XII CONVEGNO INTERNAZIONALE

Diagnosis for the Conservation and Valorization of Cultural Heritage

Napoli, 9 - 10 dicembre 2021

ATTI DEL CONVEGNO

a cura di

Luigi Campanella, Ciro Piccioli,
Anna Rendina, Valeria Romanelli

A.I.E.S. BENICULTURALI
Associazione Italiana Esperti Scientifici

POLO MUSEALE SAPIENZA

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Diagnosis for the Conservation and Valorization of Cultural Heritage

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> Summary / Sommario

pag. 13 **Prefazione**
Luigi Campanella, Ciro Piccioli, Anna Rendina, Valeria Romanelli

pag. 15 **La Valorizzazione ed il Tempo**
Ciro Piccioli

pag. 19 **> DIAGNOSIS / DIAGNOSI**

pag. 20 **The Visible Induced Luminescence photography (VIL) useful for detecting hidden particles of the Egyptian blue pigment on archaeological finds.** / *A. Scognamiglio, I. Simeone, E. Fusco*

pag. 31 **Structural restoration in archaeological context: the case studies of "Nuraghe Iloi" and "Nuraghe Talasai" in Sedilo (OR), from relief to the project.** / *M. Dessì, M. Ciudino*

pag. 40 **Impiego del laser scanner su un'opera d'arte cartacea.** / *E. Mazzoni*

pag. 50 **Il ruolo dei modelli digitali 3D nell'analisi e nella rappresentazione di un'architettura rupestre. Il caso studio della chiesa rupestre di S. Lucia alle Malve a Matera.** / *G. Fortunato, A. A. Zappani*

pag. 59 **The 3D survey for the documentation, reading and analysis of the byzantine architecture of San Marco in Corigliano-Rossano.** /
G. Fortunato, A. A. Zappani

pag. 68 **A multidisciplinary diagnostic approach for the restoration of the inside surfaces decorative plaster of the vault of the late seventeenth-century Sant'Angelo Church in Monopoli (Bari, Italy)** / *P. Acquafredda, M. De Tullio, G. Eramo, F. Laviola, N. Mongelli, A. Quartulli, L. Scrano*

pag. 77 **Multianalytical approach for the characterization of ancient mortars and plasters from the archaeological site of Campi Flegrei (Italy)** /
C. Gallo, S. Carannante, M. P. Cibelli, N. Messuti, C. Napoli, P. Talamo, E. Caliano

pag. 89 **> CONSERVATION / CONSERVAZIONE**

pag. 90 **Seismic classification through the CARTIS protocol of a building aggregate located in the historical center of Cosenza.** / *R. S. Olivito, A. Verano, C. Scuro*

pag. 100 **The Castle of Cirò Marina (KR): seismic analysis and consolidation interventions.** / *R. S. Olivito, C. Scuro, D. Gaudio, S. Porzio*

pag. 110 **Intervento di restauro per il recupero e la conservazione delle colonne dell'atrio tetrastilo della Casa del Fauno di Pompei. /** *M. Osanna, V. Calvanese, A. Mauro, E. Gravina*

pag. 123 **Il museo di Palazzo Venezia a Roma: proposta di riallestimento dei depositi museali del palazzetto nel quadro di nuove strategie per l'efficienza energetica. /** *B. Lorenzi, G. Concordia*

pag. 134 **Analisi preliminare all'intervento di restauro: il caso della Villa romana di Minori. /** *E. Civale*

pag. 146 **Analysis of XVIII- XIX century construction techniques in the ancient terra laboris: Tarcagnota and Sementini palaces in Mondragone /** *F. Miraglia*

pag. 156 **Un metodo efficiente che integra le schede di rilevamento conservativo e i dati strumentali per i piani di prevenzioni e manutenzione nei musei. /** *C. Bertorello, A. Giovagnoli, M. Gittins*

pag. 170 **Il sito nuragico di Sant'Imbenia ad Alghero (SS). Stratificazioni, conservazione e il tema della protezione delle aree archeologiche. /** *B. Billeci, M. Rendeli*

pag. 181 **In merito ad un restauro per le mura di Castel S.Angelo (RI). /** *F. Festuccia*

pag. 195 **The so-called "Casina Borbonica" in Mondragone. Constructive analysis and restoration interventions. /** *F. Miraglia , E. Pagliaro*

pag. 209 **> VALORIZATION / VALORIZZAZIONE**

pag. 210 **Il diritto d'autore tra profili temporali della tutela, la digitalizzazione e il coordinamento con il Codice dei beni culturali e del paesaggio. /** *M. Leonetti*

pag. 220 **Museo e genere: il ruolo della donna nell'antichità. Proposta di un nuovo percorso di visita tra il Museo Nazionale di Atestino e le aree archeologiche delle città di Este. /** *C. Ahumada*

pag. 230 **Il Museo Morgagni di Anatomia Patologica: innovazione e nuove tecnologie per la valorizzazione museale dei resti umani. /** *G. Magno, A. Zanatta*

pag. 235 **Valorizzazione e restauro dei reperti archeologici dal Museo MAR di Positano.** / *A. Hansen, S. Metozzi, T. Peluso, S. Pacifico, G. Galasso*

pag. 248 **Narrare le connessioni invisibili dell'archeologia.** / *M. L. Nappi, S. Capece*

pag. 261 **Fundraising per i beni comuni: una strategia di recupero e valorizzazione anche dei beni culturali. Il caso delle comunità dei beni culturali napoletani.** / *V. Romanelli, R. Nicchia, M. Coen Cagli*

pag. 271 **"Qui i muri parlano". Proposta progettuale pratica e ragionata per la Chiesa Maggiore della Gravina di Riggio oltre i limiti dello storytelling culturale.** / *A. Racioppi*

pag. 284 **Testing the relevance of each of the eight mechanisms that drive charitable living in the context of Italian cultural organisations.** / *G. Giacomel, A. De Carolis, F. Costanza*

pag. 291 **> MATERIALI MICRO E NANO STRUTTURATI / MICRO AND NANOSTRUCTURED MATERIALS FOR INNOVATIVE CONSERVATION**

pag. 292 **Toward a green approach to the massive production of titania nanoparticles for application in Cultural Heritage.** / *E. Marconi, I. Luisetto, L. Tortora*

pag. 298 **Removal of silver corrosion products (tarnishing) using kaolinite (aluminium silicate).** / *R. Reale, E. Dell'Aglio, S. Francone, L. Campanella*

pag. 306 **Nanocoating antibatterici per l'inclusione sociale dei non vedenti in ambito museale.** / *C. M. Vitale, F. Palla, F. Fernandez, S. Germinario, R. Montagno*

pag. 313 **Culture Economy: Innovative strategies to sustainable restoration of artistic heritage. Part I - Development of natural gels for cleaning the stone materials of cultural heritage from iron stains and biodeteriogenic microorganisms.** / *L. Campanella, E. Dell'Aglio, R. Reale, F. Cardellicchio, A.M. Salvi, C. Casieri, G. Cerichelli, F. Gabriele, N. Spreti, G. Bernardo, A. Guida, V. Porcari*

pag. 325 **Culture Economy: Innovative strategies to sustainable restoration of artistic heritage. Part II - New materials and diagnostic techniques to prevent and control calcarenite degradation.** / *G. Bernardo, A. Guida, V. Porcari, L. Campanella, E. Dell'Aglio, R. Reale, F. Cardellicchio, A.M. Salvi, C. Casieri, G. Cerichelli, F. Gabriele, N. Spreti*

pag. 335 **A nonlinear parabolic model arising in marble sulphation: qualitative analysis.** / *R. S. Olivito, I. Torcicollo, G. Alì, C. Scuro*

pag. 345 **> NEW DIGITAL TECHNOLOGIES FOR CULTURAL HERITAGE / TECNOLOGIE DIGITALI EMERGENTI PER I BENI CULTURALI**

pag. 346 **Museo diffuso e percorsi digitali: il successo di BEPART.** / *A. Scognamiglio, P. Scognamiglio, E. De Cesare, S. G. De Cicco*

pag. 354 **Riproducibilità di sculture e opere d'arte antica in pietra da taglio per mezzo di sistemi tecnologici integrati.** / *V. Santoro*

pag. 366 **3D fruition of burials: physical limits and 3D reconstruction of the burial 14 of necropolis of Piovego (PD), Italy.** / *F. Adesso, E. Faresin, G. Salemi*

pag. 377 **3D Lab-Sicilia: un'infrastruttura di ricerca per la promozione e la conservazione di beni culturali.** / *R. Barbera, L. Licata*

pag. 382 **MArTA Lab: un fablab per i beni culturali.** / *U. Betocchi, N. Madeddu*

pag. 389 **> BEST THESIS AWARD 2021 / PREMIO MIGLIOR TESI 2021**

PhD

pag. 390 **Color measure, reproduction and assessment: from colorimetry to movie restoration.** / *A. Plutino*

Tesi Magistrali

pag. 402 **Photo fire: a digital archive to preserve analogue film technical heritage.** / *A. Crespi*

pag. 410 **Photophysical properties changes of organic dyes induced by accelerated UV aging.** / *A. De Carlo*

pag. 422 **Semantic & Interactive Technologies for Civic Education: Ancient Classical Polychromy as a Case Study.** / *D. S. Martinez Pandiani*

Ciò che chiamiamo realtà
deriva dalla nostra partecipazione attiva
alla sua costruzione.

Ilya Prigogine

Prefazione

Il XII convegno AIES giunge, nuovamente in presenza, in un tempo profondamente segnato dalla pandemia da Covid-19 e da tutto quello che da essa è scaturito. La profonda crisi generata dal virus non è stata solo sanitaria ma ha colpito vari settori della nostra esistenza.

Ci ha colpiti nella nostra fragile umanità, costringendoci a ripensare i tempi, a scandirli in una maniera nuova e condividerli più di quanto fossimo abituati a fare.
Ci ha colpiti nel delicato processo di formazione scolastica dei nostri giovani; ne sono testimoni i giovani ricercatori le cui tesi - di laurea magistrale e di dottorato - sono state ammesse agli atti.

Ci ha colpiti nell'economia, costringendoci spesso a rivedere interi processi di marketing e a guardare alle tecnologie informatiche con più fiducia e maggiore interesse. Anche in questi atti ce n'è testimonianza.

La pandemia ha anche colpito particolarmente il settore dei beni culturali e della cultura in generale che, solo da poco, grazie alle nuove normative di accesso e fruizione, riesce a ripartire. Questo ci rende doppiamente felici di essere nuovamente al MANN, testimoni che la vita nei luoghi della cultura è finalmente ripresa.

La ripartenza del convegno AIES si presenta ricca di lavori: abbiamo ricevuto infatti un numero molto consistente di contributi di altissimo valore scientifico e di variegata natura che ci permetteranno di esplorare lo stato dell'arte da diversi punti di vista. Accanto a temi classici e cari ad AIES come diagnosi e conservazione, verrà affrontato il tema delle nuove tecnologie a supporto del complesso lavoro di valorizzazione ma anche studi di natura sociologica e culturale, come quello sui fattori che spingono le persone a donare per progetti culturali.

Un ulteriore aspetto di novità che merita una particolare attenzione è rappresentato dalla presenza fra i lavori di contributi dedicati alla *Chimica Green* applicata ai beni culturali. .La chimica europea deve affidarsi alla green chemistry per competere con successo nel mercato.internazionale con la concorrenza dell'estremo e del medio oriente favorita da bassi costi di manodopera e di infrastrutture. La green chemistry ha trovato applicazioni di successo nell'alimentare, nell'energetico, nel farmaceutico, non ancora nel campo dei beni culturali che pure per un Paese come il nostro e per l'Europa in generale rappresenta un' eccellenza. Siamo felici che il nostro Convegno possa rappresentare una tappa di avvicinamento in questa direzione.

La scienza riflette il tempo. Questo tempo in cui si avverte la necessità di ritornare ad una spensierata socialità, lascia il segno anche negli articoli che leggerete. Non è un caso che quest'anno, in allegato agli atti, verranno distribuite le "Linee-guida per la promozione e lo sviluppo della raccolta fondi ad uso delle comunità attive per la gestione e valorizzazione dei beni comuni e per le loro reti", un piccolo libretto di indicazioni sul fundraising per i beni comuni che parte dall'esperienza dei beni comuni napoletani che coincidono con beni culturali di pregio.

Dietro a questo convegno c'è un lavoro che parte da lontano e che scandisce un tempo lungo 12 mesi. Parafrasando Cesare Brandi nella sua descrizione della struttura ritmica del tempo dell'opera d'arte, si può ritrovare la struttura ritmica persino nel convegno AIES:

- la durata: che non è di due giorni a dicembre ma che ci vede tutti impegnati per tutto l'anno: a ricercare, a studiare, a sperimentare, a connettere tutto ciò
- l'intervallo: o gli intervalli che sono le scadenze imposte nella call; le dead line sono indispensabili per la buona riuscita del convegno
- l'attimo: il momento in cui ci ritroviamo di nuovo insieme, a Napoli, per parlare di scienza, di cultura, di nuove sinergie, di futuri progetti
-

Tutto questo non sarebbe possibile senza lo straordinario supporto del Comitato Scientifico che, con molta cura e dedizione, valida tutti i lavori che ci vengono sottoposti e stimola, di anno in anno, la crescita del convegno sottoponendo nuovi temi e nuove strade da percorrere per arricchire sempre di più questo momento di incontro.

Senza la coniugazione di tutti questi lassi di tempo non esisterebbe il convegno AIES che, di anno in anno, cresce e si fortifica grazie a due punti cardine: il coordinamento delle attività ed il conforto della tecnologia.

Infine, non saremmo mai capaci di guardare abbastanza lontano senza il connubio tra lungimiranza ed esperienza che sono alla base della storia di AIES e la caratterizzano, rendendola un'esperienza sempre nuova e multidisciplinare.

Questa particolare edizione del convegno ci spinge ad una riflessione profonda sul Tempo, una variabile familiare ma forse poco approfondita della nostra vita, ed è per questo che in apertura degli atti vogliamo proporre una considerazione sul rapporto tra la valorizzazione ed il tempo.

Luigi Campanella
Ciro Piccioli
Anna Rendina
Valeria Romanelli

> Diagnosis
> Conservation
> Valorization
> Micro and nanostructured materials for innovative conservation
> Emerging digital technologies for Cultural Heritage
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> Diagnosi
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> Tecnologie digitali emergenti per i Beni Culturali
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Culture Economy: innovative strategies to sustainable restoration of artistic heritage. Part II - New materials and diagnostic techniques to prevent and control calcarenite degradation

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Key words > calcarenite microstructure, analytical techniques, materials innovation, consolidating and protective products, non-destructive test

Abstract > A wide spread of multidisciplinary results was obtained for the Smart Cities and Communities and Social Innovation project supported by MIUR and entitled "Product and process innovation for the maintenance, conservation and sustainable restoration of cultural heritage". The project basically suggested the opportunity to avoid emergency closures of sites of high historical, architectural and cultural value, to proceed with planned interventions that regularly guarantee the well-being of the site and its conservation. A new organization of the protection, conservation and maintenance system was thus proposed (Part I and Part II). The choice of the test materials was suggested by their widespread use in the monumental heritage of Southern Italy and in particular in 'The Sassi and the Park of the Rupestrian Churches of Matera – UNESCO World Heritage Site" which was the site where part of the lab activity was transferred "in situ". In particular, the reported activity in Part II describes the microstructural characterization of calcarenite and the identification of some compounds of its degradation by means the integrated use of different analytical technique. The paper also presents some non-destructive test results evaluating the behavior of both a newly developed acrylic-based product developed by the project's industrial partner (Icap Leather Chem Spa) and commercial consolidating and protective products.

1. Introduction

Calcarenite is the typical material of the architectural heritage of the city of Matera (Bernardo, Guida, 2015). It is a sedimentary rock of biochemical origin with a low degree of alteration in which fossil shells and skeletons of marine organisms can still be discerned. Calcarenite has different compositional and structural characteristics depending on the size grains and on the type and quantity of the matrix and of the cement. Consequently,

the properties of the calcarenite materials, such as porosity, mechanical strength and durability, have a high variability as well. Typically, they have a low mechanical strength, a high open porosity and a poor durability (Andriani, Walsh, 2002). The severity of alteration and degradation of calcarenite materials varies according to microstructural properties. It is not uncommon to note contiguous ashlars of the same wall face, one strongly alveolized, the other one only slightly damaged. In addition, the high variability of the microstructural characteristics of calcarenites also affects the behavior of the materials used as consolidating and protective agents. Since the 1980s, when a strategic plan for the recovery of the Sassi was launched, numerous restoration projects have been carried out. Various techniques and commercial formulas have been used to consolidate and protect the degraded materials, with results that have not always been effective. Yellowing and detachments have occurred over time due to the chemical, physical and mechanical incompatibility of acrylic and siloxane-based materials with the stone substrate. In addition, the repair and rebuilding of the mortar joints in the masonry with unsuitable materials and inadequate thicknesses have profoundly altered the aesthetic and formal value of the wall faces. Despite the numerous restorations that have been carried out, there is an almost complete lack of scientific data to assess the effectiveness of restoration techniques and materials. As part of the research activity, the aim was to fill this information gap by carrying out an in situ experimental campaign, the results of which were shared on the He-Main platform. In the case studies identified in the context of the ecclesiastical heritage, thanks to the collaboration of the Archdiocese of Matera, the building materials were first microstructurally characterized, forms of deterioration were identified. Subsequently different cleaning, consolidation and protection materials and techniques were tested. The present work reports on the microstructural characterization and identification of the forms of degradation carried out on the façade of the Church of San Pietro Barisano in the Sasso Barisano and on the interior of the Church of Santa Lucia alla Malve in the Sasso Caveoso. The paper also reports the results obtained by the treatment of the surfaces of the façade of the Madonna dei Derelitti by means some commercial products and a newly developed acrylic-based product by the project's industrial partner Icap Leather Chem Spa.

2. Microstructural characterization of calcarenite and identification of degradation products

2.1 Materials and Methods

Six samples, labelled SP1, SP2, SP3, SP4, SP5, SP6, were taken from the façade of San Pietro Barisano. Sampling was carried out by taking small amounts of material of up to a few tens of grams from areas considered particularly significant. Some microsampling was also carried out indoors in areas where salt efflorescence was particularly evident. Four samples, labelled SL1, SL2, SL3, SL4, were taken from the church of Santa Lucia alle Malve. Sampling was carried out on the non-frescoed areas affected by widespread biological degradation. The experimental activity was carried out through the integrated use of the following analytical techniques: 1) optical microscopy; 2) X-ray diffraction on powders; 3) micro-Raman spectroscopy; 4) X-ray microtomography. Optical microscopy provides an initial description of the sample, giving morphological and textural information. Diffractometric analysis allows the identification and semi-quantitative estimate of the mineralogical phases. Micro-Raman analysis using a microscope connected to the spectrophotometer provides information on individual crystals

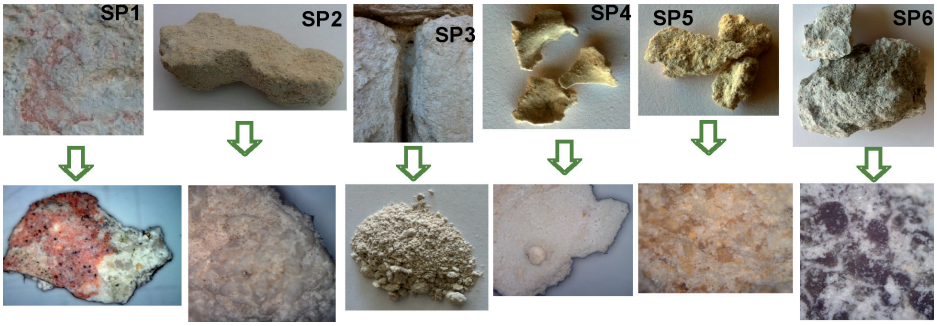

Figure 1. Samples of the façade of San Pietro Barisano observed by reflection light microscope.

or very small portions of the sample. X-ray microtomography allows the evaluation of the porosity of the sample. The optical microscope observations were carried out using a Zeiss microscope equipped with an AxioCam Erc 5s camera. Analyses of the sample were carried out in reflected light, while samples from which it was possible to obtain thin sections were observed in transmitted light. Diffraction data were collected using a Panalytical Empyrean powder diffractometer with Bragg-Brentano geometry, equipped with a Pixel 3DI detector and operating at 40kV/40mA. The scans were performed in θ-θ mode exploiting the Kα radiation of copper (1.54 Å) monochromatized with a large β-nickel filter. Diffraction data were collected over a range of 2θ 5-85°C with a step size of 0.013°, an acquisition time of 149 s and a scan rate of 0.0001°/s. The sample was first ground in a mortar and then deposited on the sample holder. A plexiglass plate was used as the sample holder in random mount mode. The analysis of the phases present was carried out using electronic databases implemented in the Highscore 4.0 software. The Raman spectra were acquired by means of a LabRAM HR Evolution spectrometer, equipped with an Olympus binocular microscope, an Ultra-Low Frequency (ULF) filter, 600 and 1800 g/mm grids and a Peltier-cooled CCD detector. Raman spectra were excited using a He-Ne laser (632 nm); the laser input power was reduced with a filter whose optical density was selected in each experiment to prevent sample heating effects; the power focused on the sample was never more than a few mW. Exposure times of 100 s were generally used; the final spectra are the average of 3 repeated acquisitions. The spectra were calibrated using the 520.5 cm-1 line of a silicon wafer.
The porosity analyses were carried out using a Bruker Skyscan 1172 high-resolution X-ray microtomographer equipped with a polychromatic X-ray tube and operating at 100kV/100mA. The 3D scans were carried out using the following operating conditions: pixel size of 5 m; field of observation (FOV) of 20 x 10 mm; Al+Cu filter; rotation step of 0.26°; frame average of 5. After acquiring the projections of the samples, the cross sections (slices) were reconstructed with Bruker's NRecon software using the Feldkamp algorithm (Feldkamp et al, 1984). During reconstruction, a Gaussian smoothing of 6 was applied to reduce noise. The effect of beam hardening was minimized by setting the beam hardening correction value to 36%. Ring artefacts were reduced by using a ring artefacts compensation value of 5 (Sijbers, Postnov, 2004). Quantitative 3D analyses were performed with Bruker's CTAn software. A volume of interest of 1500 mm3 was selected for each sample. The pore and solid size distribution were determined using the sphere-fitting algorithm (Remy, Thiel, 2002).

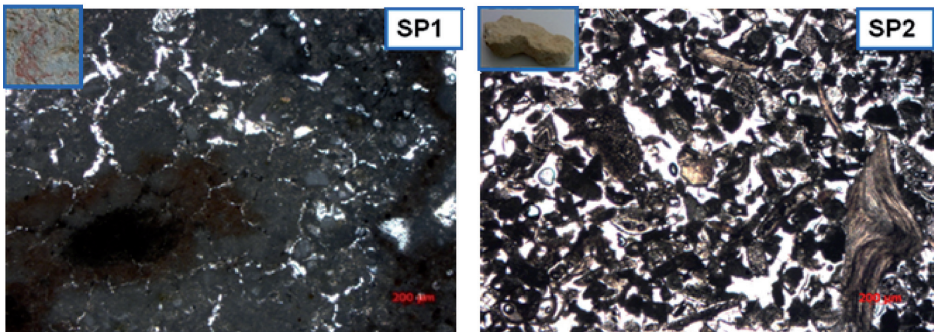

Figure 2. Thin sections of samples SP1 and SP2 of San Pietro Barisano observed by transmission light microscopy.

2.2 Results and discussions

The light microscopy observations in Figure 1 showed that sample SP1 is a calcarenite covered with abundant red and brown-black crystals. Small fractures containing yellow acicular crystals can also be recognized locally. Sample SP2 consists almost entirely of calcite crystals with a few orange crystals. Sample SP3 is a powder taken between one tuff and another, while sample SP4 is a smooth and compact white patina. Sample SP5 is a calcarenite with calcite crystals ranging in color from white to yellow and rare brown crystals. Sample SP6 consists of calcite crystals that are sometimes brown to black on the surface. Sample SP2 is the most representative of the calcarenite with which the church façade was built, while sample SP1 was of particular interest as the façade of the church appears to be diffusely affected by reddish patches that are also very extensive in places. Therefore, these two samples, were selected to make thin sections and carried out optical microscope observations (Fig. 2). According to the classification of Folk (Folk, 1959), sample SP1 is a biomicrite with secondary porosity not filled by cement or matrix. This sample also shows local portions occupied by reddish patinas. Sample SP2, on the other hand, is a bioclastic calcarenite with considerable primary unfilled

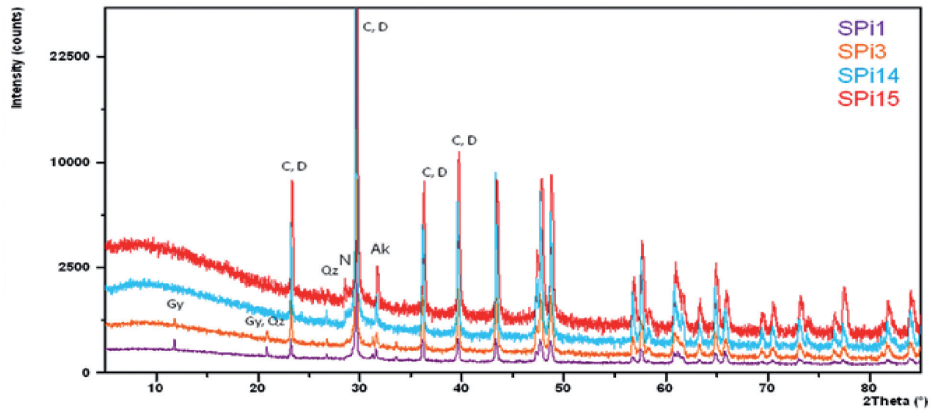

Figure 3. Diffractiongrams of samples taken from the interior of the Church of San Pietro Barisano.
Caption: C = calcite ($CaCO_3$); D = dolomite ($MgCa(CO_3)_2$); Gy = gypsum ($CaSO_4$); Qz = Quartz (SiO_2); N = nitratin ($NaNO_3$); Ak = akermanite ($Ca_2MgSi_2O_7$).

	SP1	SP2	SP3	SP4	SP5	SP6
Calcite ($CaCO_3$)	64	85	62	100	97	100
Dolomite ($MgCa(CO_3)_2$)	20	8	18	-	-	-
Anhydrite ($CaSO_4$)	13	5	10	-	-	-
Quartz (SiO_2)	3	2	3	-	3	-
Halite (NaCl)	-	-	7	-	-	-

Table 1. X-ray diffraction analysis of samples taken from the façade of San Pietro Barisano, percentage by mass

porosity. Semi-quantitative X-ray diffractometric analysis was conducted on all samples to identify the mineral phases present (Table 1). Some samples taken inside the church on the right-side wall of the apse affected by degradation due to salt efflorescence were also analyzed by X-ray diffractometry. Calcite is the main mineral in all the facade samples. Dolomite and anhydrite are present as secondary phases in samples SP1, SP2 and SP3. Small amounts of quartz are present in samples SP1, SP2, SP3 and SP5. Halite was also detected as a minor component in sample SP7. In some samples taken from inside the church, the X-ray diffraction analysis revealed the presence of gypsum, nitratin and akermanite (Figure 3). Except for sample SP3 in powder form, all samples were subjected to micro-Raman spectroscopy analysis. In the red and brown-black crystals of sample SP1, the presence of hematite and magnetite, respectively, was detected. For samples SP2, SP4 and SP5, Raman analysis of the crystals on the surface provided spectra typical of calcite. In the case of sample SP6, in addition to the typical calcite spectra, bands due to C-C vibrations were evident in the darker areas (Figure 4). Finally, X-ray microtomography analyses were carried out on samples SP2 and SP6 (Table 2). Both samples have a high porosity almost exclusively of open type.

Figures 5 and 6 show the images in optical microscopy of the samples taken from the interior of the Church of Santa Lucia alle Malve. Based on Folk's 1959 classification (Folk, 1959), sample SL1 is a biomicrite containing silico-clastic material with secondary unfilled porosity. This sample also shows a patina of alteration in which the original material is hardly recognizable. Samples SL2 and SL3 can be classified as a bioclastic calcarenite with significant primary unfilled porosity. Patinas and silicoclastic material are also present in the sample SL3. Sample SL4 is a calcarenite with unfilled primary porosity. It also differs from the other samples due to the presence of many intraclasts.

	SP2	SP6
Average granule diameter (μ m)	73.5	103.3
Average pore size (μ m)	72.9	92.3
Closed porosity (%)	0.3	0.4
Open porosity (%)	42.7	36.1
Total porosity (%)	43.0	36.4

Table 2. X-ray microtomography analysis of samples SP2 and SP6 taken from the façade of the Church of San Pietro Barisano-façade of San Pietro Barisano, percentage by mass

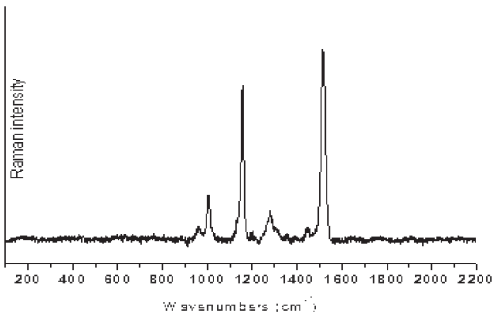

Figure 4. Micro-Raman spectra of the dark areas on the surface of the SP6 sample taken from the façade of the Church of San Pietro Barisano.

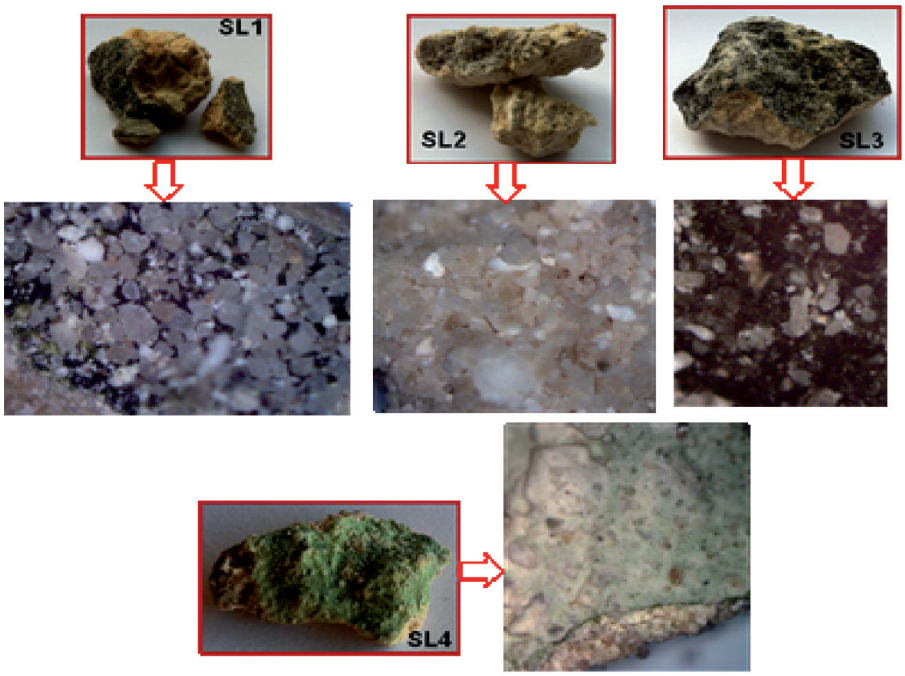

Figure 5. Samples of the interior of the Church of Santa Lucia alle Malve observed by reflection light microscope.

Finally, sample SL2 was selected for the X-ray microtomography investigations as representative of the calcarenite in which the church of Santa Lucia alle Malve was excavated. The sample has an average granule diameter of 84.3 μm, an average pore diameter of 59.9 μm, a closed porosity of 0.1 %, an open porosity of 36.5 % with a total porosity of 36.6 %. The results obtained show that the porosity of the calcarenite of the church of Santa Lucia alle Malve is slightly lower than that of the calcarenite (sample SP2) of the façade of the church of San Pietro Barisano.

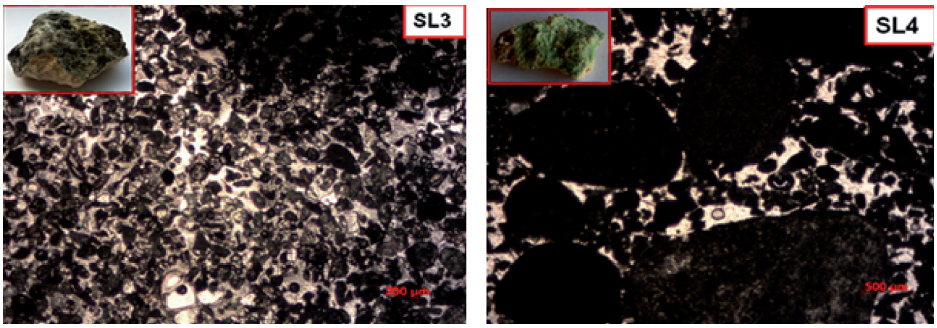

Figure 6. Thin sections of the samples SL3 and SL4 observed by transmission light microscope.

Semi-quantitative X-ray diffractometric analysis showed the presence of calcite and quartz in all samples (Table 3). Anhydrite was detected as minor component in samples SL1, SL2 and SL3. Halite and kaolinite are also present in SL1 and SL2, respectively. Results of Raman spectroscopy analysis showed in Table 4 revealed the presence of calcite crystals, calcium oxalate dihydrate and carboxyl groups on the surface of all four samples analyzed.

3. Evaluation of the behavior of commercial and newly developed consolidating and protective products

3.1 Materials and Methods

Four homogeneous zones, labelled A, B, C and D, of the façade of the Church of Madonna dei Derelitti were treated with both commercial products and the consolidating agent, named HR2, based on acrylic resins, and developed during the research activity by the industrial partner Icap Leather Chem Spa (Fig. 7). The products were applied with a 5 cm soft brush until the surface was saturated. During the treatment of the surfaces, the quantities of product used, the environmental conditions (temperature and humidity) and subjective criteria such as ease of application, tendency to foam, ease of absorption etc. were recorded. The surface A was divided into 4 zones, A1, A2, A3, A4, in which the newly developed HR product was applied with a dilution ratio of 1:4, 1:6, 1:8 and 1:10 in distilled water, respectively. On surface B, first an ethyl ester-based consolidating agent in solvents was applied and, successively, a protective coat based on silanes and siloxanes in solvents supplied by the same manufacturer as the consolidating agent. A single product marketed as a consolidating and protective agent containing ethyl ester was applied to the C surface. A self-cleaning protective coating based on titanium dioxide nanoparticles was applied to surface D.

Six months after application of the products, Colorimetric Test, Scotch Tape Test and Water Absorption Test were carried out to verify the effectiveness of the products through non-destructive in situ tests (Fig. 8). Colorimetric test determines the chromatic variations induced by treatments (UNI EN 15886:2010). The instrument used is a Konica Minolta CM-2600d spectrophotometer. The chromatic values are expressed according to the L*a*b*color space of CIE (Commission International d'Eclairage), where L* is the brightness coordinate, a* (red/green) and b* (yellow/blue) are the coordinates that define the hue (positive values of a* fall in the red zone, negative values in the green zone; positive values of b* fall in the yellow zone, negative values in the blue zone). Measurements were carried out using an aperture with a diameter of 8 mm, specular component excluded (SCE), illuminant D65 and viewing angle 10°. The results obtained represent the average of 3 measurements taken on the untreated area and on each

	SL1	SL2	SL3	SL4
Calcite ($CaCO_3$)	90	84	92	98
Quartz (SiO_2)	3	2	2	2
Anhydrite ($CaSO_4$)	5	7	6	-
Halite (NaCl)	2	-	-	-
Kaolinite ($Al_2Si_2O_5(OH)_4$)	-	7	-	-

Table 3 - X-ray diffraction analysis of samples of the interior of Santa Lucia alle Malve, percentage by mass

Wavelength number (cm^{-1})	Compound
158; 283; 711; 1088	Calcite
432; 494; 607; 1263	Weddellite ($CaC_2O_4 \cdot 2H_2O$)
1327; 1389; 1666; 1702; 1761; 1772	Carboxylic groups-COOH

Table 4. Raman spectroscopy on samples from Santa Lucia alle Malve

Figure 7. Façade of the Church of Madonna dei Derelitti with indication of the treated areas.

area treated with different products. Scotch Tape Test or Peeling Test is a test used in situ to assess the degree of cohesion of a stone surface (ASTM D3359-08, 2008). A strip of adhesive paper with known dimensions (50 x 22 mm) is weighed with an analytical balance with a sensitivity of 0.1 mg; it is then pressed onto the stone surface and left to adhere to it for 90 seconds. The difference between the initial weight and the final weight of the strip corresponds to the weight of the stone material detached from the support; this quantity reflects the cohesion characteristics of the stone. The test was repeated four times at each point. Water absorption test using the contact sponge method to assess the degree of water repellency and reduction of porosity determined by products applied to stone surfaces (UNI 11432:2011). The measurements were carried out on treated and untreated areas, using a quantity of water equal to 5 ml and a contact time of 1 minute.

3.2 Results and discussions

About color variations, only zones B and C on the façade, showed color differences below the threshold ΔE=5 perceptible to the naked eye (Table 5). As regards evaluation of the cohesion of the stone, the measurements of the total mass of the material detached from the stone support reported in Table 6 show that zones B and C, released the greatest mass, comparable to that obtained from the untreated stone surface. The lowest release of mass was recorded for zone A4 treated with newly developed product HR2 in demineralized water with dilution ratio of 1:10 and for zone D, on which commer-

Figure 8. Non-destructive in situ tests to evaluate products behavior. Colorimetric Test on the left; Scotch Tape Test in the middle; Water Absorption Tape Test on the right

Area	L*	a*	b*	ΔE	ΔL	ΔL/ ΔE	Co	C'	ΔC
Untreated area	66,67	3,63	13,89				14,36		
A1	73,40	2,44	12,05	7,08	6,73	0,95		12,29	-2,07
A4	72,87	3,06	12,45	6,39	6,20	0,97		12,82	-1,54
B	71,56	3,52	14,09	4,90	4,90	1,00		14,52	0,16
C	70,81	4,08	14,69	4,24	4,14	0,98		15,25	0,89
D	71,52	2,66	11,26	5,60	4,85	0,87		11,57	-2,79

Table 5. Colorimetric test on the façade of the rupestrian Church of Madonna dei Derelitti

cial self-cleaning protective product was applied. In all cases, there was a progressive decrease in the weight of the material from the first test (point 1) to the last test (point 4). The results obtained by water absorption test show that all the treatments imparted an excellent water-repellent capacity to the stone. The absorption water of the untreated surface has been equal to 38,59 mg·cm2/min. The best values have been obtained on the surface A1 treated with HR2 with dilution ratio of 1:4 (1,46 mg·cm2/min) and on the surface D (0,92 mg·cm2/min). The water absorption values on zones A4, B and C were 3,34, 3,55 and 1,84 mg·cm2/min. In conclusions, the innovative product HR2 and the commercial self-cleaning protective products are more effective in terms of consolidation and impermeabilization but impart color variations to the stone that are perceptible to the naked eye.

Caption: ΔE= em to continuously monitor the environmental parameters by means of a network of sensors installed inside and outside the historical and artistic monuments and information modeled on objective parameters that describe the rate of degradation, identified by experimental results and analytical research and on accounting records of previous work eventually already performed. Documents and research experiments on real cases can be shared through the reproducible HeMaIn methodology in all intervention processes on the Architectural Heritage to be restored, preserved and enhanced.

Acknowledgements

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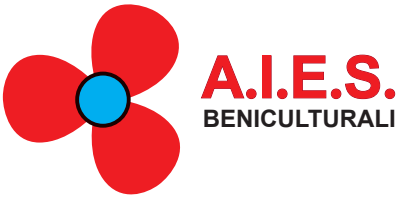

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