



# 5<sup>th</sup> Historic Mortars Conference

**HMC 2019**

## **Book of Abstracts**

University of Navarra, Pamplona

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Universidad  
de Navarra



# 5<sup>th</sup> Historic Mortars Conference (HMC 2019)

## Book of Abstracts

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# **Foreword**

**Prof. José I. Álvarez**

University of Navarra, Pamplona

Chairman 5<sup>th</sup> HMC 2019





It is my pleasure to write this preface on the occasion of the 5<sup>th</sup> Historic Mortars Conference and to welcome you to Pamplona, and, in particular, to the University of Navarra. The HMC series reaches a good number and takes us the opportunity to look back, remembering the previous editions in Lisbon, Prague, Glasgow and Santorini. I would like to thank the effort and underline the success of my colleagues who were at the head of the previous editions: Rosário Veiga - who had the intuition and good judgment of promoting the first edition -, Jan Válek, John Hughes and Ioanna Papayianni. Without forgetting Caspar Groot, who, as former chairman of successive RILEM Committees on historic and repair mortars, encouraged and contributed to the success of the previous events.

Thanks to the effort of all of them and their respective teams, we can confirm that HMC's are fully consolidated as a one-of-a kind forum to deal with historic mortars, renders and grouts, the variety of their compositions and raw materials, diverse methodologies of study, archeological sites, dating, preservation, durability, obtaining of repair materials and testing methods, consolidation, and even the upgrade of the repair mortars by nano-technology.

This HMC 2019 gathers 140 scientific and technical high-levelled contributions of participants from the five continents. Up to twelve different topics are displayed in the Conference Programme, thus emphasizing the wide range of approaches that enrich the final outcome of the event. The profitable exchange between the science advances and the practice taking place during the Conference is supported by contributions focused on a more theoretical scientific point of view combined with those works more oriented towards real applications, including case studies, assessment of the state of conservation of some ancient materials or monitoring of the effectiveness of some rehabilitation procedures and repair materials.

The old Kingdom of Navarre offers you a thriving, avant-garde community with a great natural and cultural diversity. Pamplona is a medium-sized city with bi-millennial history. Enjoy its modern and calm character and its monuments, ancient walls and historical passages. The gastronomic side is also guaranteed for all palates and tastes. In good connection with the framework of the Conference, the preservation and restoration of the Built Heritage is made reality in Navarre. The institution Príncipe de Viana of the Government of Navarre takes care of the vast Architectural Heritage, part of which is ascribed to the Way of Saint James, which passes through these lands.

I take for granted that the University of Navarra, with its always welcoming spirit, is a venue where the great eagernesses devoted to this conference bear fruit. I must thank many people who have made this event possible: those responsible for the University itself, the sponsors for their invaluable help and my colleagues of the Department of Chemistry who have cooperated in the organization.

Finally, the success of this HMC 2019 stems from the know-how and competence of these people below that are gratefully acknowledged: the members of the Scientific Committee for their intense work and constructive criticism of all the submitted contributions, Prof. Antonia Moropoulou for delivering such a remarkable inaugural lecture, Leopoldo Gil Cornet for being an unbeatable guide to observe good practices of restoration, the chairpersons and, of course, all the speakers and participants.





## **Inaugural Conference**

**Historic mortars of the Holy Aedicule and the Tomb of Christ supporting the design and evaluation of performing and compatible restoration mortars and revealing the history of the monument**

**Prof. Antonia Moropoulou *et al.***

National Technical University of Athens, Greece



The rehabilitation of the Holy Aedicule of the Holy Sepulchre in Jerusalem, an extremely demanding project, was completed in March 2017 by the National Technical University of Athens (NTUA) interdisciplinary team. The project was based on the results and proposals of an interdisciplinary diagnostic study, conducted by the same team in 2015, and aimed to address the severe deformation and intense buckling of the stone facades, enveloping this complex structure with multiple structural layers and construction phases. The diagnostic study, revealed that rising damp from the underground, in addition to the effect of rain water precipitating through the open oculus of the Rotunda dome on the Holy Aedicule in the past had lead to the disintegration and swelling of the historical mortars of the inner construction layers, enclosed within the marble facings, thus playing a critical role in the deformation of the structure.

Throughout the project the role of mortars, both joint mortars and filling mortars, was decisive in achieving the structural integrity of the monument, as well as in ensuring sustainability of the interventions. The analysis of the historic mortars of the structure was the basis for the design of repair mortars, which were required to present both compatibility with the historic materials and performance in relation to dynamic stresses and the negative environmental factors affecting the monument. At the same time, the analysis of the historic mortars, revealed the history of this important monument.

Based on the experience and relevant research results of the NTUA team regarding both historical (over 400 historical mortar samples across the Mediterranean basin as well as the historical mortars of Hagia Sophia in Istanbul, historical buildings in Venice) and restoration mortars (designed through the reverse engineering methodology) a study was conducted to select a compatible repair mortar with early and fast acquisition of mechanical strength, as well as with a high degree of resilience in high humidity conditions. Lime-metakaolin mortars were selected as the most compatible and performing type of repair mortar for the rehabilitation of the Holy Aedicule structure, based on physicochemical and physico-mechanical criteria, as well as on the level of mechanical performance of the structure under static and dynamic loads, through the use of a finite element model (FEM), within the interdisciplinary framework applied.

A restoration “concrete” type mortar was also necessary for the rehabilitation works, to be applied between the masonry and the stone facings, and it was decided to use the same type of mortar as the joint repair mortar, however with the addition of lightweight aggregates of higher gradation. Grout injections were performed to homogenize the different construction phases and layers. For this purpose a lime-metakaolin based grout was selected to ensure the highest degree of compatibility and performance. The applied materials and rehabilitation interventions were assessed during the rehabilitation through non-destructive techniques and at the completion of the project, utilizing the interdisciplinary tools of the NTUA team, to assess the response of the rehabilitated Holy Aedicule structure to dynamic stresses and the diminution of the deformations. Through this exemplary project the role of mortar study, design and application proved crucial within the integrated scientific support to decision making, highlighting the importance of materials in restoration interventions.





## **Topic 1**

Earth-based plasters and mortars on archaeology and  
historic constructions

**Chaired by**

Paulina Faria

Universidade Nova de Lisboa, Portugal





## **Earth-based plasters: the effect of anhydrite stabilization**

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### **Abstract**

Earth-based plasters have been used in buildings construction for millennia. Unstabilised earth-based mortars harden by drying and, when in contact with water, they get plastic again due to their high hygroscopicity. That is an advantage in terms of reutilization, allowing a dynamic use all along the plaster service life, but it can also be a drawback when these applications become in contact (even accidentally) with water. Furthermore, nowadays requirements claim for increased durability because maintenance actions tend to decrease. Therefore, durability towards water action is foreseen, but without jeopardizing both, compatibility with the substrates where earth-based plasters are applied, and their main technical advantages, such as the contribution to indoor humidity equilibrium by moisture buffer capacity and recyclability. Stabilization of earthen materials has been tried through the use of several types of additions. In the case of earthen plasters, one of the most common is the addition of low binder content, such as air lime. Although increasing durability towards water, it changes the colour of the plaster, decreases mechanical strength and, particularly, it turns the earthen plaster like an artificial stone (no longer dynamic) and decreases hygroscopicity.

Previous studies established the positive effects of the stabilization through the addition of calcium sulphate hemihydrate to earth-based plasters. Calcium sulphate hemihydrate is obtained with very low firing temperature of gypsum rock. With a higher firing temperature, anhydrite – calcium sulphate dehydrate – can be produced. Nevertheless, the firing temperature of anhydrite is much lower in comparison with the one to obtain other binders, such as calcitic air lime.

In this article, the effects of the addition of anhydrite to a ready-mixed earth-based plaster are assessed and discussed, in comparison with the same plaster stabilised with calcium sulphate hemihydrate. Therefore, samples of the unstabilised ready-mixed earth-based plaster were prepared, as well as samples of the same plaster with 5 %, 10 % and 20 % of anhydrite and with 20 % of hemihydrate, by volume of the ready-mixed product. The physical properties of these mortars were characterized in fresh state and after one month, namely in terms of shrinkage, bulk density, flexural and compressive strength, surface cohesion, dry and wet abrasion, capillary absorption and drying, sorption and desorption. The advantages and drawbacks of anhydrite stabilization of the ready-mixed earth plaster are highlighted.

### **Keywords**

Anhydrite; Clayish Earth; Durability; Gypsum; Mortar

## **Similar appearance of mortar and brick masses in Algiers Casbah houses during the Ottoman period (16<sup>th</sup>- early 18<sup>th</sup> centuries)**

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### **Abstract**

The traditional mortars used in the Algiers Casbah houses during the Ottoman period (16<sup>th</sup>-early 18<sup>th</sup> centuries) are particular because their mass has a similar appearance to the bricks. This is due to the reddish colour of these earth-based mortars stemming from the use of clay and other component such as sand, large grains of fired crushed ceramics of 5 mm or more, pebbles from sedimentary stones, shales, remains of nummulite limestones from the Algerian Atlas and ashes from the lime calcination. The mortars were applied to the mixed masonry walls in layers, thicker or equal to the brick rows that they bind; whereas in the ceilings they were layered in thick beds interspersed with bricks until 30-40 cm. Lime nodules without hydration is due to the artisanal mixing with limited water to maintain the compactness of the mortar. For the purpose of this study, XRD, DTA, SEM and other tests were carried out on material samples. In light of this study, it is concluded that the Roman tradition of using lime and cooked crushed ceramics is maintained in the earth-based mortars of the Ottoman period; knowing that their appearance is similar to the bricks; one can argue that the bricks look like a baked mortar.

### **Keywords**

Casbah; Ottoman Period; Mortar; Lime; Fired Crushed Ceramics

## **Assessment of adhesive strength of an earth plaster on different substrates through different methods**

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### **Abstract**

In recent years earthen mortars and plasters have been calling the attention of the building community not only because they are compatible with historic masonries that included earth or rubble stone, are reversible, but also because they can be ecological and efficient even when applied on common contemporary masonries. Particularly the contribution earthen plasters can give to relative humidity indoor equilibrium, based on the high hygroscopicity of clays, classifies them as passive technologies to achieve indoor comfort.

Nevertheless, the knowledge on the application of earthen plasters was almost lost in developed countries and is being re-gained. One of the aspects that needs to be addressed is the adhesive strength on substrates. The DIN 18947:2013 assesses this characteristic based on EN 1015-12: 2016. However, there are problems to assess the adhesion by this standard even for air lime plasters and renders. Therefore, this study intends to contribute on how to assess adhesion of an earth plaster to a masonry.

The adhesion capacity of an earth plastering mortar formulated in laboratory is performed by tensile and shear tests, on two different substrates, such as adobe and hollow fired clay bricks. The substrate are prepared differently, with and without prior application of a clay slurry. The test samples were produced also differently, by cut while fresh, cut after hardening and directly sample moulding with aimed test dimensions. Tests were performed in two different relative humidity environments: 65 % and 95 %. The results are compared, evaluating the influence of the different parameters, and compared with results of other plasters. Results show that the earth plaster have a good performance regarding adhesion on both substrates studied, being advantageous the preparation of the support with a clay slurry. The cutting procedure of the samples influences the test results. The relative humidity increment has a negative effect but even a high percentage does not compromise the stability of the plaster adhesion. The shear test proved to be a valid instrument, requiring, however, dissemination in the scientific community in order to be improved.

### **Keywords**

Clay; Earth; Mortar; Adherence; Test Procedure

## **Earth-based plasters: the influence of clay mineralogy**

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### **Abstract**

Earthen mortars were the first type of mortars to be used by mankind when ancestors mixed excavated earth with water to fill the space between wood branches to build shelter. Thenceforth earth-based mortars have been used all over the world, just as wall masonry mortar or as wall protective or decorative layers, in an extensive range of building types, from vernacular architecture to monuments. Each earth is a natural raw material characterized by its own and unique properties, due to its diverse composition comprising certain types and content ratios of clay, silt, sand and gravel. Therefore earth-based mortars properties are highly dependent on the type of earth used. Nowadays plastering is one of the main applications of earth-based mortars, for which raw earth is disaggregated and sieved, to eliminate gravel. If a too high content of clay is present, sand (or other artificial aggregate) has to be added in order to control shrinkage. For this purpose a recurrent proportion between the fine particles of earth (clay and silt) and sand is 1:3 in volume, with water addition of 20-30 % of dry components weight.

Clay minerals play a key role in this type of mortars being responsible for its setting process, associated only with drying, and when used for plastering, its contribution to equilibrium of indoor hygrometric conditions due to clay minerals high hygroscopicity.

Montmorillonitic clays are known for having a very high hygroscopicity but also a very high shrinkage. Kaolinitic clays, at the other end, are known for having the opposite: low shrinkage but also low hygroscopicity (for clays). Illitic clays normally present average conditions, with a compromise between shrinkage and hygroscopicity. Although these effects are known, they were not yet quantified when the clays are used to produce plasters, as well as its influence on plasters strength and durability.

To assess the influence of different types of clays, mortars were produced and plaster samples were prepared with montmorillonitic clay, kaolinitic clay and illitic clay, considering the same volume content ratio of siliceous sand. Mixing and testing followed the DIN 18947 standard. Mortars fresh state characterization and plasters hardened characterization were performed and are presented in this study. Results are compared between the different clayish plasters and with results obtained with other plasters, from previous studies.

### **Keywords**

Clayish Earth; Montmorillonitic Clay; Kaolinitic Clay; Illitic Clay; Mortar; Shrinkage; Hygroscopicity

## **Earth-based and binder-based mortars comparison**

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### **Abstract**

Earth-based mortars and plasters are present in archaeological sites, historical constructions and vernacular architecture all over the world. Nevertheless, the traditional knowledge once associated with formulation, mixing and application of this type of mortars by craftsmen is not commonly known by historic building conservation professionals as well as other professionals evolved in general building rehabilitation. Also physical and mechanical properties of earth-based mortars and their hygroscopic dynamic behaviour are not yet as deeply studied by scientific community as other more conventional binders-based mortars are, like gypsum, air lime or natural hydraulic lime mortars.

This study aims to compare physical, mechanical and hygroscopic properties of mortars based in three different binders and with an earth-based mortar. Binders-based mortars were formulated with respectively hemi-hydrated gypsum, air lime and natural hydraulic lime, at a volume ratio of 1:3 (binder:siliceous sand). The earth-based mortar was formulated with the same volume ratio of a clayish earth and siliceous sand. The clayish earth, characterized by the authors in previous studies, presents prevalence of ilite clay mineral, responsible for medium conditions regarding the balance between shrinkage and hygroscopicity.

The mixing of binder-based mortars as well as fresh state and hardened state characterization followed EN 1015 standards, while preparation and test of earth-based mortar followed DIN 18947 standard (which partially remit also to EN 1015 standard series).

Results are presented and discussed through the comparison between the assessed binder-based mortars and the earth-based mortar, as well as results obtained by other authors for other sets of mortars. The key property differences are highlighted in terms of advantages and drawbacks for building conservation and rehabilitation.

Conclusions highlight the earth-based mortar mechanical resistance, clearly higher than air lime mortar and also its significant hygroscopic dynamic behaviour, even compared with the gypsum mortar. The assessed earth-based mortar presented a very high adsorption and desorption capacity, which allow to anticipate that these type of mortars, when used for plastering, can act as moisture buffers, significantly contributing to the equilibrium of indoor hygrometric conditions of buildings.

### **Keywords**

Clayish Earth; Plaster; Gypsum; Air Lime; Natural Hydraulic Lime

## **Earth-based and current plasters: assessment of efficiency and contribution to indoor air quality**

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### **Abstract**

Over the last years constructive techniques have evolved with the aim of contributing to thermal and acoustic comfort of buildings occupants. Nevertheless, this evolution has not been verified with regard to indoor air quality, which depends of the building materials (namely coatings), type of occupancy, heating and ventilation systems. The concentration of local contaminants, like the emission rate, is also a feature that should be considered. The indoor air quality is very important for comfort but also for health of buildings inhabitants, since poor indoor air quality can lead to adverse health effects, like allergies and chronic diseases such as asthma. It is important to note that people spend much of their time inside buildings. For this reason the indoor air quality is a major issue not only for new construction but also for the conservation of old buildings.

Earthen mortars present several advantages: the raw material is natural, non-toxic, low costly, with low CO<sub>2</sub> emissions – low embodied energy cost, reusable (when not chemically stabilized), presenting the ability to adsorb and release relative humidity of the indoor spaces due to hygroscopic capacities of clays. But earth plasters characteristics are not often tested in comparison with current plasters.

It is considered that it is extremely important to understand the relationship between indoor air pollutants and the ability of a plaster system, based on mortars, to capture these pollutants and contribute to the regulation of temperature, relative humidity and, consequently, indoor air quality. Thus, in this study the physical and mechanical characteristics of three earth-based plastering mortars are evaluated, a ready-mixed earthen mortar, a laboratory formulated earth plaster and an earth-air lime mortar, in comparison to two ready-mixed plasters commonly used in Portugal, one based on hemi-hydrated gypsum and the other on hydraulic binder. The capacity of regulation of indoor air quality of buildings is evaluated through the assessment of sorption-desorption performance and a newly test to assess the capacity to capture pollutants.

### **Keywords**

Current Mortars; Earth Mortars; Eco-Efficiency; Hygrometric Equilibrium; Interior Air

## **Earthen plasters based on illitic clayish earth – the influence of calcitic lime addition**

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### **Abstract**

Earth-based mortars can be considered an eco-efficient solution for interior plastering in rehabilitation of vernacular building heritage as well as in contemporary architecture. Apart of their low embodied energy, earth-based plasters can promote physical and chemical compatibility with historic masonry. Furthermore earth-based plasters may give a significant contribution for health and comfort of building inhabitants due to high hygroscopicity of clay minerals. By acting as a daily moisture buffer these plasters can contribute to balance relative humidity of indoor environment and therefore promote building energy efficiency by decreasing the needs of mechanical ventilation and air conditioning.

This paper presents a study developed as part of a comprehensive ongoing research regarding earthen plasters based on illitic clayish earth extracted from a clay quarry located in Algarve sedimentary basin (South of Portugal), which revealed a high potential for interior plastering as presented in previous campaigns of this research.

This study aims to assess the influence of calcitic lime stabilization in the vapor adsorption and desorption capacity of these illitic earth-based plasters and its eventual contribution to enhance other properties of these plasters. For this assessment the German norm for earth-based plasters, DIN 18947, was followed, along with EN 1015 standards and other applicable standards and protocols. Four sets of samples were prepared considering the same volumetric proportions of clayish earth and siliceous sand, respectively 1:3. One was considered the reference mortar, without any lime addition, while the other three mortars were added with different amounts of calcitic lime putty, respectively 5 %, 10 % and 20 % regarding the volume sum of clayish earth and sand. Results from the assessment of the four mortars are presented and discussed in terms of fresh state, hardened state characterization, namely mechanical resistance, adhesive strength, abrasion resistance and liquid water erosion, as well as dynamic vapor adsorption and desorption.

Conclusions highlight that calcitic lime addition, and its increment, significantly reduce vapor adsorption and desorption capacity of this kind of illitic earth-based plaster. The expected improvement of mechanical strength and surface abrasion resistance was not confirmed; in fact, the addition of low amounts of calcitic lime presented a significant negative effect, decreasing these properties. However, this negative effect was minimized in formulations with higher contents of calcitic lime addition.

### **Keywords**

Earth; Mortar; Air Lime; Mechanical Characterization; Vapor Adsorption

## **Rescuing the manufacturing process of traditional mortars present on XIX-century earthen buildings in Brazil**

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### **Abstract**

Traditional earthen architecture techniques such as of wattle-and-daub, rammed earth and adobe were the usual approach to building in inland Brazil up to the end of the XIX century. As a result, a legacy of historical constructions of exceptional value has been passed onto our generation and its preservation is essential to guarantee the permanence of tangible records of building practices throughout the history of the country. This heritage, though, is at risk of disappearing and demands suitable preservation policies. In turn, these policies depend on the support of research actions, encompassing a profound characterisation of the composition of materials. The present paper deals with the issue of finishing mortars used on earthen buildings in a territory of southeast Brazil that is historically linked with the first economic coffee cycle, i.e., the basin of the river Paraíba do Sul (Vale do Paraíba). The aim was to draw a picture of the different solutions adopted in the conception of such materials in terms of composition in different historical moments. The research was based on the assembly of a bank of samples of mortars of earthen buildings and their physical and chemical characterization by thermal analysis (TGA and DSC), FTIR and MEV. The results obtained show that, in the majority of cases, the earlier mortar layers present on the building surfaces are most likely made of clayish earth without significant addition of any other material, such as lime or organic substances. Coarser texture than the typical local soils is preferred, a solution that were obtained either by searching less weathered soils or by correcting the soil with river sand. In two cases, a low percentage of lime (approximately 5 % in weight) was recorded: one single farmhouse, which is considered to be a specific exception yet not entirely explained, and a luxurious urban palace where the addition of lime is associated with the adoption of a more modern wattle-and-daub system (called tabique and totally uncommon in the area), possibly suggested by a Portuguese influence. Furthermore, the addition of lime was observed on all mortars used in the same premises on stone walls. Later layers overlapping the earlier ones denote the adoption of more industrialised raw materials, with increasing signs of lime, sometimes reaching the proportion of 25 % in weight, which clearly result in much more rigid final mortars, where in most cases disconnection pathologies could be identified.

### **Keywords**

Earthen Mortars; Brazil; Paraíba River Basin



## **Macroscopic high resolution techniques to the characterization of the mortars structures in the Sé-Cathedral's archaeological complex in Idanha-a-Velha (Portugal)**

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### **Abstract**

The medieval archaeological complex placed in the St Maria Sé-Catedral (Idanha-a-Velha, Portugal), preserves a first Christian structure that it is the most ancient evidence of a Christian community in the city at end of the 4<sup>th</sup> c.- beginning of the 5<sup>th</sup> c. Although, the urban centre reaches a special reputation in the middle of the 6<sup>th</sup> under the control of the Suebi Kingdom, when an Episcopal See was founded in this place and called Egitania. The medieval Church keeps the structure of a previous building, that is, it is divided into three naves with supports and capitals recycled from Roman buildings. After, it was used across the Islamic period too. Other Roman and late antique structures also integrate the site where the St Maria Sé-Catedral is placed. Some of them, such as buckets and masonry walls, have been related to the early Christian occupations. After we have identified the different chronological phases, we started a comparison of the existing mortars in different structures of this complex, carrying out, first, a visu study, and second, we following a complete macroscopic characterization based on the use of high definition images and on the chromatographic analysis of cross sections. In addition, some of the samples collected -IV1 to IV8- were selected to conduct an optical microscopy analysis. Both techniques have allowed us to establish a granulometric typology according to the Raymond Classification, as well as to reach a differentiation between mixtures with calcareous and clayey component. Results obtained have been differentiated between lime mortars mostly with siliceous aggregates, quartz, micas and feldspars. The quality of mortars varies according to the factory. For example, mortars IV6 and IV8 correspond to plasters of clays. A certain correlation has been identified between mortars and chronologies, being more homogeneous and consistent those dated in the late antique period. These aspects have been proven with the use of software tools such as JMicrivation, Micam or ImageJ, biomedicine programs that facilitate the classification of mortars and the identification of their components.

### **Keywords**

Microscopy; Software; Chromatography; Idanha-A-Velha; Mortars





## **Topic 2**

Use of nanotechnology for high performance mortars

**Chaired by**

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## **Enhancing clay mortars' properties**

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### **Abstract**

In an effort to find ways to enhance the properties of clay-based materials in an efficient and compatible way, this study examines the effect of metakaolin and nanoparticles on the properties of clay mortars. Metakaolin is a known hydraulic binder and it has been widely used as a pozzolanic material, mainly as a replacement of cement in mortars and concretes. The clay binder, which is rich in lime by nature, was partially replaced by 10 and 20 % w/w of metakaolin, while nanoparticles were also used as to act synergistically on the increase of the physical and mechanical properties of the mortars. The nanoparticles used as additives were a combination of nano-silica and nano-alumina in percentage of 1 and 0.5 % w/w respectively. All specimens were cured in air conditions at 25 °C, while the experiments were conducted after a curing period of 28 days. The fresh and hardened properties of these mixtures are reported, including workability, compressive and flexural strength, capillary absorption, volume stability and porosity, while all the samples were examined stereoscopically and through Scanning Electron Microscopy (SEM). The results indicate the positive effect in terms of microstructure for the case of 10 % metakaolin replacement with a 13.72 % improvement in volume loss, while the use of nanoparticles also contributed to the stabilization of clay mortars. The slow hardening effect of metakaolin on cement mortars was also reported in the case of clay mortars as far as the mechanical test results are concerned. The compatibility of the materials mixed was on the benefit of their properties.

### **Keywords**

Clay Mortars; Metakaolin; Physical; Mechanical Properties; Microstructure; Nanoparticles

## **Active photocatalytic-superhydrophobic coating with TiO<sub>2</sub>-ZnO nano-heterostructures for lime mortars**

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### **Abstract**

Active coatings to be applied onto hardened surfaces of lime rendering and masonry mortars and stones of the Built Heritage were developed. Nano-heterostructures of TiO<sub>2</sub>-ZnO (50:50 and 10:90) were obtained by Flame Spray Pyrolysis as photocatalytic agents with expanded sensitivity towards solar light, instead of the restricted UV dependence of the pure TiO<sub>2</sub> or ZnO. A superhydrophobic medium was simultaneously prepared and photocatalytic nanoparticles were added to obtain the coatings. The active products were expected to prevent the water absorption of the substrates and the subsequent degradation effects as well as to allow the stones and mortars to act as self-cleaning materials, reducing the dirt deposition and the biological colonization. Dispersions were applied onto the surface of lime mortars and siliceous stone. Measurements of the photocatalytic oxidation activity of the coatings were carried out by means of the NO degradation, showing a very good efficiency of the nanoparticles even at long term tests (values of NO oxidation of ca. 35%). Water contact angle (WCA) assessment evidenced a strong hydrophobization of the treated surfaces, with WCA values higher than 140°. The results proved the synergistic effect of these coatings with respect to the durability of the treated substrates, giving rise to a promising way of preventive conservation for building materials of the Cultural Heritage.

### **Keywords**

Lime Mortars; Nanoparticles; Photocatalytic Activity; Coatings

### **Acknowledgments**

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## **Evaluation of SiO<sub>2</sub> nanoparticles as additive for lime mortars: changes in the microstructure and mechanical properties**

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### **Abstract**

Lime has been traditionally used as binder in mortars. In those, the use of aggregates and additives, such as amorphous silica, was common in order to harden under humid conditions, thereby also increasing its strength and behaviour. In this context, the lack of volcanic materials which were commonly used in the manufacture of these historical mortars, forced the use of ceramics as an incorporation. Historic bricks have shown greater reactivity than the modern ones since they contained clay minerals which were calcined at low temperatures. As alternative, in recent years, nanoparticles had been considered as a potential addition to boost the reactions in calcium hydroxide. Amongst all nanomaterials, silica nanoparticles (SiO<sub>2</sub> NPs) have shown to be a promising additive for mortars for application in historical buildings of cultural heritage. The enhanced performance of the SiO<sub>2</sub> nanoparticles is mainly based on their filling action and/or their chemical reaction with the Ca(OH)<sub>2</sub> to form a gel of calcium-silicate hydrate phases (C-S-H).

In this research, effects induced by the addition of a commercial nanosilica to aerial lime mortars containing different aggregates (standard siliceous sand and crushed and grinded Roman ceramic) were studied. Specifically, the microstructural modifications produced by the presence of nanoparticles into the lime-based mortars were studied related to the mechanical and hydric properties.

Overall, the incorporation of nanoparticles modified the micro and mesopore structure for the different lime-based mortars. Thus, the use of SiO<sub>2</sub> nanoparticles, as additives for mortars, induced changes in the porosity because of the formation of C-H-S compounds. However, contrary to expectations, adding amorphous nanosilica into lime mortars with siliceous sand provoked a sharp decrease in the strength. These results are attributed to the microstructural consequences of nanoparticles addition on lime mortars due to the carbonation shrinkage and/or the cracks produced in connection with the dehydration and reactions of the hydrous silica products.

### **Keywords**

Lime Mortars; Pozzolan Lime Mortars; Nanoparticles; Nanosilica; Pozzolan

## **Evaluation of the influence of nano-SiO<sub>2</sub> and nano-Al<sub>2</sub>O<sub>3</sub> in physico-mechanical properties and microstructure of calcareous clay**

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### **Abstract**

From the beginning of construction history, clay had a starring role in traditional building systems. Nowadays, it is still used as an intervention material for the conservation and restoration of historic constructions. As a binder, however, clay has a certain disadvantage: i.e., its low resistance to humidity. The last decades, nanoparticles are studied for their influence in building materials. They have been used to improve certain properties of cement pastes, lime based systems and pozzolanic traditional systems. Up to now, few studies have been published in relation to the nano-modification of clay systems. This research work is an effort to evaluate the influence of nanosilica (NS) and nanoalumina (NA) in calcareous clay binder.

NS and NA were incorporated in calcareous clay in proportions 1.5 % and 3.0 % by mass of binder. Clay pastes were produced (shaped as prisms 25x25x50 mm and 25x25x100mm in size). The reference system was prepared with standard consistency (EN 450-1, Vicat 15±1) and a superplasticizer was added in nano-modified systems. Physico-mechanical properties were studied in order to determine whether nanoparticles influence positively the structure of clay. Compressive strength and open porosity were recorded up to 365 days. Capillarity was determined at 90 and 365 days, and volume changes were recorded up to 45 days. Specimens were observed with SEM-EDS at 120 days, in order to verify the influence in microstructure.

Calcareous clay with NS 1.5 wt % yielded impressive results. The system withstood capillarity tests, both at 90 and 365 days. Samples did not collapse upon impregnation of water by capillarity. Physico-mechanical properties were favored. Compressive strength was improved especially at 90 days (increase 35.41 %), but also was constantly improved up to 365 days. On the other hand, NA benefited the systems' early age mechanical properties only up to 90 days and did not stabilize the structure during capillarity test. A higher nanoparticle content (3 wt %) did not benefit the binder. More specifically, this percentage hampered later age compressive strength and open porosity. Overall, the incorporation of NS in calcareous clay could lead to promising results for further scientific investigation.

### **Keywords**

Calcareous Clay; Nanosilica; Nanoalumina



## **Studies of the mechanical properties of lime mortars treated with alkaline earth hydroxide nanoparticles**

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### **Abstract**

In the last decades a special attention has been devoted to the development of nanomaterials for conservation and restoration of Cultural heritage. Nanomaterials exhibit distinct properties when compared to their bulk analogues and have been seen as a good alternative of compatible materials for long-term preservation of monuments and artefacts [1-3]. Our studies have been focused on the elaboration and optimization of preparative strategies of micro- and nanolimes and on the study of their efficiency in the consolidation of lime mortars and lime paintings [4, 5]. In this communication we report the synthesis approaches to prepare calcium and magnesium hydroxides nanoparticles,  $\text{Ca}(\text{OH})_2$  and  $\text{Mg}(\text{OH})_2$ , with chemical precipitation from aqueous solutions. The synthesis is carried out at temperatures range Troom - 90 °C from equal volumes of NaOH and  $\text{CaCl}_2$  or  $\text{MgCl}_2$  solutions with different concentration. We also report our attempt to achieve a good particles morphology by the addition of surfactants. The possibility of the application of their alcohol dispersions in cultural heritage restoration for consolidation of wall paintings on lime mortar renders is anticipated. We discuss the laboratory tests conducted to assess the efficiency of the nanolimes on lime mortar specimens. Furthermore, we report out preliminary studies of the compatibility of the nanoconsolidants with inorganic pigments which have been used in the preparation of the paint layer of wall paintings.

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### **Keywords**

Nanolime; Nanoconsolidants; Magnesium Hydroxide; Calcium Hydroxide

## **Synthesis of nanolime in sugary solutions**

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### **Abstract**

This study explored the viability of synthesizing particles of nanolime at room temperature by through the formation of complexes with dissolved sugars. The size of the nanoparticles will be determined by TEM analysis followed by image analyzer software (Digimizer).

Nanoparticles were synthesized by dissolving  $\text{CaCl}_2$  water solutions in 5-10 % sucrose solution. Samples were stirring at ambient temperature for 4 h or 24 h.

The percentage yield, observed to vary with synthesis conditions, was calculated with thermogravimetry. The DTA/TG curves were in temperature range of 20–1000 °C using an open aluminum crucible. Four distinct weight loss zones could be identified on the thermograms: 25 °C to 120 °C (elimination of moisture); 120 °C to 330 °C (amorphous calcium carbonate dehydration); 345 °C to 420 °C (calcium hydroxide dehydroxylation); and 430 °C to 800 °C (calcium carbonate decarbonation). The sugary media also favored the formation of amorphous calcium carbonate. Different heating rates (5, 10, 15 and 20 °C/min) were used to determine sample size to compare with TEM analysis were hexagonal nanoparticles synthesized ranged in size from 600 nm to 25 nm.

### **Keywords**

Nanolime; TEM; DTA

## **The use of nanoparticles to improve the performance of restoration mortars**

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### **Abstract**

Restoration mortars designates a group of products used to repair masonry. They can be used to rebuild damaged or missing parts of buildings, monuments or sculptures, as well as to make replicas of architectonic or sculptural elements of high cultural value that will be placed in buildings or museums to ensure their conservation.

The goal of this collaborative project is to study the effects of nanoparticles on lime mortar characteristics and performance. As a starting point we used a commercial lime mortar, Artopierre TM (Parexlanko), with two different kinds of studied nanoparticles, SiO<sub>2</sub> and TiO<sub>2</sub> (Sigma-Aldrich). The mineralogy of the nanoparticles, as determined by X-Ray diffraction, is amorphous silica for SiO<sub>2</sub> and a mixture of rutile and anatase for TiO<sub>2</sub>.

For each of the nanoparticles, different quantities were added to the Artopierre mortar. In the first stage, three concentrations were tested; two for SiO<sub>2</sub> (1 % and 2.5 %) and one for TiO<sub>2</sub> (1 %). Different properties were measured in the 28 day old mortar samples:

- Microstructure and hydric properties. Porosity and pore size distribution by MIP, water absorption by drop test and microtube test
- Pozzolanic reaction with nanoparticles SiO<sub>2</sub>, determined by TGA and FTIR
- Mechanical properties, flexural and compression strength
- Durability verified by salt crystallization and freeze thaw weathering tests

After this first set of samples we decided to continue the test with only the SiO<sub>2</sub> nanoparticles, as the titanium particles did not have any significant effect on the mortars. In the second set of samples we tested mortars with different quantities of SiO<sub>2</sub> nanoparticles: 1, 2, 3, 5 and 7 %. The samples were tested after four months in the laboratory to allow for carbonation. Using a broader range of concentrations allows us to better interpret the role nanoparticles play in changing the physical and chemical properties of mortar.

In this second set of samples several properties were observed. Some of them are similar to the previous set: porosity, durability, and water absorption. Complementary properties were also measured: vapor absorption, wave propagation velocities.

Conclusions have not yet been completely established, but it seems that the mechanical properties of the modified mortars are affected significantly by the procedure of mixing in nanoparticles during mortar preparation. Further studies are still necessary, mainly using “laboratory made” mortars that allow for greater control over the binder and aggregate compositions. Also, another nanoparticle composition will be tested – calcium carbonate.

### **Keywords**

Repair Mortar; Admixture; Additive; Nanoparticle; Microstructure; Durability

## **Study of the role of different nanoparticles in lime pastes**

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### **Abstract**

The incorporation of nano-silica (NS) and nano-alumina (NA) in building materials has been extensively studied the last decades. The utilization of such nanoparticles in cement and traditional binders has brought up certain advantages and disadvantages. The main benefit seems to be the contribution of nanoparticles to mechanical properties especially at early ages and to microstructure. Certain drawbacks are related to water demand increase and effective dispersion. The incorporation of NS and NA in different hydraulic pastes has shown the possible connection between the type of nanoparticle and the chemical content of the binding system. On the other hand, nano-lime (NC) has been connected to healing properties in cement-lime mortars and porosity reduction in cement pastes.

Lime is a critical and basic binder that is used along with other binders or alone for centuries. The evaluation of the behavior of different nanoparticles in aerial lime could result to useful conclusions, concerning the applicability of nanoparticles in lime and the changes that can cause to its properties. For that reason, lime pastes with NS, NA, and NC and combination of the nanoparticles were prepared and tested. More specific, nanoparticles were dispersed in water through ultrasonication and the colloidal suspensions were incorporated in lime pastes. Superplasticizer was used to control the quantity of water. Compressive strength (EN 450-1) and porosity evolution (RILEM CPC11.3) of (25x25x50)mm specimens, shrinkage and capillarity (EN1015-18) of (25x25x100)mm specimens, showed the different influence of each nanoparticle. The carbonation of lime pastes was recorded by DTA-TGA and demonstrated the more benefited system; nanoparticles played a key role in carbonation kinetics. Mechanical properties were connected to the microstructure, displayed by SEM images. NS incorporation lead to almost doubled compressive strength values at 180 days. NC is believed to contribute in microstructure by boosting portlandite flourishing and therefore to calcite formation. NA worked as filler that did not benefited significantly the system at early age. Nanoparticles lead to differentiations in lime pastes properties. The kind of the nanoparticle addition behaves differently. If the advantages of each combination will be taken into consideration and after further study of the durability, they could be used for different required applications.

### **Keywords**

Aerial Lime; Nanosilica; Nanoalumina; Nanolime



## **Topic 3**

Gypsum-based plasters and mortars in historical constructions

**Chaired by**

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## **Stucco marble in the Portuguese architecture: first insights in mineralogical, physical and mechanical properties**

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### **Abstract**

Stucco-marble is one of the most complex man-made decorative techniques. It has its origin in the Italian scagliola, an inlay work of coloured stucco pastes and was extensively used in the baroque period in central and southern Europe to imitate marble stone, being often referred to as “Baroque artificial marble”. According to the literature, the materials used consisted mainly of a mixture of calcined gypsum, pigments and water with animal glue. The glue had the function of retarding the setting of gypsum and enhancing its hardness, while keeping it easier to polish.

The magnitude of the use of stucco-marble in the Portuguese architecture is still not known, partly due to the difficulty in distinguishing this material from real stone and from stucco-lustro, another decorative technique used to imitate stone materials, based on lime plaster and painting.

The correct identification of stucco-marble and the knowledge of the original materials used to perform it are essential for the definition of adequate conservation and restoration procedures, based on the principles of authenticity, minimum intervention and use of compatible materials and constitute the main objectives of this research work. Several examples of the use of stucco-marble in buildings from the 18<sup>th</sup> to the 20<sup>th</sup> century have been identified so far and samples were collected. A multi-analytical study comprehending the determination of the mineralogical composition using X-ray diffraction (XRD), optical microscopy observations of fractured and polished samples, capillary absorption and dynamic modulus of elasticity was performed.

The results showed that gypsum (calcium sulphate dihydrate) is the main constituent with the exception of one sample, where only calcite (calcium carbonate) was detected. Anhydrite was also found in most part of the samples though in less quantity than gypsum. Similar results had already been observed in previous studies of two samples that intended to simulate stone and it was concluded that anhydrite, obtained from gypsum calcination at higher temperatures, was intentionally used in the binder to produce materials with improved physical and mechanical performance.

In this paper the characterisation results of a set of samples of stucco-marble belonging to several case studies are presented and discussed. A relationship between composition, techniques of application and the intended performance of the materials is established.

### **Keywords**

Stucco-Marble; Gypsum; Anhydrite; Characterisation; Heritage Surface Coatings

## **Detailed studies of gypsum renders and plasters from the Ishrat Khane Mausoleum in Samarkand, Uzbekistan**

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### **Abstract**

The paper focuses on detailed investigations of gypsum plasters and stuccos from the Ishrat Khana mausoleum in Samarkand.

The Ishrat Khana mausoleum is constructed in 1464, damaged by earth quakes and is, today, a ruined protected monument. Badly damaged by two earthquakes and then left to decompose over more than one hundred years, the Ishrat Khana mausoleum was investigated in detail to create an appropriate conservation concept for the materials and decorating surfaces. The historic mortars, renders, plasters and stucco applications are analysed in detail by using polarizing microscopy of thin sections, SEM/EDX, DSC/DTG and chemical analyses. All original materials contain gypsum as a binder and aggregates of marl (quartz, clay minerals, feldspar, mica and calcite). Different binder/aggregate ratios enable a varied application of this building material in Ishrat Khana. This type of historic mortar is called ganch in Central Asia.

### **Keywords**

Gypsum; Plaster; Marl; Samarkand; Uzbekistan



## **Historic gypsum mortars from Emilia Romagna (Italy). Mineralogical and petrographic analysis**

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### **Abstract**

In the northern Apennines, particularly in Emilia Romagna region, there are large outcrops of evaporitic rocks both of Triassic and Messinian age. It was therefore natural to use gypsum both in the vernacular architecture and, as regards Bologna, in the monumental architecture. In the upper valley of the river Secchia (province of Reggio Emilia) the Triassic gypsum outcrops constitute a high and long white cliff that strongly characterizes the territory. Sologno (Villa Minozzo, Reggio Emilia) and all the surrounding villages until fifty years ago had a unique pink hue due to the use as "lime" of the local burned gypsum. In fact, the poverty of raw materials and the difficulties of transport in these mountain areas stimulated the ingenuity of the inhabitants who learned to extract the gypsum that, burned in artisan furnaces (fornelle) were able to provide a "powder" to use as binder of the stones in the buildings, in the interior flooring and as interior and exterior finishing plaster of all their houses.

In the hilly area of the provinces of Bologna and Ravenna there is the Messinian Gessoso Solfifera Formation (the so-called Vena del Gesso), forming characteristic rocky crests with a jagged profile with respect to the surrounding clayey hills. In this area the gypsum has been exploited for many centuries, both for the production of binder for diverse uses as mortar for joints, render (both internal and external) and for decorative purposes: stucco, stucco marble ("Scagliola") in diverse architectural elements as statues, altar frontals, capitals, etc, in Renaissance and Baroque churches; and, in the variety with large crystals (selenite), as a dimension stone. Bologna is full of evidence of this use (the base of many towers, the first circle of walls, cornerstones, architectural elements). Two kind of gypsum mortars from historic constructions were sampled and tested. Several XRD, MOP and EDS-SEM tests were carried out from samples of external renders and structural masonry mortars. Gradually in the last twenty years, thanks to scientific studies in several European countries (France, Germany, Italy, Spain, Cyprus), the knowledge of this material so present in our architectural heritage has deepened. At the same time, the European Union promotes policies in which the Cultural Heritage should form part of the core of values that all Europeans share. In this scenario gypsum is presented as a material of high interest for its study and recovery.

### **Keywords**

Gypsum Mortar; Petrographical Analysis; Emilia Romagna

## **The use of stucco-marble to restore veined polished limestone. The case of the pavement in the major sacristy of the Cathedral of Seville**

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### **Abstract**

This paper shows the results of the intervention on the floor of the major sacristy of the Cathedral of Seville. The theoretical proposal for this restoration was conceived and directed by the heritage restoration company Artyco SL, from Spain, and the execution was carried out by the restorers Pepa Parra Granell and Antonio González Portillo. The pavement dates from the beginning of the 19<sup>th</sup> century and was made of white and black tiles of marble, as well as of red tiles of limestone which can be polished.

This difference between the typology of materials was translated into uneven resistance to wear, leading to a faster decay of limestone. The result is now inappropriate, both from the aesthetical and the security of the users point of view. In fact, the main reason to carry out this restoration was to avoid people stumbling on the largest holes that exist in the tiles of limestone, subjected to daily pedestrian trafficking in one of the most visited monuments in Spain. Due to the abrasion caused by the large influx of people, it was not possible to use a pictorial technique on a previous plain stucco, being necessary to reintegrate the lost volume and at the same time preserve the veined morphology and colours of the limestone, keeping those despite the abrasion actions through time.

It was decided to use traditional technique of stucco-marble, also known as “scagliola”, since with it a marble-like finish of a great aesthetic quality could be achieved. At the same time, depending on the type of plaster chosen, we could obtain surfaces with a great resistance to abrasion, similar to the stone in this sense.

One year after the intervention was completed, the reintegrations maintain quite a remarkable appearance, conserving a very similar brightness to that of the tiles of limestone which had been reintegrated, in spite of the abrasion derived from continued pedestrian trafficking.

### **Keywords**

Stucco-Marble; Scagliola; Alpha Plaster; Stone Floors Restoration; Compatibility

## **Thermal monitoring of a traditional gypsum oven in Ribera d'Ondara (Lleida) and simulation of the calcination process**

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### **Abstract**

The durability of the heritage built with traditional gypsum mortars that has endured over the years indicates that this conglomerate is more resistant to inclement weather than today's industrial gypsum. Certainly, most of the buildings located in gypsum areas were built using this material, both in load-bearing walls and in slabs, vaults, flooring, interior and exterior cladding.

The reason for this broad range in the uses of traditional gypsum can be found in the fact that, unlike industrial gypsum, and due to the conditions in which it is calcinated (different temperatures), compounds are formed that give this conglomerate properties that are different from today's biphasic and monophasic products. The formation of these compounds provides substantially different benefits to those which we are used to find in current gypsum. There is an extensive bibliography that indicates the phases that occur during the calcination of the mineral gypsum (in environmental conditions and in autoclave) as well as the temperatures at which they are obtained. However, for the manufacture of traditional gypsum, a monomineral stone is not used, but the raw stone (Algez) contains other minerals such as clays, limestones, coal and even quartz.

In order to know the calcinations temperatures of traditional gypsum, in October 2018 a furnace was built in the quarry of Cementos Tigre (Lleida). It was monitored with 18 sensors of temperature distributed by zones inside the furnace. This work shows the thermal evolution of the furnace. Both the raw and calcinated stones are also analysed. The calcinated stones are studied separately according to the zone where they were located in the kiln, in order to establish a correlation between calcination temperatures and the obtained mineral phases. A computational study to simulate the distribution of temperature inside the furnace is also presented. The goal is to propose a way to "industrialise" the calcinations process to obtain a traditional gypsum, with all its advantages but with a more controlled and reproducible calcination.

### **Keywords**

Traditional Binder; Traditional Gypsum; Traditional Gypsum Calcination Temperatures; Built Heritage; Restoration

## **Study of properties of gypsum plasters of Araripe's pole for application in restoration mortars**

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### **Abstract**

Plaster is the earliest binder known. Findings have revealed that this material was already used to manufacture containers and plasters for decorative purposes around 9000 B.C.. Since then, it has been used extensively for indoor masonry coatings.

In order to obtain the gypsum plaster powder, it is necessary to make the proper gypsum mineral selection, to have a production control in relation to the type of furnace, calcination temperature, cooking time, type and concentration of additives, impurities, particle size, water/plaster, time of mixing, among others, that will influence the type and quality of the plaster.

Brazilian northeastern has an advantage in relation to the other regions of the world, because it presents abundance and quality of the gypsum raw material, especially in the Araripe's Pole, where gypsum plaster is produced in companies of different sizes and production control. There is a need for physical and mechanical characterization of these materials for application as gypsum-based restoration mortars. One of the most important requirements for historic masonry mortars is bonding, followed by mechanical strength. In this context, it is verified that the gypsum of the Araripe's Polo can be an excellent option for execution and repair of historical coatings.

In order to know in detail the material and to understand the relation between the form of production of the plasters and influence in the characteristics of these materials, the relationship between gypsum production control characterized by firm size and gypsum properties was evaluated.

The experimental program of the study was divided in two stages: study of characterization of the gypsum plaster powder and evaluation of the bond strength of the coatings executed in gypsum paste. Six samples of gypsum powder were collected from different producers in the region. In the first stage, the characterization study was carried out with anhydrous, fresh and hardened state by means of technological, chemical, physical and microstructural tests. In the second stage, bond strength tests were performed on gypsum paste coatings.

With the results, it is verified that, the plasters are of high quality and independent of the manufacturer, presents satisfactory performances and alike. This is explained by the characteristics of the raw material existing in the region, allowing its use in restoration mortars.

### **Keywords**

Gypsum Plaster; Plaster Properties; Gypsum Quality; Restoration Plaster

## **Clay and gypsum mortars used during antiquity in Cyprus**

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### **Abstract**

Mud and gypsum constitute the most ancient and at the same time the most friable mortars and plasters used in architecture in Cyprus as well as in other East Mediterranean countries during antiquity. When used as plasters they were mainly placed over mud structures due to their compatibility with these structures, whereas mud was diachronically used as the main binding material between stones and adobes. It is well known that mud plasters have a perfect adhesion with adobes due to the similarity between the raw materials used for their preparation. Gypsum can also be applied very easily above adobes, very often with the help of small timber posts inserted on adobe surfaces. The very simple methodology required for their preparation and the very limited use of fuel, made them perfect ecological materials. At the same time, the use of mud plaster over adobes lead to a very simple aesthetic result to the structures. In some cases the use of mud mortars of a different color from the color of the adobes led to an interesting result. This was not really, intentionally, as these structures were then plastered.

The research presented in this paper derived from a phd thesis as well as research programs financed by the Research Promotion Foundation of Cyprus. The results showed the close relationship between the raw materials used and the geology of the immediate environment of each settlement. On the other hand, the research showed a rather limited use of gypsum in the architecture of the island during the first periods of antiquity, a fact that remains without a solid explanation. Despite its limited use during antiquity, gypsum was wider used during medieval time as plaster and mortar as well as in the vernacular architecture of the last centuries especially as a plaster in the interior of the dwellings.

### **Keywords**

Gypsum; Mud; Adobes; Plasters; Mortars

## **Physical- mechanical comparison of the traditional gypsums from Albarracín and Pallars**

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### **Abstract**

The traditional gypsum of Albarracín is, probably, the only one currently on market in Spanish territory.

In the Pyrenees of Lleida, specifically in the districts of Pallars Jussá and Pallars Sobirá, the traditional constructions are executed mainly with traditional gypsum.

In 2016 in order to obtain a traditional gypsum, with properties similar to those of Albarracín, a traditional oven was made in the Pyrenees of Lleida, in Senterada (Pallars), obtaining about 500 Kg of plaster.

This present work is a comparative experimental study of two traditional gypsums (Albarracín and Pallars), on physical-mechanical level, using different water / plaster dosages and different kneading times.

The results show many similarities between both materials and demonstrated, once again, how the manufacturing factors can affect them.

### **Keywords**

Traditional Gypsum; Pallars; Albarracín; Traditional Kiln

## **Characterisation of Gypsum Renders in the Paris Region and Determination of the Traditional Fabrication Process**

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### **Abstract**

Gypsum renders are omnipresent in the Paris region. Used since the Middle Ages, they were widely employed in all building typologies including farms, stables, castles, churches, apartment buildings, Parisian's "Hôtels particuliers"...Gypsum renders can be very durable: some of them have endured hundreds of years. With the technological evolution that began in the mid-19<sup>th</sup> century, traditional materials and the plasterer's know-how disappeared. Currently, old buildings are renovated with gypsum-lime mortars, which is a type of render developed in the 1980s. This study aims to characterise the traditional gypsum renders in order to understand their traditional fabrication process. A large sampling campaign of renders covering the historical region of Paris and all building typologies from the 16th century to the early 20<sup>th</sup> century was conducted.

The cross sections of renders were first observed with the naked eye and then under the microscope to characterise the number, thickness, and granular typology of their layers. An X-ray diffraction and mercury intrusion porosimetry were performed on each render layer and an estimation of the proportion of each crystallised phase was performed using the Rietveld method. Calcite and quartz contents vary from building to building and even between two layers in a single render. On average, however, their content is low (80 % of the layers have less than 7 % of calcite and less than 2.5 % of quartz) and gypsum appears to be the main and often only component. In the quarries of the Paris region, gypsum naturally contains around 2 % (and up to 15 %) of calcite, depending on the quarry and stratum. Only calcined gypsum is used to make renders, especially for the most prestigious urban monuments. For this building typology, the renders are made up of several layers. These layers are applied successively and prepared with a single coarse plaster powder sieved at different grain sizes and mixed at specific water ratios. Traditional know-how, beginning with the gypsum powder fabrication and ending with the implementation techniques mastered by the plasterer, seems to create remarkably durable renders.

### **Keywords**

Gypsum Render; Traditional Know-How; Porosimetry

## **Figural Renaissance stucco in the Czech Republic – Technological and material characterisation**

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### **Abstract**

Figural stucco, three-dimensional figural artwork, is a particular art of stucco technique, in which sculptures are created in mortar instead of in stone. The importance of such a technique, rare during the Renaissance period yet not particularly exceptional, is remarkable for many reasons; the main level of its importance lies in the stucco technology, of which there is currently only superficial information.

This paper contributes to a closer understanding of this technique by providing a detailed study of several Renaissance figural stucco works, decorating the important Renaissance residences in the Czech Republic, that have been analyzed on a technological, structural and material level. The figural stucco works of art from Chateau Telč (UNESCO), Chateau Bučovice, Rondel Jindřichův Hradec, are all of high quality, carried out in all cases to order for the respective owners by artists invited probably from Italy or Central Europe. The detailed exploration into the stucco work was possible thanks to participation in the national project of the Czech Ministry of Culture that funds the research of Renaissance stucco in the country.

An overall complex artistic and historical survey has been carried out and incorporates archival research mapping the fate of the monuments since their creation. It includes the execution of the restoration survey and detailed material exploration, as well as the investigation into the structural features of the stucco work, which was carried out using advanced non-invasive techniques. On their basis, it was possible to reconstruct the technique and the material range of the materials used from the internal structures to the surface / painting layers of the work. Non-invasive techniques included the use of X-ray radiography, and videoscopy, with the stucco surface characteristics studied using RTI (Reflectance Transformation Imaging). Multispectral analysis based on the use of different spectral ranges of light complementing the visible spectrum was used for evaluation of surface properties. The chemical and mineralogical composition, microstructure and texture of collected samples of the stucco mortars were studied in detail by means of Polarizing, SEM microscopy and X-ray diffraction.

The results highlighted a wide range of techniques and technologies ranging from the layering of the stucco on the supporting structure and the lamination technique, through to the use of terracotta combined with stucco mortars. The knowledge about the materials and techniques is essential for increasing the understanding of this particular stucco technique. At the same time however, the obtained results provide suitable comparative material for studies into Renaissance figural stucco work not only within the territory of the Czech Republic, but also within a Central European context.

### **Keywords**

Renaissance; Figural Stucco; Czech Republic; Technology; Reconstruction





## **Topic 4**

**Functional mortars for the conservation of historic  
and modern cultural heritage structures**

**Chaired by**

**Ioannis Karatasios**

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## **Hydrophobized lime grouts prepared with microsilica and superplasticizers**

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### **Abstract**

This work reports the obtaining of lime-based grouts as repairing materials. Microsilica was added as pozzolanic additive to enhance the compressive strength of the hardened grouts. Sodium oleate, as water repellent admixture, and different superplasticizers were also incorporated to reduce the water absorption and to enhance the injectability of the grouts. Polycarboxylate ether (PCE), polynaphthalene sulfonate (PNS), melamine sulfonate (MMS) and polyacrylic acid (PA) were tested as plasticizing agents. Regarding the fluidity of the grouts, PCE was seen to improve the injectability, followed by PNS, MMS and PA. However, PCE addition was also accompanied by a severe delay in the setting time. The other three superplasticizers did not provoke significant delays in the hardening of the samples. The water contact angle underwent an increase pointing to an effective hydrophobization of the surface as a consequence of the water repellent admixture. The combination with PCE was the most effective in keeping the water repellency in comparison with the control sample (lime grout + oleate). MMS yielded high compressive strengths and durability of the mortars, in the face of freezing-thawing cycles, was enhanced.

### **Keywords**

Grout; Microsilica; Injectability; Water Repellent

### **Acknowledgments**

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## **Self-cleaning of previously healed historic mortars with multi-functional coatings**

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### **Abstract**

Aging and deterioration of mortars represent specific challenges in conservation of historic and modern cultural heritage structures. One of the solutions could be sought in surface protection and development of self-cleaning photocatalytically active materials. The use of these advanced materials could provide preventive and lasting surface protection from atmospheric pollution and microbiological corrosion. The main attention of our research was focused on the development and application of a new self-cleaning coating for previously healed historic and modern mortars. Aiming to achieve high compatibility, laboratory mortar models were prepared based on the results of historical mortars examination from two case studies, medieval Bac Fortress in Serbia and baroque Dornava Manor in Slovenia, both located in continental climate regions. The examination involved chemical-mineralogical, textural, microstructural and microbiological tests, which served to establish composition and production technology for mortar models and set aging procedures for artificial weathering and healing process. The components of the models of mortar were as follows: lime putty, micronized quicklime, sand and water (hot lime mix method). The used aggregate was based on quartz, calcite, mica, feldspar, chlorite and dolomite with the grain size within 63  $\mu\text{m}$  and 250  $\mu\text{m}$ . Comparative characterization of historic mortars and aged/healed models revealed that these two groups possess very similar composition, textural properties (total porosity 32.2 % of the historical samples compared to 38,6% of the aged samples) and patterns of materials deterioration (presence of soluble salts, microbiological growth, cracks due to the freezing/thawing cycles). After aging and healing performed with external healing agent (microbiological healing), the prepared models were treated with multi-functional photocatalytic material based on  $\text{TiO}_2$ -layer-double hydroxide (LDH) suspension, with proven self-cleaning efficiency. The LDH provided high compatibility with mortar samples, while intercalation of nano-sized  $\text{TiO}_2$  into the LDH ensured that the suspension is harmless for environment, treated surfaces and operator. After the suspension application by spray, the treated mortar models were examined regarding the water vapour permeability, colour change, photocatalytic activity and durability (water rinsing, exhaust gases, freezing/thawing). The obtained results showed good compatibility and good durability of the examined model mortar-coating system. This solution represents functional system which allows historical mortars and modern structures exposed to sun, rain, freezing and thawing to actively clean themselves from atmospheric pollutants and microbiological corrosion in the long-term.

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## **Use of natural zeolite aggregate in restoration lime renders**

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### **Abstract**

The increasing attention for restoration and conservation of historical construction and a more elevated environmental sensitivity (reduction of CO<sub>2</sub> emissions in atmosphere) has re-awakened the interest for lime-based binders. Lime mortars are not suitable for use in the moist environment because they lack hydraulic properties and have low frost resistance especially in non-carbonated state. The use of mortars with hydraulic properties is therefore often preferred for renovation purposes. Significant change of material characteristics of air lime mortars can be achieved by addition of pozzolanic admixtures or aggregates with pozzolanic properties. Porous pozzolan active aggregate can be used to prepare lightweight non-hydrophobic remedial mortars with a high ability to salt accumulation from masonry and enhanced water vapor permeability. These mortars facilitate easy moisture transport to the plaster surface. Natural zeolites have been used with limes already in the Roman times and its perspectives of effective use as a pozzolanic additive to lime or cement composites were studied by a number of authors. The use of zeolitic materials for the production of lightweight aggregates in concrete is already known. However, the application of natural zeolite as an aggregate in lime renders (suitable for restoration and conservation) has not been investigated yet. In this study, the utilization of natural zeolite as lightweight aggregate in lime renders and natural hydraulic lime renders has been investigated. Compressive and flexural strengths were tested at mortar age of 28, 56 and 90 days. Additionally, scanning electron microscopy was performed to investigate the microstructure of the renders, while the pore size distribution was analyzed with mercury intrusion porosimetry. The carbonation rate of the renders was determined by thermogravimetric method. The transport of liquid water in the studied materials was characterized by the water absorption coefficient, sorptivity and the coefficient of moisture conductivity. The durability of the prepared renders was monitored by frost-resistance tests and salt crystallization resistance tests. The results revealed improved strengths of renders with zeolite (air lime renders especially) despite the fact that the renders showed higher open porosity and water absorption than those with quartz aggregate. Increased porosity of renders resulted in improvement of their frost resistance and salt crystallization resistance. The analyzed renders with natural zeolite aggregate have a very good potential for practical applications because their properties are significantly enhanced, as compared with the common lime renders with quartz aggregates.

### **Keywords**

Natural Zeolite; Aggregate; Lime Render; Pozzolanic Effect; Durability

## **Addressing safety and durability requirements of architectural heritage by developing functional conservation mortars**

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### **Abstract**

The safety of historic buildings and the protection of people that works in or visits the monuments are two of the main requirements that define the framework for designing the interventions and selecting/developing the most suitable restoration mortars, especially when the buildings will be re-used. This work discusses the development of novel conservation mortars, reinforced at the nano-scale by the incorporation of functional carbon nanostructures. The incorporation of carbon nanostructures such as carbon nanotubes (CNTs) aims to enhance the crack-resistance of conservation mortars, increase their strength - especially in flexure-, increase the toughness of the mortars and thus reduce/limit the crack propagation and increase the service life of conservation interventions. The incorporation of carbon nanostructures creates a conductive network within the mass of mortars attributing to the mixture new functionalities, such as the ability to act as piezoresistive material. When the mortar is subjected to mechanical loadings the electrical resistance of the material is altered as well. Thus, the strain under static or dynamic conditions can be detected by measuring - continuous and remotely - the electrical properties of the nano-composite material.

The above technology was tested for the first time in lime-pozzolan-cement ternary conservation mixtures, by incorporating multiwall carbon nanotubes (MWCNTs) in three different amounts: 0.05, 0.1 and 0.2 % wt. The microstructural properties of the nano-composites were studied by MIP and SEM. Their mechanical properties under compressive and flexural loading conditions were investigated, after curing for 28 days. The electric resistance of the CNT-modified conservation mixtures was simultaneously recorded during the above-referred mechanical tests and a correlation between mechanical loading and electrical measurements was established.

The results indicate the beneficial role of nano-reinforcement on the mechanical performance of nano-composite mixtures when compared to the reference ones. The compressive and flexural strength increased up to 22 and 43 % respectively. The incorporation of CNTs modified the electrical performance of all the nano-composites investigated, so as to follow the applied stress. Overall, this work highlights a new perspective on the design of conservation mortars, giving emphasis on the new functionalities and advantages that nanotechnology can provide to cultural heritage conservation.

### **Keywords**

Cement-Lime-Pozzolan Mixtures; Carbon Nanotubes; Mechanical Properties; Piezoresistive Properties; Nanostructure

## **Autogenic vs. autonomic self-healing process in conservation mortars with crystalline admixture**

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### **Abstract**

Self-healing cement can serve as a promising solution for enhancing the efficiency of conservation interventions, extending service lifetime and safeguarding built heritage. In self-healing mortars, microcracks can be repaired without any external intervention. This could contribute greatly to the longevity of historic structures by prohibiting the degradation phenomena due to penetration of aqueous weathering solutions.

In this study, the autogenic self-healing capacity of natural hydraulic lime (NHL) mortars is contrasted with the autonomic self-healing capacity of the same mixture with the addition of a commercially available crystalline admixture (NHL-CA). Crystalline admixtures are used mainly for waterproofing applications when they are mixed in cement mortar or concrete. Therefore, their use as self-healing agents could be promising since these admixtures could promote the production of secondary healing products within cracks, contributing to the water tightness of the mortars. More specifically, controlled cracks were generated by three-point bending, in both reference mortar specimens and NHL mixtures modified with a commercially available crystalline admixture, after 15 days of curing. Thus, early-age damage was simulated. After cracking, the specimens were cured under water for another 28 days. The healing capacity of both mixtures was evaluated by water permeability and capillary absorption tests.

Moreover, the reduction of crack width due to self-healing was measured by means of optical microscopy, while the secondary healing phases produced inside the cracks were characterized and quantified by XRD, DTA/TG and SEM/EDS. It was found that secondary healing products in NHL mixtures were mainly composed by C-S-H and calcite phases whereas the addition of the crystalline admixture has mainly resulted in the formation of ettringite. In terms of performance, it is shown that the formation of ettringite crystals inside the micro-crack results in the reduction of water permeability and capillary absorption.

### **Keywords**

Self-Healing; Natural Hydraulic Lime; Crystalline Admixture; Water Permeability; Capillary Absorption

## Lime-based rendering mortars with photocatalytic and hydrophobic agents: assessment of the water repellency and biocide effect

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### Abstract

Different rendering mortars were prepared by mixing air lime and air lime-pozzolanic nanosilica with TiO<sub>2</sub> and sodium oleate as, respectively, photocatalytic and water repellent agents, added in bulk. The aim of the work was to design and obtain new rendering mortars with improved durability focusing in the reduction of the water absorption of these materials and in their self-cleaning and biocide effect. To achieve a better distribution of the TiO<sub>2</sub> particles, which was expected to enhance their efficiency, different dispersing agents were also incorporated to the fresh mixtures. Four diverse polycarboxylate ethers superplasticizers and a poly-naphthalene-sulfonate were tested. Workability and fluidity of the fresh rendering mortars were determined to guarantee the applicability of the final products. Water contact angle was monitored with the aim of assessing the hydrophobicity of the mortars lent by the water repeller. The biocide effect was studied by means of the culture of a strain of *Pseudomonas fluorescens*. The colonization of the mortars' surface was analyzed by determining the number of colonies forming units (CFU) after several days subjecting the samples to suitable T and RH conditions. At the same time, the surface of the mortars was irradiated with solar light to activate the photocatalyst. Results showed the efficiency of the sodium oleate in reducing the water uptake of the rendering mortars. Good compatibility between the water repellent agent, the pozzolanic additive and some of the polycarboxylate superplasticizers was observed. The presence of the photocatalyst was found to be very effective in preventing microbiological colonization.

### Keywords

Lime Superplasticizers; Titania; Biocide

### Acknowledgments

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## **Self-healing lime-based mortars using biological mechanisms and microvascular networks**

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### **Abstract**

Lime is considered one of the most predominant binders used in the production of hydraulic mortars. Its use can be identified in prehistoric, historic and contemporary structures. Although lime-based mortars can be relatively durable, and have survived over the centuries, the application of self-healing mechanisms could further enhance their longevity and the overall performance of the structures they belong to, reducing at the same time the need of maintenance.

This study aims to investigate the self-healing concept in hydraulic lime-based mixtures using microvascular networks for the efficient application of biological mechanisms, compared to a sodium silicate healing agent.

Natural hydraulic lime (NHL 3.5) constitutes the binder fraction, while for the aggregates fraction locally available fine aggregates of carbonatic nature are used. The binder to aggregate ratio is 1:3. Five mixtures have been designed and 40x40x160 mm specimens are produced in the laboratory, keeping the workability constant ( $165 \pm 5$  mm). Two of the mixtures are the reference materials; one comprises lime and aggregates, while in the other one, empty 3D printed microvascular networks are added, which are later used as the storage space for the self-healing components. A microvascular network is a system of small diameter channels, able to break when damage occurs and release the hosted components required for healing.

For the biological self-healing application, *Sporosarcina pasteurii* are added in the mixture, with the relevant nutrients stored in the micro-vascular networks. An additional mixture without bacteria but only nutrients is produced to evaluate their individual effect on the studied composite material. In order to compare the efficiency of the mechanism, an extra mixture of lime and aggregates is produced with sodium silicate stored in the micro-vascular networks. Damage induced by means of three-point bending loading in displacement control, takes place after 14 days of curing. All specimens are then stored for healing in an incubator until being re-tested to determine the level of restored performance. Microscopical observation of the specimens before and after each step of this study provides further information about the materials' microstructure. At the same time intervals, permeability measurements are performed for highlighting possible changes in the liquid transport of the materials under study.

The results of the present research may be valuable not only in the use of compatible, yet optimised, materials for conservation purposes, but also in modern construction industry.

### **Keywords**

Lime-Based Mortars; Self-Healing; Biological Mechanisms; Microvascular Networks; 3D Printing

## **Comparative evaluation of the morphological and rheological characteristics of nanolime dispersions for the consolidation of architectural monuments**

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### **Abstract**

This paper focuses on the comparative evaluation of different laboratory synthesized nanolime dispersions in terms of morphological and rheological characteristics and consolidation effectiveness.

Nanolime dispersions were produced following the main bottom-up (CaCl<sub>2</sub>/NaOH replacement reaction) and break-down methodologies. The modification of the surface tension between the dispersant and nano-particles was realized by the addition of Triton X-100 (non-ionic surfactant) and the replacement of the aqueous solution with a mixed polar solvent and a nano-bubbles enriched aqueous solvent.

The morphological, chemical and the mineralogical characterization of the nanoparticles produced were conducted by Scanning Electron Microscopy (SEM), Dynamic Light Scattering (DLS), BET surface area analysis, Fourier-Transform Infrared spectroscopy (FTIR) spectroscopy, and X-Rays Diffraction (XRD) analysis. The stability of the dispersions was determined with the use of UV-VIS spectroscopy.

The evaluation of the consolidation effectiveness was focused on the examination of the potential modification of microstructural and physical characteristics of a natural stone and a lime-based mortar.

The modification of the surface tension with the addition of the non-ionic surfactant and the replacement of the aqueous solvent led to the reduction of the nanoparticles size and the absence of conglomerates. Moreover, the incorporation of the oxygen nano-bubbles resulted the formation of hexagonal Ca(OH)<sub>2</sub> nano-particles and the improvement of the stability of the newly formed dispersions.

### **Keywords**

Nanolime; Conservation; Consolidation; Surface Tension

## **Photoactive Fe-TiO<sub>2</sub> lime Plasters for Building Protection**

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### **Abstract**

In outdoor conditions, buildings, monuments and any structure are affected by the environmental pollutants which induce alterations to the original color of the surfaces, causing a darker appearance. In recent years, new materials based on semiconductors, such as titanium dioxide (TiO<sub>2</sub>), have been applied as self-cleaning agents for lithic surfaces. In recent years, there has been a growing attempt to develop advanced photocatalytic systems that can be effective under the visible light. In this work, we have synthesized visible photoactive Fe-doped TiO<sub>2</sub> nanoparticles with the purpose to be used as additives to lime plasters, in order to enhance their carbonation procedure and the self-cleaning properties. More specifically, Fe-doped TiO<sub>2</sub> nanoparticles were synthesized through a simple sol-gel method containing various Fe concentrations (ranging from 0.05 up to 1.0 w/w %). Before the nanoparticles being incorporated into the plasters, they were studied through various analytical techniques, such as Infrared Spectroscopy (FTIR), X-Ray diffraction (XRD) and UV-Vis Spectroscopy (Diffuse Reflectance Spectra, DRS). For comparison purposes, undoped TiO<sub>2</sub> nanoparticles were also studied. The obtained results revealed the presence of both anatase and brookite crystalline forms of the Fe-doped TiO<sub>2</sub> nanoparticles, as well as the incorporation of the Fe<sup>3+</sup> ions into the TiO<sub>2</sub> crystalline lattice. Furthermore, the photocatalytic efficiency of Fe-doped TiO<sub>2</sub> nanoparticles was assessed through the decomposition of Methyl Orange (MO) under UV and solar irradiation. The Energy gap values obtained from the DRS fully justify the photocatalytic properties of the synthesized photocatalysts. This photocatalytic assessment of the Fe-doped TiO<sub>2</sub> nanoparticles indicated that the TiO<sub>2</sub> nanoparticles, doped with Fe in low concentrations (0.05 and 0.1 w/w %), have a positive effect on the photocatalytic degradation of MO, especially under the solar irradiation, compared to the undoped TiO<sub>2</sub> nanoparticles. All the synthesized Fe-doped TiO<sub>2</sub> nanoparticles were incorporated as additive into lime plasters and the evaluation of the carbonation process was assessed through the FTIR analysis over 40 days of curing. The results pointed out that the TiO<sub>2</sub> nanoparticles into the plasters played an important role in the carbonation process. The optimum carbonation rate was observed for the plasters containing doped TiO<sub>2</sub> nanoparticles with the low Fe concentration (0.05 and 0.1 w/w %).

Therefore, the addition of Fe-doped TiO<sub>2</sub> nanoparticles into the lime plasters enhances the carbonation procedure and simultaneously provides self-cleaning façades improving the aesthetic value of the constructions.

### **Keywords**

Plasters; Lime Mortars; Fe-Doped TiO<sub>2</sub>; Carbonation; Nano-Additives

## **SRG composite systems for strengthening masonry structures: from laboratory to field applications**

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### **Abstract**

The use of Fiber Reinforced Polymer/Grout (FRP/G) composites for the external strengthening and repair of masonry structures has met over the years an increasing consensus at both the academic and industrial levels.

One of the most promising systems consist of Ultra-High Tensile Strength Steel galvanized micro wires twisted in cords or ropes, assembled parallel to each other to form unidirectional fabrics. Steel textiles can be externally bonded to the substrate via wet lay-up, by using either epoxy resin/mineral mortar, obtaining a composite known as Steel Reinforced Polymer/Grout (SRP/G). The inorganic matrix offers several advantages such as a great reduction in material costs, a higher vapor permeability and resistance to high temperatures as well as a much higher compatibility without compromising bond with the substrate and overall performance. A number of research studies have been carried out on SRP and SRG systems in the last fifteen years. Fundamental mechanical properties, durability and shear bond performance on masonry substrates have been investigated through tests on small- and medium-scale specimens (panels, brickwork pillars and masonry arches). These studies have shown that SRP and SRG are an effective, durable and cost efficient solution for the externally bonded reinforcement of structures. This paper briefly introduces to the main properties and mechanical characteristics of steel fibers, presents few research results of tests performed on real scale structural members (walls, arches) and introduces some case studies on masonry structures demonstrating the different field applications of this new and effective strengthening solution.

### **Keywords**

Steel Reinforced Grout; Steel Fibers; Masonry; Hydraulic Lime Based Mortar; Strengthening; Seismic Retrofit.

## **Fibre reinforced mortars for cultural heritage protection**

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### **Abstract**

Addition of natural fibres into mortar mixtures is the most important and all over the world applied technology improving their performance. Historic fibrous materials are being replaced by modern synthetic ones, however, the natural fibres still may be competitive due to their ecological as well as economic qualities. The paper presents selected results of comprehensive investigations into behaviour of lime mortars modified with natural or synthetic fibres and fibrous particles. It summarizes technical data of fibre reinforced mixtures made of lime matrix, sand and natural fibres (goat's and horses' hairs) or fibrous particles (saw dust, husk) as well as polypropylene fibres. There are presented observations of the impact of fibres on the physical characteristics of resulted matured mortars, on the process of carbonation and the associated volume and porosity changes. Results of mechanical tests illustrate influence of fibres on the strength of mortars as well as toughness characteristics. The positive effect on the tension strength is documented on examples of application of such mortars as reinforcing layers on low strength masonries. Weathering effects were tested on small plaster uncovered areas exposed to one year outdoor environment. In the conclusions recommendations for fibre selection, mixture preparation and suitable applications are summarized.

### **Keywords**

Lime Mortar; Natural Fibres; Synthetic Fibres; Material Characteristics





## **Topic 5**

**Characterization of historic mortars and masonry structures. Sampling and test methods**





## **A map is worth a thousand pictures: The application of FTIR-mapping to the analysis of petrographic thin sections of historical and experimental mortar**

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### **Abstract**

A wide variety of analytical methods are available to researchers when choosing an investigative path for examining historical mortar. While every method has its advantages and disadvantages, this contribution proposes that imaging-based analytical techniques are the best methods for examining the compositional and micro-structural inhomogeneity frequently encountered in the layered structure of a mortar sample. Comprehensive, image-based analysis is best performed on high quality, polished (uncovered) petrographic thin sections, which allows for a sample to be examined successively by optical microscopy (OM), scanning electron microscopy combined with energy-dispersive X-ray spectroscopy (SEM-EDX) and Fourier transform infrared spectroscopy, here used in reflection mode to create spectral maps (FTIR). The preservation of spatial relationships and context between a mortar's micro-features is imperative to understanding the methods, materials and techniques employed an artisan in producing the mortar, as well as the alteration phenomena that have taken place since.

This approach is illustrated here by its application to a number of mortars from Roman antiquity as well as experimental mortars produced in the laboratory and in the field. All of these mortars consist of an air-lime binder mixed with a reactive aggregate - either ceramic or volcanic pozzolan. All mortars analysed, both historical and experimental, are from structural applications, such as opus caementicium or signinum. FTIR-mapping was found to be particularly useful in illustrating both binder matrix composition and alteration phenomena when the analysis rests on a foundation established by OM and SEM-EDX and is combined with digital image analysis. It can be used to confirm the identity of mortar constituents observed by the previous types of analysis. Further, it can be used to study the location, morphology and density of reactive silicates, degradation products and hydration products, the latter of which are nearly impossible to detect and/or conclusively identify by OM or SEM-EDX. This approach can thus be used to make a wide variety of correlative analyses all on the same sample preparation.

### **Keywords**

Mortar Analysis; FTIR-Mapping; Image Analysis; Experimental Mortar

## **Calcite or quartz powder as aggregate of Roman plasters (Lombardy, Italy)**

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### **Abstract**

Various kinds of mortar aggregate were mentioned by Vitruvius' *De Architectura*: sand (sand pits, river beds etc. - 2,4); marble powder (shining flakes, splinters - 7,6), pozzolana (from the region of Baia - 2,6) or potsherd (2,5). The real world of Roman artefacts of Lombardy shows a rather divergent situation: river sand, potsherd, crushed limestone, marble (calcite) powder, quartz powder. This study points out the finishing coat (marmoratum) of painted plasters: the aggregates are totally different, containing calcite powder (A - eastern Lombardy) or quartz powder (B - Milan). Optical microscopy on thin section and X-ray diffraction on powders were employed to perform the investigations.

A) Calcite crystals (Brescia site via Trieste; Civate Camuno multiple sites; Breno Sanctuary; Calvatone Domus Labirinto; Sirmione Grotte Catullo; 1<sup>st</sup>-2<sup>nd</sup> centuries CE).

- i) Poly-crystalline clasts (size 0.5 - 3.5 mm), sub-rounded; containing few calcite crystals often with polysynthetic twinning, each crystal ranges from 0,1 to 2 mm.
- ii) Single crystals (size up to 5 mm), clear, euhedral, angular according to the calcite rhombohedral cleavage and lacking of twinning.

Poly-crystalline clasts (i) derive from the crushing of fine to coarse grained marble. Single crystals (ii) derive from the crushing of very coarse grained calcite veins; these veins are diffused in all kinds of marbles and also in limestones or sandstones.

B) Quartz crystals (Milan - sites via Bossi, Broletto, Fontana and Meda; 1<sup>st</sup>-4<sup>th</sup> centuries CE).

- i) Single (size 0.1 - 0.2 mm), transparent, angular, irregular shape, low sphericity, unit extinction.
- ii) Poly-crystalline (size 0.1 - 2.2 mm), opaque, angular to rounded, regular shape, medium sphericity, undulose extinction.

Single crystals (i) seem to be originated by crushing of quartz pebbles coming from alpine gneisses and widely occurring in river beds (Ticino, Adda, Oglio) of Lombardy. Single quartz crystals, personally obtained from the crushing of these pebbles, are exactly transparent, angular and irregularly shaped, as detected in finishing coats.

Poly-crystalline quartz (ii) originates from river sand; this kind of aggregate was employed in render coats (harenatum) of the same Lombard sites.

To explain the different composition of aggregate of finishing coat of Roman painted plasters, two reflections are noticeable: quartz pebbles (milky-white), without mineralogical skill, may be easily mistaken for white marbles; quartz and calcite crystals, if obtained by crushing, show comparable features. Therefore, the use of quartz powder could be an unintentional surrogate of the calcite powder in a territory (Milan) lacking in marble outcrops.

### **Keywords**

Roman Plaster; Aggregate; Calcite; Quartz

## **Characterisation methodology for lime based materials – A case study of the Rajagopuram of Pundarikaksha temple in Tamil Nadu, India**

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### **Abstract**

The focus of this paper is to discuss the techniques for characterization of lime-based materials from historical monuments. The case study of the lime mortar samples collected from the grand entrance tower (raja gopuram) of the Pundarikaksha temple in the southern state of India, Tamil Nadu, is discussed. Most part of the monument is exposed masonry, and the historic mortar is observed to be identical throughout the structure. The historic lime binder is non-hydraulic in nature and the inert phases are mostly quartz and feldspar. Even though the aggregates are well graded, the mortar is highly porous. The critical parameters to be considered for a performance based design of the repair mortar, and the suitability of different characterisation techniques in determining the mineralogical composition, secondary phases and the porosity and pore size distribution of the mortar is discussed in detail.

### **Keywords**

Historic Lime Binder; Characterisation Scheme; Compatibility; Repair Mortar

## **Characterisation of concrete structures along the Reschen frontier, South Tyrol, Italy**

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### **Abstract**

In the late 1930s an extensive line of defence was created along the Reschen frontier, South Tyrol, Italy. The impressive concrete structures are still well preserved although being exposed to the harsh environment of the Alps for several decades. Despite the large number of studies dealing with the durability of concrete, the factors being crucial for the outstanding durability of historic concrete structures are still not completely understood. Furthermore, the amount of studies dealing with the mineralogical characteristics of the cement binder used at the beginning of the 20<sup>th</sup> century are rather sporadic. Accordingly, within this study specimens of concrete structures along the Reschen frontier will be investigated by petrographic and scanning electron microscopy as well as X-ray powder diffraction trying to point-out the factors being crucial for their durability. The knowledge on the microstructure and binder composition of historic concrete study is believed to be beneficial for both, the development of alternative binders and the planning of potential repair work in the future.

### **Keywords**

Historic Concrete; Durability; Binder; Microstructure; Petrographic Microscopy

## **Chemical, mineralogical and hydraulic characteristics of Roman mortars in Turkey**

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### **Abstract**

The binders of Roman mortars are composed of fine siliceous aggregates and carbonated lime ( $\text{CaCO}_3$ ) that give hydraulic character and high strength to the mortar. In this study, hydraulic properties of binders, pozzolanic activities of fine siliceous aggregates and content of binders of mortars used in some ancient Roman buildings located Xanthos (Antalya), Patara (Antalya) and Tlos (Muğla) in Southern Turkey were investigated. Hydraulic properties of binders were evaluated by determining the weight loss at the temperatures between 200 °- 600 °C due to the loss of the structurally-bound water of hydraulic products and weight loss at the temperatures over 600 °C due to carbon dioxide released during the decomposition of carbonated lime by TGA analysis. The pozzolanic activity of the fine aggregates was determined by measuring the differences in electrical conductivities before and after the addition of fine aggregates into saturated calcium hydroxide solution. The weight ratios of carbonated lime and fine siliceous aggregates were found by FTIR analysis developed by previous study. TGA analyses showed that  $\text{CO}_2/\text{H}_2\text{O}$  ratios of lime mortars were less than 10 indicating their hydraulic character. Pozzolanic activity measurements by electrical conductivity measurements were indicated that the fine aggregates in the binders have good pozzolanicity. The FTIR analysis results indicated that the carbonated lime and fine aggregates ratios of all mortars varied between 0.3 and 0.7 by weight. Analysis results showed that binders of Roman mortars are composed of pure lime and pozzolanic fine aggregates. Due to the use of pozzolanic fine siliceous aggregates, mortars are hydraulic.

### **Keywords**

Roman Mortar; Binder; Pozzolanicity; Siliceous Aggregates

## **Provenance study of raw materials used for lime making at Prague Castle during Medieval times**

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### **Abstract**

Number of historic mortars sampled from archaeological finds at Prague Castle area were compared in order to study the provenance of raw materials used for lime production in Medieval times. These mortar samples contained considerable amount various lime lumps and particles that can be related to the original raw material. It was possible to identify some of these particles as relicts of several limestone members from the Prague surroundings. Historic mortars were after sampling processed by several analytical techniques. Polarized light microscopy (PLM) and scanning electron microscopy with energy dispersive spectrometer (SEM-EDS) were used to determine petrographic characteristic and mineral composition of the present particles. According SEM-EDS it was possible to analyse these particles in more detail and to find out their semi-quantitative chemical composition. Based on these results, it was possible to compare these technological particles with collected limestone samples from historic localities (e.g. Prague Braník) or currently active quarries.

It turned out that the limestone raw material in historic mortars originated mostly from the Prague Basin, Palaeozoic sedimentation area located close Prague agglomeration. Several lithostratigraphic members out of which limestone is historically the most used one in building construction sector form this geological unit. The most represented limestone particles were possible to classify as Dvorce-Prokop, Zlíchov and Slivenec limestone or as members of Devonian Lochkov or Silurian Přídolí formation. Moreover, achieved degree of hydraulic properties of historic binders was comparable with hydraulicity of selected limestone members from the Prague Basin (e.g. Dvorce-Prokop or Zlíchov limestone).

### **Keywords**

Prague Castle; Historic Mortars; Building Material; Hydraulic Properties; Palaeozoic Limestone

## **Roman vs. medieval crushed brick lime mortars: A comparative study**

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### **Abstract**

Buildings from the Roman era are well-known for their exceptional durability. One reason for the well-preserved state of these buildings is the addition of pozzolans to the lime-based binder of the applied mortars. The result of the pozzolanic reaction between those components is the formation of calcium-silicate-hydrates, which contribute to the mortar's high durability. For a long time, it was believed that the knowledge of adding pozzolana to lime mortars widely got lost during medieval times.

According to recent research, mortars with pozzolanic additives like crushed bricks were also used in the Alpine region during medieval times. A detailed mineralogical characterization of these building materials has not yet been performed for objects situated in the Alps. Within the present study crushed brick lime mortars from cisterns in five medieval objects in South Tyrol and East Tyrol were examined and compared to samples from Roman times. The analyses were performed using petrographic microscopy, scanning electron microscopy equipped with X-ray spectroscopy, X-ray diffraction and simultaneous thermal analysis. First results of microscopic investigations on thin sections revealed that the medieval as well as the Roman binders are inhomogeneous concerning their colour. The colour differences are attributed to differences of the chemical composition of the binders. Diffuse, cloudy areas, which occur in most thin sections, are hints on the hydraulic character of the binder. On average the medieval mortars contain less brick fragments than the comparison samples from Roman times. The coarse brick components most likely behave similar to siliceous aggregates, while the fine brick dust seems to work as a pozzolanic component. Calcite, aragonite, lizardite and brucite as well as C-S-H phases and M-S-H phases are identified as components of the binder, indicating the formation of hydraulic phases depending on the type of raw material.

In summary, it can be stated that Roman and medieval mortars differ in their appearance and mineralogical composition. Reasons for that can be different raw materials, reactions in the binder during the centuries and a varying extent of alteration.

### **Keywords**

Pozzolanic; Crushed Bricks; Binder; Cocciopesto

## **Sampling cataloging methodology procedures for the conservation of historical colours in urban landscapes**

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### **Abstract**

Climate change and human activities are deterioration factors that are having a severe impact in the conservation of cultural heritage. At this point in time and in a European context, many vernacular buildings of historical centres are suffering the replacement of their original renders and colours with Portland cement and modern chemical binders. These new materials are completely opposed to the historic and original finishes. As consequence, the important heritage value of authenticity is disappearing.

Retaining this heritage value requires a fundamental framework for recording historic architectural finishes and a drive towards sustainable conservation practices to preserve and restore the historic landscape.

Therefore, the research initial question is: What do we know about the colour and texture of Neoclassical style buildings, that historical moment when the architects from the Royal Academy of Fine Arts of San Fernando built on this precepts? How can conservators tailor their materials and methods to its restoration?

The authors of this paper are sampling and analysing the colour palette of domestic neoclassical architecture (19<sup>th</sup> century) from wall to wood and metalwork in the neighborhood of Arenales, Las Palmas de Gran Canaria city.

An interdisciplinary approach will be taken followed by practical colour research and sampling methods.

The resulting map of colour will then be used to create a tool for decision-making by restorers when selecting materials and pigments for the restoration of the historic aesthetic during building conservation.

### **Keywords**

Coloured Surfaces; Historic Masonry; Sampling Methodology; Valuation of Building Finishes



## **Analytical and chromatic characterization of the interior walls finishes in the Batlló House of Gaudí in Barcelona. A surprising discovery**

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### **Abstract**

It is well known that Modernism implemented and promoted the decorative arts in architecture. In Barcelona, chief town of the avant-garde in the bend from XIX to XX centuries, the use of coloured lime stucco in the interior and exterior coatings of buildings was very prolific. This paper presents the outcome of our chromatic study of the interior walls of Gaudí's Casa Batlló, built in 1904 at the Pg. de Gràcia boulevard. We have inferred not only the materials and colours of finishing but also the execution technique, which was innovative and involved a high degree of difficulty.

The systematic study of the interiors of the Casa Batlló's noble floor through different analytical techniques has revealed the modernist finishes, serving as a basis for its restoration process. The main rooms have been inspected and documented, and micro-samples have been extracted 'in situ' to verify the presence of original coatings. The combination of several analytical techniques carried out in different laboratories, together with the deep knowledge of the artisan techniques of the stucco, the sgraffito and the ironing of the lime-stucco from master artisans, have led to outstanding results presented here in a novel way. The laboratories involved are the X-Ray Laboratory of the Institute of Environmental Assessment and Water Research (IDAEA) from CSIC, the Center of Conservation-Restoration of the Generalitat de Catalunya-CRBMC, the Arte-Lab Laboratory, and our own Laboratory of the Gabinet del Color.

The conclusion of this study is that in this Barcelona bourgeois house Gaudí exalted the creative modernist movement, designing for the walls and interior ceilings some imitations of its famous "trencadís" made with coloured lime stucco, performing then sgraffito to mark the "trencadís" and later finishing it with hot iron, and even with the presence in some rooms - as in the office of the owner and patron Mr. Güell - of extremely pure gold leaf. In this particular case, the genius of Gaudí was a challenge for the stucco masters and artisans to execute smooth finishes on surfaces, both walls and ceilings, very often with sinuous forms, where the chromatic treatment distinguished the rooms depending on its usage. The combination of the different analytical techniques, together with a global vision of the study object from different disciplines (analytical, architectural, restorative, historical-artistic and craftsmanship) have allowed to establish the execution technique of these extraordinary coloured stuccoes, unknown to date.

### **Keywords**

Gaudí; Coloured Lime Mortars; Chromatic Analysis; Historic Architecture; Sgraffito

## **Mineralogical characterization of historical cement-based mortar from Rupnik military fortification line**

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### **Abstract**

The paper will present results of historical cement-based mortar characterization taken from surface layer of military bunkers in Rupnik Line. The Rupnik Line was conceived as part of the fortified defence system protecting the newly formed Rapallo border between the Kingdom of Italy and the Kingdom of Yugoslavia. The border established by the treaty in Rapallo in 1921 became the focus of intense defence infrastructure works in the second part of the 30ies in the pre – WWII armament race. The Italians raced to protect the northern borders with the Vallo Alpino 1850 km fortification system stretching from Genova on the Ligurian sea to Reka on the Adriatic sea. The Yugoslavs responded with the 289 km long Rupnik fortified system protecting the entire border with the Italy and partly also the northern border with Austria. Due to complex and demanding geomorphological attributes of the land where the Rapallo border was defined and the fortifications constructed, construction works were demanding from points of view of logistics, technical and technological solutions, design features and spatial organisation. Considering the »zeitgeist« the fortifications of the Rapallo border represent today a magnificent and unique structure in space and offer a very specific historical case study, also from the materials point of view.

The aim of work that will be reported is a detailed investigation of historical cement-based mortar that was most probably used for the camouflage of the bunkers. It presents the rendering layer that was applied to the massive concrete walls. At first different types of damages visible with naked eye are addressed, followed by in-depth analyses. Results of petrographic examination, mineralogical analysis (XRD), and microstructural and chemical analysis (SEM-EDS) are presented. The first results confirmed that local carbonate sand was applied as an aggregate in the mortar. Binder mixture of cement and blast furnace slag was used in order to obtain special greenish colour of the mortar to ensure camouflage characteristics.

### **Keywords**

Historic Mortar Characterization; Cement-Based Mortar; Blast Furnace Slag; Microscopic Observation; Rupnik Military Fortification System

## **Algarve vernacular architecture facade ornaments: chemical, physical and mechanical characterization**

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### **Abstract**

In Algarve, southern region of Portugal, vernacular architecture presents extensive facade decoration, characterized by the presence of colored relief ornaments. These ornaments are mainly made with air lime mortars modelled by in situ carving, run out or cast moulding. In the region ancient ornament fragments were found in archaeological sites dated from the 11<sup>th</sup> to 13<sup>th</sup> century, indicating that craftsmen skills and ornamentation traditions are present at least since then. In 18<sup>th</sup> century some examples of erudite genesis, made in situ by direct modelling, are specifically known by their ornamental figures based on animal and human compositions. From the end of 19<sup>th</sup> century towards the first half of 20<sup>th</sup> century, a period of economic prosperity took place in Algarve region, along with the improvement of the moulding techniques, allowing the extensive use of casting moulds rather than in situ direct modelling.

This study aims to assess if these different types of modelling techniques required specific mortars compositions, namely by the use of specific binders, sand types and binder-sand ratios. For this propose 27 samples were selected among three historic periods, 15 samples from mid-18<sup>th</sup> century, six from early-20<sup>th</sup> century and six from mid-20<sup>th</sup> century, corresponding respectively to in situ carving, cast moulding and run out cast moulding. Mortars samples were chemically and mineralogically characterized through wet chemical analysis, X-ray diffraction analysis (XRD), thermogravimetric and differential thermal analysis (TGA-DTA) and scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM-EDS). Mortars were also physically and mechanically characterized comprising tests of water absorption by capillary action, drying curve (evaporation curve), open porosity, dynamic modulus of elasticity and compressive strength.

Results showed predominance of calcitic air lime along all samples with traces of other binders as, hydraulic lime, gypsum and portland cement in cast moulded ornaments from early-20<sup>th</sup> century, as well as significant amounts of dolomitic lime in run out cast moulded ornaments from mid-20<sup>th</sup> century. Different types of sand with distinct particle size distribution curves were also found, probably due to different extraction sites. In situ carved and run out moulded ornaments presented a multi-layer technique comprising distinct mortars while cast moulded ornaments showed a single mortar composition. Ornaments were colored through limewash pigmented layer applied over dried mortars surface. These results contribute to mitigate the present lack of scientific knowledge regarding mortars composition of Algarve vernacular architecture ornaments and provide information for its adequate restoration.

### **Keywords**

Algarve; Vernacular Architecture; Facade Ornaments; Moulding Techniques; Mortars Composition

## **Medieval mortar, stone and repair mortar of an abandoned Medieval Church, compatibility issues: example from Hungary**

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### **Abstract**

In order to understand the processes of deterioration of medieval masonry walls, it is important to study the composition and use of its mortar, plasters and stone. Furthermore, the structural behaviour of stones and renders, the construction history and the micro-environmental conditions also influence the preservation of these construction materials. Medieval architectural heritage structures are rare in Central Europe and most of them are in ruins. The current paper deals with one of these ruined churches, which is located near Budapest (Central Hungary) close to village Tök. In the first phase of the study a 3D model of the remaining walls was compiled based on photographic documentation. Mortar and stone were studied on site by using non-destructive test methods, such as portable moisture meter (GANN Hydromette UNI), Schmidt hammer and Durosokop. Small samples were taken for X-ray diffraction analyses and thin-sections. Selected stone and mortar samples were studied by Scanning Electron Microscopy. Three different stone types representing various lithologies were used in the walls. Varieties of mortars were also identified from Medieval lime-based mortars to portland cement containing latest repair mortar.

The moisture content measurements clearly indicate the diversity and complexity of moisture distributions within these often-heterogeneous walls, which have also had long histories of decay and conservation. Replacement stone patches and portland cement show consistently lower moisture conditions than the surrounding stones or lime renders. By analysing historic lime mortar information on the binder aggregate ratio, chemical composition of the mortar was obtained. This helps in the design of an appropriate repair mortar with properties and composition similar to the original mortar.

The study reveals that studying physical properties and composition of medieval mortars in combination with the detailed analyses of stone masonry can contribute to our understanding of the medieval stone heritage and allows designing a better repair mortar.

### **Keywords**

Lime Mortar; Masonry Wall; Decay Mapping; Non-Destructive Testing

## **Interpretation of scientific data derived from analytical techniques used in the characterization of Roman mortars**

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### **Abstract**

Mortars made by lime and lime and pozzolans were a standout amongst the most critical contribution of Romans to the architectural history. Roman lime mortars were exceptionally renowned for their quality and durability which generally relied upon the resources utilized in imperial territories. A comprehensive examination of this past technology leads us to better conservation practice and more sustainable construction.

To have a broader perspective this research was carried out in different Roman provinces. This paper aims at presenting the characteristics of Roman mortars from three archaeological sites in Spain (Mérida), Egypt (Alexandria) and Turkey (Labraunda). Characterisation was carried out through polarized optical microscopy (POM), X-ray diffraction (XRD), thermal analysis and differential scanning calorimetry (TGA-DSC), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), X-ray fluorescence (XRF), Fourier Transform Infrared (FTIR),  $\delta^{13}\text{C}$   $\delta^{18}\text{O}$  isotope analysis.

The central focus of the study is to understand the usefulness of the analytical methods which give clue about the construction technology, selection of raw materials according to the function and conservation state of mortars. We will also discuss to what extent we can find answers concerning aggregates, natural and inorganic additives and their effect on shrinkage, the manufacture of lime, the effects of environmental conditions and the interaction between adjacent building materials.

### **Keywords**

Roman Lime Mortar; TGA; FTIR; XRF; POM

## **Petrography of Historic Mortar Materials: Polarising Light Microscopy as a Method for Characterising Lime-Based Mortars**

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### **Abstract**

In studying historic, repair and restoration mortars comprised of lime-based material there is an analytical method that answers more questions than any other; transmitted (and reflected) polarised light microscopy (PLM). The present study is based on thin-section analysis of historic and restoration mortars made by the authors in Sweden and Scotland since the 1980's, previous research (i.e. Elsen 2005, Blaeuer & Kueng 2007, Middendorf et al 2005, Lindqvist & Sandström 2000) and petrographic atlases (Ingham 2010, MacKenzie, Adams 1994). It is illustrated by a selection of microphotographs to show the variety of questions that the method can give answers to, such as, in the study of:

- lime binder; source materials, hydraulicity, slaking methods, lime lumps, cracking tendencies
- aggregates; grading, mineralogy/lithology and texture
- additives; inorganic puzzolanic materials and organic materials
- paint layers; different binders, pigments, thicknesses
- pore structure; durability, vapour transport and capillarity
- craftsmanship; various application and working techniques

The aim of this paper is to illustrate the usefulness of Polarising Light Microscopy as a method for characterising lime-based mortars and to contribute with references on parameters that can be analysed.

### **Keywords**

Petrography; Thin Sections; Lime Mortar; Polarisation Light Microscopy; Optical Microscopy

## **Colors and grains: study on the composition and characteristics of mortars of the 18<sup>th</sup> and 19<sup>th</sup> centuries in São Luís, Maranhão – Brazil**

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### **Abstract**

The historic centers present traditional traces of constructions, materials, textures and architectural expressions. In the coastal areas of Brazil, there are still many historic buildings that were built with lime mortars. In the city of São Luís, in the state of Maranhão, in addition to the use of lime from beach shells, it was also possible to detect local sand and clay, which give an interesting variety of colors to the mortars and their functionalities. The materials, techniques and colors are prominent in São Luís, highlighting a diverse and peculiar architecture, representative of a specific historical-constructive context. This was possible through the in loco and laboratory observation, based on a historical-documentary, iconographic and bibliographic research, besides the metric and photographic registers. Therefore, awareness of the need to obtain accurate information on the materials has resulted in growing interest in the detailed study of lime-based mortars and their composition. The article presents and characterizes mortars used in ten historical constructions, from chemical and mineralogical tests, which allow the knowledge of the used materials and the observation of their unique characteristics. The aim is to contribute to the scientific and technological information of the materials used in São Luís in the 18<sup>th</sup> and 19<sup>th</sup> centuries, and thus to the preservation and rehabilitation of this heritage.

### **Keywords**

Mortars; Colors; Composition; Historic; São Luís-Brasil

## **DB-Heritage: A database of mortars composition and characteristics**

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### **Abstract**

Mortars are materials that are present in constructions from the antiquity to nowadays, being complex in terms of their constituents, incorporating traditions and techniques related to the local knowledge. Furthermore, mortar characteristics are also dependent of its role and use in a particular local and environment.

The knowledge of the mortars constituents, characteristics and behavior in their specific conditions is important in several contexts, namely in terms of materials history and for better definition of conservation strategies. That knowledge, obtained by many different researchers and practitioners of the area, should be easily available for all the people who need it, both for research and for direct use in the field.

This was the context of the DB-HERITAGE database project (<http://dbheritage.lnec.pt/>), which intends to build a repository for historical building materials, and a free-access IT-tool to collect the related data on this construction material, including from conservative interventions made on mortars applied on historic constructions. An overview of the database tools will be presented in this paper, exemplifying some of its outputs and potentialities related to historic mortars in different case studies in Portugal.

### **Keywords**

Building Materials; Building Heritage; Free ICT Tools; Web-Based Database



## **Characterization of Lime Mortar from a Tabique Pampango Wall Technique in the Philippines**

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### **Abstract**

The architectural style of old Philippines was heavily influenced by the Spanish Colonial Empire, being its former colony in South East Asia for almost 380 years. Manufacturing techniques used in old structures were introduced by the different religious orders that came in the Philippines together with the original Spanish contingent. One of the enduring legacies of this period are the massive and elegant church structures that still stand until today. This study focuses on the chemical composition of the wall making technique called Tabique Pampango that was obtained from the old convent of Loboc, Bohol in the Philippines. Tabique is Spanish for a wall partition and Pampango refers to the local people from a province (Pampanga) in the Philippines, where the technique originally was adapted and flourished. This procedure is also known internationally as “wattle and daub”. A screen of wood or bamboo is fixed on a lime mortar base and the whole surface is coated with a thick layer of mortar. The earliest documentary evidence of it being used in different Spanish Colonial Churches is as early as the 18<sup>th</sup> century. Despite its usefulness on wall constructions, it has been outlawed by the government in the late 19<sup>th</sup> century due to earthquake ordinances.

The lime mortar sample from Loboc was subjected to Energy Dispersive X-ray Fluorescence (EDXRF), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric techniques (TGA). Possible organic compounds were also analyzed using various wet chemical analyses such as Ninhydrin, Biuret and Xanthoproteic tests for albumin (eggs) and Molisch’s, Barfoed’s and Fehling’s tests for sugar additives. Results show that the lime mortar is dominated by CaO (39.30 %) and SiO<sub>2</sub> (3.51 %) from the EDXRF. This was supported by the XRD results which has intense peaks for the minerals, calcite and quartz. Furthermore, the FTIR and TGA confirms its calcitic nature. Possible organic compounds were not detected on the sample based on the different qualitative tests for albumin and sugar.

### **Keywords**

Tabique Pampango; Organic Additives; X-Ray Techniques; FTIR; TGA

## **Roman mortars of floor substrates and walls from Arroyo de la Dehesa de Velasco site: petrographic and mineralogical characterization**

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### **Abstract**

Samples were taken from the floors and walls at Arroyo de la Dehesa de Velasco Roman archaeological site (Burgo de Osma, Soria) for petrographic and mineralogical analysis. The site is related to a bath complex, possibly for public use, integrated into the settlement around the city of Uxama Argaela. Archaeological evidence provides a relative chronology for the first building structure related to baths in the early Roman Imperial Period. The coatings of stucco and their pictorial elements are noteworthy and the pictorial style dates the building to the 2<sup>nd</sup> century AD. Petrographic analysis is essential for the study of the composition of the building materials, including aggregate composition, size and shape and binder characterisation, and to establish the construction technique, as well as for the development of repair mortars for the reconstruction process. Eight samples of mortar-based materials were collected for the study, two from the floors and six from the walls. Most of the samples show some multi-layer technology except the most recent mortar on the floor. The oldest floor mortars show a multi-layer construction system whereas the most recent floor consists of a single layer. The multi-layer floor mortar is formed by four layers: an internal layer with moulds of hand palm-size river pebbles, a second internal layer (4 cm thick) with smaller rounded pebbles, a third layer (5-6 cm thick) of crushed rock fragments and finally a thin outermost lime layer (1 mm thick). Regarding wall mortars, the old mortar exhibits five layers under the pictorial surface. An external surface made by a thin (<1mm) whitish micritic matrix with angular calcite that is well attached to the next layer. The next whitish layer (2 cm thick) is constituted by silicate grains and some fragments of ceramics. The third reddish layer (2-2.5cm thick) exhibits significant amounts of pottery fragments. The fourth beige layer (1-2 cm thick) contains silicate grains and is poor in lime. The innermost fifth whitish layer (1-1.5 cm thick) is richer in lime with silicate grains. According to this study the Roman workers at the Dehesa de Arroyo de Velasco site used refined and standardised techniques for both floor and wall constructions.

### **Keywords**

Floors Lime Mortar; Walls Lime Mortar; Bath Complex; Roman Period

## **Hydraulic mortars at Caesarea: underwater and on-land pozzolanic reactions through chemical and mineralogical examinations of Herodian, Roman, and Byzantine constructions**

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### **Abstract**

During the rule of the Roman Empire in the Levant, Caesarea was a valuable commercial hub with large scale government, religious and recreation centers, and one of largest open sea harbors at the time. The construction projects at the ancient city of Caesarea reflect the tight relations between the Roman Empire and King Herod, showing the use of Roman concrete in under-water and on-land mortars. The Roman concrete in the underwater wave breaker and on-land harbor are especially unique since they contain pumiceous materials (pozzolana) that have been associated with the Gulf of Naples, Italy (Brandon et al. 2014).

Ancient hydraulic mortars undergo different hydraulic pathways depending on the original formulation, the extent of the hydraulic reaction, and the chemical environment in which the material evolved. Since many of the hydraulic products are amorphous and poorly crystalline, their identification is often problematic, and multi-analytical approaches are required for a proper characterization of the binders.

Here we show results from archaeological mortars and plasters during the Herodian, Roman, and Byzantine periods at Caesarea. We discuss the different information obtained by XRD, XRF and FTIR measurements, aimed at understanding the extent of the hydraulic reaction of mortars that contain pumiceous materials in underwater and on-land chemical environments.

The study shows that different local materials were used in Roman concrete, changing the recipe as described by Vitruvius, and the extent of pozzolanic reaction. Interestingly, the hydraulic mortars in the Herodian underwater wave breaker are similar in composition to the on-land harbor vaults, showing presence of pumiceous materials with varying amounts of local materials. However, the mortars of the on-land temple which is also associated with King Herod are based only on local materials. In addition, different materials were used at different periods, with gypsum, surprisingly, being a consistent during the Byzantine period in on-shore constructions. These observations contribute to our understanding of the technological choices involved in mortars production in this UNESCO site.

Brandon, Christopher J., Robert L. Hohlfelder, Marie D. Jackson, and John Peter Oleson. *Building for eternity: the history and technology of Roman concrete engineering in the sea*. Oxbow Books, 2014.

### **Keywords**

Caesarea; Pozzolanic Reaction; X-Ray Powder Diffraction; Fourier Transform Infrared Spectroscopy; X-Ray Fluorescence

## **Characterization of historical mortars from the Portuguese Citadel in Ksar Seghir (Morocco)**

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### **Abstract**

Ksar Seghir was a privileged place to control the Strait of Gibraltar throughout the centuries, being implanted in its southern bank, nowadays in Moroccan territory. Its prominence led to the construction of a unique circular wall and monumental doors during the Marinid dynasty in the late 13th century. The defensive system was later reformulated during the Portuguese occupation between 1458 and 1550, which sought a technical update, namely the introduction of new defensive devices adapted for artillery, that was gaining importance in the Mediterranean context.

After its abandonment by the Portuguese, the town remained unoccupied, being today an archaeological site. Since 2008, the Moroccan government has undertaken a program to value the site and its structures, namely through various conservation and restoration actions. From 2011 a team of Moroccan and Portuguese researchers began a project to deepen the study and restore Ksar Seghir's archaeological contexts, which led to the characterization of its structures, specifically the type of construction materials and techniques used. One of our main focuses has been the defensive system, for its impressiveness and historical richness. In this context, the work that is being carried out at Laboratory HERCULES, aims the material study of several mortar samples, collected from the Portuguese Citadel. The analytical methodology employed is an adaptation of standard analytical procedures used in historical mortars studies.

The results of textural, mineralogical and chemical characterization allow, among others, the identification of mortars binder and aggregates and also the determination of the binder: aggregates ratio. This provides useful information regarding the history and the construction techniques of the different spots according to its purpose. Moreover, the acquisition of data on the mortars textural, chemical and mineralogical features is essential to assist conservator-restorers in the correct choice and production of replacement and/or repair mortars that are compatible from a physical as well as chemical point of view with the original ones.

### **Keywords**

Ksar Seghir; Historical Mortars; Mineralogical and Chemical Characterization

## **16<sup>th</sup> century decorative elements in the Convento dos Capuchos (Serra de Sintra, Portugal)**

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### **Abstract**

The Convento dos Capuchos, founded in the 16<sup>th</sup> century by Don Alvaro de Castro and reformed in the 18<sup>th</sup> century, is one of the most emblematic monuments of the Parques de Sintra-Monte da Lua. Built in an isolated and inhospitable place, their occupation would favour a life austere dedicated to meditation. The structure of capuchin convent is characterized by an irregular succession of several of poor and small spaces that embraces the topography and granitic geology of the Serra de Sintra. Reopened to the public in 2001 the convent was in an advanced state of deterioration, particularly in terms of renders. In this way it was possible to identify, under the murals of the 18<sup>th</sup> century, the existence of a black coating that would cover the majority of indoor spaces. Under the intervention diagnostic-conservation project conducted by In-Situ company at the request of Parques de Sintra-Monte da Lua, a sample campaign was design in order to cover decorative elements such as mortars, renders and black coating. Sampled spaces include indoor and outdoor locations such as the chapel of Senhor do Horto, front-porch, cells, cloister and church. The material characterization by X-ray diffraction, thermogravimetric analysis, scanning electron microscopy coupled to X-ray dispersive energy detector and optical stereomicroscopy, was performed in HERCULES laboratory.

With regard to mortars (indoor and outdoor) it was used as a binder a lime of calcitic nature. The TGA allows to estimate a ratio of lime to aggregates of 1:3 or 1:4 being these identified as quartz, feldspar, mica and granite. Aggregates exhibit generally angular shapes denoting short transportation of grains compatible with granitic material from Sintra region. As in mortars, the binder of the black coating is always of a calcitic nature. Just in two samples, registered as an exception, it was detected calcium and magnesium in the binder used on the chapel of Senhor do Horto, denouncing the probable use of dolomitic lime in this space, probably as an later intervention. The black pigment was identified mainly as charcoal, rarely char.

The characterization of the materials from the black coating did not indicate any type of functionality, namely of an isolating nature. Nevertheless its application, along with the volume and layout of the ecclesiastical-architectural complex, suggests an intention to mimic the surrounding landscape by recreating dark spaces as the image, namely the natural cavities generated by the chaos of granite blocks, of the Serra de Sintra.

### **Keywords**

Black Render; Multi-Analytical; Sintra; Charcoal





## **Topic 6**

Historic production, processing and application of mortars, renders and grouts. Lime technologies





## **Blast furnace slag in historic mortars of Bergslagen, Sweden**

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### **Abstract**

Water cooled granulated slag is today commonly used as a binder mixed with cement. Slag contributes with both hydraulic and pozzolanic properties. Since the late 18<sup>th</sup> century blast furnace slag was used as an additive in bedding and pointing mortars of lime in the area of Bergslagen (the iron producing area in the middle of Sweden). It was developed in order to reach some hydraulic or pozzolanic properties in the mortar. This type of mortars has not been documented and described in Sweden. The aim with the present project has been to document possible traditional use of slag in mortars in iron producing area in Sweden and to see if it is possible to identify pozzolanic or hydraulic reactions.

The study is based on:

- a literature review on historic literature describing the production of slag and the use of slag mortars,
- inventory of historic mortars *in situ*,
- thin section analyses in polarization microscope and micro chemical analyses in SEM/EDS.

During the studied time period granulated slag was invented, but this is not reflected in the studied material. The unsuitable chemical composition of the slag gave it a very low reactivity. As this study only included a small part of the production sites, is it still possible that some furnace produced slag with a composition that could give a better reactivity.

### **Keywords**

Thin Section; Slag Mortar; Polarisation Light Microscopy; Blast Furnace Slag; Pozzolanic Additives

## **A Mortar Maker's guide to evolving mortar specifications in 18<sup>th</sup> and 19<sup>th</sup> C France and England and their implications today**

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### **Abstract**

In France and England, the 18<sup>th</sup> and 19<sup>th</sup> centuries saw the publications of essential treatises on construction and building materials. Some gave general principles on architectural styles, concepts and materials, such as the “Cours d’Architecture” by Jean-François Blondel in 1777 and the “Traité sur l’Art de Bâtir” by Jean-Baptiste Rondelet in 1803 and some were more specific such as George R. Burnell’s “Rudimentary Treatise on Limes, Cements, Mortars, etc.”, 1780 and Valentin Biston’s “Manuel théorique et pratique du chaffournier” published in 1836. In all of them, the way to burn lime is detailed along with methods to mix lime with sand and recipes of a range of mortars are compared and given. This work proposes to explore how these formulations and recommendations can inform the choice of materials and the methods of mixing for the design of nowadays conservation mortars. Many of the repair mortars used today were already used in the past. It is therefore important that the knowledge of many centuries these treatises bring together inform what we know of mortars today and how we use them. By combining detailed literature review of these treatises and beyond with a knowledge of current mortars in conservation this work links history of construction and conservation science to help inform conservation decisions. Findings include discussions on the type of lime to use and how, such as how to mix sand with fat lime, whether and when to add quicklime and what are the benefits, as well as how to choose aggregates and additives. Those topics often raise discussion in current research and amongst practitioners, and this work will help understand what was historically done.

### **Keywords**

Treaties; Archives; Conservation; Mixing Methods

## **Preliminary research on potential raw material sources for dolomitic lime mortars at St John convent at Müstair, Switzerland**

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### **Abstract**

The sourcing of carbonate rocks for air lime production is a topic scarcely investigated in historic mortar research. According to the scientific literature, provenance studies are based on a geochemical approach (Ortega et al., 2008; Miriello et al., 2011) and textural analysis between inner structure of lime lumps with potential sources (Bartz et al., 2017).

The Benedictine convent of St John at Müstair (UNESCO world heritage site) represents an ideal site for such research due to the availability of well documented and preserved dolomitic lime mortars dated from Carolingian to Baroque Age. Dolomitic potential raw material sources were collected from potential sources of the Upper Austroalpine domain (Engadiner Dolomiten) close to the site. Mineralogical analysis carried out through X-ray powder diffraction analysis (XRPD) allowed for a preliminary distinction of the collected materials. Dolomitic raw material sources can be easily differentiated on the basis of the presence of accompanying minerals. Therefore, the sources can be classified as rocks containing just dolomite (Piz Umbrail), dolomite with minor amount of gypsum or quartz (Pass dal Fuorn), dolomite, quartz, calcite, muscovite, and feldspars (Lü village). Samples collected in Val Vau are generally composed of dolomite. Therefore, the mineralogical composition also integrated and supported with textural analysis under the Polarizing Light Microscope (PLM) and geochemical data of major, minor and trace elements obtained through Inductively Coupled Plasma Mass Spectrometry (ICP-MS) indicates that the analysed dolomitic sources are clearly distinguishable and therefore can be possibly correlated with the compositions and textures of the historic mortars lime lumps in order to understand the provenance and eventually the changes of the sourcing areas.

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### **Keywords**

Dolomitic Lime; Lime Lumps; Müstair; Provenance; St John Monastery

## **Pozzolanicity beyond Vitruvius: insights into the exploitation of reactive silicates throughout the Roman world**

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### **Abstract**

The discovery of pozzolanic reaction through the employment of pozzolanic additions, amorphous aluminosilicate compounds capable to react with lime and water and precipitate nanostructurally-polymerized insoluble phases with excellent mechanical, permeability and durability properties, constituted the most relevant technological evolution in the field of inorganic binders since the beginning of pyrotechnology.

After the first applications by ancient Mediterranean societies of the Second and First Millennium b.C., the potentialities of pozzolanic binders were fully exploited by ancient Romans, which used them for large-scale structural purposes through the formulation and development of Roman concrete (*opus caementicium*). Driven by the necessity of parameterizing its production techniques, the Roman technology of pozzolanic binders pushed since the beginning to the systematic quarrying of the most suitable geological sources of pyroclastic materials, firstly individuated in the unconsolidated pumiceous ash levels from the Campi Flegrei volcanic district, and then, by the Augustan era on, in the loose sediments of the mid-Pleistocene Pozzolane Rosse pyroclastic flow from the Monti Sabatini/Alban Hills volcanic districts. Nevertheless, several evidences indicated that such established supply criteria were often overcome by the employment of non-standardized local pozzolanic materials, both natural and anthropogenic, with obvious geographic and social advantages.

In this study, a combined mineralogical-spectroscopic-microstructural analytical approach has been adopted for the characterization of the mineralogical and crystal-chemical features of Roman pozzolanic binders, considering several materials collected from various archaeological sites in the Italian peninsula and around the Mediterranean region. The samples have been analyzed by a combination of XRPD, MAS-NMR, FTIR and SEM-EDS, correlating when possible the compositional and microstructural information with the results obtained from mechanical tests. The study demonstrated that, besides a diffuse transport of the traditional pyroclastic compounds, several alternative materials were used by Roman craftsmen, including natural products such as volcanic breccias, nano-clays and microcrystalline sedimentary silicates, and artificial compounds such as ceramic byproducts and charred plant remains. Furthermore, results allowed defining the advanced levels of technical knowledge and the exceptional final performances of the Roman structural materials, both when standardized and non-standardized pozzolanic additions were used.

### **Keywords**

Pozzolanic Reaction; *Opus Caementicium*; X-Ray Powder Diffraction; NMR; SEM

## **Composition and Technology of the 17<sup>th</sup> Century Stucco Decorations at Červená Lhota Castle in the Southern Bohemia**

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### **Abstract**

Stucco decoration from the Great Chamber of castle Červená Lhota in the South of Bohemia Region was surveyed and characterised as part of its planned conservation. The works that are still to be specified in detail should be based on a good knowledge of the original materials and techniques. The main focus was on the fruit decorating the ribs of vaults. Altogether, over twenty mortar samples were collected from the bedding mortar, plaster, drawn elements and stuccos. Additional samples were also taken from the pieces of fruits, that were loose and had to be temporarily removed. The characterisation of the binder, aggregate and additives was carried out using optical microscopy, scanning electron microscopy with EDS, thermal analysis, acid attack, sieve analysis of the unsolvable residue, X-ray powder diffraction, Fourier transform infrared spectroscopy, nano-liquid chromatography-mass spectrometry. The characterisation led to the understanding of the uses of various mortar mixes and determination of their recipes. A typical fruit piece was composed of a modelling core mortar made of lime-gypsum binder and a coarser aggregate that was covered with a 5–10 mm thick lime rich stucco layer containing pure calcitic air lime and fine siliceous sand. The stucco mortar was also used for modelling all various fine and thin shapes like leaves, cherries and grapes. These were attached to the main piece by a fine gluing mortar made of gypsum and lime in various proportions. The use of gypsum and the differences in lime to gypsum ratio corresponded to a certain fresh mortar performance. This finding was a key parameter that was able to contribute to the reconstruction of the original application technique and procedure. The whole pieces were attached to the vault by a gypsum-lime mortar typically without any reinforcing pin. Qualitative analysis of animal proteins determined presence of collagen – an animal glue that was used to modify the setting of gypsum and which also played a role in the final finish appearance. The characterisation and the interpretation of the results are discussed as an example of a procedure that aims at a detailed replication of the original technique. A concept that stems from a detailed analysis, identification and location of similar raw materials and ability to process them in a similar way in order to have a mortar that genuinely replicates the original one.

### **Keywords**

Historic Production; Processing and Application of Mortars; Renders and Grouts; Lime Technologies

## **Hot applied lime mortar – assessment of a traditional technique used in modern restoration**

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### **Abstract**

Lime was one of the most important binder groups used for mortars in historic building activities and is often favoured in restoration work due to its mechanical and chemical compatibility to the historic structures. Different slaking methods of lime allow adjusting the respective mortar for special purposes and are often regional tradition. Slaking and processing lime at the same time provides hot mortar with temperatures of 40° to 80°C and unique properties. According to our research, within Europe, there are just a few craftsmen using this technique today, most of them working in the United Kingdom and Germany. They postulate from practical experience that hot applied mortars - “Heißkalk” in German - show very fast setting, give a higher strength, have better adhesion and are more salt resistant. In addition, high shrinkage, a constraint of lime mortars with high binder to aggregate ratio, seems to be less problematic.

A project carried out between 2017 and 2019 tried to keep this tradition alive and to get reliable parameters to assess hot applied lime mortars. For that, a workshop concept which shall continue also in future was planned and realized in two test trials and a final colloquium. Based on the use of powdered quicklime, the possibilities and limits of hot applied mortars as bedding mortar, render or harl, pointing mortar, floor and lime wash are demonstrated and discussed. Another focus is on the evaluation of recent works with hot lime technique on historic buildings and monuments with respect to their durability. Results of the assessment of different hot applied lime mortar mixtures on test areas of historic stonework in Kloster Buch (Saxony) after one year of exposure are presented.

Material tests on laboratory samples aimed at confirming or disproving the properties described from the practical experience of craftsmen. For this purpose, measurements of mechanical properties like bending tensile strength and compressive strength, but also water uptake and mercury intrusion porosimetry were carried out. Furthermore, the samples were investigated by polarising microscopy on thin sections and scanning electron microscopy on cracked specimens. The microstructure of the hot applied lime mortars, compared to normal “cold” ones, seems to have a crucial effect on the properties.

### **Keywords**

Hot Lime; Lime Mortar; Case Studies; Materials Tests; Properties

## **Warm applied Mortar (WAM) – An insight into the historical technique of “Heiße Speis” and its use for renders**

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### **Abstract**

Aside some manuals from the 18<sup>th</sup> and 19<sup>th</sup> Century, the technique of using “Heiße Speis” (hot mortar in German) or “hot applied lime mortar” is mainly reported orally as an old traditional craftsman’s method in Austria. For this, lime mortar was used immediately after dry slaking quicklime together with wet sand and applying the reactive mixture while still hot or warm. This hot mixed and applied mortar was used widely as grout, but is also said to work as a render with special qualities such as better workability. The process was also used when there was no possibility for building a lime pit or no time left to wait until the reaction was over. The approach of using quicklime still warm and reactive contradicts most conventions for producing durable and stable renders as such material is considered “still thirsty” and therefore useless and dangerous. To test this unconventional technique under field conditions quicklime was hot mixed (arranged in layers with sand from a nearby river in wooden boxes and slaked by adding water) but not stored to rest. As soon as the exothermic reaction slowed down, the materials were immediately applied to the wall still hot as a single layer render. As the material had no time to properly cool down, it was still warm and reactive when troweled up. As eight masons worked simultaneously on a rather small building the amount of fresh mortar needed in a very short time guaranteed that the whole façade was plastered with hot/warm mortar. After finishing in just a few hours the render was inspected the next day and lime washed as a final coating. In its practical application as a single layer render the “hot applied mortar (HAM)” showed very good workability and stripping the surface with a mason’s trowel was very easy. Even when there was no visible damage to the surface next day, the application was heavily doubted by the local craftsmen as it broke nearly every rule of conventional plastering. Nevertheless, even after an extreme winter in harsh weather conditions the coating showed surprisingly no significant signs of damage and was completely stable and compact. Only at the base of the wall near the street, where large amount of road salt was used for winter the plaster had to be replaced by another material. When the samples and parts of the plasters were finally tested and analysed in the laboratory, the SEM pictures showed signs of a specific structure of micro-cracks, which are characteristic components of this type of material. The lecture shows the practical process of producing and working with hot lime mortar and the experiences with this special technique.

### **Keywords**

Hot Mixed and Applied Mortar; HAM; Heiße Speis; Dry Slaking; Microstructure

## **Mortars and renders from Roman villa Horta da Torre (Portugal): a multi-analytical approach**

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### **Abstract**

The archaeological site of villa Horta da Torre (4<sup>th</sup> century) is situated near Cabeço de Vide in the municipality of Fronteira, Alentejo region in central Portugal. The villa presents a good state of conservation of the structures revealing unique architectural and aesthetic characteristics in the context of the Roman villas in Iberia. The villa had a large room with an area of about 90 m<sup>2</sup> – triclinium, that at one end presents a stibadium and around it a double apse structure (water reservoir). As an eccentric structure the triclinium had an artificial cascade from the abside wall, that would cover the triclinium floor with water. The walls of triclinium were covered with the panels of mosaic tesserae and the skirting with marble slabs. The mural paintings were preserved in peristylum. The unique indoor environment of the villa and sophisticated technical interventions and decorations should give an impressive outlook enjoyed by people invited by the rich owner of the villa.

In present work eleven mortar samples from different context and functions were characterized. After register and physical sample preparation several complementary techniques were used; stereomicroscopy, petrographic microscopy, variable pressure scanning electron microscopy coupled with energy dispersive spectroscopy, X-ray powder diffraction and thermogravimetric analysis. Together were used to determine the textural, mineralogical and chemical composition of different type of mortars from the villa.

Despite the refined taste and aesthetic peculiarities there seems to have been no great special attention in the preparation of the different mortars: the selected sands are coarser compared to other Roman villas and without distinction in their use, that is, same granularity for masonry as well render. Also there is not a specific layer - intonaco - as verified also in other contemporary villas of Iberia, but only a very thin discontinuous lime layer being that the coarse aggregates even come out to the chromatic surface. Nevertheless it was observed some intentionality such as the preparatory layers for the supranucleus and nucleus tesserae where the presence of ceramics works as an additive for the improvement of the mortar.

The binder used in all the samples is calcitic aerial lime obtained from the calcination of crystalline limestone probably sourced from the vicinity of the villa. The nature of aggregates is diversified but in agreement with a local geological provenance of less than 3km and in accordance with the two sand exploration sites.

### **Keywords**

Raw Materials; Mortar Function; Roman Villa; Provenance





## **Topic 7**

**Mortars in archaeological sites. Construction history.  
Archaeometry**



## **Technical analysis on materials and characteristics of mortar-based compounds in Roman and Late antique Aquileia (Udine, Italy). A preliminary report of the results**

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### **Abstract**

The present research reports the results of a series of investigations performed on Roman mortars and concretes employed in Aquileia during the Roman Age (II century b.C. – VI century A.D.). This study is part of a larger research promoted by the Department of Cultural Heritage and the Department of Geosciences of the University of Padova aimed at investigating binder's formulation and use in ancient times within the history of Greek and Roman building techniques. The overall project concerns samples collected from several archaeological contexts in the Mediterranean, including sites in Italy (Aquileia, Padova), Sardinia (Nora), Greece (Gortyna), Turkey (Hierapolis) and Israel (Caesarea Maritima).

The research project on Aquileia binders has been supported by the Superintendence of Archaeology, Fine Arts and Landscape of Friuli Venezia Giulia and by the research teams of the Universities of Padova, Udine, Venice – Ca' Foscari and Verona, currently involved in annual archaeological excavations in the ancient city. The samples represent the entire variety of architectures built with mortar-based materials in antiquity, encompassing walls, foundations, water infrastructures, floors and floor beddings, wall-paintings. Materials characterization was achieved through a multi-analytical approach comprising OM, XRPD, SEM-EDS and Raman analyses. XRPD analyses on separated binder fractions have been undertaken on selected samples for a quantitative discrimination of hydraulic phases, combined with trace element analyses for the definition of the provenance of pozzolanic materials and sourcing areas, in order to decipher commercial trades of raw materials. Subsequently, the analytical data have been statistically tested via PCA to define trends and clustering of compositionally related samples. Exploiting the potentialities of the single context approach, this research allowed to fully define the evolution of techniques and recipes of mortars and concretes adopted over six centuries in a Roman town. By cross-checking the results with complementary studies on Aquileia building techniques (Previato 2015) and with databases related to Aquileia mosaic art (TESS database: Ghedini et al. eds. 2018) and pictorial culture (TECT database), the study has developed a mature interpretation of technical competences and workmanship abilities of artisans in a wide range of construction and/or decorative activities, and their relationships both with the geological context and with socio-economic factors, such as the contacts with other regions of the Roman world, the economic power of patronage, the use and functionalities of individual structures in the buildings, and the chronological developments of the architectural structures.

### **Keywords**

Aquileia; Mortars; Plasters; Archaeometry; Archaeology of Building Techniques

## **M.N.I.A.R. techniques of macroscopic characterization from the colorimetry and chromatographies analysis applied to the mortars in the archaeological site of Los Hitos (Arisgotas, Toledo, Spain)**

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### **Abstract**

The late antique settlement of Los Hitos is one of the most important archaeological sites that is located in Arisgotas (Toledo, Spain), in centre of the Iberian Peninsula. This architectural complex is integrated by different buildings, between them ecclesiastical constructions, and preserves other relevant occupations across time. The whole preserves aspects that could be related to the presence of a important religious centre that can be compared with other places in the Region of Toledo, such as the near church of Pedro de La Mata and St Maria de Melque. The first excavations conducted in 70s in one of the main buildings discovered several external buttresses and a tripartite floor plan nave of a building with two floors that was utilised as funerary space. However, our excavations have shown different functionalities and occupation phases in the framework from Late Antiquity to the Medieval periods, which are: The set includes three well-differentiated spaces: 1) a prestigious rural residential building in the 6<sup>th</sup> c. AD; 2) a reoccupation of this previous space as a funeral pantheon in the 7<sup>th</sup>-8<sup>th</sup> c. AD and new buildings were built in this moment, such as a church; 3) an medieval Islamic farmstead, transforming and adapting spaces and adding other structures, from the 9<sup>th</sup> to the 11<sup>th</sup> c. AD. Mortars of different nature are identified in almost all these spaces (inside and outside buildings), which have been analysed applying a new macroscopic technique, called M.N.I.A.R. (high resolution non-intrusive macroscopy).

In the data collection phase, two procedures were carried out: first, a non-destructive sampling using a M.N.I.A.R.; and second, a sampling by manual and mechanical extraction. The M.N.I.A.R. has not caused any alteration on the original walls, making more than 2000 photographic microtomes with a portable binocular loupe of 400 magnifications. These high-resolution shots were processed at the time to an electronic device, which calibrated the images and stored them on a hard drive for treatment in the laboratory. The images were processed thanks to different software programs (VehoCapture 1.3, Gimp 2.8, JMicrovision 1.2.7 and Micam 1.6,) from which we were able to generate three-dimensional surface maps and granulometries based on the spectral calculation of the sections generated in high quality. The main result was to discriminate the mortars of the first phases respect to the latest occupatios, identifying pathogenic elements, such as microcracks, and documenting microstratigraphies belonging to old plasters and plasters. Finally, this work used a precise technical sequence of analysis that has allowed us to take a step further in the macroscopic characterization of mortars.

### **Keywords**

M.N.I.A.R.; Los Hitos; Chromatographies; Granulometry; Mortars

## **Insights into Carolingian construction techniques – results from archaeological and mineralogical studies at Müstair Monastery, Grisons, Switzerland**

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### **Abstract**

The project “Mortar Technology and Construction History at Müstair Monastery” is addressing the question about specific recipes for different uses of mortars and plasters through the medieval and early modern period. Thanks to decades of archaeological investigations at the World Heritage site (Goll, 2016), the remains of five mechanical mortar mixers (Hueglin, 2017) and more than 5000 mortar samples have been recovered, dating between about AD 775 and AD 1800. Müstair acts as a case study to tell about materials and methods in medieval and early modern construction sites. It also exemplifies the substantial changes that architecture, landscape and society underwent during these periods.

The proposed paper will focus on the earliest construction phase of Müstair monastery, which has been dated by dendrochronology to around 775 AD. A large number of archaeological features can be attributed to the Carolingian construction site: trenches, post-holes, fills, deposits of lime and plaster, as well as imprints on soil and mortar. They offer insights into the organization of the building site and the progression of the work. The petrographic analyses of mortars and raw materials further complement and expand our knowledge about Carolingian building techniques. Different mortar groups have been identified with different petrographic characteristics: one comes from the main church and one from the large adjacent building complex to the west, the so-called "Carolingian quadrilateral". This use of different materials could indicate consecutive construction phases of the monastery, the presence of two teams of builders, or different construction techniques chosen on purpose to differentiate between buildings with different functions and status.

J. Goll, Müstair, Kloster St. Johann. In K. Papajanni and J. Ley (eds.) *Karolingerzeitliche Mauertechnik in Deutschland und in der Schweiz*. Schnell&Steiner, Regensburg 2016, pp 343-360.

S. Hueglin, Historic Mortar Production in the First Millennium A.D. New Results from Archaeology and Scientific Dating. In M. Santhanam et al. (eds.) *Advances in Construction Materials and Systems, Proceedings of an International Conference (ICACMS) Chennai, India, 3-8 September 2017*, RILEM Proceedings PRO 118, Vol. 4, pp. 609-613.

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### **Keywords**

Historic Mortar; Building Technology; Medieval Archaeology; Carolingian Period; Monastery; Mortar Mixer; Polarised Light Microscopy; AMS Radiocarbon Dating

## **Animal, vegetable or mineral? Characterising shell-lime, maerl-lime and limestone-lime mortar evidence from the Late Norse and Medieval site of Tuquoy, Orkney**

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### **Abstract**

Recent examination of an extensive curated assemblage of mortar samples, removed from the Late Norse and Medieval site of Tuquoy (Orkney) during excavation in the 1980s, suggested the collection was associated with distinct groups of compositionally contrasting materials related to discrete constructional events. Subsequent petrographic analysis supported this early interpretation and presented evidence for a remarkable series of phase-specific mortars, bound with a range of different biogenic and geogenic lime source materials - including marine shell, coralline algae (maerl) and limestone. Wider landscape survey highlighted the broad range of exposed calcareous materials in the coastal and sedimentary environments dominating the Northern Isles of Scotland today, and that many of these different potential lime sources were exploited by craftspeople at different times in the medieval and later period is now clear.

Given the high significance of the Tuquoy mortar study for our understanding of the development of this culturally important site, and as a prelude to more general publication of the wider archaeological project, a further investigation of selected samples from the mortar assemblage is now being undertaken through a range of geoscientific techniques. This paper presents emerging evidence from a comparative petrographic, SEM-EDS and XRD study designed to further characterise these various mortar materials, and challenge those previous interpretations of contrasting building lime sources. Like most environmental archaeological investigations, this study is essentially concerned with interpreting the depositional histories of surviving materials, but with a particular focus on establishing the distinction between (anthropogenic) kiln relict and (natural) added temper mixtures when both contain biogenic and geogenic clasts.

### **Keywords**

Shell-Lime; Maerl-Lime; Petrography; Archaeology; Medieval

## **Analysis of mortars from the Tarragona Roman Aqueduct as a study case to document original building and restoration materials**

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### **Abstract**

Les Ferreres Roman aqueduct, also called Pont del Diable (Devil's bridge), is an important monument of the Archaeological Ensemble of Tarraco (Tarragona, Spain). It is one of the best preserved Roman aqueducts and, since 2000, has been listed as a World Heritage Site along with other remains of the Roman Tarraco. It was built about 4 km north of Tarraco to negotiate the Els Arcs valley. The structure, composed of two levels of arches, is 217 m in length and its maximum height is 27 m. Archaeological and historical information suggest that it probably dates from the time of Emperor Augustus (63 B.C.–14 A.D.) and that it was still in use during the Middle Ages. Despite some undated minor repairs, the first well-documented restoration was carried out in the mid-19<sup>th</sup> century under the supervision of the architect B. Hernández Sanahuja. From 2010, the aqueduct has benefited from an integral restoration financially supported by the Spanish Ministerio de Fomento and the Tarragona City Council. Within this work the study of their mortars was included to increase the knowledge about the monument and to assess the restoration process.

Different types of mortars can be distinguished depending on their use. Even though ashlar blocks are apparently stacked without mortar, a very thin layer of mortar was used to adjust the stone blocks. Different mortars were also used to set up the water channel, for the foundations of pillars, for coatings on the north wall, and even for ashlar replacements on the south wall. Other types of mortars were used for repairs, the most abundant being those used in the mid-19<sup>th</sup> century restoration. All types of mortar were accurately mapped on aqueduct front elevations designs to select the most representative for sampling. Sampled mortars were first analysed by petrographic optical microscope (POM). Chemical and mineralogical analyses were carried out by scanning electron microscopy (SEM-EDX), X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR).

Results indicate that the ancient mortars, particularly those employed in Roman times, are made with air lime and aggregates. They present different mixing ratios depending on whether they were used for the ashlar joints, for the Roman concrete, or for foundations. Raw material sourcing studies allowed assessing the provenance of the aggregates composing the mortars, indicating that these come from environmental sources. Results also suggest that repair mortars are different; those employed during the mid-19<sup>th</sup> century of special interest since they feature the presence of hydraulic phases.

### **Keywords**

Historic Mortars; Repair Materials; Construction History; World Heritage Monument; Spectroscopic Techniques

## **Characterisation of Roman Mortar from the Archaeological Site of Mirobriga**

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### **Abstract**

Mirobriga is a Roman site located in the municipality of Santiago do Cacém, in Setúbal, a district in the southwest of Portugal. Mortar samples were collected from the domestic, commercial buildings, and public buildings from the site. The samples may be divided according to their function – filling, rendering, and plaster. The chemical, mineralogical, and microstructural characterisation of the samples was performed using a number of complementary techniques – stereomicroscopy, polarised light microscopy, chemical and granulometric analysis, thermogravimetric analysis (TGA), powder X-ray diffraction (XRD), and variable pressure scanning electron microscopy-energy dispersive spectrometry (SEM-EDS). The results show that in most of the samples, the aggregates consist of quartz sand, whilst the binder is lime-based. The exception, however, may be seen in the samples where stratigraphy is present. In such samples, the external layer of the mortar functions as a plaster, and both the aggregate and the binder consist of calcite. In addition, the majority of samples have a binder to aggregate ratio of 1 : 3. As a whole, the raw materials used for the production of the mortars may be said to be quite similar, and shows little difference between the filling and rendering mortars, though the aggregates of these two types of mortar are different from those functioning as plaster.

This project was made possible thanks to Dr. Manuela de Deus and the Direção Regional de Cultura do Alentejo, the Laboratório HERCULES/Universidade de Évora, and the ARCHMAT Consortium.

### **Keywords**

Mirobriga; Roman Mortars; SEM-EDS; XRD; TGA



## **Analysis of Mortar Samples from the Church of the Saints Sergius and Bacchus at Umm as-Surab (Jordan)**

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### **Abstract**

Northern Jordan has a complex system of ancient villages built almost entirely in basalt which, thanks to particular historical, cultural, geological and environmental convergences, have survived until today in a reasonable state of conservation. This is a built landscape located in a border zone between the steppe and desert areas (the so-called Hawrān) which for centuries has been part of a network of important communication routes between the Mediterranean coast and the Arabian Peninsula. For this reason, it was the melting pot of various cultures such as Nabatean, Roman, Byzantine, Sassanid, and Islamic. In such context, the architecture mirrors this cultural variety and becomes a fundamental source of information, especially for the Late Antiquity and the Middle Ages, not only from the point of view of material culture but also for the study of the social and cultural phenomena behind the historical crucial changes. Because of the rich history of the buildings in the area, the only way to unlock the information contained in them is to use an archaeological approach. The most advanced archaeological approach to the analysis of buildings and structures is the Building Archaeology that allows unravelling the complex construction sequence common to a large number of historic buildings, sometimes very difficult to decode due to the reuse of building materials and to the continuity in the use of specific construction techniques. Building archaeology methods and techniques were applied to the Church of St. Sergius and Bacchus at Umm as-Surab (Jordan) and this has allowed highlighting its complex stratigraphy. To support the results of the stratigraphic analysis and to provide further information on the construction materials and technologies used in its construction at various stages, archaeometric analyses were carried out on mortars samples from various part of the building. In this paper, we report the initial results of XRD and SEM analyses of 7 mortars samples taken from specific stratigraphic contexts of the religious complex. Results suggest the use of lime as a binder together with gypsum and clay.

### **Keywords**

Jordan; Hawrān; Mortars; XRD; SEM

## **Microbiological diversity of ancient architectural structure of Wawel Royal Castle in Krakow, Poland**

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### **Abstract**

Historic masonry and mortars of cultural heritage buildings, for centuries had been exposed to physical, chemical, and biological factors causing the degradation of materials used for the construction of buildings. Often these degradation processes are associated with the loosening of the structure of masonry and mortars and supposedly the release of ancient microorganisms and various compounds which could be scavenged by these microbes. Additionally it appears that in historic buildings there is commonly observed salt efflorescence which in turn leads to the formation of extreme niches colonized by specific microorganisms.

In our study we investigated “The Lost Wawel” complex of Wawel Royal Castle which is one the most significant historical museum in Poland. In 2017 it was visited by over 1.4 million tourists. The Lost Wawel is composed of buildings built in different periods (from 10<sup>th</sup> to 16<sup>th</sup> century) and using different materials such as irregular sandstone plate bound with lime mortar or slab broken limestone bound with gypsum mortar. The most valuable are the remains of the Rotunda of Sts. Felix and Adauctus but the exhibition also encompasses the former royal kitchens and coach house. The objective of our study was to characterize microorganisms colonizing the surfaces of historic buildings. The microbiological diversity was determined by analysis of deep sequenced amplicons covering hypervariable regions V3V4 of 16S rDNA gene and ITS2 located between 5.8S and 26S rDNA.

### **Keywords**

Cultural Heritage; Microbiological Diversity; High-Throughput Sequencing

## **Characterization and durability analysis of coral stones in a marine environment**

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### **Abstract**

Coral stones are a variety of limestone composition, rare masonry units used in many world historic structures but least understood. Many structures are in the verge of deterioration. In current situation where the World Wide Fund has banned the further mining of coral stones, the continued existence with proper protection of structures is critical in preserving the cultural value. Coral stones collected from a historic structure of 15<sup>th</sup> century from Southern India was examined for physical characteristics. The chemical composition was analyzed using X-ray diffraction where calcite and aragonite were observed. The pore structure and porosity of the highly porous stone was determined using mercury Intrusion Porosimetry. The stones were then subjected to accelerated weathering tests in laboratory using immersion in sodium sulfate and sodium chloride solutions allowing dry-wet cycles. The mass loss was determined after each cycle, and microstructural characterization was carried out to see the modified pore distribution and porosity after the weathering cycles. It was found from the investigation that even though coral stones possess low compressive strength, they have extremely good performance with respect to durability. From the results, sodium chloride was found to be not a detrimental weathering agent for coral stones. Whereas long-term exposure of sodium sulfate in heavy dosages can cause slow but homogeneous kind of weathering. Wind could be a major possible factor that cause weathering in such structures. Hence, providing water repellent coatings is not a suggestive remedy for preventing further deterioration.

### **Keywords**

Coral Stone; Historic Masonry; Salt Crystallization; Weathering; Characterization

## **Monitoring of bio-aerosols, gaseous and Particulate Matter (PM) pollution and microbiological contamination of stones and mortars of the reserve “The Lost Wawel” of Wawel Royal Castle in Cracow, Poland**

Magdalena Dyda; Ewa Wilkojc; Beata Kwiatkowska-Kopka; Oliwia Buchwald-Ziecina; Karolina Szlek; Sławomir Korzeniowski; Paulina Drabik; Aleksandra Skłodowska

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### **Abstract**

Air pollution resulting from anthropogenic sources have increased the atmospheric concentration of inorganic and organic compounds and their deposition on stone surfaces. The complex physical and chemical interactions of these agents with the mineral material has dramatically accelerated the decay of stone materials. Additionally, organic pollutions can influence on the intensity of the microbial contamination of stones, mortars and bricks. In Cracow, one of the cities with the worst air quality in Europe, protection of historic monuments and buildings have been introduced to the city's strategic policy documents. The oldest place in Cracow is Wawel Hill – a Jurassic limestone rock formed about 150 million years ago. In addition to the Wawel Royal Castle, there is an archaeological reserve on the hill - “The Lost Wawel”. These exhibition is dedicated to the history of Wawel Hill and centered on the remains of the Rotunda of Sts. Felix and Adautus from the late 10<sup>th</sup>/early 11<sup>th</sup> century. This is the most important architectural site within the reserve. The cylindrical rotunda with four apses is built directly on limestone bedrock. The building is constructed of irregular sandstone plate bound with lime mortar. The exhibition also encompasses remnants of medieval structures: the pre-Romanesque walls of the tomb annex with a partly disturbed burial site, a Gothic defensive wall, and the foundations of the Gothic upper castle. This unique exhibition combines an archaeological-architectural reserve, architectural elements from various periods and traditionally displayed objects uncovered in archaeological excavations, requires special conservation care.

In our study we were monitoring Indoor Air Pollution (IAP) including gaseous, particulate matter and microbiological pollutants of the reserve “The Lost Wawel”. IAP concentrations were monitoring once every two months. The microbial communities presented on mortars and stones surfaces were also studied. Microbiological samples were taken from different heights of the Rotunda walls and mortars.

### **Keywords**

Cultural Heritage; Particulate Matter Pollution; Microbiological Contamination; Mortars Deterioration; Stone Deterioration.

## **Fernandina old fortress of Lisbon – contribution to its preservation**

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### **Abstract**

The Fernandina old fortress of Lisbon started to be built in 1373. It is composed by 76 towers and 35 entrances along the fortress that is closed with an extension of 4.69 km, having two main sections: East, limited between St. George's Castle and Terreiro do Trigo Street, and West, starting also in the St. George's Castle and finished at Misericórdia Street. The construction technology of those sections is mainly rammed earth with an average height of 8 m. Some sections seem to be totally homogeneous but there are other composed of two parallel stone masonry walls with a nucleus filled with compacted earth. The thickness of the fortress wall is variable between 1.75 and 2.20 m.

To build with rammed earth technique, humid excavated earth was placed in layers on a wood formwork and manually compacted reducing the thickness of each layer. A succession of compacted layers completed the formwork, which was laterally displaced to build the next rammed earth block. After completing the rammed earth level, the formwork was displaced to the upper level and the same cycle continues up to the top level of the wall fortress. Sometimes, particularly in defensive structures, air lime was added to stabilize the humid earth, obtaining the so called "military rammed earth".

In the other case, the humid earthen materials were placed in layers and compacted inside the two parallel masonry walls that acted as formwork.

The fortress is nowadays completely "emerged" and surrounded by the city. Several interventions mainly performed on old buildings confining with, or including, the old fortress have been held in the last years. Some of the sections present renders and repointing mortars that are not originals. Nevertheless, so far there is a lack of information on the materials, originals and applied in the history of interventions.

Therefore, this study intends to present the characterization made in situ by visual observation and non-destructive techniques and in laboratory on samples that was possible to obtain from some sections of the old fortress walls.

It is expected that the information from the material characterization will be useful to support decisions on future interventions, namely on the definition of repair mortars that need to be compatible and assure efficient conservation of sections that are being accessible of the old wall.

### **Keywords**

Rammed Earth; Repair Mortar; In Situ Characterization; Laboratory Samples Characterization; Preservation

## **Characterization of historical mortars from the Botanic Garden of the National Palace of Queluz**

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### **Abstract**

The National Palace of Queluz and its historical gardens are one of the most remarkable examples of the harmonious link between landscape and palatial architecture in Portugal. They illustrate the evolution of the Court's tastes in the 18 and 19<sup>th</sup> centuries, a period that was marked by the baroque, rococo and neoclassicism. Built in 1747 at the orders of the future King Pedro III, the Royal Palace of Queluz was initially conceived as a summer residence, becoming the royal family's preferred place for their leisure and entertainment. They lived there permanently from 1794 until their departure for Brazil in 1807, as a result of the French invasions.

The National Palace of Queluz was designated a National Monument in 1910 and has been a member of the Network of European Royal Residences since 2013.

In 2015, the Parques de Sintra – Monte da Lua (PSML) began the work on the restoration of the Botanical Garden in the National Palace of Queluz. This intervention falls within the framework of the overall restoration project for the National Palace and Gardens of Queluz. In this context, the work that is being carried out at Laboratory HERCULES in collaboration with the PSML, aims the material study of several mortar samples, collected from different spots, namely, the entrance portico, the foundation walls of the greenhouses, dividing walls, benches and flower boxes.

The analytical methodology employed is an adaptation of standard analytical procedures used in historical mortars studies. The data acquisition techniques consisted of X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscopy coupled to energy dispersive X-ray spectrometry (SEM-EDS) and thin-section optical microscopy. The results of textural, mineralogical and chemical characterization allow, among others, the identification of mortars binder and aggregates and also the determination of the binder: aggregates ratio. This provides useful information regarding the history of gardens and the construction techniques of the different spots according to its purpose. The results obtained show that the mortars are lime-based mortars composed of calcitic lime with siliceous aggregates, mainly quartz. The silicates phases identified in the aggregate fraction suggest a sedimentary source for the aggregate raw materials. The results point out for different technical solutions depending on the mortars function, including the binder to aggregate ratios.

### **Keywords**

Botanic Garden of The National Palace of Queluz; Historical Mortars; Textural; Mineralogical and Chemical Characterization

## **Petrographic and chemical-mineralogical characterization of plaster and mortar from the Renaissance cistern at Amaiur Castle (Navarre, Spain)**

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### **Abstract**

Throughout history water has always been one of the most important resources and has been stored and reserved in areas where water could be scarce, especially at time of drought or a siege. Cisterns have been used to supply water in a regular and independent way since the Neolithic Age. These infrastructures have varied from simple clay structures to large underground structures with specific requirements such as waterproof linings and moisture resistant. Advances in building skills have allowed long-term conservation of cisterns resistant to highly deteriorating factors.

Amaiur Castle (Navarre, Spain) has two cisterns, the oldest cistern dated in the medieval period, and the Renaissance period cistern (16<sup>th</sup> century). This study is focused on the castle's Renaissance cistern built between 1512 and 1522. It was a period of war between the kingdom of Castile and the kingdom of Navarre, after the conquest of Navarre by the Crown of Castile in 1512. The cistern is housed between the first medieval wall (13<sup>th</sup>-14<sup>th</sup> centuries) and the second medieval wall (14<sup>th</sup>-15<sup>th</sup> centuries). Walls at different heights and the beginning of the barrel vault structure that would have closed the cistern are the preserved remains today. This study aimed to carry out an accurate petrographic and chemical-mineralogical characterization of mortars and plaster from the Renaissance cistern at Amaiur Castle. Polarized Light Microscopy (PLM) observations of thin sections, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Raman Microspectroscopy and Fourier-transform Infrared Spectroscopy (FT-IR) analysis have been performed to determine the main features of the mortar and plaster in order to achieve a better understanding of some technological aspects of the manufacturing processes.

Multianalytical results indicate the tank cistern plaster consists of a red pigment layer composed by a mixture of lime, iron oxide and an organic compound as a waterproof lining sealing off the structure.

### **Keywords**

Cistern; Waterproof Plaster; Lime Mortar; Renaissance







## **Topic 8**

Dating of historic mortars



## **Structural characterization and thermal decomposition of lime binders allow accurate radiocarbon age determinations**

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### **Abstract**

Radiocarbon dating of anthropogenic carbonates ( $\text{CaCO}_3$ ) such as ash, lime plaster, lime mortar, and concrete, has proven a difficult task due to the occurrence of a number of contaminants embedded within the  $\text{CaCO}_3$  pyrogenic binder. These include radiocarbon-free geologic components and/or secondary phases bearing an unknown amount of radiocarbon, which nucleate upon recrystallization processes (calcite) and pozzolanic reactions (e.g. layered double hydroxides). Thus, the alteration of the original pyrogenic isotopic signature of the material results in major age offsets when carbon recovery for dating is performed through acid hydrolysis of the binder. Here we present a characterization/quantification approach to anthropogenic carbonates that includes Fourier transform infrared spectrometry (FTIR), X-ray diffraction (XRD), thin section petrography, and scanning electron microscopy coupled with high-resolution cathodoluminescence (SEM-CL), with which we identified the pyrogenic  $\text{CaCO}_3$  fraction in archaeological lime binders from different regions and periods. The preserved pyrogenic component was then isolated by density separation and its purity checked again using FTIR. Carbon was recovered through thermal decomposition in vacuum. Carbon aliquots obtained in the range 500-600°C allowed avoiding secondary phases and geologic components, which decompose at lower and higher temperatures, respectively. The resulting radiocarbon ages match the expected age of the sample, based on radiocarbon dating of organics embedded within the binder, or on archaeological assessments. When the pyrogenic fraction was not identified due to severe recrystallization of the lime binder, we did not proceed with dating, but rather used the carbon aliquots evolved at different temperatures to obtain insights into the diagenetic history of the material.

### **Keywords**

Carbonate; Mortar; Plaster; Diagenesis; Radiocarbon

## **An Ecology of Castle Construction: geoarchaeology, archaeobotany & radiocarbon analysis in the ecotone of Lochindorb Castle**

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### **Abstract**

Lochindorb Castle is situated on a small island, in an isolated and exposed freshwater loch, high in the Grampian Mountains of Scotland. The building is currently ruinous and surrounded by an almost treeless landscape, with wide views dominated by rough infertile pasture and non-calcareous metamorphic rock outcrops. Various strands of documentary evidence, however, suggest that Lochindorb Castle was a building of significant regional and national import in the medieval period, and subsequently remained a navigational landmark in one of the main routes across Highland Scotland for some considerable time. Moreover, the surviving complex is extensive and multiphase, and contains a sophisticated primary phase enclosure which can be confidently associated with a refined mid-late 13th-century date.

In line with the established methodologies of the Scottish Medieval Castles and Chapels C-14 Project (SMCCCP), Lochindorb Castle has been subject to a programme of landscape, buildings and materials analysis; including geoarchaeological, archaeobotanical and radiocarbon analysis of surviving constructional materials. With a particular concern for material provenances and depositional histories, this investigation has considered: possible geological sources for various stone and mortar materials; how contrasts in mortar composition relate to structural phasing; and the relationship between relict fuel taxonomies/morphologies and radiocarbon data interpretations. Ultimately, this SMCCCP case study is presented to further highlight the importance of examining how buildings relate to their surrounding environments, and how a palaeoenvironmental approach to materials analysis and radiocarbon dating can inform our understanding of the physical, cultural and chronological context of masonry construction.

### **Keywords**

Radiocarbon; Buildings; Environment; Archaeobotany; Petrography

## **The latest advances on Single grain OSL dating of mortars and their integration in early medieval archaeology**

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### **Abstract**

Single grain OSL dating of mortars is nowadays becoming a new support in analyzing the construction history of ancient buildings which is particularly stimulating for the research on early medieval architecture. The present paper will show the progress achieved in the last few years, consisting in the transition from the methodological research towards its real applications in archaeology. The SG-OSL method is applied on the aggregate in mortar which makes it applicable not only on lime-based materials, but also on all the types of clay mortars containing quartz sand. The basic premise in OSL dating of mortars is that quartz in the sand used for making mortar is optically zeroed during the preparation process. The moment to be dated is the last exposure of mortar to light.

The first OSL dating tests on known-age archeological mortars were performed by a standard single aliquot OSL technique, indicating potential but raising also some important shortcomings linked in particular with the problems of poor bleaching. A deeper research, focused on dating of known-age monuments, has been carried out since 2012 in the Bordeaux laboratory IRAMAT-CRPAA. It consisted in an important methodological innovation: a systematic use of so-called "single grain analyses" (SG-OSL). This technique, relatively recent in the field of luminescence dating, allows overcoming the problems of heterogeneous light exposure. However, the change of scale of analyses from hundreds to single grains required to reassess our perception of the material in terms of its heterogeneity and to develop new concepts of data treatment. Therefore, the tools for convenient assessment of "single grain" measurements, taking into account the bleaching mechanism of mortars, have been developed. Contrary to usual practice, we insist on systematic characterization of studied mortars by different analytical techniques in order to evaluate their microdosimetric characteristics. Thanks to the all aforementioned methodological advances, we are currently able to assess reliability of dating results obtained which is a fundamental requirement for the integration of the SG-OSL dating in archaeological research.

Since the last 6 years, 15 monuments from France, Switzerland, Italy, Croatia and Spain were studied by this novel approach, providing dating results for more than 80 mortar samples from different historical periods. The majority of them indicates conclusive results. Rather than the individual case studies, the paper will attempt to discuss the geographical potential of this dating approach with respect to the experimental data collected up to now within the Western Europe.

### **Keywords**

Mortar Dating; Building Archaeology; Medieval Architecture; Single Grain; Optically Stimulated Luminescence

## **Characterization and Radiocarbon dating of complex mortars in Historic Buildings**

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### **Abstract**

Since centuries, mortars were used in building technology and their dating is often requested in order to establish the chronology of different mortared constructions in archaeological and historical contexts. Radiocarbon dating was recently used to date mortars considering the atmospheric CO<sub>2</sub> signal absorbed during the hardening process by the lime putty, which is converted back into CaCO<sub>3</sub>-binder containing the signature of the construction time. The anthropogenic carbonate must be isolated from other carbonate sources as well as other contaminants which could affect the dating process and then it has to be subjected to radiocarbon dating [1–3]. Despite the large efforts devoted by the scientists to find an efficient binder isolation method and control, the routine dating of complex mortar systems seems to be elusive, and dedicated studies in this peculiar field are needed. On one hand, simple lime-based mortars with a one-step carbonation history are known to yield reliable dates of the binder emplacement. On the other hand, such an ideal situation is hardly encountered in practical cases, where radiocarbon dating of the carbonated binder is variously affected by geologic carbonate, delayed hydraulic reactions, formation of long-term phases containing carbonate, and secondary alteration processes.

It is evident how an appropriate mineralogical characterization of the mortar greatly enhances the understanding of the reaction history of the material allowing the preliminary assessment of the “dateability” of the binder, and afterwards, the choice of the needed pre-treatments for a reliable radiocarbon dating.

The mineralogical composition and microstructural features of common historic lime mortars investigated in different cases of study are described, and a series of parameters are proposed that can be efficiently used to assess the chances of extraction of datable binder fractions. Furthermore, innovative treatments of the fine binder fraction in order to eliminate the influence contaminants are shown.

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### **Keywords**

Historic Mortars; Radiocarbon Dating; Characterization

## **Characterization of historic mortars: techniques used to establish a construction chronology. Case study: “Aragoneses mill” as it belongs to popular architectural heritage**

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### **Abstract**

Restoration actions in building construction require complete preliminary studies. It is essential to perform tests for the characterization and identification of materials. In this work, mortars and bricks characterization as a method of dating constructive techniques have been used. A case study for the “House of Aragoneses”, an ancient watermill sited in Monachil, Granada, is presented.

According to the building historical studies, there was an oil mill on that place that worked due to the Huenes River waterflow from the 15<sup>th</sup> to the 16<sup>th</sup> centuries. It may have disappeared because of the ravages of the Monachil Moorish properties in the riots prior to 1570. But from the documentary information existing in the archives, the construction works from XV to XVI century are not clear. The building underwent changes throughout history but there is not any documentation related to these interventions.

By means of different tests and characterization techniques, dating and identification of existing masonry works at the “House of Aragoneses” has been accomplished. In the first construction phase, on 18<sup>th</sup> century, mixed masonry with natural stone and burnt clay bricks -- with bricks burnt at low temperature-- was carried out,. Lime mortars with low lime/aggregate ratios and densities were detected.

In the second construction phase, from the 19<sup>th</sup> or early 20<sup>th</sup> century, the same type of masonry work was executed, but in these cases with bricks burnt at higher temperature and lime mortars with a richer proportion of binder.

Therefore, mortars analysis has helped to establish a construction chronology differentiating ancient and modern phases of the mill architecture. It has also allowed for a better interpretation and understanding of the remains of this building adding value to popular heritage.

### **Keywords**

Masonry; Restoration; Historic Mortars; Diffraction; Thermal Analysis







## **Topic 9**

Natural and Roman cement mortars



## **European natural cements - their key technical properties under standardised conditions**

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### **Abstract**

This paper provides the first comparative information on 7 commercially available brands - 4 from Spain and 1 each from France, Poland and the UK. In the absence of a European Norm the testing procedures have followed a standard protocol proposed by the EU FP7 funded ROCARE project, which itself is based upon various ENs for cement and building limes. Thin-section microscopy identifies characteristic features of natural cements. XRD shows similar belite patterns and quantities in all cements but with a range of sulfate content which is reflected in ettringite production in the first 24 hours of hydration; 1 cement being sulfate-free. Other early age hydrates include hemi-carboaluminate and a carbonated AFm phase in varying proportions. Five cements are typified by rapid setting whilst two display prolonged setting times of 90 – 135 minutes. Mortars for evaluation of physical and microstructural characteristics, up to an age of 270 days, were produced at identical mix proportions. Substantial variation in consistency was observed such that a second, more limited series was produced at a common flow for comparison at ages of 7 and 91 days only. Mortar strengths remained generally static during at least the first 7 days of curing before displaying rapid strength gain with 91 day strengths in range 7.5 – 21.3 MPa. Whilst at ages of 28 and 91 days the relative performance of each cement was similar, different rankings were observed at ages of 1 and 7 days. Varying the w/c ratio to achieve constant workability yields greater strength variability and relative performance at both ages. The Water Absorption Coefficient was shown to reduce between the ages of 28 and 91 days. Later age pore structure is more varied than previously reported. SEM investigation reveals characteristic features associated with natural cements with hydrate phases of acicular or tabular habits in the  $\mu\text{m}$  size, arranged in a more or less porous fabric the density of which varies between the different cements and with mortar age. The matrix compactness visible by SEM correlates well with mortar strength and WAC and increases with age.

Retarded mortars were produced using a pre-hydration technique. The response to this process varies across the range of cements and may relate to their sulfate content. Using combinations of de-activation water, storage time and variations to the mixing protocol it was possible to achieve a workable life up to 90 minutes.

### **Keywords**

Natural Cements; Mortars; Comparative Performance; Physical and Chemical Properties

## **From marlstone to rotary kilns – the early development of Portland cement**

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### **Abstract**

The invention and evolution of Portland cement (PC) emerged from a continuous development of different hydraulic binders starting in the late 1700s. Around the turn of the 20<sup>th</sup> century the increasing requirements of construction projects along with the technological developments in the cement production provoked that PC became the most widely used hydraulic binder in building construction. Based on the optical and electron microscopic investigation of different PC-based materials from structures in six Central European historic objects built between 1868 and 1908 completed with data from historical sources, the present contribution discusses the phase composition and microstructural properties of unhydrated residues and hydrated cement phases and thus the evidence of technological development of cement production. As an example of the early stage of PC production, the natural PCs in Austria in the 1860s were characterized by coarse and very heterogeneous cements containing, besides C3S, C2S, C3A and ferrite, a wide range of under and over burned components. Although the PCs produced between the late 1870s and the turn of the century were less heterogeneous, the amount and distribution of cement phases suggest the absence of accurate control of the raw mix. Finally, the PCs produced in the early 1900s show features that are characteristic of clinker burned in rotary kilns for the first time. Although the cements of the early 1900s were more homogenous compared to their 19<sup>th</sup> century precursors, some features indicate the progressive introduction of new kiln types. Nevertheless, the analyses of residual cement grains suggest that the evolution of milling technique could not keep up with the new calcinations technologies. The compositional differences of hydration products between the samples produced in different periods were predominantly a consequence of by-mixed calcium sulfate and the effect of secondary reactions. The results obtained contribute to gain more knowledge to better understand, preserve and maintain historic PC structures.

### **Keywords**

Portland Cement; Production Technology; Hydrated Cement; Microscopy

## **Drying Shrinkage of Historic Portland Cements: Factors to be Considered for Successful Repair**

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### **Abstract**

Drying shrinkage of building materials containing Portland cement is a key concern in their conservation. While drying shrinkage can lead to cracking of hardened cementitious materials, it is also an important consideration for repair materials, as failure to match the shrinkage of the original and new materials can result in a repair that is poor. However, this is a complicated issue as changes in manufacturing technology and processes, available raw materials, and the introduction of material standards, have all had an effect on the physical and chemical properties of cementitious building materials. As such, historic materials vary considerably from those that are manufactured today, and there is limited understanding on how to address drying shrinkage, both of the in-situ material and of repair materials. In order to better understand this issue, a drying shrinkage study of materials containing historic Portland cements was undertaken. A statistical analysis of the results was then performed in order to determine which physical and chemical properties have the most influence on drying shrinkage, and the implications posed for the conservation of historically-significant structures.

### **Keywords**

Portland Cement; Repair; Analysis

## **Restoration techniques using 1930's Portland cements at Porte de l'Est in the Roman city-wall of Aventicum, Switzerland**

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### **Abstract**

Historic masonries using hydraulic cement were extensively used for construction and restoration at the turn of the 20<sup>th</sup> century. Different cements such as Roman (or natural) cements, natural Portland cements or dolomitic cements, were used according to their local availability, the choice and experience of architects and workers. The Roman city Aventicum (now Avenches) was built at the beginning of the 1<sup>st</sup> century. Unique in Switzerland, classified of national importance the wall of the Roman enclosure of Aventicum was built during the second half of the first century AD. 5.5 kilometers long, this vast perimeter crowned the capital of the Helvetians and showed the power of Rome. Subsequently to a large campaign of archaeological and restoration works initiated from 1845, the restoration of the (one of the four) original main entrance of the city, Porte de l'Est, consisted in the construction (in the 1930's) of a protection wall above the roman vestiges. The walls were made of artificial and joined stones composed of three materials (core in concrete, intermediate layer of mortar finishing layer mortar local tuffstone). This study presents first the technological details of the artificial stones and the wall construction. Then the identification of the cements (natural Portland cements with different sulfates contents) and the compatibility properties between the successive layers are discussed.

### **Keywords**

Hydraulic cement; Natural Cement; Portland Cement

## **Repairs to Historic Concrete Pavement at Jacob Riis Park Utilizing Natural, Roman and Portland Cements**

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### **Abstract**

When Hurricane Sandy devastated the community of Breezy Point, NY in 2012, destroying some 350 houses, the Federal Emergency Management Agency (FEMA) utilized the large concrete parking field at Jacob Riis Park as a debris collection area in their emergency response. The Park is part of the Gateway National Recreation Area and is designated as a National Historic Landmark.

Extensive damage caused to the historic concrete pavement by the storm-relief operations required repairs involving placement of over 900 cubic meters of natural cement concrete to match the original pavement.

Due to the overlapping authorities of the New York City Department of Parks, the US Army Corps of Engineers and FEMA, a mix design meeting the City's historic restoration standards, the Army Corps of Engineers performance standards and FEMA's budgetary requirements had to be developed. The Parks Department historic preservation office prioritized use of a natural cement binder to reproduce the original concrete mix as closely as possible. The Corps of Engineers specified a minimum 28-day compressive strength of 31 MPa to meet their pavement performance standards, a figure generally beyond the 28-day strength development capabilities of natural cements alone. A repair mortar based on a combination of Roman Cement, American Natural Cement and Portland Cement binders, with fine aggregates matching those used in the original concrete materials, was developed to meet the objectives of all parties. Matching coarse aggregates were blended with the repair mortar on site to produce a concrete matching the appearance of the original pavement, meeting the 31 MPa minimum strength and incurring reasonable costs for FEMA. The approach to meeting these design criteria, the testing program and the final results are discussed and illustrated.

### **Keywords**

Historic Cements; Natural Cement; Roman Cement; Catastrophic Repairs

## **The use of mortars in Palau Güell by Antoni Gaudi**

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### **Abstract**

In Catalonia during 19<sup>th</sup> century there was a process of industrialization following the Llobregat river. One of the traditional materials used historically in building had been lime mortar. Natural cement began to be used from the middle of the 19<sup>th</sup> century in order to improve quality and characteristics of mortar elements. Barcelona experimented a huge urban expansion during the second half of 19<sup>th</sup> century. In order to do new buildings it was necessary to develop new techniques and materials.

Antoni Gaudi finished his studies in 1876 and he began working in Barcelona since then. He had learned from traditional materials and techniques at his father workshop in his hometown. He also learned from new technologies at School of Architecture in Barcelona.

Guell family were a wealthy family who owned different industries around Catalonia, following Llobregat river. They were the founders of a Portland factory in Spain, at Castellar de N'Hug in 1904. Before that date some Portland cement shipments had arrived and had been used in Barcelona's buildings imported from France.

They asked to a young architect, Antoni Gaudi to build their family house in the centre of Barcelona, near the harbour. Palau Guell built from 1886 to 1890 was the first important project done by Antoni Gaudi. In this building, Gaudi was able of studying and experimenting different technologies and materials that he would later use in other buildings as Mila's House or Sagrada Familia.

Palau Guell is built during a period of confluence between traditional and industrial techniques. There are some traditional techniques as paintings or plaster coatings with other more industrialised as the use of pavements and finishing which they answer to a will of seriation and standardizing. There is a neat debate between technique and art and the relationship between production and design. Gaudi was focused in order to introduce new technologies to buildings and to enhance traditional ones.

In Palau Guell different mortars are used as binder materials and as fastening material for façades and pavements. This different way of using mortars lead us to do research in some areas of Palau Guell obtaining interesting results in the use of natural cements.

This lab research has allowed us to understand the development of mortars by Antoni Gaudi. This research proves how Gaudi was an architect related to traditional techniques but looking to its development through research and experimentation.

### **Keywords**

Natural Cement; Historic Mortars; Gaudi; Technology



## **From lime to cement. Historic binders in Catalonia**

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### **Abstract**

This article deals with the results obtained from the research being carried out on natural cement in Catalonia, from the beginning of its local manufacture in the mid-19<sup>th</sup> century until the late 1930s, with the widespread use of Portland cement and the implementation of the new structural construction system based on reinforced concrete.

At this stage of the research, great importance has been given to knowing the coexistence of the different lime-based conglomerates; aerial limes, artificial and natural hydraulics limes, weak or eminently hydraulic limes, the different types of cements, first the natural ones and then the artificial ones, puzzolans, etc. Another important aspect that will be detailed is the interesting discussion on nomenclatures and how sometimes the reference of the same product has been found, with two different ways of naming it and that can sometimes confuse, especially in times of very diffuse borders between different materials, scarce regulations, incipient testing technology, multitude of inventions and patents, and so on.

Likewise, some significant information will be presented on production and the different types of ovens and technologies, the marketing of the product, the diversification of products marketed by the same manufacturer and the communication networks for their distribution, both near and far, with the aim of completing the global image of these materials in a specific time and geographical delimitation.

As far as the uses are concerned, we can affirm that natural cement was used in all the construction systems; mortars for the masonry of foundations and walls, Catalan vaults, pavements, coatings; renders and plaster, and even in prefabricated format, blocks or drainage collector pipes. With regard to prefabrication products, one of the most characteristic uses for their performance was that of modelling, for the realization of artificial stone in the form of decorative and ornamental elements, above all, in facades.

All this study, basically historical, has to be an aid to complement together with other disciplines, the reading of architecture, its cultural values, of use, etc. and, above all power to improve the interventions in rehabilitation and restoration of the heritage, with compatible materials.

### **Keywords**

Natural Cement; Traditional Binders; Industrial Heritage; Restoration; Artificial Stone

## **When Portland cement meets natural cement**

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### **Abstract**

The development of Portland cement industry gradually replaced the natural cement production by the end of the 19<sup>th</sup> century. But a the pivotal period in between, in some construction, natural cement that was still used for its unique ability to mold ornaments, could neighbor Portland cement used for the structural elements such as beams and walls. In this paper a dramatic combination of natural and Portland cement in one of the first reinforced concrete building of Paris will be presented.

In 1898, François Hennebique decided to build his headquarters in Paris city center using his newly patented reinforced concrete system. In 1900 a splendid 36 meters-high building exhibiting arches, balusters bow-windows and molding, was achieved, with additional ornamental flamed stoneware sculptures in a pure art-nouveau style.

In 2016 a restoration of the façades of this building was planned, mainly in the aim of recovering a clean aspect and eliminating the thick layers of paint accumulated with time. When the paint removal operation was achieved, incredibly detailed finishing of the concrete emerged with light tools marks. Locally important cracking was also observed leading to a deeper diagnosis. Binocular observations and SEM examinations combined with EDS analysis of cross sections and fractured surfaces evidenced a massive sulfate reaction in the Portland cement structural elements. The origin of this reaction could clearly be attributed to the presence of high-content sulfate natural cement in the vicinity of the Portland cement concrete.

### **Keywords**

Natural Cement; Portland Cement; Sulfate Reaction

## **Methodology of identification of natural and historic Portland cements. Application and study in mortars of Madrid and Barcelona**

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### **Abstract**

Natural cement was patented in England in 1796, but did not arrive in Spain until 1835. Its arrival supposed a natural coexistence with the rest of traditional mortars that had been used (lime, hydraulic limes and gypsum). However, its use was not extended in time, and soon it was replaced by the use of artificial Portland cements.

Although in Spain the natural cement came later, it was massively used in the decorations of historicist and modernist facades during the 19<sup>th</sup> and 20<sup>th</sup> century.

Nowadays, after more than 100 years since its construction, it's necessary to conserve all this heritage. In order to achieve a good restoration, it is essential to correctly identify the mortars originally used.

The restoration of these mortars with incompatible materials can cause irreparable damage to the original elements of the buildings.

The aim of this research is the development of a simple, practical and economical method for the identification of natural cements and historic Portland cements mortars.

We have use petrographic microscopy techniques in laboratory samples to establish the identification patterns of these cements. Then, this methodology has been applied in samples of buildings of Madrid and Barcelona where it is documented the use of historical cement mortars.

The differences in the formation of alite and belite crystals between Portland and natural cements have been analyzed, as well as the differences in the petrographic textures between the cements of Madrid and Barcelona.

### **Keywords**

Natural Cement; Historic Mortars; Roman Cement; Roman Cement Industry





## **Topic 10**

**Conservation issues concerning mortars, plasters, renders and grouts. Diagnosis. Decay and damage mechanisms. Case studies**



## **Evolution of mortars composition and characteristics during the 20<sup>th</sup> century– Study of Portuguese buildings awarded with Architecture Valmor Prize**

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### **Abstract**

20<sup>th</sup> century is most known for the changes that have led to a new paradigm of construction techniques and the increasing use of new materials on the verge of modernity. Reinforced concrete has become the wide spread new construction material, letting behind traditional lime mortars that were used massively throughout the past centuries. Thus, Portland cement gradually substituted lime-based mortar's binders, both aerial and hydraulic, and quickly bestowed an unmatched material to all construction agents, however almost incompatible with the traditional ones.

The Valmor Prize, one of the most prestigious prizes for Architecture in Portugal, was established in the beginning of 20<sup>th</sup> century to distinguish the most architectural striking buildings in Lisbon. In this paper the results of physical, mechanical, chemical, mineralogical and microstructural characterization of renders and plasters collected in several Valmor awarded case studies will be presented. Their study aims to understand the evolution of composition and physical and mechanical characteristics of wall renders over the 20<sup>th</sup> century, since this knowledge and the evaluation of conservation state will have a huge contribution for valorization and preservation of built heritage.

### **Keywords**

20<sup>th</sup> Century; Renders; Mortars; Valmor Prize

## **The restoration of the church of Our Lady of the Assumption, Daus, Bohol, Philippines**

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### **Abstract**

The study presents the historical significance of Daus church in Bohol, Philippines, the timeline of its construction, the role and actions made by cultural agencies in the immediate rescue and restoration effort on the damaged structure brought by a magnitude 7.2 earthquake that struck last October 2013. It discusses the extent of damage to the structure. It also tackles the underlying principles and restoration guidelines that govern its actions and the basic issues and concerns that influence the conservation methods, approaches and program of work based on architectural and engineering studies and investigation of composite materials such as mortars, plasters and consolidation of rubble core. On site test (surface strength determination, Coring, Wall Scanning, Elemental analysis and Environmental condition) and laboratory analysis (Mechanical, Physical and Chemical analysis) were performed. Daus Church was built using Coral Stones which is a challenge in the restoration works. Collaboration with different government agencies were conducted to look for alternative compatible materials for the losses.

### **Keywords**

Composite Material; Coral Stones



## **The analysis of the proportion of mortar for Japanese roof tile (Ibushikawara) in Taiwan by applying of Taguchi Method**

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### **Abstract**

Cultural Heritage Preservation and rehabilitation have been gradually emphasized recently in Taiwan. A lot of studies have been carried out on the various buildings and material. However, due to the lack of knowledge in mortar, some restoration works use the cement to repair old Japanese roof tile (Ibushikawara) in Taiwan. The old roof tile would be damaged by cement and it cannot be reused. Therefore, this paper focused on the effect and optimization of using different types of mortar for interface on Ibushikawara by using Taguchi experimental design method. The experiments were conducted by 8 control factors, the control factors are like adjusting weight proportions of lime and sand, weight proportions of lime and fiber, varying types of lime etc. The collection of mortar proportions data is based on craftsmen, historical documents and related research. The preliminary result revealed that the adhesive bond strength of many experimental groups met the standard stress of 0.09 kg/cm<sup>2</sup> in Taiwan and all experimental groups are reversible.

### **Keywords**

Mortar; Taguchi Method; Japanese Roof Tile; Lime; Mix Proportion

## **The use of dolomitic lime in mortar samples from a 15th-century buttress of York Minster (York, UK)**

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### **Abstract**

This contribution describes the results of a series of analyses carried out on mortar samples from a 15<sup>th</sup>-century buttress of York Minster (York, UK). The aim of this research is to shed some light on the construction technologies and materials used in the North East of England in medieval time. York Minster is one of the most important religious places in North England and has been the center of Christianity since the 7<sup>th</sup> Century CE. The Minster is one of the largest Gothic Churches in Northern Europe. Its architecture shows the development of the Gothic style in England through a time-span of almost three centuries and is still nowadays a major destination of great religious and artistic interest. Despite its vast cultural relevance, little is known about the materials and techniques used in the construction of the building. It is general knowledge that in the past, construction skills, knowledge, and expertise were developed empirically and orally passed between members of the same guild with a high risk of being lost (if not properly recorded) when the related materials and technology changed. The lack of knowledge on the use of traditional materials (e.g. lime) currently hinders the planning and execution of correct conservation works. In the framework of the restoration works carried out at York Minster, the production of new, compatible repair mortars is a priority. The choice of appropriate compatible materials for the restoration of the Minster is of fundamental importance and is possible only through an accurate and detailed understanding of the materials and techniques used in the past. In order to provide an insight on the material and technology used in the construction works carried out in the 15<sup>th</sup> century at York Minster, the characteristics of a mortar sample collected from one of the buttresses currently under restoration, was investigated using a variety of analytical techniques such as optical and electron microscopy and X-ray diffraction analysis.

The analyses provided an initial characterization of the mortar including the binder-aggregate ratio, the aggregate characteristics, the characteristics of the binder (that was found to be produced by using a locally sourced dolomitic limestone) and the technology used for producing the mortar. These results are of great importance for future conservation works at York Minster.

### **Keywords**

Lime; York Minster; Hot Lime; Dolomitic Limestone; 15<sup>th</sup> Century

## **Digital image analysis as a basic for the evaluation of mortars in architectural conservation**

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### **Abstract**

Undoubtedly, mortars have always played a crucial role in the conservation of built heritage. Although this is not today's knowledge, it is astonishing that there has been hardly any systematic investigation into this type of conservation-agent under a historical perspective. The restoration mortars, that have been used in the last decades or centuries, have passed through different stages of development and differ in their execution. Yet we cannot quantify these measures, nor can we make a statement about which materials or implementations have been most successful in the long term. Despite the constantly growing internationalisation of cultural heritage research, the conditions for conservation-restoration treatments still vary so much from place to place that it is indeed difficult to draw an overall picture. Consequently, to fill the research gap on the efficiency of conservation mortars, a self-contained environment is required which, in addition to a long history of restoration, also reflects the most advanced ideas for conservation of the respective epoch. The ancient city of Pompeii offers these possibilities. During a four-year research project it was possible to systematically investigate various historical conservation mortars that were applied between the 19<sup>th</sup> and 20<sup>th</sup> century. The focus lies on the edging repairs of plasters, which were applied to preserve wall paintings and plasters in situ. The repairs could be classified chronologically based on historical photos. In addition to the documentation of location, exposure, condition and shape, material samples were taken and made into thin sections for further laboratory testing. Along with the chemical analysis the aim was to develop a method based on the digitized images of thin sections that would allow a targeted reproduction of the historic conservation mortars. Digital Image Analysis has proven to be a powerful tool for extracting all the required parameters, such as grain size distribution, stratification, porosity and binder content, necessary for the characterisation of the mortar. With its low analytical effort, in comparison to elaborate analyses methods, the easy availability is a great advantage of this method. To ensure a successful implementation, the susceptibility to errors was determined by adjusting all important parameters with supplementary methods such as SEM/EDS/XRF/XRD/ $\mu$ CT and laser diffraction analysis.

The aim of the research was to develop an iterative analysis pipeline for a systematic assessment of the efficiency of conservation mortars, accessible to everyone. The results also add to information of the chemical composition and can be used for reconstructions.

### **Keywords**

Pompeii; Architectural-Conservation; Mortars; Digital-Image-Analysis; Thin-Section

## **Decorative renders simulating stone of middle 20<sup>th</sup> century in the region of Lisbon**

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### **Abstract**

Housing and public buildings of the middle 20<sup>th</sup> century period, in Portugal, are characterized by a sober, rather severe, architectural and constructive design. This is the so-called Estado Novo period. In the region of Lisbon, many of those buildings were covered with very durable unpainted decorative renders, simulating stone, called “marmorite” and obtained by a special technique consisting on: application of a finishing render with a mineral binder and aggregates selected to obtain an aesthetic effect, usually of limestone or marble nature; short drying period for partial hardening; washing of the final skin of the binder in order to let the aggregates visible; very careful curing to avoid cracking of the render, which was rich in binder to provide a good key for the aggregates. This finishing technique for façades is thought to be based in techniques of the same type used in Central Europe in the period between wars.

Considering the period – middle 20<sup>th</sup> century – in which cementitious binders were already often used, both in the structural elements and in coatings, and due to the darkening effect of fungus, soiling and pollution, there was a generalized belief in the technical milieu that these decorative renders were cement based mortars. This belief was enhanced by the existence of an official specification document referring the use of a cement mix. As a consequence, repair interventions on those buildings used materials and techniques chosen for cementitious renders. In 2006 a restoration of the main building of the National Laboratory for Civil Engineering, in Lisbon, constructed in 1950-1952 and with a “marmorite” decorative render, was planned, and tests were carried out to characterize the mortar. It was verified that the “marmorite” render was composed by air lime as binder, and limestone and marble as aggregates. Was this building an exception? A large experimental campaign was later accomplished to clarify the composition of “marmorites” of that period, in the Lisbon region. Samples of 21 buildings localized in 3 different districts were collected and characterized from the chemical, mineralogical, microstructural, physical and mechanical points of view.

It was concluded that “marmorites” of that period, in the region of Lisbon, were generally composed by air lime, sometimes coloured with a pigment, and limestone and/or marble aggregates of different colours selected for their aesthetic effect. Hydraulic binders were found only in a building and it was interpreted as possibly corresponding to a repair intervention.

### **Keywords**

Decorative Render; Marble Aggregate; Marmorite; Construction Technology; Material Characterization

## **Towards an integrated approach to mortar analysis - The Pompei Arch&Lab Project**

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### **Abstract**

The conservation and care of archaeological sites is one of the great challenges of the present day. Pompeii, the only ancient city that has come down to us almost intact. Pompei Arch&Lab (Restoration Archive and Exposition Laboratory) is a cooperation project, launched in 2015, between the science and humanities.

Combining the skills of the different disciplines involved, the project aims to investigate for the first time, from both a scientific-technological and a historical-cultural perspective, the 250 years of excavations, restorations and opening to the public of the ancient city of Pompeii in order to provide practical answers to the issue of the relationship between ancient evidence and the preservation of the monumental heritage.

The methods and effectiveness of the restoration techniques applied thus far to the original works of Pompeii are analysed in detail through physical and chemical investigations, with particular attention to the study of the wall surfaces and mortars. From a historical and artistic perspective the city of Pompeii is examined as an archive of the history of restorations from the eighteenth century to the present day, in the various historical, technical and cultural contexts.

Recently unexplored sources and documents like drawings, paintings, photography and historic guidelines to the preservation of Pompei relating to the history of restoration and the musealization of the ancient city serve as knowledge base for the development of new materials for restoration, and as the prerequisites for innovative approaches in the field of historical investigation. Thus the different disciplines are brought closer together.

The processes currently used by the science of construction materials – Raman spectroscopy, micro-CT, high resolution 3D microscopy and computerized statistical image analyses – in combination with the traditional methods of analysing mortars, allow an integrated approach to the reconstruction of the mixture of materials up to the level of the particles: the preparations of the mortars used in the first stages of restorations at Pompeii can thus be classified and assessed and, in turn, they can contribute to the development of new construction materials for the sustainable conservation *in situ* of wall paintings.

### **Keywords**

Mortar; Analysis; Edging Repair; Conservation History; Pompei

## **Practical application of lime-pozzolan mortars to damp masonry**

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### **Abstract**

In the Czech Republic, there are examples of cultural heritage non roofed buildings/structures that are exposed to severe weathering conditions as wind-driven rain, hygroscopic salts, rising damp, and freezing-thawing cycles.

The fencing cemetery wall in Krchleby (about 100km from Prague) is an example of such a construction to be plastered according to cultural heritage authorities. Rising damp is one of the main sources of damage, but its treatment is very expensive and, in most cases, ineffective.

In the present experiment, which preceded the repair of the cemetery wall, renders composed of lime and lime-pozzolan were applied to the structure. The mortars included a local raw pozzolanic material (so-called Czech clay shale), with a similar composition metakaolin, but with a higher content of alumina.

This study is focused on the visual and chemical-mineralogical monitoring of the lime-metakaolin mortars applied on the cemetery wall (5 panels 2x5 m); each render panel was 50 cm width and covered the entire height. The wall was covered up to half of its height by earth from the interior side, hence constituting the main source of moisture and salts. The plasters were exposed throughout the 2-year monitoring period to the influence of rising damp, rain water, and frost. All these factors affected the course of hydration and carbonation of the binder. One year after the render was applied, samples were taken from both the lower and upper parts of the wall and analyzed in terms of water and water-soluble salts' content, thermal analysis (TGA), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The analyses showed that water and water-soluble salts' distribution on the panel was strongly affected by the type of binder of the tested mortars.

Air lime mortars had significantly lower water and salt content than pozzolanic mortars. Pozzolanic samples collected from the lower part (area covered with earth from the interior side) was fully saturated with water, which on the one hand led to the formation of calcium silicate and aluminate hydrate phases, and on the other hand it hindered the carbonation reaction.

Deposits of halite (NaCl) crystals were detected by SEM on the surface of the mortars from the upper (drier) part of wall.

### **Keywords**

Repair Mortar; Damp Masonry; Pozzolan; Metakaolin



## **Topic 11**

**Preservation. Consolidation materials and techniques. Development of new products.**

**Preventive conservation**





## **Comparative analysis of permeability values of traditional aerial lime mortars for preventive conservation**

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### **Abstract**

The suitable materials should always be used in historic building and this compatibility is defined in relation to the substrate and the existing mortars on historic renders and plasters. These papers refer to the influence of aerial lime characteristics in water vapour permeability capacity in pre-dosed mortars of powder and in putty lime. It is interesting defined this behavior as traditional construction permeable material for its application in render mortar and plaster mortar in Cultural Heritage conservation. The coatings must guarantee the breathability of the wall to prevent its deterioration and to avoid efflorescence or subflorescence deposited on the surface of masonry. To realize this study have been elaborated selected mortars sample following UNE-EN 1015-19:1999/A1: 2005 that determines the degree of permeability to the mortars water vapor in render mortar and plaster mortar. The mortar permeability is directly related to the porosity of the product which, in turn, is determined by the granulometry, therefore it is considered important to measure the ascending flow of water vapour across the material and in the opposite direction. This analysis allows to check the influence of the carbonation process in the permeability coefficient measured in different periods of time, until 180 days, addition to considering the importance of the type of aggregate (composition and granulometry) and relation binder/water in its properties. This study establishes the values of coincidence and those which demonstrate significant variations in the characterization of the materials used for every mortar. One of the principal advantages of the aerial lime mortars use resides in the aptitude to regulate moisture content of environmental humidity. In the tests results are defined the values obtained to determine the degree of symmetry in water vapour data and exchanges; later are analyzed the singular circumstances (room temperature, humidity) and, to finish, are indicated the permeability behaviors of the different dosages.

Experimental data according to the dimensions of the test are discussed defined its properties. Permeability is critical to evaluate the respirability of the wall. It is possible to conclude that this aerial lime mortars guarantee durability and effectiveness for the renders and plaster with a controlled dosing of the composition. However, sometimes it is not possible to obtain enough data about old materials, especially concerning masonry which is more difficult to test than mortars. The characteristics of the mortars to use can be established, based on the results obtained, in order to ensure compatibility and functionality.

### **Keywords**

Aerial Lime Mortar; Permeability; Testing Procedures; Conservation

## **Comparative study of ethyl silicate versus acrylic resin consolidation of wall painting with high water and salts contents: a case study at the Chapter Hall of Chartres cathedral**

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### **Abstract**

The consolidation of wall paintings is particularly challenging when high contents of water and of hygroscopic salts are present in the masonry. Many wall paintings present such a situation, but only a few of them offer an easy access and a long-term monitoring possibility. The 14<sup>th</sup> century wall painting located on the East wall of the Chapter Hall of Chartres cathedral is one of them. This painting, covered by several whitewashes, is suffering from severe deterioration, mostly strong paint detachment. A partial mechanical uncovering revealed powdering and flaking paint layers characterized as a lime-based paint colored mainly by ochre and black carbon pigments. It turned out recently that a consolidation had to be considered because the Chapter Hall is bound to be opened to visitors. New environmental conditions might likely lead to damaging side-effects with serious risks of salt crystallization and hence destruction of remaining pictorial layers.

The aim of this study is to assess the compatibility, efficiency and long-term behavior of ethyl silicate (Silres BS OH 100, Wacker Chemie AG) and acrylic resin (Primal E330S, C.T.S. France) for the consolidation of wall paintings showing high salts and water contents. Ethyl silicate was applied only once by brush, up to visual saturation. No further application was performed because whitening was observed shortly after this treatment. Acrylic resin was sprayed three times one month apart in a single pass. Several observations and tests were performed before, three and nine months after the treatments. Dry matter was calculated to know the actual amount of product applied after evaporation of the solvent. Macro and micro-pictures of the surface were taken. Color changes were controlled by spectrophotometry. Scotch and cotton swab tests were performed for the assessment of superficial cohesion. The water absorption capacity and the surface water-repellency were assessed respectively by sponge and micro-drop tests. Diachronic follow-up of water content was realized with dielectric measurements. Temperature and relative humidity were continuously monitored next to the consolidated areas in order to correlate the potential evolution of the surface aspect and physical properties with the changes of microclimate.

Nine months after application, ethyl silicate does not show any measurable consolidating effect and keeps its initial hydrophobicity, suggesting an incomplete hydrolysis. On the contrary, a significant consolidating effect is achieved with the acrylic resin without important changes of surface properties and water absorption capacity.

### **Keywords**

Wall Painting; Consolidation; Hygroscopic Salt

## **Preliminary results on the use of ammonium phosphate solutions for the consolidation of lime-based mortars**

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### **Abstract**

In this study, a preliminary evaluation of the effectiveness of aqueous solutions of diammonium hydrogen phosphate (DAP) for the consolidation of lime-based mortars is reported. So far, the use of DAP solutions has proved successful for the consolidation of carbonate stones, such as marble, limestone and sandstones. The core idea is that phosphate ions supplied by the DAP solution react with calcium ions supplied by the substrate (or externally added) to form calcium phosphate (CaP) minerals, ideally hydroxyapatite (HAP). Thanks to HAP formation inside the pores, grains can be more effectively bonded and mechanical properties of weathered materials can be restored. Starting from the promising results obtained on stones, in this study the use of DAP solutions was explored for 4 different types of mortars: 2 lime-based mortars (containing either siliceous or calcareous aggregates), 1 lime-based mortar also containing brick powder as a pozzolanic addition (so-called "cocciopesto" mortar) and 1 mortar based on natural hydraulic lime. Two different formulations of the DAP solution were considered, differing in terms of DAP concentration,  $\text{CaCl}_2$  addition as a calcium source and ethanol addition to boost CaP formation. First, for the various types of mortar, the nature and the morphology of the new CaP phases were investigated by FT-IR and SEM, for increasing treatment duration (ranging from 30 minutes to 7 days). Once identified the most promising treatment conditions (in terms of formulation of the DAP solution and duration), the mechanical benefit resulting from the new CaP was evaluated. The increase in cohesion was evaluated by ultrasonic measurements and scotch tape test, while the increase in mechanical properties was assessed by measuring the compressive and the flexural strength. The treatment compatibility was also evaluated, by measuring the colour variation and the alteration in water absorption. The obtained results suggest that the DAP-based treatment is highly promising for conservation of lime-based mortars, as it allows achieving a significant mechanical improvement, with minor alterations in stone colour and water absorption.

### **Keywords**

Hydroxyapatite; Calcium Phosphates; Inorganic Consolidants; Lime-Based Mortars; Cocciopesto

## **Frost resistance of reproduced mosaic mortars**

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### **Abstract**

Flaxseed oil has been identified by FTIR spectroscopy as admixture used in mosaic mortars from beginning of 20<sup>th</sup> century, collected in Central European region. The present paper deals with influence of this admixture on engineering properties of lime based mortars, which composition was inspired by the historic mosaic mortars. The flaxseed oil has been dosed to mortars in amount of 0.5, 1, 1.5 and 2 % with respect to the slaked lime. The admixture has influenced both fresh state, as well as hardened properties of mortar. Thanks to its nature, flaxseed oil has acted as hydrophobic agent, reducing the rate of water absorption to the mortar and increasing the amount of entrained air; both of these effects improved frost resistance. On the other hand, the oil also reduced the rate of mortar carbonation, what resulted to the slower strength evolution compared to control mortar without oil.

### **Keywords**

Frost Resistance; Historic Mosaic Mortars; Flaxseed Oil Admixture

## **Black pigmentation by fungi in Romanic churches' wall paintings in Northern Portugal (15<sup>th</sup> and 16<sup>th</sup> century). Challenges and strategies of preventive conservation in places of worship**

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### **Abstract**

Wall painting in Portugal was developed with intense activity between 15<sup>th</sup> and 16<sup>th</sup> centuries. During an on-going inventory of wall painting ensembles in the north of the country, a problem of biodeterioration, which is manifested by the abundant production of a blackish film that stains both granite supports and paintings, is often encountered. Internationally, there are few case studies investigated that can be considered similar to national ones. Wall paintings present biological colonization by different organisms, but few reports mention paintings with black stains or the existence of black fungi in them.

The visual impact resulting from this biological alteration is a matter of importance in the aesthetic restitution of the painted surfaces, and a problem that frustrates mural paintings' conservators, without a solution on site. Whereas the microbial reduction is usually achieved through the use of biocide treatment, these products are ineffective in removing black pigmentation resulting from their growth. The traditional construction system that lasted hundreds of years is beginning to show weaknesses that a mere passive climate control is incapable of slowing its degradation. Due to rural and isolated location of the majority of churches, and the cessation or decrease of cult duties in several of them, implementation of technology-based control methods is not sustainable.

Towards ensure a holistic approach of the problem and sustainable conservation protocols, a comprehensive study both nationally and internationally was carried out, including the commonality aspects that make wall paintings in religious buildings in northern Portugal susceptible to fungi degradation. The survey focused on data gathering comprising variables for both architectural heritage (tutelage, frequency of cleaning/opening, location, area (urban/rural), among others) and the non-dissociated sets of wall paintings (date, characteristics, paintings location, restoration interventions, indoor/outdoor temperature and relative humidity evaluation, among others) and research on solutions applied in Portugal and abroad for risk mitigation in similar cases, leading to the elaboration of an all-inclusive preventive conservation plan. The methodology combines risk mitigation, implementation of organizational procedures through new strategies for community involvement in articulation with the respective dioceses, sustainable, durable and affordable solutions, the application of a protective layer imbued with biocide that does not need systematic application and the use of mural painting prototypes or thematic mappings for quantification of lacunae and degradation areas. On this regard, an extensive research was conducted for the compilation of solutions.

### **Keywords**

Preventive Conservation; Black Stains; Wall Paintings; Sustainable Solutions; Holistic Approach

## **Coatings in the conservation of built heritage with earth in Santiago de Chile**

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### **Abstract**

Despite the seismic risk, Chile has a considerable number of works built with earth, where the adobe is the best known technique. Its conservation depends on elements such as roof or foundations, but also the quality of the coating, which is vital for protection against erosion and infiltration of rain; as well as for natural hygrometric control of the inner wall structure. This study aims to contribute to the updating, dissemination and promotion of traditional techniques and materials used in coatings whose structure and / or filler is based on earth as its main construction material.

Through the survey and graphics, comments on the subject of study records, and sampling on site, we proceed to analyze dice, and their historical and theoretical context. Different samples of coatings on various structures of earth (adobe, and mixed (wood/earth) structures), in the historic districts of Santiago are chosen. Based on the above results, we proceed to a practical job of checking the possibilities of improving the behavior of the original coating systems founded. The disclosure of the study has considered conferences and workshops. We have shown good practices of intervention on the original walls, as well as presented various proposals for improvements to the original coatings compatible with its conservation. With the disclosure activities we've trained an audience interested and related with built heritage subjects, that could contribute to the promotion of good conservation practices.

### **Keywords**

Traditional Earth Plasters; Heritage Preservation

## **Highly transparent TiO<sub>2</sub>-SiO<sub>2</sub> layers for cultural heritage preservation**

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### **Abstract**

In the field of cultural heritage preservation all new products and solutions must meet strict requirements. They cannot change the appearance of the original matter; the materials used for reconstruction should imitate the original. But above all, they must be safe in a long term perspective. This means they must undergo a long list of tests before being introduced on the market.

The subject of our work is to develop highly transparent TiO<sub>2</sub>-SiO<sub>2</sub> protection layers suitable for historic buildings. This means we will be testing a whole range of substrates: natural and artificial stones, bricks but also mortars used for joints. We will study the preparation process of colloidal systems with extremely small particles of titanium oxide and silicon oxide, their stability and photocatalytic activity in relation to ratios and concentrations of both components in the mixture. An important part of our project will be focused on the influence of pH and its optimization. In order to avoid negative impact of photocatalytic properties of TiO<sub>2</sub> particles on the organics possibly present in the original layer a protective interlayer will be introduced. A system based on gels will be tested, as gels should not soak into the original material but should stay on its surface.

Armed with several years of expertise in the field of TiO<sub>2</sub> layers application on plasters, two years ago we started an experiment on two types of material from a museum collection, i.e. marble – representing flat and polished material, and sandstone – representing coarse and porous structure. Based on experiences gathered during this experiment, we want to prepare the restoration mortar for repair of historic masonry.

We believe TiO<sub>2</sub>-SiO<sub>2</sub> protection layers, thanks to their self-cleaning properties, could be a good solution for historical monuments struggling with high pollution and microbiological attacks.

### **Keywords**

Titanium Dioxide; Transparent Layer; Restoration Mortar

Cancelled



## **Calcium alkoxide as an innovative product to consolidate cracks in cement mortars**

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### **Abstract**

Building and construction material decay is a consequence of combined factors which cause physical, chemical and biological weathering [1]. Conservation treatments have a key role for the protection of construction materials, but several products used in this field show an unsatisfactory long-term performance which requires frequent re-application [2, 3]. Therefore, scientists are constantly involved in the research of stable, efficient, compatible and long-lasting consolidation treatments. Within this context, in the present work a novel product developed during the NANOMATCH European project for the conservation of carbonate stones, calcium tetrahydrofurfuryloxide  $\text{Ca}(\text{OTHF})_2$ , has been applied to consolidate Portland cement mortars, which simulated the surface layer of concrete. This product, dissolved in a proper organic solvent, can react with  $\text{CO}_2$  and humidity of the air to form calcium carbonate. Before the application of the treatment, cement mortar samples were pre-cracked to simulate a damaged or deteriorated surface layer of concrete. In fact, even if the presence of thin cracks (<0.1 mm in width) does not constitute a structural issue for a reinforced concrete building, it may compromise its durability and aesthetic features. The properties of cement mortar samples were investigated before and after the cracking process and, finally, after the application of the consolidation treatments. Performance and effectiveness of the new products were evaluated in terms of efficacy and compatibility and explored as follow: consolidation effect, reached penetration depth, visual aspect, variation in water transport properties and changes in the superficial morphological characteristics.

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### **Keywords**

Cement Mortars; Conservation; Calcium Alkoxide





## **Topic 12**

**Repair mortars and grouts. Requirements and design. Compatibility issues. Durability and effectiveness. Repair mortars: Adequacy of testing procedures**



## **Characterization and compatibility assessment of commercial stone repair mortars**

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### **Abstract**

Commercial, ready mix stone repair mortars have often been applied in conservation practice for the restoration of natural stone, brick and terracotta over the last decades, with varying success in terms of durability of the mortar itself and compatibility with the substrate. It is well-accepted that, for a stone repair mortar to be compatible, its properties should be as much as possible similar to those of the substrate on which it is applied. This implies that both the properties of the substrate and those of the stone repair mortar should be known, in order to make the choice of a compatible repair mortar possible. Unfortunately, information sheets of commercial repair mortars do not always provide the relevant properties of the product, making a substantiated choice difficult for the user. Besides, different producers are using different methods to assess these properties, which complicate even more the choice a compatible mortar.

In this research, 4 commercial stone repair mortars, commonly used in Dutch conservation practice, have been characterized in laboratory. Those properties considered as most relevant for the assessment of the technical compatibility of the mortar with the substrate were determined.

The composition of the repair mortars was studied by means of polarizing and fluorescence microscopy on thin sections. The effect of curing was assessed by comparing specimens cured in laboratory (28 days) and in outdoor conditions (28 days and 1 year). The porosity and pore size distribution of the mortar were determined by Mercury Intrusion Porosimetry and related to the measured capillary water absorption and drying behaviour. The hydric dilation both at different RHs and after saturation with water was measured. The flexural and compressive strength of the mortars were assessed, as well as their pull-off strength.

Finally, based on these results an attempt was made to evaluate the compatibility of the mortars with two limestone substrates of different total porosity and pore size.

### **Keywords**

Stone Repair Mortar; Mortar Properties; Compatibility

## **Improvements to Water, Salt-Scaling and Freeze-Thaw Resistances Of Historic Mortar Replication Mixes**

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### **Abstract**

While the long-term destructive potential of liquid water in masonry assemblies is well known, the potential for disruption of saturated mortars exposed to freezing conditions can represent a more challenging and more immediate problem. High permeability mortars, valued in historic preservation work, also have the potential to become rapidly saturated. Projecting elements such as buttress caps, water tables and copings are particularly vulnerable. The problem is exacerbated in situations such as pavements and stairways, where water ponding and saturation prior to freezing may be further complicated by the presence of de-icing salts. Early freeze thaw failures of air-entrained Type O Portland cement-lime masonry mortar on projecting elements at the East Block on Parliament Hill in Ottawa led to initiation of a testing program aimed at evaluating a potential amendment for improving freeze-thaw resistance. Early freeze-thaw failures of several natural cement pavement mortars at the Foley Courthouse in Albany, NY led to a second study, examining a variety of historic mortars in pavement joints, including natural cement, natural hydraulic lime and portland cement-lime mortars. The paper reviews the testing programs and methods, the materials evaluated, the results of testing, and the mortar amendments determined to be most effective in improving freeze-thaw resistance while maintaining high moisture vapor permeability. Saturation coefficient reduction was determined to be the most effective strategy for improving performance in salt-scaling resistance, as determined by ASTM C672: Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals.

### **Keywords**

Historic Mortars; Freeze-Thaw; Polymer-Modification; Air Entrainment

## **Use of ultrafine mafic rocks for the enhancement of carbonation reaction in lime renders**

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### **Abstract**

Lime renders have been used since the earliest periods of antiquity. These composites set and harden through carbonation; the latter is a rather slow chemical reaction between calcium hydroxide and atmospheric carbon dioxide (CO<sub>2</sub>) to form calcium carbonate. In view of the slow rate of carbonation, which is considered a major drawback for the use of lime renders in vernacular and contemporary architecture today, several studies in the literature focus on the mechanism and kinetics of the aforementioned setting/hardening reaction, as well as on the factors affecting its rate. This paper reports on the effect of ultrafine mafic rocks on the carbonation of hydrated lime renders. The latter have been modified using ball-milled olivine basalt and dolerite quarry waste material. Both ultrafine rock additives were added to the mixtures at a percentage of 15 % (w/w in replacement to the lime binder). The results confirm the increase in the carbonation rate of the lime renders under investigation, with the simultaneous transformation of portlandite into calcite; this is corroborated through thermogravimetric (DTA/TG) and XRD analyses, as well as through the phenolphthalein test. The modified end-products are suitable for use not only in restoration projects, but also in contemporary architecture.

### **Keywords**

Hydrated Lime; Mafic Rocks; Waste Material; Ball Milling; Carbonation

## **The impact of elevated temperatures on the properties of lime-based mortars**

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### **Abstract**

The preservation state of historic mortars is closely related to a synergy of factors regarding their type and consistency, their properties, the environmental conditions of each area, as well as accidental actions that may occur during their service life (i.e. earthquakes, floods, landslides and fire). Although exposure to fire and elevated temperatures seems to be a significant decay factor of structures and building materials, the topic is not adequately studied up to now. Despite the fact that many monuments were subjected to fire, as identified by archaeological remnants or historic sources, not much has been done in relation to the behavior of the traditional mortars to elevated temperatures. On the other hand, the adaptation of fire safety criteria on the restoration of monuments is a critical aspect that should be taken into account, in order to determine the post-fire residual strength of historic masonries or to apply effective repair materials, resistant to elevated temperatures (specific applications i.e. churches, buildings of public use).

In the present study, two series of mortar mixtures based on lime-pozzolan and lime-pozzolan-brick dust were manufactured and tested. In the first series the aggregates were natural, of siliceous origin (gradation 0-8mm), while in the second one 40 % of the natural aggregates were substituted by crushed brick of the same gradation. Twenty-eight and ninety days after their manufacture, 2 specimens of each mortar composition were subjected to elevated temperatures (200 °C, 400 °C, 600 °C, 800 °C and 1000 °C), under a specific heating procedure. Subsequently, their physical (volume change, ap. specific gravity, porosity) and mechanical (dynamic modulus of elasticity, flexural, compressive strength) properties were recorded. The test results were comparatively evaluated, in order to determine the performance of the compositions to elevated temperatures, as well as to define the parameters that have to be taken into account when the mortars' resistance at high temperatures has to be studied. According to the results, lime-based mortars present high performance until 800 °C, while at 1000 °C they still maintain their volume stability, which is contradictory to the cement-based mortars' performance as presented by literature. In addition, the presence of brick dust and crushed brick in their matrix enhances their resistance.

### **Keywords**

Lime-Based Mortars; Elevated Temperatures; Properties; Fire-Resistance



## **A grout and mortar system for fine cracks and shallow surface fills in Carrara marble**

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### **Abstract**

Three Carrara marble sculptures in outdoor exposure at the one site, The Royal Agricultural Society of Victoria (RASV), have been conserved using a grouting and mortar system based on the binding capability of lithium silicate to calcitic matrices.

In the first two examples, a full-length portrait pair depicting Victoria and Albert, the Carrara marble had characteristic pitting, assumed to have developed after the portraits were completed or where the artist may have filled the pits with now lost fill material. The pitting was widespread and in the case of Queen Victoria, had substantially disfigured the face due to bio-colonization. The pitting was in the range of 5-20mm in diameter by no more than 3 mm deep, and thus presented a challenge in adhesion and durability. Earlier attempts to fill the pits with lime-based mortars failed after 18 months due to the shallow depth and poor adhesion. Earlier acrylic modified lime repairs the author has applied elsewhere gave improved adhesion and gloss, but with a propensity to crack and not support the parent stones biological regime. Client approval was granted to apply a lithium silicate bound mortar with marble powders and granules as the substantial filler. A small addition of silica flour (20-110 microns) was added to around 5% total aggregate.

For mortars the marble was ground and sieved to pass through a 500 micron mesh, with no lower cut-off size. The key aggregate size determinant was that required to best achieve a matching granular surface appearance. The mortar was applied to the surface with no preparation or other working of the surface, relying solely on the remarkable ability of lithium silicate to bond to the calcitic matrix. The surface was worked to achieve the desired texture and after curing received local tonal adjustments with diluted lithium silicate and pigments provided a satisfactory and durable tonal match. The general body colour of the marble could be achieved through mortar formulation alone, however localized patination meant that a more precise chromatic integration was achieved.

The third work, a life-sized bull with attendant handler, had spent its early life indoors, then removed to the current outdoor exposure in the 1960s. The upper surfaces, to midway down the body had developed an extensive network of very fine cracks, measured to be in the range 0.2-0.4 mm wide at their outer opening. No depth measurements were established.

The decision to fill the cracks was driven by client concerns that the marble was not durable and that it might be better placed in a more protected environment. The conservator proposed that before relocation to an indoor or sheltered location, the object should be treated to determine whether stabilization could be achieved to keep the work outdoors.

A grouting system based on lithium silicate was applied to the cracks, commencing with unfilled lithium silicate, followed by increasingly filled applications.

### **Keywords**

Carrara marble, lithium silicate, fine crack filling

## **An innovative way for testing adhesion of non-structural injection grouts for the stabilisation of historic plasters**

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### **Abstract**

Adhesion is an important property for non-structural injection grouts which aim to stabilise delaminated historic plaster layers. The adhesion of grouts has been tested in previous studies according to standard UNI EN 1015-12 (pull-off strength) on multi-layer panels including, in stratigraphy, support-rough plaster-grout to be tested-fine plaster. This setting may have few disadvantages: it involves multiple interfaces and heterogeneous materials; testing requires drilling which, providing vibrations, can influence the adhesion at the various interfaces, and therefore it may be not fully reliable. The present research tries to improve adhesion testing, proposing a sandwich system (plaster-grout to be tested-plaster) which simulates real situation- conditions for the setting and hardening of the grout and eliminates the need of drilling. The sandwich preparation, repeatable and reproducible, is presented in this paper; the sample was used to evaluate adhesion following standard DIN 1048-2, in which the specimen undergoes an uni-axial tensile force in the two opposite directions until failure. The results were assessed both qualitatively (where the rupture surface occurred within the sample) and quantitatively (the pulling force sustained before rupture). Compared to the panels tested in previous studies, such sandwich testing method (i) reduces the number of different materials involved (4 for the panel vs. 2 for the sandwich system), the number of interfaces, and therefore decreases the potential ambiguity of the qualitative result; (ii) does not require core drilling, which may have an influence on the grout adhesion, particularly for the weak lime-based grouts; (iii) has a specimen with a larger loadbearing surface (50 mm diameter for the panel according to UNI EN 1015-12 vs. 95 mm diameter for the sandwich system), and thus determines a potentially higher accuracy of the result compared to the pull-off test. Replicas were also prepared (support-rough plaster-void to be filled by the grout-fine plaster), in which the grouts were injected and left to set and harden; the replicas were cut and adhesion was visually assessed. The same standard used for the sandwich system, DIN 1048-2, was used to test the cohesion of the grouts separately, i.e. not in relation to the plaster; compressive strength was assessed, according to UNI EN 1015-11. An overall assessment of the grouts regarding their adhesion and mechanical strength is carried out, also in relation to other properties such as porosity. Possible advantages and disadvantages of this alternative method to test adhesion are here assessed and discussed compared to other methods employed.

### **Keywords**

Injection Grout; Adhesion; Testing

## **NHL-based plasters and renders – Assessing the influence of mixing method on workability and hardened mortar properties**

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### **Abstract**

The term “workability” summarizes essential properties of plaster mortars that underlie subjective interpretation resulting in different approaches for its basic description. Generally, workability is influenced by composition and treatment of mortar, which affect the fresh mortar properties. A comprehensive overview of fresh and hardened mortar characteristics allows to generate data, which can be used to predict durability qualities. Thus, within the present study two NHL-based recipes were evaluated with regard to the influence of different mixing methods. Varying the mixing time aims at simulating different workmanship methods on the building site. The properties that were investigated within this study cover mechanical and transport properties of NHL-based plaster mortars in order to estimate the influence of mixing method and water content, respectively, on fresh and hardened mortar characteristics. First results revealed that the interaction between mixing time, water content as well as type of lime hydrate affect the mentioned properties. The gained insights into the interaction between “workability” and resulting durability properties of NHL plaster mortars is beneficial for the understanding of modern and traditional NHL-based systems.

### **Keywords**

Plasters; Renders; Mixing Method; Workability; NHL-based systems

## **Comparing the moisture permeability of limecrete and concrete floor slabs**

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### **Abstract**

Retrofitting impermeable ground-bearing floor slabs to an old building is thought to 'drive' soil moisture up adjoining walls. Historic England has commissioned the University of Bath to conduct research into water vapour and liquid permeation through typical floor slab materials, and their influence on local soil moisture. The project comprises computer modelling to examine the response of soil moisture to slab installation, laboratory tests to analyse moisture movement rates through slab materials, and field monitoring to measure the effect of different slab materials in situ. Initial laboratory tests on two materials, concrete and NHL5-based limecrete, used a bespoke modular soil-slab-air apparatus developed to establish evaporation rates through slab materials. Subsequent material characterisation tests were conducted to compare their microstructural properties and moisture transfer characteristics including mercury intrusion porosimetry (MIP), sorptivity tests, and scanning electron microscopy (SEM). Preliminary results showed the NHL5-based limecrete slab was only marginally more permeable than the concrete slab, suggesting that a NHL5-based limecrete slab might be more effective than a concrete slab in reducing water rise in a wall. Further testing of limecrete mixes and in situ monitoring is proposed to verify these results.

## **Impact of guar gum and chitosan ethers on physico-mechanical properties and durability of natural hydraulic lime mortars**

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### **Abstract**

The addition of guar gum derivatives and chitosan ethers has been studied on the natural hydraulic lime (NHL) mortars. These types of polysaccharides can be possibly used as viscosity enhancing admixtures or water retention agents for the mortars where, in the current state, cellulose ethers are used in the most cases. The water retention capacity is an essential property of mortars to enhance hydration of hydraulic components of NHL and its adhesion to a substrate. The usage of these alternative admixtures in hydraulic lime-based mortars has been scarcely explored, for the cement is the most common binder in our time thus most studies focus on cement based materials. The main focus of this study is laid upon the physico-mechanical properties and durability of the NHL mortars, but the microstructure study is also involved using high pressure mercury intrusion porosimetry and water absorption coefficient due to a capillary suction. The constant water:binder ratio has been used with binder:aggregate volume ratio 1:1 by using siliceous sand and admixtures in doses of 1, 5, and 10‰ of binder weight. The admixtures used were hydroxypropyl- and carboxymethyl- chitosan and hydroxypropyl- and carboxymethylhydroxypropyl- guar. The addition of admixtures decreased the workability of the mortars. Hydroxypropyl- derivatives of the studied biopolymers showed air-entraining function, which is beneficial for the durability of mortars and affects porosity of mortars, as well as water absorption coefficient due to an interaction of air filled pores with capillary network. The biopolymer addition slightly improved initial flexural strength, with the increasing difference for later ages; the only exception for hydroxypropyl guar, which showed slight decrease of flexural strength. Compressive strength was decreased by the addition of biopolymeric admixture in all cases; the degree of decrease was dependent on the type of the admixture and its dosage.

Some of the admixtures showed slightly different behaviour in comparison with their use in aerial lime-based mortars because of different chemical composition of binders.

### **Keywords**

Natural Hydraulic Lime Mortar; Guar Gum Derivatives; Chitosan Ethers; Durability; Strength; Porosity

## **Lime-based grouts for architectural surface repair. Comparison of their performance by using laboratory and field test methods**

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### **Abstract**

Three often used compositions of lime-based grouts, containing lime-pozzolan (L-P: 1:1 by mass), lime-pozzolan-white cement (L-P-WC: 1:0.8:0.2 by mass) and hydraulic lime NHL 3.5 were tested, by using laboratory and field test methods. The properties tested were fluidity, penetrability, volume stability and shrinkage tendency, as well as compressive and flexural strength at the age of 28, 90 and 180 days. For the measurement of fluidity ASTM C939-02 (Marsh cone) and EN 445:2007 (Cone for fluidity test) methods were used, while for penetrability EN 1771:2004 and a relevant field test proposed by the Getty Conservation Institute. Volume stability was measured by following ASTM C940-98A and the relevant field test proposed by Getty, by which shrinkage tendency is also indicated. Shrinkage, compressive and flexural strength were measured in 4x4x16cm specimens, according to ISO 1920-8 2017, ASTM C191-81 and EN 196-1 respectively. Field test methods are rather qualitative measurements but they are simple and easy methods, allowing local conditions and circumstances to be taken into account in a real project.

All grout compositions met the criteria of properties at fresh state, but L-P and L-P-WC performed better in fluidity and penetrability, in particular the L-P composition. Field tests for penetrability showed that L-P and L-P-WC compositions also performed better, characterized as “easy”, while NHL3.5 was characterized as “feasible”. Thus the results from laboratory and field tests were comparable and in harmony. Field test for volume change (by using the mortar cups method) is rather indicative of the shrinkage tendency and could be compared with the laboratory shrinkage measurements. According to field tests the compositions L-P and NHL3.5 were the best and only after 10-12 days cracks appeared at the periphery of the mortar cups. Laboratory volume change measurements for these compositions showed that at the age of 10-12 days were 1.5 % and 0.2 % respectively, while for L-P-WC the volume change was around 1 %. In this case the remarks from field and laboratory testing did not fully agree. Regarding the compressive strength of the grout compositions, strength level of 4.7MPa was achieved by L-P compositions at 180 days, while at 90d the strength of all compositions reached 2-2.5MPa. The cement content in L-P-WC seemed to favor (as expected) the early strength development, as well as the flexural strength development.

Based on the results of all tests, it seems that the L-P composition is more advantageous in relation to the performance at fresh and hardened state. However, more experimental work is needed to validate the field test methods.

### **Keywords**

Lime-Based Grouts; Laboratory Test Methods; Field Test Methods; Fluidity; Penetrability

## **Limestone-filled, hydraulic-lime mortars for historic and traditional fabrics**

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### **Abstract**

Hydraulic limes (HLs) are used in repair mortars as their properties are compatible with historic fabrics. Their chemical and physical compatibility relies on the fact that they contain a mixture of free lime and clinkers (of which belite is the most abundant) that result in materials of high water vapour permeability, lower strength and a greater plasticity than cement-based materials. This paper investigates natural, eminently-hydraulic lime (NHL5). As belite hydrates slowly, the strength of NHL5-based materials will keep rising well after the material has been set into the fabric. This has raised concerns within the conservationists as, in practice, a NHL5 mortar will keep developing strength for up to a year.

This paper replaces NHL5 with increasing amounts of ground limestone to lower the (potentially too) high ultimate strength that can be detrimental for some historic fabrics.

Limestone cements (where Portland cement-PC-is partially replaced with limestone) are widely used however, limestone is not added to HLs. In PC, limestone accelerates hydration acting as nuclei for portlandite and C-S-H precipitation which enhance early compressive strength. It is known that limestone reacts with C3A (aluminat) forming calcium carboaluminate hydrates in place of calcium sulfoaluminates (AFm phases); and that C3S hydration in the presence of limestone results in the formation of calcium carboaluminate hydrates. HLs contain clinkers identical to those in PC. This paper investigates the hydration of NHL5 in the presence of limestone and the impact on strength and hygric properties. The limestone consists of microcrystalline calcite-CaCO<sub>3</sub>- with traces of silica-SiO<sub>2</sub>- and significant porosity. It qualifies as a filler as it comprises 95.8 % CaCO<sub>3</sub> by mass with practically no clay, and 63 % of the particles are under 63µm.

The SEM/EDX results evidenced that the limestone is active in HL: calcium carboaluminate hydrates (resulting from C2S - CaCO<sub>3</sub> reaction) and carboaluminate hydrates (C3A-CaCO<sub>3</sub> reaction) were evidenced on interfaces and in the matrix. The limestone increased early strength: the 10 % replacement enhanced the 28 day strength of the NHL5 (compressive by 36 % and flexural by 56 %). However, after 180 days, all the limestone-filled mortars reached lower compressive strength than the NHL5 mix and, after 90 days, all the limestone-filled mortars reached lower flexural strength than the NHL mix (except for the 10 % with similar strength).The superior early strength of the limestone-filled mortars is attributed to their lower water demand; the increase of early hydrates and their placement: strengthening transition zones and inter-particle links. The strength raise does not affect moisture and vapour permeability, and the composites remain 'breathable'.

### **Keywords**

Hydraulic Lime; Limestone Filler; Hydration; Strength; Hygric Properties

## **Lime based mortars. Relationships between composition parameters and mechanical strength**

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### **Abstract**

Lime based mortars are used for repairing Heritage Structures (HS), since they are considered more compatible to old mortars compared with cement based ones. Apart from hydrated lime they often contain a hydraulic component such as pozzolan, brick dust or small percentage of cement (usually  $\leq 30$  % by mass of the binder content). Hydraulic lime such as NHL3.5 is also used for repairing HS. Lime based mortars are characterized by low strength potential and slow strength development rate. The main parameters influencing the mechanical strength are the type of binder or binding agent, the water to binder ratio (W/B) and the binder to aggregate ratio (B/A). Furthermore, the curing regime analogous to the binder type and compaction or workmanship applied to mortar play also an important role in mortar's strength development. Trying to find interrelationships between mechanical strength and composition parameters, a peer review of literature made and 123 different mortar compositions were listed and categorized according to aforementioned influential parameters. The relevant regulative frames were kept for the curing conditions and compaction and the water content was the required for workability  $15 \pm 2$  cm extension measured with flow table (according to EN1015-3). The data concerned the compressive and flexural strength of  $4 \times 4 \times 16$  cm mortar specimens at 28-d and 90-d ages. The influential composition parameters were the binding system W/B and B/A ratios. The binding systems compared were hydrated lime CL90 (EN 459), hydraulic lime NHL3.5, lime:pozzolan (1:1), lime:pozzolan:brick dust (10-20 % by mass of binder), lime:cement (10-20 % by mass of binder). The evaluation of the results showed that:

- The strength development depends mainly on the type of binding systems and W/B ratio. The stronger the hydraulic content the higher the strength development. The lower the W/B ratio for the demanded workability the higher the strength development.
- The effect of B/A is relatively low in these mortars which have been cured properly up to testing the mechanical strength.
- In the evaluated lime based mortars, the rate of strength development ( $f_{c28}/f_{c90}$ ) ranges from 45-70 %, that means the 28-d strength is not representative of the final strength capacity of these mortars.
- The 28-d flexural strength of these mortars constitutes the 30-40 % of their compressive strength while in cement based the corresponding percentage is 12-15 %.

The paper contributes to lay out the proportioning of lime-based mortars on a scientific basis since diagrams of strength-W/B have been plotted for each of the studied binding systems.

### **Keywords**

Compressive and Flexural Strength; Binding Systems; Strength Development



## **Lime-pozzolan injection grouts with ovalbumin and ethanol added as water-reducing agents: grout design and assessment of the mineralogical evolution**

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### **Abstract**

Non-structural lime-pozzolan injection grouts with water-reducing agents were designed for the stabilisation of delaminated historic plasters in water-sensitive situations, such as plasters contaminated with soluble salts. Water used as suspension medium in a grout can activate and mobilise soluble salts in the porous structure. Salts activation is problematic, as it can potentially cause disaggregation problems related to their crystallisation. Superplasticisers have been extensively used and studied as water-reducing additives to improve fresh properties of cement-based mixtures and, more recently, as additives for lime-based mortars. Another way to reduce the undesirable effects of water has been explored in this study. Two water-reducing components were considered for the fluid mixture: ethanol and ovalbumin. Ethanol, as a partial substitute for water, is a less effective solvent for soluble salts; ovalbumin is a protein found in egg white, which has been studied as a water-reducer, water-retainer and air-entrainer. Both ethanol and ovalbumin have been used in grouting in the conservation of porous building materials. The present study focusses on assessing the influence of these water-reducing components on the grouts' chemical reactions and final composition. Ovalbumin, experimentally verified to act as a water retainer, may lead to slower carbonation and hydraulic reactions, while the presence of ethanol may delay hydration reactions in hydraulic systems such as lime-pozzolan mixes. X-ray powder diffraction (XRPD) coupled with quantitative phase analysis (QPA) by means of the Rietveld method was employed to assess the evolution of the chemical reactions in grout samples 7, 28 and 150 days after their preparation. Microstructural and microchemical analyses were performed by scanning electron microscopy-energy dispersive microanalysis (SEM-EDS) on cross-sections of grouts aged 150 days, to characterise the overall packing geometry of the hardened material and the microchemical profile of the binder matrices. The influence of both ethanol and ovalbumin in the formation of mineral phases and on the material internal structure will be discussed, as well as how this affects other properties of the grouts (e.g. mechanical strength).

### **Keywords**

Injection Grout; Ethanol; Ovalbumin; XRPD; SEM

## **Microstructure of lime pastes with the addition of vegetable oils**

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### **Abstract**

Vegetable oils can be used as water-repellent additives for mortars and coatings for improving their durability when applied in the protection of structures exposed to severe weathering conditions involving water ingress. Previous studies have shown that, besides imparting water-repellence, vegetable oils can significantly affect the microstructure of the lime paste. The influence of the addition of different doses (0.5 and 1.5 wt %) of linseed, stand, and rapeseed oil on the microstructure of lime pastes has been investigated with thermogravimetry (TG), Fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRPD), and scanning electron microscopy (SEM) up to 90 days of age. The wettability of the pastes was analysed by measuring the contact angle of water drops on the pastes' surface. The results of TG showed that up to 28 days of curing, samples with oil were more carbonated than the reference, but after 90 days the calcium carbonate content was lower, namely in the specimens with 1.5 wt % of oil. Stand oil added in the lowest amount had the least effect on the delay of the carbonation reaction. A small amount of aragonite and vaterite was identified with XRPD and FTIR in specimens containing stand and rapeseed oil. Linseed and rapeseed oil added in 1.5 wt. % promoted the development of amorphous phases, which were reduced over time down to a similar amount to that of the pure lime paste. SEM analysis indicated that the pastes with linseed oil exhibited larger crystal size and a more amorphous and porous structure in relation to the reference. Rounded macropores with a mean diameter of 50  $\mu\text{m}$  were observed. The texture of the pastes with stand oil was very similar to that of the reference, apart from the higher number of bubble-like pores. The paste with rapeseed oil showed a higher number of macropores in comparison with the linseed paste. The matrix was also more porous and amorphous than that of pure lime. Only the pastes with 1.5 wt % of linseed and rapeseed oil exhibited hydrophobicity.

The overall results indicate that linseed and rapeseed oil are more reactive with lime than stand oil, thus affecting the microstructure of the paste more significantly. The lower reactivity of stand oil is assigned to the significantly lower amount of C-C double bonds as compared with the other oils. A higher amount of stand oil may be required to achieve water-repellence in the lime paste.

### **Keywords**

Lime; Vegetable Oils; Water-Repellence; Microstructure

## **Effects of natural zeolite addition to lime based render layers for restoration of historical buildings**

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### **Abstract**

Renders, as the most exposed elements of historical buildings, have limited durability and their periodical replacements are necessary. Various studies have been made with a purpose to develop renders with improved resistance to environmental conditions, yet compatible with originally used materials (mortars and masonry). Although traditional renders were applied in several layers with different properties, most of the studies are based on only one layer production and testing. Natural zeolites are crystalline hydrated aluminosilicates with a specific three-dimensional framework structure. They have been used as a pozzolanic addition in lime mortars since ancient times. This paper presents the effects of natural zeolite addition on physical and mechanical properties of lime-based mortars, representing both base and superficial layer of traditional renders. For this purpose, two reference and four lime mortar mixtures containing zeolite were produced. Render for base layer was designed with volumetric lime to sand ratio of 1:3 using aggregate with the grain size of 0/4 mm. Second reference mixture, designed as uppercoat, had volumetric lime to sand ratio of 1:1, using aggregate with the grain size 0/0.5 mm. Other four mixtures were produced with natural zeolite as 20 % and 40 % replacement of lime, for both reference mixtures. Lime putty with 80 % of lime content was used for production of all of the mixtures. Only locally available and produced materials were used in the mix design. Capillary water absorption coefficient, ultrasonic pulse velocity, open porosity, flexural and compressive strength were measured on prismatic samples after 14, 28 and 60 days, and compared with the recommendations for each render layer properties. Mixture with lime:aggregate ratio 1:3 and 40 % of natural zeolite showed improved mechanical properties at the age of 28 days, when compared to reference mixture whose compressive strength reached 1.0 MPa at this age. Compressive strength of both mixtures with zeolite was improved already after 14 days for mixtures with lime:aggregate ratio 1:1. The results demonstrate positive effects of the zeolite addition for both render layers.

### **Keywords**

Natural Zeolite; Lime Renders; Render Layers; Compatibility

## **Formulated lime mortars as a sustainable practice for Built Heritage conservation in Mexico**

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### **Abstract**

In Mexico, restoration lime mortars are developed with regional materials and prepared and evaluated on site. To deepen the knowledge of properties such as the effectiveness of the pozzolanic material and the characterization of the mortar as permeability, resistance to compression, setting speed and resistance to saline attack, provides tools to analyze compatibility with the historical mortars and the correct operation of restoration procedures. Several studies have demonstrated the ability of pozzolans to modify those properties of natural lime mortars. In contrast to the usual practice of restoration in Mexico, where traditionally a minimum of 10 % cement is added to achieve some of these properties, formulated lime mortars are a compatible option in terms of physical and chemical qualities with original lime mortars, meanwhile it is a sustainable practice.

Mortars made with formulated lime mean a considerable reduction of CO<sub>2</sub> emissions in contrast to those made with cement. Also, it is possible to recycle material by using pozzolans derived from bioproducts of industrial processes, waste from the brick industry or natural resources available on the region; this promotes the reuse of materials considered useless and that would otherwise be a polluting waste.

The most important issue of the sustainable nature of these actions lies in considering the plural and social vision as the foundation of the restoration projects include the participation of the communities; In this way, restoration transcends and evolves towards the preservation of constructive culture and local identity. The society inherits its built heritage, but also the traditional knowledge and, if it has an option to participate actively, the recognition of the local constructive culture is promoted through the exchange of knowledge between the multidisciplinary restoration group and the ancestral technology that society guards. This approach is adequate for the reality of the restoration works in Mexico, that is, with limited resources of time and economy; enhancing the virtues of discipline in our country, such as the use of material resources available in the region, promoting a possible compatibility between the materials, the study and the use of accessible materials at a regional level, and economic and sustainable practices.

### **Keywords**

Formulated Lime Mortars; Sustainability; Mexico

## **Efficiency of field test methods for evaluation of non-structural injection grouts in Slovenian conservation practice**

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### **Abstract**

Field test methods for the evaluation of properties of non-structural injection grouts were designed for conservators. The main aim of the methods is fast and easy control of the grout mixture properties on-site. Moreover, adequate workability of the grout in realistic environment, at the particular conservation site, can be evaluated.

The main advantage of the field tests is their adaptability to conditions at the site and application of simple and no expensive test equipment. On the other hand, the field tests are not as precise as the laboratory tests and are rarely reproducible, due to fluctuating environmental conditions and varying substrates.

The aim of the research presented in the paper was to assess the reproducibility and efficiency of the field test methods by Slovenian conservators. A custom-mixed grout and a commercial grout were used in the study. The reproducibility was evaluated within the framework of a workshop where the Slovenian conservators tested each grout mixture with the selected field test methods. Three workshops were carried out, with 15 participants at each workshop. These 15 conservators were divided in 3 groups of 4 conservators. To assess the efficiency and reproducibility of selected field test methods, the Slovenian conservators evaluated each test based on their testing experiences during the workshop. The selected field test methods were injectability with a syringe, flow with a syringe, flow on a plastered tile, expansion and bleeding, wet density, water retention and drying shrinkage with a mortar cup. The results show that the field test methods are efficient in evaluation of the workability of non-structural injection grouts in the field. The results show that the same test performed by different groups of conservators give comparable data in most cases. The main reason for the difference in the test results is probably preparation of the grout mixture and the inaccuracy of the field tests. The conservators chose injectability with a syringe, expansion and bleeding, wet density, water retention and drying shrinkage with the mortar cup as tests that enable efficient evaluation of properties in the field.

### **Keywords**

Field Test; Non-Structural Injection Grout; Conservator; Workshop

## **Evaluation of the rheological behaviour of a natural additive of vegetal origin in restoration lime mortars as an ecological and sustainable alternative**

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### **Abstract**

Recent investigations tend to reinforce the idea of incorporating organic components into the binder matrix of historic mortars. It is debated whether these additives modify the properties of the fresh mortar and if the use of non-biodegradable and irreversible synthetic resins as additives for lime mortars is a viable and ecological option. In this sense, this research has tested how the incorporation of an additive into the binder matrix of lime mortar in variable proportions, which constitute a natural water retainer, modifies its rheological behaviour. In order to study and assess the properties of the fresh mortars, the consistency of the mortars, as well as their bulk density, air content, water retention capacity, and workability were determined.

The conducted research shows that the tested additive modifies all the rheological properties tested so far, and that the mortars' behaviour is conditioned by the additive chemical nature and its percentage added to the mortar (0.5 %, 1 %, 1.5 %, and 2 %). Results show that the tested additive can be classified as a modifier of air content (aerator), as a water retainer and as a modifier of the workability of the mortar and that it guarantees appropriate application benefits: ease of application, long periods of workability which allow for a better finish, and a high water retention capacity, which prevents a quick evaporation and reduces the tendency for the consequent shrinkage cracks. This natural additive is also an ecological and sustainable alternative to synthetic additives.

Knowing the functions of these historic organic additives in mortars is a valuable asset to design new repair mortars and to guarantee a higher compatibility with the original materials in restoration of monumental buildings of patrimonial interest.

### **Keywords**

Compatible Mortars; Historic Additive; Slaked Lime Putty; Properties of Fresh Mortar; Water Retainer

## **Diethyl oxalate-based microgrouts in calcium carbonate systems: formulation, field testing and mineralogical characterisation**

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### **Abstract**

In wall paintings and historic plasters, delamination between plaster layers (or between support and plaster) may vary from few mm to several cm. Such delamination, resulting in lack of adhesion of the layers involved, is typically addressed with injection grouting. The grouting of very shallow delaminated areas, often referred to as 'microgrouting', poses particular challenges: it requires the introduction of a material with suitable flow to travel into the thin delamination, but at the same time with sufficient bulking properties, cohesion and adhesion upon setting. In the conservation of 1730s wall paintings in Malta, a microgrout was required to address a thin delamination (< 2 mm) between limestone (support) and a thin layer (3 mm circa) of painted plaster, composed of lime and calcium carbonate aggregates. Microgrouts were formulated mixing calcium carbonate aggregates with 3 different inorganic binders: nanolimes [Ca(OH)<sub>2</sub>], ammonium oxalate [(NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>] and diethyl oxalate [(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>]. Nanolimes ensure chemical compatibility with the substrate, since their carbonation lead to the formation of calcium carbonate. Ammonium oxalate reacts with calcium carbonate producing calcium oxalate and proved to be compatible with a calcium carbonate substrate. Diethyl oxalate is a relatively new chemical in the field of conservation and has been recently studied for the consolidation of carbonatic substrates in comparison to ammonium oxalate; the final product obtained from the reaction is still calcium oxalate, compatible with a calcium carbonate system, but the product is reported to achieve better penetration than ammonium oxalate. This characteristic may be advantageous in the microgrouting treatment, since it can potentially improve adhesion between grout and substrates.

Field testing was performed in order to define and compare the microgrouts developed in terms of working properties (injectability, flow, setting time) and performance characteristics (shrinkage, cohesion, adhesion). The most promising microgrouts were applied on site, in order to assess their performance in the wall painting system. The diethyl oxalate-based microgrout proved to be the one with the most suitable properties, providing good injectability and flow in a < 2 mm delamination, minimal shrinkage, good cohesion and adhesion. Such microgrout was also tested in the laboratory to characterise the mineralogical phases formed; XRPD coupled with quantitative phase analysis (QPA) by means of the Rietveld method was employed to assess the evolution of the chemical reactions in the microgrout samples 7, 14, 28 and 180 days after their preparation. The microgrout showed to develop calcium oxalate in the first days after its preparation, ensuring therefore good cohesion at an early stage.

### **Keywords**

Microgrout; Diethyl Oxalate; Field Testing; XRPD

## **The initial reactions of lime-pozzolan pastes for conservation of masonry**

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### **Abstract**

Historic buildings constructed from masonry are often in need of conservation. Damage can occur over the years due to a range of different environmental factors. Microstructure plays an important role in understanding how the properties such as vapour permeability, porosity or coefficient of thermal expansion of historic and repair materials influence the longevity of the repair as the physical, mechanical and chemical characteristics of materials need to work together, particularly mortar and brick/stone in the masonry. Mortars of appropriate strength are essential to allow buildings to accommodate movement and thereby reduce the generation of harmful stresses in the masonry. That's why, traditional lime mortars have one tenth the compressive strength of masonry units which have been approximately between 1N/mm to 2 N/mm. However, the strength is not only factor for lime mortars to be a deformable material. The porosity, lime demand, hydraulic set become crucial to be able to enhance distinctive features of mortars for conservation. Throughout history, pozzolans with siliceous and aluminous components have been employed in the presence of water to improve the properties of lime mortar. These pozzolans give a hydraulic character to the mortar through their pozzolanic reactivity. In the mortar, the pozzolanic reaction and carbonation process of lime mortar are crucial to define factors and specify the demands for the lime mortars. Therefore, it is necessary to characterise the properties of pozzolans which can form lime mortars with favourable properties. This study will develop pozzolanic lime mortars of lower strength and higher permeability compared to the stone/brick they will bind. Porosity and pore structure of the enhanced mortars will be investigated through mercury intrusion porosimetry (MIP). The morphology of the enhanced mortars will be analysed through the scanning electron microscopy (SEM). The Chapelle test will be performed to obtain information about the reactivity of the lime mortars enhanced with different pozzolanic materials. Pozzolans such as wood ash, volcanic ash, brick dust, diatomaceous earth, metakaolin as an additive for lime mortar will be tested to reach high durability for moisture and water diffusion and weak compressive strength to facilitate flexibility accommodating movement within the masonry. It is expected that the mortars enhanced with pozzolanic materials will deliver superior durability of the whole masonry unit.

### **Keywords**

Lime Mortar; Pozzolan; Conservation of Masonry; Durability



## **Comparative analysis of the mechanical properties and workability of lime mortars: examples from Hungary and Cyprus**

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### **Abstract**

Lime mortars are widely used in restoration works in many countries, where historic limestone structures are common. The properties of these mortars depend on the quality of lime, the size and composition of aggregate, binder/aggregate ratio, water/binder (w/b) ratio. These parameters control the workability and also the applicability of the mortar. The present study compares lime mortars prepared in Hungary and Cyprus. Local materials were used for the tests; lime binder available in the two countries, limestone sand and quartz sand. The water/binder ratio was 0.850 to 1.587 depending on the mixtures. The consistence was measured following the guidelines given in EN 1015-3, while the flexural and compressive strength of the hardened mortar were tested according to EN 1015-3. The aims of the tests were to compare the flexural and compressive strength, as well as the workability of the mortars prepared in the two countries, and also to determine the factors controlling the aforementioned properties. Using the same type of aggregate and binder, specimens were prepared with different w/b. Lime binders of CL80 and CL90 were used. The highest compressive strength was recorded for the quartz sand containing mortar with 0.850 w/b, but this high strength was linked to a relatively low workability. The tested mortars from Cyprus had a better workability. When limestone sand was used as an aggregate, workability values were better. The results clearly demonstrate that there are significant differences in the lime renders and the ingredients that are used to prepare lime mortars in the two countries. They also point out differences in the techniques of preparation of lime mortars, which lead to differences in the properties of the hardened mortars. There is some dissimilarity in the practical applications of these lime mortars in the two countries.

### **Keywords**

Lime Mortar; Water/Binder Ratio; Strength; Workability

## **Evaluation of the fresh state properties of lime-based grouts through inter-laboratory comparative testing**

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### **Abstract**

The need for evaluating the efficacy of laboratory testing methods concerning the fresh state properties of lime-based grouts, emerged from the activities of RILEM TC SGM and continued in the frame of RILEM TC 277 LHS. To this direction, a collaboration of three laboratories was achieved (AUTH, POLIMI, ITAM) for following a round robin protocol of testing grouts. The experimental study focused on identifying the parameters influencing the fresh properties of different types of grouts (commercial and custom-made). Four types of grout mixtures were manufactured (compositions repeated 3 to 8 times) and tested. The binders used concerned Ledan TB1, PLM-A, NHL 3.5 and lime : pozzolan (1:1) + 1 % w.w. superplasticizer. The fresh state properties measured were fluidity (ASTM C939-02, EN 445: 2007), penetrability (EN 1771:2004) and volume stability (ASTM C 940-98A). The type of mixer (Hobart or high speed), the time of mixing, as well as the temperature of the grout (after manufacture) and the room temperature were recorded. From the comparative evaluation of the results taken from the 3 Labs, the following conclusions were asserted:

- Different types of grout presented different mixing requirements. For example, custom-made grouts (i.e. lime+pozzolan, NHL 3.5) performed better at high speed mixing (up to 8000rps) while commercial grouts (i.e. Ledan TB1) at low mixing speed (Hobart mixer) and time.
- Testing flow time according to ASTM C939-02 Marsh cone, seemed to be reliable for the custom-made grouts, while for the commercial ones, EN 445: 2007 cone was more appropriate.
- Testing penetration time according to EN 1771:2004, seemed to be reliable for all grout mixtures, while the type and gradation of the aggregates in the sand column was of great importance.
- Testing volume stability according to ASTM C 940-98A, seemed to be adequate reliable for all grouts. However, it was better to be referred as volume change, since grouts may present expansion or reduction, according to their type.
- Other parameters seemed to slightly affect the grouts' performance, such as the mixing time and the room temperature.

The scope of the experimental study was to identify the reliability and the applicability of the test methods for fresh grouts. It was concluded that all test methods applied were reliable, in relation to measuring the grouts' performance, but the grout type (custom-made or commercial), affected the mixing way, the rheology and volume changes of the grout mixtures. These differences should be taken into account in measuring the performance of commercial grouts in fresh state.

### **Keywords**

Grouts; Round Robin Testing; Fresh State Properties; Fluidity; Penetrability

## **Investigating differences in the performance of lime-based mortars**

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### **Abstract**

Lime based mortars in general, are commonly considered the mortars used in repairing historic structures, in which hydrated lime dominates in the binding system. They are of low strength capacity (lime:cement 1:1 is not usually included) and consist of hydrated lime (CL) or CL plus a hydraulic component, such as cement or pozzolanic or combination of them. Natural hydraulic lime (NHL) could also be considered as lime-based binder since there are hydraulic constituents in its composition. It is well known that two setting and hardening mechanisms occur in these mixed-Otype binding systems.

In the paper a number of lime based mortars, such as pure CL 90, NHL 3.5 (according to EN 459), CL90-natural volcanic pozzolan (Poz.) (1:1) and CL90-Pozz-cement (1:0.7:0.3) are monitored by applying tests in fresh and hardened state. Physico-chemical properties, mechanical and elastic characteristics have been recorded up to the age of 180 days. Remarks concerning the behavior of them such as the capillary elevation, the rate of strength development, shrinkage deformation are made. Generally, it seems that in spite of some differences the results of testing the properties of lime-based mortars with a hydraulic component form a separate bundle from the pure CL 90 mortars. This shows that many alternative compositions of lime-based mortars can lead to mortars of similar performance.

### **Keywords**

Lime-Based Mortars; Differences; Physical and Mechanical Properties

## **Influence of the substrate on the mechanical characteristics of the applied mortars**

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### **Abstract**

The façades of the historic buildings are mostly left in stone or coated with mortars, usually based on aerial or hydraulic lime.

As they are the most external elements of these constructions, they are the most exposed and the first to undergo degradation, and therefore the first to require maintenance/rehabilitation/replacement.

The choice of replacement/repair mortars for historic buildings requires very special care in order for them to completely fulfill the function for which they are intended. These mortars should not contribute to accelerate degradation of pre-existing mortars and existing supports, they must be capable of protecting and preserving the elements, and should be durable and reversible.

According to the available standardization, the performance of these mortars is performed on laboratory-produced test samples, with standard specimens' dimensions and under standard curing conditions.

However, when mortars are applied over the real supports, their final behavior will not be the same. After application of the mortar to the support, whatever it is, an interface will be formed, ie the interactions that occur after the contact of the mortar still in the plastic state with the substrate will change over time due to the hydration kinetics and absorption of the substrate. The behavior of the mortar, over time, will then be influenced by the characteristics of the support and by the interface that will be created between these two building materials (support and mortar).

The present study is part of a broader investigation within the scope of the IF Mortar project, which aims to analyze the influence of the characteristics of several substrates on the characteristics of different mortar types, in order to predict, more rigorously, which will be the final performance of the mortars after their application.

In this paper, some of the results already obtained in previous experimental campaigns, related to the mechanical characteristics of several mortars applied on different substrates, will be presented.

### **Keywords**

Historic Mortars; Interface; Mechanical Characterization

## **Impact of aggregates on fresh mortars' properties**

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### **Abstract**

The preparation of a mortar mix with high plasticity is important to facilitate the mason's work, since mortars must be easy to place and spread, but it also is essential to obtain mortars with good performance in the hardened state.

Each component (binder, aggregate and water), as well as its relative proportion in the mix, affect the rheological properties of fresh mortars, as well as their microstructure and their physical and mechanical properties.

Since aggregates are, by weight or by volume, the major component of mortars, their characteristics, such as size distribution, shape, mineralogy, and surface texture, have a significant impact on workability, especially on flowability, segregation resistance and density.

An experimental program is presented in order to evaluate the influence of aggregates mineralogy, shape and size distribution on the fresh state of mortars. 30 compositions were prepared using three different binders, five aggregates of different mineralogy and two size distributions. The workability and water retentivity were examined.

The results indicate that there are two main characteristics of the aggregates that affect the water content of a mix. In general, a greater surface area of the mix constituents will also increase the amount of water required to lubricate the surface of the particles and enhance the water retentivity. However, a large surface area can be obtained either by having a fine grain size distribution or a large proportion of sharp, angular particles, which also lead to an optimization of the packing density of solid materials, thus offsetting the effect of the surface area through their own lubricating mechanism. No direct relationship between the mineralogy of the aggregates and the workability of the fresh mortars was observed.

### **Keywords**

Aggregates; Flow; Fresh Mortars; Water Retention; Workability





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