FUNCTIONAL MATERIALS for Cultural Heritage

Fun4Heritage

September 5th-6th, 2019

"University campus" UniBAS in Via Lanera 20,

Matera (Italy).

Book of Abstract

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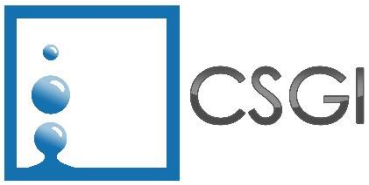

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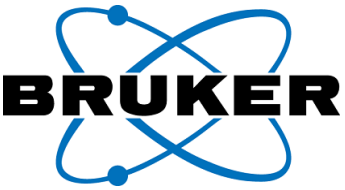

ABOUT

The Workshop "*Functional Materials for Cultural Heritage*" (**Fun4Heritage, Matera, Italy, September 5-6, 2019**) wishes to be an opportunity to present the state of the art in the design and synthesis of functional materials with application in the context of the restoration of *cultural heritage* and *conservative recovery of architectural and artistic heritage.*

The field of hybrid materials and functional nanocomposites is one of the most extensive multidisciplinary research areas of major importance for the archaeological, artistic and architectural sectors, so as to find a natural place in the setting that will see Matera, in 2019, the *European Capital of Culture*.

TOPIC KEYWORDS

- Geopolymers
- Functional Nanomaterials
- Smart Systems
- Antimicrobial Activity
- Controlled Release
- Sol-gel Technique
- Nanocomposites
- Pharmaceuticals
- Nutraceutics
- Innovative Materials

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All speakers and poster contributors will have the opportunity to publish a scientific paper (in English language) based on their presentation/poster. The collection of the accepted contributions will be published as a single issue of IOP Conference Series: Material Science and Engineering, adopting a perpetual Open Access policy.

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-Opening speech-

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New methods and materials for the conservation of modern and contemporary art

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We pioneered one of the most exotic application of soft matter and materials science to conservation of cultural heritage. Art Conservation poses a formidable and exciting challenge to soft matter scientists in two respects. First, the majority of the most performing and environmentally safe cleaning and consolidation agents for artworks are soft matter systems. Second, the interaction of these agents with the artifact involves an exceptionally complicated range of interfacial interactions. Works of art surfaces interacting with the environment are the most prone to aging and decay; accordingly, soiling is a prime factor in the degradation of surfaces, chemical and mechanical degradation are often associated to soiling and lead to the disfigurement of a piece of art. The effects of these processes are usually strongly amplified in the presence of protective coatings (mainly acrylic and vinyl polymers), applied in previous restoration treatments. We pioneered the synthesis and the application of several advanced systems for the consolidation and the cleaning of works of art, as hydroxides nanoparticles, microemulsions and chemical/physical gels. These systems mark a paradigm shift in modern conservation and have been used on classic, modern and contemporary artifacts as Beato Angelico, Piero della Francesca, Picasso, Lichtenstein, Pollock, de Chirico, etc. Finally, I will summarize the main perspectives that this field can disclose for Chemists and Conservators communities.

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Sol-gel-technique

a versatile approach to create functional coatings

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Sol-gel-technique as a topic of the chemical nanotechnology offers far reaching possibilities to create functional coatings. Those functional coatings can be employed for a variety of substrate materials made of polymers, ceramics or metals. Development of inorganic-organic hybrid-polymers following the general sol-gel-approach allows producing coatings with a lot of properties sought-after. Some researchers even speak of the possibility to tailor properties of such coatings. Depending on the specific precursors as well as appropriate reaction conditions like pH-value, temperature, kind of solvent, water content or concentration of the different precursors coating materials can be produced that, e.g., protect against UV-light, abrasion, heat, wetting or microbial contamination.

The basic approach to synthesize nanosols and to apply those will be introduced. Examples will be presented basically but not solely for modification of technical textiles.

-Closing speech-

HE-MAIN-Heritage Maintenance in Smart Cities and Communities: Smart Maintenance, Conservation and Restauration of Cultural Heritage

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HE-MAIN is part of the Italian Ministry of Education and Research (MIUR) programme named <Smart Cities and Communities: Smart Maintenance, Conservation and Restauration of Cultural Heritage>

The subtitle of HE-MAIN is: 'Product and process innovation for maintenance, preservation and sustainable programmed restoration of cultural heritage'

The main objective is: Application of new methods, protocols and chemical products with low environmental impact for a more effective planning of the operations of the cultural heritage restoration.

The increasing of environmental pollution, climatic changes and micro and macro-biological activities are responsible for the growing deterioration in the last decades of the cultural heritage, such as monuments, ancient buildings and statues having historical, archaeological and artistic value.

The visible results of these damages (aesthetic and structural) and decay phenomena are: erosion, soiling (dirtiness) of surface, biological contamination, etc.

Some of the techniques and products that have been used so far in the field of maintenance and conservation of historical stone buildings were often based on the use of materials potentially harmful for the environment and human health. For this reason, the preservation of cultural heritage requires the development of more innovative, effective, long-lasting and cheap cleaning and protection strategies.

The specific aims of the department research group are:

-to develop innovative BIO-remediation techniques based on the use of natural 'organic biocides', with particular attention to the bioactivity of glycoalkaloids extracted from Solanaceae plants;

- to use as BIO-mineralization system native colonizer microorganisms that are able to induce carbonate precipitation and, therefore, structure consolidation.

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Advantages of controlled antibiotics release nanosystems for diagnostic, restoration and conservation of Cultural Heritage

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The increased attention on Cultural Heritages has pushed scientists from different fields to provide a contribution in this direction. Moreover, conservators are aware that investigation of a work-of-art needs to use a scientific approach. Modern nanotechnologies are strategic and they can open unexpected new scenarios on conservation of Cultural Heritages. On one side, advanced techniques based on investigation at nanoscale domain can help to investigate art-works, while a modern set of nanomaterials with potential uses in treatments for restoration and conservation of Cultural Heritage play a relevant role. Perspectives are devoted to obtaining nanomaterials with smart and stimuli responsive features. These characteristics are strategic in many aspects of conservation such as controlled cleaning or development of smart protective coatings and active consolidants. Examples of applications include, but are not limited to, conservations of stone, metals, paints, paper and wooden artifacts. In particular, microorganisms (bacteria, archaea and fungi), in addition to lichens and insect pests, cause problems in the conservation of Cultural Heritage because of their biodeteriorative potential [1-2]. This holds true for all types of historic artefacts, and even for art made of modern materials, in public buildings, museums and private art collections. The variety of biodeterioration phenomena observed on materials of cultural heritage is determined by several factors, such as the chemical composition and nature of the material itself, the climate and exposure of the object, in addition to the manner and frequency of surface cleaning and housekeeping in museums [3]. This study aims to present recent advances in nanotechnology applied to cultural heritage and in particular for the production of dispersions of polymeric-based nanoparticles as an antimicrobial agent, micellar solutions, microemulsions, gels and in order to offer new reliable pathways to restore works of art by combining the main features of soft and hard-matter systems for cultural heritage conservation [4-7].

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New approaches based on nanostructured materials for the sustainable conservation of metal works of art and concrete monuments

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Nowadays, the conservation of cultural heritage based on metals is still a critical issue since these types of artworks are typically affected by complex degradation phenomena. Among them, the corrosion processes have received great attention because they can induce irreversible damages with a consequent loss of the objects. The inhibition of corrosion processes is necessary to preserve the chemical-physical stability of different classes of artworks including metal objects and concrete monuments. For example, the rebars corrosion is one of the main degradation causes in concrete structures.

The identification of new conservation strategies is mandatory to avoid the loss of a unique and valuable cultural heritage based on metals. In this contest, nanostructured materials can offer novel solutions to tackle problems due to corrosion and to achieve long-lasting protection, environmental sustainability and cost-effectiveness by preserving the aesthetic features of artworks.

To face these issues, our research efforts were addressed to the development of nanocomposite coatings able to provide an "active" protection of modern metal works of art [1-2]. A green polymer matrix based on chitosan was functionalized with stimuli responsive nano-containers loaded with corrosion inhibitors. These materials provide a local and tailored release of protective molecules only under stimuli related to corrosion processes, as acid pH or chloride ions. These new materials are more effective and safer than commercial benchmarks and also satisfy aesthetic requirements.

Based on our findings, the stimuli responsive approach was used also for the conservation of concrete monuments within the EU InnovaConcrete project. The first step was the proper selection of corrosion inhibitors to protect the steel reinforcements and then they were confined into nanocarriers able to release them under stimuli related to degradation processes occurring in reinforced concrete. To achieve a long-lasting protective efficacy, the nanocarriers acting as inhibitor reservoir are incorporated into consolidants thus producing innovative multifunctional systems.

After laboratory validation, the optimized products will be applied on European XX century concrete monuments identified as representative case studies that have been characterized to get information about the compositional-structural features and state of conservation. In Italy, the case studies include: i) War Memorials selected for their social values, as symbol of collective identity of European citizens, and ii) Flaminio Stadium as relevant monument designed by Pier Luigi Nervi.
These approaches based on nanostructured materials opens the way to novel scenarios for the conservation of cultural heritage.

This work has been carried out within the NANORESTART project funded by the European Union's Horizon 2020 Research and innovation programme under the grant agreement No 646063 and within the InnovaConcrete project funded by the European Union's Horizon 2020 Research and innovation programme under the grant agreement No 760858.

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A lightweight cement-free alkali-activated slag plaster for the restoration of poor quality masonry structures

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The growing attention to the preservation of the historical heritage has remarkably boosted research in the field of the restoration and conservation, leading the scientific community toward the investigation for new construction materials and techniques. One of the most interesting solution proposed to replace Portland cement is the alkaline activation of ground granulated blast furnace slag (GGBFs) [1]. Alkali activated slag-based mortars are characterized by a very low environmental impact linked to the reuse of industrial by-products which allows a reduction of disposal in landfill, the consume of not-renewable raw materials, the energy consumption and the CO_2 emissions [2] and present excellent mechanical strength values both for short and long age [3]. This paper deals with the development of an innovative Portland-free lightweight structural plaster to improve the seismic performance and the energy efficiency of poor quality stone masonry buildings. In particular, one-part alkali-activated slag-based mortars were manufactured with different lightweight glass aggregate contents to be mechanically compatible with historic stone walls and to serve as a thermo-insulating layer. Results indicate that the Portland-free alkali activated-based plaster is able to provide a 28-day compressive strength equal to 8 MPa and a thermal conductivity of 0.35 W/mK due to density close to 700 kg/m^3. Moreover, problems related to shrinkage, adhesion, micro-cracks and detachments have been resolved by the addition of methylcellulose, modified starch, polypropylene fibers, shrinkage reducing admixture (SRA) and silane-based surface treatment.

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Application of microscopy to the diagnosis of the degradation level of stones used for historical buildings

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Scanning electron microscopy techniques are employed in various types of research and tests, especially in natural sciences, like biology, chemistry or geology, but also in forensic sciences, as well as in material science, architecture, and archeology. Due to a wide range of magnifications, which exceeds the abilities of light microscopes, and high resolution, scanning electron microscopy can produce images that bring a set of interesting data, which would have not been obtained with light microscopic techniques. Moreover, the electron microscopy can be used to analyze the composition of the tested object, including elementary analysis and the distribution of composites. During the lecture, we want to describe some of the techniques employed in the analysis of buildings and heritage. Next, we will present the preliminary results of our tests on the growth of bacteria and production of mineral substances by these organisms on various types of stones obtained for the rupestrian churches of “San Pietro Barisano” and “Santa Lucia alle Malve” in Matera (Italy). The study of the microbial community that can be found on lithic cultural heritage may be useful for the insight of the colonization and deteriorating processes. These processes are tightly connected by a cause-effect relationship which leads to the chemical-physical alteration of the stone matrix and an esthetical change resulting in a loss of harmony and beauty of the artwork.

Synthesis, characterization and antimicrobial activities of new NHC Ag(I) and Au(I) complexes for cultural heritage conservation

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The discovery and introduction of drugs such as: penicillin (1940), erythromycin (1950) and methicillin (1960) has contributed greatly to control bacterial diseases [1]. Subsequently, negative aspects emerged (rapid increase of bacterial resistance, reduction antibiotic efficacy etc.) and hence the need to development of new antimicrobial agents. Metal-based drugs find application both as diagnostic and therapeutic agents. The selection of the ligands used for the synthesis of metal complexes allows modulating of their lipophilicity, solubility and reactivity, which results in a better therapeutic activity. Scientific research has focused its attention on synthesis of new and more effective antimicrobial compounds and Youngs reported silver *N*-heterocyclic carbenes (NHCs) as a new class of antibiotics in 2004 [2]. Several other metal complexes, such as gold derivatives, have been evaluated as potential therapeutic agents [3]. In the light of these knowledge, we have aimed to synthesize, characterize and test as antibiotics, new silver(I) and gold(I) complexes stabilized by NHC ligands (Figure 1).

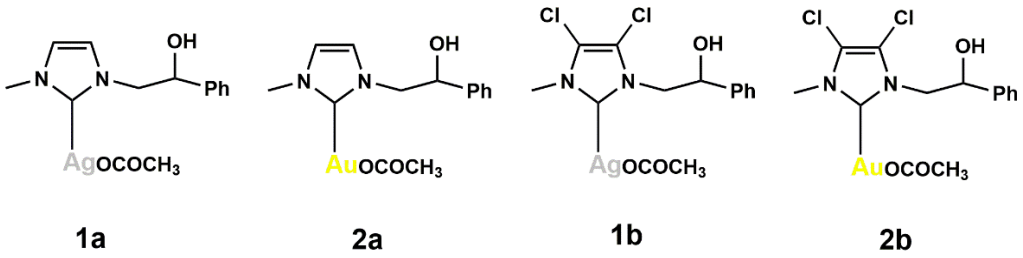

Fig. 1. NHC-silver(I) and gold(I) complexes.

The zone of inhibition test of four complexes against selected bacteria (i.e. enterococcus, escherichia, listeria, pseudomonas) was evaluated. Results showed that they inhibited the growth of the almost all these bacteria.

Since, microorganisms (bacteria, archaea, fungi) induce problems in the conservation of cultural heritage because of their biodeteriorative potential, we have solubilised the complexes in poly(e-caprolactone) and by electrospinnig we have made some membranes. These will then be placed on works of cultural interest and could show an effective, lasting and easily localized action, without the need to move the work.

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Geopolymer production for conservation-restoration using Sicilian raw materials: feasibility studies

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Geopolymers have been recently proposed in built heritage conservation-restoration interventions as substitutes for conventional materials. In the framework of the project Advanced Green Materials for Cultural Heritage (AGM for CuHe), local raw materials, as well as industrial wastes are being used as precursors for geopolymers. Moreover, the project aims to promote the fruition of cultural sites during the restoration campaign, allowing the general public to become familiar with practices and technological advances in the field.

Structural and decorative elements of ancient Sicilian masonries, from black volcanic stone, to white calcarenites and clays, from red bricks to mortars and plasters, reflect the availability of local resources. All these materials are still available today and the feasibility of their use as resources for the synthesis of advanced, smart and high-tech products, is promising in terms of chemical, mineralogical, mechanical and aesthetic compatibility, as required by good conservation practices.

Volcanic aluminosilicate precursors are widely available in the vicinity of Mt. Etna and are problematic, since volcanoclastic materials are considered waste according to current legislation. Also, the Eolian Islands pumices could be used for geopolymers production but the quarrying of these volcanic rocks is not allowed anymore. Clayey sediments, widespread in Sicily, are chosen in order to restore plasters and ancient mortars at a larger scale, in substitution to the traditional Portland cement. Finally, ceramic wastes as bricks and tiles appear to be compatible with the wall substrates and are therefore studied in order to be used as geopolymer precursors for substitution, consolidation and repairing of brick masonries of archaeological interest. The ceramic waste reuse addresses as well sustainability issues.

Moreover, the preparation of fiber-reinforced composite materials represents an ambitious task of the project, with the aim to reinforce structures, and to mitigate the brittle behavior of traditional masonries.

The characterization of raw materials and of geopolymers is achieved by a multi-analytical approach (XRD, XRF, FT-IR, Raman spectroscopy, SEM-EDS, integrity test, mechanical tests) in order to understand the composition of the starting materials, to optimize the formulation design, and to evaluate the chemical stability (including the development of efflorescences) and mechanical properties of geopolymers for use in conservation-restoration intervention. The results of these preliminary tests are a fundamental step before testing the innovative materials on historical buildings, and before their use in conservation-restoration campaigns.

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The role of flax fibres in the cultural heritage field

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Sicilian raw materials for the industrial production of geopolymers

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This study focus on the identification of clay geological outcrops belong to the Sicilian territory - usable as raw materials to produce geopolymeric materials (Davidovits, 1991) - for applications in the fields of industry (Buchwald, 2006) and Cultural Heritage. In this perspective, Sicily can be considered as an "open-air laboratory" for both the wide variety of natural resources and the mining purposes (Oil & Gas; REE; raw materials). In order to obtain mapping and geological information about the sicilian clay, the Piano Regionale Cave of the Sicily Region has been consulted. At the same time, researches based on the available literature have been conducted in order to provide information about the mineralogical-chemical composition of some clay geological Formations.

Specifically, SEM-EDX and XRF analysis of clay from the Messina Province have been carried out with the aim to define the mineralogical and chemical composition. In laboratory, a mixture of siliceous component represented by sicilian diatomite (Tripoli Formation) and slag component made of iron scraps (from Acciaierie di Sicilia Spa) have been tested to produce geopolymers with good results. Since the most production industries of geopolimers are concentrated in the Central-Northern area of Italy, the creation of environmental-friendly (Duxson et al., 2007) geopolymers industries in Sicily, based on the use of "on-site" clay raw materials, can represents a significant innovation/opportunity for the Sicilian territory. [1-3]

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Gold-nanoparticles grafting for potential architectural and Cultural Heritage applications.

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Nanomaterials have been designed to guarantee the long-term restoration and preservation of cultural heritage; in particular, scientists are involved in the cleaning, the deacidification and the consolidation of different kinds of artistic substrates. Cleaning the surface of artworks is an irreversible and delicate intervention that implicates the replacement of shallow layer materials. In some cases, as hard materials such as marble and stone, this can be achieved using laser or plasma techniques. However, these procedures can produce local heating and mechanical shocks, especially for painted surfaces. Chemical methodologies are the favorite options because they may result more ecological, practical and not invasive. Nanoparticles (NPs) are produced using different materials such as metals, metal oxides, carbon, polymers and lipids.[1] The potential of NPs for conservation of buildings has been established for the consolidation of decayed materials, production of de-polluting surfaces, self-cleaning or biocide surface enhancement to reduce biodeterioration. [2] Suspensions of opportune NPs, even dispersed in polymeric matrices, have been tested and employed for their efficacy against biofouling of building wrappers.

The aim of this work is the green synthesis and preparation of colloidal systems tailored to the preservation and consolidation of wall paintings, cements and stones.[3] In particular, the present study will show the grafting of gold-nanoparticles on three different substrates: 1) montmorillonite clay, which was previously functionalized with a thiolic group, useful for application in the restoration and conservation of building materials; 2) glass surface, for the restauration of glass artifacts; 3) fabrics, for 'wearable materials' properties implementation. Unfortunately, under particular conditions, deterioration of an artwork may induce the nanoparticles release into the environment; in order to inhibit this undesired phenomenon, the colloidal nanoparticles could be stably bonded to the treated artwork (based on clay, glass or textiles), by means of a grafting sol-gel polymeric 3D-matrix. [4]

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Advanced Green Materials for Cultural Heritage: from research to the tourism market

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AGM for CuHe project aims at developing eco-friendly, smart and high-technological products for conservation and restoration of Cultural Heritage, thanks to a complex interaction between universities and industrial partners. The outcomes of the project are expected to be in terms of novel restoration products to be applied on built heritage. These materials are expected to be sustainable in terms of carbon footprint and use of raw materials, but also compatible with the existing structures, durable and aiming at safeguarding built heritage in high seismic risk areas thanks to adequate mechanical properties. The development of such materials, their use and impact on built heritage and its fruition involve academic and industrial partners, as well as decision makers. The target objectives of AGM for CuHe are in agreement with the European guidelines, in which actions focused on improving the impact of Cultural Heritage in economy, tourism market, societal challenges and employment rate is widely encouraged. The achievement of the target objectives is supported by: the highly qualified human resources and the presence of well-equipped laboratories at a national level for the development of research in geology, biology, chemistry, engineering, physics, material sciences; the support of economic and juridical expertise for the sustainability assessment of the project; the presence of a well-experienced industrial leadership assured by companies involved in management, production, and restoration actions. Moreover, AGM for CuHe is expected to improve the employment rate, especially in southern Italian regions, with the formation of new experts in the field of extraction and recycling of wastes for the production of innovative materials; production of nanoparticle-based products for restoration; standardisation of protocols for restoration; innovation in cultural heritage fruition. Finally, AGM for CuHe will have an impact on the tourism industry, by favoring the fruition of restored cultural heritage by using innovative materials and promoting the attractiveness of the built historical environment thanks to advanced tools. Tourists will have the possibility to follow all the research, experimental and applicative phases of the project, for a more responsible and conscious fruition of culture and cultural heritage.

The research is supported by the AGM for CuHe project. PNR 2015-2020. Area di Specializzazione "CULTURAL HERITAGE". CUP E66C18000380005.

Polymeric Microcarriers to Deliver an Antioxidant and Chemopreventive Extract (HSE) From Hazelnut Shell Waste

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A large volume of waste and by-products are generated by agro-industrial processing and their treatment and disposal is becoming increasingly difficult. The recover, re-use and re- and up-cycling of wastes in a circular economic vision is a key global issue in the light of the demand of sustainability. Chemistry-led innovation for sustainability is provided by the present research with reference to a research on agro-food wastes and by-products from Hazelnut, *Corylus avellana* L.; in fact, shell and skins are the main by-products of the kernel industry processing [1].

The research was aimed to design and develop new stable and bioactive particle powder systems as ingredients for nutraceutical and cosmetic industry.

Extraction and chemical and biological studies of the wastes showed an interesting composition in phenolic derivatives with antioxidant, antimicrobial and chemopreventive activities, the last as an inhibitory effect on human melanoma and cervical cell lines, inducing apoptosis by caspase-3 activation [2].

The extract HSE was loaded and carried in natural-based polymeric matrix by spray-drying. The tandem polymeric matrix was based on L-proline (P) as loading carrier, Hydroxyethyl cellulose (HEC) and Pectin as additional coating co-polymers. Moreover, in order to improve the solubility of HSE raw material, lecithin (L) and ethanol were used in the feed suspension. The study and setting of process parameters such as polymeric phase concentration and the hot-cold-hot method used led to satisfactory results, showing a high loading efficiency (LE) (95%). Particles resulted well-formed and free of fractures with unaltered antiradical activity, and an improved in vitro water dissolution profile. The developed method is suitable to transform HSE raw material in a stable micro-particulate powder to be enclosed in a topical or oral dosage form as chemopreventive and antioxidant ingredient.

Active ingredients based on food wastes/by-products combined with biodegradable and biocompatible polymers with coating or film-forming ability, appears an attractive approach for the up-cycling and valorization of wastes addressing the formulation of health products.

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Thalidomide Correlated Compounds as new promising tools for breast cancer treatment

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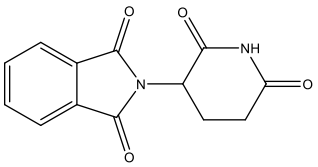
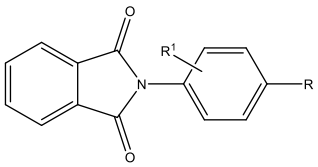
Thalidomide is an old well-known drug firstly used as morning sickness relief in pregnant women and then withdrawn from market because of its dramatic effects on fetal normal development. Over the last few decades, the interest in this old drug has been renewed, because of its efficacy in several important disorders as, for instance, multiple myeloma, breast cancer, and HIV-related diseases. It became clearer that thalidomide exerts multifaceted properties, directing the efforts of many research groups toward the synthesis of several derivatives and the study of their effects, mostly as new ant-cancer agents [1,2]. A recent work on thalidomide correlated compounds [3] allowed us to select active compounds which are very effective in inducing cancer cells death by triggering TNFα-mediated apoptosis. The most active compounds were able, as well, to reduce drastically the migration of breast cancer cells, through the regulation of the two major proteins involved in epithelial-mesenchymal transition (EMT), vimentin and E-cadherin. They diminished the intracellular level of vascular endothelial growth factor (VEGF), primarily involved in the promotion of angiogenesis that sustain tumor progression. Following this work, a small library of phthalimide derivatives were synthesized and studied for their activity on breast cancer cells, particularly on the estrogen-positive (ER+) MCF-7 and the triple negative MDA-MB-231 cells.



thalidomide



R = Alk-O, ArO, Hal
R^1 = H, Alk-O, Hal

In this study we report preliminary and promising results of these new compounds, as new antitumor agents.

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Morphological and structural properties of wood by Micro-imaging NMR and FTIR Spectroscopy.

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The use of non-invasive and non-destructive techniques is of great importance in the cultural heritage studies for investigating its composition and state of preservation without compromising the integrity of the material. In this work, an innovative approach based on Nuclear Magnetic Resonance (NMR) was adopted. Results were correlated with the standard FTIR spectroscopy characterization. By magnetic resonance microimaging (μMRI) methods, images characterized by a spatial resolution of approximately 100 μm or smaller are obtained. These images are particularly useful for investigating heterogeneous and porous systems soaked with water. Specifically, μMRI techniques are able to retrieve high resolution information about the mean diameter, the shape and the distribution of microfeatures of porous systems. Moreover, it is possible to obtain indirect information about the density of material and its submicrostructure (smaller than image resolution) by measuring several NMR parameters, such as relaxation times T1, T2, T2*, diffusion and perfusion coefficients D and D*[1]. On the other hand, FTIR spectroscopy allows to estimate an eventual gradient of water absorbed (more precisely, hydroxyl groups, OH) between the surface and the inner part of woods, to understand chemical changes in wood forming tissue, in turn, providing unique insights into wood formation or aging [2]. For the first time to our knowledge, we have correlated the results coming from micro-NMR and FTIR to systematically study the morphological/structural construction and/or metabolism of wood wall matrix components.

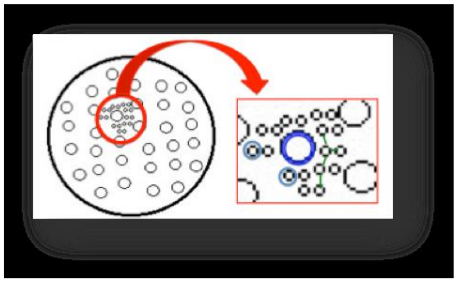

Fig. 1 - free water and weakly bound water (vessels) was obtained by NMR; bound water (cell wall) was obtained by FTIR.

This work was supported by European Union FSE Grant.

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Total phenolic flavonoid contents, antioxidant and antitumor activities of *Annona cherimola* leaves

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Annona cherimola Mill. is a subtropical fruit tree, which belongs to the Annonaceae family Cherimoya is native from the inter-Andean valleys of Peru and Ecuador but it is cultivated as well on the coast of Granada-Malaga (Spain) and South of Italy (Calabria and Sicily). Recent studies have highlighted that the extracts of *Annona cherimola* leaves exhibited several health benefits, which in general are attributed mainly to its bioactive composition; flavonoids were the main sub-class of phenolic compounds followed by phenolic acids [1]. They play an important role in health because of their numerous biological properties [2-5]. The present study investigated the cherimoya leaves, picked from trees grown in Reggio Calabria areas, for their total phenolic and flavonoid contents, and for their biological activities, in order to provide basic support for the functional uses of these by-products.

In this context, the lyophilized and pulverized leaves were subjected, separately, to extractions with three different solvents: water, methanol and ethanol. Methanol and water yields were the highest ones. The dried extracts were tested with the Folin Ciocalteu, flavonoid and DPPH assays. Ethanol extract exhibited the highest amount of flavonoids and phenolic compounds followed by methanol extract, whereas total phenolic and flavonoid contents resulted lower in water extract. Ethanolic and methanolic extracts showed a significant antioxidant effect. Moreover, all the extracts are effective in reducing the proliferation of two cell lines of breast cancer, MCF-7 and, above all, MDA-MB-231. In particular, the ethanolic extract provided the better cytotoxic profile. As a conclusion, the obtained results present *Annona cherimola* leaves as potential sources of bioactive compounds with applications in the pharmaceutical field.

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A novel spectroscopical approach to assess the synthesis and characterization of BPA-free epoxy resins designed for stone conservation

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The development of stone conservation products is an active field of research since the achievement of environment-friendly multifunctional products able to ensure, at the same time, long-term key properties such as consolidating and hydrophobic effects, is a hard task. In this sense, novel products derived from Bisphenol A (BPA)-free epoxy resins have become increasingly appealing due to the easy tailoring of their physical, thermal and chemical properties coming from their combination with compatible inorganic precursors and/or their nano-reinforcement [1]. Pursuing this challenge, a cycloaliphatic diol derived from the isobutyric acid, with minor associated health and environmental concerns than phenolic-based ones, was selected to synthetize the corresponding BPA-free epoxy precursors, 2,2,4,4-tetramethyl-1,3-cyclobutanediol (CDBO) (Figure 1). To characterize the product, as well as to follow and assess the clean-up and curation process, FT-IR, ATR-FTIR and Raman spectroscopies were employed.

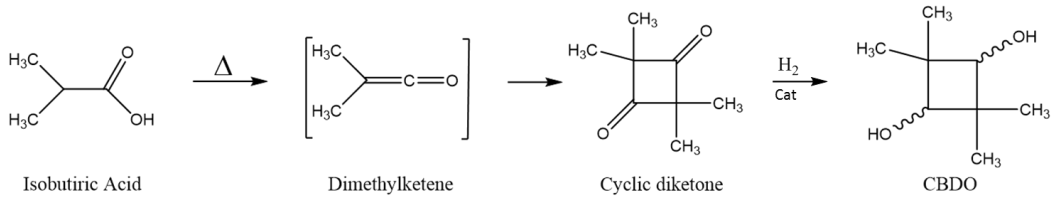

Fig. 1. Synthetic route of CDBO from isobutyric acid

H-NMR and ^{13}C-NMR were also used to determine the structure and purity of the obtained epoxy resins. Then, the ratio epoxy-amine used for its thermal cure was optimized with the help of ATR measurements [3]. The thermal behavior of the most promising product, to be exploited as stone conservation material, was studied by TGA and DSC investigations.

This work has been financially supported by the project PHETRUM (CTQ2017-82761-P) from the Spanish Ministry of Economy and Competitiveness (MINECO) and by the European Regional Development Fund (FEDER). Pablo Irizar also acknowledges his grant from the MINECO (PRE2018-085888).

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Role of Vitamin B12 and Folate, in patients with hyperhomocysteinemia, in the conversion from Mild Cognitive Impairment to Dementia

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Homocysteine (Hcy), a Sulphur containing amino-acid present in all cells, has been identified as a probable risk factor for the human health if accumulated in the blood because of being endowed with a high pro-oxidant power. The homocysteine values in the plasma are considered physiological when they are in the range of 5-15 μmol / L. However, rare genetic polymorphisms (CBS and MTHFR gene) can cause severe hyperhomocysteinemia, with values of 100-200 μM/L or mild to moderate hyperhomocysteinemia with values of 10-100 μM / L, very common in elderly people. Hyperhomocysteinemia (Hhcy) is considered an independent risk factor for the development of cardiovascular, cerebrovascular and peripheral vascular systems. With genetic factors, which are the main cause of the imbalance of homocysteine (Hcy), nutritional and hormonal factors contribute to raising their levels in the body. Hyperhomocysteine is a surrogate marker of the group vitamin B deficiency and a neurotoxic agent. An integration of B vitamins (in particular vitamin B12 and folate) has an efficacy in reducing the well-validated homocysteine; therefore, this could be an easily modifiable risk factor for cognitive impairment and dementia. The objective of this study was to compare homocysteine levels in subjects with MTHFR C677T_T and MTHFR A1298C_C, to verify if there was an increased risk, in the presence of hyperhomocysteinemia, of converting from Mild Cognitive Impairment to dementia and whether the treatment of these subjects with Vitamin B12 and Folate lengthened the conversion time. In our opinion, it would have been very interesting to be able to verify if patients not treated with Vitamin B12 and Folate converted more quickly than those treated but, considering it ethically incorrect, we could not pursue this path.

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Qualitative characteristics, nutraceutical profile and shelf life in soft drinks

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The objectives of the study were two: the first, the measurement of antioxidant capacity and, the second, the link between oxidative processes and the Shelf Life of citrus products.

The study of three different types of matrices, was used to evaluate the qualitative characteristics, the nutraceutical profile and the shelf life of citrus-based soft drinks (lemon and orange).

Beyond the routine analyzes (carried out when the concentrate and drinks were produced, at which point the Shelf Life begins) we evaluated the antioxidant capacity through the assays: DPPH, ABTS and the Folin-Ciocalteau method.

Furthermore, the use of a climatic chamber was possible to carry out an ASLT test (it provides for accelerated conditions on the samples) in order to evaluate the Shelf Life of the matrices being studied.

The excellent results obtained have confirmed the bibliographic studies and our hypotheses. Both matrices, while losing their antioxidant capacity during the Shelf Life however, maintain an always positive trend. Unfortunately, through colorimetric analysis (also perceptible sensorially), a browning of all the samples was recorded, especially in the lemon matrix.

Antimicrobial activity of new *N*-heterocyclic carbenes metal complexes

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New complexes of silver and gold metals containing N-heterocyclic carbenes (NHC) were synthesized and fully characterized by NMR and mass spectroscopic methods [1]. The analogous complexes have as counterion the acetate group because acetate salts are completely soluble in the physiological environment and this could facilitate the bioactivity. The antimicrobial activity and the minimal inhibitory concentration (MIC) were evaluated against selected bacterial strains of significant importance for human health and food production, by using the agar well diffusion assay [2]. A total of thirty-two Gram-negative and Gram-positive bacteria were employed as screening microorganisms to determine the antimicrobial effect, the action spectrum and the antimicrobial effectiveness of each extract. Results showed that all complexes provided a good antimicrobial activity against a wide range of bacteria, with a higher effect of Au-complexes than Ag-complexes. The inhibitory activity showed a good efficacy on the growth of gram-negative and gram-positive bacteria in presence of different extract concentrations; in particular complexes were found very effective against *Enterococcus* spp. strains, *Listeria monocytogenes* and *Staphylococcus xylosus* with a high antimicrobial activity. Overall, the complexes with acetate group as counterion proved a better inhibition activity, probably due to a greater solubility.

The data underlined the importance of these new synthesized metal complexes, by making them potential candidates to be used as antimicrobial agents.

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Cyclodextrin matrices as photoprotective systems of anti-inflammatory agents for topical use

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Photostability studies applied on topical formulations containing anti-inflammatory drugs have confirmed the sensitivity to light of many of these drugs [1]. For this reason, their formulation in cream or gel is often avoided in favor of other forms, such as tablets or suspensions.
In this work, the behavior of nabumetone (NA), (4- (6-methoxy-2-naphthyl) butan-2-one) in aqueous solution was tested, revealing the 6-methoxy-naphthalene-aldehyde as the main photoproduct [2]. Photodegradation of NA was then investigated in both liquid and gel formulations. The experiments were monitored by spectrophotometry and the data processed by Multivariate Curve Resolution (MCR), able to estimate spectra and concentration profiles of the components involved in the kinetic process. Photoprotection of NA was tested by entrapping the drug into cyclodextrin matrices (Fig.1). The complexes prepared in liquid and gel formulations were exposed to forced degradation to test their ability in improving the light-stability. Several type of cyclodextrins were evaluated to increase the encapsulation percentage of the drugs, showing an increase of the drug solubility in water in the order HPβCD>MβCD>βCD. No significant photoprotection of NA from the light was measured in liquid formulation. On the contrary, gel formulation containing the βCD-complexes showed relevant stability respect to the liquid and gel formulation of the drug, maintaining a 90% of the starting concentration, after 300 min of light exposure set at 350 W/m^2 of irradiance power.

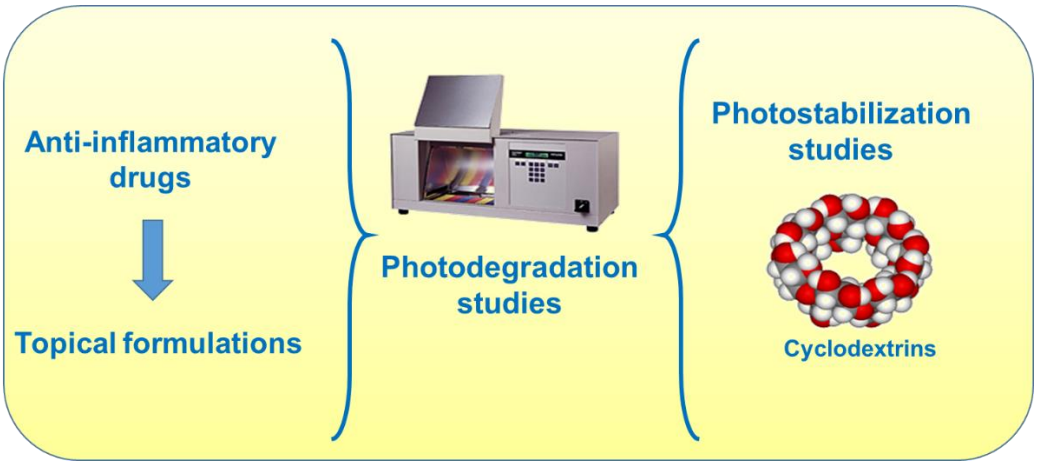

Figure 1. Inclusion of FANS into cyclodextrins.

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Fosfomycin analogues as potential antitumor agents

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Fosfomycin represents a broad-spectrum antibiotic belonging to the class of phosphonic with a bactericidal activity against both gram-negative and some gram-positive microorganism. The aspect that makes it particularly interesting is the minimal occurrence of side effects which consist only of: rash, headache, nausea, rhinitis, vaginitis, etc. [1]

In addition to bactericidal qualities, literature data report that several antibiotics, including Fosfomicyn, are possible inhibitors of DNA synthesis. This characteristic could lead them to become efficient antitumor agent. [2]

Actually, a lot of study demonstrates that the co-administration of cis-platin (a well-known anticancer drug) and Fosfomycin is a good approach to decrease the onset of adverse effects due to the use of cis-platin alone such as ototoxicity and nephrotoxicity. [3] For the reasons abovementioned (overcoming cis-platin's toxicity and verify if there's the possibility to create innovative and safe anticancer drugs) the aim of our work was to synthetize fosfomycin's derivatives. These analogues have been prepared with a simple and cheap one pot reaction using dimethylphosphonate and appropriate α-halo-ketones in the presence of sodium methoxide (CH3ONa) in methanol. However, in order to establish their potential antiproliferative activity against tumour cell lines and possible prevention toward non tumoral cells *in vitro* assays will be carried out.

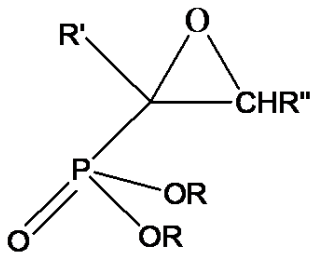

Fig. 1: General structure of Fosfomycin's analogues

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Design and development of hybrid coatings for conservation and restoration of cultural heritage

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Geopolymers are inorganic materials that form long-range, covalently bonded, networks; they are materials similar to ceramics, whose components are mainly aluminum and silicates Any inorganic source of silica and alumina, dissolved in the alkaline solution, acts as a precursor of geopolymers and therefore lends itself to geopolymerization. The type of clay most commonly used in the synthesis of geopolymers is based on kaolinite. A green method to implement these properties consists of incorporation of coupling silane agents[5] and organic fillers during the geopolymerization process. The methods of functionalization of the geopolymers shown in this work are: (i) the treatment of the geopolymeric surface, post-drying, through the application of the sol-gel directly on the monolith, for a modification of the surface properties; (ii) the pretreatment of the geopolymeric mixture, through the addition of the alkoxysilane which can induce a homogeneous consolidation of the molecular structure, implementing the properties of the entire monolith. In this work we show the design and development of hybrid coating sol-gel formulations to apply on geopolymer surface to improve characteristics such as mechanical strength[6], chemical inertness, heat resistance and porosity for possible use in the restoration and conservation of cultural heritage. The surface treatment is carried out using different alkoxysilanes[7], which differ according to the length of the hydrocarbon chain and the presence of halogen atoms or epoxy groups. Hydrophobicity and antifouling tests were performed on these formulations.

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Coupling of nanomaterials and bacteria for development of new strategy of bioremediation: a perspective

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The development of new eco-friendly, sustainable and economically-sound technologies to clean up the environment from contaminants is one of the most challenging task of actual biotechnology. In the last years, different physical, chemical and biological technologies have specifically focused on actions to protect and restore environment from xenobiotic (e.g. oil hydrocarbons).
Bioremediation techniques have been proved to be a new an effective method for cleaning up residual pollutants in a variety of environments and a quite flexible management option to be implemented, also at a large scale, in marine ecosystem. Stimulation of the growth of indigenous microorganisms (biostimulation) or inoculation of foreign oil-degrading bacteria (bioaugmentation) were recognized as effective measures for accelerating the detoxification of a polluted site with a minimal impact on the ecological system [1]. Although bioremediation provides an excellent and flexible recovery strategy for different pollutants, it is poorly effective when dealing with high concentration of the pollutants and xenobiotics or refractory compounds, causing unsustainable treatment efficiencies and recovery time [2]. In this context the development of nanotechnology and the integration of the use of Nanomaterials (NMs) and Nanoparticles (NPs) can represent [3] a promising innovation able to enhance the bioremediation action and to go beyond the actual limitations for in-situ or ex-situ applications. The combined approach appears to embrace a wider range of potential applications with reduced costs and increased control of undesired byproducts formation [4]. In this work different strategies have been tested for evaluate the efficiency of recovery of oil polluted seawater.

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Sustainable development of geopolymer technology for cultural heritage application: synthesis set-up and morphological characterization

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The term "geopolymer" describes a wide variety of natural or synthetic composite materials showing a polymeric structure. They are materials similar to ceramics, whose components are mainly aluminum-natural silicates. The geopolymerization takes place in three phases: dissolution of the aluminosilicate source, polycondensation of aluminosilicate oligomers and finally precipitation of geopolymeric particles, which form a gel. The sol-gel synthesis is an eco-friendly approach to functionalize geopolymers, without high temperature treatments, perfectly in accordance with the principles of circular economy and green chemistry. [1] The present work concerns the modification of the geopolymer at the chemical and nanostructural level, through the condensation process in alkaline conditions [2]. The alkoxysilane agents may be chosen for the implementation of specific properties of the geopolymer materials that allow the production of various types of coatings, such as protective coating, reflective or anti-reflective coatings, refractory linings, coatings with controlled porosity, all these properties may result determining in the restoration of cultural heritage. The morphology of the geopolymer surface depends on the degree of cross-linking of its molecular structure and varies according to the quantity and type of alkoxysilane added. The methods of functionalization of the geopolymers described in this work are the following: a) the treatment of the geopolymeric surface, *post-drying*, through the application of the sol-gel directly on the monolith, for a modification of the surface properties; b) the *pre-treatment* of the geopolymeric mixture, through the addition of the selected alkoxysilane, can induce a homogeneous consolidation of the molecular structure, implementing the properties of the entire monolith.

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Enhancing photophysical properties mixing dyes and silica

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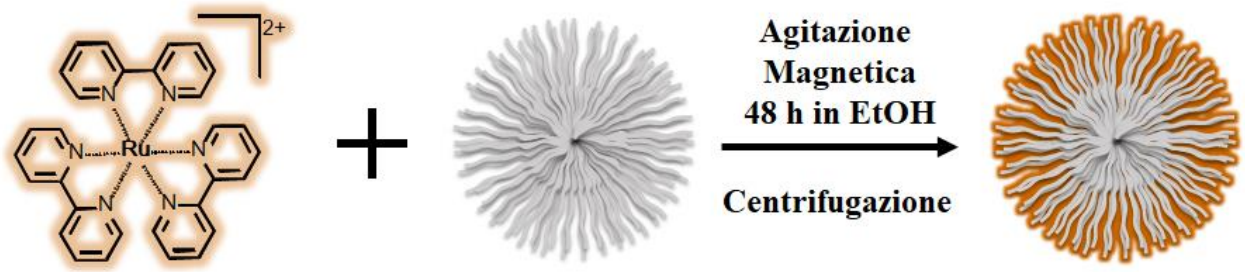
Because of abundance, distribution and ease of formation, singlet oxygen can be predicted as one of the most disruptive agents of degradation. The colored nature of fine art works makes them virtual generators of singlet oxygen which is the activated excited state of molecular oxygen and as such, potentially more destructive than the fundamental triplet state oxygen. Besides being the cause of fading of the color of the pigment itself, singlet oxygen may be the cause of damage of the binding materials for the pigments and of the varnishes composing the artwork, greatly contributing to its final deterioration. For example, in a polymer case, by photo-oxidation reaction induces a chemical change that reduces the molecular weight and the material will become more brittle, with a reduction in its strength.

So, a way to reduce the interaction of pigments (or of binders) with oxygen is needed. To this aim a new silica scaffold, able to enclose dyes and to protect them, have been prepared. In order to evaluate the performance in terms of photo-protection of these new materials, new hybrid systems based on polypyridine Ru (II) complexes within fibrous silica nanospheres have been prepared and their photophysical properties studied into details.



Life cycle assessment of geopolymers: case study of green industries in Sicily

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Geopolymers are materials obtained from alkaline activation of natural raw materials (e.g. kaolin/metakaolin) or secondary raw materials (industrial wastes like fly ash or slag) and are used to make binder systems as alternative to Portland cement besides for restoration applications in the field of cultural heritage. Portland cement manufacture causes high emissions of carbon dioxide (5- 10% of global anthropic emissions) and its production process needs a large amount of energy (mainly from fossil fuels)[8]. Geopolymers production, instead, reduces the consumption of primary raw materials and the use of waste for the geopolymer production could contribute to relieve the waste disposal system. This study aims to provide an evaluation of the complete life cycle of geopolymeric materials - from production until disposal. Moreover, the environmental impact assessment of the industrial production of geopolymers in Sicily (Italy) has been also carried out.
The method usable to evaluate the potential impact on the environment during the geopolymer production is named as Life Cycle Assessment (LCA). Life-cycle assessment (LCA, also known as life-cycle analysis, eco-balance, and cradle-to-grave analysis)[9] is a technique to assess environmental impacts associated with all the stages of a product's life from raw material extraction through materials processing, manufacture, distribution, use, repair and maintenance, and disposal or recycling. LCAs can help avoid a narrow outlook on environmental concerns by: 1) Compiling an inventory of relevant energy and material inputs and environmental releases; 2) Evaluating the potential impacts associated with identified inputs and releases; 3) Interpreting the results to help make a more informed decision.
In conclusion, evaluation of the LCA results will enable a Sicilian cement industry (Edil Ponti SCARL) to produce geopolymeric materials by building a pilot plant. Preliminary results of our study show, for different scenarios, a considerable reduction of CO_2 emissions and of energy consumption but also a general improvement of the environmental indicators.

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Calabrian kaolin as raw material to produce geopolymers for application in the field of cultural heritage

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This research is focused on the use of the Calabria kaolin to produce geopolymersfor application in the field of cultural heritage. Kaolinis a phyllosilicate mineral, consisting of alternate layers of silica and alumina in tetrahedral and octahedralcoordination, respectively. Geopolymers can be considered as green materials because they can be synthesized from natural resources and their chemistry are environmentally friendly without the production of CO_2 emissions. They are inorganicpolymeric materials obtained by mixing of a dry solid (aluminosilicate) with an alkalinesolution and otherconstituents, ifnecessary. The raw material must be rich in silicone (Si) and aluminium (Al), and itwould be a naturalmineralsuchaskaolin.Kaolin-based geopolymers were produced by the alkali-activation of kaolin with an activator solution (a mixture of NaOH and sodium silicate solutions).The obtained geopolymers are materials of great interestfor their peculiarphysical and chemical properties (thermal insulation, fireresistance,low-tech building materials, decorative stoneartifacts,etc.).
In order to provide information on the Calabrian kaolin geological surveys have been carried out. The results put in evidence that kaolin was reported inthe Serre Massif and Longobucco-Bocchigliero area belonging to the Calabria-Peloritani Orogen (CPO; Perri et al., 2008; Calcaterra and Parise, 2010).The Serre Massif occupies the central position of the CPO and it mainly consists of a crystalline basement made up of Variscan metamorphic and plutonic rocks mainly granitoids with subordinate gneiss and phillites.The weathering processes of granulitic rocks from the Serre Massif transformed feldspar and muscovite in kaolin (Calcaterra and Parise, 2010). In the Longobucco-Bocchigliero area,in Northeastern Calabria, kaolin are reported into the Mesozoic sandstones. The sandstones are mainly quartzarenites, which display heterogeneous distributions of authigenic quartz, kaolin, illite, feldspar,and minor carbonate cementation(Zuffa et al., 1980).
In conclusion, the results of our study demonstrate that the calabrian kaolinitic natural materials represent a good raw material for the production of eco-friendly geopolymers.

- **Study on Properties and Morphology of Kaolin Based Geopolymer Coating on Clay Substrates** - C. M. Ruzaidi1, a, A. M. Mustafa Al Bakri1, b, M. Binhusain2c, M. S. Siti Salwa1,d, A. Alida1, e, M. T. Muhammad Faheem1, f, S. S. Azlin1, g
- **Clay Mineral Assemblages and SandstoneCompositions of the Mesozoic Longobucco Group, Northeastern Calabria: Implications for Burial History and Diagenetic Evolution** - Francesco Perri , Rosolino Cirrincione, Salvatore Critelli , Paolo Mazzoleni &Anna Pappalardo
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Iron slags from iron scraps as raw material to produce geopolymers

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In the last years, researchers and "green thinking" engineers have focused their attention on developing of alternative binders which do not require the use of fossil fuel to heat large kilns and natural raw materials. One alternative can be "geopolymer" technology, which requires mixing of post-industrial and municipal waste as well as naturally occurred aluminosilicate materials with an activation agent at the ambient temperature or slightly increased temperature produced eco-friendly cementitious materials (Bajare et al., 2014).

In line with this perspective, the current study provides an opportunity to advance the understanding about the use of iron-scraps as possible raw material to obtain geopolymers. To reach this aim, the identification of supply sites in Sicily (Italy) and their localization on map of the region, has been firstly carried out. Acciaierie di Sicilia Spa was chosen as supplier industry to provide iron scraps. During the next stage, experiments have been conducted in the laboratory. Different tests on micronized samples (<100 μm) characterized by different ratios of silicon, calcium and aluminum (Si> Ca>Al for all samples) have been carried out. The raw material has been mixed and grinded, and then heat treated (at 300°C for 48h) and dried (80°C for 7 days). Our results showed that the produced geopolymeric materials area characterized by excellent mechanical properties (e.g. a good compression strength). The best result has been obtained from the mixing of: 1) 300 g of iron scraps; 2) 40 g of siliceous diatom shells and 3) 220 ml di NaOH 9 M. This produced geopolymeric material also passed the immersion test (into water for 72 h).

Overall, we propose that further studies on introduction of different waste materials in the production process of geopolymeric materials are needed in order to improve the knowledge about the possibility of their re-use.

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An innovative artworks cleaning approach: PEO-based nanostructured polymer systems

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The search for innovative, smart and performing cleaning agents in the field of cultural heritage is one of the main issues of modern conservation science. The development of novel smart nano-structured cleaning systems requires the comprehension of their structural behaviour and interactions with other materials down to the nanoscale in a fluid environment. Complex fluids based on amphiphilic formulations such as micelles, microemulsions and (hydro-gels) represent emerging materials, in the field of conservation of artworks, as safe and effective nano-structured systems for the removal of hydrophobic polymeric coatings. In this respect the comprehension of the cleaning mechanism represents a key information for the design and engineering of tailored fluids for this purpose. In spite of the number of recent studies in this field, the mechanism and the interaction processes between nanostructured fluids and hydrophobic polymer films is still poorly understood [1-3]. We show some recent results from a study about the mechanism of the cleaning process (organic components removal) by means of nano-structured materials consisting of PEO-based micellar polymer systems of block copolymer polydimethylsiloxane-b-polyethyleneoxide (PDMS-PEO). The obtained results may help to identify the main relevant parameters that influence the increase of the rate and efficiency of the cleaning process.

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Sol-gel silica coatings for the protection on textiles from cultural heritage

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Ancient and archaeological textiles are a precious historical-artistic heritage, which must be protected against the deterioration induced by environmental agents (light, temperature and humidity changes, atmospheric pollutants, etc.). Recent scientific advances led to a wide array of new coatings consisting of nanostructured materials for the preservation of the cultural heritage and nowadays many scientific reports are concerning the application of such materials synthesized by the sol-gel method in the conservation and consolidation of stones and glasses [1]. According to this technology, it is possible to synthesize advanced materials with high degree of homogeneity at molecular level and unique physical-chemical properties. Based on subsequently hydrolysis and condensation reactions, this versatile and cheap synthetic strategy, performed at low temperature, conducts to inorganic or hybrid organic-inorganic networks by starting from proper metal alkoxide precursors [2]. The thin silica films can be applied as protective coatings on materials surface, among which textile fabrics: it has been found that the colloidal silica improves the mechanical properties of the substrate allowing good optical transparency, and provides further properties such as hardness, anti-corrosion, wear resistance and so on [3,4]. Tetraethoxysilane (TEOS) is a silica inorganic alkoxide widely employed in the sol-gel technique and several studies have been conducted in order to study the adhesion of silica coating on the surface of different materials such as glasses, plastics, metals, and textiles [5], as well as the influence of catalyst on the gelation process [6].

A study dealing with the relationship between catalyst amount and the number of coating layers deposited on cotton fabrics was conducted in this work. Dibutyltindiacetate (DBTA) was employed as the catalyst for the sol-gel reaction of TEOS. The catalyst was solubilized in alcohol and employed as the last layer in the multistep process (1 to 6 TEOS depositions) for the realization of a multi-layer architecture on textiles. The influence of the architecture, the combination of the number of silica layers with the catalyst amount on several properties of treated fabrics were investigated. Synthesized coatings were characterized by several chemical-physical characterization techniques. The chemical structure of thin films and their morphology were studied by ATR FT-IR spectroscopy and scanning electron microscopy (SEM), respectively. The thermal and thermo-oxidative stability, the flammability and the combustion behavior of the sol-gel treated cotton fabrics were analyzed as well as the durability of the coatings after different washing cycles and their abrasion resistance.

Experimental findings demonstrate the efficiency of sol-gel based materials specifically tailored to the protection on and preservation of textiles from cultural heritage.

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