment methods suited for organic Archaeological artefacts) which aims not only to broaden knowledge on the processes occurring in wooden remains and in the surrounding environment but also to explain the temporal relationships between changes in the chemical and physical properties of wood and changes in the conditions of their deposition.

Analytical pyrolysis (Py-GC/MS and EGA-MS) proved capable to highlight the chemical changes that occurred both in the lignin and in the polysaccharides of the wooden archaeological remains. It allowed us to evaluate the depletion and depolymerization of polysaccharides and side chain shortening, oxidation or demethylation processes that occurred in the lignin polymer in wood. Extractives will be characterized by GC-MS analysis and the results compared between the various wooden artefacts studied.

The aim of this work is a contribution to the understanding of the biochemical processes in wood degradation, and to relating them to the chemical-physical environmental changes of the long-term burial environment.

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## Conservation and Enhancement of Osteoarchaeological Remains: Aims and Purposes of the Database IsoMedIta

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In the last decades there has been a growing interest about archaeometric researches upon osteoarchaeological remains as a source of information to investigate past lifestyles. Stable isotope analysis of collagen, the organic component of the bone, allows us to gather a wealth of information ranging from paleo-diet, climate changes, social status, exploitation of local resources, migrations, weaning age, breeding and farming practices of ancient populations. However, these data could be affected by the conservative state of the bone, the degradation of which could compromise the reliability of the analysis. The PH of the soil, changes in temperature and the presence of groundwater are only a few factors that can lead to cracks in the bones and direct exposure to substances present in the deposition soil. As a result, the degradation of the mineral component of the bone, the hydroxyapatite, could directly lead to collagen exposure to microbiological attacks by fungi and bacteria. It is therefore important to assess the diagenesis rate of the collagen also as a tool to ensure the accuracy of the stable isotope measurements. In this framework, there is a growing need to go beyond the analysis of individual case studies and adopt a more global approach towards the osteoarchaeological remains and how their conservation could also affect the information they retain. With this goal, has been created IsoMedIta, an Isotopic database for Medieval Italy born with the purpose of collecting stable isotope measurements ( $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ ,  $\delta^{18}\text{O}$ ,  $\delta^{34}\text{S}$ ,  $^{87}\text{Sr} / ^{86}\text{Sr}$ ) of human, plant and animal samples from the Italian Middle Ages, alongside with biological, historicalarchaeological and environmental meta-data. The stable isotope values are only considered in correlation with acceptable ranges of collagen diagenesis, ensuring the reliability of the collected data. The integration of archaeometric and archaeological researches will guarantee a more accurate overview of the informative potential of osteoarchaeological remains and promote sustainable initiatives to limit the environmental factors that can accelerate their degradation and therefore a loss of invaluable information. On this account, one of the main aims of IsoMedIta is to offer a tool for the conservation and enhancement of this area of the cultural heritage, in which an increasingly marked emphasis must be placed on an interdisciplinarity approach.

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## Mapping stones and deterioration morphologies distribution at the *Torre dell'Orologio* (St. Mark square - Venice) in the frame of the Hyperion EU project

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The *Torre dell'Orologio* (clock tower) in Venice is an early Renaissance (1499) building in Lombard style overlooking the entrance to the *Mercerie* on the north side of St. Mark's square. Its historical and artistic importance in the city as well as in the general landscape of the Italian Renaissance, is undeniable, as it is the representative expression of the architectonic stone materials most in vogue in that historical period. As part of the European project HYPERION (Horizon 2020), which is dealing with resilience and sustainable reconstruction of historical areas, the main façade of this elegant monument was mapped in order to obtain an overview of the composition of the building, its state of conservation and the history of its restoration.

The mapping of deterioration morphologies was carried out following the ICOMOS-ISCS glossary of decay forms together with a quantification of the same inspired by the work of Fitzner and Heinrich (2001).

To support the drafting of the maps, in-depth investigations were carried out on a series of micro-samples of both stone and deterioration products following a multi-analytical approach including petrographic and biological analyses by optical and scanning electron (SEM-EDS) microscopy, as well as through powder X-ray diffraction, ion chromatography and infrared spectrometric investigations using  $\mu$ FTIR.

The final output was the production of a series of monographic maps: one concerning the building materials, a series of maps focused on the five macro-categories of deterioration morphologies as defined in ICOMOS-ISCS (i.e. cracks and deformations, detachment, features induced by material loss, discolouration and deposit, biological colonisation). A further map was produced to graphically summarise the total state of decay of the building by reporting a Total Decay Index (TDI).

In general, the most abundant and/or intense forms of deterioration detected were black crusts, patinas, discolouration and patterns related to erosion processes. The stones used in the façades are: regional (*Rosso Verona* and *Scaglia Rossa*) and extra-regional limestones as well as a series of marbles and stones already used in classical times: three crystalline marbles (*Carrara* and *Pavonazzeto Toscano from the Apuan Alps*; *Proconnesian from the micro-Asiatic island of Marmara*), the *Rouge de Languedoc* (a French limestone in Italy called *Rosso di Francia*), and the famous Egyptian volcanite known as *Porfido rosso antico*.

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# How to design an affordable homemade pXRF spectrometer for Cultural Heritage applications: the FUXYA2020 experience

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The FUXYA2020 project was conceived with the aim of creating a cost-effective portable prototype for energy dispersive X-ray fluorescence (pXRF) analysis. The design and prototyping of FUXYA2020 was intended to improve accessibility and availability of the capabilities of XRF analysis for all those institutions that would not be able to purchase a commercial device due to financial constraints. In fact, a homemade project allows to spread the costs over different periods of time and it is, therefore, the most suitable approach when funds are low but constant over the years. FUXYA2020 was designed primarily for Cultural Heritage applications. Indeed, the geometric configuration of the spectrometer was optimized to suit the requirements for both low Z matrix objects, such as ceramics and glasses, and medium-high Z materials, such as metals. Moreover, a positioning system, run through an Arduino UNO board and connected to several components, was developed in order to verify the investigated area and to allow repeatable and comparable measurements. To make a low-cost and lightweight device, all the structural components of the FUXYA system were 3D printed in PLA (polylactic acid) except for the base, which was cut out from a PVC sheet for greater structural strength. FUXYA system is indeed very compact and its dimensions are comparable to those of an A4 sheet of paper (Figure 1).

The FUXYA2020's performance was tested for qualitative, semi-quantitative and quantitative analysis on pigment layers, archaeological ceramics and certified gold-based alloys respectively, thus covering three main materials in the CH field. In the case of ceramics, their classification was carried out based on multivariate analysis obtained through R environment, while for metal alloys Axil-QXAS software was used for data processing. Again, in the perspective of a low-cost project, both software used are available free of charge.

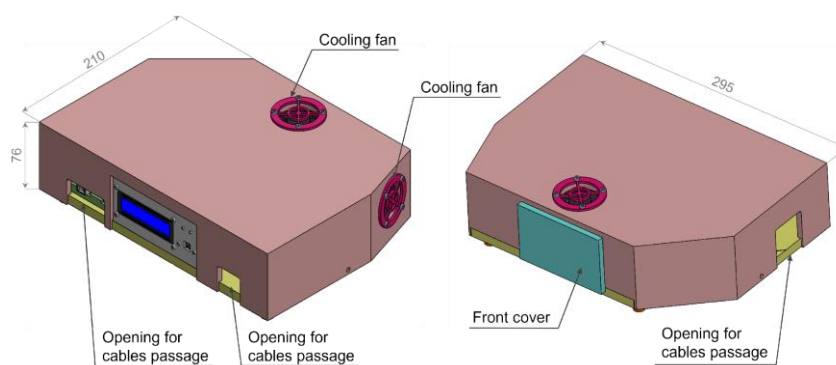


Figure 1 - FUXYA2020 external case with dimensions given in mm

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# Comparison of analytical methods in the characterization of two - and three - layer samples using the HHXRF technique

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X-Ray Fluorescence (XRF) technique is widely applied in the chemical analysis of artefacts in the field of cultural heritage [1,2] where very thin and multi-layered objects are often investigated. They may have a complex structure and coating layer that is can be difficult to characterize, especially in terms of thickness. Paintings are also composed of several layers (support, preparatory layer, binders, pigments and varnish) that are often useful to study.

In this contribution, the results of the comparison of different analytical methods used to study multilayer samples (two and three layers) based on spectra acquired with a HandHeld XRF spectrometer (HHXRF) are presented. Six methods were used for thickness determination: (a) direct measurement with an electrometer; (b) analytical approach implemented in PyMca (by D.K.G. de Boer [3]); (c) theoretical equations relative to characteristic lines using experimental values; (d) fitting curves of the measured ratios of characteristic lines [4,5]; (e) partial least square regression [4]; (f) fitting curves of elemental percentage concentrations in the layers [6].

To obtain accurate calibration curves, sixty standard samples with different layers and thicknesses were constructed. Each has a one mm thick lead substrate covered by a gold layer without or with a silver coating. The different configurations were obtained by varying the gold and silver thicknesses. We present a useful overview of the methods used and the results highlighting the reliability and ability to determine the thickness with each of the methods and to identify the most appropriate choice according to the configuration analyzed.

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**SESSIONE 6:**  
**INNOVAZIONE, SOSTENIBILITA' E APPROCCI**  
**"GREEN" NELLA CONSERVAZIONE**

## Evaluation of gelling agents in the biocleaning of the 16th century painting “Coronation of the Virgin with the Saints Ambrose and Jerome”.

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The subject matter of the study is the restoration of a late 16<sup>th</sup> century painting on canvas representing the Coronation of the Virgin with the Saints Ambrose and Jerome, attributed to the Tuscany-Umbria area. Following previous restoration treatments, a triterpenoid resin (mastic) and an animal glue were applied on the paint layer of the canvas. In particular, the natural resin was used as a varnish, while the adhesive was employed to glue paper on the paint layer before the lining process. Over the years the two superimposed substances underwent degradation processes due to natural aging and environmental factors (e.g., particle pollution), making it necessary to remove both the glue and the resin.

The research was carried out to experiment the bacteria-based removal of the two top layers. Six non-pathogenic bacterial strains, belonging to the “ENEAMIRRI” culture collection (<http://www.mirri.it/index.php/associated/enea/>), inoculated into nutrient-free gels, were tested in biocleaning trials.

The use of gelling agents in biocleaning is essential to keep bacterial cells on the surface of the paint layer, to reduce the penetration of water within the work of art and to increase contact time with the surface. Specifically, five different viscosity modifiers, made of natural and synthetic polymers, were tested: Agar (a natural polysaccharide), Klucel™ H (hydroxypropylcellulose), Vanzan® NF-C (xanthan gum), Hydrogel (based on the reaction between sodium alginate and calcium chloride) and Velvesil™ Plus (made of silicone polymers). Each gel was applied on reproductions of the painting, made with the same materials of the artwork, and it was tested three times in order to obtain relevant statistical data.

The aim of the experiment was to select both the most suitable bacterial strain and the gelling agent, as well as the minimum amount of time required to remove properly the top layers. Four tests were conducted overall, during which gels were left on for 24, 12, 8 and 6 hours respectively.

The tests demonstrated that prolonged contact times did not correspond to a better removal of the resin and the glue. Results, however, depended on the chemical and physical properties of the gelling agents. Velvesil™ Plus had negative effects on bacterial metabolic reactions, forbidding biocleaning operations. On the other hand, Agar and Klucel™ H did not inhibit bacterial metabolism, but their application on the paint layer was complicated by their rigidity and adhesiveness. The most remarkable results were obtained by Vanzan® NF-C and Hydrogel, both of which led to the removal of the glue and the resin in combination with the strain *A. calcoaceticus* LAM21. The combination of LAM21 and Vanzan was then applied on the painting for the biocleaning.

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## Deep eutectic solvents (DESs): new green solvents for the cleaning of nonpolar coatings

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Deep eutectic solvents (DESs) are a mixture of two or more solid components leading to a strong depression on the melting point when compared to their individual counterparts [1]. They form eutectic mixtures of a hydrogen-bonding acceptor (HBA) and a hydrogen-bonding donor (HBD) able to self-associate via hydrogen bonds and Van der Waals interactions. The properties of the final DES can be adjusted through the selection of the individual components based on their chemical structure and molar ratio. The discovery of these non-toxic formulations generated a breakthrough in the world of green chemistry. As eco-friendly solvents, DESs are being used in many areas of science and technology due to their excellent physicochemical properties, such as low volatility, low cost, and non-toxicity [2]. However, only a few attempts have been made so far to use DESs in the field of Cultural Heritage Conservation [3,4].

In this framework, we choose to explore the potentialities of DESs for cleaning treatments on works of art, specifically for the removal of nonpolar coatings – such as waxes. The experimentation was performed using laboratory specimens consisting of one layer of wax applied on microscope glass slides. Four different types of waxes were chosen for the testing, namely two natural waxes (beeswax and carnauba wax) and two microcrystalline waxes, which are commonly used as coating layers on Cultural Heritage materials. The cleaning tests were performed using three different operational modes, i.e., swab cleaning, 1-minute-application using Japanese paper (Tengujo) as an intermediate layer, and 3-minute-application using the same intermediate layer.

Multispectral imaging was carried out using visible (VIS) and ultraviolet (UV) light both before and after cleaning tests. The analysis was performed on samples, swabs, and Japanese paper to assess the effectiveness of the tested DESs in solubilizing the four waxes. Fourier Transform Infrared spectroscopy (FT-IR) in Attenuated Total Reflectance (ATR) mode was carried out both before and after cleaning tests as well, to evaluate the presence of solvents residues on the samples' surface. The analysis was also carried out on the swabs and the Japanese paper sheets used for the cleaning treatments to detect the presence of wax and assess the effectiveness of the cleaning tests.

The experimental process provided the first valuable results, proving DESs' potential of being used as sustainable solvents for cleaning treatments on Cultural Heritage materials and, therefore, giving reason to further research.

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## Circular economy approach for the restoration of ancient ceramic: the example of ceramic-waste geopolymers

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The actual scenario of the conservation and restoration field is dominated by the dramatic climatic conditions and by the foresaw aggressive changes. Ceramic materials are one of the most abundant material found in archaeological contexts, in fact it was for long time one the most used materials for ordinary objects and constructions. Its conservation is fundamental for the preservation of the memory of social, cultural and technological aspects. Nevertheless, even if it is a durable material, it could undergo very important damages, which could sometimes completely compromise its integrity, particularly when traditional obsolete restoration materials are used (e.g. cement mortars). Thus, to find the right conservation and restoration practices, in order to make a restoration intervention at the same time compatible, efficient and durable; and – considering the environmental issue – sustainable, rises as a challenge of the modern world. A new class of materials seems to be the right alternative, the alkali activated materials, among which geopolymers. These are solid materials obtained by mixing at room temperature - thus reducing the CO<sub>2</sub> emissions - powdered alumino-silicate precursors with an alkaline liquid solution. The application of these products on Cultural Heritage is still not deeply investigated, but the few results available are encouraging. In this study, in order to involve the restoration field in a local circular economy, industrial tiles waste - one of the prominent source of waste in Europe - have been used as main precursor for the implementation of restoration mortars for brick masonries and for the pre-casted substitutional bricks and other three-dimensional elements. Once investigated the geopolymeric gel formation on the binders developed; mortars and three-dimensional elements have been realized and their physical-mechanical characteristics have been tested by means of Mercury Intrusion Porosimetry, Capillary absorption tests, Mechanical and Flexural tests, and the colour compatibility was verified by colorimetry. Furthermore, in order to observe the applicability, tests have been performed at the interface of the mortars applied on archaeological bricks, as well as attempts of synthesis in situ were performed (Fig.1). Today, their monitoring is ongoing.



Fig. 1 – Ceramic-based geopolymeric mortar during in situ application (left) and a totally green masonry prototype (right), both exposed at the Odéon in Catania.

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## **Gentle cleaning operations. A sustainable approach in the conservative project on four Michelangelo masterpieces**

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The paper reports a sustainable approach in cleaning operations of marble masterpieces by Michelangelo.

An important campaign of analytical investigations was conducted in the Museo di Casa Buonarroti, focused on two famous works by Michelangelo, the Madonna della Scala (1491) and the Battle of the Centaurs (1492) belonging to his early production. The two reliefs were subjected to a soft cleaning and maintenance operations in occasion of the renewal of the dedicated expositive room into the museum.

The restoration project followed the previous one carried out in 2020 on the Medici Chapel in the Sacrestia Nuova in San Lorenzo church (Florence), which aimed to study the materials and to support the restoration of the funeral monuments of Giuliano Duke of Nemours and Lorenzo Duke of Urbino, sculptural complexes created by Michelangelo. The restorations that all the marble artworks had undergone during the centuries left some residues or altered protective materials, such as waxes and oxalates which changed to a yellowish-orange tone over time.

The analytical study was based on non-invasive analyses, applied directly in situ, and integrated with micro-invasive insights on superficial deposits taken from the artworks. The main goal was to characterize the patinas and deposits in order to better understand the issues related to the state of conservation but primarily to monitor the cleaning operations, by means of a detailed check of the colorimetric parameters, addressing the restorers in their choices during the whole intervention. Hue and homogeneity of the colour of the patina, as well as the comparison of the chromatic coordinates of different areas were guiding factors for the evaluation of operational choices. The results allowed to select a sustainable and mainly solvent-free procedure that helps in thinning the patinas without altering the precious surfaces.

## Enhancing antimicrobial activity of plant essential oils

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Essential oils (EOs) have been used for centuries as antimicrobial, antioxidant, pesticidal agents in several fields (agriculture, cosmetic, food, medicine, pharmacy) and recently also to perform green conservation strategies of cultural asset (Palla & Barresi 2017). Biological systems as bacterial and fungal colonies are able to induce the biodeterioration of cultural objects. Conventional treatments to control these processes provides for the use of synthetic chemical biocides, whose toxicity for humans and environments is well known, as well as their persistence in natural environments, contaminating areas far from the site of treatment (Arias-Estévez et al. 2008).

In order to develop alternative biocides, several studies have been focused to various natural products with potential antimicrobial activity such as plant essential oil or hydro-alcoholic extracts. In our previously studies *Thymus vulgaris* L. and *Origanum vulgare* L. (Lamiaceae family) essential oils have been applied to counteract microbial colonization or insect infestation, exposing the artworks to EOs volatile compounds (Ebadollahi et al. 2020; Palla et al. 2020).

Here the exposure to EO volatile compounds is carried out in *ad hoc* clean chambers, under vacuum or environmental conditions, comparing the antimicrobial efficiency. Preliminary, the EOs chemical compounds were identified by GS-MS analyse and antimicrobial activity *in vitro* tested by Agar Diffusion Disk and Well Plate Diffusion methods. Results from assays performed on different works of art (wooden and parchment artworks, and wood specimens) highlight that vacuum conditions speed up the disinfectant processes.

Plant extracts have different modes of action, such as activation or blocking of enzymatic reactions, direct effects on enzyme synthesis, regulation of intermediate metabolism or alteration of membrane structures; the interaction of its hydrophobic components with the lipids present in the cell membrane of microorganism, resulting in metabolic damages and cell death.

Although further investigations are needed, plants essential oils activity combined with specific time and exposure conditions allow to hypothesize their use through a green strategy, replacing toxic conventional biocides.

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## 15-17th century Frescos of Riga Castle and their Conservation with lime-based Materials

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Riga Castle, in its beginnings, during the 14th to mid-16th centuries, was a residence for the Livonian Order. After that, the castle as a materialized symbol of power was used as the residence of King of Poland Stephan Batory, general governor of Polish-Lithuanian Kingdom, General Governor of Sweden, and General Governor of Russia. In 1922, it was adapted to the residence of the President of Latvian State.

In the medieval period, the castle had a classic structure with private chambers, dormitory, and rooms for technical purposes – kitchen, warehouse, brewery, etc. The most presentable premises in the castle was a chapel and a refectory, where frescos of great value have been discovered: traces of medieval figural fresco in Chapel and late renaissance paintings in Refectory.

As the fortification building, we may not expect a great richness of decorative elements on the castle's facades. Due to frequent destruction, reconstructions, and adaptations during the seven centuries of its existence, the castle inside has lost a lot of its decorative elements. Each repair or transformation to the new rulers demands integrated into the historic body new elements of interior decoration. Until now, by methods of investigation, we may fix a few Gothics, Renaissance, Baroque, and Neo-classical details and traces of wall paintings.

The current conference report is devoted to the annotation and description of remnants of medieval fresco-secco on the eastern wall of the Chapel and decorative paintings of the 17-th century on the vaults and vaulting capitals of Refectory.

The state of preservation of the old paintings was as fragile as the building itself. Both premises – chapel and refectory – need meticulous restoration work. Fragments from frescos that have remained from 500-year-old history are very fragile. Some material investigation was done beforehand, in 2017-2018, so until 2020, when restoration started, the program and materials for conservation and adaptation of premises were prepared for the needs of the Museum of History. Both frescos are on lime plaster with lime-based colors (lime tempera), so it was convenient to use only lime-based materials in restoration – lime mortars, lime-based injections, and nanolime consolidant.

After the conservation, all paintings shall be exposed as an essential part of the interior, demonstrating the historical transition of a long-living object of architecture and illustrating characteristic features of style and time.

## The era of plastic: from micro-destructive to non invasive approaches for a more sustainable preservation of plastic artworks

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The rapid and drastic growth in global plastic production occurred since the last century have made plastic one of the most versatile materials, employable in a great variety of application areas, including cultural heritage. Nowadays, an increasing number of objects made of plastic are common in worldwide heritage collections. Modern and contemporary artists experimented with different types of synthetic polymers mixing them with traditional atelier products, making the composition of final artworks extremely heterogeneous and complex to characterize, maintain and preserve over time. The short-term stability shown by some plastic materials, especially by the newest biodegradable and recyclable ones, makes conservation practices highly challenging considering the wide variety of degradation mechanisms in which different plastic formulations may incur, strongly influenced by the presence of additives and exhibition conditions. A further issue is constituted by the scarce and undetailed information on plastic objects provided by museum catalogues, in which generic and not informative terms are often reported for plastic objects. Thus, a proper knowledge of the chemical composition of the starting materials is fundamental for characterizing their physical and chemical properties, studying degradation pathways and fine-tuning suitable preservation strategies.

In this study, the characterization of Acrylonitrile-Butadiene-Styrene (ABS) based polymer, chosen for its favorable mechanical proprieties, easy availability and wide application in plastic art and design objects, have been carried out exploiting the combination of non invasive and micro-destructive analytical techniques. LEGO® bricks are made of ABS, and were selected for optimizing an analytical approach applicable to plastic art objects. Firstly, the composition of unaged LEGO® building blocks of different colors were determined in terms of polymer composition, additives, inorganic/organic pigments through the application of both non invasive techniques such as Infrared, UV-Vis-NIR, Raman and X-rays fluorescence (XRF) spectroscopies, and micro-destructive ones such as pyrolysis online with gas chromatography or evolved gas analysis both coupled with and mass spectrometric detection (Py-GC-MS and EGA-MS). Despite ABS short-term stability in proper conditions is already known, a non invasive approach able to determine the degradation state of ABS based objects is still missing. Thus, two approaches have been tested on LEGO® bricks: (i) UV-Vis photo-oxidative accelerated ageing in weather chamber followed by characterization by spectroscopic and mass spectrometric techniques; (ii) online micro-irradiation with an UV-Vis source directly in the pyrolysis chamber of Py-GC-MS performed both in oxygen and inert atmosphere. The comparison of the two approaches allowed us to evidence the role played by ageing parameters, additives, extenders, and synthetic pigments in the degradation pathways of polymer providing hints for aware preventive conservation measures.

This work aims at exploiting the potentialities of robust micro-destructive methods in polymer analysis for developing a high valuable non invasive approach for plastic characterization in cultural heritage. The proposed protocol will promote green analytical methods, applicable without the aid of organic solvents or any other risk for the user. This study constitutes a pilot study to be expanded to further classes of polymers, in order to monitor the conservation state and early degradation signs of plastic artworks and design objects ensuring their sustainable conservation for fruition and future preservation.

## From characterization to innovative conservation compound: a project aimed to mosaics preservation.

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Mosaics are complex systems composed by several layers each characterized by a wide variety of mainly inorganic materials [1]. Due to the heterogeneity of the materials composition, several deterioration process can occur, and conservation process result difficult. Thus, prior material characterization is a first fundamental step to understand the nature of the mosaic. In this work, 2000 pieces of archaeological material of mosaic gathered during surface recognition activities in the fields around Aquileia are considered. Among these, 150 entities are selected for the research purpose, in accordance with their colour and their distribution within an area of interest of 4 km radius. Petrographic characterisation in thin section is performed in order to assess the nature of the rock types and the composition of the mortar. Sampling procedure is carefully planned in order to minimise the quantities of material used. Analyses are performed with optical microscope (OM), scanning electron microscopy (SEM) coupled with EDS probe, micro-Raman. Additionally, complementary and innovative analysis for the study of superficial patinas is carried out using a SEM coupled to a focused ion beam (FIB-SEM). XRD analysis will be applied to determine the mineral composition of mortars.

The number of specimens studied in this work is high enough to perform a statistical study of the most used type of materials in the region of Aquileia during the early roman period. This information will be important to ascertain the relationship of the local population with the areas of supply of rock types. The nearness between the fields of recognition and the most famous mosaics of Aquileia will be considered in order to compare the unknown material consider in the research with the one present on studied sites. On the basis of the characterization results, new formulations for the stabilisation and consolidation of damaged mosaics will be developed. The design will rely on the theory of reverse engineering, which is based on the reproduction of the original ancient material through modern material methods. In this way, it is possible to produce new materials with chemical composition equal to the ancient one [2]. The addition of natural organic compounds as figs or fats in the preparation of mortars is a practice that cross along the centuries, cited also by Pliny the Elder [3]. Nowadays, the use of plants products as additive is renovated and transferred to the production of green and sustainable compounds. This idea will be at the base for the development of an injection mortar useful for the consolidation of mosaics.

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## Study and development of silica nanoparticles strengthening agents for stones preservations

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Study of innovative products aimed to the conservation of natural building stones and lithoid materials is fundamental in order to approach the consolidation of structural and decorative architectural elements in a conscious and safe way. Application of suitable compound can reduce the deterioration phenomena extending the preservation of materials. Ethyl silicate (TEOS) was widely used for the treatment of mortars and stones (Wheeler, 2005), as base for the development of consolidating and protecting mixture (Tesser *et al.*, 2014), as well as precursor for the synthesis of silica nanoparticles (SNP). Several studies demonstrate how the application of products based on silica particles of nano dimensions allow a greater penetration depth in porous matrix avoiding problems of superficial cracks induced by ethyl silicate sol-gel process (Milani *et al.*, 2007). In this regards, a research was developed in order to assess the effectiveness of SNP as strengthening agent for silicate stones with different porosity (Stucchi *et al.*, 2022). Two SNP products based on well monodispersing particles having dimensional range of 50 nm and 120 nm (SNP50 and SNP120, respectively) were synthesized and dispersed in a water-ethanol solution. The solutions were applied on *Pietra di Firenzuola* and *Pietra di Muggia* sandstones, and on *Bianco Sardo* granite. Their application demonstrated plain chemical compatibility between the compound and the substrates, whereas aggregation phenomena of the particles was assessed preventing the penetration of the products in the substrate.

For this reason, polysiloxanes and calcium ions functionalizations of SNP were considered (SNP-PDMS and SNP-Ca, respectively).

Adopting the same analytical and methodological approaches used in the previous work, the functionalized SNP were analysed by chemical and microscopic investigations (SEM-EDS, FTIR, NMR) showing pro and cons of each one. The comparison of the results obtained allowed to select SNP-PDMS as the best one. The product was applied on the same silicate rocks tested in the previous work and the results of microscopic investigation, porosimetric analyses, colorimetric measurements and sponge tests compared. SNP-PDMS seems improve SNP120 performances, limiting the aggregation phenomena and enhancing water repellency of treated surfaces.

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**SESSIONE 7:**  
**VULNERABILITA', TUTELA E**  
**VALORIZZAZIONE**

## Byzantine wall paintings from San Marco d'Alunzio, Sicily: knowledge, diagnostics and restoration for valorizing the widespread Cultural Heritage

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The wall paintings preserved in the Museum of Byzantine and Norman Culture and Figurative Arts in the Municipality of San Marco d'Alunzio (ME) belong to a church discovered in 1953. The building, referred to as the "Church of the Four Saints Doctors" due to the significant subject present in one of the apses, was the subject of surveys and studies that would have suggested a pre-Norman existence. The frescoes in the central apse and in the right one (*prötesis*) are of considerable interest for the knowledge of Byzantine figurative culture in the Norman age, dating back to the 12<sup>th</sup> century. The subjects should not have been rare in ancient times, even if the loss of many pictorial complexes actually prevents the evaluation of its iconographic fortune and diffusion. Similar themes can be recognized in buildings of the Byzantine-Norman age in Southern Italy (Cattolica di Stilo, Panaghia di Rossano), certainly imported from mainland and insular Greece where there are numerous similar examples, documented for a longer period of time.

Diagnostic investigation was carried out during the recent restoration works of the remains of the frescoes. First, the wall paintings were analysed using the XRF and the FORS portable spectrometers to obtain a non-invasive identification of the original palette. Then, three fragments were sampled for a microstratigraphy study to identify the chemical composition of the mortars and of the blue and black pigments non unequivocally identified through non-destructive techniques. Polished sections were observed and analysed with OM and SEM-EDS. The palette included mainly earthen pigments like red and yellow ochres, green earth and more valuable lapis lazuli blue applied on bone black layer. The analysis of mortars found on the different apses showed the same manufacturing technique and constituent materials: lime-based binder with the addition of monocristalline quartz, polycristalline quartz and rare calcareous lithic fragments as sandy aggregate. The obtained results allowed to know the pictorial technique used for the wall paintings and to compare the Sicilian cycle painting with coeval byzantine wall paintings preserved in Sardinia and Southern Italy.

The example here presented wants to focus attention on the possibilities that the characterization of techniques and pictorial materials represents a starting point for defining new strategies for tourism circuits. Comparisons between geographically distant sites but which refer to the same temporally coeval artistic production and cultural movements occurring over wider territories could contribute to define different stages of a single visit itinerary. This broadened perspective based on archaeometric studies and historical-artistic studies can improve knowledge of the widespread culture heritage. Moreover, this approach enhances on a large scale the tourist offers by promoting sustainable growth of the numerous small villages far from the large sites best known to national and international tourism.

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## Study of indoor air quality and microclimatic conditions in the Sanctuary of the Beata Vergine dei Miracoli in Saronno, Italy

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The Sanctuary of the Beata Vergine dei Miracoli was built between the XV and XVII centuries and is located in Saronno, a small town in the Lombardy region of Northern Italy. Once the architecture of the sanctuary was completed at the start of the XVI century, some of the most renowned and influential artists of the time were summoned to work on the interior decorations [1]. Thanks to the work of these artists, the sanctuary is still today the home of several masterpieces, including frescoes, wooden sculptures and carvings. Poor indoor air quality and microclimatic conditions are two factors which contribute significantly to the degradation of works of art such as the ones previously mentioned. For this reason, museums have imposed concentration limits on the major air pollutants, along with temperature and relative humidity ranges that need to be respected [2]. However, the same regulations do not apply directly to sanctuaries and other indoor sites which attract large numbers of people acting as vehicles for the penetration of pollutants from outdoor. Hence, a proper and thorough air quality characterization is of the utmost importance for the safeguard of the works of art in such places.

In the present work, microclimatic conditions have been monitored using temperature and relative humidity data loggers (USB Mini TH, XS Instruments). Instead, an optical particle counter (P-Dust Monit, conTec Engineering Srl) was used to carry out the particle count and to determine the concentration of particulate matter (PM). The particles were classified into fifteen different dimensional classes and PM concentrations were expressed as PM<sub>10</sub>, PM<sub>2.5</sub> and PM<sub>1</sub>. Furthermore, an air quality monitoring unit (MQA, conTec Engineering Srl) was used to evaluate the concentration of gaseous pollutants such as: CO, NO, NO<sub>2</sub>, SO<sub>2</sub>, O<sub>3</sub> and H<sub>2</sub>S. Finally, diffusive passive samplers (Radiello®, Fondazione Salvatore Maugeri-IRCCS) were used to determine the concentration of BTEX (benzene, toluene, ethylbenzene and xylene). In order to identify possible spatial variations, the studies were conducted in different sites and at different heights in the Sanctuary. Moreover, air quality was monitored before, during and after religious ceremonies in order to evaluate the impact of the presence of people on the concentration of specific pollutants. Particular focus was given to the Easter Mass and the Easter week during which liturgical services which attracts a large number of people were carried out. Also, a comparison with outdoor values were performed, in order to highlight accumulation phenomena and other variations in the concentration of the species. Preliminary analyses on the conditions of some wooden sculptures present in the two main chapels of the Sanctuary were carried out using X-Ray Fluorescence (XRF) directly on the works of art and Scanning Electron Microscopy coupled with Energy-Dispersive X-ray spectroscopy (SEM-EDX) on the dust deposited on the sculptures, which was collected with an appropriate brush. This enabled to establish the presence of degradation phenomena originating from poor indoor air quality and microclimatic conditions.

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## Study of the catalytic action of heavy metals in the sulphation process through experimental tests in climatic chambers

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Research conducted on the degradation of cultural and architectural heritage caused by the deposition of air pollutants indicates that both sulfur dioxide and particulate matter produced by the combustion of fossil fuels are the main agents responsible for the deterioration of carbonate materials (marble, limestone, etc.). The main chemical degradation process is the sulphation of the substrate and consists in the initial conversion of sulphur dioxide (SO<sub>2</sub>) into sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) and the subsequent reaction of sulphuric acid with calcium carbonate. This leads to the formation of dark heterogenous encrustations known as black crusts, which are composed of gypsum inside which particulate matter is embedded. Heavy metals adsorbed on the carbonaceous particles enable the sulphation of the substrate by acting as catalysts in both stages of the process. Despite the presence of numerous studies related to black crusts formation, a specific role of the single metals is still unclear. Aiming to unveil the catalytic action of different metals, a series of experimental tests was carried out in specific climatic chambers for accelerated aging.

Accordingly, several Carrara marble specimens were prepared for exposure in the chambers by treatment with particulate matter, metal ion solutions, or mixtures of metal ion solutions. Graphitic carbon was also added into the samples (except for the ones covered with particulate matter) to simulate the behaviour of elemental carbon present in polluted outdoor environments. The simulation of the aging process was carried out with the use of a two climatic chambers for corrosion tests in a humid atmosphere with sulphur dioxide and exposure cycles simulating solar irradiation (xenon arc climatic chamber). The exposure of the specimens was performed for four consecutive weeks and, after each week, a portion of the samples was retrieved for analysis. The chemical characterisation was carried out before and after the accelerated aging tests by means of a multi-analytical approach involving different techniques. Colorimetric analysis was used to evaluate the variation of the chromatic coordinate L (brightness), which is related to the formation of gypsum on the surface. Scanning Electron Microscopy coupled with Energy Dispersive X-ray spectroscopy (SEM-EDX) was employed to study the elemental composition and morphology of the surface and degradation products. Moreover, X-Ray Diffraction (XDR) was used to identify the mineralogical composition of the degradation layer and Ion Chromatography (IC) was performed to identify and quantify soluble salts, with particular focus on the sulphate ions. Finally, Thermogravimetric Analysis (TGA) was carried out to quantify the amount of gypsum formed, along with organic and elemental carbon present in the black crusts.

All the experimental data collected will be processed in the next stage of the project to create a predictive mathematical model. This will help predict the formation of black crusts on carbonate surfaces based on the outdoor pollution present in a given site. In fact, this research work is part of the interdepartmental SEED 2019 project of the University of Milan entitled SciCult (mathematical modelling and Scientific analysis for Cultural heritage: prediction and prevention of chemical and mechanical degradation of monumental stones in outdoor environments).

## Guaranteeing the authenticity of VENINI Murano glass: handheld XRF analysis as a tool for a first compositional database

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Non-destructive and in situ analyses of unique and fragile objects, such as glass and enamels, are currently a significant challenge of the archaeometric research. In this context, handheld X-Ray fluorescence spectrometry (p-XRF) can assure a large contribution for studying the chemical composition of the artifacts without causing damages [1][2][3].

Thanks to the financial support provided by Pentagram Stiftung Foundation, the results reported in the present work concern the qualitative and semi-quantitative analysis of sixty-four transparent vases from a private collection made from VENINI furnace on Murano between 1921 and 1980s. The main goal of the study was to establish a solid database of chemical elements concentration ranges, in order to compare the composition of vases of certain provenance and epoch with objects of uncertain/doubious origin and period, preserving and promoting the authenticity of Murano glass. To do this, the method of analysis has been deepened by exploring the instrumental limits and developing an analytical protocol to ensure a good analytical quality as well as to provide reliable data comparable with the bench technique. Taking into account the well-known constraints of the analytic technique, preliminary quantitative calculations tests using standard glasses of similar composition were performed in order to define and validate the analytical protocol. [4][5]

The results allowed to identify elemental compositional ranges for VENINI base glass of the '20ies, 50'ies and '80ies, respectively, and to define the elements added for colouring or opacifying the glass. Some outsiders were detected and their attribution was questioned by requiring a stylistic verification of the objects.

Colorimetric measurements were carried out for proposing the association between the values of colorimetric coordinates ( $L^*a^*b^*$ ) and the chemical composition of the objects. Further quantitative studies are necessary for a sound confirmation of the hypothesised relationship between slight chromatic differences and chemical features.

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**SESSIONE 8:**  
**DIGITALIZZAZIONE E MODELLIZZAZIONE 3D**  
**NEI BENI CULTURALI**

## Virtual archaeology and 3d printing: applications in the field of scientific research and communication

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**Introduction:** In recent years, the spread of 3D modelling and printing technologies has provided new tools and different solutions in the field of archaeological research and communication. The aim of this research is to experiment the possible applications of this tools in the funerary archaeology, studying anthropological remains and grave goods, to verify the possibilities offered for the reconstruction of the deposition context and for the dissemination to the public of multimedia contents, that reflect the FAIR principles (<https://www.go-fair.org/fair-principles/>).

**Content:** The modern technics of 3D modelling and printing of archaeological findings and contexts help integrating traditional research methods and communication activities.

One of the fields in which the contribution of photogrammetry and 3D printing technologies has been experimented was the study of osteological findings, pertinent to the skulls of buried individuals, found in the archaeological excavation of the late roman necropolis of Vaste (Lecce, IT).

In this case, the modelling and printing of the archaeological finds proves to be of great addition to develop the research activity, through the attempt to reconstruct the physiognomy of the individual, using the 3D model as a basis for the modelling of the physiognomic traits.

Furthermore, the 3d printed replicas of the finds and the context allow the tactile experience within didactic laboratories and tactile paths in the museum exhibition.

The stereolithography 3D printing technique was also used to reproduce the headpiece composed by 21 deer canines discovered inside a woman's grave, dating at the early stages of the Upper Paleolithic, in the site of the Grotta delle Veneri of Parabita (Lecce, IT). The restitution of the set was made possible thanks to a 3D scanning process of the deer teeth, made by Laboratorio 3D Cordinamento SIBA, University of Salento, and the following process of 3D printing and characterizing of the archaeozoological finds.

In this case, the use of 3D printing proved to be useful for reproducing the original appearance of the burial garment worn by the woman. Indeed, in the Historical-Archaeological Museum of the University of Salento, thanks to this work is possible to observe, next to the ancient remains, the original appearance of the headdress exposed.

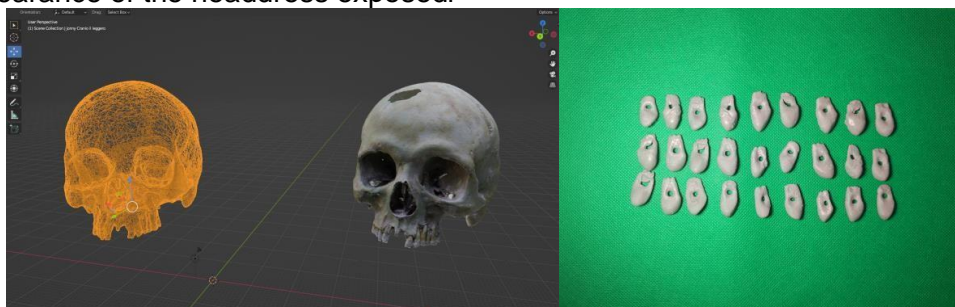


Figure 1: Virtual model of the skull found in the late roman necropolis of Vaste (left); the 3D prints of deer teeth found in the Grotta delle Veneri, Parabita (right)

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# Music in the digital era: X-ray CT imaging analysis and 3D reproduction of historical wind instruments

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Historical musical instruments preserved in museum collections are objects originally made to communicate and entertain with the music they produced. Unfortunately, playing historical woodwinds is particularly problematic; indeed, the humidity produced by the musician's breath during a performance represents a significant risk in terms of conservation, such as possible formation of cracks and deformations of the wood. As a result, most museums do not allow historical wind instruments to be played, thus depriving them of their musical meaning. Until now, the creation of replicas has been based on manual measurements of the originals, and both measurement and manual manufacturing leave a considerable margin of choice to the maker, which makes unfaithful copies very common. Today, the possibility of using non-invasive analysis techniques and digital technologies for measurement and modelling offers new opportunities in this field. The presented project has the purpose of developing a methodology for the reproduction of ancient wind musical instruments from museum and private collections by means of digital technologies. In particular X-ray Computed Tomography (CT), computer modelling and 3D-printing, can be employed to recreate the sounds of ancient instruments for the contemporary public, preserving at the same time the physical integrity of the originals. X-ray CT scanning is a non-invasive technique already successfully applied for the study and analysis of internal structures and features of different types of cultural artefacts, in some cases also by means of synchrotron radiation [1]. The use of imaging techniques in the field of musical instruments has been validated by recent studies [2], in which shapes complexity, variety of materials and objects size make high-quality CT acquisition non-trivial. In this work, starting from indications and tips provided by the MUSICES project [3], in particular on the woodwind instruments measurements, tomographic analysis on three wind instruments made of wood with some ivory details were carried out: a piccolo flute of unknown manufacture and a baroque flute (Traversiere) by Lorenzo Cerino both from the late Eighteenth Century, and a copy of a renaissance recorder by Fulvio Canevari from the 1970s. The CT measurements were performed at the Physics Department of the University of Turin, in collaboration with the Chemistry Department. After the analysis of the obtained data and the digital modelling of the objects, high quality and precision copies were realized by means of 3D printing technology, comparable in terms of size with the original instruments. 3D printed parts were fitted and finished with traditional flute-making techniques, resulting in good sounding instruments. Acoustical analysis, based on both physical measurements and testing sessions with professional musicians, shows that the technology has a high potential for producing faithful copies of ancient instruments, although some present limitations need to be addressed.

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## CAVE, Archeology and virtual reality: visiting unreachable places

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'Beyond Archaeology' is a methodological project that implements a digital approach from the excavation plan to the interpretation of the finds and the exhibition of the results ([www.bearchaeo.com](http://www.bearchaeo.com)). In particular, the BeArchaeo database hosts all the information on the finds and the excavation activities carried out at the Tobiotsuka Kofun, located in Soja city in Okayama Prefecture of Japan. Together with other Kofun burial mounds and the related archaeological material in ancient Kibi and Izumo areas, researchers aim to develop a transdisciplinary vision in studying the archaeological site and other archaeological materials now stored in museums and laboratories. The photogrammetric surveys have produced a comprehensive 3D model of the Tobiotsuka Kofun, also put in relation to the photogrammetric surveys on the finds.

Going from the acquisition of the photogrammetric data to the exhibition of the results, we have developed a narrative virtual interactive model for a Cave Automatic Virtual Environment (CAVE), that is a video theater with three walls made up of rear projection screens and the floor with a downward projection screen. The CAVE is implemented by four computers that work by coordinating the projections on the screen. The user wears 3D glasses that receive stereoscopic images in dependence of a motion capture system that acquires the user's position in real time. The installation focuses primarily on the relief of the burial chamber, thus allowing a visit to the tomb exactly as it appeared to the eyes of archaeologists immediately before the start of the activities of excavation. In the same way, being in possession of the reliefs of the excavation trenches along the sides of the mound for a number of days of excavation, activators were inserted to show the transformation of the excavation along the progress of the archaeologists' activities.

This paper illustrates the realization of the interactive narrative virtual environment, with a non-immediate solution, as the CAVE is configured as a complex system that synchronizes four independent displays harmonized through to a forced perspective that focuses on reference users. We introduced a reference cursor which, in addition to having the possibility of interacting with the space around it, also projects a shadow, essential for increasing the sense of presence in the simulation. The surrounding landscape implements a progressive definition reduction system downsizes the processing load by extrapolating the relief curves directly from the satellite images. The most representative finds that were found during the excavation and other contemporary ones stored in nearby museums are displayed to represent the reference culture of the structure, together with explanatory tables edited by the archaeologists.

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## Reference Information System in 3D for the exploitation of heterogeneous archaeometric data. Case study of a cave

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A Referenced Information System in 3 Dimension (RIS3D), inspired by GIS, has been developed by the Archeovision platform (Dutailly 2022). This tool is adapted to the multi-nature collected data. Each data is geolocalized in a 3D model acquired by digital photogrammetry. All data are gathered in a dedicated database. The database is integrated in the RIS3D. The end-user is able to query the RIS3D about any kind of data, taking advantage of the localization, aiming at a providing a better spatial interpretation. The case study is a cave named *Leye*, situated in the Vézère Valley (UNESCO site in Dordogne, France) where some famous ornated caves stand (Lascaux, Font-de-Gaume, Combarelles, etc.). This *Leye* cave offers many advantages: (i) it does not present any painting or drawing, therefore any type of scientific instrument can be set in, and any sample can be taken out, (ii) it shows the same environmental parameters as for the ornated caves, and (iii) more specifically it shows the presence of two types of crystallized whitish materials called moonmilk and coralloids which appear as a white veil covering areas on wall surfaces (Lacanette *et al.* 2013, Bassel 2017, Chapoulie *et al.* 2017, Mauran *et al.* 2019). In this cave, it was possible to register many different types of data either of physical or chemical nature: temperature, CO<sub>2</sub> content, relative humidity, air speed, chemical composition, physical structure. Data were collected using *in situ* dedicated sensors and mobile analytical equipments (OM, pXRF, pRaman), or by sampling and further analyzing into laboratories (XRD, SEM-EDX, micro-Raman). To know more of this veil (identification, origin, growing process, chronology), observations and analyses have been carried out for more than 15 years. All analyses provided a big amount of data : texts (comments, operator, date of work, reference number), pictures, 2D graphs... The RIS3D includes a database and enables to see all the stored data thanks to their location, helping to simultaneously test several hypotheses for understanding the role of each component or parameter into this alteration process. This work opens up promising prospects in the field of prehistory and geoarchaeology as well as in conservation-preservation issues.

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## Decay assessment and 3D surface modelling of historical brick masonries in Venice

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In many noteworthy buildings in Venice we can observe a strong loss of material at the bottom of the façades (usually in the 1-2 meters from the soil level). Capillary rise, water condensation, evaporation sub-florescence of salts can induce scaling and cracking in bricks with a subsequent reduction of the thickness of the wall section and therefore of its load-bearing capacity. This work proposes a new approach to assess the vulnerability of historical brick masonry patrimony, the most used in Venice. This new approach provides for three main steps: i) the minero-petrographic characterization of the materials (bricks and mortars); ii) the 3D photogrammetric survey and the measurements of the cross-sections of brick walls; iii) the decay assessment. For this purpose, the Church of Santa Maria dei Servi (Cannaregio, 14th century) was selected as case of study. Color, mineralogy and texture of the bricks and mortars, and secondary weathering products were studied by means spectrophotometry, X-ray diffraction, optical and scanning electron microscopy, and hyperspectral analysis. 3D surface modelling by empirical descriptions based on photogrammetry was used for understanding the effects of water and salts interaction in the bricks wall of the main façade. Results will be useful to provide previsions of long-term deterioration of that monument and they can be implemented in other brick buildings in Venice, which have the same decay pattern.

# POSTER

## Non-invasive Diagnostic Techniques to Protect Cultural Heritage from the Threat of Climate Change

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The growing attention paid towards the devastating effects of climate change on the environment has progressively led to a renewed interest in the wide range of damage also produced on Cultural Heritage. It is increasingly necessary to adopt environmental monitoring techniques in sites of historical and artistic interest as well as museum interiors, with the main purpose to gain a deep understanding of the factors that can accelerate the degradation of Cultural Heritage, in an effort to adopt preventive measures instead of invasive restorations. Monitoring methodologies are in continuous development, as evidenced by the following case studies from the Royal Site of Carditello (Caserta, Italy) an 18<sup>th</sup> century Bourbon palace, and from the village of S. Agata dei Goti (Southern Italy) where an environmental monitoring campaign was recently carried out in collaboration with the startup 'Energreenup s.r.l.'. The project required the installation of an integrated system of wireless sensors for the microclimatic control of the site, both indoor (measuring the parameters of Temperature, Humidity, NO<sub>2</sub>, SO<sub>2</sub>, Brightness) and outdoor (Temperature, Humidity, NO<sub>2</sub>, SO<sub>2</sub>, O<sub>3</sub>, PM, Rainfall, Anemometry, Seismic Probes, Visible and Ultraviolet Radiation), in association with non-invasive archaeometric analyzes to investigate the state of decay of the decorative elements of the sites and monuments. The use of non-destructive diagnostic techniques, such as IR thermography, UV fluorescence, X-ray Fluorescence (XRF) and infrared reflectography, were useful not only to evaluate the conservative state of the mural painting located at the noble floor of the Royal Site of Carditello, but also to distinguish the original pigments from the materials used during the restorations dating from the 19<sup>th</sup>-20<sup>th</sup> centuries. All the detected measures were transmitted to a web platform for data collecting, allowing a real-time monitoring of the environmental parameters of the site. The results achieved from the monitoring campaign allowed to gain a more precise perspective upon how thermo-hygrometric variation can produce physical alteration, distress and chemical reactions depending on the materials affected and how non-invasive analyzes conducted on the mural paintings could also be useful in planning future consolidation works based on their state of decay. In the long term, the data analysis could be useful to anticipate the damage before it even occurs, thus optimizing the safeguard of Cultural Heritage with minimally invasive maintenance interventions. This type of innovative approach has proved to be ideal for identifying chemical, physical and biological alterations caused by atmospheric agents and pollutants on Cultural Heritage, but it is clearly necessary to invest more in the application of monitoring techniques on a larger scale, while raising public awareness on the need to limit the atmospheric agents that jeopardize its conservation.

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## Archeometric study of two tanagrine's statuettes

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In the context of forensic investigations and in the field of cultural heritage the multidisciplinary approach is very important to collect data and valuable information on historical and archaeological finds. Such investigations take on particular value when artefacts come from clandestine archaeological excavation contexts or are the result of investigations addressed to the fight against illicit trafficking of cultural goods of unknown origin. The multidisciplinary approach analyzes and investigates the various aspects of a work such as: raw materials, minero-petrographic features, construction techniques, etc. The analysis of raw materials through archaeometric investigation allows in fact to increase the possibility of obtaining important compositional and technical data, also relating them to possible historical-artistic and archaeological contexts, considering the stylistic connotations of the artefacts. This study highlights the data obtained from stylistic and archaeometric analyses, carried out on two statuettes of the Tanagra's type confiscated by the Cosenza Carabinieri Unit for the Protection of Cultural Heritage and Anti-Counterfeiting (Calabria, Italy). The stylistic production turned out to be close to the votive statuettes made in the Greek Magno area, especially in the territories of the ancient Apulia, Sicily, Locri Epizephiri and ancient Lucania. For these purposes, the analytical approach involved the use of minero-petrographic and physical analysis, as follows: petrographic analysis (OM), X-ray diffraction (XRD) and thermoluminescence tests (TL). The preliminary observation, which highlights differences in the stylistic features of the two statuettes as well as in the colour, morphology and distribution of the white-greyish patina, is further confirmed by the TL investigations. The TL test revealed an ancient production only for one of the analysed finds and the investigations on the raw materials allowed to relate this to a possible local historical-artistic context. The second statuette, on the other hand, is attributable to a modern production as confirmed by TL measurement.

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## Ancient polychromy and terracotta technology in the Sanctuary of Athena at Castro (Lecce) – Preliminary results of a MOLAB access

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Systematic archaeological research carried out since 2000 brought to light the ancient sanctuary of Athena in the indigenous settlement of Castro (Lecce), on the Adriatic coast of the ancient Messapia region (southern Apulia) (D'Andria 2009; D'Andria 2020). The settlement played an important cultural and religious role during the Archaic, Classical and early Hellenistic period (7th-2nd century BC), as a place of integration and exchange between local groups, the Greek inhabitants of Tarentum, and the Greeks from Macedonia, Epirus and the Corinthian colonies in the Adriatic Sea. Among the many significant discoveries, sculptural and architectural materials allow us to deeply investigate the craft productions both of Messapians and Tarentum. Indeed, to highly specialized Tarentine workshops are attributed i) a late Archaic clay roof of a monumental temple (about 520 BC), ii) a colossal statue of Athena in local limestone (350-300 BC) (D'Andria 2020), iii) some gigantic relief slabs decorated with 'peopled' scrolls (350-300 BC) (Ismaelli 2020), iv) Doric frieze blocks of a late Classical calcarenite monument.

A recent MOLAB access, financed by the Italian node of E-RHIS-IT (AthenaInColor), offered the opportunity to investigate ancient polychromy, terracotta and stone technology, benefiting from the exceptional state of conservation of the archaeological materials. Since the potential of archaeometric approach to these craft productions has been so far neglected, the MOLAB project intended to fill the gap on the technical know-how of Magna Graecia artisans.

For the limestone Athena colossal statue, the peopled friezes and the Doric friezes, a sustainable protocol based on imaging techniques (VIS, UVL/VIL) coupled with non-invasive techniques (p-XRF, External reflection mid-FTIR and reflectance spectroscopy) was conducted in order to detect traces of ancient polychromy. Besides the identification of yellow and red ochres, Egyptian blue, and the presence of preparatory layers UVL observations demonstrated the extensive use of organic treatments on all the finished limestone surfaces, a technological solution not attested so far for limestone sculptures in Magna Graecia. Analyses on fragments sampled on these areas are still ongoing. On architectural terracottas, the research focuses on raw materials, the supply of selected stone for the aggregate, and the technology of firing. Special attention was devoted to the painting technique and pigments analyses, since many questions remain open on the technology of Archaic terracotta architectural elements.

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## Investigating provenance with a proton microbeam: the case of red figure pottery from Locri Epizephiri

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Proton Induced X-ray Emission is a well-established technique for the study of trace element concentrations inside materials, ensuring at the same time a high sensitivity and a complete non-destructive approach, suitable for the investigation of valuable Cultural Heritage objects [1]. In cases where different sources of the raw material employed for artworks present different chemical composition features, even at the trace level, the element concentrations obtainable with PIXE can become strong markers for provenance; many interesting applications of this concept can be found in the literature concerning a wide variety of materials, such as lapis lazuli [2], gold [3] and pottery [4]. Furthermore, the possibility of focusing the proton beam down to the micrometric scale allows to investigate very small areas of the sample having a high spatial resolution.

These features of PIXE have been exploited for a research initiated by the group of the Department of Chemistry of the University of Turin more than 10 years ago and still ongoing on archaeological pottery decorated with vitrified black slip found in Locri Epizephiri (southern Italy) [5]. Black-glaze ware and red figure pottery was produced in Southern Italian workshops during the 6<sup>th</sup> to the 4<sup>th</sup> century BCE, according to a Greek technological process [6]. Previous archaeometric studies by means of SEM-EDX and ICP-OES analyses [5] unveiled the possibility of discriminating between a Greek and an Italian origin starting from major elements concentrations. Instead, provenance markers for distinguishing Calabrian from Sicilian objects could lie in the trace element composition of the very thin vitrified gloss (some 20 nm) on the pottery shards, considering the hypothesis that different workshops would have used raw materials from different sources to obtain their products. The PIXE investigation on this thin vitrified layer performed at the microbeam line of the AN2000 accelerator at INFN-LNL will be presented.

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## From site to ceramic petrology: the pottery of the earliest foundry of Pre-Roman Padua and its economic entanglement

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The earliest foundry of Pre-Roman Padua (north-eastern Italy), and other nearby spaces used for pottery production were only partially excavated at the courtyard of the Questura building in 2000-2001 (Michelin, 2021). A large “earthen block” with a dense insulating filling of ceramic sherds below the floor of the metallurgical unit was removed to be excavated in laboratory and record all operations with videos and 3D digital reconstructions of the findings. The ceramics from this and other foundry's context include specific types formally coherent with pots common in the hinterland of Friuli (north-eastern plains and Adriatic coast). A hypothesis is that such pots might have been used in a systematic trade of salt from the sea coast, and that salt, in the central markets of early Padua, could have been a medium of exchange with other high-cost goods, such as copper and textiles.

Previous studies (Tenconi et al., 2013) on this type of pottery found in various sites of the Veneto region, pointed out that the clay was deliberately tempered with crushed fragments of speleothems, probably coming from karst environments whose geographical distribution is limited to few localized areas of north-eastern Italy.

We will take advantage from the clear evidence of local pottery production. The present study presents the preliminary analysis of pottery, which includes large fragments of firing mis-products. We will apply an innovative approach which links each potsherd to a 3D model and, through the thin-section and microstructural analysis, aims to define with high precision the production technology of this ceramic class. Quantity of added temper and its grain-size will be measured by digital image analysis, whereas the firing temperature estimated by X-ray powder diffraction on the fine fraction (micromass), as well as microstructure of the calcite-based temper.

The provenance of the clay will be determined by microchemical analysis at the SEM-EDS, and on the basis of type and quantity of heavy minerals. The provenance of the temper will be defined using stable isotope analysis by mass-spectroscopy on the fragments mechanically extracted by the ceramic body. All these pieces of information will address provenance and production technology of this exotic ceramic class, into a unique and comprehensive archaeometric study.

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## Between archaeology and archaeometry: a multidisciplinary approach for the study of the Roman building materials of Aquileia (Udine, Italy)

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Nowadays Aquileia is a small village in the North-East of Italy, but in ancient times it was one of the richest cities of the Roman Empire and an important commercial knot amongst the Po plain, Central Italy, the Alpine passes and the Balkan peninsula.

Today the traces of the great past of Aquileia are barely evident because of the intensive spoliation activities occurred during the Middle Ages and in modern times. Nevertheless, extensive archaeological excavations carried out in the last two centuries of many houses, public buildings, roads and infrastructures allowed to propose a valid reconstruction of the ancient urban layout.

In recent years, the architectural remains of these buildings have been the object of a multidisciplinary research project carried out by a joint collaboration among archaeologists, geologists and archaeometricians of the University of Padua, aimed at reconstructing the dynamics of supply of building materials, the technical knowledge of Roman builders and their ability to deal with construction in marshy environments.

The research focused on the characterization of a conspicuous number of samples collected both from building stones (local and imported) and mortar-based materials, which were employed in walls, mosaics and infrastructures of the city.

In order to locate the origin of the raw materials used in Aquileia, the study of the territory's resources was deepened by means of a survey of ancient quarry areas, from which samples were collected for comparison with the archaeological materials.

In last years, a specific attention was devoted also to perishable materials, and in particular to timber, usually preserved in Aquileia only in anaerobic conditions (i.e. piles in foundational environments). Adopting dendrochronological and radiocarbon analysis the wood samples were used to get information about both the provenience of the raw material and the chronology of the infrastructures.

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## Imaging and punctual analysis to support the restoration of the 17th painting “Coronation of the Virgin with the Saints Ambrose and Jerome”, attributed to the Tuscany-Umbria area.

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The aim of this work is to combine non-invasive imaging with chemical characterization analyses in order to study original and restoration materials of a late 16<sup>th</sup> century painting on a canvas representing the “Coronation of the Virgin with the Saints Ambrose and Jerome”, preserved in the Diocesan archive of Orte, a town in the district of Viterbo (Italy). The diagnostic campaign was addressed to support the restoration activities and the choice of the most suitable and sustainable cleaning operations, also by using green solutions and methodologies. For this reason, both traditional analytical techniques and multispectral imaging were applied to solve the diagnostic issues and best address the restoration of the painting. Specifically, hypercolorimetric multispectral imaging (HMI), X-ray fluorescence spectroscopy (XRF), Fourier transform infrared spectroscopy (FT-IR) and gas chromatography coupled with mass-spectrometry (GC-MS) were combined to obtain information on the general conservation state of the artwork and the characterization of pigments, binders and superimposed materials, these last being particularly important to identify ancient and not-documented restoration intervention, enabling a correct choice of the most suitable and effective cleaning intervention.

Multispectral data allowed to differentiate and map original materials through infrared and ultraviolet false colour images and spectral reflectance-based similarity maps, suggesting pigments attribution and focusing punctual analysis for characterization. This approach was particularly successful to identify and locate the presence of unaltered smalt blue in a first painting coat then covered with other pigments and to suggest the use of organic dye in mixtures with cinnabar and ochres. Spectroscopic and chromatographic techniques enabled to respectively identify the painting palette and to confirm the use of oil-based binder for the pigments and characterize the altered top layers, made with a natural resin and an animal glue. Their characterization was essential to select the most suitable methods and materials for the biological cleaning experimented during the restoration activities.

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## The contribution of archaeometric-diagnostic analyses to the restoration of the narthex of the San Marco Basilica in Venice (Italy)

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In the narthex of St. Mark's Basilica in Venice, at the sides of the San Pietro portal, there are two pairs of ornamental columns, installed in the early decades of the thirteenth century using ancient spolia. The capitals, dating back to the 9th century, are believed to have been taken from the imperial palace in Constantinople after 1204. The shafts are made of a monogenic black and white breccia and were probably taken from the ruins of an ancient building in Aquileia, broken into two or three parts. When relocated to St. Mark's, they were repaired with *doroni* and iron brackets. The bases, which are adorned by small lions, are spolia from an unidentified building.

The columns – rendered unsafe due to serious fractures in the bases of the shafts caused by the oxidation of metal pins as a result of many centuries of contact with high tides and flooding – were recently dismantled, their capitals removed and their shafts raised to allow structural consolidation. The mineralogical-petrographic (optical microscopy and XRD) and chemical-physical (pXRF and FTIR) investigations that accompanied the restoration enabled the breccia used for the shafts of the columns and the crystalline marble of the capitals to be identified, respectively, as *Marmor Celticum* (from Aquitaine, in the central French Pyrenees) and Proconnesos white marble (from the island of Marmara, Turkey). They also proved possible to identify both the initial decorative finish of the capitals (cinnabar-based polychrome and gilding accompanied by small blue cobalt glass inserts), and techniques and procedures used in the 13th century for the reconstruction of fractured shafts: stone tenons and mortises accompanied by the application of metal pins fixed with colophony (rosin).

## Decay Assessment of Stone-Built Cultural Heritage: The Case Study of the Gerace Cathedral (South Calabria, Italy)

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The sustainable management of cultural heritage is a multidisciplinary process constituted by different steps. The first one is the diagnostic approach addressed to know the main degradation and alteration processes affecting a cultural site.

The Cathedral of Gerace, reconciling Greek and Latin characters, was built on the remains of a pre-existing sacred structure devoted to Aghia Kyriaki (Saint Ciriaca) dating back to VIII century, between 1085 and 1120, under Normans domination. The building is characterized by a triple nave and Latin cross, measuring 1898 m<sup>2</sup> and is the largest Romanesque church in southern Italy. The Cathedral is the most representative monument of Byzantine-Romanesque-Norman style in Calabria and it's impressive both inside and outside [1].

The present study aims to assess the different decay phenomena affecting Gerace Cathedral (Calabria, South Italy), both in its interior and exterior part.

In order to achieve that, a multidisciplinary approach was taken into account, combining in-site diagnostic with Non-Destructive and Micro-Destructive Techniques (NDTs and MDTs).

In particular in-site diagnosis, through InfraRed Thermography (IRT) [2, 3], allowed to identify and evaluate the intensity of the present decay forms, and choose the sampling area, in order to obtain information about the main degradation agents.

Specimens were undertaken to a complementary analytical approach, precisely: Polarizing Optical Microscopy (POM), Ion Chromatography (IC) [4], X-Ray Diffraction (XRD) [5], and Scanning Electron Microscopy coupled with EDS (SEM-EDS) [6].

The results archived highlighted that the main degradation forms are related to crystallization of different salts solution such as thenardite (Na<sub>2</sub>SO<sub>4</sub>) and hexahydrate (MgSO<sub>4</sub>).

The stratigraphic thin section and the SEM analysis allowed to evaluate the decay degree occurred on the stone materials and enabled proposing an intervention priority scale that can be helpful to institutions when planning a prompt restoration intervention.

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## Reuse of mining waste in the construction industry

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The present study takes place in the framework of REDiRECT project (Prog. n. F/160016/03/X41 – CUP: B22C21001250005) [1].

REDiRECT is an Industrial Research and Experimental Development Project promoted by Gruppo Ceramiche Gresmalt and Eurit in collaboration with the University of Modena and Reggio Emilia, the University of Calabria and the University of Sassari.

One of the project's purposes is to reuse mining waste, produced by national sites, in the ceramic industry.

The following study seeks to verify the suitability of those waste into another field of application: the construction industry and the restoration field. In particular, mining waste from Calabria region (South Italy) will be treated, using the same samples already characterized for REDiRECT project.

Those samples, after been characterized via a set of in-lab analysis, such as Polarizing Optical Microscopy (POM), X-Ray Diffraction (XRD) [2], and X-Ray Fluorescence [3], will be used as aggregate for mortar production. The mechanical properties of the mortars will be evaluated via compression [4] and ultrasonic tests [5].

At the same time, colorimetric test, water absorption and ultrasonic analysis were carried out in order to evaluate the compatibility with the support and also the mechanical features.

The results of this study may be useful to define an alternative use of mining waste and making mortar production a more eco-friendly process.

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## Archaeometric characterisation of wall paintings from Isera and Ventotene Roman Villas

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The poster aims at presenting the first results of a archaeometric research project carried out jointly by the Rovereto Civic Museum Foundation and the MUSE - Trento Science Museum, which consists in analysing wall paintings samples coming from different areas of the ancient Roman territory, in order to highlight similarities and differences related to the technique of decoration, the composition of mortar and the nature and origin of pigments. In particular, the first data collected concern the characterization of third style samples coming from the Roman Villa of Isera (Trentino) and the Roman Villa of Ventotene (Lazio) by analysis in thin section under the optical microscope and SEM-EDS microanalysis.

Keay words: Isera, Ventotene, Wall Paintings, Archaeometry, Thin Sections, SEM-EDS microanalysis.

## A chemometrics approach on XRF data to rediscovery the historical Cremonese lutherie

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Over the last ten years, a large set of spectroscopic XRF data has been collected on a large number of historical stringed musical instruments, the majority of which manufactured by Cremonese violin makers over the period from the 16th to the 19th century as a whole. All the instruments have been non-invasively investigated using the XRF spectroscopic technique by means of contactless and portable instrumentation, in order to highlight elemental peculiarities of each string instrument [1]. The analyses have been conducted in the Arvedi Laboratory of Non-Invasive Diagnostics of the University of Pavia, hosted in the Museo del Violino (Cremona).

The present work aims at providing a workflow driven by chemometric approaches to manage hundreds of XRF spectra [2]. To the purpose, a consistent database - consisting in a matrix (objects x variables) - has been constructed and preliminary explored by PCA (Principal Component Analysis). As visible in Figure 1a, the PCA decomposition allowed the identification of sample groups based on the instrument type. In addition, a more accurate visualization of groups based on the code of each single instrument allowed us to identify single violins that grouped together (Figure 1b). As the main goal of the project, the supervised classification approach, after a robust calibration, will allow us to discriminate objects (i.e. spectra) into classes based on materials, thus describing differences linked to violin makers and manufacturing methodologies. The proposed workflow has been tested for the first time on the entire XRF spectroscopic dataset currently available, in a perspective of continuous implementation and ampliation of it.

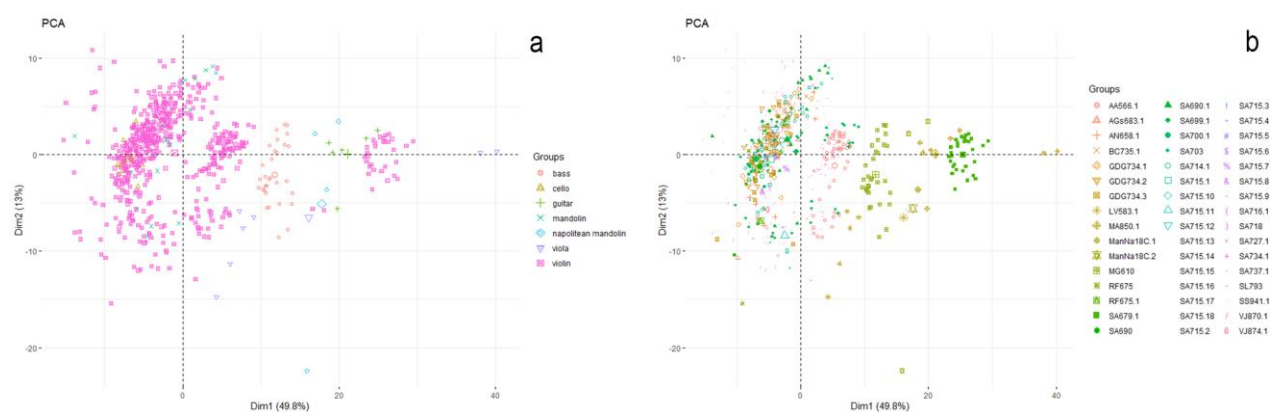


Figure 1. Score plots (PC1 vs PC2) obtained on the dataset collected on 61 string instruments.

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## Multianalytical investigation of pigments in the *Madonna della Croce* wall painting (Triggiano, Italy)

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The *Madonna della Croce* portrait is painted on a lunette, previously detached from a wall of unknown origin and now preserved on the right of the main altar in the *Madonna della Croce* church, in Triggiano (Bari, Italy). The wall painting, which depicts the Virgin and Child with St. Sebastiano and St. Rocco with their symbols of martyrdom, was realised by a local artist who worked between 1550 and 1570 in the town.

The numerous legends about the wall painting, mixed with historical events, talk about miraculous healing stories which has strengthened the strong relationship of the Triggiano inhabitants with this religious symbol, such as they built a dedicated church and chose this Virgin as patron Saint of the town.

The presented research mainly focusses on the investigation of pictorial layers of this painting by a non-invasive multianalytical approach. Firstly, shape, size and optical features of pigments have been observed using a portable digital microscope equipped by a polarizing filter. By means of a portable spectrophotometer, colour of painting areas has been measured and expressed in the CIEL\*a\*b\* system and for each of them reflectance spectra of visible light (400-700 nm) have been recorded. The identification of pigment composition has been carried out by FORS (fiber optic reflectance spectroscopy) and XRF (X-ray fluorescence spectroscopy). Produced spectra have been compared with databases available in literature.

Results have provided information about features and compositions of pigments, highlighting the use of red ochre, yellow ochre and carbon black for red and yellow paintings and for black profiles; in addition, mixing of red ochre and green earth and mixing of carbon black, lime and yellow ochre have been revealed respectively in correspondence to green background and blue Virgin mantle, as attested in other Apulian wall paintings (Fioretti et al. 2020; Fioretti et al. 2020).

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## Characterization of plasters from wine production vats

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The production of wine has very ancient origins and is one of the main characteristics of Mediterranean culture. During the Roman times, most of wine production installations are characterized by the presence of vats where the grape juice is collected before it is transferred in dolia or amphorae for the fermentation process. This work presents the study of plasters from Roman production installations located in North-eastern Iberian Peninsula. In particular, it focuses on the plaster coatings of vats used for collecting/storing grape juice. The aim is to investigate the characteristics of the plasters used in the coating of vats, their mineralogical, petrographic and physico-chemical properties, the production techniques and the raw materials used (De Luca *et al.* 2013, 2015). Moreover, we want to investigate if specific technological characteristics distinguish the mortars used in wine processing structures from other types of mortars and to verify their hydraulicity. The function of the structures has been confirmed by archaeological data and/or organic residue analysis (Pecci, 2021; Pecci *et al.* 2013).

The work is part of the activities of the ERAAUB, the IAUB, the Museo de Badalona, the projects RACAMed II (PID2020-113409GB-I00), CLT009/18/00045 (Generalitat de Catalunya) and the Cella Vinaria Project.

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## Analytical methodologies for the characterization of pigments from ancient Egyptian artefacts

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One of the most compelling and inspiring challenges in archaeometry is the identification of pigments in artefacts of historical and archaeological interest. Chemical-physical analyses allow the characterisation of the pigment's nature and possibly the origin and/or the manufacturing techniques employed. This information may enable a geographical and historical contextualisation and may also help in the dating of the artefact. Research studies conducted in this field were able to identify the vast array of colours the ancient Egyptians had at their disposal.

It is worth noting that every different shade was associated with a specific, symbolic meaning. The ritual and social use of colour resulted from theological beliefs, and every different hue had a precise, cultural value. This fact inevitably led to the utilisation of numerous pigments to decorate the works of art produced [1, 2].

The aim of this work was to characterise different samples of ancient Egyptian artefacts by identifying the employed pigments and highlighting, where possible, the presence of organic materials. The whole sample set was composed of sixteen fragments from three different artefacts coming from a private collection. A group of six micro-fragments were taken from the cover of an anthropomorphic sarcophagus belonging to the period between the XXVI and XXX dynasties. Seven other samples were collected from a wooden fragment of a different sarcophagus belonging to the Late Period of ancient Egypt (664-332 BC). Finally, three samples were obtained from the exterior and interior surfaces of a red container, better defined as a cylindrical wooden ciborium from the Imperial Roman period (I-III century AD).

The investigation of the fragments was conducted utilising a multi-analytical approach. The first observations of the pictorial textures were performed by optical microscopy. Then the semiquantitative determination of the elemental composition of the pigments was carried out by SEM-EDX (Scanning Electron Microscopy coupled with Energy Dispersive X-ray spectroscopy) analysis. Subsequently, investigations were carried out through molecular spectroscopic techniques, such as Attenuated Total Reflectance Infrared Spectroscopy (ATR/FT-IR) and Raman spectroscopy, to highlight the nature of the pigments. Finally, Visible Induced Luminescence (VIL) was employed as a further tool for pigment recognition. The VIL technique proved to be particularly useful in identifying Egyptian blue in the samples since it permitted the determination of its presence even where the other techniques did not.

This multi-technique approach applied to the study of the Egyptian fragments allowed the complete characterisation of the pigments used to decorate the artefacts. Also, some preliminary information about the presence of organic substances, both in the pictorial layers and in the sample coming from the ciborium's inside, was achieved.

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## Chemical characterization of a reddish coating on Egyptian coffins

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The polychrome decoration of Egyptian coffins characterized by a reddish superficial finish which covers the hieroglyphs were investigated. On all the artefacts analyzed, the finish is distributed in specific areas, suggesting a ritual or symbolic value. In some cases, it is locally affected by a strong blackening that partially hides the inscriptions and the underlying paint layer, the legibility of which can however be easily recovered with infrared reflectography.

The research was aimed at exploring the possibility to recover the darkened hieroglyphs and figures and make them readable again. Most of the polychrome wooden coffins object of this study were found excavating two XX Dynasty tombs built for two sons of Ramesses III (Setherkhepeshef, QV43 and Khaemaset, QV44) tombs reused later on; in fact, they are dated to the XXV-XXVI Dynasty. Moreover, they belong to three generations of the same family, over a period of about 50 years. In consideration of this, we initially hypothesized the use of the red finish as related to a family workforce or workshop, handed down from generation to generation. However, detecting the same reddish finish on another artefact (a fragment of a coffin lid of different chronology, New Kingdom, included in this study), we tend to speculate its widespread use.

The multi-technical and interdisciplinary study of these artifacts by non-invasive and microdestructive, spectroscopies (MO, SEM-EDX, XRF, FORS, FTIR, and UVF, UVR, RX, TAC imaging) and chromatography (GC-MS, HPLC-MS) based techniques allowed mapping the materials and unveiling the chemical composition of the red finish.

GC-MS and HPLC-MS analyses highlighted that the finish material is composed of vegetable oils, a triterpenoid resin (the use of a *Pistacia* resin is hypothesized) and a *Pinacea* resin, which are all commonly used materials in Egyptian funerary artifacts. In addition, brominated compounds were also determined suggesting the possible presence of purple.

## Pigments and glass powders: their use by Rogier van der Weyden

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Rogier van der Weyden (1399-1435) is considered one of the most important and influential Flemish painters of his time, among contemporaries such as Jan van Eyck. In this study, the painting "Entombment of Christ" belonging to the collection of the Uffizi gallery in Florence was analysed during conservation treatments at the Opificio delle Pietre Dure with the aim to characterise the materials and possibly to understand the painting technique.

Macro X-ray fluorescence (MA-XRF) was employed for this purpose as first non-invasive mapping approach: although alone cannot provide a comprehensive characterisation, it is nonetheless an invaluable tool for providing an initial overview or hypothesis of the painting materials and techniques. It is indeed one of the most essential analytical methods exploited in heritage science: providing spatial distribution elemental maps, not only it gives indications on the materials employed but also on production techniques of an analysed object, as largely demonstrated in this study. INFN-CHNet, the Cultural Heritage Network of the Italian National Institute of Nuclear Physics, designed and developed a MA-XRF scanner aiming to be a lightweight, easy to transport piece of equipment for use in in-situ measurements. This was the instrument employed for this study.

With a degree of uncertainty due to the technique itself, traditional Renaissance materials were determined or hypothesised. These include azurite, ultramarine, lead-white, vermilion, Cu-based green, Fe oxides/hydroxides (earth/ochres) and lead-tin yellow. The detection of Ca traces associated with some areas possibly suggests the use of dyes or lakes in which a calco-potassic glass, likely present as a drier/siccative, as consistent with the Flemish technique during those times. It is of course highly recommended to combine, when available, other analytical methods for an accurate identification of the compounds. However, MA-XRF is undoubtedly a powerful technique which is a source of relevant information, especially when employed as an early non-invasive and non-destructive analytical method, complemented by subsequent complementary scientific analysis.

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## Iron sulfides formed in freshwater in an archaeological context

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Archaeometry studies on ancient objects of common use give us insights on the ordinary life in the past. In this work, framed in the P.R.I.N. grant “food & S.T.O.N.E.S.”, we focused the attention on 31 of soapstone vessels fragments that presented baked encrustations presumably related with the presence of food remains.

Samples have been collected in the High Medieval village of Nogara (Verona) a centre collocated in the low plain south of Verona, on the riverbanks of the Tartaro river, that in the centuries 8<sup>th</sup>, 9<sup>th</sup> and 10<sup>th</sup> knows important development and structuration that culminates with the construction of a castle in the year 906 after a Diploma by King Berengario, main objective of the castle was the defence from the incursions of the Hungarians, but certainly there was also the will to give strategic importance to a centre in strong rise.

Nogara is in a plain context rich of freshwater; the large amount of fresh water saturating the natural and archaeological layers creates anoxic or strongly reducing environments in them, which can inhibit the action of many microorganisms and makes it easier to preserve perishable material such as wood, which is why these contexts are often valuable from an archaeological point of view. At the same time these conditions can facilitate other processes as we found during the analysis.

Stereomicroscopic observations have been carried out on the samples and on a selection of them have been performed SEM-EDS analysis.

During the SEM-EDS analysis performed on the encrustations, the presence of iron sulphides emerged; these iron sulphides probably have been formed by the actions of Sulphate-reducing microorganism that proliferates in strong reducing and anoxic environments.

The presence of the iron sulphides is not an archaeological data per se but, despite this, is very interesting for a couple of indirect information that can give us: first, these minerals generally have an important biologic contribute during their formation, confirming that the encrustations probably contained organic material (maybe food residues, or maybe plant residues from the nearby area); this organic material was metabolized by the Sulphate-reducing microorganisms; Second and possibly most important, the presence of iron sulphides in the encrustations can help us in the assessment of the paleoenvironment in Nogara during the Middle Ages and after, possibly confirming other historical and archaeological data that shows the presence of swamp phases during the Medieval and Modern age in the riverbank area archaeologically excavated; these phases in fact probably created, with the abundance of water in the layers, the ideal situation for the formation of the sulphides.

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## The “Byzantine house” of Giuseppe Torres in Venice: first characterization of the building materials and related deterioration products of a visionary home of the early 1900s

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As part of the project "*Venezia 2021. Programma di ricerca scientifica per una laguna regolata*", financed by the *Consorzio Venezia Nuova* and coordinated by CORILA, the private "Byzantine house", designed by Giuseppe Torres in 1905, was chosen as representative architecture largely built with concrete. Reinforced concrete and “artificial stone” (i.e., architectural concrete unit manufactured to simulate natural cut stone) are here combined with traditional materials that characterize the Venetian architecture, and this impose a specific reflection on their conservation in the aggressive lagoon environment. Various stone and lithoid materials as well as the related deterioration products, sometimes spectacular (Fig.1), were sampled from the masonry and decorations of the interior rooms located on the ground floor.



Figure 1. Efflorescence and loss of material on masonry.

The investigations concerned a wide range of materials (i.e. plasters, mortars, natural and artificial-reproduced stones, bricks, glass mosaic tesserae and painted finishing); they were carried out by means of a multi-analytical approach comprising: minero-petrographic and stratigraphic studies using scanning electron (SEM-EDS) and optical microscopy (the latter in both reflected and polarized transmitted light); ion chromatography; Raman and Infrared (FTIR) spectroscopy; powder X-Ray diffraction analysis. The results show a certain variability in the compositions of the mortar mixtures and a significant homogeneity of the mineralogical and chemical features of bricks and glass, respectively. The stones identified indicate a strict local supply clearly linked to the Venetian architectural tradition (Euganean trachyte, Istrian stone). In addition, the sands recognized in the mortars are mainly of regional origin and the glass are fully compatible with traditional Venetian recipes. Of particular interest is the presence of a peculiar *intonachino* (plaster) charged with ground fluorite and used as preparation layer for painting. The deterioration products are clearly linked to the extensive and intense phenomenon of capillary rising and efflorescence deposition induced by the lagoon environment.

This analytical study is part of a larger interdisciplinary research aimed at developing a methodology for reading and interpreting Venetian architecture. The multi-disciplinary contributions related to the characterization and mapping of materials and deterioration products, stratigraphic survey, identification of construction techniques and environmental monitoring, talk together and merge, with the ultimate aim of producing an overall vulnerability assessments of the building.

## Broad spectral range reflectance spectroscopy (from VIS-SWIR to Mid-IR) for *in situ* analyses of the painting “Mobili nella stanza” painted by Giorgio De Chirico

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Although multi-analytical approaches represent a good practice for the study of artworks [1, 2], in recent years, multi-sensor strategies have been developed leading to optimise the results obtained from several techniques into integrated data through chemometric approaches [3, 4]. From this prospective, broad spectral range reflectance spectroscopy from VIS-SWIR to mid-infrared (IR) (400 to 28000 nm) turns out to be very useful for providing information about organic and inorganic artists' materials used to realise their paintings [5].

In this work, we combined hyperspectral data from reflectance spectroscopy (VIS-SWIR) and External Reflection-FTIR spectroscopy in order to identify the pictorial technique and the chemical composition of the materials constituting the oil painting “*Mobili nella Stanza*”, painted by Giorgio De Chirico (currently stored at “Carlo Bilotti Aranciera di Villa Borghese” Museum in Rome). In detail, Visible (VIS) and Short-Wave Infrared (SWIR) reflectance spectra were collected in a non-invasive way by using an ASD FieldSpec® 4 Standard-Res portable spectroradiometer (350–2500 nm), while FT-IR spectra were acquired by using Alpha II (Bruker) spectrometer (7000–350 cm<sup>-1</sup>) equipped with the External Reflection module. The spectral data were combined in a multisensory way in order to create a classification model in continuous spectrum from visible to mid-infrared.

Complementary and non-destructive techniques (i.e., X-ray fluorescence, UV fluorescence and Raman spectroscopy) were performed to confirm the achieved results and to add other information to the pictorial layers.

This study is a part of the ARTEMISIA project (ARTificial intelligence Extended-Multispectral Imaging Scanner for In-situ Artwork analysis), funded by the Lazio Region.

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## Preliminary characterization of mortars from *Anfiteatro Flavio* (Pozzuoli, Campania region)

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The *Anfiteatro Flavio* is one of the most important archaeological sites of the Campania region (southern Italy) and one of the largest amphitheatre arenas of the Roman Empire, third in size only to the Colosseum in Rome (Lazio region) and the *Anfiteatro of Santa Maria Capua Vetere* in Caserta (Campania region). The site is located in an area of great archaeological and geological interest, the Phlegraean Fields volcanic field and in particular in the city of Pozzuoli (*Puteoli*).

The study and the analysis of the materials that constitute the *Anfiteatro Flavio* allows a better understanding of the roman civilization, whose legacy is still collected nowadays.

Thanks to the permission and collaboration of Archaeological Park of Phlaegrean Fields, we performed a non-invasive, but representative, sampling of 12 mortar samples in order to reach our characterization scopes and provide useful information on probable future restoration activities.

6 mortar samples date back to 1<sup>st</sup> century A.D and are bedding mortars between *opus reticulatum*, 6 samples dating 2<sup>nd</sup> century A.D and are bedding mortars between *opus latericium*.

Collected samples were studied by multiple methodologies (OM, XRPD, SEM-EDS). The results confirmed that Roman engineers extensively used tuff aggregate, hydrated lime, and in some samples ceramic fragments. In particular, the typical mineral association of phillipsite > chabazite > analcime points out the provenance of tuff aggregate from the Neapolitan Yellow Tuff (NYT) formation, related to the Campi Flegrei volcanic activity (de Gennaro et al., 2000).

EDS analysis have been performed on both the binder and aggregate fractions. *Lime lumps* and matrix are mainly formed by CaO. Hydraulicity Index (HI; Boynton, 1980) is relatively lower for *lime lumps*, which can be thus classified as aerial lime, whereas it is relatively higher for binder fraction, allowing a classification as weakly to moderately hydraulic. HI values confirm that hydraulicity of mortars is closely linked to the presence of pozzolanic material (represented by volcanic fragments). EDS analyses of glass of juvenile fragments and of mineralogical phases detected in the binder fraction confirmed a local provenance as they are in line with literature data relating to the Phlegraean Fields volcanic products.

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# Ultraportable system for LED-induced fluorescence spectroscopy coupled on a multi-technique scanner for the characterization of pictorial materials: a feasibility study

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The ARTEMISIA project (*ARTificial intelligence Extended-Multispectral Imaging Scanner for In-situ Artwork analysis*) aims to implement an innovative technology for the in-situ identification of pictorial materials. The prototype that is being developed involves the combination of hyperspectral imaging (VIS-NIR spectral range) and infrared spectroscopy in Fourier Transform (FT-IR) in reflection mode (MIR spectral range). The project includes feasibility studies for the coupling of complementary techniques, such as LED-induced fluorescence spectroscopy and X-ray fluorescence spectroscopy (XRF). Through LED-Induced Fluorescence spectroscopy in the UV range of 220-360 nm it is possible to obtain information on some painting materials such as synthetic binders (that are known that have a spectral signature in that spectral range [1]), dyes and pigments [2]. Therefore, the capability to couple UV LED sources on the ARTEMISIA prototype could be useful for the characterization of the possible presence of retouching, paints, fixatives, consolidants, biodegradation products, etc., on a paint surface, that are the information required during a restoration process. The aim of this work is to carry out a feasibility study on laboratory samples of painting materials by using an ultraportable LED-induced fluorescence system. Among the advantages of the proposed system, the ones that should be mentioned are its very small dimensions and weight, and the capability to use three different excitation wavelengths (280, 365 and 470 nm) allowing to study the spectral behaviour of the sample at different excitation wavelength. Furthermore, the possibility to adjust the power values and to illuminate the surface by using a probe, allows to perform LED-induced fluorescence analyses safely both for the artwork and for the operator. [3]. Once tested the LED-induced fluorescence system will be mounted on the ARTEMISIA scanner which will effectively become a multi-technical mobile station which is an aspect of increasing attention in the field of cultural heritage. This study is part of the ARTEMISIA project (*ARTificial intelligence Extended-Multispectral Imaging Scanner for In-situ Artwork analysis*), funded by the Lazio Region.

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# **New insight on metal finds, Cu-based nails and lead sheathing, of the Punic Ship of Lilibeum. Composition and conservation state**

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During the organization of the new exhibition of Museo Lilibeo, inaugurated in March 2017, numerous metal finds, belonging to the wreck of the Punic ship, were rediscovered in the deposits. These are very important elements for understanding the ship's construction method and, for this reason, a showcase has been dedicated to them, which is one of the most admired in the exhibition. The batch of finds consists of lead sheets used for the hull lining below the waterline and of several countless pegs and residual heads used for fixing the above sheets, of long and folded nails used to ensure the frames of the hull to the planking, of wooden dowels having nails inside them and of concretions of iron nails.

One of the questions asked by the archaeologists themselves at the time of the exhibition concerned the composition of the alloys. In the literature, the nails are defined tout court as "copper nails", but the detailed excavation report, published by Honor Frost, addresses the question in a critical and scientifically way, and deserves further investigation [1]. Then, the other questions concern the unusual corrosion, evident in the nails and in the lead sheets, and with a big variability of thickness and colors of patina and concretions.

Here, the non invasive investigation of 30 nails and 3 fragments of lead sheathing, belonging to the wreck of the Punic ship, is reported. Portable X-ray Fluorescence (XRF) and Raman Spectrometer allowed us to identify the elements and compounds constituting them making some discussion deductions about their composition. X-ray Diffractometry and Optical microscopy of a cross section permit to explain the degradation occurred in the underwater environment.

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## Study of Colantonio's paintings in the Capodimonte Museum using IR and XRF analyses

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Colantonio (1420 ca - 1470 ca) was the main Neapolitan painter of the fifteenth century and protagonist of the southern Renaissance, capable of making a synthesis between the Flemish model and its Mediterranean variants [1].

His experience in Naples is evidenced with panel paintings '*Saint Jerome in the studio*', '*Saint Francis of Assisi delivering the Rule to the Franciscan orders*', '*Deposition of Christ from the cross*' and the polyptych '*Episodes from the life of Saint Vincent Ferrer*', all dated between 1440 and 1460 and preserved in the Capodimonte Museum (Naples, Italy) [2].

Infrared Reflectography (IR) and X-Ray Fluorescence (XRF) analyses were performed on these paintings in situ to provide an initial overview and hypothesis of paint palette and techniques used by the artist.

To study the paintings by looking beyond the visible layers of paint and therefore to analyze the preparatory drawings and the regrets or changes made during the painting process [3], reflectographic scanning was performed using the Apollo camera by Opus Instruments.

X-ray fluorescence analysis [4,5] made it possible to determine the elemental composition of the paintings allowing the identification of pigments and materials used in paint and preparatory layer. XRF measurements were collected using the portable spectrometer Elio by XGLab Bruker using the point mode. For some regions of particular interest, elemental XRF mapping was also carried out. In this contribution we intend to present some results obtained from the application of the two noninvasive techniques, showing how important they are in enriching the technical and scientific documentation of Colantonio's paintings.

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## Combined spectroscopic techniques in a bench-top device for archaeometry applications

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Since 2002 the Solid State Physics group at the University of Torino has been working in the field of physics applied to cultural heritage in collaboration with other institutions, in particular with the National Institute for Nuclear Physics [1]. Studies have been conducted in several areas, of which the majors are the provenance of lapis lazuli by means of proton related techniques [2,3], and X-ray imaging and compositional analysis of historical and archaeological finds [4,5]. The measurements have been carried out in national and international laboratories (e.g. INFN-LNL in Italy, New AGLAE in France) as well as using facilities developed by the group [6].

In 2022, twenty years after the beginning of the activities, the development of a transportable and multi-technique instrument is underway. The project is carried out within the INFN-CHNet collaboration and has been also funded by the NEXTO project.

The device, based on the technology of the INFN-CHNet MA-XRF [7] consists of a handheld X-ray source and a multi-detector system, located on a three-axes motor stage. Two of the spectroscopic techniques available are X-ray fluorescence (XRF) and X-ray induced luminescence (XRL). In addition, the integration of other X-Ray based techniques is at the moment under development, such as digital radiography (DR). This device opens to the possibility of a multi-technical approach of X-ray techniques available *in situ*. Preliminary combined measurements with XRF and XRL techniques will be presented.

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# A combined XRF and $\bar{\nu}$ -Raman investigation of the Monument to the Fallen by Francesco Jerace in San Ferdinando (Reggio Calabria, Italy)

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The Monument to the Fallen in San Ferdinando (Reggio Calabria, Italy) is an imposing copper-based alloy sculpture, created between 1920 and 1926 by the famous sculptor Francesco Jerace (1853-1937), dedicated in memory of the sub-lieutenant Vito Nunziante and the fallen soldiers from the Great War. It has an important heritage value for the city of San Ferdinando, of which it is considered an identity symbol. The historical importance of the monument equally extends to all of Southern Italy, where Francesco Jerace's sculptures have the most valuable artistic representation [1].

This research highlights the results of a multi-analytical study, carried out *in situ* and in the laboratory by portable X-ray fluorescence (XRF) and  $\bar{\nu}$ -Raman spectroscopy, respectively, intended to characterize the elemental and molecular composition of the superficial layers of the alloy and the related corrosion products, gathering, thus, important information on both the materials and the state of conservation of the sculpture [2].

XRF data revealed an alloy mainly composed of Cu, Sn, and Zn, with Pb and Fe as minor elements. As far as corrosion products are concerned, the presence of oxides, chlorides, and carbonates was revealed by  $\bar{\nu}$ -Raman technique, mainly ascribed to the interaction between the alloy surface and the local weather factors, such as the effect of proximity and exposure to the marine environment.

The research was carried out on the occasion of the recent restoration of the sculpture, in the framework of a project promoted by the municipality of San Ferdinando, assisted by the Department of Mathematical and Computer Sciences, Physical Sciences and Earth Sciences of the University of Messina.

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## Italian maiolica between the 18<sup>th</sup> and 20<sup>th</sup> century: the contribution of Ion Beam Analysis

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Maiolica, i.e. tin-glazed earthenware, has been an important art form in Italy since the 14th century up to date. The technique to make the white tin-glaze, which characterizes it, and the colours to decorate it has remained very similar for centuries. Even after the Industrial Revolution ceramists continued to use the same main components (sand, lead, tin, wine lees, etc.), but some compositional modifications did take place thanks to the introduction of new chemicals on the market (Manca 2021).

This study aims at improving our knowledge of the technological evolution occurring in the production of Italian maiolica after the Industrial Revolution and, specifically, at acquiring information on the introduction of boron in the glazes.

Boron acts as both network former and flux and improves the chemical, mechanical and optical properties of a glaze (Beauvoit et al. 2021). Its use became more common in the 19th century, when new sources were discovered first in Tuscany and then in North America. However, boron is extremely light (atomic number  $Z=5$ ) and cannot be identified either by SEM-EDS, which is the golden technique for the analysis of maiolica, or by other common techniques, such as XRF spectroscopy, so that there is a scarcity of data about its presence in glazes.

The use of Ion Beam Analysis, and specifically Particle Induced Gamma-ray Emission (PIGE), allowed us to check the presence of boron and quantify it in a completely non-invasive way in more than twenty maiolica wares made between the 1750s and 1950s by important Tuscan manufactures, such as Ginori (Doccia), Cantagalli (Firenze), Chini (Firenze-Borgo S. Lorenzo) and the Società Ceramica Colonnata (Colonnata).

The set of reference data acquired in this study provided relevant information for the reconstruction of the history of maiolica technique and of the exploitation of boron, which will be useful also in future authentication studies.

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## A preliminary study of artificially corroded Cu alloys by neutron-based imaging

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When a copper-based artefact is buried in soil for a long time, the result is a corrosion patina characterised by complex chemical and metallurgical structures [1]. The study of these patinas requires the combination of several analytical techniques to characterise the corrosion products and to identify the degradation phenomena that have occurred during burial [2-3]. As a matter of facts, the interaction of the metallic artefacts with the archaeological environment plays an important role in the corrosion rate. Among the most widespread analytical techniques, non-destructive methods represent a very important tool for conservators and art historians to obtain valuable information about works of art without being too invasive. In the last decades, neutron imaging techniques have acquired great importance thanks to the results obtained in the Cultural Heritage field.

In this study, several artificially patinated Cu-based alloys with composition and microstructure like the one of archaeological artefacts were prepared in laboratory with the aim of providing a reference for neutron imaging. Firstly, several ingots with defined compositions (Cu-Zn, Cu-Sn, Cu-Sn-Pb) were prepared in laboratory. The final chemical composition was confirmed by Optical Emission Spectroscopy (OES) analysis. Afterwards, artificial patinas were produced on the samples surface by means of different chemical and electrochemical methods. The morphological and chemical characterisation of the artificial patinas was performed using Raman spectroscopy, X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). Finally, the samples were analysed with neutron imaging techniques (digital radiography and computed tomography). The aim of these measurements is to estimate the specific attenuation coefficient value and its spatial distribution in the samples, distinguishing between the alloy (inner part) and the patina (external part) which potentially have different density and composition in different areas. The goal is to determine the nature and properties of archaeological artefacts of unknown structure using the data obtained by analysing the Cu-based reference alloys. All neutron analyses were performed at the L.E.N.A. Centre in Pavia, where a thermal neutron reactor is available and a new facility for imaging studies has been recently developed in the framework of the CHNet-NICHE project and is currently under optimisation.

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## Towards an anamnesis of Troia Cathedral. History of diagnostic investigations and monitoring.

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Placed as a hinge between analyzes and decisions on interventions, the diagnosis involves interpretations on whose correctness may depend to a great extent the outcome of structural restorations and the future life of the building.

In the context of diagnosis, an important role is played by the knowledge of the history of restoration interventions and the investigations connected to them in order to orient and finalize new diagnostic investigations and to compare data acquired at different times with the dual purpose of integrating them and discovering constructive and structural variations occurred.

This paper is a review of the history of the diagnostic investigations and monitoring performed on the Cathedral of Troia between 1992 and 2004, which represent a valuable cognitive tool for further studies and research on the architectural structure.

The Cathedral of Troia is the emblem of religious building that have stigmatized Romanesque architecture in Puglia Region. The use of materials of different lithological nature gives to the façades a complex compositional language, unique and original to the other contemporary, Apulian, religious buildings. Between the end of the twentieth century and the early 2000s, some restorations and structural interventions, diagnostic investigations aimed at defining the mine-petrographic and physico-chemical characterization of lithotypes and cleaning and consolidation tests have been carried out. In addition, several monitoring campaigns of the state of conservation have been conducted to qualify and verify the experiments and interventions.

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## Fluorescent paints in contemporary murals: a case study

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Street art, here intended as visual art in urban contexts, has been recognized part of our cultural heritage only in the latest years. The ephemeral character, free access, and exposure to the environment and anthropic actions make public paintings vulnerable to neglect, removal, vandalism, and degradation. Beyond that, the strategies aimed at their preservation and fruition are rather unclear or lacking.

The target of our research is the development of an integrated protocol for the diagnostic of the of modern paint materials that constitute street artworks, aiming at a long-term sustainable monitoring and conservation. The optimisation of analytical methods is part of the PRIN2020 project "SUPERSTAR -Sustainable Preservation Strategies for Street Art" (2022-2025) an Italian network project setting as a goal the definition of innovative guidelines for the preservation strategy of street art (<https://prin2020superstar.dcci.unipi.it/>).

The results are useful to shed light on the chemical-physical properties and vulnerability aspects of street artworks, focusing on paint binders and inorganic and organic pigments. The latter are particularly prone to degradation as fading and the identification of the specific formulation used by the artists is pivotal for proper restoration and fine-tuning suitable conservation treatments.

Here we present the results of a diagnostic campaign entailing a combination of non-invasive and micro-invasive techniques (Raman, SERS, Py-GC-MS, HPLC-DAD, HPLC-MS) applied to study the recent "UBUNTU" mural created in July 2018 on the external wall of Dalla Chiesa middle school (Reggio Emilia, Italy) by the artists Ivan "Hang" Pontevia and Daniele Castagnetti in collaboration with city Municipality and Officina Educativa. UBUNTU painting is included among the selected artworks for the Erasmus+ project "CAPUS-Conservation of Art in Public Spaces" (Erasmus+ Knowledge Alliances, 2018-2021, Project N° 588082-EPP-A-2017-1-IT-EPPKA2-KA)" <http://repository.capusproject.eu/artwork/ubuntu#>. Even if the mural is quite recent, direct sunlight and intense traffic pollution are high-risk factors, and fading is already observed, mainly affecting the fluorescent spray.

The application of the multi-analytical approach allowed us to identify the binders as alkyd and acryl resins by Py-GC/MS. Raman and liquid chromatography detected all the fluorescent dyes and pigments present in the spray paints. For some colors, such as those based on rhodamine, the molecular profiles show the presence of photo-oxidation products of the original dyes, highlighting that an early degradation is already occurring, consistently with the observed fading.

The results of the analytical investigation will help curators in monitoring the state of conservation of the mural, ensuring a sustainable preservation of this peculiar work-of-art.

## Aerospace technology as part of our heritage: characterization of aircraft materials and study of their degradation processes by analytical pyrolysis

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Historical airplanes have entered museum collections to show the development of aviation and are part of our technical heritage. The period between the beginning of the aviation history and World War II is characterized by giant progressions in the chemical industry. During these years a wide increase in the production of new materials and coatings was observed, that replaced the natural materials used in the aircraft industries due to their improved properties. The study deals with the characterization of the materials used in the paint layers of three airplanes, an Ansaldo A.1 (Comune di Casale Monferrato, complete plane, 1918) and two Messerschmitt Bf 109 (Private owner, original cabin roof, 1937; Deutsches Museum, complete plane, 1938-repainted until 1975), including overpainting layers from later use and museum presentations. The study is aimed at understanding the materials chosen in aviation technology, also in relation to the developing chemical industry, and to understand if and how the paint composition can be related to the conservation condition of the paint layers and of the aircrafts. In this study we applied for the first time analytical pyrolysis coupled with gas chromatography and mass spectrometry for the characterization of the painted layers. The analyses were carried out directly on the paint fragments, in some cases also with the use of hexamethyldisilazane as derivatizing agent. A selection of samples was also analyzed by infrared spectroscopy. The paint samples were characterized by a very complex stratigraphy, with extremely thin paint layers well-adhering to each other, and thus impossible to be mechanically separated. A careful sampling campaign allowed us to obtain information on the sample build up and the history of the planes. For the plane from the First World War the analyses allowed us to characterize the constituting materials and to highlight the ongoing degradation processes. The study carried out on samples from the two Messerschmitt planes allowed us to characterize the original materials used to produce the planes and to study those used in the different restoration campaigns which have been carried out until 1974. The use of analytical pyrolysis was crucial to characterize a wide range of natural and synthetic materials, allowing to resolve complex mixtures. Data show an interesting evolution of the painting materials used in the two different historical periods, which include natural materials, as a drying oil and Pinaceae resin, but also several synthetic materials, including cellulose acetate, alkyd resin, nitrocellulose, phenol formaldehyde resin as original materials, and more than other six different synthetic polymers used during the restoration campaigns.

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## SuPerStAr - Sustainable Preservation Strategies for Street Art: a new Italian project on the safeguard and preservation of street art

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Street art has been recognized part of our cultural heritage only in the latest years. The ephemeral character, free access, and exposure to the environment and anthropic actions, make public paintings vulnerable to neglect, removal, vandalism, and degradation. Beyond that, the strategies aimed at their preservation and fruition are rather unclear or lacking.

The project PRIN-2020 SUPERSTAR *Sustainable Preservation Strategies for Street Art (2022-2025)* sets as a goal the definition of innovative guidelines for the preservation strategy of street art, aimed at safeguarding its powerful social and cultural message in the urban context. The combination of non-invasive and micro-invasive techniques will shed light on the chemical-physical properties and vulnerability aspects of modern paint materials that constitute street artworks. The studies performed in the laboratory on reference materials will be supported by research performed on case studies, focused on the materials used by the artists, the environmental risks and anthropic stress. Relevant case studies have been selected, located in different environmental urban contexts in Milan, Torino and Pisa, in collaboration with municipalities and with urban art curators.

Thanks to the effective collaboration among a wide team of researchers with complementary expertise, involved in the various participating units, and to the interaction with conservation institutions and entities engaged in safeguarding public urban art, the project will contribute to define future preservation strategies. The following outputs are expected: optimized innovative cleaning procedures for the restoration of outdoor murals and for the removal of vandalistic graffiti; selected protective coating materials with particular attention to durability aspects; and an integrated protocol for long-term sustainable monitoring and conservation.

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<https://prin2020superstar.dcci.unipi.it/>

## The 2-step procedure with Di-ammonium phosphate to consolidate carbonate stones used in cultural heritage

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The degradation of stony materials is an open issue in the field of restoration; consolidation treatments are often required due to the action of pollution, acid rain and weathering especially for artifacts that are exposed outside. There is not a single product or a single methodology to counteract this decay since different materials and degradations develop each time. In this research Di-ammonium phosphate (DAP) has been studied since this product is environmentally friendly and was specifically designed for carbonatic matrixes. DAP reacts with carbonate substrates to produce different phases of calcium phosphates, that are insoluble. However, the depth of penetration is still questioned and deserves a detailed study to identify the best application protocol of DAP. The 2-step procedure was applied on the samples in very low percentages compared to previous studies. Stone of Lecce and Stone of Finale have been used to test the treatment due to their different fabrics in specimens of 5x5x2 cm size. In the first step a poultice with 0,5% of DAP in water solution was applied and left on the samples for 24 hours. Then, the first poultice was removed and the second one was applied in the same way, with 4% of DAP in water. After 24 hours, the second poultice was also removed. Later, different analyses were carried out to test the consolidating effect and to detect calcium phosphates. Thanks to Planar Abrasion Meter (PAM) the entire specimens' surfaces were tested; the treated stones had doubled their abrasion resistance up to a depth of 1 mm. Mercury Intrusion Porosity (MIP) showed an increase of small pore volume in treated stone together with decrease of coarser porosity. The capillary absorption test showed that treated stones absorbed less water than untreated ones, while the evaporation test demonstrated that all absorbed water is completely released. Elemental maps performed by Scanning Electron Microscope coupled with Energy Dispersive Spectrometry, a thin layer of phosphorus was detected on the surfaces of treated samples and in some of the shallowest cavities. Ten consecutive samples were taken on the surface of the treated specimens and the exported material was analyzed in transmission in a diamond cell by FTIR (Fourier Transformation Infrared). The tenth specimen no longer showed the presence of phosphorus. As a conclusion even detectability of phosphates is controlled by the heterogeneity of stones and very low amount of product utilized, the effectiveness of DAP is evidenced by physical-mechanical tests. The DAP penetration seems to attain 1 mm in depth nevertheless there are evident limits of detectability cannot exclude greater penetration. Ongoing experiments address a poultice-free methodology to avoid a waste of material, especially for large construction sites.

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# Testing Sustainable Restauration of Sandstone

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In this work the effects of consolidation products usually used in stone restoration were studied, with the aim to compare their applicability to sandstone, evaluating the best performance also in terms of eco-sustainability. Several commonly used consolidating products were selected and tested in this study [1]: limewater, diammonium phosphate, ammonium oxalate, nanosilica, ethyl silicate. They were chosen because they do not contain volatile organic compounds (except ethyl silicate, commonly used) and they are not harmful or toxic, therefore safe for the operators' health and for the environment. Some products are especially effective on carbonatic-rich rocks, therefore, in this work their applicability was tested on Camerino sandstones, a lithotype rich in carbonatic component (43-53 wt%  $\text{CaCO}_3$ ). Camerino sandstone (Camerino formation, FCI), is widely used in the construction of historic buildings in the Marche region and presents many conservation problems connected to heterogeneity of the mineralogical composition and high porosity, strongly affecting the aesthetical characteristics but also, with time, the stability of the structures. Samples were artificially weathered by heating them at 400°C for an hour, then followed by the application of the cited products on the surface. The behaviour of the samples after treatment was evaluated testing water absorption by capillarity and total water immersion for 24 h, Scanning Electron Microscopy (SEM), optical microscopy, colorimetry and X-ray Diffraction (XRD).

The results after the treatment with different products showed that:

- artificial weathering affected strongly the porosity and permeability of the samples;
- unwanted variations of surficial color were detected and it turned out to be unacceptable for samples treated with ammonium oxalate poultice for 72 h or with pure nanosilica;
- all the consolidation treatments decreased the rock surface permeability, especially ethyl silicate, which showed an unacceptable hydrophobicity of the treated surface;
- diammonium phosphate provided best results since the color variations are acceptable and it hasn't caused hydrophobicity. Moreover, it demonstrated a good compatibility with the substrate.

These preliminary results suggest that ethyl silicate could be successfully substituted by diammonium phosphate in restoration of these sandstones, with advantages for the sustainability of the restoration process and safeguard of operators' health.

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## Multi-Analytical Investigation of the Oil Painting “Il Venditore di Cerini” by Antonio Mancini and Definition of the Best Green Cleaning Treatment

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This work shows the results obtained from the multi-analytical investigation of the oil painting “Il Venditore di Cerini”, made in 1878 by Antonio Mancini. The aim of the research was twofold: the characterization of the constituent materials of the artwork and the application of newly developed green solvents for the removal of film-forming substances. SEM/EDS analysis showed the presence of pigments already detected in other paintings by Antonio Mancini [1]. Multispectral imaging, DinoLite microscopy, and FT-IR ATR spectroscopy revealed significant data regarding the invention of a technique implemented by Mancini, also proving the presence of an aged layer of non-original shellac on the surface. The yellow/brownish tone of the varnish required selective removal of the aged coating. Indeed, the multi-analytical approach implemented enabled the acquisition of significant elements to guide the cleaning treatment performed on the artwork. The selection and implementation of potentially well-performing cleaning systems were selected among sustainable alternatives available in the market, in compliance with the guiding criteria of Green Chemistry. The tested cleaning systems are: Green Varnish Rescue (acetals' mixture) in both the liquid form and thickened, thickened Polar Varnish Rescue (anionic surfactant and acetals), and Nanorestore Cleaning (nanostructured water-based fluid with anionic surfactant and mixture of 1-pentanol, ethyl acetate, and propylene carbonate) [2,3,4]. The selection was made according to the Fd parameter of the cleaning systems – which defines the energy from dispersion forces between molecules – in relation to what is defined in the literature as the suitable Fd value for the removal of shellac. The best-performing green cleaning system proved to be the Polar Varnish Rescue GEL — a gelled acetals mixture developed by YOCOCU APS – for its effectiveness in selectively remove the aged shellac while preserving the integrity of the original stratigraphy. The reason for its effectiveness is twofold: the Fd value of the product, which is similar to the Fd known to be used for the removal of shellac, and its tendency to establish hydrogen bonds, which plays a significant role in the solvation of shellac. The Polar Varnish Rescue GEL can be easily applied, showing overall valuable qualities for cleaning treatments on works of art. Most importantly, it represents an alternative to more toxic solvents that are still widely used in the conservation field, since its components are known to be green chemicals capable of safeguarding both human health and the environment. Indeed, the present study aimed at promoting the use of new green materials for conservation purposes, fostering sustainable development.

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## Deep Eutectic Solvents as Green Solvents for the Conservation of Cultural Heritage

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Deep Eutectic Solvents (DESs) are a relatively new class of unconventional green solvents. DESs can be obtained by mixing two or more solid components [a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA)], which form a liquid at room temperature, because they have a lower melting point compared to the starting materials. DESs are solvents that are easy to prepare starting from economical, non-toxic and biodegradable components. The use of DESs can be very advantageous in many applications, because they have peculiar chemical-physical characteristics, such as they are stable in the presence of water, non-flammable, with a high boiling point and eco-friendly [1, 2].

In the cultural heritage sector, the use of new biocides with a lower impact on the environment and on the operator represents an important result. On the other hand products with biocidal action are few and they must possess appropriate requirements, such as being effective at low concentrations, not interfering with the material, not dangerous for the operator and the environment.

DESs are non-toxic, environmentally friendly, non-volatile, safe and inexpensive compounds and although are being used in various fields, they still represent a novel frontier in the sector of cultural heritage, as they have not been used so far for cleaning and conservation treatments.

In this communication, we report for the first time DESs as potential biocidal agents in the field of cultural heritage. Choline Chloride: Urea (1: 2), Choline Chloride: Glycerol (1: 2), Choline Chloride: Ethylene glycol (1: 2), Choline Chloride: Malonic Acid (1: 1), Choline Chloride: Oxalic Acid (1: 1) and Choline Chloride: Zinc Chloride (1: 2), have been prepared and tested as biocides in laboratory and in situ at the archaeological park of Ostia Antica (Rome) and during the conservation treatment of the frescos "Cristo Pantocratore" in Motta San Giovanni (Reggio Calabria).

The results obtained from this study are very promising for a green application of DESs in the field of cultural heritage.

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## **Old and new uses of traditional materials between sustainability and durability: from laboratory samples to historical surfaces**

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In the plasters of the pre-19th century tradition it is possible to recognize forms of surface protection based on the use of inorganic materials, such as lime (in the form of cream or milk), or organic substances, of a vegetable type (carnauba wax, oil of flax, soap) or animal (beeswax). From the analyzes carried out on samples of historical plasters and in relation to the information present in the traditional literature, it can be said that these protections from external invasion water have always been considered an essential element to ensure a better duration of the entire coating, which is linked in particular to the reduction of surface porosity. The protective application technique, also recognizable by the degradation phenomena and by the microstratigraphic sections of samples, was based on the fresh application of the last finishing layer in order to exploit the cohesion with the liquid part of the lime present on the surface when the plaster begins carbonation.

In consideration of the close relationship between durability and type of surface treatment, a research path has therefore been developed which intends to highlight the properties acquired by the coating. The study is divided into both laboratory tests and verification of applications carried out on historic walls during some construction sites.

A laboratory experiment, which aimed to compare the effects of traditional materials and commercial products applied on two series of samples (with tests performed before and after artificial aging in a climatic chamber: drop test, sponge test, stereomicroscope verification, colorimetric measurements), highlighted how traditional protective products have a greater ability to maintain their water-repellence and reduced capillary absorption properties over time. In another test relating to uses of soaps of different origins (Aleppo soap, Marseille soap, coconut soap), and consistency (hard soaps, soft soaps), he highlighted that there are different fields of application in relation to the application and under the conditions of the substrate surface.

In the context of the use of soap, as an economical and effective solution to obtain high waterrepellent performance, the tests carried out in six construction sites of historic buildings made it possible to face different situations and methods in relation to the materials present, the type of surface porosity, the environmental conditions and the design choices of the final effect. The situations present ranged from exposed brick masonry, to the thin layer of plaster, to the thicker coating, both in aggregate of sand and in stone material; furthermore, there were situations of old plaster and new plaster, with different ambient temperatures in relation to the season of application. In situations of already carbonated surfaces, historical or recent, the combination of applying lime water and soapy water in succession was very effective. The verification of the gradual reduction of porosity made it possible each time to calibrate the dosages and the application steps.

The combination of laboratory experiences and verifications (through controlled water spray test) on built surfaces constitutes a test field for a better understanding of the parameters of the solutions to be adopted to increase the performance of the surfaces, both in terms of water repellency and compatibility and sustainability for the characteristics of durability that derive from it.

## "Arte è(‘) Scienza" exhibition: a successful integration between cultural heritage and scientific techniques

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Since 2014 “Arte e(‘) Scienza” days are one of the most expected events the Italian Association of Archaeometry (AIAR). The event, which involves the entire network of AiAr researchers, provides for the simultaneous realization of conferences and laboratory activities in museums, archaeological sites, restoration laboratories, institutions and research institutes, throughout Italy.

The main goal is to offer the public, students or visitors, the opportunity to see scientists at work with their handheld instrumentation in close contact with curators or archaeologists to understand, preserve and know works of art. At the same time “Arte e(‘) Scienza” represents a unique opportunity to discuss on the vital relationship between cultural heritage and scientific methodologies. In this paper we present a panoramic of the activities carried out during these eight years and the new perspectives for the future.



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## “An Ocean of Science” Project: A journey to the seabed

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The protection of underwater cultural heritage, as an integral part of the cultural heritage of Humanity, is exercised according to the same general principles envisaged for the underground archaeological heritage. These principles are reaffirmed and extended in a fundamental international legal instrument, the Convention on the Protection of the Underwater Cultural Heritage, adopted in Paris on 2 November, 2001 by the General Conference of UNESCO Member States. Archaeological materials coming from underwater environments are of great relevance in order to study technologies, origin and progressive evolution of ancient civilizations and for a better understanding of historic events. Underwater sites are highly dynamic environments and undergo the influence of the marine system; they are also of considerable interest for their natural, geomorphic, physical, biochemical characteristics, with particular regard to marine flora and fauna as well as for their scientific, ecological, cultural, educational and economic importance. Being the umpteenth testimony of the vast cultural that enriches our territory, the submerged heritage bears witness to our cultural identity. Undermining the beauty of the hidden treasures protected by our waters, in addition to the well-known illegal trafficking activities, contributes and aggravates the environmental damage and, consequently, the conservation of the submerged cultural heritage.

The project "An Ocean of Science" (CUP H23D21002060001), promotes effective interventions and innovative educational paths to support the growth of knowledge and skills in schools, helping the relevant institutions to ensure more inclusive cohesion policies with a high cultural and scientific impact. The project is aimed at high school students with the aim of raising awareness of the enhancement and enjoyment of the finds and evidence of the past culture that lie on the seabed and also to study the pollution of the seas, as one of the major threats to the marine ecosystem. The results will be aimed at increasing students' knowledge and awareness of their own cultural identity and at promoting the enhancement of the national submerged cultural heritage, also acquiring awareness of its environmental importance.

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## Integrating p-XRF data from different equipment: A challenge for the investigation of large collections of Iron Age glass

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The INGOT-EL project (INvestigation of Glass Origin and Technology in Etruscan Lands) aims to bring new evidence on glass production and circulation by analysing a large set of glass beads uncovered in many Iron Age burials in central Italy.

One of the first steps of the investigation accounts for a non-invasive in-situ approach with portable-X-Ray Fluorescence (p-XRF) equipment operating in several archaeological museums involved in the research.

The aim of this preliminary step is to obtain a first overview on the elemental composition of the beads and guide the selection of representative samples that will be included in the following analytical steps with micro-invasive techniques, overcoming some limitations of the p-XRF approach such as the lack of data on light elements. The p-XRF survey is therefore crucial to enhance the sustainability of archaeometric investigations in large archaeological collections, as the preliminary non-invasive step reduces the number of the samples that will be subjected to micro-invasive approaches, without reducing the quality of the overall information. It is in fact mandatory that analytical procedures guarantee the general accuracy of the compositional data, so that they can be treated (and re-treated in the future) in the broader perspective of the archaeometric investigation of glass.

There are many factors affecting the results of p-XRF analyses, therefore comparing data obtained with different spectrometers is far from obvious [1]. Configuration of the equipment (in terms of energy of the primary radiation and the angles of take-off and incidence), density of the analysed material and energy of the element's characteristic fluorescent radiation may influence the intensity of the signals and, therefore, lead to case-study dependant data, without a possibility for comparison with other datasets.

To manage these issues, we performed a dedicated study using four p-XRF spectrometers with different sources of incident radiation, geometry of measurement and analytical spot sizes. Data were systematically collected from Corning glass reference materials that represent different compositional groups of historic glasses and several archaeological glass pieces with known composition. All spectra were treated with the free XRF toolkit PyMca [2]. The procedures for data treatment are presented and discussed in terms of statistical bias and precision of the data. Three of the considered spectrometers – which allow the analyses of small areas thanks to their focussed incident ray - have been then employed in the museums involved in the INGOT-EL project to analyse archaeological glass beads.

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## Transdisciplinarity goes digital: the BE-ARCHAEO endeavour from the Semantic Web perspective

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The involvement of many disciplines has long been a distinctive feature of archaeological research and this trend continues to grow. Nowadays, a large amount of digital data is produced by the many processes performed both at the archaeological site and in the scientific laboratories. These data can be vehicles of new knowledge if they are stored in the digital world according to proper representational models, ensuring the coherence of information and knowledge as contributed by each intervening discipline and – according to the Semantic Web perspective – allow different audiences to query the data and evaluate (or re-evaluate) conclusions. Many representational models have been developed to account for the encoding of the archaeological processes in a data base, with recent semantic models relying on the CIDOC Conceptual Reference Models (CRMs) [1]. Nevertheless, the many archaeometric processes have yet to be addressed.

The BE-ARCHAEO project represents the perfect playground to develop the relevant computational ontologies for transdisciplinary endeavours related to archaeological records from the Semantic Web perspective: the BE-ARCHAEO team carries out archaeological excavations at the Tobiotsuka Kofun (Soja city, Okayama Prefecture, Japan) and performs archaeometric analyses of archaeological materials found at several burial mounds in the ancient Kibi and Izumo areas (present Okayama and Shimane prefectures).

The aim is the development of a transdisciplinary vision in preparing and managing archaeological records, including their digital interlinked representations. BE-ARCHAEO researchers are leading the development of a modular ontology based on the CRMsci model (a member of CIDOC-CRM family) by covering the large variety of processes related to archaeometry: from pedological and microbiological analyses of soil to the micro-morphological and compositional characterization of archaeological pottery, glass, and metal finds. Representation and management of the digital assets produced within BE-ARCHAEO follows the criteria of Digital Data Curation [2], to enhance their subsequent exploitation.

The BE-ARCHAEO ontology comprises three modules: archaeological knowledge, archaeometric knowledge and the catalogue record knowledge, with connections to standard ontologies and the inclusion of non-ontological resources [3]. The overall effort, besides effectively supporting reflectivity and fertilising transdisciplinarity, is implementing the ontology-based approach to encode transdisciplinary knowledge and is offering the archaeometric community new digital strategies to face the task of transforming the present “archipelago” of diverse, specialised, but rather isolated information systems into interlinked transdisciplinary databases through archaeology and archaeometry. Some significant examples for the archaeometric module of the database - presently displayed as an installation of the Content Management System Omeka-S [4] - are presented.

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# Underground Triggiano: photogrammetry 3D reconstruction of the historical centre hypogea

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This paper introduces a specific study based on the presence of hypogeum environments below the street pavement of the historical centre of Triggiano, (Bari, southern Italy). The research started from previous studies concerning the St. Maria Veterana archaeological site (XI-XVI) (Fioretti et al.2020), an example of a Medieval church dug in the surrounding local calcarenite rock. During the 1980s archaeological excavations, there was found a maze of underground rooms near the church, also in different places within the walls of the Medieval town (Campobasso 2021). These environments consisted of hypogea used in the past as oil mills, tanks, subterranean storages, paths and walkways connecting different points of the ancient village. Previous local history studies highlighted that the frequentation of these underground rooms, both for work and domestic purposes, was very intense from the Middle Ages up to 1800.

In order to explore the “Underground Triggiano”, the digital photogrammetry was used to obtain both the 3D model of each environment and the 3D reconstructions of the building above by linking the hypogeum with the upper level. Two different photogrammetric methods were adopted: the first used an optimized digital photogrammetric technology (Nikon D800-16mm FX, crop sensor), the second, based on the use of a spherical camera (Ricoh Theta S 360°) is faster than the other, however it is characterized by less detailed pictures. A 360° camera was adopted in inaccessible places with the support of anchorage systems and with the aim to produce a 3D reconstruction of the interconnected environments with each other.

The research illustrated, on the one hand, the information technology applications on cultural heritage and, on the other hand, demonstrated how 3D reconstructions can favour the enjoyment of inaccessible places. Finally, the most innovative virtual reality technologies make easier the dissemination of the digital products with immersive experiences and help to remember the past and preserve the memory.

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## **Recostones: an app for provenance studies of natural stone materials**

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This work presents a new software tool, named “*RecoStones*”, developed within the SILPI project (acronym of “Sistema per l’Identificazione di Lapidei Per Immagini”) financed by POR Calabria FESR-FSE 2014-2020 (De Luca et al., 2021). It is an app for smartphone able to classify stone materials and to determine their provenance through image recognition techniques. This app was created using stone samples coming from twenty-five Calabrian quarries, representative of the five provinces (Catanzaro, Cosenza, Crotona, Reggio Calabria, and Vibo Valentia) and many of them used in historical times for the construction of artefacts of artistic and archaeological interest. For each quarry 10 samples were collected and for each sample images through smartphone and flatbed scanner were acquired, obtaining a set of 750 images (250 acquired by smartphone and 500 by scanner). To evaluate the best methodology to use, different images process techniques were performed. The method that provided the most successfully results is based on the combination of the Convolutional Neural Network Inception-v3 (Szegedy et al. 2016) and the feature descriptor called “Median Robust Extended Local Binary Pattern” (MRELBP) (Liu et al. 2016). Using this methodology is, therefore, possible to recognize a typology of rock without sampling and analyses, but only using a simple image of the material, acquired under standard conditions. The testing phase of the app, carried out *in situ* on different local known materials belonging to monuments and buildings of the Calabrian Cultural Heritage, confirmed that *RecoStones* is a useful, smart and cheap tool which can help geologists, restorers, architects, art historians, engineers and all the staff involved in restoration and conservation projects, to determine the typology of a stone material and its provenance. Currently this app is able to identify only Calabrian stone materials, but in the future, it can be upgraded by sampling other outcrops and/or acquiring further images related to other quarries located in different national or international contexts, becoming a new tool of global interest. In addition, the samples collected from the Calabrian quarries were also studied by optical microscopy, X-ray Powder Diffraction and X-ray Fluorescence, in order to obtain their chemical and minero-petrographic characterization and to create the first database on the Calabrian rocks.

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# **The CAST Project. Exploring the knowledge over the dark. Advanced multidisciplinary archaeology investigations from Pertosa caves, Italy**

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The CAST project (Caves through Archaeology, Science and Technology) is configured as a methodological intervention program, which makes use of innovative technologies, enabling the acquisition of significant information on timeframes and on how the caves of Pertosa-Auletta (southern Italy) can be used. If on the one hand the objectives are the continuation of historical and archaeological research, on the other we want to develop an integrated museum system between nature and high impact popularization models using virtual survey technologies. Using innovative methodologies, the aim is to make the information acquired during the interventions available through a platform for continuous monitoring of the progress works. The direction marked by this pandemic phase has made the use of these methodologies even more urgent, favoring the spread of a New Digital Humanism that can determine a social impact capable of guiding future approaches to the management and scientific treatment of data. In this regard, through the web platform dedicated to the Virtual Reality Experience it is possible to carry out a virtual tour of the 3D reconstruction of the pile-dwelling settlement, identified at the entrance to the Caves, developed on the basis of archaeological data and the high-resolution three-dimensional survey carried out through the integration between laser scanner and photogrammetry. During the virtual tour it is also possible to interact with the 3D models of the finds that can be explored obtaining information of a different nature. This platform contains virtual reproductions connected to the archaeological and natural history of the Caves, with diversified levels of access to information, which constitute a model of virtual experience with a strong scientific and social impact. The purpose of the platform, constantly implemented and updated, is also to allow you to check the progress of the research in a single environment that is easy to access.

Keywords: Caves Archaeology, New technologies, 3D survey, Virtual reality, Digital Museum



## 3D multispectral imaging for Cultural Heritage preservation

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Digitalization techniques, such as photogrammetry, are attracting the interest of experts in the field of cultural heritage, as they enable the creation of three-dimensional virtual replicas of historical artefacts by using 2D digital images. Photogrammetry allows acquiring data regarding the overall appearance of an item, its geometry, and texture.

Among several image-based techniques exploited for the conservation of artefacts, multispectral imaging (MSI) finds great application in the study of the materials employed for the creation of historical items. Indeed, MSI is useful for the non-invasive investigation of pigments, binders, varnishes, and other materials since it may aid their characterization by taking advantage of their different response when exposed to specific wavelengths of the electromagnetic spectrum [1].

Despite their great usefulness and diffusion, MSI techniques are traditionally applied only at a 2D level. In addition, photogrammetry and multispectral imaging are often used as separate tools. The possibility of integrating data coming from MSI and photogrammetry can notably expand the information carried by a 3D model. Indeed, the combination of geometric and radiometric information in a 3D model could represent a powerful tool for experts in the field. Taking into consideration that 3D models can represent virtual documentation useful for study, preservation, and research aims, preventing the acquisition of incorrect dimensional and metrical data is of paramount importance. In spite of this, nowadays there is no unique way to assess the dimensional accuracy of 3D models in the Cultural Heritage field [2].

Therefore, this paper presents a metrological approach for the integration of geometrical and spatial information coming from photogrammetry and radiometric data of multispectral imaging in a unique 3D model. In particular, a novel research methodology and experimental setup, that enable the acquisition of multispectral 3D models, combining the outcomes of photogrammetry and multispectral imaging in a single coordinate system, is presented. This approach has been developed together with a specific novel reference object, for application in the cultural heritage field. This has the main aim of being employed as a dimensional reference for the assessment of the dimensional accuracy of 3D models [3]. In addition, this item integrates several pictorial preparations, with materials historically employed on cultural heritage items, thus it can be used as a multispectral reference. The above-mentioned approach has been exploited for the study of the state of preservation of a wooden sculpture belonging to the collection of the Museo Egizio di Torino. In particular, the creation of multispectral 3D models was performed to investigate the materials present on the surface, both the original ones and the ones applied in previous treatments, to provide information that could support the design of suitable conservation treatments.

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## The Statue of Venus Lilybetana: digitization and virtual restoration

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Here, the result of a 3D survey campaign carried out on Venus Lilybetana saved at Parco Archeologico di Lilibeo-Marsala (TP), is reported.

The marble statue of Venus Lilybaetana is the most important artifacts included in the exhibit, found in 2005 during the archaeological excavation in the area belonging to the Church of San Giovanni Battista in Marsala. The statue, dating back to the second half of 2<sup>th</sup> century AD is headless and lacking of other parts: half of the right arm, which covered the breast, more than half of the left arm, which held the himation, half of the right leg and part of the left leg.

The statue was subject of a 3D survey aimed at realizing a digital replica. It was performed integrating two techniques: terrestrial laser scanning (using a Faro Focus 3Ds 120) and digital photogrammetry (using a Canon Eos 7D Reflex Camera). The first is a point-based technique based on an active sensor that measures the distance between the instrument and the target, providing very accurate metric data; the second is an image-based technique based on the triangulation principle, through which a set of photos taken from different viewpoints are turned into 3D models thank to specific algorithms (structure from motion).

The outputs derived from the two processes were finally combined in order to obtain a complete 3D reconstruction, where the geometric accuracy is given by the laser scanner points while the graphic rendering by the photographic texture. Working on this basis, we are proceeding with the creation of a first virtual restoration, which also includes the reconstruction of the missing parts. The reference model for this further process is the well-known iconography of the Callipygian Venus.

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# Interdisciplinary approach to the dynamics of data collection, cataloging and homologation of photogrammetric models

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With the increasing diffusion of photogrammetry and, more generally, of the three-dimensional survey of finds and materials, new issues arise for the entire production chain and for the professionals involved. Especially for the models obtained with photogrammetry, we face problems in cataloging, homologation and indexing of the 3D models as well as of the original photographic material (i.e., the pre-existing matrix to any processing activity). In recent years, there have been many proposals for a methodological approach to recording, from the pioneering Inception Protocol.

In this paper, we address an operational loop in the recording of the materials, which occurs between the cultural heritage professionals (in particular, archaeologist and archaeometrists), who work on the interpretation of the finds, and the technicians, who carry out the three-dimensional surveys. In fact, the record description requires an interpretation process that may have not produced any usable result at the time the three-dimensional materials are produced. Actually, the 3D materials themselves can contribute to the interpretation process. The decisions taken about the model underlying the database schema must necessarily go beyond the traditional archaeological concepts of finds and stratigraphic units.

Here, we propose a methodological approach for the identification and the recording of the items that are produced in the several steps of the production of 3D models. In particular, for each step, we assign an evaluable feature and establish the minimal requirements to be achieved, for the survey to be valid. Also, the formal annotation of the metadata has to be coherent with the traditional recording methods.

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# Reflectance transformation imaging of archaeological finds

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Reflectance Transformation Imaging (RTI) is a computational photography technique that can reveal details hidden to the naked eye on objects, such as shape and traces of colour and details of the surface. A camera and a mobile light source are all the equipment needed to operate. Thanks to the photographic footage taken, an algorithm can determine the various responses to light issued by the object, recognizing the presence of rough or smooth areas along the surface. The process does not record the information on the colour of the object only, like a normal image file, but also recognizes the normal of the surface (CHI 2021, p. 13). Subsequently, it is possible to interact with the elaborated model and vary the lighting conditions, thus obtaining a re-illuminable image (Scopigno, Montani 2015, p. 21), and evaluate the variation in shading. This procedure allows the observer to see and perceive the irregularities of the surface. It is also possible to accentuate these aspects by automatically removing the colour information and increasing the reflectivity of the surface (Fiorini 2018, p. 242). The use of open-source software makes this survey technique widely accessible and therefore also applicable in the context of excavation and survey operations. Thanks to these characteristics, RTI is a versatile tool for archaeological research, suitable for the study of the finds and to verify their conservation conditions. In recent years RTI use has grown in the archaeological field, with applications both on artifacts and finds and on the remains of structures. This contribution aims to present the results obtained from the analysis of some materials from the late antique necropolis of Vaste (LE). The objective of the research was to develop new forms of documentation processing, useful for evaluating the conservation conditions of the finds and, moreover, to highlight any traces of processing not perceptible to the naked eye. The integration of RTI with the most common photogrammetry techniques makes it possible to have an informatics tool to be used for dissemination purposes and for any 3D printing of the finds, to ensure greater accessibility and use for the public.

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# Methods of acquisition and processing of 3D models as a support for the conservation, enhancement and use of Cultural Heritage

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This work has contributed to the development of a wider project, entitled “The Virtual Museum of Piazza Duomo in Parma”, which is a tour route that can be used entirely online by PCs, smartphones, tablets and virtual reality viewers. The users, in particular, can discover the entire monumental complex of the square, the Cathedral, the Baptistery and the Diocesan Museum in Parma, watching seventy-three 3D models and fourteen 360-degree photographs.

This study is divided into several phases. First of all, the reliefs of fourteen artworks conserved in the Diocesan Museum were realized through the use of structured light lasers and automatic photogrammetry. More specifically, the first acquisition method was used for the Roman period lamp, while for the other works of art the photogrammetric technique was used.

In the second phase the previously acquired data were processed, using different types of software, such as Zephyr and Agisoft Photoscan, to obtain 3D models (figure 1).

The data thus obtained were initially loaded onto the Sketchfab platform, to allow an improvement of the 3D models and to set the visualization through the virtual reality viewers, which allow an immersive and very realistic use.

Lastly, the pages were created, one for each art work, on the 3D Virtual Museum website, in order to implement new museum collections online and to expand the number of users who, for example, cannot visit the monumental complex of Piazza Duomo to see online the works preserved there.



Figure 1: 3D model of the “Lastra dell’ambone antelamico della Cattedrale” art work

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## A 3D multimodal and multiscale approach for the study of Upper Palaeolithic ground stone tools

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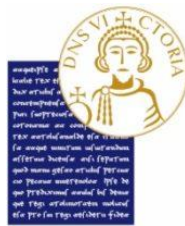
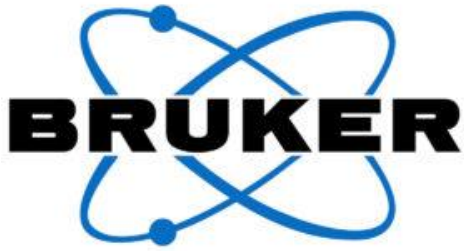
The term “ground stone” refers to non-flaked industry, and includes a multitude of different instruments that have not undergone an intensive stone shaping. This natural and non-modified appearance of the stones lends to them the high potentiality of being utilised in the most varied uses as the processing of different materials such as bone, mineral and plant [1]. Among them we are here referring to the elaboration of vegetal resources that leave evanescent traces and perishable residues on the stone surfaces, which call for a tailored approach. Of great support for the comprehension of these stone tools’ function(s) is the use of analytical techniques that allow a 3dimensional characterisation of the item and of wear affecting the surfaces as well as of morphotextural properties within the sample volume [2]. Our approach is based on multimodal and multidimensional morphological approach, by applying imaging techniques ranging from macro to sub-micro scales aiming at elaborate a digital model of the tools and at performing a wide range of analysis. In particular, the procedure exploits the advantage of (i) photogrammetric acquisition of the ground stones and 3D model elaborations to inspect their geometry and macroscale investigation of their surface, (ii) profilometry measurements of selected areas of the stone surface to characterise the microtopography and highlight the presence of use-wear traces, (iii) high-resolution synchrotron X-ray tomographic imaging to investigate the volumetric microstructure by a non-destructive approach and to verify the presence of crevices that served as putative traps for biogenic residues deriving from the plants grinding and pounding.

We present the pipeline developed for the analysis of the artefacts from several Marine Isotopic Stage 3 (MIS 3, 60-25 ka) sites, and in particular, tailored for the ground stones retrieved in the Aurignacian cultural level III of the Brînzei I cave (north-west Moldova) [3]. The proposed analysis is tested on experimental replicas used to treat plants starch-rich organs compatible with the biome of the Pontic steppe during the MIS 3 [4].

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