XVII CONVEGNO NAZIONALE
AIMAT2023

ATTI DEL CONVEGNO

Sessioni Tematiche

1. Sintesi, caratterizzazione e modellazione dei materiali
2. Nuove Funzionalità dei materiali
3. Applicazioni tecnologiche, nuovi processi e nuova manifattura
4. Progettazione sostenibile e ciclo di vita dei materiali
5. L'ambiente, il costruito e la conservazione del patrimonio in collaborazione con CINCOMINET

SESSIONE 1

Codice	Titolo	Corresponding Author
S1.1	ALUMINIUM-ASSISTED ROOM TEMPERATURE DEPOSITION FOR ELECTROLESS Ni AND NiP COATINGS: A COMPARATIVE STUDY	Marco Conti
S1.2	NOVEL SILICA AEROGEL-POLYURETHANE COMPOSITES FOR THERMAL INSULATION	Alessandro Fraleoni
S1.3	DEXTRAN-BASED SYSTEMS FOR BIOMEDICAL USE: SYNTHESIS, CROSSLINKING AND CHARACTERIZATION	Miriam Cappello
S1.4	ENVIRONMENTAL APPLICATIONS OF ANODIC TiO_2 FILMS FOR WATER AND AIR PURIFICATION	Maria Vittoria Diamanti
S1.5	ENHANCED PERFORMANCE OF MAGNETRON SPUTTERED LEAD FREE PEROVSKITES THIN FILMS BY THERMAL TREATMENT	Nicola Calisi
S1.6	FABRICATION OF MULTIFUNCTIONAL GO-CNR@PCL CORE-SHELL NANOCOMPOSITES WITH SPIDER LEG BIOINSPIRED HIERARCHICAL ARCHITECTURES USING WET ELECTROSPINNING-ASSISTED SELF-ASSEMBLY	Michele Gammino
S1.7	DEVELOPMENT OF A NOVEL DRUG DELIVERY SYSTEM FOR ORAL CANCER TREATMENT	Alessia D'Andrea
S1.8	DEVELOPMENT OF ASYMMETRIC GEOPOLYMERIC MEMBRANE FOR WASTEWATER TREATMENT	Giulia Masi
S1.9	CHITOSAN-BASED POROUS MICROSPHERES FOR TUNING FOOD PACKAGING FILMS PERMEABILITY	Andrea Fiorati
S1.10	INVESTIGATING THE ACTIVATION OF PASSIVE METALS BY A COMBINED INSITU AFM AND RAMAN SPECTROSCOPY SYSTEM: A FOCUS ON TITANIUM	Luca Casanova
S1.11	INNOVATIVE MATERIALS FOR HYDROGEN STORAGE	Giulia Calabrese
S1.12	THE INFLUENCE OF ELECTROSPUN VEILS POSITION ON THE DAMAGE TOLERANCE OF CARBON FIBRE REINFORCED LAMINATES	Irene Bavasso
S1.13	INNOVATIVE COMPOSITE COATING FOR LOW-TEMPERATURE THERMOCHEMICAL ENERGY STORAGE	Emanuela Mastronardo
S1.14	CHARACTERIZATION OF IN718 ALLOY FOR ADDITIVE MANUFACTURING AND ITS APPLICATION IN LATTICE STRUCTURES	Annalisa Ferrarotti
S1.15	VANADIUMEFFECT ON HEAT AFFECTED ZONE OF HIGH STRENGTH STRUCTURAL STEELS	Andrea Di Schino
S1.16	SURFACE AND INTERFACE DRIVEN ENVIRONMENTAL RELIABILITY AND CRACK PROPAGATION RESISTANCE OF 3D-PRINTED ALD-COATED NANOCERAMICS	Marco Sebastiani
S1.17	ADVANCED CHARACTERIZATION OF MICROSTRUCTURAL PARAMETERS INFLUENCINF SERVICE LIFE OF THERMAL BARRIER COATINGS	Giulia Pedrizzetti
S1.18	VISCOELASTIC CHARACTERIZATION OF REFORMABLE EPOXY VITRIMERS COMPOSITES	Barbara Palmieri
S1.19	BIOREINFORCEMENTS ACHIEVED FROM DWARF PALM FOR THE FABRICATION OF GREEN COMPOSITES WITH STIFFNESS-TOUGHNESS BALANCE	Andrea Maio
S1.20	TAILORED RELEASE OF CORROSION INHIBITORS FROM SMART ENGINEERED MESOPOROUS SILICA NANOPARTICLES	Federico Olivieri
S1.21	DESIGN STRATEGIES FOR BIOACTIVE COLLAGEN-BASED SCAFFOLDS TO SUPPORT BONE TISSUE REGENERATION	Giorgia Montalbano
S1.22	THE INFLUENCE OF INFILL ON CHARACTERIZATION AND MODELING OF FUSED FILAMENT FABRICATION THERMOPLASTIC PARTS	Marco Zanelli
S1.23	INFILL DENSITY INFLUENCE ON A COPPER PLATED ABS OBTAINED BY FUSED FILAMENT FABRICATION	Valentina Mazzanti
S1.24	ADVANCED NANOINDENTATION MEASUREMENTS: HIGH-SPEED 3D MAPPING ON COMPLEX MATERIALS FOR 4.0 DESIGN AND ERRORS MITIGATION STRATEGIES	Edoardo Rossi
S1.25	THE INFLUENCE OF GRAPHENE OR STAINLESS STEEL FLAKES ON THE MECHANICAL BEHAVIOR OF PORCELAIN ENAMEL	Stefano Rossi
S1.26	LIFE CYCLE EVALUATION OF DOUBLE-STEP COLD BONDING PELLETIZED INDUSTRIAL WASTE LIGHTWEIGHT ARTIFICIAL AGGREGATES	Narinder Singh
S1.27	INSIGHT INTO THE ROLE OF INTERPARTICLE SPACING ON THE MECHANICAL BEHAVIOUR OF BRICK-MORTAR NANOCOMPOSITES	Michele Giordano
S1.28	FUNCTIONALISED 3D PRINTED SCAFFOLDS AND IMPLANTS FOR CRANIOMAXILLOFACIAL APPLICATIONS	Ilaria Cacciotti
S1.29	BIOCHAR FROM RESIDUAL AGRO-BIOMASS AS ENZYMES CARRIER	Marianna Guagliano
S1.30	FIRE-RESISTIVE PERFORMANCE OF AGED SUSTAINABLE INTUMESCENT COATINGS FOR NAVAL APPLICATIONS	Elpida Piperopoulos
S1.31	ELECTROCHEMICAL HYDROGEN CHARCGING ON CARBON STEELS AND LOW ALLOY STEELS, EFFECTS ON FRACTURE SURFACE AFTER J-INTEGRAL MECHANICAL TEST	Luca Parterlini
S1.32	OPTIMIZATION OF A FRIT-FREE GLAZE FORMULATION FOR CERAMIC GLAZE AND ENGOBE PREPARATION	Erika Iveth Cedillo-González
S1.33	A MULTISCALE INVESTIGATION OF WEAK INTERACTIONS BETWEEN POLYETHERIMIDE AND CARBON DIOXIDE	Giuseppe Scherillo
S1.34	CHARACTERIZATION OF CYCLIC OLEFIN COPOLYMERS AS CONSTITUTIVE MATERIALS FOR INSULIN RESERVOIRS	Mario Milazzo
S1.35	MULTIFUNCTIONAL ALUMINUMSURFACES	Amani Khaskhoussi
S1.36	REDUCED GRAPHENE OXIDE/WASTE-DERIVED TIO2 COMPOSITE MEMBRANES: A NEW MATERIAL FOR HYBRID WASTEWATER TREATMENT	Anna Dotti
S1.37	DIGITAL LIGHT PROCESSING OF TEXTURED ALUMINA CERAMICS: SLUTTY ELABORATION AND MICROSTRUCTURAL TAILORING	Bartolomeo Coppola
S1.38	ANODIZING OF AlSi10Mg ALLOY PRODUCED BY L-PBF: EFFECT OF SURFACE FINISHING AND SEALING	Maria Francesca Bonilauri
S1.39	NOVEL APPROACHES FOR THE MANUFACTURING OF HOLLOW COMPOSITES PARTS USING NOVEL WAX-BASED BLENDS	Giuseppe Recca
S1.40	THERMOMECHANICAL CHARACTERIZATION OF THERMOPLASTIC NANOCOMPOSITES BASED ON THE IN-SITU ANIONIC POLYMERIZATION OF POLYAMIDE 6	Giulia Fredi
S1.41	CORROSION OF AL ALLOYS TANKS IN BIO ETHANOL BLENDED GASOLINE FUEL	Federico Scaglione
S1.42	MICRO AND NANO-STRUCTURE OF POLY-LACTIC ACID WITH THERMAL, MECHANICAL, TRANSMISSION ELECTRON MICROSCOPY AND X-RAY DIFFRACTOMETRY ANALYSES	Giovanna Molinari
S1.43	EFFECT OF DIFFERENT SURFACE TREATMENTS ON GRADE 5 TITANIUM ALLOY FOR IMPROVING THE ADHESION WITH THERMOPLASTIC BASED COMPOSITES	Riccardo Miranda
S1.44	TITANIA NANOTUBES ARRAYS DECORATED WITH COPPER: EFFECT OF THE SUBSTRATE PRETREATMENT ON MORPHOLOGY AND ELECTROCHEMICAL PERFOMANCE OF THE CATHODES	Elisabetta Petrucci

S1.1 ALUMINIUM-ASSISTED ROOM TEMPERATURE DEPOSITION FOR ELECTROLESS Ni AND NiP COATINGS: A COMPARATIVE STUDY

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Electroless nickel plating baths typically consist of various components such as complexing agents, nickel salts, reducing agents, buffering agents, and stabilizers. Among these, reducing agents such as NaH2PO2, KBH4, and N2H4 · H2O have been widely used to prepare nickel coatings. The electroless reactions require a sufficient potential barrier, which is usually achieved through heating the bath. For the deposition of pure Ni and NiP coatings, the typical temperature range for the deposition bath is between 80 and 95 °C. Maintaining high temperatures in industrial tanks with a high capacity involves a significant energy expenditure that contributes to high production costs and adverse environmental impact. Lowering the deposition temperatures is a potential strategy to mitigate this issue, but it requires overcoming the activation energy barrier for deposition reactions. The feasibility of using lower-temperature baths, including room-temperature baths, is dependent on identifying and utilizing a non-thermal energy source. It is widely recognized that aluminum is an effective material for initiating electroless nickel plating on some substrates through a process known as aluminum-induced activation. Specifically, aluminum can serve as an electron source to promote the plating process. Drawing inspiration from the phenomena discussed above, here is present a novel and straightforward electroless plating method for depositing Ni coatings. In this study, we utilized aluminum as non-thermal energy source, replacing traditional and expensive thermal energy source. The plating bath employed in our method contained both Ni source and the reducing agent but is performed at room temperature. To perform the plating process, substrates connected to aluminum were directly immersed into the plating bath at room temperature. This method yielded both pure Ni and NiP coatings. To assess the effectiveness of this approach, a comparative analysis was conducted between the Al-assisted coating and a coating produced using the traditional electroless heating bath method.

S1.2 NOVEL SILICA AEROGEL-POLYURETHANE COMPOSITES FOR THERMAL INSULATION

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Polyurethanes (PUs) are well known for their chemical versatility, which allows to prepare them as either rigid or flexible materials, depending on the chosen starting monomers. They also have an inherently low thermal conductivity λ (about 0.18 W/mK), which can be further lowered to 0.015-0.030 W/mK in foamed PUs, i.e. porous PUs filled with gases having a low thermal conductivity. However, in PU foames the filling gas tends to escape with time upon relatively short times (days/weeks), causing a progressive increase of λ towards the 0.18 W/mK value typical of the solid (i.e., non-foamed) PU.

In order to achieve an effective thermal insulation performance able to last in time, it is hence desirable to identify appropriate PU-based formulations. One possible strategy is to realize a composite with solid PU as the matrix and a solid filler characterized by very low λ, such as silica aerogel (SA), which when taken alone can reach indefinitely lasting thermal conductivities down to 0.015 W/mK.

Here we present some results of this approach, related to the thermal behavior of a composite based on elastomeric PU as the matrix and silica aerogel as the filler. In more detail, we found that it is possible to realize homogeneous composites with SA loadings up to 20% W/W, and that such composites have a thermal conductivity about 13% lower than that of the pristine (i.e., pure), starting PU. Tests on the evolution of the thermal conductivities of such composites are ongoing, with preliminary results indicating that the lower λ of these composites does not decay appreciably over time spans of about two weeks.

S1.3 DEXTRAN-BASED SYSTEMS FOR BIOMEDICAL USE: SYNTHESIS, CROSSLINKING AND CHARACTERIZATION

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In the last decades, natural polysaccharides have gained great interest in cosmetics, pharmaceutical, and biomedical applications. Chitosan, alginate, hyaluronan, gellan gum, and dextran are the most used polysaccharides as biomaterials. In recent years, dextran-based materials were developed for drug delivery use, column chromatography application, and cell culture technology. The dextran macromolecules show high resistance to protein adsorption due to their hydrophilicity. This is an important property required by implantable medical devices and drug or protein-targeting carriers, so dextran-based materials are suitable for the coating of biomedical implants and drug delivery systems. Polyelectrolytes such as chitosan, polyallylamine, polystyrene sulfonate, and polyacrylic acid, are commonly used for layer-by-layer coating techniques. In this work, new materials based on dextran methacrylate (DexMA) and polyacrylic acid (PAA) were developed to obtain bioartificial materials that combine polyelectrolyte properties with high anti-fouling characteristics due to polysaccharide moieties.

DexMA/PAA systems were prepared with different weight ratios and crosslinked by UV and thermal treatments. The hydrogels obtained were characterized by a morphological (SEM) and physico-chemical (FT-IR, DSC, TGA) analysis before and after the crosslinking. Swelling and mass loss tests were also performed.

Samples showed good chemical homogeneity due to molecular interactions between DexMA and PAA. Untreated samples had a dense structure, on the contrary, the crosslinked samples showed an inhomogeneous porosity. Concerning water stability, the crosslinking treatments increased this property. The highest stability in water was shown by DexMA/PAA samples photo-crosslinked by UV at 256 nm followed by thermal treatment.

S1.4 ENVIRONMENTAL APPLICATIONS OF ANODIC TiO2 FILMS FOR WATER AND AIR PURIFICATION

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The interest in titanium and its oxides keeps growing on account of their peculiar engineered properties, which find applications in several fields, from architecture to bioengineering, from automotive to photovoltaic cells and photocatalytic devices. There are several methods that allow to grow titanium oxides, among which anodic oxidation has the nice advantage of growing TiO_2 nanostructures directly immobilized on a substrate, avoiding the issue of nanostructure recovery from the medium. The experimental data here presented will then focus on latest achievements in the field of environmental cleanup with anodic TiO_2 oxides. Such materials are often tested in purification devices, for both wastewater and air treatment: the main goal is to achieve large surface area and high oxide crystallinity, in order to ensure the formation of oxides with high photocatalytic activity. In this direction, the production of self-aligned TiO_2 nanotubes that stem vertically from the metal substrate (Fig. 1) is particularly attractive, as it allows to achieve specific surface areas even two orders of magnitude larger than the nominal one. Oxides are then tested in the degradation of organic dyes as representative of possible water contaminants, and in the degradation of volatile organic compounds (VOCs).

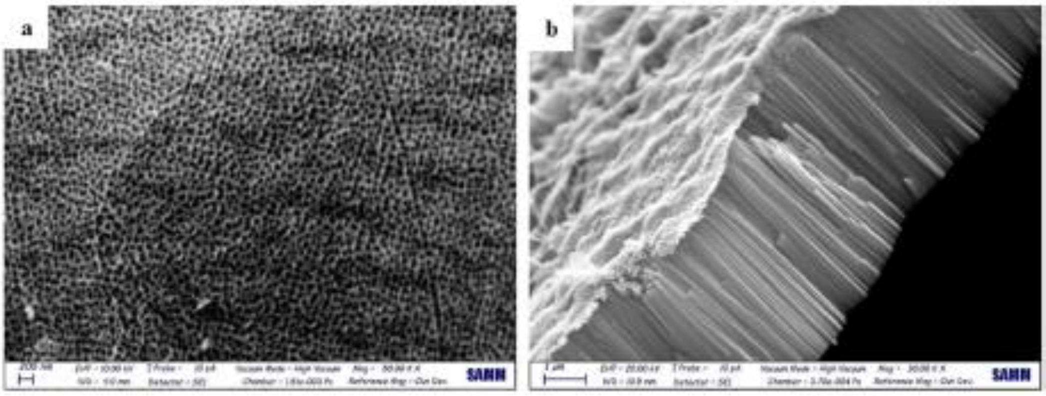

Figure 1. Morphology of nanotubular oxides employed in this work.

While dyes are often used to assess photocatalytic activity, typical tests are very far from real wastewater treatment, as they involve one single dye in distilled water; to better characterize the potential of these oxides, here we present the purification of multiple dye mixes and the influence on the process of possible ions present in water. On the other side, air purification was analyzed with a prototypal plug-flow reactor with different concentrations of toluene, representing medium-high pollution levels typical of an industrial *context. Results indicate a good potential of these oxides in practical applications (Fig. 2), with a correct choice of the target environment.*

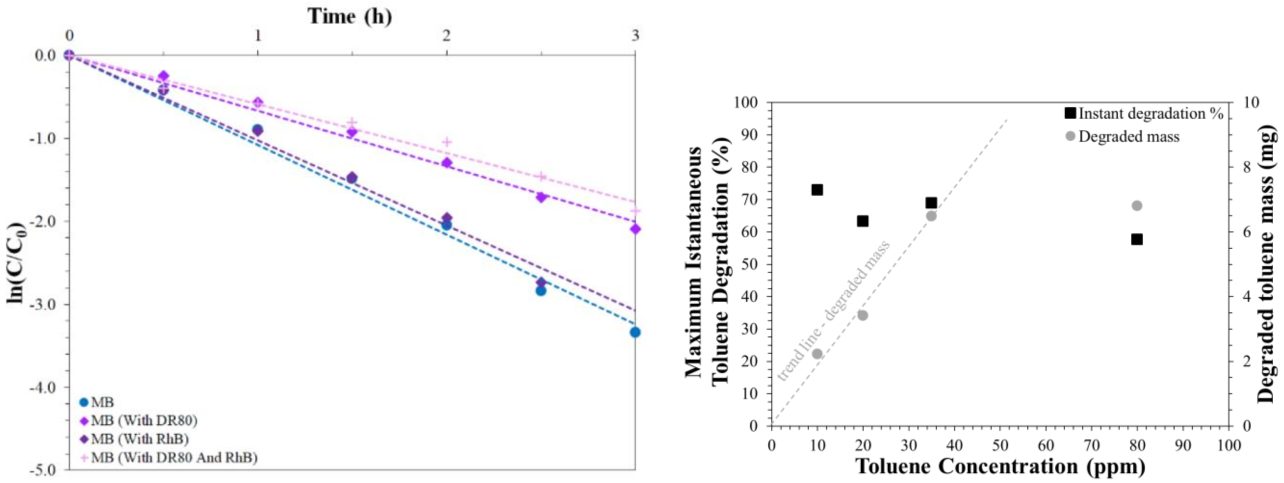

Figure 2. Left: Variation of degradation kinetics of methylene blue dye (MB) alone, in presence of rhodamine B dye (RhB), direct red (DR), or both. Right: Toluene degradation extent (instant percent degradation and mass degraded in 30 min of test) as a function of inlet toluene concentration, in presence of 50% RH.

S1.5 ENHANCED PERFORMANCE OF MAGNETRON SPUTTERED LEAD FREE PEROVSKITES THIN FILMS BY THERMAL TREATMENT

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Perovskites are ones of the most promising materials for the realization of the new generation of solar cells. Mixed organic-inorganic perovskites were the first ones proposed but they presented low stability on Ambiental condition. By substituting the organic cations with Pb2+ the stability of the materials increases but the presence of lead, toxic both for environment and humans, is not practicable. Here we present an innovative way for the deposition of the lead free totally inorganic Cs3Bi2I9 perovskite from vapor phase. By taking use of a magnetron sputtering system equipped with a radiofrequency working mode power supply and a single target containing the correct ratio of both CsI and BiI3 salts, it was possible to deposit a perovskitic film on silicon and soda lime glass. The target composition was optimized to obtain a stoichiometric deposition and the best compromise was founded with a mix enriched with 20% w/w of CsI. Secondly, the effect of post deposition thermal treatments (150 °C and 300 °C) and of the deposition on a preheat substrate (150 °C) were evaluated by SEM, XRD and XPS techniques. The thermal treatment at 150 °C improved the uniformity of the perovskite film without degrading it, the one at 300 °C damaged the perovskite deposited. By deposition the perovskite on a preheated substrate at 150 °C the obtained film showed a higher crystallinity, and no degradation phenomena was observed. An additional thermal treatment at 150 °C on the film deposed on the preheated substrate was evaluated and it showed that the crystallinity remains high, like the one of the not treated film, but the morphology was more uniform, and no degradation phenomena was observed.

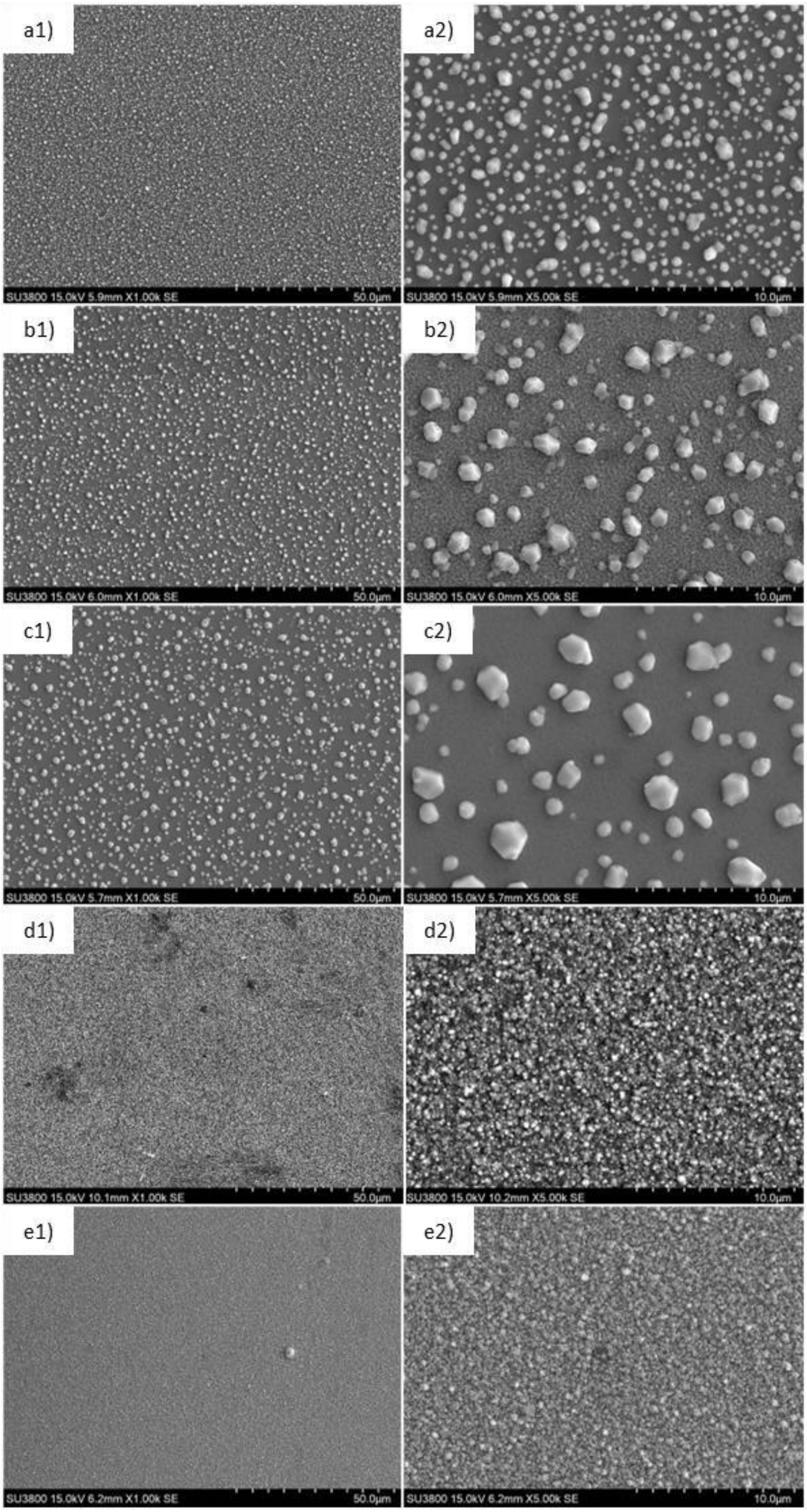

Figure 1. SEM images of (a) pristine perovskite film, (b) perovskite film annealed at 150 °C, (c) perovskite film annealed at 300 °C, (d) perovskite film deposed on preheated substrate at 150 °C and e) perovskite film deposed on preheated substrate at 150 °C and annealed at 150 °C.

S1.6 FABRICATION OF MULTIFUNCTIONAL GO-CNT@PCL CORE-SHELL NANOCOMPOSITES WITH SPIDER LEG BIOINSPIRED HIERARCHICAL ARCHITECTURES USING WET ELECTROSPINNING-ASSISTED SELF-ASSEMBLY

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This work highlights the importance of developing efficient fabrication and modification strategies for the simultaneous removal and recovery of chemical contaminants from water, in compliance with circular economy and zero-waste guidelines. The optimization of absorbent materials requires a multidisciplinary approach, including knowledge of processing technology and surface engineering. Adequate levels of porosity, surface area, and roughness, along with tailored wettability, are crucial to allow efficient removal, while mechanical robustness is necessary to withstand severe external stresses without causing secondary pollution. Nanomaterials such as metal organic frameworks, nanoclays, carbon nanotubes, and graphene or graphene oxide have shown excellent sorption performances. However, their difficulty in handling and removing from water requires them to be incorporated or anchored into various substrates with well-defined morphology. Porous core-shell nanocomposites, having a flexible polymeric skeleton coated with GO-CNTs as active compounds, offer the possibility to conjugate the easy processability and good mechanical properties of polymers with the adsorption performance ensured by the surface characteristics of external sheath. Unfortunately, the current protocols involve multi-step procedures, whose laboriousness increases as the morphological and functional complexity of hierarchical structures increases. This study presents a novel approach to fabricating nanohybrid structures, consisting of a 3D fibrous network of polycaprolactone (PCL) wrapped by graphene oxide (GO) sheets with carbon nanotube (CNT) brushes anchored onto the GO sheets. The method involves electrospinning PCL solutions onto a suspension of GO and CNTs in ethanol, resulting in self-assembly through electrostatic wrapping of GO sheets around PCL fibers and π-π stacking between GO and CNTs. The resulting monoliths are lightweight with 99% porosity and exhibit a 1575% stiffness improvement. The nanopatterned surface allows for efficient removal of methylene blue and/or methyl orange from stagnant water with approximately 100% efficiency. Overall, this approach offers a fast and efficient way to design multifunctional nanohybrid structures with enhanced mechanical properties and improved removal of chemical contaminants from water.

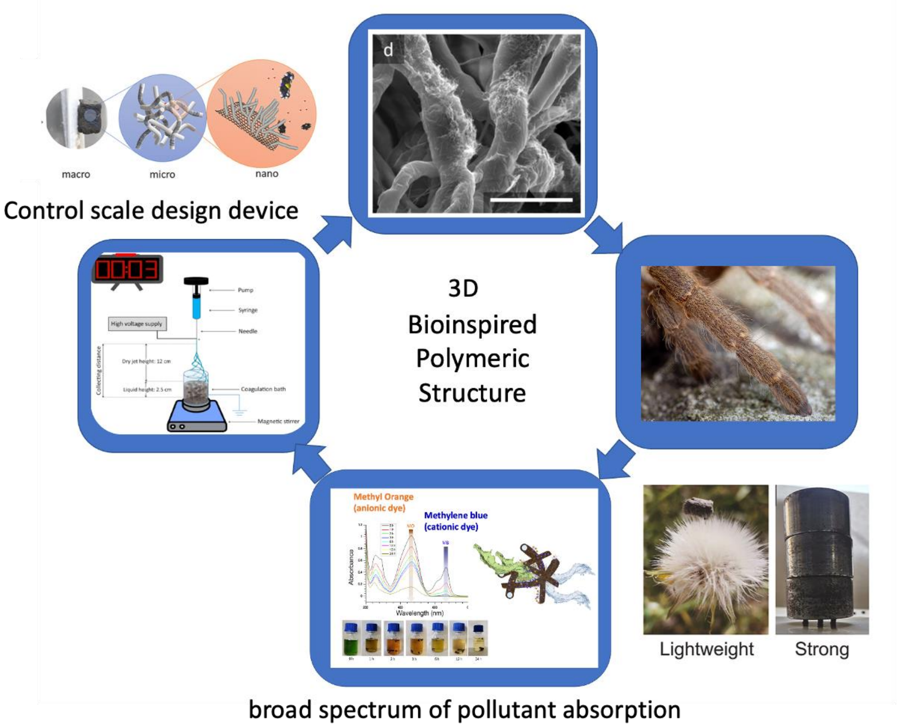

Figure 3. Schematic representation of design Multifunctional GO-CNT@PCL Core-Shell Nanocomposites with Spider Leg Bioinspired Hierarchical Architectures(from high adsorption capacity to Strength).

S1.7 DEVELOPMENT OF A NOVEL DRUG DELIVERY SYSTEM FOR ORAL CANCER TREATMENT

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Squamous Cell Carcinoma (OSCC) is one of the most common forms of oral cancer [1]. It can affect every section of the oral cavity, and among its risk factors alcohol consumption and smoking [2], but also infection by Human Papilloma Virus [3] can be cited.
This kind of cancer is often diagnosed in advanced stages, thus complicating the treatment and decreasing the survival rate [4].
The most common therapies involve surgery, ionising radiation treatment, or a combination of both [5], but they all present several drawbacks, both physical and psychological, among which a demanding recovery and general toxicity effects on healthy cells, tissues and organs due to chemo/radiotherapy.
Hence, the rising effort to devise alternative approaches and promote a pharmacological strategy. In this work, we tried to develop an innovative Drug Delivery System (DDS) to ensure an improved local treatment of the cancer with sustained release of corticosteroids, such as dexamethasone.
The DDS we developed and analysed consisted in poly-ε-caprolactone (PCL) and poly-lactic acid (PLA) microspheres. Their production procedure was devised and honed. The microparticles were prepared both empty and loaded with the drug. The obtained particles were observed at Scanning Electron Microscopy (SEM), and their thermal properties studied by Differential Scanning Calorimetry (DSC). The loading capacity was determined, the drug release was studied and cytotoxicity tests with murine fibroblasts were performed.
After the consolidation of the production process and the characterisation of the system, we proceeded with the engineering of a polysaccharide paste to encapsulate and carry the microspheres, in order to promote mucoadhesion on the treatment site and further modulate the drug release kinetics from the microparticles.

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S1.8 DEVELOPMENT OF ASYMMETRIC GEOPOLYMERIC MEMBRANE FOR WASTEWATER TREATMENT

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Microfiltration membranes for wastewater treatment are usually produced with ceramic or polymeric materials. While ceramic membranes exhibit great durability, polymeric ones are often preferred due to the production costs. An emerging route is the use of geopolymers to produce asymmetric and self-sustained membranes. This material exhibits outstanding durability properties, considerably low production costs and environmental impacts, because the sintering process could be avoided by using geopolymers. For this reason, this study aimed at developing an asymmetric membrane for microfiltration, completely produced by geopolymer. Firstly, the preparation of a pressed geopolymeric support was optimized by ranging the fundamental molar ratios of the mix designs, the water content, and the forming pressure, as shown in Figure 1.

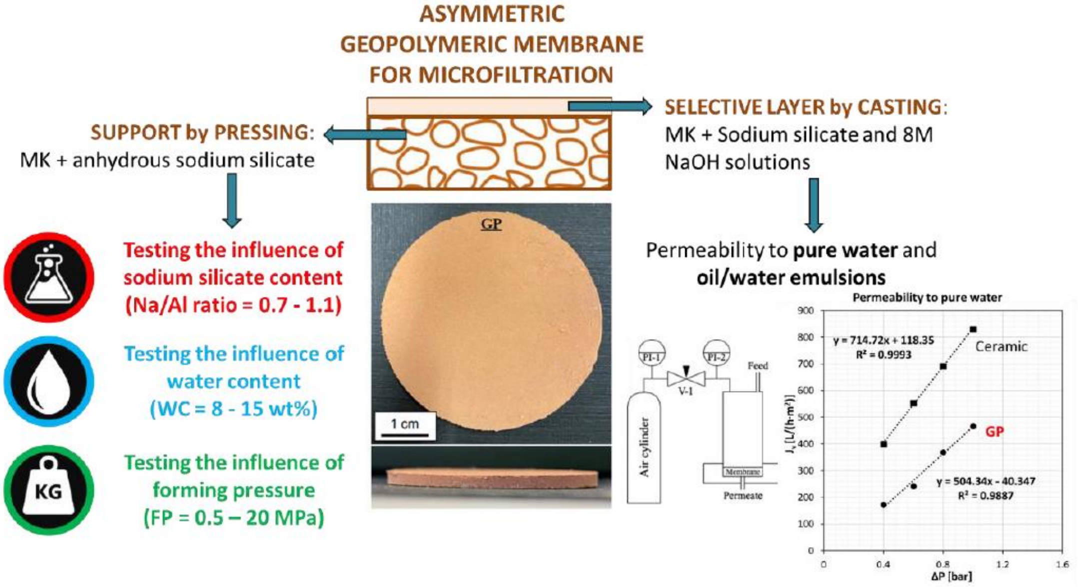

Figure 1. Schematic representing the ratio of this study: on the left, the optimization of the membrane support obtained by pressing and on the right the performance of the whole membrane in terms of permeability to pure water.

The optimised parameters for membrane support preparation are the application of 5MPa of uniaxially pressing to a dry mixed powder consisting of metakaolin and anhydrous sodium silicate, sprayed with 12 wt% water. It was found that the porosity and the permeability to pure water (obtained with a dead-end geometry at a trans-membrane pressure in the

range of 0.2 - 0.5 bar) of the pressed supports are comparable to those of ceramic membranes for microfiltration.
Then, a selective layer prepared by mixing metakaolin, sodium silicate and 8M sodium hydroxide solutions was deposited by spatula coating. The obtained layer shows a thickness of 40 µm and a pore size in the range of 0.1.-1.0 µm. Permeability and rejection of the obtained asymmetric membrane to pure water and oil-water emulsions was carried out with transmembrane pressures below 1.2 bar, showing comparable performances with commercial ceramic membranes (Figure 1). This suggests that the developed microfiltration geopolymeric system is a suitable alternative to ceramic membranes, in terms of performance, production cost and environmental sustainability.

S1.9 CHITOSAN-BASED POROUS MICROSPHERES FOR TUNING FOOD PACKAGING FILMS PERMEABILITY

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Edible and compostable films with tuneable barrier properties are particularly interesting for food packaging applications: the biomimetic approach simulating plant stroma recently emerged as a strategy to design active barrier properties.1 In this work, we prepared porous chitosan microspheres employed as filler for methylcellulose(MC)-based films (figure 1a) to modulate O2/CO2 permeation in fresh produce packaging. The microspheres are obtained by a sharp pH change of a chitosan/PEG in paraffin water-in-oil emulsion, followed by a PEG removal. The process results in porous particles with a diameter of 10 ± 3.5 μm and submicrometric porosity, which can be tuned by post-producing treatments such as freeze-drying (figure 1b and 1c). The microspheres were then treated with tannic acid to reduce oxygen permeability.

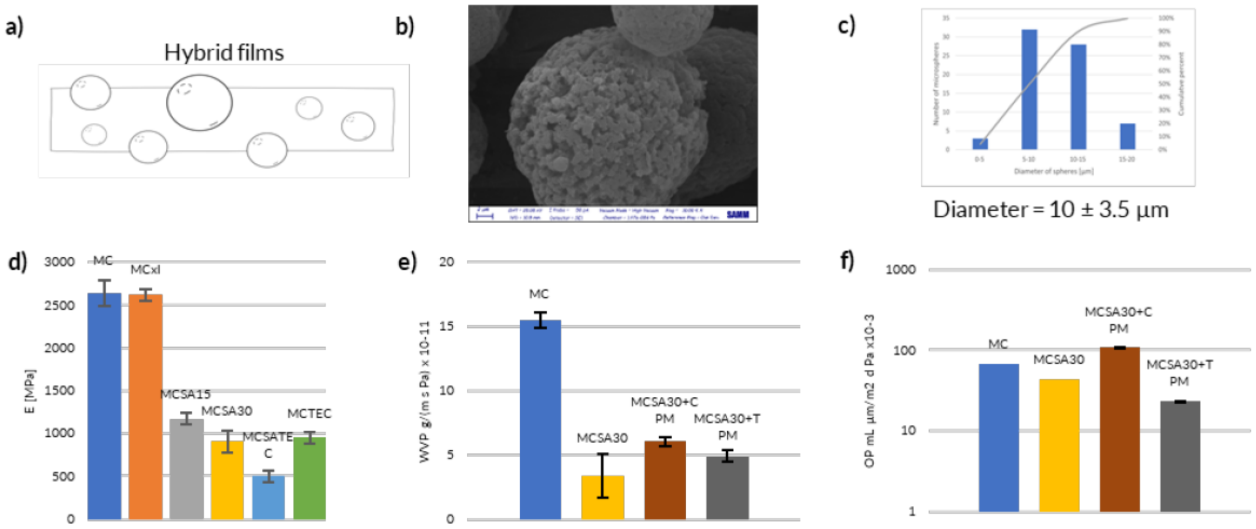

Figure 1.(a) Schematic representation of hybrid film design approach, (b) SEM micrograph of a porous chitosan microsphere, (c) dimensional analysis of the microspheres, (d) Young's modulus of hybrids films, (e) water vapor permeabilities, and (f) oxygen permeabilities.

The films were prepared by casting solutions containing MC and different amounts of stearic acid (SA) and microspheres and then characterized via quasi-static tensile tests, DMA, water vapour, and oxygen permeability.

Adding Stearic Acid decreases the water vapour and oxygen permeability (80% and 20%, respectively) compared to pure methylcellulose. In addition, SA showed a plasticizing effect (lowering the σB of 65% and Young's modulus of 65%, compared to pristine MC) comparable to the one obtained by a common plasticizer (triethyl citrate), figure 1d.

Adding the porous particles modulates the permeability to water vapour and oxygen (190% and 250% increase, respectively). The tannic acid particle treatment increases by 54% the water vapour permeability while further reducing the oxygen permeation by 45%, compared to the control (figure 1e and 1f).

These results demonstrate the fine-tuning of the barrier properties of hybrid films simply by controlling the microsphere morphology, concentration, and chemical modification, paving the way for a customizable platform for materials for food packaging.

Acknowledgement
This study was carried out within the Agritech National Research Center and received funding from the European Union Next-GenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) – MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.4 – D.D. 1032 17/06/2022, CN00000022).

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S1.10 INVESTIGATING THE ACTIVATION OF PASSIVE METALS BY A COMBINED IN-SITU AFM AND RAMAN SPECTROSCOPY SYSTEM: A FOCUS ON TITANIUM

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Understanding the main steps involved in the activation of passive metals is an extremely important subject in the mechanical and energy industry and generally in surface science. The titanium-H2SO4 system (Figure 1a) is particularly useful for this purpose, as the metal can either passivate or corrode depending on its potential. Although several studies tried to hypothesise the surface state of the metal, there is no general consensus about the surface state of Ti in the active-passive transition region.
In the present work, by combining in-situ atomic force microscopy (AFM) and Raman spectroscopy, operating in an electrochemical cell containing 40 %v/v H2SO4, we show that the cathodic polarization of Ti electrodes causes the dissolution of the upper TiO2 portion of the passive film, with formation of electronic defects (Figure 1b), leaving the electrode covered by only a thin layer of titanium monoxide.

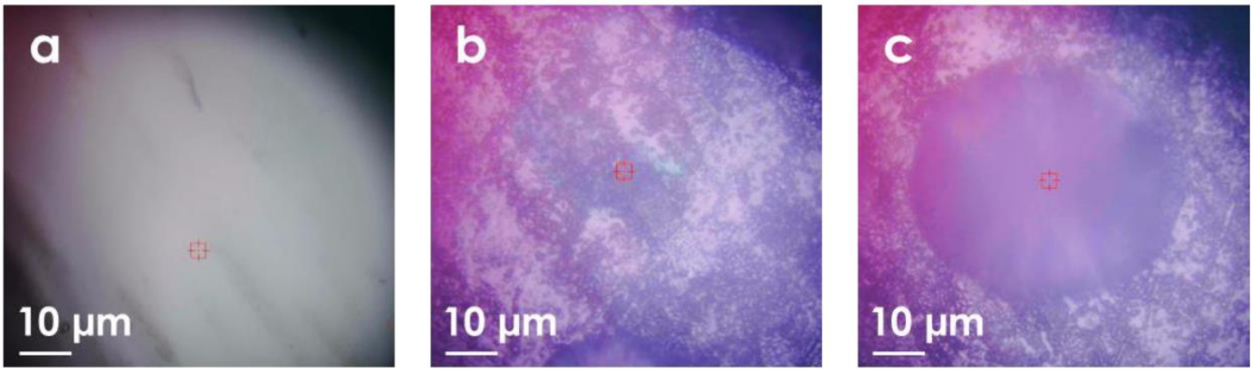

Figure 1. (a) pristine Ti Gr. 2 surface; (b) optical image showing fluorescence due to oxide reduction; (c) image showing formation of a concentration cell.

Fast anodic reactions involved the local acidification of the solution and accumulation of sulphur containing anions at the metal surface. This produces a local increase of the solution turbidity, as in Figure 1c, allowing to distinguish favourable regions for the precipitation of TiOSO4 ·2H2O.

These results give a clear answer to the long-stated question of the physical origin behind the formation of negative polarization resistances, sometimes occurring in corroding systems, and a rationale about the proton-induced degradation of passive surfaces in presence of sulphur containing species.

S1.11 INNOVATIVE MATERIALS FOR HYDROGEN STORAGE

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One of the main challenges for today's society is to find alternative energy sources to fossil fuels. Hydrogen is the most suitable candidate because it has a high energy density (33.33 kWh)and its only reaction product is H_2O. Due to extremely low density of this gas, solid-state storage system is the most viable solution.[1] As alternative and promising materials for this purpose we are evaluating perovskite oxides because these materials have a high thermal stability, they are relatively active, they allow the exploitation of a great variety of elements in the composition while maintaining the basic structure unchanged and, to improve its efficiency, it is also possible to easily vary the stoichiometry.[2] Specifically, we are considering $ABO_{3\pm\delta}$ type perovskite oxides with a non-stoichiometric content of oxygen to investigate whether the presence of oxygen vacations can promote the amount of hydrogen absorbed. We are also evaluating the addition of small amounts of Pd^0, which should promote the dissociation of hydrogen and, therefore, improve the ab/desorption kinetics of this gas. Among the investigated materials, $CaMnO_3$ (CM) is the most promising in terms of hydrogen storage. The H_2 absorption and desorption analysis as a function of pressure were carried out with a High Pressure Gas Sorption Analyzer (i-Sorb Anton Paar). Measurements carried out at 40 bar of H_2 and at different temperatures have shown that the non-stoichiometric amount of oxygen affects the hydrogen storage capacity. In particular, the structures with an excess of oxygen exhibit a higher H_2 absorption capacity. In fact, at 100 °C, CM absorbs 0.44 kg/m3 of H_2 and the uptake of H_2 for $CMO_{3\pm\delta}$ is 0.98 kg/m3. Moreover we observed that, in the same conditions, these materials in presence of Pd^0 absorbs 12.88 kg/m3 and 44.95 kg/m3 of H_2 respectively. Ongoing activities are focused on investigating the gas release conditions and on eventual structural modifications.

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S1.12 THE INFLUENCE OF ELECTROSPUN VEILS POSITION ON THE DAMAGE TOLERANCE OF CARBON FIBRE REINFORCED LAMINATES

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Carbon fibre reinforced polymer (CFRP) laminates represent a class of materials widely used in different sectors (automotive, aviation, aerospace) thanks to their excellent properties as high specific stiffness and strength, and durability (higher corrosion resistance compared to metal alloys). However, because of their anisotropic nature, CFRP suffers delamination as the main internal damage which leads to a significant decrease in the mechanical properties of the laminate. Different techniques have been proposed to mitigate this issue and, among them, interleaving thermoplastic veils can be considered a solution that allows to obtain a homogeneous distribution of the reinforcing material in specific locations. Thanks to the electrospinning process, it is possible to produce veils with a high surface/volume ratio without altering the overall thickness and weight of the final laminate. In this work, commercial polyamide electrospun veils have been used as interleaves to toughen the CFRP laminates. The laminates were manufactured by stacking 20 carbon/epoxy prepreg plies and the neat CFRP properties were compared with those of two configurations (interleaved with six electrospun veils) to study the effect of the veils position (Figure 1). All materials were tested under dynamic (low velocity impact- LVI) and quasi-static (four-point bending test) conditions. LVI tests were performed at three energy levels (10 J, 20 J and 30 J) and three temperature conditions (-50 °C, 25 °C and 100 °C). The effect of the impact event was analyzed by ultrasonic tests (damaged area through-the-thickness), non-contact laser profilometry (dent depth) and scanning electron microscopy. The results collected clearly showed the effect of the veil position on the mechanical properties and the damage tolerance of the laminates.

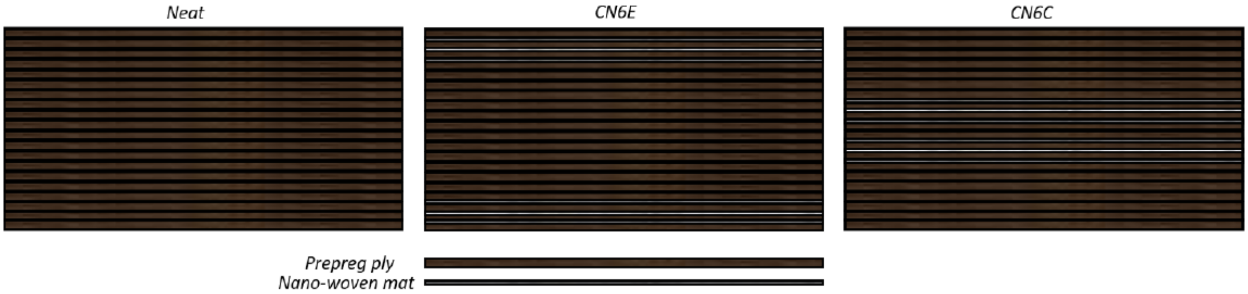

Figure 1. Schematic representation of the tested laminates.

S1.13 INNOVATIVE COMPOSITE COATING FOR LOW-TEMPERATURE THERMOCHEMICAL ENERGY STORAGE

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Low-temperature thermochemical heat storage (TCS) is fundamental for increasing the efficiency of several industrial processes by storing wasted heat and re-using it on demand. This technology exploits a reversible dehydration/hydration reaction of salt hydrates: $A \cdot mH_2O(s) \rightleftarrows A \cdot (m-n)\ H_2O(s) + n\ H_2O(g)$ $(m \geq n)$*. We aim at progressing here by developing innovative composite coatings made of an active TCS material and a water vapor permeable polymeric matrix. Specifically, calcium L-lactate (CaL) pentahydrate has been selected as a novel TCS candidate. This salt, other than a large heat storage capacity of 1021 kJ/kg, is inexpensive, non-toxic, non-corrosive, and non-flammable with respect to its inorganic counterparts. Sulfonated polyether ether ketone (SPEEK) has been selected as the matrix due to its water vapor permeability. For the coatings preparation, SPEEK was solubilized in dimethylformamide (DMF), and then CaL is added at varying content (50-80% wt.). The coating solution was deposited by drop-casting technique on aluminum strips. The coatings undergo dehydration/hydration cycles under controlled temperature and humidity conditions for testing their storage performances and integrity. Morphological, structural, chemical, and thermochemical behaviors were investigated through environmental scanning electron microscopy, X-ray diffraction, Fourier-transform infrared spectroscopy, adhesion tests, thermogravimetric analysis. As a main result, it was demonstrated that SPEEK, being water vapor permeable, not only favors the mass transfer during the CaL hydration and dehydration steps but also enhances the re-hydration kinetics with respect to CaL loose powder. Additionally, the flexibility of the matrix allows the volume shrinkage/expansion of the salt caused by the cyclic dehydration/hydration reactions without significant damage. These encouraging results demonstrate that CaL/SPEEK composite coatings are a highly promising solution for TCS application.*

S1.14 CHARACTERIZATION OF IN718 ALLOY FOR ADDITIVE MANUFACTURING AND ITS APPLICATION IN LATTICE STRUCTURES

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In this work, we investigated the structural, microstructural and mechanical properties of Inconel 718 alloy employed in Laser Powder Bed Fusion processes for producing bulk samples and lattice structures. The work was aimed at: 1) comparing fresh and reused powders in terms of particle size distribution and powders morphology; 2) investigating the structural, microstructural and mechanical properties of bulk samples produced by AM; 3) studying the structural, microstructural and mechanical properties of three different lattice structures: diamond (A), body diagonals (B) and rhombic dodecahedron (C) with relative densities of 30%, 50% and 30%, respectively. Concerning the morphology of the powders, no significant differences were observed between the fresh and reused batches. In the as-built additively manufactured bulk sample, melt pools followed on xy plane (perpendicular to growth direction) the laser scanning path, thus generating a microstructure elongated in the scanning direction, while on xz plane (parallel to growth direction) melt pools assumed an arc shape due to the presence of a thermal gradient, as shown by SEM microstructural analysis. The overall microstructure shows a fine cellular/dendritic structure, indicative of a very high thermal gradient and intermediate interfacial solidification rate, with equiaxed and elongated grains on xy and xz planes, respectively.

In the lattice structures, structural and microstructural features similar to the bulk sample were observed. Also the calorimetric traces of the bulk sample and lattice structures showed similar signals, indicating that there are not significant differences between the two sets of samples. Finally, concerning the compressive behaviour, the stress-strain curves of structures A and C showed a stress plateau, suggesting a bending dominated behaviour, while structure B did not show such a feature and revealed larger values of elastic modulus and yield stress as consequence of its higher density.

S1.15 VANADIUM EFFECT ON HEAT AFFECTED ZONE OF HIGH STRENGTH STRUCTURAL STEELS

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Recent developments of high strength low alloyed steels (HSLA) for energy sector focused on the need to target optimized combinations of strength, toughness and weldability at industrial scale at affordable prices. Welds and heat-affected zones are critical when considering structural integrity, specifically toughness and fatigue properties. In particular, the inter-critically reheated grain coarsened heat affected zone (IC GC HAZ) has been reported as one of the most brittle section of HSLA steels welds. The presence of microalloying elements in HSLA steels induces the formation of some microstructural constituents which can be detrimental to the mechanical performance of a welded joint. Following double welding thermal cycle, with second peak temperature in the range between Ac1 and Ac3, the formation of residual austenite (RA) can lead to a strong loss of toughness and fatigue resistance in the IC GC HAZ. The scope of the present study is to investigate the effect of the vanadium addition on the behavior of the IC GC HAZ of a HSLA steel (S355 grade). The influence of different V contents addition on microstructural variation, formation of the RA fraction and precipitation state of samples subjected to double pass welding thermal cycles is shown. Welding thermal cycles were reproduced using a dilatometer at five different maximum temperatures of the secondary peak in the inter-critical area, from 720 °C to 790 °C.

However, the amount of RA formed is not significant for inducing detrimental effects in terms of fatigue resistance (from the EBSD analysis the values of RA are always less than 0.6% (Fig.1).

Moreover, the variant with the highest content of V (0.1 wt.%) showed the presence of Vrich precipitates as measured by TEM with size smaller than 60 nm (Fig. 2), which was even below 15 nm for 50% of precipitates.

Moreover, the variants with higher content of V (0.1 wt.%) showed the presence of V-rich precipitates with size smaller than 60 nm (Fig. 2), which was even below 15 nm for 50% precipitates.

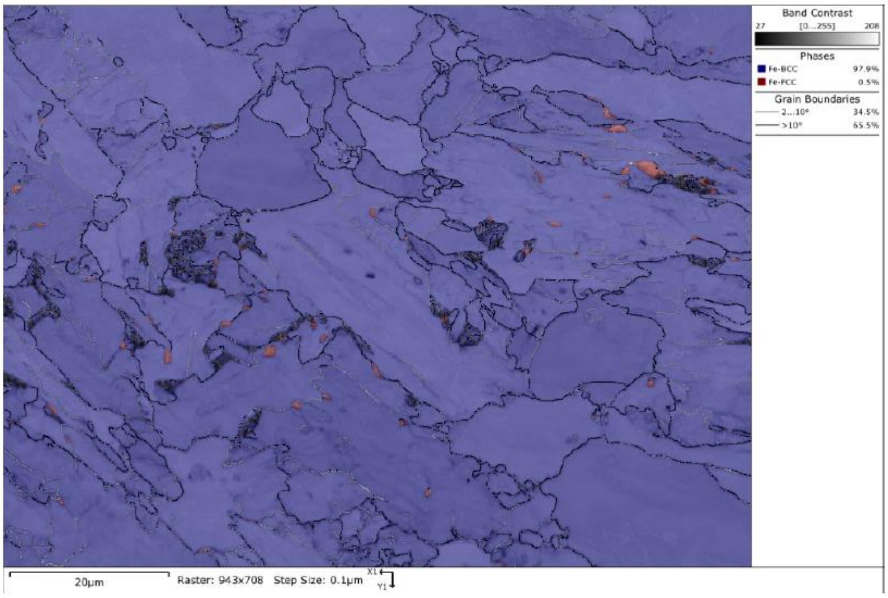

Figure 1. Example of EBSD phase maps of S355 steel grade micro-alloyed with 0.1 wt.% of vanadium. Red zones are the RA phase.

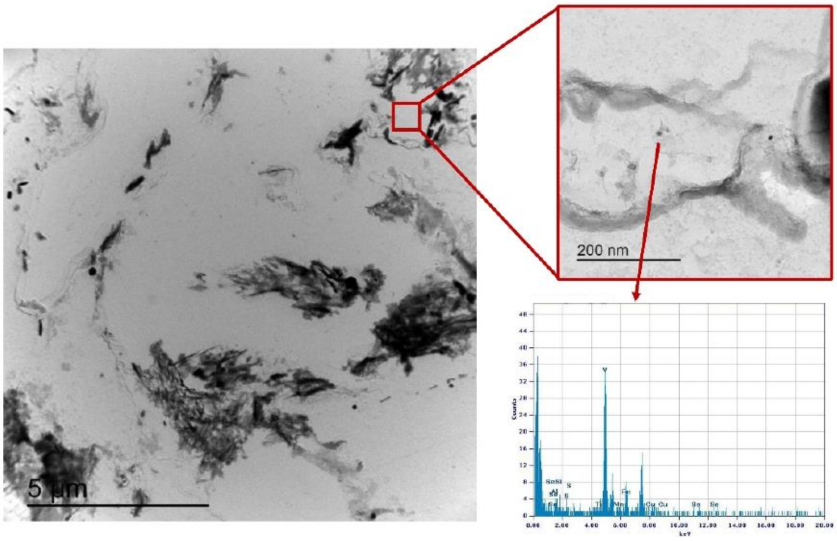

Figure 2. TEM micrograph of S355 steel grade micro-alloyed with 0.1 wt.% of vanadium. Highlighted are the fine V-rich precipitates in the matrix.

S1.16 SURFACE AND INTERFACE DRIVEN ENVIRONMENTAL RELIABILITY AND CRACK PROPAGATION RESISTANCE OF 3D-PRINTED ALD-COATED NANO-CERAMICS

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The applicability of innovative 3D-printed micro- and nano-architected ceramic metamaterials is hampered by the lack of knowledge of their mechanical reliability. Recently, the sensitivity of nano-ceramics' crack propagation resistance to environmental conditions has been evidenced [1]. In this work, we present a further step in understanding and improving the environmental reliability of those materials via the possibility of controlling their surface characteristics and obtaining enhanced independence of the crack propagation resistance to the testing relative humidity. To this scope, a population of TPP glassy carbon micro-pillars and uniform 2.5D structures have been coated with highly conformal ALD-deposited Al2O3 thin films (having a thickness of 50 nm, Figure 1).

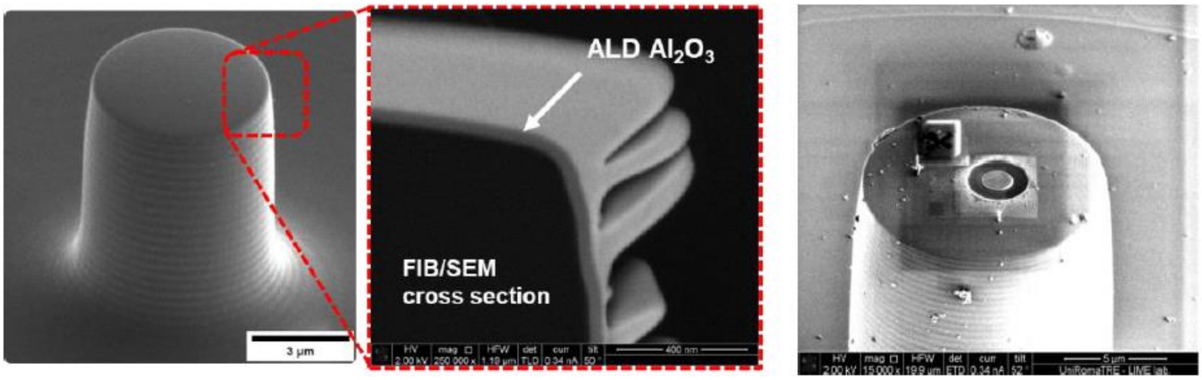

Figure 1 - Testing configuration for a TPP 5.3 μm micro-pillar coated with ALD conformal 50 nm thick Al2O3 film.

The study focused on the application of pillar splitting protocols and localized residual stress measurements [2] to elucidate the effects of the deposited films on the fracture toughness of 5.8 μm diameter micro-pillars (see Figure 1) as a function of two extremal testing RH levels (<5% "and">60%). It is evidenced that the fracture toughness of ALD coated pillars is incremented from that measured for pristine defective high-humidity tested pillars (Figure 2), gaining independency from the relative humidity conditions. Moreover, the average Kc value for the ALD coated samples does not reach the values recorded for the non-coated *pillars at low RH. It was found that tensile residual stresses within the coating interact with the crack as an additional reservoir to the opening system.*

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S1.17 ADVANCED CHARACTERIZATION OF MICROSTRUCTURAL PARAMETERS INFLUENCING SERVICE LIFE OF THERMAL BARRIER COATINGS

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Failure of thermal barrier coatings (TBCs) is generally caused by the development of cracks near the bond coat/top coat interface caused by both the mismatch in thermal expansion coefficients of layers and by the stress field induced by growth of the protective oxide. Since failure is mainly an interface-driven mechanism, thermal sprayed dense-vertically-cracked (DVC) YSZ top coats and highly rough NiCoCrAlY bond coats are developed to increase strain accommodation of the system. Nevertheless, correlation between these features and their influence in determining coating lifetime has not been fully established yet. This work proposes a new method to characterize cracks distribution and roughness parameters based on image analysis procedures implemented in MATLAB. The method for crack analysis allows to introduce a new crack parameter, defined as the ratio between the sum of crack length and the top coat area, which represents the equivalent density of through-the-thickness cracks (pttc). Furnace cycle tests of different TBC systems on ReneN4 superalloys demonstrated that higher pttc corresponds to higher lifetime of coatings and that this parameters is more representative than the standard crack density. The correlation with BC surface parameters showed that too high Rsm, which indicates a higher spacing between peaks, relates with lower interlocking efficiency. Influence of microstructural features on the mechanism of coating failure was investigated exploiting a new method of online monitoring of spallation based on thermographic image. It was observed that higher pttc and lower Rsm associates with effective dissipation of stress through the development of a network of microcracks near the interface. Conversely, less durable coatings with lower pttc associate with failure that occurs through the progressive growth of a single long crack along the interface. To conclude, the combination of pttc and Rsm demonstrated to be relevant in determining durability of TBCs.

S1.18 VISCOELASTIC CHARACTERIZATION OF REFORMABLE EPOXY VITRIMERS COMPOSITE

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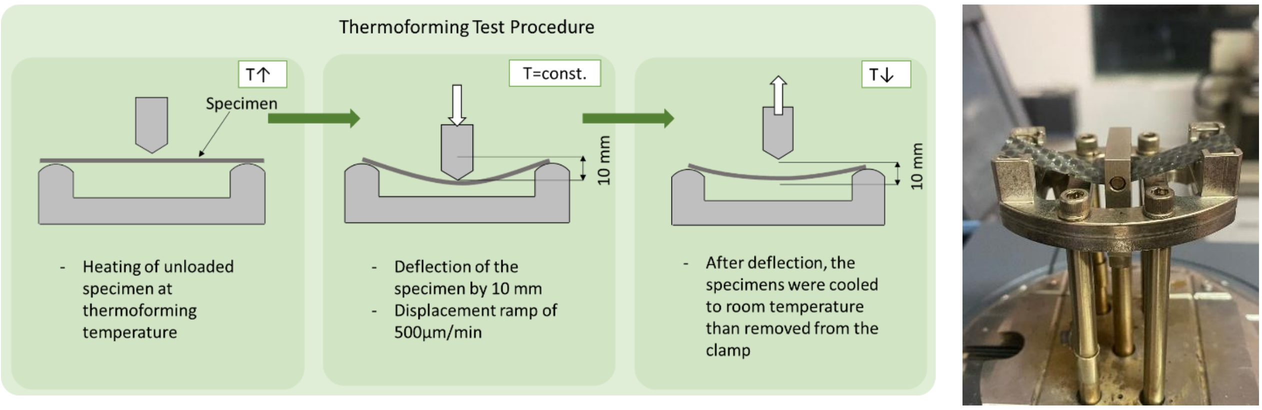
Increasing environmental awareness and industrial competitiveness encourage the use and development of repairable and recyclable structural materials to reduce polymer waste and extend their service life. Recently, the development of thermoreversible cross-linked networks has been introduced as a viable alternative to produce composite components that can be recycled and reformed as thermoplastic materials.

Introducing a thermoreversible covalent bond in the cross-linked backbone allows the topological reshuffling of polymeric networks and makes it possible to rework and reform the cross-linked material. One possible mechanism that enables the vitrimeric behaviour in epoxy resins is based on transesterification exchange reactions between esters and beta-hydroxyls formed by reacting epoxy precursors with suitable acids/anhydrides. Incorporating a catalyst activates the transesterification reaction and induces topological variations, stress relaxation and flow in the cross-linked networks, even though the total number of cross-links does not change. During transesterification, the network's connectivity is altered via exchange reactions, inducing stress relaxation and plastic flow at elevated temperatures without depolymerisation.

In the present work, a commercial epoxy system (araldite® LY 3508 and aradur® 917-1 by Huntsman Corporation) suitable for CFRP manufacturing was selected for its modification as a vitrimer. Furthermore, $ZnAc_2$ was selected as a transesterification catalyst, and two formulations at different ratio between epoxy precursors and curing agent have been prepared and modified by adding the catalyst.

Vitrimer carbon fiber reinforced composites were produced using hand lay-up and vacuum consolidation. The thermal properties of vitrimeric matrix have been investigated by DSC and DMA tests. The thermal reshaping capability of vitrimer-CFRP has been investigated by reprocessing CFRP coupons.



- Heating of unloaded specimen at thermoforming temperature

- Deflection of the specimen by 10 mm
- Displacement ramp of 500µm/min

- After deflection, the specimens were cooled to room temperature than removed from the clamp

Figure 1. thermal reshaping experiment by 3-point bending procedure.

S1.19 BIOREINFORCEMENTS ACHIEVED FROM DWARF PALM FOR THE FABRICATION OF GREEN COMPOSITES WITH STIFFNESS-TOUGHNESS BALANCE

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Two fibrous fillers were achieved from stalks and leaves of Chamaerops humilis dwarf palm and tested as reinforcing agents for poly(butylene adipate co-terephthalate) (PBAT)-based green composites. A robust map of processing-structure-properties relationships was constructed by integrating several techniques, including morphological, spectroscopic, pycnometric and mechanical analyses. Depending on the type and content of filler, a broad array of properties can be achieved. The results of tensile tests (Fig. 1) pointed out that both fillers are strong candidates to overcome some typical drawbacks of PBAT, namely, the lack of both stiffness and cost-effectiveness, while preserving its ductility and environmental sustainability.

The remarkable stiffness increments (up to 300%), combined with fair retention of stretchability (33%) and doubled resistance, enable constructing green composites with toughness values as high as 20–25 MJ/m3. The outstanding mechanical reinforcement was prompted by the formation of an extensive and strong inter/intraphase region, likely arising from either reactions occurring during compression-moulding, or supramolecular interactions between aromatic domains of lignin and terephthalate units of PBAT. The mechanical behaviour of these systems can still be modelled by introducing opportune corrections to Halpin-Tsai model. Both materials and the different fabrication routes herein proposed show a promising palatability for a wealth of applications, including for instance panels for automotive and furnishing, gathering excellent performance from a mechanical, environmental and economic point of view.

S1.20 TAILORED RELEASE OF CORROSION INHIBITORS FROM SMART ENGINEERED MESOPOROUS SILICA NANOPARTICLES

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The improvement of the protection of metal substrates from corrosion phenomena is an important content, which interests several fields, from aerospace to cultural heritage. Mesoporous silica nanoparticles (MSN) show promising potentialities as nanoreservoirs/nanocarriers for the loading and the controlled release of anticorrosive agents [1,2]. Here, two different strategies based on the synthesis of engineered MSN, with the aim to maximize the loading and to tailor the stimuli-responsive release of corrosion inhibitors, are reported. In details, MSN were synthetized by a high-throughput synthesis, loaded with benzotriazole (BTA) and treated with a silver salt to realize new MSN/BTA/Ag stimuli-responsive systems. Indeed, these nanoparticles showed a pH-controlled BTA blocking/release mechanism, depending on the dynamic equilibrium of formation and dissolution of the BTA/Ag complex [3]. It has been proved that BTA guaranteed a passive barrier to corrosive media, while Ag ions were able to sequestrate chloride ions, adding an active barrier contribute. Moreover, a new amino-functionalized MSN (MSN-NH_2) were developed, in order to chemically induce a reaction with benzoyl chloride (BC). Indeed, the stability of the amino bonds between the benzoyl groups and the MSN-NH_2 are pH-dependent, tailoring the release of the anticorrosive agent benzoic acid. The realization of protective coatings was performed by embedding both the engineered MSN in acrylic matrices, which showed effective protection of metal substrates.

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S1.21 DESIGN STRATEGIES FOR BIOACTIVE COLLAGEN-BASED SCAFFOLDS TO SUPPORT BONE TISSUE REGENERATION

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Among the bioactive and biomimetic biopolymers, type I collagen is considered a strategic biomaterial to design biocompatible constructs and biomedical devices aimed at the regeneration of various functional tissues such as bone. In this scenario, the processing of collagen-based materials by 3D-printing and electrospinning technologies can support the design of complex architectures, improving the biomimetic features of the scaffolds. However, these fabrication techniques require a complex optimization of the rheological and physico-chemical properties of the materials while preserving high biocompatibility of the process. Hence, type-I-collagen was successfully combined with different bioactive and smart inorganic phases such as mesoporous bioactive glasses, nanohydroxyapatite and superparamagnetic nanoparticles to design micro- and nano-structured bone scaffolds. The properties of the hybrid formulations were finely modulated according to the chosen manufacturing technology and to guarantee the preservation of collagen functionality. Concerning the use of extrusion-based 3D printing techniques, the formulations were optimised ensuring the shear thinning behaviour and thus their good extrudability, while supporting gelatine slurries were exploited to enable the design of high-resolution complex scaffolds. In parallel, the use of electrospinning was explored to produce nano-fibrous membranes able to mimic the structure of native extracellular-matrix. In this case, the proper selection of solvents and process parameters represented a key factor to develop environmental-friendly approaches while preserving the native physico-chemical properties of collagen. Moreover, the optimised procedure confirmed the processability of hybrid formulations containing up to 10% in weight of inorganic phase, while the high surface area of the scaffolds was exploited to improve the functionality of the final device by grafting of bioactive molecules.

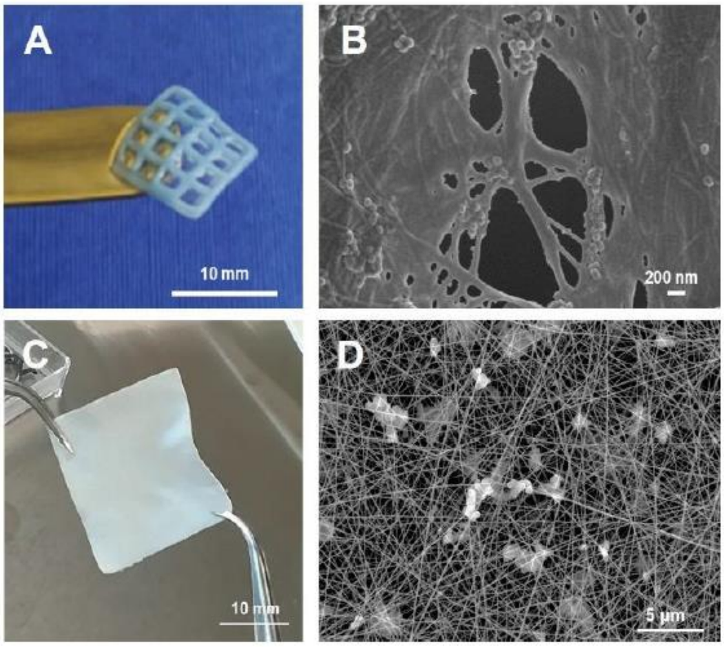

Figure 1. Images of the macro- and microstructure of collagen-based scaffolds produced by means of 3D printing (A, B) and electrospinning technologies (C, D).

S1.22 THE INFLUENCE OF INFILL ON CHARACTERIZATION AND MODELING OF FUSED FILAMENT FABRICATION THERMOPLASTIC PARTS

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Among all the parameters that influence Fused Filament Fabrication (FFF) 3D printing of thermoplastics, infill density is among those that affect mechanical properties the most. Recently, it has been shown that 3D-printed objects can be modeled as composite materials using the Classical Lamination Theory (CLT), in which each layer is represented as a unidirectional lamina. CLT works successfully when the objects are completely filled (100% infill), but it is not clear whether the same occurs for components with lower infill percentages and continuous fiber reinforcement. One idea is to define an equivalent continuous material representing the inner portion, thus neglecting the infill pattern (grid, triangle ...) and orientation. A 3D CAD model starting from the G-Code file can be used and suitably characterized to find such an equivalent material, therefore parts will be modeled as sandwich structures.

Flexural tests were performed on specimens with different infill densities to characterize the mechanical properties. Samples were made both of unfilled and continuous fiber-reinforced (CFR) materials. The experimental measurements were compared with FEM simulations and theoretical estimates through CLT.

From the design point of view, CLT allows results to be achieved quickly, at the expense of accuracy, whereas with FEM modeling much more accurate and representative results are obtained. This is due to a more realistic definition of both geometry and material, but requires more time and effort from the preparation side.

The results showed that continuous fiber reinforcement is very effective in increasing the flexural properties of 3D printed parts. Both qualitative and quantitative agreement was found between modeling and experimental measurements concerning unfilled and CFR materials, especially with the more realistic 3D CAD model.

S1.23 INFILL DENSITY INFLUENCE ON A COPPER PLATED ABS OBTAINED BY FUSED FILAMENT FABRICATION

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Fused Filament Fabrication (FFF) is an additive manufacturing technology that is diffusing more and more in the academic and industrial field. FFF is characterized by various advantages such as a relatively low cost, a high level of customization and simplicity of obtaining complex shapes. In particular, many benefits derive from the possibility to control the so-called infill, a parameter that defines the shape and the amount of material used in the 3D internal structure. On the other hand, FFF parts have often some problematic issues such as high surface roughness and mechanical properties that are lower than those obtained with traditional forming techniques.

A possible solution to solve some of these problems is to use an electroplating technique that permits to coat a polymeric part with a suitable metal layer. This procedure is common in automotive and in electronic industry to improve surface appearance and thermal and electrical properties of injection molded products, but it is less common on parts made with FFF.

In this study, a two-step electroplating method that is free from toxic and precious metals, was performed directly on an Acrylonitrile-Butadiene-Styrene (ABS) substrate that was 3D printed previously. Specimens were built with various infill densities from 30% up to 100% and then plated with copper. Mechanical and thermal properties were measured to study the effect of this parameter. The mechanical properties were also modeled with the Finite Element Method (FEM) to evaluate the interface quality between copper and ABS.

The main results showed that an infill density between 50% and 60% is optimal to obtain adequate mechanical and thermal properties, since the infill density is sufficiently low that copper can penetrate into the sample creating a good mechanical interlocking with ABS, but at the same time it is sufficiently high to create several conductive bridges through the sample.

S1.24 ADVANCED NANOINDENTATION MEASUREMENTS: HIGH-SPEED 3D MAPPING ON COMPLEX MATERIALS FOR 4.0 DESIGN AND ERRORS MITIGATION STRATEGIES

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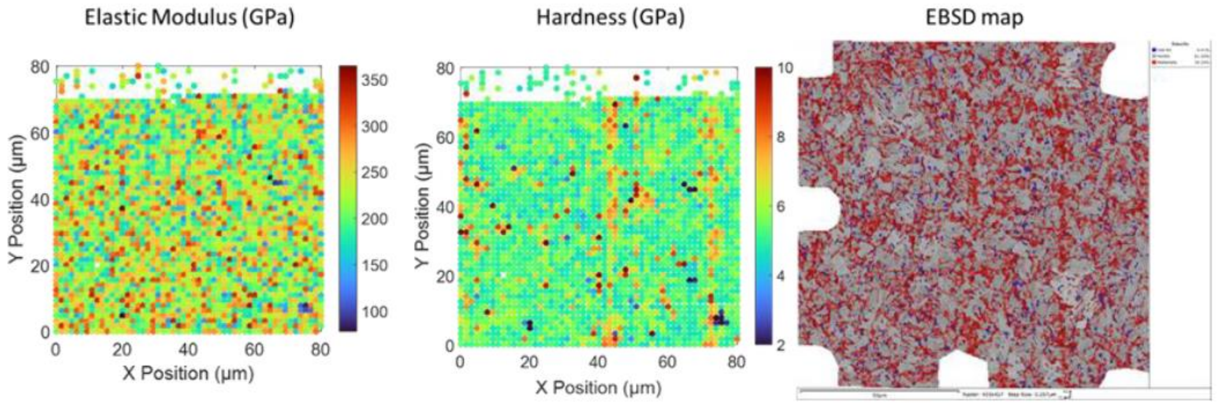
High-speed nanoindentation mapping can provide rapid (less than 1sec), high-resolution mechanical property measurements across the surface of a material. The technique allows, therefore, novel structural design routes for complex and heterogeneous materials. However, some essential considerations on measurement errors must be drawn from the intrinsic characteristics of this advanced methodology. This work will give an overview of this advanced technology, specializing in the use case of duplex stainless steel mechanical phase recognition (EBSD-aided) (Figure 1). This work introduces a novel robust methodology proposed to overcome one of the thorniest errors of all above: pile-up.
In those cases where this phenomenon is relevant, the most effective solution is to perform time-consuming direct measurements of the actual contact area on individual indentation marks (e.g., by Atomic Force Microscopy).



Elastic Modulus (GPa)

Hardness (GPa)

EBSD map

Figure 1 – Elastic modulus, Hardness high-speed nanoidentation maps in correlation with EBSD mapping for automotive duplex steels.

This work presents an alternative fast and straightforward protocol that uses high-speed nanoindentation to produce a large array of indents, generating a measurable change in the surface roughness quantifiable by fast optical profilometry analysis. It is demonstrated that the change in surface roughness (Sa) caused by the array of indents is strongly and uniquely correlated to the pile-up height. As validation, several materials (bulk and thin films) were investigated at different indentation depths to induce distinct pile-up levels.

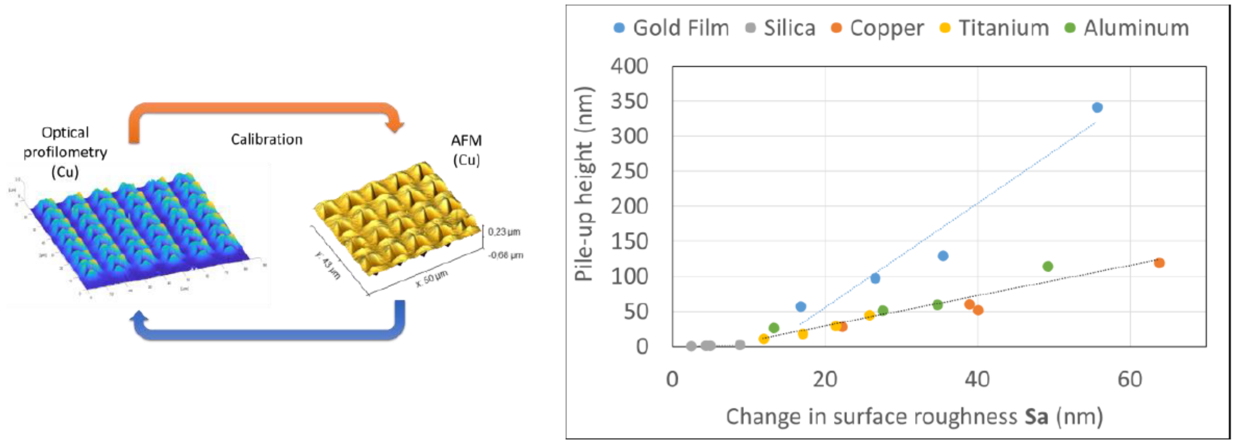


Figure 2 – (a) conceptual representation of the optical AFM-aided pile-up correction protocol (b) calibration curves for different materials according to pile-up correction protocol.

Correlation functions are demonstrated (Figure 1b) that allow the estimation of pile-up height and consequently perform correction of the hardness and elastic modulus quickly and reliably, overcoming limitations imposed by AFM acquisitions.

S1.25 THE INFLEUNCE OF GRAPHENE OR STAINLESS STEEL FLAKES ON THE MECHANICAL BEAHVIOR OF PORCELAIN ENAMEL

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Vitreous enamel coatings deposit on metallic substrates presents very interesting properties. High corrosion protection performance, good resistance against aggressive chemicals, weathering agents and thermal stresses are the most important properties.

However, same applications are limited by the brittle nature of the glassy matrix. A non-excellent abrasion resistance and high susceptibility to tensile stresses characterize enamel coatings. In some case, the resistance of the surface to mechanical stresses and damages influence the durability of the coating.

It is therefore important to improve enamel mechanical properties.

The aim of this study is to investigate how the addition of graphene platelets (0.25 – 1 wt.%) or 316L stainless steel flakes (1 – 5 wt.%), could influence the mechanical properties of enamelled AA5005 aluminium alloy.

The frit was mixed with a blue cobalt-based pigment, selected platelets or flakes, and screen-printing medium to obtain chosen formulations. A 2A/1F (2 applications and 1 firing at 570 °C) process was used.

As a first step, the microstructure of the deposits is evaluated to check their uniformity and adhesion with the substrate.

The abrasion behaviour of the coatings are investigated by P.E.I. measurement. The efficacy of the metallic additive in increasing the cracking resistance of the coatings was assessed by means of in-situ 3-point bending tests.

The addition of graphene platelets cannot be considered as an effective way to increase the abrasion resistance of the coatings, as agglomeration phenomena cannot be completely avoided.

The addition of 5 wt.% flakes is beneficial in increasing the coating's abrasion resistance. The stainless steel flakes undergo plastic deformation during the abrasive process. The presence of a high concentration of these flakes is beneficial in improving the cracking resistance of composite enamel coatings.

S1.26 LIFE CYCLE EVALUATION OF DOUBLE-STEP COLD BONDING PELLETIZED INDUSTRIAL WASTE LIGHTWEIGHT ARTIFICIAL AGGREGATES

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The researchers have been exploring ways to produce lightweight artificial aggregates (LWA) from industrial waste materials, reducing the demand for natural resources. This study focuses on the life cycle evaluation of double-step cold bonding pelletized industrial waste lightweight artificial aggregates (LWA). The study conducted a cradle-to-gate life cycle assessment, evaluating the environmental impact product life cycle stages, including transportation, raw material extraction, processing, and end-of-life disposal. The study also compared the environmental impact of double-step cold bonding pelletized industrial waste LWA aggregates with traditional natural aggregates. In this experimental study, fly ash derived from municipal waste incineration, marble sludge, and powdered granulated blast furnace slag (GBFS) were combined with cement to produce three different mixtures. The percentage of fly ash remains relatively stable (80%). The mechanical, environmental, and economical characteristics of LWA mix were investigated by altering the blast furnace slag and cement content. The AHP was performed to allow the establishment of optimal conditions with regard to the three combinations, on which convergence and compliance may exist in terms of environmental consequences (evaluated with LCA), economic repercussions (evaluated with LCC), and technical details. The results indicated that LWA aggregates comprised of fly ash (80%), blast furnace slag (5%) and a greater proportion of cement (15%) are the optimal solution, allowing for the sustainable preference of LWA. The results show that the production of double-step cold bonding pelletized industrial waste LWA consumes less energy, emits less greenhouse gases, and generates less solid waste than traditional natural aggregates. Additionally, the use of industrial waste materials reduces the need for natural resources, further reducing the environmental impact.

S1.27 INSIGHT INTO THE ROLE OF INTERPARTICLE SPACING ON THE MECHANICAL BEHAVIOUR OF BRICK-AND-MORTAR NANOCOMPOSITES

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Building nanocomposites with superior mechanical properties is an ongoing challenge. Innovative materials inspired by nature have raised huge interest thanks to their remarkable mechanical properties and impressive performance. Hierarchically organized microstructures, such as that of nacre, are capable of reproducing on large scale the mechanical properties of the microscopic reinforcement. However, a nonmonotonic dependence of the mechanical properties of the nanocomposite with filler content is found in the literature when the nanosheet content strongly increases.

In this work, the authors investigated the mechanical behaviour of graphite nanoplatelets (GNPs)/epoxy nanocomposite with a brick-and-mortar (BM) architecture vis-a-vis filler content. Samples with different filler content (from 60 to 90 vol%) have been fabricated following a top-down approach. A modified shear lag model is proposed to quantitatively describe the effectiveness of reinforcement and the behaviour of the elastic modulus, accounting for a discontinuous polymer distribution over the nanoparticle's surface at high filler content.

At low filler content, the polymer covers the entire nanoparticle surface, allowing a complete contribution of the particle surface to the stress transfer (Figure 2a). Increasing the filler content, the InterParticle Spacing (IPS) becomes very low, reaching the size of the polymer chain (2Rg), and its critical value (cIPS). In this condition, the particle surface is not entirely covered by the bounded polymer, and partially contributes to the stress transfer, as shown in (Figure 2b). The stress transfer is limited to a smaller area and the efficiency of reinforcement drops. Finally, a design parameter is proposed to determine the upper fraction of the nanofiller that ensures the achievement of the maximum elastic modulus in a well-aligned nanoplatelet composite.

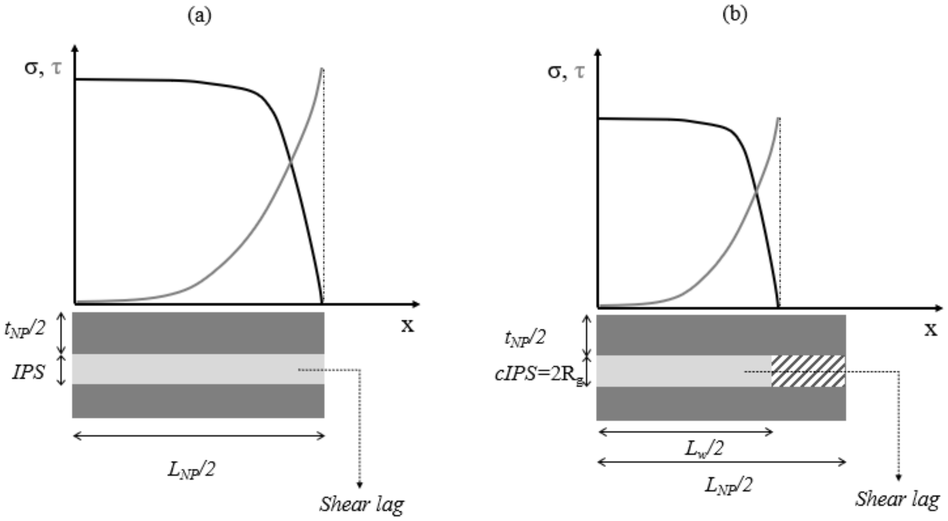

Figure 2. Stress state in nanocomposite in the case of (a) continuous matrix layer and (b) discontinuous matrix layer

S1.28 FUNCTIONALISED 3D PRINTED SCAFFOLDS AND IMPLANTS FOR CRANIOMAXILLOFACIAL APPLICATIONS

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The additive manufacturing technologies are gaining a lot of interest for biomedical applications, mainly for the production of custom made implants and scaffolds for the regenerative medicine [1]. The craniomaxillofacial sector is progressively growing and its market is expected to register a compound annual growth rate (CAGR) of 9.1% from 2022 to 2030. The custom-made strategy presents several advantages with respect to the traditional approach, e.g., shorter surgery times, reduced morbidity, lower infection/inflammation risk, higher aesthetical results, less blood loss, better correspondence between the implant and the graft site [2]. Biopolymeric materials are commonly used, but they present several criticisms, i.e. induction of inflammation, lack of osteointegration, low mechanical properties. In this context, graded scaffolds, as well as of implant prototypes, were produced by fused deposition modelling (FDM) technique is proposed. Different biopolymers (e.g., polylactid acid (PLA), polymethylmethacrylate (PMMA), polycaprolactone (PCL)) were tested, as well as different pattern depositions [3-5]. The functionalization of the produced structures with calcium phosphates and selected antibiotic/anti-inflammatory agents was proposed in order to promote the osteointegration and to avoid the post surgery inflammation occurrence, respectively. The thermal and mechanical properties of the produced scaffolds were investigated by differential scanning calorimetry (DSC), and tensile and compression tests, respectively. The efficacy of the coating deposition procedure was demonstrated by observation at scanning electron microscopy (SEM), and infrared spectroscopy (FTIR/ATR) measurements. The controlled release of the chosen anti-inflammatory agents was studied through tests in appropriate solvents and in phosphate buffer solution. Preliminary in vitro cytotoxicity tests were carried, as well as osteoblastic differentiation tests with osteosarcoma cells.

Acknowledgements

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S1.29 BIOCHAR FROM RESIDUAL AGRO-BIOMASS AS ENZYMES CARRIER

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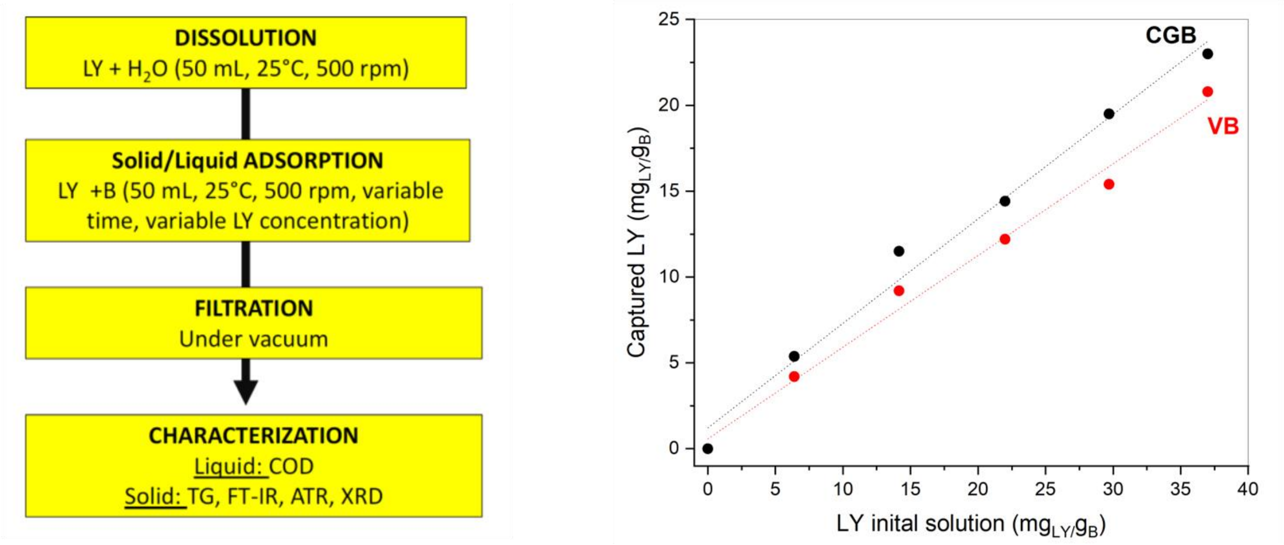
Biochar is the carbonaceous porous by-product of residual biomass gasification, known for its contribution to mitigate climate change. Indeed, during biochar production, carbon from the photosynthetic process is incorporated into biochar in a stable form. This means no CO_2 emissions when it is recycled for other purposes. In this context, the use of biochar as carriers of lysozyme (LY), selected as representative of bioactive molecules with antimicrobial properties, represents an innovative biochar application. Indeed, one of the challenges during oral administration is to avoid gastric degradation. Two biochar (B) derived from different residual biomass sources (vine and chestnut, VB and CGB respectively) have been tested as LY carriers. An experimental approach, based on a solid/liquid adsorption, was applied for LY immobilization (Fig. 1a). The effects of lysozyme concentration, pH and reaction time have been tested to find the better conditions for a complete LY adsorption.



(a) (b)

Figure 1. a) Biochar-supported LY preparation procedure, b) captured LY as function of the initial LY content.

For both the biochar a linear dependence was found between the LY initial concentration in solution and the amount of LY captured (Fig.1b). A maximum lysozyme loading of 21-23 mg/g$_B$ was achieved under the following operating conditions: 1.5 mg/mL as LY initial *concentration, pH 4.5 and 90 min of reaction time. Lysozyme – carriers interactions were proven by DTG analysis, where LY decomposition in hybrid material occurred at a different temperature than free LY, but did not seem to be dependent on the morphology of the support: LY (165 nm^3) is thus definitely too large to be hosted in the carrier pores (at most 40 nm). Considering biochar ZPC (-40mV), and LY ZPC, highly positive, interactions between opposite charges are expected to govern LY-B interactions. Finally, LY release efficiency by biochar will be tested in vitro conditions.*

S1.30 FIRE-RESISTIVE PERFORMANCE OF AGED SUSTAINABLE INTUMESCENT COATINGS FOR NAVAL APPLICATIONS

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Structural steel is largely used in naval construction sectors, due to its excellent mechanical strength. In this context, it is necessary that this material keep its integrity, even in fire event. Steel suffers a 50 % decrease of its mechanical strength when exposed to temperatures near to 500 °C, easily reached in conventional fire events. Intumescent flame-retardant coatings are highly recommended as passive fire protection as they could prevent fatal consequences. Intumescent coatings are composed of three flame-retardant additives (acid source, carbon source and blowing agent). The coatings form an intumescence char layer when exposed to heat, which acts as a thermal barrier that effectively protects the substrate against fire, maintaining the integrity of the metallic substrate. In this work, sustainable intumescent coatings were studied. Innovative formulations, filled with low environmental impact recycling materials (i.e. cork, recycled glass, clay nanotubes) were realized and characterized. To investigate the durability of the coatings, UVA irradiation was used at different aging time (300, 600 and 900 hours). Furthermore, coated panels were hung directly on a real ferry boat, in two different locations (engine room and helm room). At different aging times in situ, the panels were removed and characterized, to verify the maintenance of the intumescent properties. In addition to thermochemical and microstructural properties (i.e. TGA, DSC, XRD, SEM), the fire-resistive performance was investigated by means of a specifically designed fire resistance test. The results highlighted that all coating configurations evidenced suitable barrier/intumescent capacity. Comparing all unaged and aged batches, the best performances were highlighted by the recycled glass filled coating, in terms of fire protective behavior and durability, indicating this filler as a promising material for intumescent coatings design.

Acknowledgements

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S1.31 ELECTROCHEMICAL HYDROGEN CHARGING ON CARBON STEELS AND LOW ALLOY STEELS, EFFECTS ON FRACTURE SURFACES AFTER J-INTEGRAL MECHANICAL TEST

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The introduction of a substantial amount of gaseous hydrogen, recently identified as a viable energy vector, inside the already existing pipeline network designed for the transport and the distribution of natural gas will soon become a reality. Nevertheless, hydrogen is widely known for its impact on the mechanical properties of several materials, just like carbon and low alloy steels, which are frequently employed in the existing distribution and transport pipeline network and related services. For this reason, the compatibility of these materials needs to be thoroughly evaluated.

To test the response of such materials to the presence of hydrogen on laboratory scale, in literature several methods has been proposed to reproduce the high-pressure gaseous hydrogen environment.

This work will focus on the electrochemical hydrogen charging method, which exploits the evolution of atomic hydrogen on the surface of a cathodically polarized surfaces. The entry of atomic hydrogen into steels is promoted by the employment of recombination poisons, which inhibit the recombination of atomic hydrogen to its molecular form.

Three different solutions (combination of acid and recombination poison) were tested to electrochemically charge thick CT samples (up to 20 mm) of carbon and low alloy steels. To optimize the charging method a set of parameters were investigated, such as time, cathodic current density and recombination poison concentration.

The hydrogen charged specimens were successively subjected to J-integral test, carried out at the Department of Mechanics, Politecnico di Milano, to investigate the effect of HE on toughness. The fracture surfaces were analysed with SEM, to link alterations of the mechanical properties of the samples with different fracture morphologies.

S1.32 OPTIMIZATION OF A FRIT-FREE GLAZE FORMULATION FOR CERAMIC GLAZE AND ENGOBE PREPARATION

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Sustainable architecture seeks to minimise the environmental impact of buildings. Within the ceramic sector, the environmental impact of porcelain stoneware tiles can be decreased by modifying and optimising the industrial formulations. Floor and wall tiles are composed of four layers: the ceramic support, the engobe, the ink-jet decoration and the glaze. Between those layers, the preparation glazes involve frits, which are granulated glasses melted at high temperatures and then quenched in water. Frits' production process is thus energy intensive. As industrial energy efficiency has been in the spotlight during the 2022 energy crisis, focused studies to overcome unstable energy prices are essential in the building sector.

This work modified a conventional industrial glaze formulation by replacing the frit with a combination of raw and secondary raw materials such as recycled soda-lime glass, chamotte, and metakaolin. A Mixture Design with 20 formulations was adopted to determine each component's role in forming a glaze with similar properties to the conventional one (thermal behaviour, coefficient of thermal expansion and cleanability), but without the use of the energy-intensive frit. Three formulations were then optimised and further characterised by optical microscopy, hot stage microscopy, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), roughness, CIELab colour, cleanability and chemical durability. Afterwards, one of the optimised glazes was used to investigate how two different operational factors (granulometry and glaze thickness) modify the gloss and luminosity (L) properties of the final fired product (firing was done at 1205 °C). It was found that depending on the values of the operational factors, glazes (high gloss and low L*) or engobes (low gloss but high L*) can be obtained from the same frit-free glaze formulation.*

S1.33 A MULTISCALE INVESTIGATION OF WEAK INTERACTIONS BETWEEN POLYETHERIMIDE AND CARBON DIOXIDE

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This contribution illustrates a procedure aimed at the understanding, on a molecular scale, of the state of CO_2 *molecules absorbed in glassy polyetherimide (PEI). To this aim in-situ FTIR spectroscopy and gravimetry were implemented to gather relevant experimental information, while MD, DFT and statistical thermodynamics approaches were adopted to model the behavior of the binary system at different scales. Based on the outcomes of FTIR spectroscopy and on DFT and MD calculations, it was determined that the carbon atom of carbon dioxide molecules establishes relatively weak interactions prevalently with the carbonyl groups of PEI, whose quantitative estimation has been provided by MD calculations. Finally, the system was investigated on a continuum thermodynamic scale by adopting the Non-Equilibrium Theory for Glassy Polymers – Non-Random (NETGP-NR). This model, specifically developed by our group, takes into account the non-equilibrium nature of glassy polymers as well as the non-randomicity of mixing between polymer, penetrant and free volume. Once the NETGP-NR parameters were retrieved by a fitting of solubility data the model allowed to predict the isosteric heat of sorption and the carbon-dioxide – polymer interaction energy in agreement with experimental findings and DFT calculations respectively. Finally, experimental data of* CO_2*-PEI mutual diffusivity have been successfully interpreted using a semi-empirical theoretical model accounting for the effects of the penetrant concentration, of energy barriers associated to the occurrence of an effective diffusive jump and of a thermodynamic factor provided by NETGP-NR.*

S1.34 CHARACTERIZATION OF CYCLIC OLEFIN COPOLYMERS AS CONSTITUTIVE MATERIALS FOR INSULIN RESERVOIRS

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Type-1 diabetes is one of the most prevalent metabolic disorders worldwide. It results in a significant lack of insulin production by the pancreas and the ensuing hyperglycemia, which needs to be regulated through a tailored administration of insulin throughout the day. Recent studies have shown great advancements in developing an implantable artificial pancreas and, overall, one of the key components is the insulin reservoir since it has to store the insulin preventing the agglomeration of such a protein.

We discuss the employment of two types of Cyclic Olefin Copolymers (Topas 5013L-10 and Topas 8007S-04, inherently hydrophobic polymers) as constitutive materials for fabricating a 3D-printed insulin reservoir, as an alternative solution to Nylon 6 and Teflon.

After a preliminary thermomechanical analysis, Topas 8007S-04 was selected as the best material to fabricate a 3D-printed insulin reservoir due to its higher strength (UTS=56 vs. 40 MPa) and lower glass transition temperature (75°C vs. 130°C). Fiber deposition modeling was used to manufacture a reservoir-like structure (Figure 1A) and a mechanical characterization of the surface texture was carried out on three specific locations of the sample. The results showed a roughness (R_a) ranging in 6.84 - 8.55 µm and localized variations between peaks and notches in a range of 0 - 40 µm, generally higher than that observed for Nylon 6 or Teflon (Figure 1B). Spectrophotometric analysis carried out up to 14 days did not detect any statistically significant insulin aggregation (Figure 2). Based on these results, the manufactured Topas 8007S-04 showed that local irregularities of the surface had less impact on insulin aggregation than a global evaluation of the pattern (i.e., roughness).

These findings may disclose new opportunities in developing insulin reservoirs for implantable devices, in terms of biomaterials and manufacturing technologies.

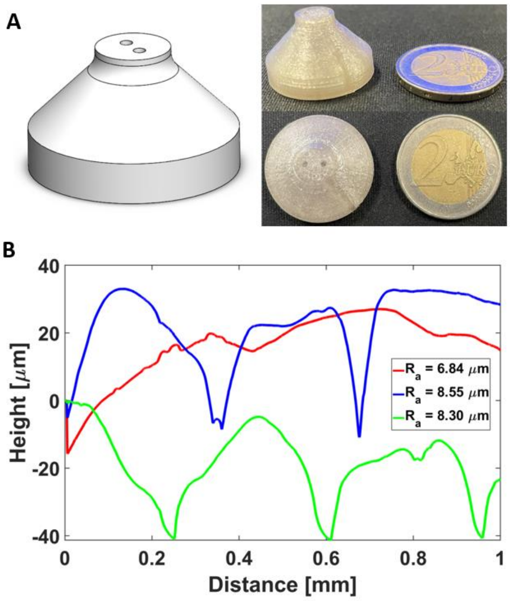

Figure 4. Fabrication and texture characterization of the reservoir to test the insulin anti-aggregation properties of the geometry/material. (A) From the CAD model to the 3D printed device (a 2 € coin for size comparison). (B)Roughness measurements on the surfaces of the insulin reservoir in three characteristic locations of the insulin reservoir.

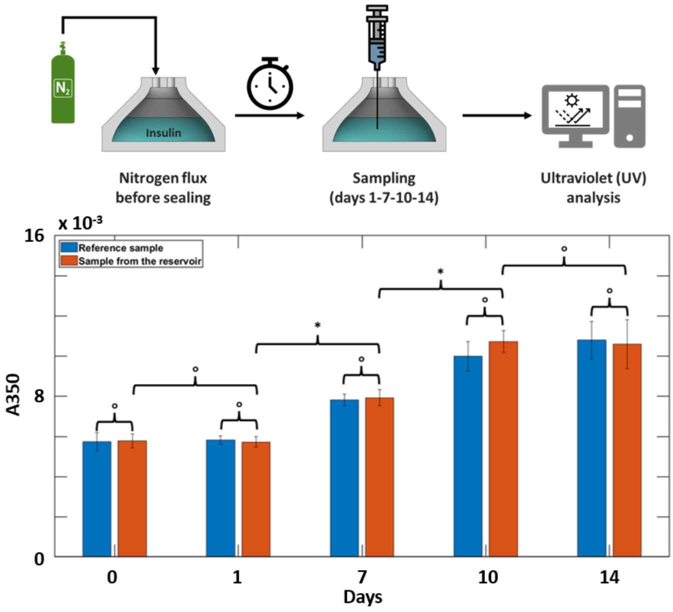

*Figure 5. Schematic showing insulin storage set-up and aggregation tests. After filling the tank with insulin, a gaseous nitrogen was fluxed to remove the air present inside, before a firm sealing. On day 1, 7, 10 and 14, a sample of insulin was taken from the tank for UV assays. Between sampling days, the tank was kept firmly sealed with an inert atmosphere made of nitrogen. Bar graph reporting the outcomes of UV analysis performed at an absorbance of 350 nm (A350) to assess the aggregation of insulin at 0, 1, 7, 10, and 14 days comparing insulin samples stored in the fabricated T8007 reservoir (orange bars) and pristine insulin, used as reference (blue bars). Results from the statistical analysis are reported with the following symbols: ° p-value ≥ 0.317; * p-value ≤ 0.005.*

S1.35 MULTIFUNCTIONAL ALUMINUM SURFACES

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High specific strength, low specific weight and relatively low cost are some of the main reasons for the widespread use of aluminum alloys in building construction, transportation industries and many other applications in daily life.

Some applications (e.g., printing plates, automotive and aerospace usage) need to have functionalized aluminum surfaces in which "functionalized" is defined as improving a feature or providing a new property to the material. In some cases, functionalization is used to improve the aluminum anti-icing and corrosion resistance (automotive and aerospace), while in other cases functionalization is used to give new properties such as a specific topography, and wettability (marine application, painting). Surface functionalization can be carried out with various methods, such as mechanical machining, chemical, laser, electrochemical, and ion beam surface texturing.

Chemical surface texturing stands out as a powerful method to conduct the functionalization of the surface because of its good properties, such as being environmentally friendly, reproducibility and low cost.

In the present paper, a superhydrophobic, self-cleaning surfaces were obtained on aluminum alloy substrate by three different surface chemical treatments. Preliminarily, the effect of aluminum surface treatments on wettability and morphology of the aluminum surfaces was assessed. The micro/nano rough structure of aluminum surfaces was evaluated combining scanning electron microscope and atomic force microscope analysis. The superhydrophobic behavior was assessed by performing water contact/sliding angles (WCA/WSA) measurements by sessile drop tests. Finally the electrochemical behavior of the surfaces was evaluated in simulated seawater solution. The results showed that all surface treatments stimulated the formation of a micro/nano structure with very high WCA. The as-prepared surfaces revealed also a good corrosion resistance behavior. Applications for this kind of surfaces range from easy-to-clean surfaces to anti-icing or anti-condensation functionalities that could be of interest for several sectors.

S1.36 REDUCED GRAPHENE OXIDE/WASTE-DERIVED TIO2 COMPOSITE MEMBRANES: A NEW MATERIAL FOR HYBRID WASTEWATER TREATMENT

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Efficient wastewater treatment and reuse are of primary importance in a world where water scarcity and droughts are increasingly frequent. Titanium dioxide nanoparticles (TiO_2NP) are widely employed in photocatalytic water treatments, despite some limitations due to their tendency to agglomerate and high bandgap. In this research, TiO_2NP were combined with reduced graphene oxide (rGO), a graphene-like material of proven metal ion capture ability, with the aim to obtain self-assembling membranes (rGO-TiO2) having double functionality (i.e. photocatalyst and adsorbent) for water treatment. Materials' integration simply consisted in the mechanical mixture of the two components followed by vacuum filtration, without the introduction of further chemicals. In a view of circular economy, a second type of membranes (rGO-TIO) was produced, according to the same procedure, replacing TiO_2NP with Tionite, a TiO_2-containing material resulting from TiO_2 production. Composition and morphology of both membranes were characterized through XRD, thermogravimetric and SEM-EDX analyses. Fe^{3+} and Cu^{2+} 3 mg/L aqueous solutions were considered to assess if the combination of rGO with TiO_2 or Tionite affects its capture ability, verifying that the produced membranes are effective in metal ions adsorption. Both types of membranes were tested in the photodegradation of Imidacloprid®, a common organic pesticide, as a proof of concept of their photocatalytic properties. Photodegradation efficiencies of 25% and 21% were registered for rGO-TiO2 and rGO-TIO, respectively. Results of capture and photodegradation experiments provide a promising, yet preliminary, demonstration of a potential use of rGO-TiO2 and rGO-TIO membranes in the remediation of wastewater containing both inorganic and organic pollutants. Decontamination performances of Tionite-based membranes are particularly significant, considering that a waste material, without any further purification, was employed in their production.

S1.37 DIGITAL LIGHT PROCESSING OF TEXTURED ALUMINA CERAMICS: SLURRY ELABORATION AND MICROSTRUCTURAL TAILORING

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Textured ceramics have attracted widely interest for applications in structural and functional ceramics. They have many superior properties compared with ceramics composed of randomly oriented grains. Indeed, fracture toughness along specific directions increases with the incorporation of oriented grains, which favour the mechanisms of crack deflection, crack bridging and grain pull-out. There are many examples of textured materials with excellent performances in nature, such as nacre, bone, and bamboo. Textured ceramics have been produced by a variety of techniques: tape casting, magnetic alignment, template grain growth (TGG) and, recently, additive manufacturing.

3D printing can overcome traditional ceramics shaping limitations, such as reduced design freedom and post-production machining, and further promote the use of technical ceramics. Up to now, only few works investigated 3D printing of textured alumina ceramics via Digital Light Processing (DLP) and no studies explored the fabrication of textured alumina/zirconia composites.

Therefore, in this work, we demonstrate the feasibility of fabricating textured alumina and alumina/zirconia composites via DLP. Alumina platelets were used to template only a α-alumina powder as well as in ternary compositions with a 3 mol% yttria-stabilized zirconia. Aim of the study is to open the path for 3D printing textured ceramics for structural and functional applications. Different photocurable slurries were prepared and studied in terms of alumina platelets/alumina/zirconia ratios and solid loadings. Optimisation of rheological properties and printing parameters was fundamental for platelets orientation and printing accuracy. Mechanical properties were strongly influenced by the orientation degree (determined via FESEM observations and XRD analysis) and density. High strength ceramics were successfully fabricated opportunely tuning the composition and sintering conditions.

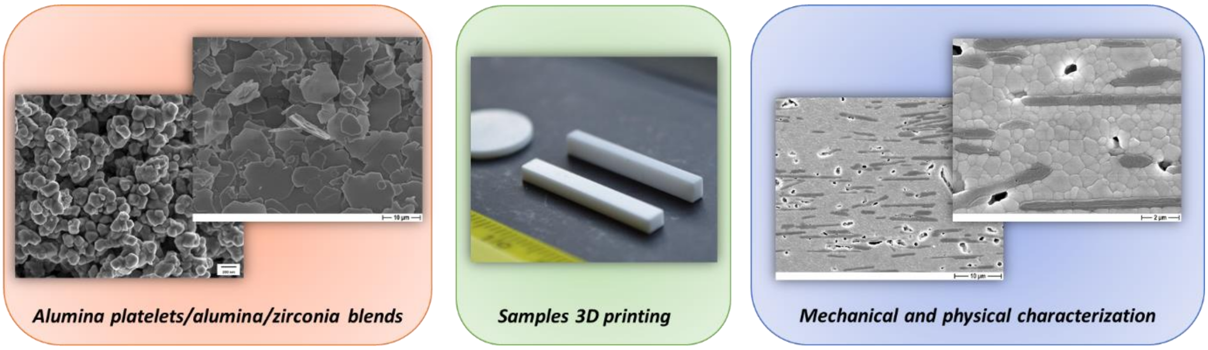

Figure 6. Schematic representation of the experimental flow: powders characterization and slurries preparation; samples 3D printing via DLP; physical and mechanical characterization (FESEM, XRD, flexural strength, etc.).

S1.38 ANODIZING OF AlSi10Mg ALLOY PRODUCED BY L-PBF: EFFECT OF SURFACE FINISHING AND SEALING

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Anodizing is an electrochemical process used to improve the wear and corrosion resistance of aluminium and its alloys. After anodizing, many Al-based alloys form compact and protective oxide layers. Conversely, the presence of large Si precipitates in cast Al-Si alloys results in defect-rich anodic layers. AlSi10Mg alloy is among the most studied materials processed via Laser-Powder Bed Fusion (L-PBF). This technique exposes the alloy to high cooling rates, leading to the formation of a fine Si network surrounding a-Al cells. This microstructure could allow to obtain good quality anodic layers even in a Si-rich alloy. In this work, AlSi10Mg samples obtained via L-PBF underwent anodizing and sealing. To evaluate the influence of surface morphology, we finished the samples by sandblasting with glass microspheres, tumbling, or mechanical polishing before anodizing. The microstructure of the oxide layers was observed by means of scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The anodic layers were found to consist of anodized Al cells surrounded by voids and partially oxidized Si particles. Inside the Al cells, the oxide showed its typical columnar structure with hexagonal-shaped cells (Figure 1). Due to the uniform distribution of fine silicon particles, tumbled and polished samples developed uniform oxide layers. By contrast, sand blasted samples produced defect-rich oxide layers, because of the irregular morphology of the surface and the presence of residual glass microspheres embedded in the material. The quality of the oxide layer influenced the corrosion behaviour of the material, tested in 3.5 wt.% NaCl solution. Indeed, the corrosion potential and current of tumbled and polished samples were significantly improved after anodizing, and sealing further increased the corrosion potential up to a value of about +150 mV. Conversely, the defects in sandblasted samples resulted in a lower corrosion resistance of anodized samples.

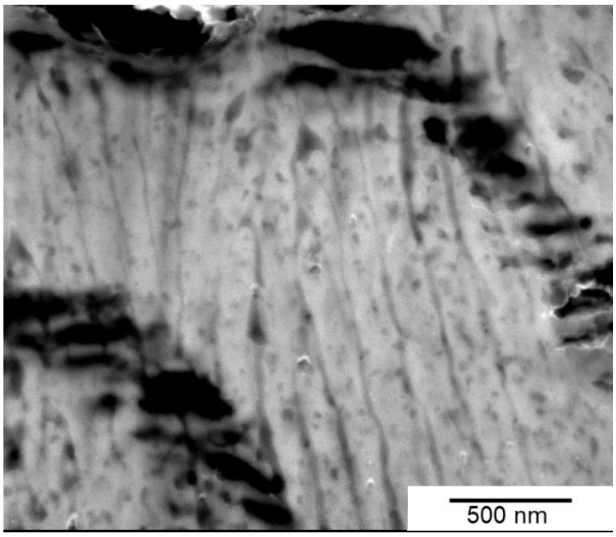

Figure 1. Cross-sectional TEM micrograph showing the columnar microstructure of the anodic layer grown on L-PBF AlSi10Mg alloy.

S1.39 NOVEL APPROACHES FOR THE MANUFACTURING OF HOLLOW COMPOSITES PARTS USING NOVEL WAX-BASED BLENDS

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Manufacturing of hollow composites materials is a challenging task that requires complex procedures using soluble or flexible materials. The use of soluble materials relies on creating a near net shape mandrel onto which shaping the uncured laminates. Once the part is cured the soluble mandrel is removed washing it away. This approach requires the composite parts to be exposed to water or even more aggressive fluids that can seriously decrease the final composite's properties. Flexible materials are used for compacting the uncured laminates during curing. The shaping is usually realized using two halves' molds. This approach makes easy to extract the flexible mandrel after curing but, it does not offer any possibility to layup directly on the mandrel. This is a limit as it binds to the use of matched molds and, for these reasons, can limit the shape's complexity while increasing costs. The research on materials for composite's tooling is raising the interest of industry but, at the same time, the correct integration within the curing cycle should be considered. In this work a novel approach combining the use of novel blends based on the use of waxes mixed with magnetic particles is presented. The use of waxes is proposed because of their low cost and possibility to be removed by simply melting back to its fluid state after composite's curing. The use of magnetic particles is proposed to overcome the low thermal inertia of waxes by using induction heating. The results obtained demonstrated that novel wax-based blends can be used for the manufacturing of mandrels by simple casting that can be simply removed from the cured parts melting it back and reusing for further processing cycles (Figure 1). A further advantage demonstrated using this approach was obtained with the controlled thermal expansion of the wax that can give a contribution to the compaction of the composite's laminates (Figure 2).

Figure 7. An example of a mandrel obtained using a novel wax-based blend.

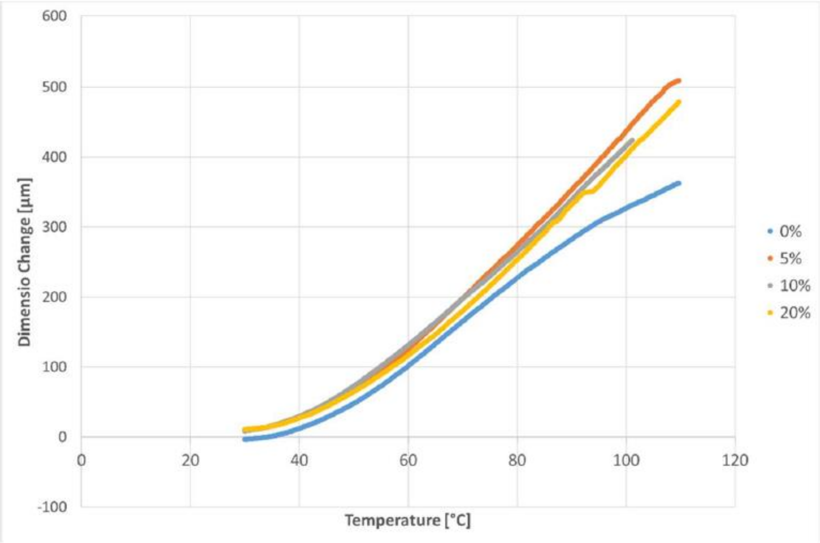

Figure 2. TMA analyses result for wax based blends with different particle's content.

S1.40 THERMOMECHANICAL CHARACTERIZATION OF THERMOPLASTIC NANOCOMPOSITES BASED ON THE IN-SITU ANIONIC POLYMERIZATION OF POLYAMIDE 6

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The in-situ anionic polymerization of polyamide 6 (PA6) is an interesting method to produce thermoplastic nanocomposites with high mechanical and functional properties. This study aims to investigate the thermomechanical properties of anionic PA6-matrix (aPA6) nanocomposites and correlate it with microstructural features such as the crystallinity degree and the nanofiller dispersion.

The nanocomposites were prepared in a lab-made small-scale polymerization plant as follows. Caprolactam (CL) was melted at 110 °C in a round-bottom flask under nitrogen flow, followed by the addition of a commercial initiator and activator in variable concentrations and the nanofiller (expanded graphite nanoplatelets (xGnPs) (Figure 1), organomodified nanoclay) in proper amount. The stirred mixture was then injected into a mold preheated at varying temperatures (140-170 °C), and left to polymerize for 20 minutes.

While all the investigated nanofillers mostly increased the elastic modulus, the quasi-static properties at break and the impact properties resulted to be strongly affected by the nanofiller dispersion. The mechanical properties of the nanocomposites were found to be influenced by the processing parameters such as the temperature and the concentration of the initiator and activator, mostly due to the profound impact of these parameters on the matrix crystallinity. All the investigated nanocomposites presented a high degree of conversion with little (<2%) residual caprolactam, as investigated by thermogravimetric analysis.

This study provides valuable insights into the properties and behavior of thermoplastic nanocomposites based on aPA6. While the results suggest that these nanocomposites have potential for use in various applications where improved mechanical, thermal, and electrical properties are required, further research is needed to fully characterize the interfacial interactions between the fillers and the matrix and to improve the filler dispersion, also with the introduction of surface functionalization.

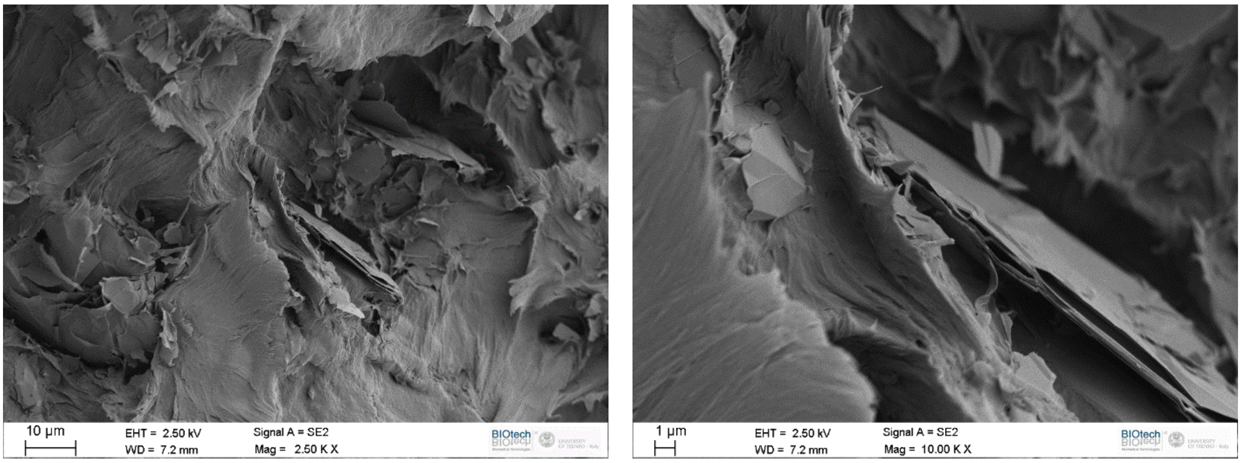

Figure 8. SEM micrographs of the cryofracture surface of PA6/xGnP nanocomposites (xGnP content = 0.5 wt%) at two different magnification levels.

S1.41 CORROSION OF AL ALLOYS TANKS IN BIO ETHANOL BLENDED GASOLINE FUEL

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Biofuels are suggested as substitutes for fossil fuels in transport applications to reduce greenhouse gas emissions. In vehicles using Al alloy tanks, contact with this type of fuel can lead to corrosion phenomena. This work investigates the evidence of corrosion, close to the welded joints, on Al alloy (AA1050) tanks in contact with E27 biofuel (i.e. 27% v/v ethanol) under incubation for 1000 hours at 60 °C. The fuel was analysed, by means of FT-IR analysis and mass spectrometry, before and after interaction with the material, checking for the presence of alcoholates, acetic acid and water, parameters that can affect the stability of the passivating layer of the alloy. The tank alloy was analysed by SEM and EDS, before and after corrosion. The presence of oxides on the samples' surface was investigated by grazing angle XRD. Using electrochemical analysis, the corrosive phenomenon was analysed on a laboratory scale.

A significant water content (about 0.025 %) was observed in the freshly produced fuel, which did not generate the corrosion, whereas the fuel used under operating conditions showed a content up to ten times lower. FT-IR analyses of the fuel excluded the formation of alcoholates. Similarly, the presence of acetate ion, and thus acetic acid, was not observed, which allowed the oxidation of ethanol to acetic acid to be ruled out as a cause of corrosion. The welded specimen showed a corrosion potential of E_{corr}*=-0.27 V when in contact with fresh produced fuel, suggesting greater corrosion resistance than the unwelded specimen, which showed a corrosion potential of* E_{corr}*=-0.36 V.*

The water content within the fuel appears to play a key role in the corrosion phenomena of the Al alloy. Freshly produced fuel containing a sufficient amount of water favours passivation of the alloy rather than its corrosion. In fact, the presence of water led to the formation of protective oxides which blocked the corrosion phenomenon, as the surface was ennobled.

S1.42 MICRO AND NANO-STRUCTURE OF POLY-LACTIC ACID WITH THERMAL, MECHANICAL, TRANSMISSION ELECTRON MICROSCOPY AND X-RAY DIFFRACTOMETRY ANALYSES

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In an age of rapid globalisation, the everyday life is essentially based on plastics which, at the end of their life, are incinerated, dumped in landfills, or left in the environment where they endure for hundreds of years. An innovative alternative to conventional plastics can be bioplastics, whose market has been growing steadily. Polylactic acid (PLA) is considered the second most important bioplastic globally by volume of consumption and by its properties, which are comparable to those of traditional petroleum-based plastics. In this study, we aim to find a correlation between the micro- to nano-structural properties of PLA and the resulting macroscopic characteristics, to render the industrial processing of this novel bioplastic as performant as possible. Different studies will be carried out both from a thermo-mechanical point of view and from a transmission electron microscopy and X-ray diffractometry. Firstly, the analysis of the thermal and mechanical properties will allow us to estimate the different contributions of the various constituent interfaces characteristic of polylactic acid and how and to what extent these may impact and/or influence the macroscopic characteristics of the system. Subsequently, transmission electron microscopy and X-ray diffractometry will enable us to correlate specific micro- and nanoscopic analyses to the various processing and manufacturing conditions of the biopolymer, whether amorphous or crystalline, plasticised, or non-plasticised. Indeed, by considering one of the major issues related to PLA, i.e., its intrinsic rigidity, more complex systems such as PLA-plasticised will also be investigated. In this way, we will propose an analysis as close as possible to the industrial world of bioplastics processing, also with a view to the circular economy, which can be, by means of appropriate modelling tools, optimised, and predicted to enable industrial manufacturing to be as efficient and feasible as possible according to world demand.

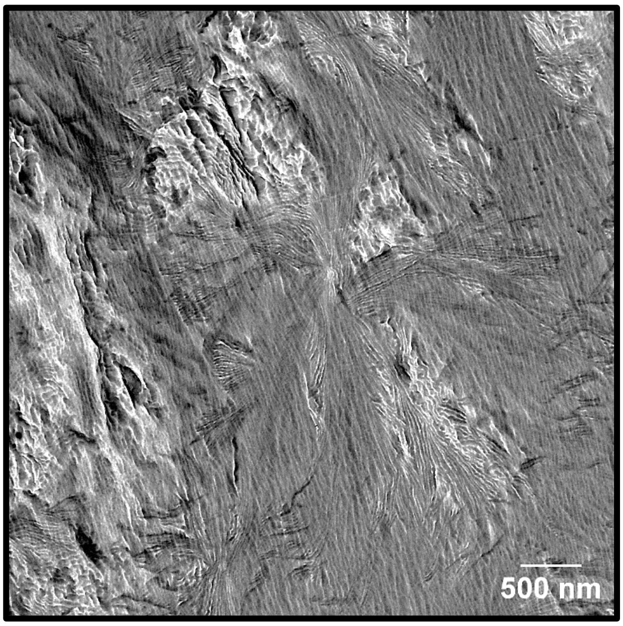

Figure 9.TEM imaging of a Poly-lactic Acid Spherulite.

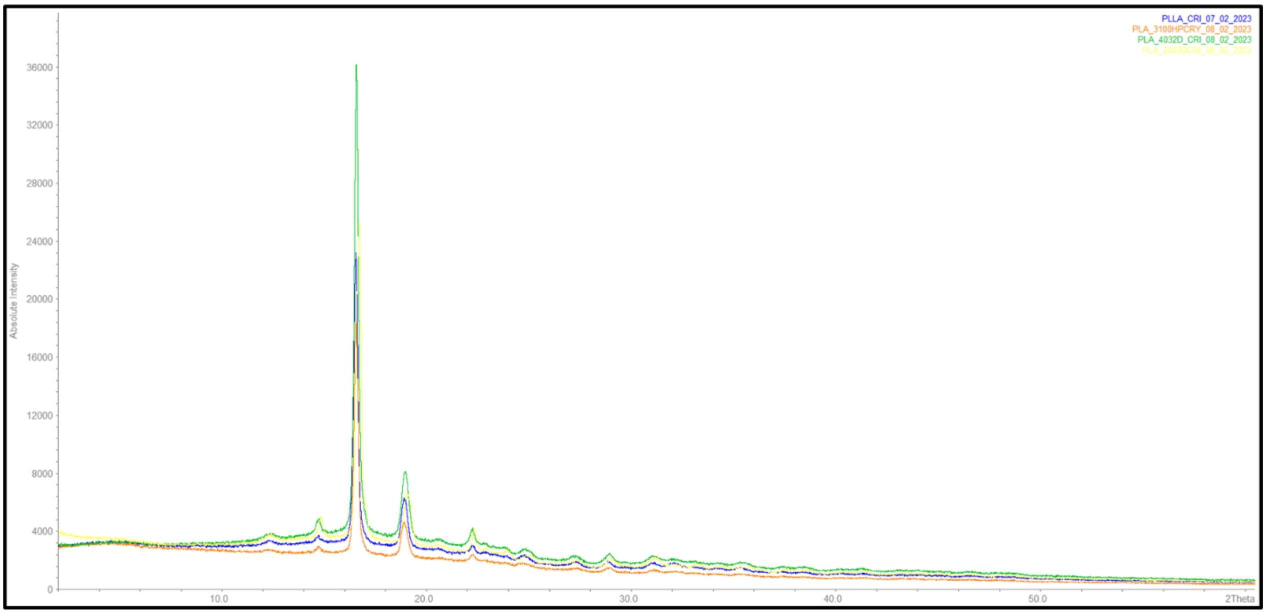

Figure 2. XRD Diffraction Studies on the influence of D-isomer percentage in Poly-lactic acid Samples.

S1.43 EFFECT OF DIFFERENT SURFACE TREATMENTS ON GRADE 5 TITANIUM ALLOY FOR IMPROVING THE ADHESION WITH THERMOPLASTIC BASED COMPOSITES

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In this study, different surface treatments were performed to evaluate the mechanical improvements of thermoplastic fiber metal laminates (TFMLs). The latter were made of a grade 5 titanium alloy (Ti6Al4V) and carbon fabric reinforced polyether ether ketone (PEEK) composite substrates, manufactured by compression molding. An electrochemical surface treatment, i.e., anodizing, that allows to grow a titanium oxide nanotubes layer on titanium alloy surface was studied to evaluate the effectiveness of this morphology in increasing the mechanical strength of FMLs. Parameters such as voltage, time and anodizing bath composition were studied to find the optimal conditions that would guarantee the maximum diameters of the nanotubes thus improving the interlocking with the matrix. Specifically, solutions of ammonium fluoride (0.5%wt), ethylene glycol and water were used. The voltage values chosen were 30V and 50V whereas the water volume contents were 5%, 20%, 50%. For each voltage and water content, the samples were immersed for 10, 30 and 60 minutes at room temperature. In order to induce the crystallization in the prepared nanotubes, a thermal treatment in a muffle furnace (i.e., 450°C for 60 min) was performed soon after the anodizing process. SEM analysis and contact angle measurements were carried out to characterize the morphology of titanium nanotubes and their wettability. After these tests, only two conditions were selected: 30V, 50% v/v water (30V50P) and 50V, 20% v/v water (50V20P), both for 60 minutes. In addition, mechanical abrasion treatment with sandpapers (MT) and chemical treatments (SIL) with commercial silane (3-aminopropyl trimethoxy silane) were performed to compare them with the proposed treatments. A mechanical characterization of FMLs was performed by evaluating their fracture toughness and interlaminar shear strength through double cantilever beam (ASTM D5528) and short beam shear (ISO 14130) tests, respectively. The overall results show that 30V50P anodizing leads to the best performance of the produced FMLs.

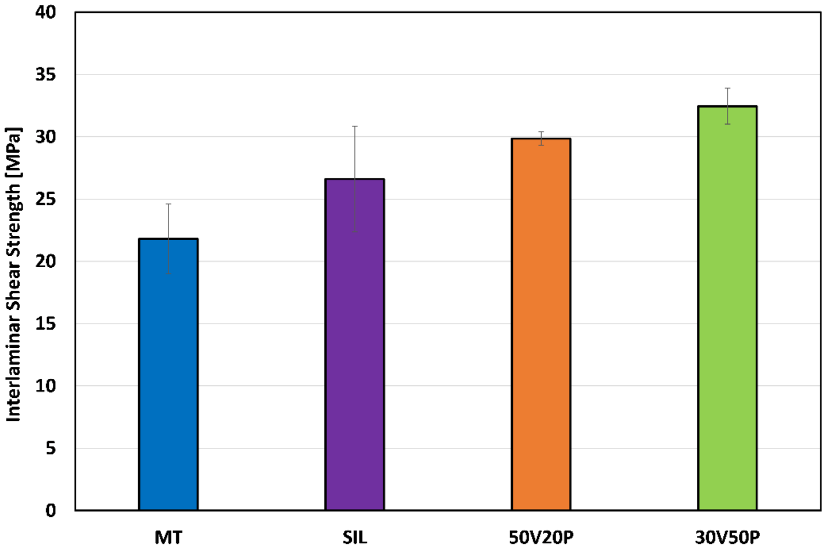

Figure 1. Interlaminar shear strength by short beam shear test varying the surface treatments on titanium alloy substrate of fiber metal laminates.

S1.44 TITANIA NANOTUBES ARRAYS DECORATED WITH COPPER: EFFECT OF THE SUBSTRATE PRETREATMENT ON MORPHOLOGY AND ELECTROCHEMICAL PERFOMANCE OF THE CATHODES

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The production of materials with vertically aligned nanotubes (NTs) entails the possibility of obtaining large surface areas and therefore enhanced electrocatalytic and photo-electrocatalytic abilities. Pristine titania NTs are generally used as photoanodes in water splitting. Much more often NTs arrays are coated with organic substances or metal oxides or even mixed metal oxides. Only a limited effort has been so far devoted to the fabrication of NTs coated or decorated with metals in the elementary state.

On the other hand, some metals such as copper, for example, have shown interesting electrochemical properties in the environmental field, particularly in the electroreduction of NOx or in the conversion of CO_2 *to organic molecules.*

This work investigates the possibility to develop cathode materials by galvanostatic electrodeposition of copper micro-particles on differently treated titanium-based surfaces. The surface modifications will include etching with hydrogen fluoride and anodization in a glycerol-containing electrolyte to promote the growth of an ordered titania nanotube array. The effect of an intermediate thermal treatment before deposition will be *also evaluated.*

The morphology, structure, and electrochemical properties of the cathodes will be characterized by SEM imaging, XRD spectra, corrosion tests, EIS spectra, and cyclic voltammetry before and after use.

Finally, the materials will be tested in the reduction of aqueous solutions containing nitrate or chlorate.

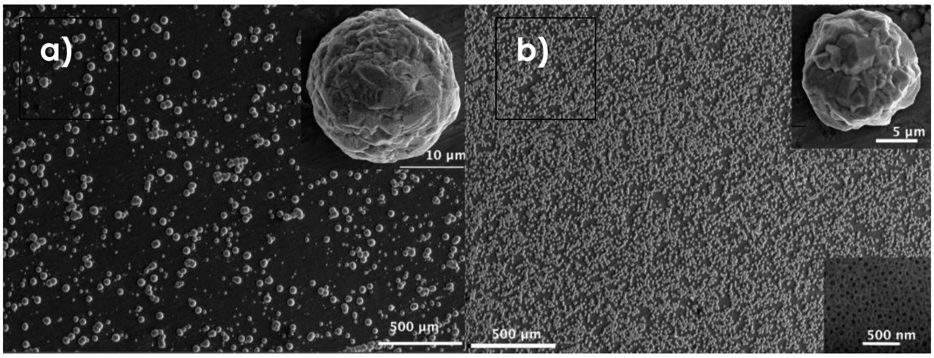

Figure 10. SEM images of electrodeposited Cu on Ti *(a) and* TiO_2 *nanotubes (b)*

SESSIONE 2		
Codice	**Titolo**	**Corresponding Author**
S2.1	COEXTRUDED FOOD PACKAGING FILMS BASED ON BIODEGRADABLE BLANDS COMBINING BARRIER PROPERTIES, THERMAL RESISTANCE AND LOW TEMPERATURE DUCTILITY	Annalisa Apicella
S2.2	ELECTROPHORETIC DEPOSITION (EPD) OF MUCOADHESIVE CHITOSAN-METHYLCELLULOSE ORAL PATCHES FOR THE TREATMENT OF LOCAL MOUTH BACTERIAL INFECTIONS	Lina Altomare
S2.3	OPUNTIA FICUS INDICA/MATER-BI® BASED GREEN COMPOSITES FOR FERTILIZER CONTROLLED RELEASE DEVICES PRODUCTION	Maria Clara Citarella
S2.4	SELECTIVE POSITIONING OF DIFFERENT CELL TYPES ON 3D SCAFFOLDS VIA DNA HYBRIDIZATION	Enrico Domenico Lemma
S2.5	A SELF-ACTIVATED MULTI-LAYER MORPHING MATERIAL	Giulia Lanzara
S2.6	THERMAL CONDUCTIVITY ENHANCEMENT OF TiN AND SiO_2 NANOFLUIDS	Veronica Testa
S2.7	OPTIMIZATION OF MICRO ARC OXIDATION COATINGS ON AZ31 MAGNESIUM FOR CORROSION CONTROL IN BIOLOGICAL APPLICATIONS	Matteo Pavarini
S2.8	MULTIFUNCTIONAL PIEZOELECTRIC NANOFIBERS FOR ACOUSTIC AND MECHANICAL APPLICATIONS	Giulia Lanzara
S2.9	ATTRACTIVE APPLICATIONS ON MAGNETIC PARTICLES	Giulia Lanzara
S2.10	THE IMPACT OF AG CONCENTRATION ON THE DURABILITY AND BIOCIDAL ACTIVITY OF WOOD PAINTS EMPLOYING COLLOIDAL SILVER AS A NEW MULTIFUNCTIONAL PIGMENT	Massimo Calovi
S2.11	BIO-INSPIRED MATERIALS FOR STRUCTURAL MORPHING	Giulia Lanzara
S2.12	ENCAPSULANT FILMS FOR BIFACIAL HETEROJUNCTION PHOTOVOLTAIC MODULES: PERFORMANCE AND DURABILITY	Nadka Tz. Dintcheva
S2.13	SUSTAINABLE BIOPLASTIC FOR SUN BLOCK PATCHES AND TAPES MADE FROM REGENERATED SILK AND DNA	Luca Valentini

S2.1 COEXTRUDED FOOD PACKAGING FILMS BASED ON BIODEGRADABLE BLENDS COMBINING BARRIER PROPERTIES, THERMAL RESISTANCE AND LOW TEMPERATURE DUCTILITY

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Despite the undoubted ecological advantages of biodegradable polymers, their applications in the food sector are still limited due to their low functionalities. In particular, there are severe limitations in the application of biopolymers over a wide temperature range, i.e. for foods subjected to hot filling processes followed by frozen storage.

In this study, we investigated both melt blending and coextrusion techniques to realize a novel bilayer biodegradable packaging with barrier performance, heat sealability, and suitable for either hot-filling and frozen food products.

The films were realized by combining an inner layer, made of Ecovio and amorphous poly(lactide) (PLA4060) blend and providing high ductility and sealability, with an outer layer made of poly(butylene succinate) (PBS) and semicrystalline PLA4032 blend contributing to barrier properties and thermal resistance. Prior to making the bilayer structures, monolayer films based on different compositions of these blends were produced and tested, to identify the optimal concentration of each blend for the target application. Then, three coextruded structures were realized by varying the relative thicknesses of both layers and were analyzed for their physicochemical, surface, mechanical, barrier and overall migration properties. All the coextruded films exhibited high interlayer adhesion and overall migration values below the limits established by the European legislation. Moreover, the bilayer films had functional performance intermediate with respect to those of single outer and inner layers, and weighted on their relative thicknesses.

Among all, the bilayer film with the thickest outer layer thickness (50 µm) showed the best balance between stiffness and ductility in the explored temperature range (-18 °C to 85 °C) and proved to be an effective sustainable alternative to conventional fossil-based films for food packaging applications involving both hot-filling and freezing.

S2.2 ELECTROPHORETIC DEPOSITION (EPD) OF MUCOADHESIVE CHITOSAN-METHYLCELLULOSE ORAL PATCHES FOR THE TREATMENT OF LOCAL MOUTH BACTERIAL INFECTIONS

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Mucoadhesive buccal patches are dosage forms promising for successful drug delivery. They show the distinctive advantage of long residence time on the oral mucosa and increased in situ drug bioavailability. This local administration of antibiotics may represent an advantage in the management of oral infections as compared to the systemic route. Different mucoadhesive films and patches based on natural polymers, have been reported in the literature to treat local mouth bacterial infections. CS-based buccal patches may suffer from weak mucoadhesion, impairing their therapeutic effect. In this work, methylcellulose (MC) was exploited to increase the mucoadhesive character of pristine CS patches. CS-MC patches were obtained in a one-pot process via EPD, and the possibility of incorporating gentamicin sulfate (GS) as a model of a broad-spectrum antibiotic in the so-obtained patches was investigated.

All specimens appeared homogeneous at SEM observation, with random porosity created by the evolution of the H_2 bubbles (Fig1). A predominant presence of CS in the CS-MC patches, with a mean CS fraction of 66.8 %, was detected.

The resulting CS-MC patches displayed high stability in a water environment and superior mucoadhesive character (σ_{adh} = 0.85 ± 0.26 kPa, W_{adh} = 1192.28 ± 602.36 Pa mm) when compared with CS control samples (σ_{adh} = 0.42 ± 0.22 kPa, W_{adh} = 343.13 ± 268.89 Pa mm), due to both the control of the patches porosity and the bioadhesive nature of MC. Furthermore, GS-loaded patches displayed no in vitro cytotoxic effects against L929 cells and antibacterial activity on both Gram-positive and Gram-negative bacterial strains.

In this study, we demonstrated the possibility to obtain, via a one-pot EPD process, novel mucoadhesive CS-MC patches with broad antibacterial activity by adding GS sulfate to the solution. The presence of MC provided CS-MC samples with superior mucoadhesive character, while GS provided a broad antibacterial activity on selected bacterial strains (E. coli and S. aureus).

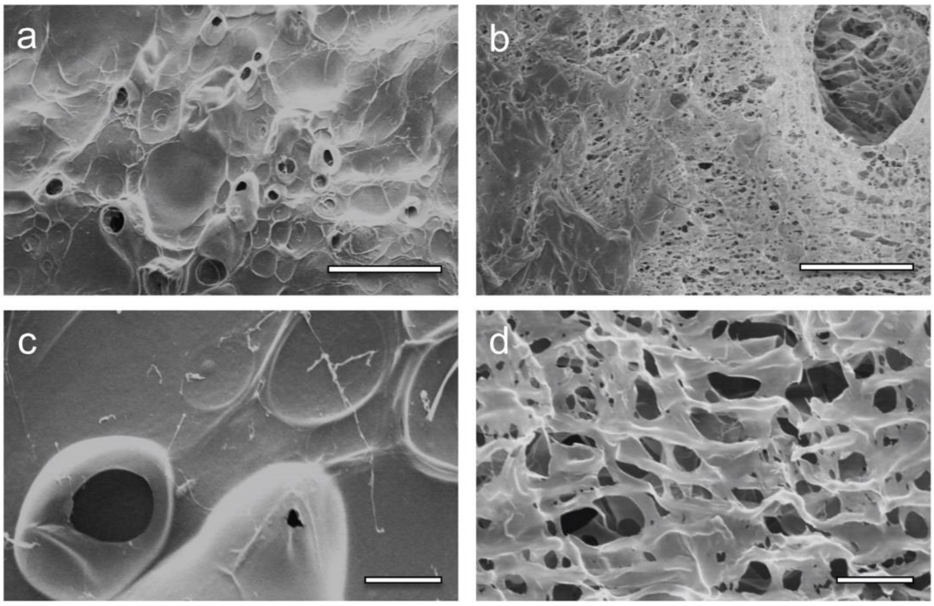

Figure 11. SEM micrographs of a) c) CS and b) d) CS-MC samples. a) b) Scale bar = 200 μm; c) d) Scale bar = 20 μm.

Acknowledgements

This study was carried out within the Agritech National Research Center and received funding from the European Union Next-GenerationEU (PIANO NAZIONALE DI RIPRESA E RESILIENZA (PNRR) – MISSIONE 4 COMPONENTE 2, INVESTIMENTO 1.4 – D.D. 1032 17/06/2022, CN00000022).

S2.3 OPUNTIA FICUS INDICA/MATER-BI® BASED GREEN COMPOSITES FOR FERTILIZER CONTROLLED RELEASE DEVICES PRODUCTION

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Excessive fertilization causes ecological concerns due to leaching issues. To solve this problem and promote agriculture sustainability an innovative green composite for controlled release fertilizers was produced by adding NPK fertilizer flour to a biodegradable polymer (Mater-Bi®) with or without Opuntia Ficus Indica (OFI) particles. Six formulations, formulations namely MB/OFI-A, MB/OFI-B, MB/NPK-A, MB/NPK-B, MB/OFI-A/NPK-A and MB/OFI-B/NPK-B, were successfully produced and employed for the fabrication of devices both for compression molding (CM) and fused deposition modeling (FDM). Both filler and fertilizer displayed good dispersion in the composites and excellent adhesion with the polymeric matrix (Figure 1).

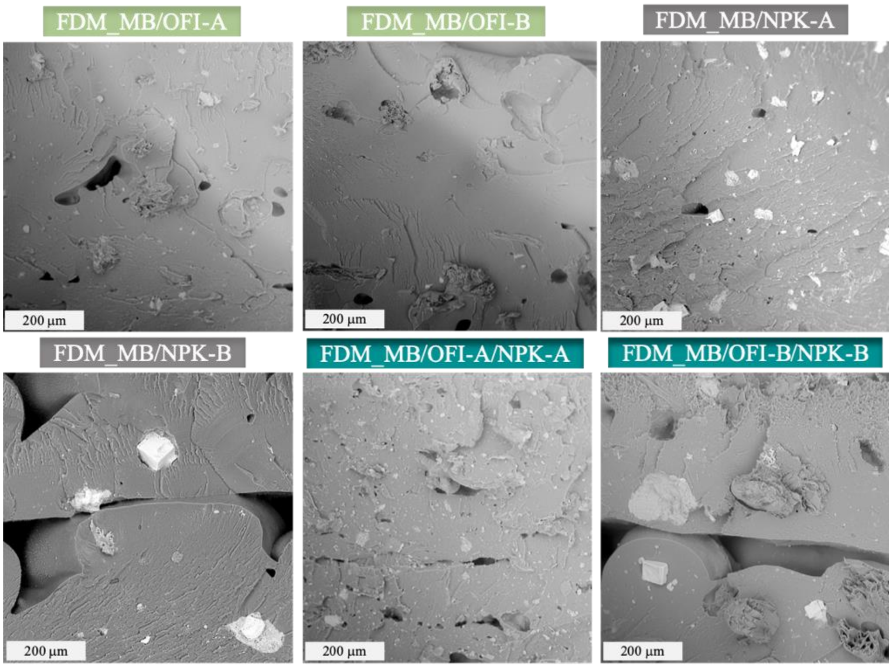

Figure 1. SEM micrograph of fractured cross-section of green composites and CRF devices fabricated for FDM.

FDM samples showed higher mechanical properties if compared with compression molded ones. Release tests of CRF devices reveal the ability of all the obtained composites to slow the release rate of NPK (up to 30 days), which proved to be tunable by modifying formulation, flours granulometry and production techniques. The porous structure of FDM samples, induced by the process itself, promote the water transport across the devices *allowing the release of the NPK also from the more inner layer. In particular, FDM_MB/OFI-A/NPK-A act as the best CRF device showing a remarkable burst delivery with about 70% of NPK released in the first 24 hours and a 100% release after 30 days (Figure 2).*

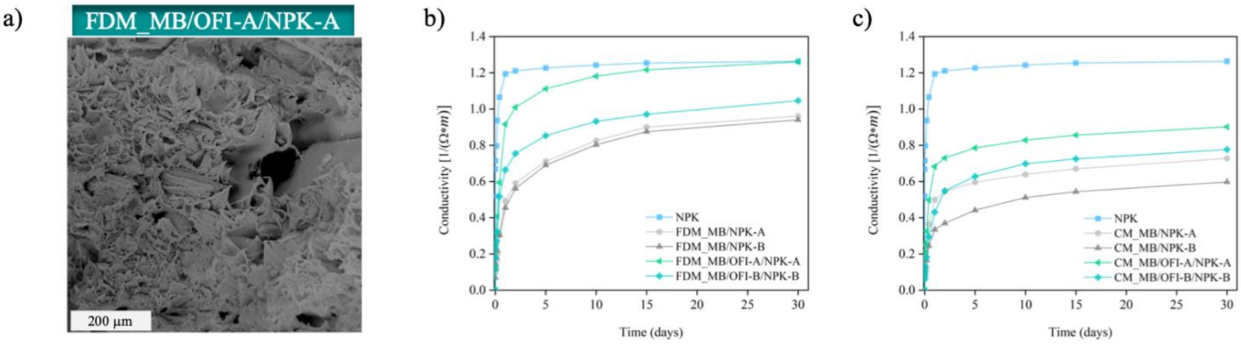

Figure 2. SEM micrograph of FDM sample after soaking in water for 30 days (a). Fertilizer release monitoring by conductivity measurements as function of time for FDM (b) and compression molded CRF devices (c).

By appropriately selecting the dimension of the particles, the addition of OFI and the production technique, was possible to modulate the NPK release rate: FDM samples containing fine particles of OFI and NPK displayed the fastest release. Release data were fitted according to Peppas-Korsmeyer model to understand the release mechanism.

S2.4 SELECTIVE POSITIONING OF DIFFERENT CELL TYPES ON 3D SCAFFOLDS VIA DNA HYBRIDIZATION

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In the last decade, a great effort has been devoted to the establishment of in vitro platforms mimicking the complexity of cellular and tissue microenvironment. Two-photon lithography (2PL, fig. 1a) has given a major contribution to the field, and several scaffolds for studying cell biology made via 2PL have been reported[1]*. A further improvement is represented by techniques for the precise positioning of distinct cell types in a 3D microenvironment. Current techniques, e.g., antigen-antibody interaction, pose severe limitations for selective single-cell-type tagging as they lack specificity*[2]*. Here, we report the decoration of 2PL-obtained 3D microscaffolds with single-strand DNA (ssDNA) exploiting light-induced click chemistry. An acrylate-based 2PL resin including an UV-reactive molecule (i.e., photo-enol, PE, fig.1b) was formulated and characterized for photopolymerization parameters (i.e., intensity and dose, fig.1c), for further surface decoration with biomolecules. The aldehyde group of PE enolizes upon UV irradiation and subsequently reacts with a carbon double bond (e.g., of a maleimide molecule) via a Diels-Alder [4+2] cycloaddition click reaction*[3]*. Therefore, a solution of biotinylated maleimide in DMF was placed on the 2PL-made 2D and 3D microstructures and exposed to a focused 405 nm laser, resulting in the covalent binding of the maleimide to the PE; streptavidin was then incubated, followed by an incubation step with a fluorescent biotinylated oligonucleotide (fig.1d). The degree of functionalization could be controlled by tuning the laser intensity and exposure time. The procedure was repeated sequentially for two different oligos both on 2D (fig.1e) and on 3D structures (fig.1f). To test the selective binding affinity of cells to ssDNA, two different cell lines (i.e., NIH3T3 and U2OS) were decorated with cholesterol-TEG complementary strands and incubated on functionalized 3D scaffolds (fig.1g). As expected, cells hybridize more effectively on complementary oligos than on surfaces with non-complementary ssDNA (fig.1h). Selectivity of adhesion on functionalized structures was quantified also with several other cell lines, including stem cells (fig.1i). Current studies focus on scaling up this methodology to the meso- and macroscale and on widening the palette of acrylate-based photoresists, thus enabling the fabrication of 3D scaffolds using consumer-grade stereolithographic 3D printers.*

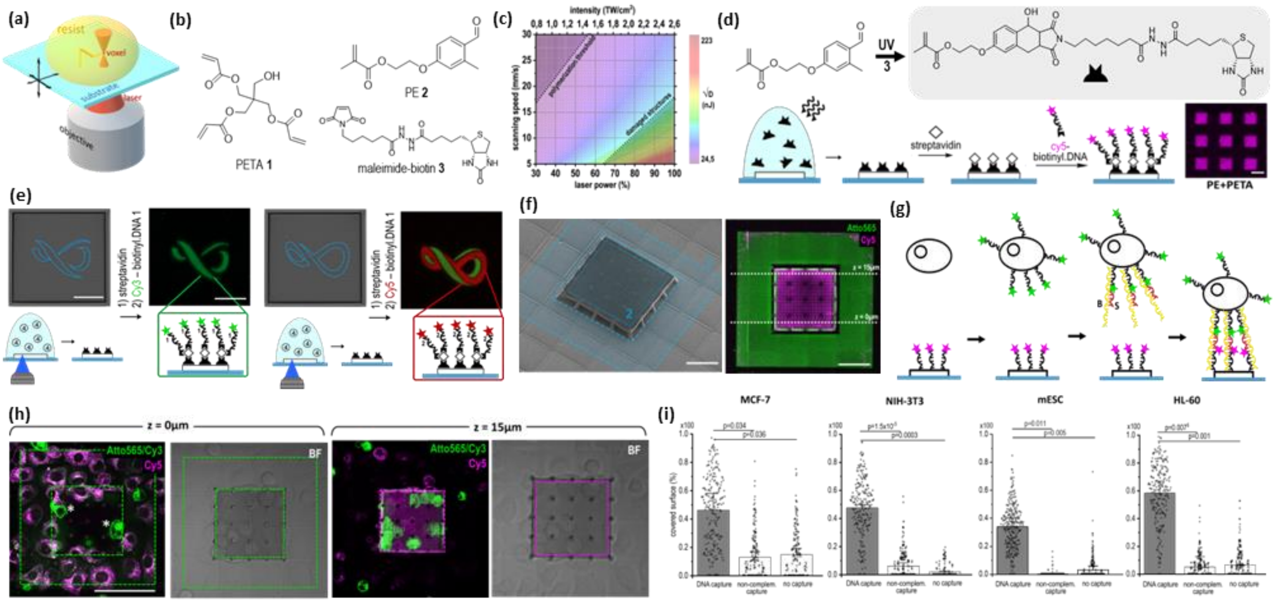

Figure 12.

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[2] *Richter et al., Adv. Mater. 29 (2017)*

[3] *Gruendling et al., Macromol. Rapid Commun. 32 (2011).*

S2.5 A SELF-ACTIVATED MULTI-LAYER MORPHING MATERIAL

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Some biological systems are able to modify their structure and functionality in response to an external stimuli. Smart and shape-changing materials try to resemble such a function via properly engineered structures. The most sophisticated among these types of materials are those that self-activate themselves. The automatic adaptation to the environment in fact has the great potential to reduce energy consumption. Here, a unique multi-layer system that adapts its shape when exposed to thermal gradients, is presented. Such a design logic finds its roots into the strategic combination of materials that possess an opposite glass transition temperature and that can be plastically deformed at the desired temperature setting. This is conceptually different from the classical multi-layer systems that exploit the difference in thermal expansion coefficient of the overlapping layers in order to induce internal stresses in the material. In particular the system presented in this paper, consists of a bottom polyethylene (PE) layer and a top polyethylene terephthalate (PET) layer. This bi-layer structure has the beauty that can be "educated" to exploit its functions at the desired set temperatures, for instance: flat at low temperature (0° C) and curved shape at higher temperatures (20° C). It is worth noting that such a temperature gradient is extremely narrow, thus the ability to induce the material to radically change shape, is, in principle, extremely challenging with conventional design approaches. One of the uniqueness's of the proposed design is that such morphing actions are cyclic without the need of re-educating the material (as typically occurs in shape memory polymers). The possibility to fine-tune with great repeatability and precision the shape of the film as a function of temperature, is demonstrated.

This study is being carried out through a research project funded by the Italian Space Agency. The described unconventional morphing material will be used to implement a bio-inspired coating for energy saving in space satellites.

S2.6 THERMAL CONDUCTIVITY ENHANCEMENT OF TiN AND SiO₂ NANOFLUIDS

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A Proton Exchange Membrane (PEM) fuel cell (Figure 1) is considered the eco-friendly and sustainable solution in term of electricity generation to substitute the fossil fuel and reduce carbon emission in automotive industries. Fuel cells operate at temperatures below 100°C and produce electricity as a result of the reaction of hydrogen and oxygen. Nanofluids are recently employed in cooling system for PEM due to great heat transfer properties: added nanoparticles, typically with a size on the order of 1 to 100 nanometers dispersed throughout the base fluid, significantly improves the thermal conduction. These nanoparticles can be made from a variety of materials, including metals, nitrides (ex: TiN), oxides (ex: SnO_2, TiO_2, Al_2O_3, SiO_2), and carbon-based materials. The recent widespread use on nanofluids in PEM fuel cells is increased by the need to reduce the cooling system size in the stacks, which is favourable in vehicle applications.

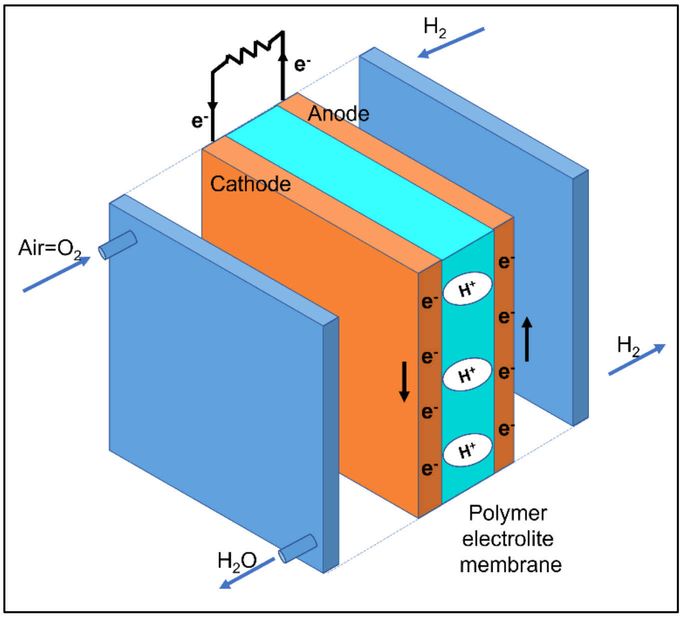

Figure 13. Scheme of a Proton-exchange membrane fuel cell system

This work is focus on the use of TiN and SiO_2 nanoparticles mixed in water – ethylene glycol at different concentration with the aim to improve the performance and efficiency of cooling systems. The increased thermal conductivity of the nanofluid can help to dissipate heat from the engine more effectively, which can lead to improved fuel efficiency and *reduced emissions. The shear viscosity, the thermal conductivity and the chemical stability are investigated in order to find the optimal composition. The performance of each nanofluid composition was observed and compared to base liquid, including a cost evaluation, potential toxicity limits and environmental risks associated with the use of the nanoparticles.*

The use of TiN and SiO_2*, alone or in combination, enhances heat dissipation capability because of associated heat transfer coefficient in comparison with base fluid water-ethylene glycol.*

S2.7 OPTIMIZATION OF MICRO ARC OXIDATION COATINGS ON AZ31 MAGNESIUM FOR CORROSION CONTROL IN BIOLOGICAL APPLICATIONS

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Introduction

Magnesium alloys represent promising bone substitutes due to their good mechanical properties combined with their bioresorbability. However, Mg corrosion in vivo is often too fast, impairing the material's load-bearing ability while also causing a strong inflammatory response due to the abrupt increase in local pH. Electrochemical treatments like Micro-Arc Oxidation (MAO) can be exploited to slow down Mg corrosion by forming protective ceramic coatings, thus also supporting bone healing at the implant site. In this work we present the optimization of MAO coatings on AZ31 magnesium alloy for prospective application in bone repair.

Materials and Methods

MAO treatments were applied to AZ31 Mg using a Si-based electrolyte with or without cerium introduction. Different MAO frequencies were tested, and their effect on the coatings' features was investigated by SEM and XRD. The samples' corrosion resistance was tested by potentiodynamic polarization in simulated body fluid, while the coatings' adhesion was assessed by scratch testing. The cell response was evaluated on hMSCs by indirect cytotoxicity in degradation products-containing medium.

Results and Discussion

All the coatings feature the porous morphology typical of macro-spark MAO. By increasing the process frequency, their composition shifts towards a higher forsterite content, gradually replacing the periclase phase. Accordingly, high frequency coatings show a greater corrosion resistance, and this is further improved by cerium incorporation; moreover, Ce also leads to a significantly higher coating adhesion, reducing the risk of delamination. Both these aspects are crucial for a longer-term stability of the material. Also, the biological assays show a strongly improved viability of hMSCs in presence of Ce-rich degradation products, ascribable to the antioxidant behavior of cerium in alkaline environments.

All these results show the strong potential of Ce-doped, MAO coated Mg as a solution for bone fixation.

S2.8 MULTIFUNCTIONAL PIEZOELECTRIC NANOFIBERS FOR ACOUSTIC AND MECHANICAL APPLICATIONS

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PVDF is an advanced multifunctional material with excellent piezo/pyroelectric properties. Electrospinning of PVDF micro/nanofibers has been extensively investigated over the past decade as a green solution over the traditional piezo materials due to their ease of processability with non-toxic chemicals and low production cost. The piezo micro/nanofibers can be applied into several novel applications ranging from advanced sensing materials to smart textiles. In this context we present the implementation of electrospun PVDF nanowires to form non-woven ultra-light and porous membranes as well to form pre-defined patterns of 3D printed nanowires, for advanced sensing materials which can also be used in structural health monitoring systems, by exploiting the direct piezoelectric effect. Here the structural vibrations are monitored in both contact and non-contact mode. In the first case the nanowires play the role of a strain/vibration sensor, while in the latter case the work as acoustic sensors. For strain sensing applications the PVDF web was installed on a fiberglass composite material resembling a cantilever beam structure that is fixed at one end and left free to vibrate on the other. The strain sensor was able to register an excellent electro-mechanical response. The alternative use of pre-defined patterns has the beauty to allow to define local deformations. In the acoustic sensing application, the electrospun PVDF membrane was suspended with elastics orthogonally to a microfilament composite material that was mechanically stretched until failure. The mechanical vibrations of the composite filament breakages were clearly registered in the electrical acquisition of the sensor over time. The corresponding breakage frequencies were finally transformed into a novel concept of 'Speech of Materials'. The detected fingerprints of such a speech will be discussed in detail.

S2.9 ATTRACTIVE APPLICATIONS ON MAGNETIC PARTICLES

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Magnetic particles are highly attractive nanomaterials due to their specific properties and biocompatibility, identifying a wide range of possible processes in modern technology. Here, the functional response of solid and liquid composite drops of Fe3O4 nano- and micro-particles is investigated. It is observed that a drop of magnetite particles in water, placed in contact with a substrate, modifies the substrate roughness and thus its wettability. The corresponding chemical, morphological and surface energy analysis will be discussed as a function of the magnetite content and interacting substrate. On the other hand, a liquid drop of a Fe3O4 nanocomposite alginate solution solidifies through instantaneous cross linking when in contact with a calcium solution. This ultra-fast cross-linking gives rise to a magnetic alginate micro-sphere whose size and shape is fine-tuned through the process optimization. The interaction of liquid and solid composite spheres with an external magnetic field, will also be discussed and will highlight the great potential of the analyzed drops for a variety of applications as micro-robots, drug delivery, smart glues as well as for the realization of magnetically activated smart materials.

S2.10 THE IMPACT OF AG CONCENTRATION ON THE DURABILITY AND BIOCIDAL ACTIVITY OF WOOD PAINTS EMPLOYING COLLOIDAL SILVER AS A NEW MULTIFUNCTIONAL PIGMENT

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The purpose of this study is to evaluate the impact of two different colloidal silver concentrations on a water-borne wood paint's durability and biocidal efficacy. Colorimetric measurements and optical microscope observations were used to assess the impact of this multifunctional pigment on the coatings' aesthetic qualities. Via a variety of accelerated degradation tests, including UV-B radiation exposure and climate chamber exposure, the samples' durability was evaluated. To verify the residual effect of silver in changing the protective behavior of the paint, scanning electron microscope observations, infrared spectroscopy analysis, and colorimetric inspections were carried out. Furthermore, the samples that incorporated silver had high antibacterial activity against Staphylococcus aureus and Escherichia coli, while colloidal silver had no noticeable fungicidal activity against Coniophora puteana and Trametes versicolor fungi. In conclusion, this research demonstrates how colloidal silver can be employed as a functional pigment in wood paint, able to change the coating's appearance and improve its antibacterial ability without adversely affecting its protective performance.

S2.11 BIO-INSPIRED MATERIALS FOR STRUCTURAL MORPHING

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Natural systems represent a great source of inspiration for the design of innovative materials. Such systems have in fact evolved through slow but extremely effective processes, and are often made of highly sophisticated hierarchical structures that exhibit multifunctional behaviors. For instance, they are able to detect an external stimulus and respond to it in a predetermined manner over time. One of the most intriguing aspects of biological systems, for instance in flying creatures, is that they are able to augment the performance at reduced energy consumption by tailoring their shape during flight. From an engineering perspective, such a behavior could be reinterpreted as the capability of materials to modify the shape while carrying loads. However, this is contradictory in terms of mechanical properties of materials: shape changes require low stiffness while load-bearing needs rigidity. Here we review some of the material design strategies that are being implemented in the STM – MultiMat Laboratory (Multifuncitonal Materials Laboratory). In particular, two key approaches are investigated by taking inspiration from the bat wing which is known to be ultra-light and flexible with the capability to tune its rigidity. A carbon fiber composite with an unsymmetric layup and with a thermosetting epoxy resin whose rigidity can be modulated with temperature, transforms the composite into an unprecedented mechanical actuator which exploits an unexpected impressive capability in lifting loads during shape transitions. On the other hand, a novel 2D patterning of silicone micro-filaments integrated with Fe_3O_4 nanoparticles, is implemented to draw magnetically activated nanoscopic textiles on an ultra-light and thin PVC film. Once exposed to a magnetic field, the textile stiffens the film over 100% with a negligible mass increase. Here we demonstrate that the patterned membrane can be induced to morph into radically different shapes, emulating the various configurations that are typically assumed by a bat wing during its flight.

S2.12 ENCAPSULANT FILMS FOR BIFACIAL HETEROJUNCTION PHOTOVOLTAIC MODULES: PERFORMANCE AND DURABILITY

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Nowadays, the energy recovery from renewable sources is a very attractive and challenging issue. Therefore, production of photovoltaic (PV) modules, as efficient device for energy recovery, becomes a consolidate and prosperous industrial practice and the formulation of suitable encapsulant materials, having good performance and durability, is a very important concern, see Figure 1.

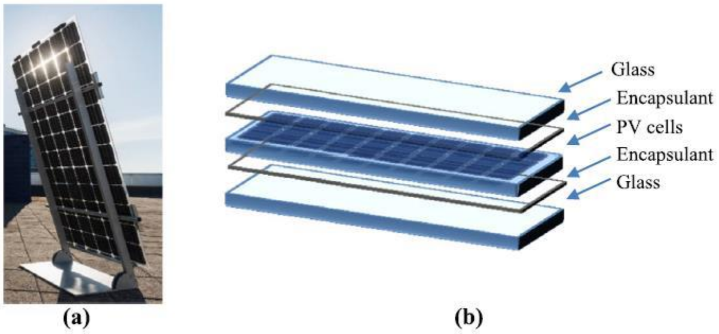

Figure 1. Novel, highly reliable bifacial heterojunction glass/glass PV module: (a) image and (b) schematic structure representation (by 3SUN - ENEL Green Power, Italy).

In this work, encapsulant films based on PolyEthylene Vinyl Acetate (EVA) and PolyOlefin Elastomer (POE), containing appropriate additives, before (pre-) and after (post-) lamination process, have been accurately characterized by differential scanning calorimetry, rheological and mechanical analysis, FTIR and UV-visible spectroscopy analyses. To simulate industrial lamination processing conditions, both EVApre-lam and POEpre-lam sheets have been subjected to prolonged thermal treatment upon high pressure and accurate analysis in terms of photo- and thermo- oxidation resistance, has been carried out. Additionally, the replacement of silan-based adhesion promoter using a polar wax for the formulation of encapsulant films has been evaluated too.
The durability, in terms of photo- and thermo- oxidation resistance, of pre-laminated and post-laminated EVA and POE sheets, has been accurately evaluated upon UVB exposure and prolonged thermal treatment, and the progress of degradation has been monitored by spectroscopy analysis. All obtained results agree that the lamination process has beneficial effect on 3D-structuring of both EVA and POE sheets, and after lamination, the POE shows enhanced rigidity and appropriate ductility. However, although both EVA and

POE can be considered good candidates as encapsulants for bifacial PV modules, it seems that the POE sheets show a better resistant to the oxidation than the EVA sheets.

S2.13 SUSTAINABLE BIOPLASTIC FOR SUN BLOCK PATCHES AND TAPES MADE FROM REGENERATED SILK AND DNA

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Bioplastics made from protein, polysaccharide, etc., have been extensively investigated in the last years. Materials such as silkworm silk and DNA can be considered as inexhaustible biopolymers, as be extracted from any organisms including plants and animals. To date, these materials have not been reported for applications to protect human skin from sunlight. In this study, patches and tapes are realized as thin films or non-woven, aligned-spun fiber textile material by exploiting the dissolution of silk fibers (e. g. regenerated silk (RS)) in different solvents capable of dissolving salmon sperm DNA. Circular dichroism, infrared spectroscopy and X-ray diffraction were used to investigate the conformational transition of RS and DNA solutions in different conditions and the structure of solid RS/DNA bioplastic. The RS/DNA bioplastic are water-insoluble and thermally stable. We therefore present the validation of our proof of concept based on biological results on cellular metabolism (MTT assay) after exposures to the simultaneous combinations of solar spectrum that consist in the emission of UVB, UVA, VIS and nIR components. The obtained bioplastic absorbs UV radiation, is pressure sensitive, adhesive once applied to the skin and improves the metabolic activity of HaCat cells. This adhesive does not leave any residue on the skin after its removal. Finally, RS fibers spun by direct extrusion in the air, create a water insoluble tape with controllable secondary structure offering a useful method for packaging and storage applications.

	SESSIONE 3	
Codice	Titolo	Corresponding Author
S3.1	PROCESS DEVELOPMENT OF CuCrZr ALLOY VIA ELECTRON BEAM POWDER BED FUSION	Stefano Felicioni
S3.2	A COMPARATIVE STUDY OF PLA/GRAPHENE NANOPLATELETS MEMBRANES PREPARED BY ELECTROSPINNING OR SOLUTION BLOW SPINNING	Emmanuel Fortunato Gulino
S3.3	Ti_6Al_4V MANUFACTURED BY COUPLING FUSED DEPOSITION MODELING (FDM) AND SINTERING: MECHANICAL PROPERTIES OPTIMIZATION	Claudia Sergi
S3.4	PURE POLY (PENTAMETHYLENE 2,5-FURANOATE) AND POLY (BUTYLENE 2,5-FURANOATE) ELECTROSPUN MATS: CHARACTERIZATION AND APPLICATIONS	Sofia Santi
S3.5	Inkjet printed Ti_3C_2 electrodes for anode-free Zinc-ion battery	Eugenio Gibertini
S3.6	TAILORING MORPHOLOGY AND MECHANICAL PROPERTIES OF PLA/PBSA BLENDS OPTIMIZING THE EXTRUSION PROCESSING PARAMETERS WITH LUDOVIC® SIMULATION SOFTWARE	Vito Gigante
S3.7	DECARBONIZING THE CONSTRUCTION INDUSTRY BY REDUCING THE CLINKER CONTENT IN CEMENT AND THE CEMENT CONTENT IN CONCRETE: THE PROMISING AND READY-TO USE SUSTAINABLE SOLUTION OF LC3 (LIMESTONE CALCINED CLAY CEMENT) CONCRETE	Beatrice Malchiodi
S3.8	HIBRID POLYMERS MEMBRANES FOR SEPARATION OF HYDROGEN/METHANE MIXTURES	Lucio Bonaccorsi
S3.9	CATHODIC PROTECTION MONITORING: DESIGN OF A NEW SOLID INTERNAL BACKFILL FOR POTENTIAL PROBES	Marco Ormellese
S3.10	DEVELOPMENT OF FeSi STEEL WITH INCREASED SI CONTENT BY LASER POWDER BAD FUSION TECHNOLOGY FOR FERROMAGNETIC CORES APPLICATION: MICROSTRUCTURE AND PROPERTIES	Giulia Stornelli
S3.11	RATIONAL DESIGN OF NEW SUSTAINABLE INTEGRATED SYSTEM FOR INDOOR CULTURES	Monia Montorsi
S3.12	STUDY OF THE INCORPORATION OF MICROPARTICLES IN COATINGS PRODUCED BY PLASMA ELECTROLYTIC OXIDATION (PEO) OF LIGHTWEIGHT ALLOYS	Federica Ceriani
S3.13	AN ASSESSMENT OF THE DIFFUSIBLE HYDROGEN RELEASE AND ITS EFFECT ON EXPERIMENTAL MEASURES	Alessandro Carrozza
S3.14	EXPANDED GLASS FROM POST-CONSUMER WASTE RECYCLING	Luca Cozzarini
S3.15	SELECTIVE LASER SINTERING OF BIO-BASED PBAT POWDER	Giovanna Colucci
S3.16	METAL MATRIX COMPOSITE APPROACH TO IMPROVE THE PROCESSABILITY OF 6061 ALLOY BY LASER POWDER BED FUSION	Elisa Padovano
S3.17	CORROSION BEHAVIOR OF 316L STAINLESS STEEL MANUFACTURED BY LASER POWDER BED FUSION	Francesco Andreatta
S3.18	CHARACTERIZATION AND DESIGN OF PURE PHOTOPOLYMERS AND SUSPENSIONS FOR DIGITAL LIGHT PROCESSING (DLP)	Carlo Antonini
S3.19	BI-LAYER YSZ TBCS: THE INFLUENCE OF MICROSTRUCTURE AND POWDER CHEMISTRY ON THE PERFORMANCES OF A GAS TURBINE COATING	Simone Bursich
S3.20	ASSESSMENT OF THE CORROSION BEHAVIOR OF AN HIGH-STRENGTH Al-Cu ALLOY PROCESSED VIA ADDITIVE MANUFACTURING	Sergio Lorenzi
S3.21	MICROSTRUCTURE INVESTIGATION OF A NI-BASED COMPOSITE FABRICATED BY LASER POWDER BED FUSION	Giulio Marchese
S3.22	METAL HYDRIDE-POLYMER COMPOSITES FOR HYDROGEN STORAGE	Marcello Baricco
S3.23	WATER OIL EMULSION FLOCCULATION THROUGH CHITOSAN DE-SOLUBILIZATION DRIVEN BY PH VARIATION	Marco Friuli
S3.24	ENVIRONMENTAL BARRIER COATINGS: NEW SOLUTIONS FOR PROTECTION OF CERAMIC MATRIX COMPOSITE COMPONENTS IN AIRCRAFT ENGINES	Laura Paglia
S3.25	INTERLAMINAR PROPERTIES AND ABLATION RESISTANCE OF CARBON FIBER COMPOSITES DERIVED FROM HIGH CHAR YIELD MATRICES	Maurizio Natali
S3.26	NOVEL SYNTHESIS AND PROCESSING BY DIGITAL LIGHT PROCESSING OF CeO_2-STABILIZED ZIRCONIA COMPOSITES: INFLUENCE OF SECOND PHASES ON PRINTING PARAMETERS AND MICROSTRUCTURAL MECHANICAL PROPERTIES	Elisa Fiume

S3.1 TITLE -PROCESS DEVELOPMENT OF CuCrZr ALLOY VIA ELECTON BEAM POWDER BED FUSION

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Among the copper-chromium based alloys, the precipitation hardened CuCrZr alloy shows excellent functional properties (e.g., electrical, and thermal conductivity) associate with moderate to high strength. Microstructural design plays a crucial role to obtain the right combination of abovementioned properties. Unfortunately, the traditional processing techniques such as forging and casting does not offer a good control over microstructural design. On the other hand, current advances in additive manufacturing (AM), make possible to have a better control of microstructural evolution as they provides high degree of freedom in design. The Powder-Bed-Fusion (PBF) techniques involve infact rapid heating and cooling rates which allow to obtain huge microstructural refinements, thereby improving the mechanical properties without any significant loss in the electrical conductivity. Compared to others Laser Powder Bed Fusion (L-PBF), the EB-PBF (Electron Beam Powder Bed Fusion) AM technology offers many advantages in the processing of copper alloys: it avoids difficulties associated to the high thermal conductivity and reflectivity of copper-based materials and, not least important, EB-PBF is performed in high vacuum environment to prevent oxidation. In this work, the process parameters optimization of the CuCrZr alloy via EB-PBF has been performed adopting a trial-and-error approach. The proper processing window for the achievement of dense and free of defects parts has been identified; the effect of process parameters on microstructure was also investigated. Moreover, a deep mechanical characterization and electrical conductivity tests have been performed on samples fabricated with optimized process parameters.

S3.2 A COMPARATIVE STUDY OF PLA/GRAPHENE NANOPLATELETS MEMBRANES PREPARED BY ELECTROSPINNING OR SOLUTION BLOW SPINNING

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Electrospinning (ES) is one the most frequently reported technique to prepare nanofibers mats. It has been recognized as promising technique to prepare nanofibres membranes. ES easily allows to produce uniform and aligned fibers with homogeneous diameters that can form membranes with high and interconnective porosity. On the other hand, ES displays several disadvantages that have prevented industrial scalability such as a high production time, high energy consumption, and operator safety hazards. Solution blow spinning (SBS) is particularly interesting because of the possibility of producing nanofibrous membranes in short times and without a high energy consumption since it uses only a pressure air flow to promote the production of nanofibers. Therefore, a fast processing time and overall low costs could make the nanofibrous membranes obtained by SBS competitive and industrially scalable. It could be an interesting challenge to evaluate the existing difference, to form the production to the application, of the same drug delivery systems produced for ES or SBS. In this work, a comparative study was performed on nanofibrous PLA-based systems for a controlled drug delivery application prepared with two different process techniques: ES and SBS. CRV was added to the polymeric solution as a liquid model molecule and, separately, CHX was added as a solid model molecule. In some cases, GNP were also added in order to study their effect on the release rate.

The PLA-based membranes were prepared by adopting two different kinds of processing, namely, electrospinning (ES) and solution blow spinning (SBS). Figure 1 provides a pictorial description of the two different routes adopted for producing different membranes.

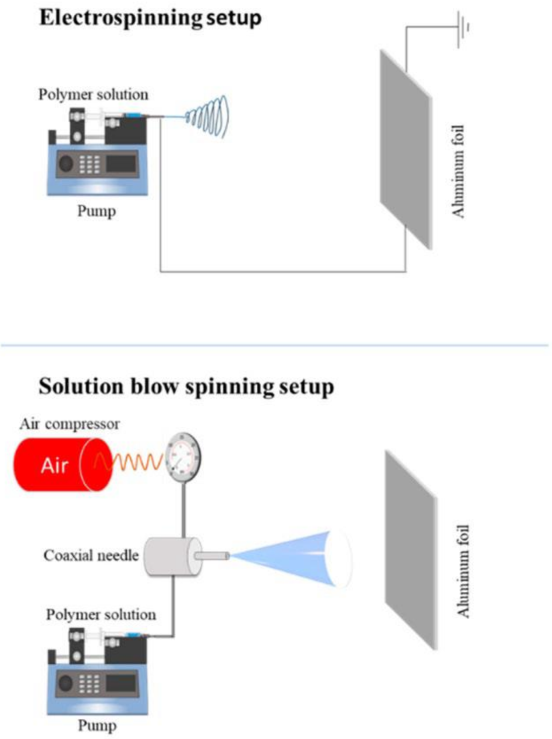

Figure 1. electrospinning and solution blow spinning setup.

S3.3 Ti6Al4V MANUFACTURED BY COUPLING FUSED DEPOSITION MODELING (FDM) AND SINTERING: MECHANICAL PROPERTIES OPTIMIZATION

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Additive manufacturing is an on the rise production technique which ensures many advantages compared to traditional manufacturing processes such as injection molding. It enables a higher design freedom allowing to produce complex structures, waste minimization, fast prototyping and promotes mass customization. If 3D-printed polymeric components proved to be cost-effective especially for low-medium production volumes thanks to a reasonable cost of the apparatus that reduces the initial costs of investment, this is not true for 3D-printed metal components which require complex and expensive equipment.

In this framework, the present work aims to overcome this drawback by coupling a common 3D-printing technique used for polymers, i.e. Fused Deposition Modeling (FDM), with well-established debinding-sintering processes to produce cost-effective dense metal parts starting from a polymeric filament highly-filled with metal powders. In particular, the work focused on a Ti6Al4V alloy which is largely used in the biomedical sector where product customization can significantly improve patient life quality. In this perspective, a lower cost of the biomedical device could increase buyers' audience allowing more people to take advantage of component customization.

To make this 2-step process effective, it is fundamental to perform an optimization of the printing parameters to achieve the highest possible mechanical properties. To reach this aim the Design of Experiment technique and the surface response analysis were used to optimize Ti6Al4V tensile and flexural properties by applying a 2-level full factorial design to three fundamental printing parameters: layer thickness (200-300 µm), nozzle temperature (140-160 °C) and printing speed (15-25 mm/s). The mechanical characterization was supported by a morphological one, i.e., surface fracture and grain size analysis and microtomography, to study the correlation between printing parameters, mechanical properties and microstructure.

S3.4 PURE POLY(PENTAMETHYLENE 2,5-FURANOATE) AND POLY(BUTYLENE 2,5-FURANOATE) ELECTROSPUN MATS: CHARACTERIZATION AND APPLICATIONS

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A great interest is turning to poly(alkylene 2,5-furanoate)s (PAFs), biobased polyesters synthesized starting from 2,5-furandicarboxylic acid (FDCA) and glycols with a variable alkyl chain length. Sustainable and bioderived furanoate polyesters are emerging as promising substitutes of petrochemical-derived polyesters. Recent studies on PAFs showed that they have very good thermal stability as well as better thermo-mechanical and gas barrier properties than their oil-based terephthalic counterparts. Among PAFs, poly(butylene 2,5-furandicarboxylate) (PBF) and poly(pentamethylene furanoate) (PPeF) have been considered very promising materials for different applications.

PBF and PPeF have a very similar repeating unit that differs in just one methyl group, but they have remarkably different physical and mechanical properties as films produced by compression molding. In the literature, any information about the development of electrospun mats based on PBF or PPeF is reported. For this reason, the presented work aims to report a detailed study on the feasibility to produce nanofibrous mats of PBF and PPeF by electrospinning and an accurate characterization of the thermal, physical, mechanical and biological properties. In particular, (i) the solubility of the polymers in different solvent mixtures, (ii) the viscosity and concentration of the spinning dopes, (iii) the spinning rate, (iv) the applied voltage and (v) the glass transition temperature were selected as the main parameters for the production of PPeF and PBF electrospun mats at controlled conditions of humidity and temperature. The optimized PPeF and PBF were also mechanically characterized by tensile tests and the potential application of PPeF and PBF electrospun mats as biomedical device was also evaluated: the optimized PPeF and PBF mats were treated under vacuum for the removal of residual solvents (Figure 1) in order to proceed with a cytotoxic test through LDH assay, cell adhesion of human fibroblasts (Figure 2) and *release tests of dexamethasone to investigate the suitability as an implanted device or a dermal patch.*

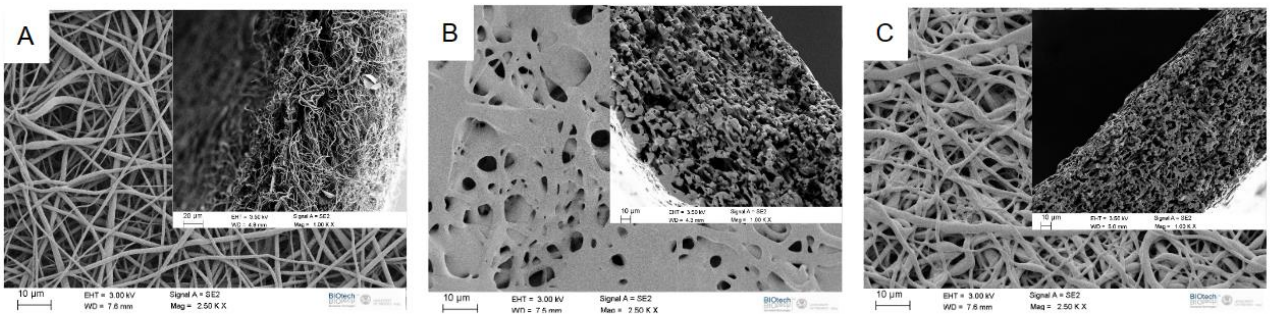

Figure 14. FESEM images of (A) PBF mats, (B) PPeF mats obtained by the electrospinning of the PPeF solution in HFIP and (C) PPeF mats derived from PPeF solution in DMF/CHCl₃ after removal of residual solvents.

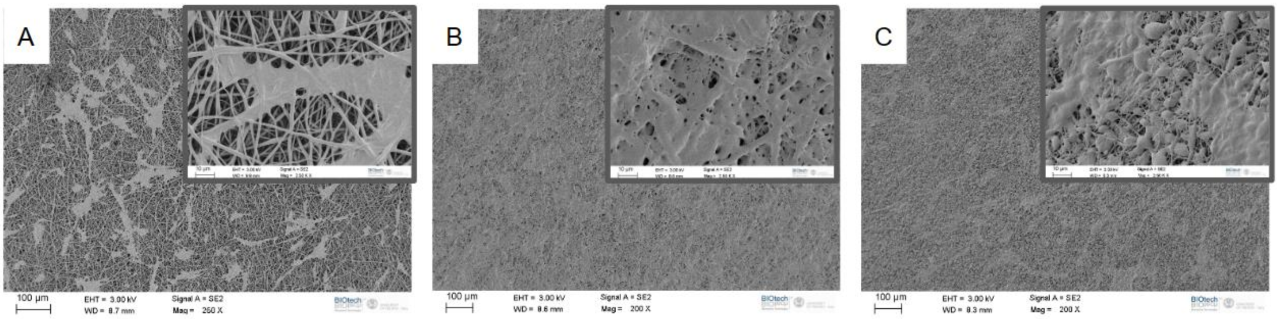

Figure 2. FESEM images of fibroblast MRC5 adhesion on (A) PBF mats, (B) PPeF mats obtained by the electrospinning of the PPeF solution in HFIP and (C) PPeF mats derived from PPeF solution in DMF/CHCl₃ after 3 days of culture.

S3.5 INKJET PRINTED TI$_3$C$_2$ ELECTRODES FOR ANODE-FREE ZINC-ION BATTERY

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The quick spreading of wearable electronics technologies and IoT (Internet of Things) devices urgently need the development of flexible and miniaturized energy storage devices [1]. In this regards, Li-ion batteries (LIBs) always poses safety concerns for the use of hazardous electrolyte, in particular when wearables are placed in close contact to human body. Zinc-ion batteries (ZIBs) are considered as a promising alternative to LIBs because of the employment of more friendly electrolytes and low cost and high theoretical capacity of the Zn metal anode [2]. However, the use of bulky Zn metal sheet is not feasible for wearable energy storage devices. The anode-free battery configuration could solve the issue and, in this regard, Ti3C2Tx MXenes have been demonstrated as promising "zincophilic" substrate for smooth and efficient zinc plating and stripping process [3,4].
In this work, we investigate inkjet-printed Ti$_3$C$_2$ MXene thin coatings as an ideal substrate for anode-free ZIBs. The zinc metal nucleation phenomena on MXene 2D sheets were investigated by electrochemical potentiodynamic polarization techniques as well as galvanostatic ones, comparing them to a zinc metallic foil. Ex-situ electron scanning microscopy (SEM) and in-situ morphological investigation allowed to reveal the surface modification of the inkjet printed MXene electrodes, defining the optimal electrochemical parameters and electrolyte composition for a stable and uniform Zn plating on the Ti$_3$C$_2$ electrode, paving the way to the development of all inkjet printed anode-free ZIBs.

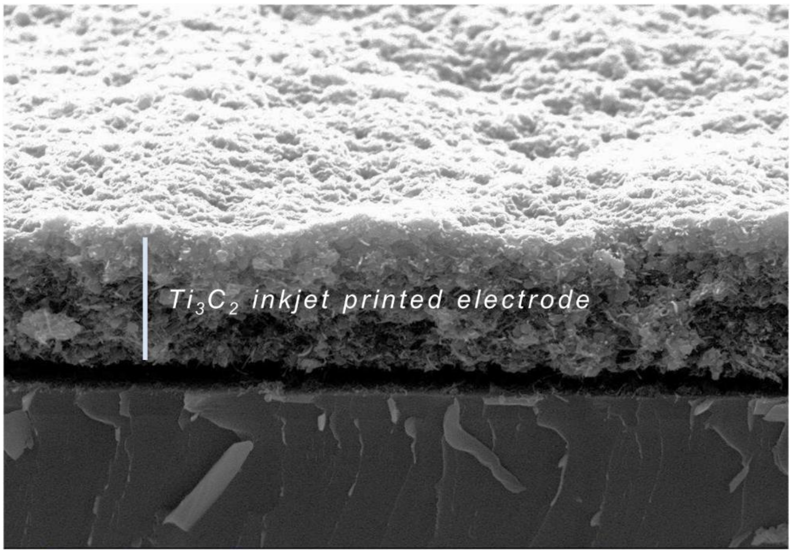

Figure 1. SEM image of the cross-section of the inkjet printed Ti$_3$C$_2$ thin film as zincophilic electrode for anode-free ZIBs.

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S3.6 TAILORING MORPHOLOGY AND MECHANICAL PROPERTIES OF PLA/PBSA BLENDS OPTIMIZING THE EXTRUSION PROCESSING PARAMETERS WITH LUDOVIC® SIMULATION SOFTWARE

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To think in a sustainable way, the use of bio-based plastics for tailored applications would be the solution to follow, especially in relation to the need of emancipation from oil resources. Nevertheless, the full exploitation of biopolymers is strictly connected to the final properties achieved and the capability to up-scale the production processes. In this context, above all for blends systems (in which it is necessary to capitalise on the advantages of the polymers involved) the evaluation of the processing behavior and the optimization of the parameters is of paramount importance. At industrial scale, polymer blending is carried out in twin-screw extruders; even if it is well known the correlation processing/morphology/properties and that the final morphology of the blends depends on the miscibility of the substances that constitute the system, the identification of the best processing conditions, such as the optimal screw speed, feeding rate and shear rate is still done by a trial and error approach.

For all these reasons, the idea of this work is to optimise the extrusion process parameters through a 1D software aided DoE to simplify and speed up the parameters selection process for a polylactic acid (PLA)/polybutylene succinate-co-adipate (PBSA) system blends with different PLA/PBSA ratios. Ludovic® software, indeed, has been developed to provide data on the evolution of many outputs (temperature, viscosity, shear rate) along the screw axis.

In the present work, the simulation results have been compared with real extrusions in a constrained range of process conditions; in addition morphological, rheological, mechanical and DMTA tests have been performed directly on the filaments produced avoiding a secondary process that could influence the final properties. The materials compounded, employing the best processing parameters, showed a satisfying accordance with the predictions, demonstrating the usefulness of the simulation tool.

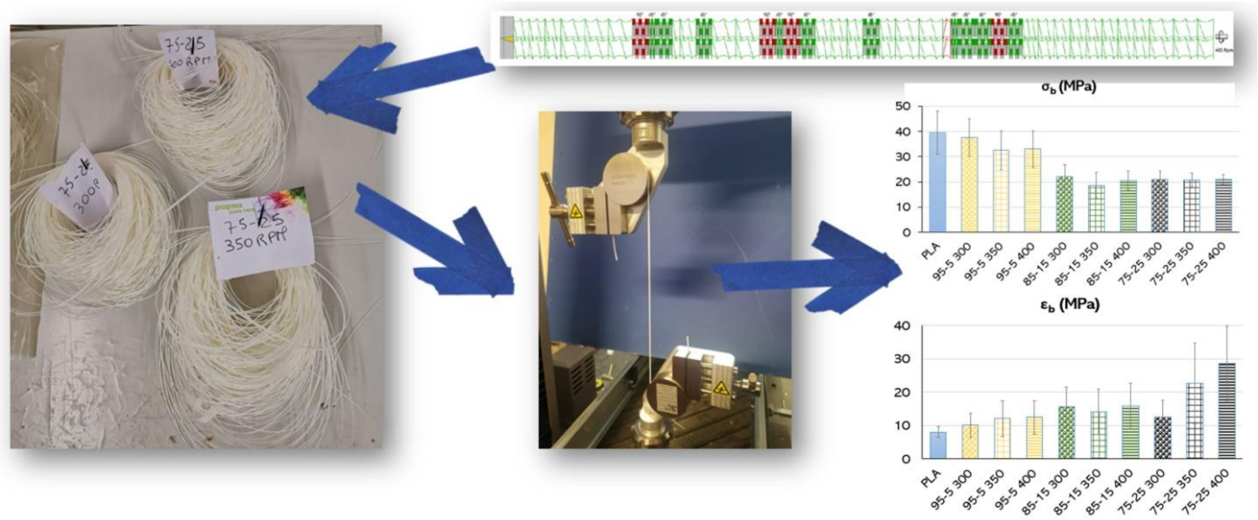

Figure 15. Example of work pathway: simulation->extrusion->testing.

S3.7 DECARBONIZING THE CONSTRUCTION INDUSTRY BY REDUCING THE CLINKER CONTENT IN CEMENT AND THE CEMENT CONTENT IN CONCRETE: THE PROMISING AND READY-TO USE SUSTAINABLE SOLUTION OF LC3 (LIMESTONE CALCINED CLAY CEMENT) CONCRETE

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The decarbonization of the construction industry will contribute to 8% reduction of the overall CO_2 emission by 2050. There is a clear consensus that reducing the clinker content in cement along with reducing the cement content in concrete is the main strategy to achieve this goal. Combining both premises, this study was designed to further reduce the carbon footprint of the most promising and ready-to-use sustainable binder, i.e., Limestone Calcined Clay Cement (LC^3), and of LC^3 structural concrete. Fifteen sustainable concrete mixes were designed to reduce the LC3 binder content below the amount prescribed by the construction standards (300 kg/m3) up to 250 kg/m3 while replacing up to 75% of the clinker content in cement.

The fresh, mechanical and durability properties of the designed sustainable concrete mixes were tested and compared to those of the correspondent Ordinary Portland cement (OPC) concrete. The results showed that a lower binder content, given proper workability maintained, does not involve a loss in performance. On the contrary, LC^3 concrete mixes with a 40-75% reduction in clinker content showed (i) acceptable slump using different levels of PCE-based SP, (ii) equivalent 7- and 28-days compressive strength and elastic modulus, (iii) incredibly lower chloride permeability in comparison with the control OPC concrete. These performance indicators were then integrated into the functional unit of a cradle-togate life cycle assessment to compare the environmental impact of each LC^3 blend and select the most sustainable in term of lowest Global Warming Potential (GWP).

S3.8 HIBRID POLYMERS MEMBRANES FOR SEPARATION OF HYDROGEN/METHANE MIXTURES

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In the upcoming shift to a hydrogen economy, membrane technologies and material development appear essential for the separation of hydrogen from natural gas. It might be less expensive to transport hydrogen through the current natural gas infrastructure than through new pipes. Currently, a lot of research is being done on the creation of novel structured materials for gas separation applications, including the blending of different additives in polymeric matrix. The mechanism for gas transport in those membranes has been clarified after numerous gas pairs have been studied. To support the shift to more sustainable energy sources, the selective separation of high purity hydrogen from hydrogen/methane mixtures is currently a major challenge and requires significant improvement. Fluoro-based polymers, like PVDF-HFP (Poly(vinylidene fluoride-co-hexafluoropropylene) and NafionTM, are among the most widely used membrane materials in this situation because of their remarkable properties, even though further optimization is required.

In this study, thick graphite surfaces were coated with thin films of hybrid polymer-based membranes. To separate hydrogen/methane gas mixtures, various weight ratios of PVDF-HFP and NafionTM polymers supported over 200 µm thick graphite foils were tested. To study the mechanical behaviour of membranes, small punch tests that replicated the testing environment were conducted. Finally, at room temperature and close to atmospheric pressure, the permeability and gas separation activity of hydrogen/methane over membranes were studied. In the developed membrane with the best performance, a 32.6% (v%) H2 enrichment was observed. The values of experimental and theoretical selectivity were also in good agreement.

S3.9 CATHODIC PROTECTION MONITORING: DESIGN OF A NEW SOLID INTERNAL BACKFILL FOR POTENTIAL PROBES

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According to ISO 15589-1, the assessment of cathodic protection efficiency is based on IR-free potential measurement, i.e., without the ohmic drop contribution. For buried structures, as pipelines or tanks, the ohmic drop is typically not negligible, due to the high soil resistivity and the presence of flowing direct currents, such as the cathodic protection current or the current dispersed by the public transportation systems.

The use of potential probes is continuously increasing, thanks to their accuracy in the potential measurements and the possibility to assess cathodic protection efficiency even in the presence of stray currents. A potential probe consists of a fixed Cu/$CuSO_4$ *reference electrode and a metallic coupon close to it (Figure 1a). The coupon is permanently connected to the structure in cathodic protection and simulates a coating defect.*

One drawback of the use of the potential probe is related to the leakage of the copper sulphate solution, contained in the plastic case of the probe, through the porous septum. At the same time, contaminants, as chloride ions, could permeate through the septum into the probe, polluting the internal electrolyte. Hence, their lifetime is limited only to a few years. Recently, internal copper sulphate gels have been proposed instead of the liquid solution even if the duration increase is limited.

In this work, a new internal solid electrolyte (backfill) for potential probes is proposed. In the first phase, some mixtures consisting of copper sulphate and a solid binder in different proportion with water were studied. Then, conductivity and mass loss measurements were carried out to investigate the stability of the backfills to the exposure in dry or wet soils. The most promising backfills were selected for the preparation of a Cu/$CuSO_4$ *reference electrodes (Figure 1b), which potential accuracy was measured by long-term tests trials. Results show the higher stability of the backfill prepared with gypsum as binder, both as lifetime increase and potential measurements.*

a) b)

Figure 1. a) Schematic view of a potential probe; b) portable CSE reference electrodes with solid electrolyte.

S3.10 DEVELOPMENT OF FESI STEEL WITH INCREASED SI CONTENT BY LASER POWDER BAD FUSION TECHNOLOGY FOR FERROMAGNETIC CORES APPLICATION: MICROSTRUCTURE AND PROPERTIES

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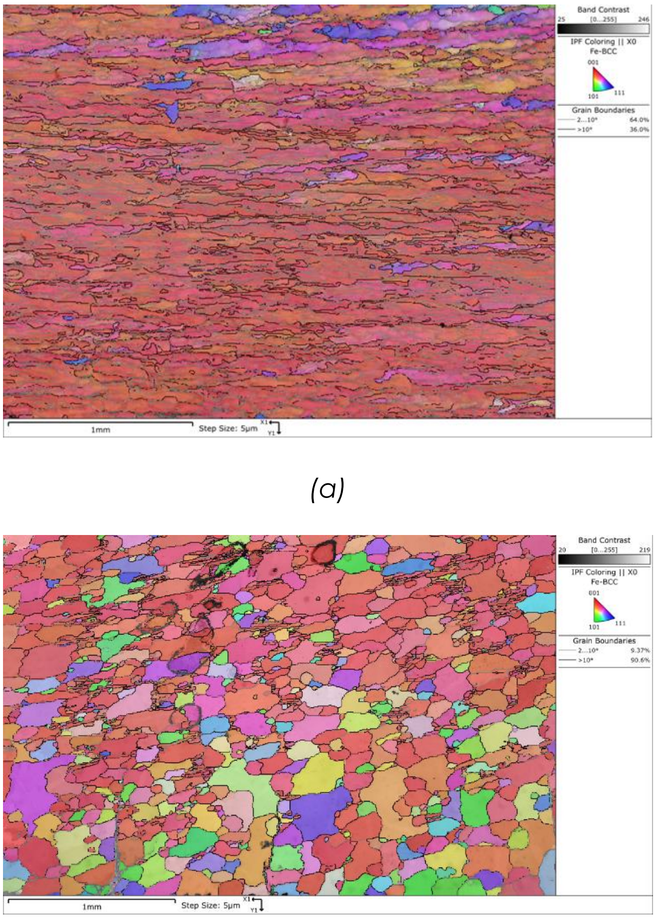
The conventional production of ferromagnetic cores involves the production of thin sheets in FeSi steel which, given the inherent brittleness and poor workability of the steel, limits the Si content to a maximum of 4.0 wt%. Additive manufacturing technologies represent a valid alternative for the production of FeSi steels components with optimized Si content. In this work, Laser Powder Ben Fusion technology was used to produce samples and components of two grades of FeSi steels, with standard (3.0wt% - FeSi3) and high (6.5wt% - FeSi6.5) Si content. The study is aimed to optimize the process parameters thus obtaining components suitable for magnetic applications. Moreover, the effect of post-production annealing heat treatment, in both microstructural and texture evolution, was investigated. The experimentation included microstructural analysis by optical and scanning electron microscope, EBSD, X-ray diffraction analysis and electro-magnetic measurements. The examined FeSi steels present some relevant differences:

- all grains of FeSi3 are columnar whereas for FeSi6.5 the microstructure is a mix of columnar and equiaxed grains;
- all columnar grains have the [100] orientation parallel to build direction whereas equiaxed ones exhibit various orientations;
- after annealing the grain size and shape of FeSi3 do not remarkably change also at the highest temperature of 1150 °C (Fig.1(a)) whereas relevant variations take place in FeSi6.5 in which, at 1150 °C, the equiaxed grains completely cover the sample surface (Fig1(b)).

Regarding the magnetic behavior, electromagnetic measurements were carried out on samples with optimized geometry and subjected to annealing at 1150°C. Results showed that FeSi6.5 samples exhibits less eddy current effects in comparison to FeSi3 resulting in higher magnetization capability and reduced power losses over than 50%. Improved post-production processes may lead to promising technology for making components for magnetic applications with high Si content FeSi steels.



(a)

(b)

Figure 1. EBSD images of (a) FeSi3 steel following annealing heat treatment at 1150 °C for 1 hour (columnar microstructure) and (b) FeSi6.5 steel following annealing heat treatment at 1150 °C for 1 hour (equiaxed grain microstructure)

S3.11 RATIONAL DESIGN OF NEW SUSTAINABLE INTEGRATED SYSTEM FOR INDOOR CULTURES

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A new integrated system for house and vertical gardens cultures is proposed in this study by synergic application of innovative fertilizer and LED lighting. Rational Design, based on Design of Experiments methods, has been used to develop new NPK fertilizers constituted by a core of light weight aggregates derived by agri-food and post-consumer waste enriched with potassium and phosphorus, coated by organic materials derived by waste valorization by using Black soldier flies. NPK fertilizers were characterized by physical and chemical properties, considering their nutrients' release capability for lettuce growth (fig1). Same rational approach has been used to derive the LEDs lighting conditions promoting plant growth and obtaining robust models correlating fertilizer quantity, compositions, LEDs light with finals plant growth parameters. A controlled environment in terms of temperature and humidity (climatic chamber) was employed, whereas differents LEDs were considered as variables in the experimental plant, as well as the quantity of NPK fertilizer in the soil. The experimental achievements of this study identify an approach to improve the environmental sustainability of indoor horticulture, especially when realized in the urban context. At the same time, well-tailored light recipes for lettuce growth were calculated, avoiding loss of beneficial and critical resource such as water and nutrients (Nitrogen, Potassium, and Phosphorous). The involvement of wastes into the process is part of their valorization in the circular economy approach and perfectly in agreement with the European guidelines.

Figure1. Scheme of fertilizer design, realization, and technological indoor cultivation box.

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S3.12 STUDY OF THE INCORPORATION OF MICROPARTICLES IN COATINGS PRODUCED BY PLASMA ELECTROLYTIC OXIDATION (PEO) OF LIGHTWEIGHT ALLOYS

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Microparticles could be used as additive in the electrolytic solution during plasma electrolytic oxidation (PEO) of valve metals (mainly Al, Ti and Mg) to improve the thickness and the compactness of the produced oxide coatings, enhancing their corrosion resistance. Microparticles can fill the film porosity, whose formation is intrinsically related to the occurrence of discharges during the PEO process, reducing the defectiveness of the oxide structure. As a result, the corrosion resistance of the coatings is improved since the number and dimension of defects, which may facilitate the penetration of aggressive substances through the oxide, are significantly reduced. Moreover, the larger the particles retention within the oxide, the higher its thickness; this leads to a further growth of the coating resistance to corrosion.

The present research is focused on the comparison of the effects of particles incorporation within PEO coatings on lightweight alloys. Two types of particles, namely rutile and anatase (TiO_2) microparticles (diameter < 5 μm), are considered. The PEO treatment is performed working in alkaline solutions based on NaOH and KOH with the addition of Na_2SiO_3. Both DC and AC electrical regimes are applied. PEO coatings are analysed by scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) analyses. In addition, electrochemical impedance spectroscopy (EIS) in NaCl 3.5 wt% is carried out to evaluate the surface activity of the PEO samples. Particles uptake increases with the plasma duration over the electrode surface, promoting the formation of thick oxide coatings. The oxide structure is characterized by a uniform fine porosity when treated under AC conditions, while working in DC regime less numerous but larger pores are generated. Thus, AC coatings show a better barrier effect against the penetration of corrosive species, which results in higher impedance values and then in an improved corrosion resistance.

S3.13 AN ASSESSMENT OF THE DIFFUSIBLE HYDROGEN RELEASE AND ITS EFFECT ON EXPERIMENTAL MEASURES

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Green hydrogen is a promising low-impact energy carrier for reducing carbon emissions, but its safe transportation through existing pipelines still requires thorough materials compatibility investigations. Several experimental approaches are possible to determine the effect of hydrogen on alloys (e.g., slow strain rate, fracture mechanics test). All these methods study the impact of hydrogen on the mechanical properties. These effects are generally related to the concentration of total diffusible hydrogen inside the metal lattice, but this value (generally measured ex-situ) is difficult to obtain. Despite the possible experimental variability, uncertainties remain in the results because of the lack of control over the amount of hydrogen released by the samples after charging. The time gap between the end of charging and testing can also affect the detected hydrogen concentration.

This study focuses on addressing these issues by using an electrochemical approach (solubility tests) to assess the time effect on hydrogen concentration on X65 pipeline steel samples. These underwent cathodic polarization to introduce hydrogen into the specimens. Then, an anodic polarization was performed, while measuring the circulating current, that can be correlated to the diffusible hydrogen released from the material. This process was conducted considering different time gaps between the two charging operations, allowing to determine the hydrogen release naturally occurring in air.

The results of this study show that the diffusible hydrogen concentration decreases at increasing time gaps, as illustrated in Figure 1a. These findings emphasize that even after very short period between the hydrogen charging and testing phases, significant diffusible hydrogen release happens (Figure 1b).

These finding emphasize that the time window between charging and testing is of primary importance when investigating the compatibility of hydrogen with metal alloys, even if this aspect is often neglected.

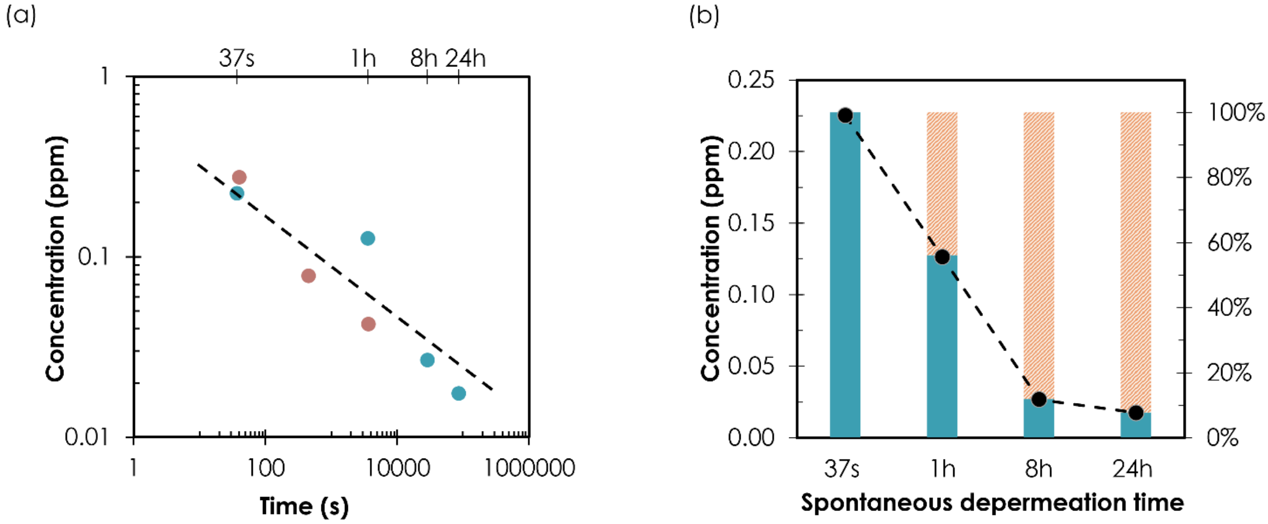

Figure 1 – Evolution of the absolute (a) and relative (b) concentrations of hydrogen in X65 pipeline steels after different time gaps between hydrogen charging and testing.

S3.14 EXPANDED GLASS FROM POST-CONSUMER WASTE RECYCLING

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Expanded glass samples are obtained from the recycling of post-consumer waste. Their mechanical, thermal and acoustic properties are characterized as a function of process parameters. Preliminary results show that it is possible to produce glass foams with thermal and acoustic insulation properties from a blend composed of 98% post-consumer glass waste and 2% expanding agent. This latter is a mixture of carbon and transition metal oxides, extracted from different sources of post-consumer waste. The glass foams are produced after heating the blend, in the form of fine powder, in a ventilated furnace at temperatures between 800 and 900°C for time ranging from 30 and 60 minutes. The glass foam samples have densities in the range of 150-250 kg/m^3, porosity of 80-90%, thermal conductivity values of 80-100 mW m^{-1} K^{-1}, noise reducing factors of 0.15-0.40 and compressive strengths of 1.0-2.5 MPa. Although the insulating performances are not as outstanding as those of polymer foams or mineral wool, these materials can emerge as competitive candidates for applications requiring moderate thermal and acoustic insulation properties in combination with low weight, mechanical strength, non-flammability and high temperature load bearing-capacity. Moreover, the use of 100% recycled raw materials limits the energy and the resource required for their production, if compared to those needed for the extraction, transportation, and processing of primary raw materials, making these foams attractive also in terms of environmental impact.

S3.15 SELECTIVE LASER SINTERING OF BIO-BASED PBAT POWDER

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Bio-based polymers are environmentally friendly materials that can be used as sustainable alternatives to the traditional fossil-based polymers.
Among these, poly(butylene-adipate-co-terephthalate) (PBAT) is one of the most important due to its biodegradability, good thermal and mechanical properties, whose applications are wide-ranging and significantly growing, from agriculture to food packaging, from textile to biomedical field.
The aim of the present work is to use for the first time PBAT as starting polymer for Additive Manufacturing, and specifically for Selective Laser Sintering (SLS). In recent years, SLS has become the most popular AM process due to its high accuracy, productive efficiency, and surface quality. However, there are very few commercially available polymeric materials suitable for this technique.
In fact, it is very difficult to produce polymeric powders with specific particle size and shape, good flowability and processability, useful for SLS applications.
The present study started with the production of PBAT powder, through an emulsion solvent evaporation method, with the purpose to investigate the 3D-printability of the bio-based polymer by SLS. The PBAT powder was fully characterized by morphological and thermal point of view. Finally, 3D printed components with increasing complexity were successfully realized, from monolayer and multilayer prints with simple geometry, to more complex-shaped structures, such as six-pointed stars or a hexagon with an intricate internal profile and holes.
This can represent a valid approach for producing novel biodegradable 3D printed objects using bio-based polymers that can replace the conventional polymers for many different added-value applications.

S3.16 METAL MATRIX COMPOSITE APPROACH TO IMPROVE THE PROCESSABILITY OF 6061 ALLOY BY LASER POWDER BED FUSION

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Laser Powder Bed Fusion (LPBF) is one of the most widespread additive manufacturing technologies due to its high process flexibility. Thanks to the layer-by-layer strategy, LPBF offers in fact the possibility to fabricate metallic components with a high degree of geometrical freedom without the use of moulds, overcoming the main barrier of traditional manufacturing processes. The advantages of this innovative process have attracted an increasing interest in processing high performant materials for which the optimization of customized parts can be exploited in a wide range of applications. In this context, Al alloys are very attractive for manufacturing parts showing an excellent strength-to-weight ratio in addition to a relatively low cost. Currently, cast alloys with near eutectic composition such as AlSi10Mg, AlSi12 and A357 are the most studied thanks their solidification properties which make them suitable to be processed for LPBF. However, there is an increasing interest in the development of other Al-based systems such as high-strength aluminium alloys (e.g., 2000, 6000 and 7000 series) characterized by good workability and high specific strength. Unfortunately, their processability for LPBF is very challenging due to their large solidification range, poor melt flowability and high cracking susceptibility.

Among the different strategies that can be used to improve the processability of these materials, this work is focused on the study of the process window for the LPBF manufacturing of AA6061 parts using a metal matrix composite approach.

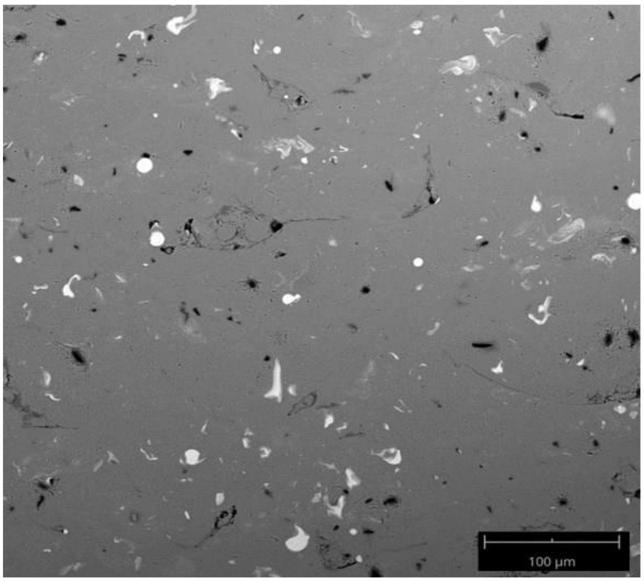

Figure 1. SEM micrograph of PLBF processed sample.

The evolution of powdered system constituted by a 6061 matrix reinforced with Ti and B4C is investigated as a function of process parameters variation in order to obtain dense composite material. The in-situ reaction mechanism is discussed in addition to the relationship between LPBF parameters, the microstructure and defects formation with a focus on cracks development.

S3.17 CORROSION BEHAVIOR OF 316L STAINLESS STEEL MANUFACTURED BY LASER POWDER BED FUSION

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This work investigates the protective properties of oxide films on AISI 316L stainless steel produced by laser powder bed fusion (L-PBF) and those of its wrought counterpart. The behavior of nearly defect-free oxide layers obtained on polished surfaces is compared to that of oxide layers present on the as printed surface.

The L-PBF specimens investigated in this work were manufactured with a Concept Laser M2 Cusing machine, equipped with a 400 W single-mode CW ytterbium-doped fiber laser with emission wavelength of 1070 nm. Process parameters were optimized in order to obtain samples with very low porosity. The L-PBF stainless steel specimens were tested in the as produced condition without stress relief or recrystallization heat treatments.

A detailed characterization of the microstructure and electrochemical behavior of the L-PBF and wrought 316L stainless steel was carried out by FE-SEM and AFM. This work combines different electrochemical methods (open circuit potential measurements, potentiodynamic polarization and electrochemical impedance spectroscopy) to evaluate the different oxide layers on L-PBF and wrought 316L specimens. In addition, a localized electrochemical approach combining the micro-cell technique and the scanning kelvin probe force microscope (SKPFM) were employed to highlight the corrosion susceptibility of the as printed surface.

S3.18 CHARACTERIZATION AND DESIGN OF PURE PHOTOPOLYMERS AND SUSPENSIONS FOR DIGITAL LIGHT PROCESSING (DLP)

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Several additive manufacturing (AM) technologies are nowadays available for layer-by-layer fabrication of complex parts, allowing reduction of costs and waste compared to conventional manufacturing technologies. Among all AM technologies, digital light processing (DLP), an advanced version of stereolithography (SLA), is emerging for manufacturing 3D objects using liquid photopolymers and suspensions (e.g. including ceramic and metal particles) as feedstock. With the rapid growth of the technology in the last few years, DLP requires a rational design framework for printing process optimization based on the specific material and printer characteristics. In this study, a systematic characterization of both pure photopolymers and suspensions is presented, discussing two radiation-independent characteristic parameters of photopolymers: the critical energy (Ec) and the penetration depth (Dp). The discussion also includes some of the basic theoretical aspects of light-matter interaction that can guide the manufacturing process development and optimization. The proposed methodology enables printing optimization for any material and printer combination, based on simple preliminary material characterization tests to define the printing space. The methodology can be generalized and applied to other vat polymerization technologies.

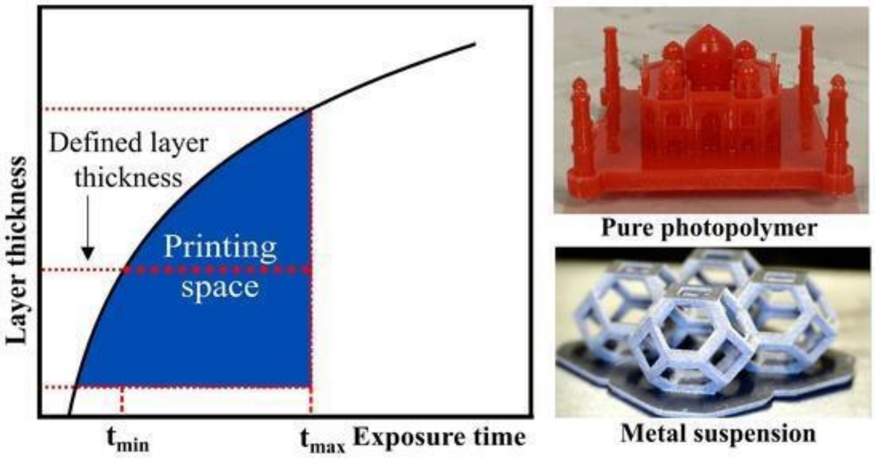

Figure 1. Optimal printing space for 3D printing by digital light processing (DLP, left), with examples of complex 3D printed objects using a pure photopolymer (top right) and a metal suspension (bottom right).

S3.19 BI-LAYER YSZ TBCs: THE INFLUENCE OF MICROSTRUCTURE AND POWDER CHEMISTRY ON THE PERFORMANCES OF A GAS TURBINE COATING

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The adoption of Thermal Barrier Coating (TBC) systems with high-yttrium zirconia is constantly increasing in the Oil&Gas and aviation fields due to demanding turbine temperatures. High-yttrium compositions represent an economical and easy choice to obtain TBCs with better phase stability than "standard" 7-8YSZ at high temperatures and chemical resistance to molten silicate deposits ("CMAS").

The aim of this work was to study the performances of ceramic bi-layer coating systems deposited by Atmospheric Plasma Spraying (APS) using an innovative cascade plasma torch. The bilayer systems comprised an inner layer of standard 7-8YSZ and a top layer of ZrO_2*-20wt.%* Y_2O_3*.*

The bottom and top layers were obtained using agglomerated powders of the respective compositions, producing systems with porous and Dense-Vertically Cracked (DVC) microstructures for both layers. Hastelloy-X substrates with a HVOF-APS "Flash" NiCoCrAlY bond coat were used for all the coating tested.

The thermomechanical behavior and bond strength of the samples were analyzed through Thermal Cycling Fatigue (TCF) and tensile adhesion (ASTM C633) tests.

Chemical attack by molten CMAS (CaO-MgO-Al_2O_3*-*SiO_2*) deposit at high temperature was also performed. FEG-SEM, XRD and Raman Spectroscopy techniques were used to characterize the coating systems before and after TCF and CMAS-corrosion tests, elucidating their degradation mechanisms.*

S3.20 ASSESSMENT OF THE CORROSION BEHAVIOR OF AN HIGH-STRENGTH Al-Cu ALLOY PROCESSED VIA ADDITIVE MANUFACTURING

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The decarbonization of our society is one of the biggest challenges faced by scientists in this century. In this framework, creating a smart, efficient and sustainable mobility system is a key factor. For instance, the transportation sector accounts for approximately 25% of the total greenhouse gas emissions in Italy.

One of the possible strategies to reduce the total emissions generated by vehicles focuses on weight reduction. Therefore, the transportation industry is experiencing a growing interest towards high-strength and low-density materials, such as aluminum alloys. In particular, Al-Cu systems (2xxx series) are very promising due to the relatively high mechanical performances achievable.

Additive manufacturing (AM) technologies represent another interesting trend in the current industry, due to their potential for material waste reduction, high strength granted, and ability to generate complex designs, allowing the production of lightweight components.

For the above reasons, the application of AM technologies to process Al-Cu alloys is a very promising strategy in the sustainable mobility framework. However, these materials are usually characterized by a lower corrosion resistance, due to the higher nobility of Cu with respect to Al, thus possibly compromising the durability of these alloys. Hence, this work focuses on the comparative analysis of AM-processed 2139 and AlSi10Mg alloys in terms of corrosion behavior in a NaCl- and NA_2SO_4*-rich solution. The specimens were investigated via electrochemical impedance spectroscopy (EIS), potentiodynamic polarization, and susceptibility to intergranular corrosion tests. In general, the 2139 alloy provided a more active behavior in the potentiodynamic polarization curves (Figure 1a) and two well-defined time constants and lower impedance modulus and low frequencies in the EIS curves (Figure 1b). These results confirm the critical corrosion behavior of the 2139 alloy, even if an AM technology is deployed.*

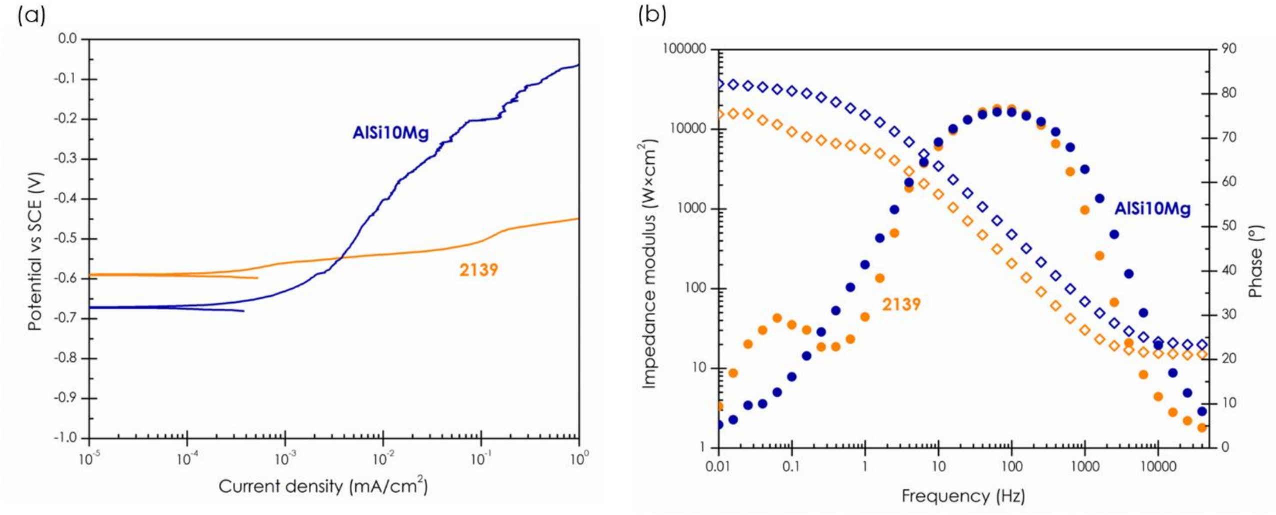

Figure 1 – Potentiodynamic polarization (a) and EIS (b) curves of the AM-processed 2139 and AlSi10MG alloys.

S3.21 MICROSTRUCTURE INVESTIGATION OF A NI-BASED COMPOSITE FABRICATED BY LASER POWDER BED FUSION

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Inconel 625 (IN625) is a Ni-based superalloy that combines good mechanical performance with elevated resistance in harsh environments at high temperatures. This alloy can be easily processed by the additive manufacturing laser powder bed fusion (LPBF) process, allowing the production of complex near-net shape components in a single step. Typically, IN625 alloy can be used for a broad range of applications in the aerospace, marine, and energy industries.

Nowadays, there is a growing interest in developing materials with superior mechanical performance for the industry. One strategy that allows the production of higher-performance materials is represented by adding ceramic particles into the Ni-based superalloy, thus creating Ni-based composites.

In the present study, a mixed powder of IN625 and TiC particles was processed by LPBF process. Initially, a process parameter optimization was performed to identify the most suitable parameters to obtain dense composite (IN625/TiC) samples. The investigation demonstrated that it is possible to obtain a densification level similar to the IN625 alloy (porosity less than 0.15 %). The microstructure of the as-built composites revealed the formation of columnar grains along the building direction, as observed for the IN625 alloy. For the composite, the TiC particles chiefly remained along the grain boundaries as well as the melt pool contours. Applying high-temperature heat treatments revealed higher microstructure stability of the composites with respect to the IN625 alloy. For instance, the solution-annealing at 1150 °C induced full recrystallization for the alloy while the composite still presented columnar grains along the building direction (Figure 1). In fact, the TiC particles hinder the recrystallization and grain growth. Finally, the mechanical performance of the composite was compared to the IN625 alloy. This study drives attention to the potential of the development of metal matrix composites by LPBF process.

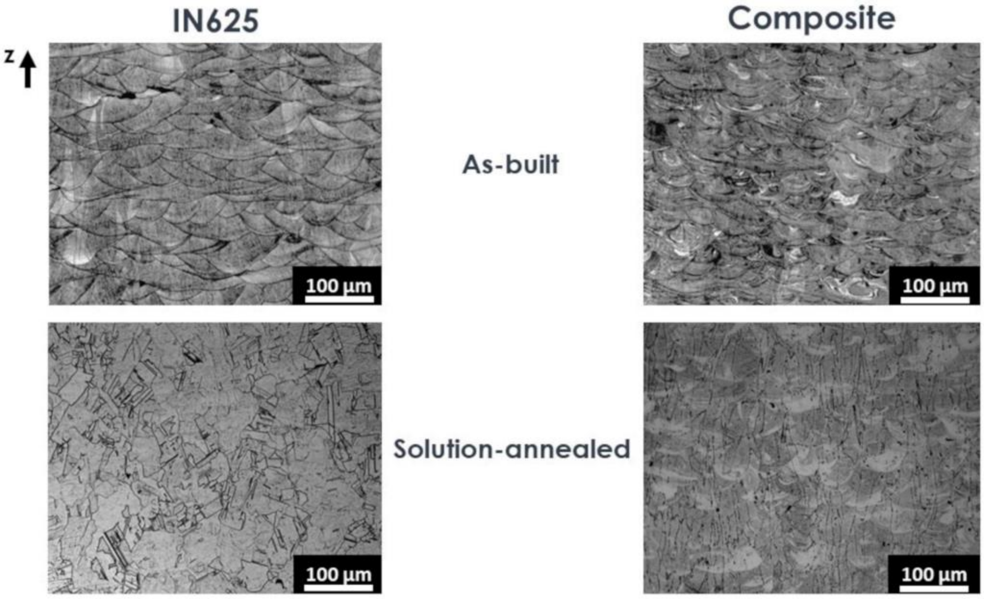

Figure 16. light optical images of the as-built and heat-treated IN625 and composite samples.

S3.22 METAL HYDRIDE-POLYMER COMPOSITES FOR HYDROGEN STORAGE

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Despite the advantages posed by hydrogen as an energy carrier, extremely low temperatures for the liquid form or high-pressure for the gas phase are necessary for its storage. Solid-state technologies, using metal hydrides (MH), allow to obtain high-volume hydrogen storage capacity and to improve the safety. For this purpose, MH is typically placed in a packed bed reactor as a loose powder. However, in the long-term applications, MH powders undergo a "breathing effect", where the several cycles of absorption and desorption of hydrogen cause the material to pulverize, causing pipe deformation at the bottom portion of the reactor and affecting the overall storage efficiency and thermal management of the system. An alternative solution is to compact the pressed powder into pellets, enhancing mechanical stability, improving thermal conductivity, and increasing mass transfer between hydrogen and the material.

The aim of this work is to synthesize a composite material of $LaNi_5$*-type MH with a polymeric binder. After suitable comparisons, PE polymer was selected. The synthesis was obtained by ball-milling the metal powder, followed by a mechanical press, obtaining suitable pellets, up to a 90:10 MH:PE ratio. Characterizations were performed with SEM, XRD, DSC, Raman and ATR. A volumetric Sievert apparatus was used to determine thermodynamic parameters through PCI curves and Van't Hoff plot. Finally, pellets were tested in a single pipe MH reactor for cycling studies.*

Ball milling induces a microstructural refinement of the metal powder. The formation of pellets does not change MH phase composition and microstructure. The hydrogen gravimetric density had an average value of 1.29 wt% for metal alone and 1.16 wt% for the composite. Moreover, pellets showed durability and remained intact after 20 cycles of hydrogen sorption, with a 3% residual weight of fine powder. These results show a promising material of PE-MH composites for stationary applications of hydrogen storage.

S3.23 WATER OIL EMULSION FLOCCULATION THROUGH CHITOSAN DE-SOLUBILIZATION DRIVEN BY pH VARIATION

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Water pollution is a major concern in our modern age. The contamination of water, as a valuable and often limited resource, affects both the environment and human health. Industrial processes such as food, cosmetic and pharmaceutical production also contribute to this problem. Vegetable oil production, for example, generates a stable oil/water emulsion containing 0.5-5% oil, which presents a difficult waste disposal issue. Conventional treatment methods based on the use of Aluminum salts generate hazardous waste, highlighting the need for green and biodegradable coagulant agents. In this study, the efficacy of commercial chitosan, a natural polysaccharide derived from chitin deacetylation, has been evaluated as a coagulation agent for vegetable oil emulsions. The effect of commercial chitosan was assessed in relation to different surfactants (anionic, cationic, and non-polar) and pH levels. The results demonstrate that chitosan is effective at concentrations as low as 300 ppm and that its flocculation mechanism relies on the de-solubilization of the polymer, which acts as a net to entrap the emulsion, rather than solely relying on electrostatic interactions with the particles. This study highlights the potential of chitosan as a natural and eco-friendly alternative to conventional coagulants for the remediation of oil-contaminated water.

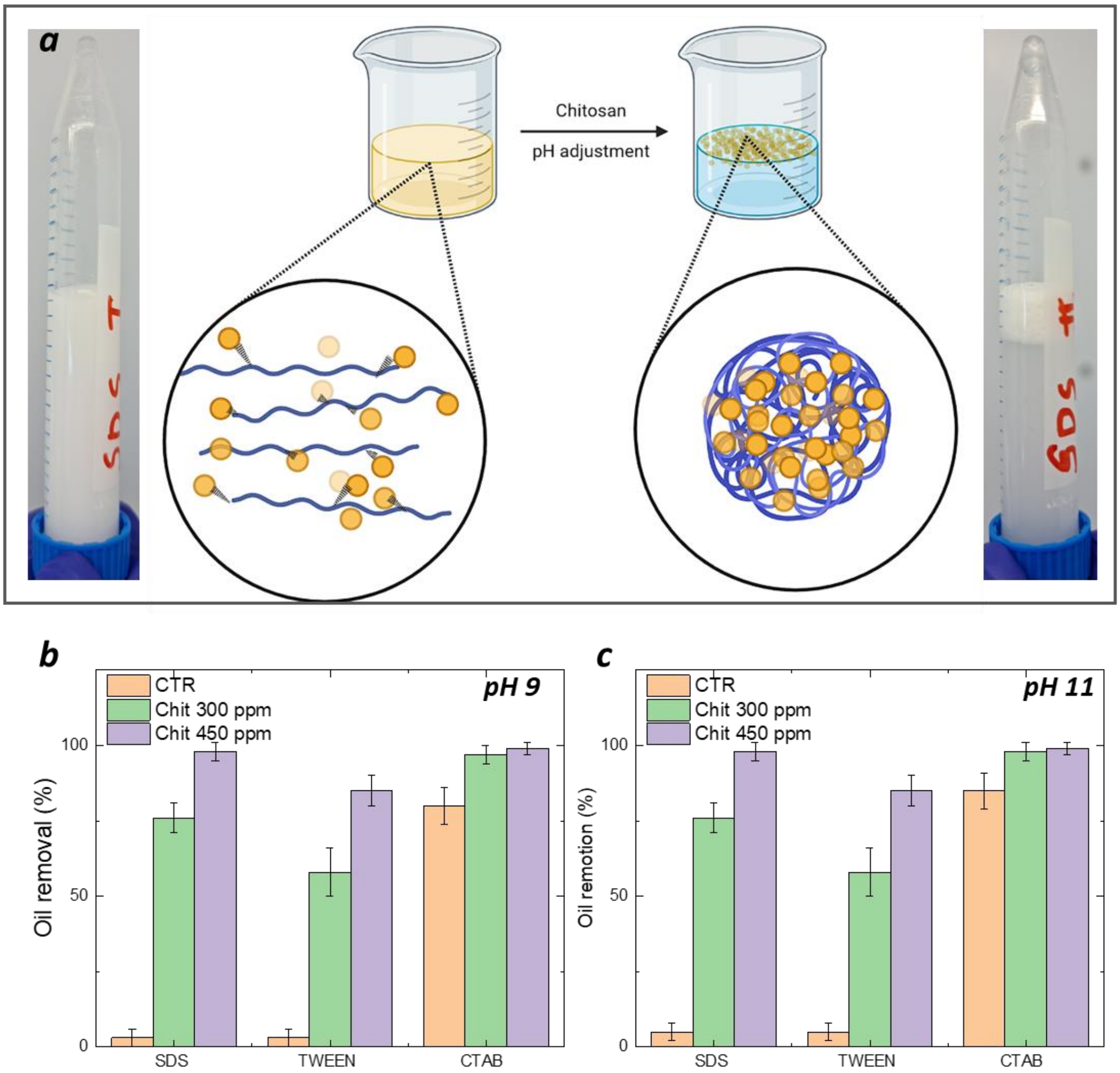

Figure 1. a) picture and schematic representation of the chitosan-driven flocculation on oil-water emulsion; b-c)Oil removal obtained with different concentrations of chitosan and at pH 9 and 11 respectively.

S3.24 ENVIRONMENTAL BARRIER COATINGS: NEW SOLUTIONS FOR PROTECTION OF CERAMIC MATRIX COMPOSITE COMPONENTS IN AIRCRAFT ENGINES

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Aircraft gas turbine engines efficiency can be improved by increasing the inlet turbine temperature of the combustion gasses, for this reason metallic superalloys protected by thermal barrier coatings need to be replaced with more performing materials. Ceramic Matrix Composites (CMC) are excellent candidates for the hot-sections of the gas turbine engines because of their low density (~1/3 of the metallic superalloys), high temperature capability, good mechanical strength and excellent thermal shock resistance. In particular SiC/SiC CMCs have been used since 2019 for the shrouds of the LEAP engine produced by General Electrics.

Despite their excellent features, SiC/SiC CMCs suffer severe surface recession phenomena in the high temperature water vapor-rich combustion gasses and a protective coating, called Environmental Barrier Coating (EBC), has to be designed.

Nowadays EBCs for SiC/SiC CMCs consist of multilayer systems: silicon is used as bond-coat and a top-coat of rare-earth (RE) silicate faces the combustion environment. In particular ytterbium silicates is chosen for its inherent phase stability up to 1850°C, its low recession rate in water vapor reach environment and a CTE close to SiC's one.

Other RE silicate can be taken into consideration for new EBC systems: in this work scandium disilicate and yttrium/ytterbium disilicate were deposited on SiC substrates with an Atmospheric Plasma Spray technique. After deposition they were heat treated at high temperature (1300 and 1400°C for 40 h in air) in order to avoid amorphous phases. Their microstructure was characterized by SEM observations and by studying crack density and porosity. EDS and XRD analysis were carried out for understanding their composition and the phase evolution during the thermal treatments. This characterization aims to understand the potentiality of these alternative RE-silicate as EBC systems and the influence of the APS deposition parameters on their final properties.

S3.25 INTERLAMINAR PROPERTIES AND ABLATION RESISTANCE OF CARBON FIBER COMPOSITES DERIVED FROM HIGH CHAR YIELD MATRICES

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Carbon/Phenolic Composites (CPCs) are used in the nozzle assembly of Solid Rocket Motors (SRMs) which are essential to preserve the independent access to space. Solid rocket propulsion is also at the base of the homeland security. In this work a new family of nanostructured, low cost, high char yield CPCs, manufactured with T300-like Polyacrylonitrile (PAN) based Carbon Fibers, have been developed. The interlaminar properties, ablation resistance and thermal diffusivity of the CPCs were studied. Starting from a commercial 0.1% formaldehyde phenolic matrix, through a modification carried out with the addition of 10 wt% epoxy resin, it was possible to increase the Inter Laminar Shear Stress (ILSS) of the composite of about 87.3% as compared to the counterpart made with the neat resol, leading to values in lines with the state of the art CPCs restricted under International Traffic in Arms Regulations (ITAR). Moreover, once exposed to an Oxy-Acetylene Torch (OAT) test, the values of the ILSS of the partly burnt CPC based on the hybrid epoxy-phenolic system (P10E) resulted to be about 137% higher than the corresponding value of the composite made with the neat resol (figure 1). Furthermore, among the different nanostructured CPCs, the formulation derived from the addition of 2 wt% of carbon black on the P10E matrix (C-P10E-2CB) showed to be the most promising system, also considering that the manufacturing process was not compromised by the use of nanofiller: the ILLS post-OAT test resulted to be about 186% higher than the corresponding value of the neat matrix composite (C-P). The in-depth temperatures (figure 2) at 3 and 5 mm from the hot surface and loss of mass trends after OAT test supported the evidence related to the ILSS results providing more insights on the ablation process of the produced CPCs.

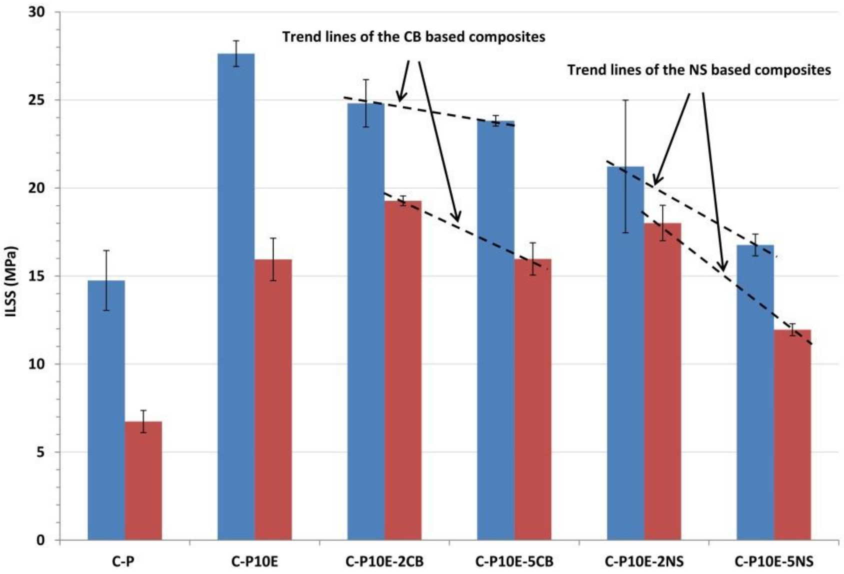

Figure 17. ILLS values pre- (blue) and post- (red) OAT tests.

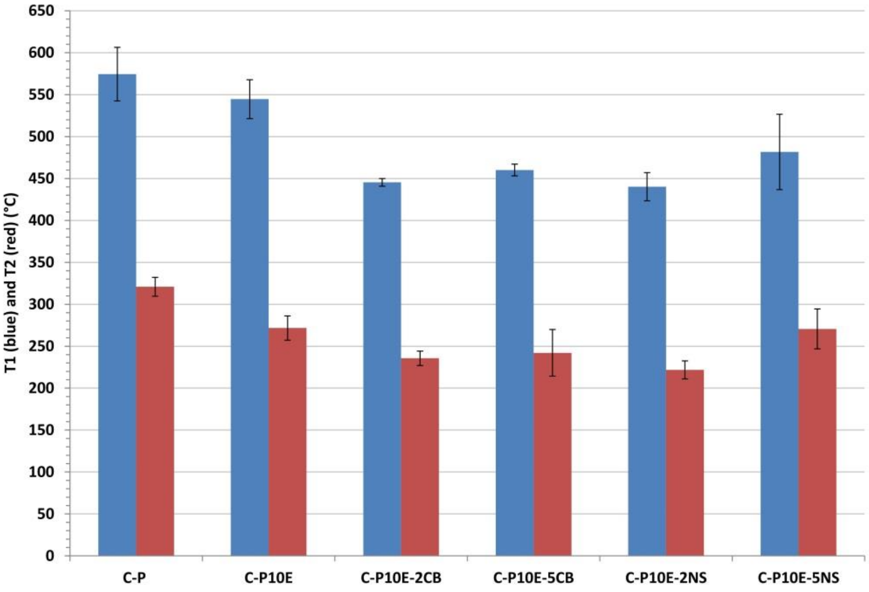

Figure 2. Maximum value of T1 (blue)registered at 3 mm from the hot surface and T2 (red) registered at 6 mm from the hot surface in an OAT test.

S3.26 NOVEL SYNTHESIS AND PROCESSING BY DIGITAL LIGHT PROCESSING OF CeO_2-STABILIZED ZIRCONIA COMPOSITES: INFLUENCE OF SECOND PHASES ON PRINTING PARAMETERS AND MICROSTRUCTURAL/MECHANICAL PROPERTIES

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In recent years, ceramic stereolithography (CS) has undergone an intense acceleration, rapidly extending to different research fields and advanced industrial applications.

To date, CS has been widely used in combination with various technical ceramics, such as alumina, zirconia and their composites, leading to obtaining high-resolution components with complex geometries and superior surface quality, not achievable by conventional manufacturing processes.

Among all, CeO_2-stabilized zirconia (CSZ) stands out for high flexural/compressive strength and toughness in moist and extreme environments, making it particularly suitable as structural component in sensors, instrumentation, probes, pumps and fluid control systems.

While superior mechanical performances can be easily achievable by adding secondary phases within the zirconia matrix to control and refine grain growth upon sintering, the significant absorbance of ceria in the UV range still represents one of the major issues in 3D printing strategies based on *light-activated polymerization.*

The present contribution aims at methodically addressing open challenges related the printability through Digital Light Processing (DLP) of CSZ composites characterized by increasing phase composition complexity, i.e., Ce-TZP, Ce-TZP/a-Al_2O_3 and Ce-TZP/a-Al_2O_3/$SrAl_{12}O_{19}$.

Novel surface modification strategies were developed to obtain high quality composite materials, then used as solid load within the printing ink composition. Slurries rheological behaviour was properly adjusted to reach the best compromise among polymerization efficiency and density of the printed parts.

The effect of secondary phases on zirconia transformability, microstructural and mechanical properties will be presented in detail, supported by X-Ray Diffraction (XRD), *Scanning Electron Microscopy (SEM) and 3-point bending experimental data, corroborated by a robust statistical analysis.*

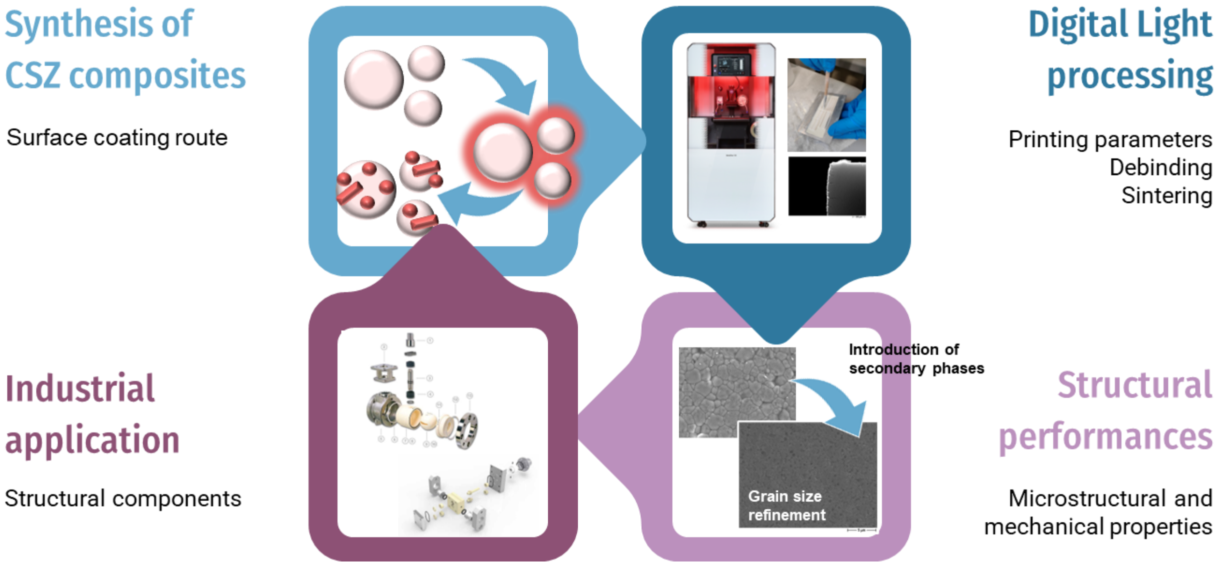

Figure 18. Graphical abstract: Digital light processing of CSZ-composites with increasing phase composition complexity.

	SESSIONE 4	
Codice	**Titolo**	**Corresponding Author**
S4.1	BIOPOLYMERS BLEND COMPOSITES CONTAINING BIOCHAR PARTICLES FROM ANAEROBIC URBAN WASTE DIGESTATE PYROLYSIS	Giulia Infurna
S4.2	CEMENTITIOUS TILE ADHESIVE WITH END-OF-LIFE TIRE DERIVED PRODUCTS: DESIGN AND CHARACTERIZATION	Sebastiano Candamano
S4.3	SIMULATING POST-CONSUMER PLASTIC WASTE BY ACCELERATED THERMAL AGEING: AN INVESTIGATION ON POLYAMIDE 6,6 COMPOSITES	Alessandro Salvi
S4.4	3D PRINTING OPTIMIZATION PROCESS PARAMETER FOR PA6,6 COMMERCIAL MATERIAL REINFORCED WITH RECYCLED CARBON FIBER FILLER	Ilaria Rossitti
S4.5	EVALUATION OF ENVIRONMENTAL PERFORMANCE OF A MATERIAL/ENERGY RECOVERY PLANT FROM ORGANIC FRACTION OF MUNICIPAL SOLID WASTE THROUGH LIFE CYCLE ASSESSMENT	Francesco Todaro
S4.6	MULTIFUNCTIONAL BIONANOCOMPOSITES BASED ON LIGNIN AND ITS HYBRIDS: USE IN PACKAGING, BIOMEDICAL AND AGRICULTURE SECTORS	Debora Puglia
S4.7	THE MATERIALS PERSPECTIVE AND CHALLENGES IN HYDROGEN STORAGE: TYPE IV CYLINDERS, METAL HYDRIDES, AND MORE	Giovanni Capurso
S4.8	COMPATIBILIZER PRECURSOR FROM WASTE VEGETABLE OIL FOR END-OF-LIFE FISHING NETS RECYCLING	Sara Filippi
S4.9	A SUSTAINABLE WAY TO TRANSFORM ETNA'S ASH INTO A RESOURCE FOR SICILY	Laura Aliotta
S4.10	CARBON MINERALIZATION OF ALKALINE WASTES: TOWARDS A CIRCULAR ECONOMY APPROACH	Paola Palmero
S4.11	INTEGRATING 2D MATERIALS INTO MESOPOROUS FILMS, AN ADVANCED FUNCTIONAL PLATFORM	Plinio Innocenzi
S4.12	DEODORIZATION OF POST-CONSUMER MIXED PLASTICS: DEGASSING AND MELT COMPOUNDING WITH ZEOLITES AS VOC ADSORBENTS	Emilia Garofalo
S4.13	USE OF A NANOFILLER FOR RECYCLING MULTILAYER BARRIER FOOD PACKAGING	Vincenzo Titone
S4.14	EVALUATION OF THE ENVIRONMENTAL IMPACT OF PLASTIC SPRAYERS THROUGH LIFE CYCLE ASSESSMENT: AN INDUSTRIAL CASE STUDY	Francesco Valentini
S4.15	POLYMER COMPOSITES INCORPORATING PERMANT MAGNETS FROM WASTE ELECTRICAL AND ELECTRONIC EQUIPMENT	Daniel Milanese

S4.1 BIOPOLYMERS BLEND COMPOSITES CONTAINING BIOCHAR PARTICLES FROM ANAEROBIC URBAN WASTE DIGESTATE PYROLYSIS

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The valorization of urban organic waste for energy production and the development of bio-based materials, are the trends undertaken by several current research programs and initiatives for sustainable development. Therefore, the formulation of bio-based materials, having good properties and performance in service and controlled end-of-life, is imperative for an effective circular economy. With the perspective of circular economy, in this work, sustainable composites have been produced adding biochar particles to biopolymers blend at different weight ratio of poly(butylene-adipate-co-terephthalate), PBAT, and poly-lactic acid, PLA. The lab-made biochar used, has been obtained as a by-product after biodiesel and syngas extraction obtained by means of slow pyrolysis of digestate (anaerobic digestion of organic urban waste). Samples of different PBAT:PLA weight ratio, in absence and in presence of 10 wt.% of BC have been produced by melt mixing and then subjected to accurate characterization by tensile test and dynamic mechanical analysis, rheological, morphological and thermal characterization. Previously BC particles characterization has been performed, by FT-IR and granulometry analysis. Moreover, viscometry test of biocomposites has been performed. All obtained results suggest that varying the PBAT:PLA weight ratio the contribution of the same amount of BC particles results diametrically opposed. In fact, a well pronounced reinforcement effect has been found with BC added to blend with a majority of PBAT. Instead, when added to blend with majority of PLA seem to have a role of pro-degradant reducing molecular weight. This results were also confirmed by viscometry test.

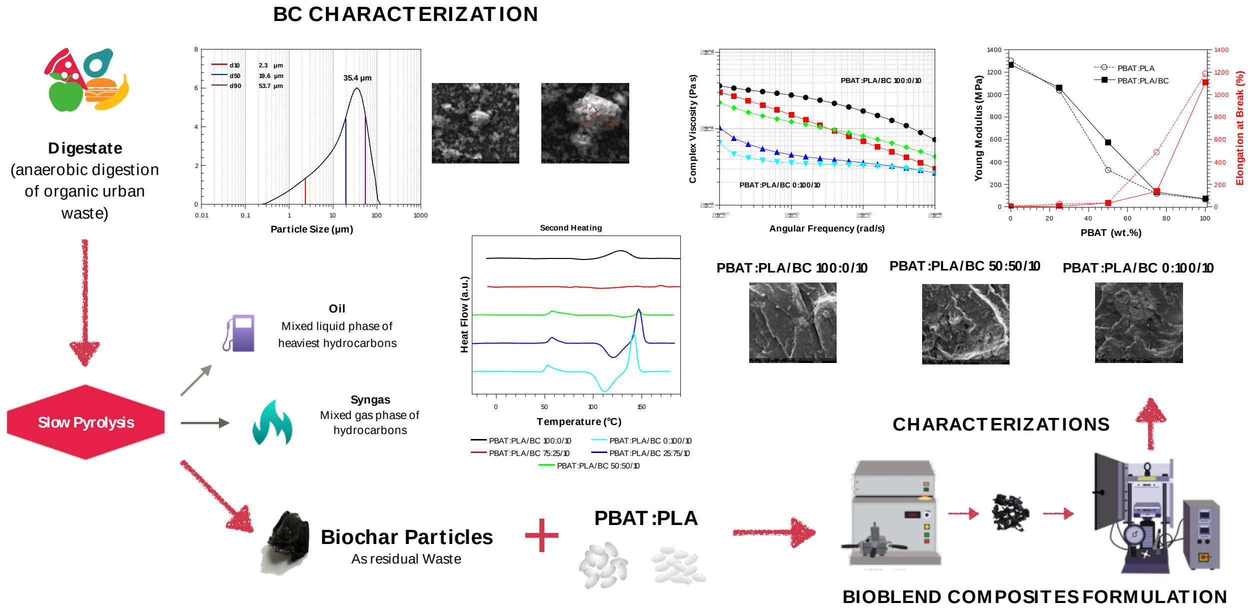

Figure 19. Graphical Abstract

S4.2 CEMENTITIOUS TILE ADHESIVE WITH END-OF-LIFE TIRE DERIVED PRODUCTS: DESIGN AND CHARACTERIZATION

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The term "end-of-life tyre (ELTs)" denotes a tyre that is no longer sufficiently safe or efficient to be reused, even if retreaded, thus turning into a waste. Its sustainable management, based on the circular economy and aimed to close the materials loop following the approach of recycling, reusing, or reducing materials has resulted in two different processes:

- *recycling, when new materials such as rubber, steel and textile fibres are obtained;*
- *recovering as energy mainly in cement factories.*

Among tyres' components, the rubber is a mix of very high quality polymers with exceptional chemical-physical characteristics. It can obtained by the means of mechanical shredding of tyres at room temperature in pieces of different dimensions, namely chips (20-50 mm), granules (0.8-20mm) and powders (<0.8mm).

They find application in the building industry, to improve acoustic insulation and produce anti-vibration elements, in sport sector with playgrounds and sports flooring and in the sector of roads in "modified" asphalts and cycle lanes.

Nevertheless, further feasible applications and markets need to be developed and maintained worldwide.

In this scenario, authors developed the mix design of a pre-mixed cementitious tile adhesive containing end-of-life tire derived rubber pieces. It was tested according to the main relevant standards and classified according their fundamental requirements. Test on tensile adhesion strength after 28day, after immersion in water, after freeze-thaw cycles, after heat ageing, deformability, open time, extended open time, vertical slip, thermal and acoustic insulation were carried out. The produced adhesive is classified as C1TS2

S4.3 SIMULATING POST-CONSUMER PLASTIC WASTE BY ACCELERATED THERMAL AGEING: AN INVESTIGATION ON POLYAMIDE 6,6 COMPOSITES

Alessandro Salvi[1,*], Francesco Marzullo[1], Marlena Ostrowska[2], Giovanni Dotelli[1], Claudia Marano[1], Francesco Briatico Vangosa[1]

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Recycling post-consumer plastic waste is challenging compared to post-industrial recycling as it unveils a series of disadvantages, specifically: (1) the waste stream has a highly variable composition, (2) it is often contaminated, and (3) it could contain degraded polymers. This last aspect is more relevant for products with a long lifespan, such as electrical equipment. Studying the recyclability of the plastic waste from such products can be a complex task because of the difficulty to foresee the properties of the degraded material after a lifespan that could reach up to 30 years.

This study aims at creating a model able to predict the mechanical properties of glass fiber reinforced plastics (GFRP) used in low-voltage circuit breakers. Specifically, the materials of choice are two polyamide 6,6 based composites, differing in glass fiber content and type of the added flame retardant agents.

Three-point bending test, DSC and FTIR analysis have been carried on the materials before and after performing a thermal aging at different temperatures and exposure times.

From the results, a decrease in flexural strength and an increase in crystallinity after aging was observed. The spectroscopic analysis suggests that the thermal treatment has caused oxidation of the polymeric molecules.

By plotting the mechanical property of the treated materials as a function of the aging time, at different aging temperatures, the time t_{20} *at which for each temperature a 20% decay of the material property has occurred was determined. Such decay has been identified by the producer as the maximum acceptable loss for the intended application of the material.*

S4.4 PRINTING OPTIMIZATION PROCESS PARAMETER FOR PA6,6 COMMERCIAL MATERIAL REINFORCED WITH RECYCLED CARBON FIBER FILLER

Ilaria Rossitti[1,2,*], Matteo Sambucci[1,2], Ilario Biblioteca[1,2], Sara Taherinezhad Tayebi[1], Marco Valente[1,2]

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The focus of this work was to study the characteristics and properties of composite materials using one of the additive manufacturing techniques, the 3D printing, thanks to the collaboration with Fab Lab SAPeri&Co of La Sapienza University, obtained by combining a PA6,6 matrix with a recycled carbon fibers (rCF)reinforcement. The used microfibers have been supplied by Carbon Task Srl and are the result of a second stream recovery process, starting from long fibers recycled carbon scraps, used to produce TNT fabrics. The production of TNT generates between 1 and 3% of fibers which are pulverized and, subsequently, a second waste stream. These waste are generally agglomerated short fibers that are impossible to untangle and therefore to use. This waste material is collected and ground in a ball mill, and this very filler was used in our work to produce a 3D filament material using a commercial PA6,6. We started from the production and characterization of three different types of filament by single screw extruder, which differ in the amount of carbon fiber reinforcement used, that are: (i) neat PA6,6 filament, made without reinforcement addition; (ii) PA6,6 + 5 wt% rCF filament; (iii) PA6,6 + 10 wt% rCF filament. Then, a first phase involved the printing process optimization with the commercial filament in PA12 focusing on specimens geometry, extrusion temperature and type of printing. The results of the analysis showed that the best choice falls on a dog bone geometry without a perimeter and with curved fittings to avoid the formation of voids, on a temperature of 265 °C and single printing. It has gone to print PA6,6 composite samples in order to be tested at tensile strength (Figure 1). While the values increase from neat to 5% rCF is excellent, it is not between 5 and 10%, reason why the printing process parameters for the latter case have been optimized, testing 3 different operating conditions: (i) printing temperature (Ts) increased to 270 °C and plate temperature (Tp) unchanged to 90 °C; (ii) Ts unchanged and Tp to 110 °C; (iii) Ts and Tp increased to 270 °C and 110 °C. From the tensile tests and microstructure analysis, the third chosen route was the correct one, which ensured the disappearance of inter-filament voids.

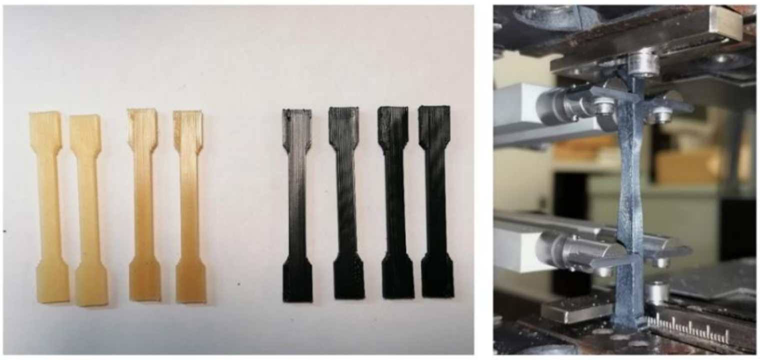

Figure 20. Tensile test specimens in PA6,6 neat and composite

S4.5 EVALUATION OF ENVIRONMENTAL PERFORMANCE OF A MATERIAL/ENERGY RECOVERY PLANT FROM ORGANIC FRACTION OF MUNICIPAL SOLID WASTE THROUGH LIFE CYCLE ASSESSMENT

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The valorization of the organic fraction of municipal solid waste (OFMSW) plays a key role in sustainable resource management. Indeed, OFMSW recycling is important for transforming the linear economy model into a circular one. In this context, the combined technology of anaerobic digestion and composting of OFMSW is configured as one of the best available technologies (BAT). Anaerobic digestion produces biogas, which can be used to generate electricity/heat or upgraded into biofuel, and digestate, which can be turned into soil improver through a composting process.

This work presents the environmental performance evaluation of the combined treatment plant of AMIU Puglia that handles the organic waste produced in the municipality of Bari. This plant was designed to process 40000 tons/year of organic waste and to produce 10 thousand tons of compost and 3.8 million m^3 *of biogas (Fig. 1).*

LCA (life cycle assessment) study was developed according to the ISO 14040 (2006) to evaluate the environmental impacts of organic waste treatment in AMIU Puglia plant. During the study, a mass balance was performed to quantify the energy produced and compost recovered, as well as the process losses.

Figure 1. AMIU Puglia plant.

The link between the waste characteristics and the technical-operational parameters of this plant was studied to maximize the production of biogas and compost. Indeed, the chemical composition of the substrate plays a crucial role among the factors responsible for digestion performance and cumulative methane production.

LCA results show that composting is characterized by major impacts due to the energy consumption associated with the treatment of exhausted air (fig. 2). Overall, the environmental benefits from the production of a good quality compost helped to decrease the impact. Anaerobic digestion shows environmental advantages from the production of electricity and thermal energy (fig. 3).

Furthermore, sensitivity analysis in the LCA will be conducted to identify parameters that can considerably change the result, which might need further investigation.

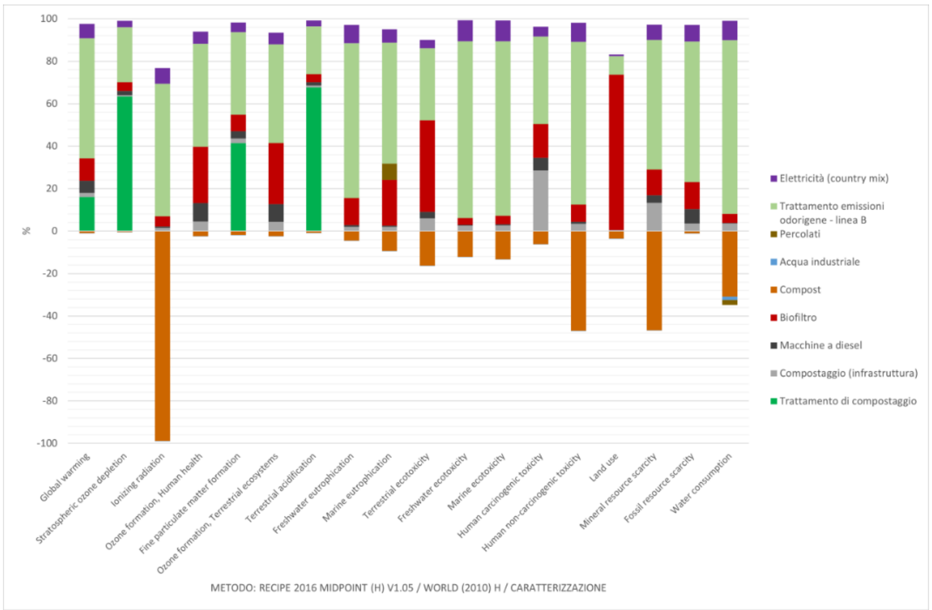

Figure 2. Midpoint characterization of composting treatment.

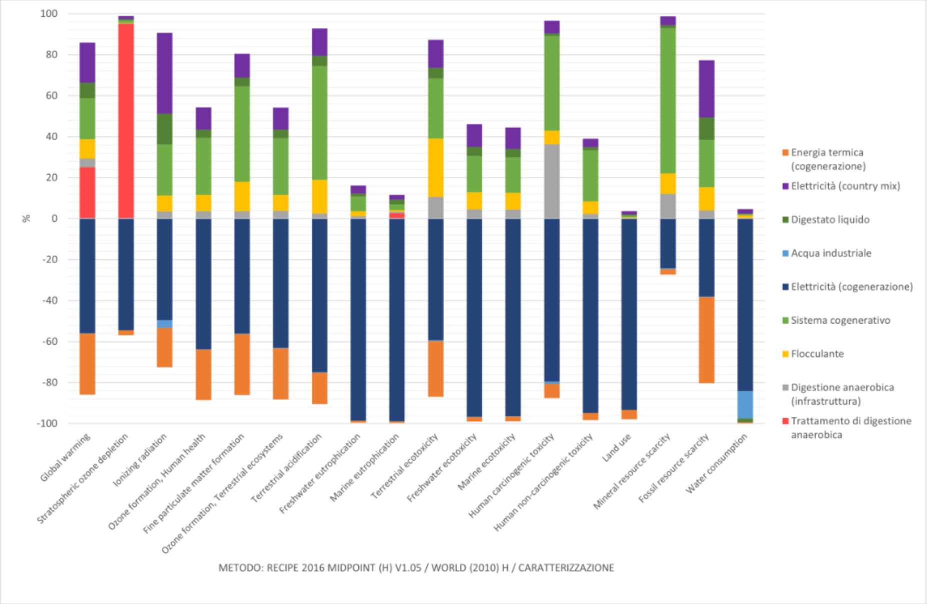

Figure 3. Midpoint characterization of anaerobic treatment.

S4.6 MULTIFUNCTIONAL BIONANOCOMPOSITES BASED ON LIGNIN AND ITS HYBRIDS: USE IN PACKAGING, BIOMEDICAL AND AGRICULTURE SECTORS

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The preparation of lignin-based nanocomposites and its hybrid systems needs different blending procedures and chemical modifications . In its original state, lignin can enhance mechanical and thermal stability of a polymeric blend or a composite, acting as a UV blocker, antimicrobial, antioxidant or flame retardant agent. However, high loadings of irregular lignin micropowders in polymers are hindered, mostly due to the lack of compatibility: in order to preserve multifunctionality, controlled size, surface shape and chemistry could overcome these issues and win on miscibility crucial effect when a nanocomposite approach is considered. Lignin nanoparticles in melt extruded polylactic nanocomposite films, if properly dispersed, confirmed the positive antimicrobial and antioxidant role of this nanofiller towards different pathogens in fresh food packaging [1]. Additionally lignin, as a natural three-dimensional network, has shown the ability to create uniform composite structures with inorganic nanoparticles, so organic–inorganic hybrid materials have similarly received wide-ranging attention [1]. The production of lignin/inorganic hybrid nanosystems represents also a new methodology for high-value reuse of this material, since the hybridization potentially combines the benefits of all constituents to acquire matching synergic properties [2]. According to this, we put emphasis on the use of lignin in a synergic role in presence of other inorganic metal oxide nanoparticles (Figure 1a), or as seeds priming (Figure 1b) [3]. Biopolymeric tridimensional scaffolds were also prepared by TIPS in presence of LNPs or ZnO@LNP LNP , confirming the active role of the hybrids towards the interaction with cellular proteins at the interface between biomaterials and cells and the capability to adsorb and photodegrade methylene blue (MB) for dye removal purposes.

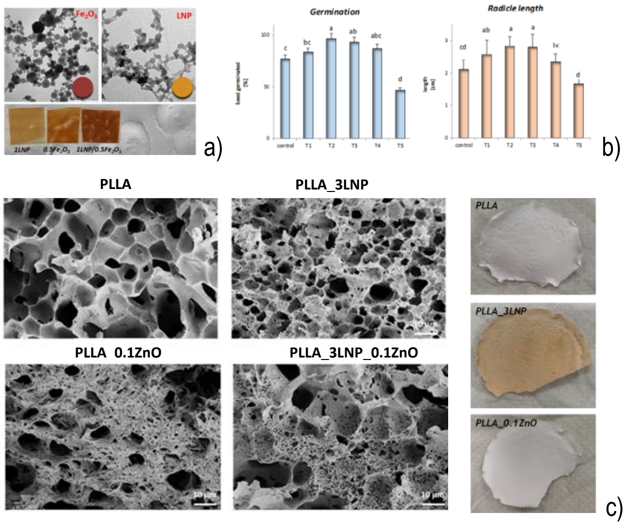

Figure 1: (a) TEM images for Fe_2O_3, LNP and visual images of PLLA films containing the two nanofillers); (b) Effect of the treatments with ZnO@LNP on the shoot and root length and fresh weight compared to the untreated controls (T1, T2, T3, T4, and T5 refer to the ZnO@LNP concentration used for seed nano-priming); (c) PLLA based scaffolds obtained by TIPS in presence of LNP, ZnO and Zno@LNP as function of dioxane/water ratio

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S4.7 THE MATERIALS PERSPECTIVE AND CHALLENGES IN HYDROGEN STORAGE: TYPE IV CYLINDERS, METAL HYDRIDES, AND MORE

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To address the threats that the most recent abrupt climatic and economic upheavals are posing, energy harvesting and storage technologies must progress just as rapidly. Among these, it is very likely that hydrogen will play a non-marginal role as an energy carrier in a renewable-based scenario. Thus, technical development should allow the most effective exploitation of supplies and the most efficient storage systems.

Even considering the drawbacks of safety and energy invested for hydrogen compression, high pressure gas cylinders are one of the most mature and applied technologies, but their manufacturing is not completely problem free. Few examples regarding Type IV cylinders are shown in this work, from the suitability of the internal liner polymer and its processing to the optimization of the composite shell. The perspective of a completely sustainable life cycle for these products and materials, as well, is motivating the research and development of recyclable materials, both concerning the polymer matrix and the reinforcement fibers.

Solid state hydrogen storage is a newer approach, and it addresses some of the issues of physical storage methods by increasing the reachable energy density, owing to a host material that allows storing hydrogen atoms closer to each other. In this field, the tasks for material development are numerous and they range from the kinetics of reaction to the thermal management of powder beds, from the risk of losing active surface by sintering or oxidizing powders to a reactor design that encompasses all possible energy saving strategies. Applying once more the LCA approach, interesting steps were done in the manufacturing, for example with the exploitation of other industrial waste materials, but the restoring and recycling of these hydride materials is still a pioneering field.

In general, in this subject, materials science challenges are numerous, which are worth investigating for a greener future.

S4.8 COMPATIBILIZER PRECURSOR FROM WASTE VEGETABLE OIL FOR END-OF-LIFE FISHING NETS RECYCLING

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Discarded, lost or abandoned fishing nets are one of the main problems of sea pollution. Giving new economic value to end-of-life fishing nets could encourage their recovery through an appropriate logistic collection system. Nets recycling would contribute both to circular economy directives and reducing marine pollution. In this work discarded fishing nets (Figure 1) are washed, cut and preliminary characterized by differential scanning calorimeter (DSC), infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and polarized optical microscopy (POM). The analysis confirmed that they are mainly constituted by polyamide-6 (PA6) and ultrahigh molecular weight polyethylene (HDPE). In the first part of the work, they have been separated, and mechanically processed to have again PA6 and HDPE pellets. The properties of the recycled materials were compared with those of virgin samples to evaluate the possibility of their reuse in a second production cycle. In the second part of the work, they were instead processed together, avoiding any preliminary separation. Assuming two different scenarios in which either polyamide or polyethylene could constitute the matrix of the final mixture, PA6/HDPE blends with 75/25 or 25/75 wt% composition were considered. To improve the compatibility between the polar polyamide and the non-polar polyethylene, few percentages of a compatibilizer precursor, were added. The latter, also coming from waste materials, was synthetized by laboratory epoxidation of treated waste vegetable oil, collected from the catering circuit. Epoxy groups react, during the blending, with the amino terminal groups of the polyamide and the vegetable oil grafted with polyamide moves to the interphase reducing the interfacial tension. All the blends were characterized from morphological, thermal and mechanical point of view.

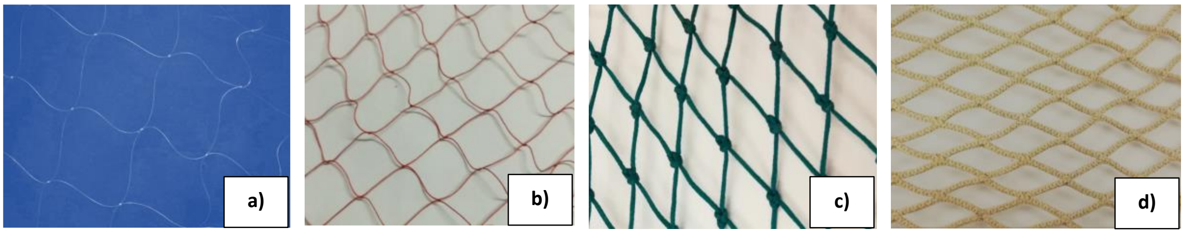

Figure 21. Example of waste fishing nets: a) gillnet, b) trammel net, c) trawl net and d) fish farming net.

S4.9 A SUSTAINABLE WAY TO TRANSFORM ETNA'S ASH INTO A RESOURCE FOR SICILY

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Circular economy is a virtuous path to be followed; however only 9% of the world's economy is circular. It is difficult to find an universal circular approach; nevertheless, by analyzing the local available wastes, it is possible to create local circular economy pathways. In this context, the use of suitable waste products as raw materials to be used in the composites industry has become an interesting matter of study.

In countries with active volcanoes, volcanic ash is considered a waste, but it can become a feasible, nature-based solution for soil improvement and CO_2 reduction; in fact, it has been proved that the soils in which volcanic ashes are present, are more fertile and carbon rich.

With its 3357 m above sea level, Etna is the highest volcano in continental Europe, as well as one of the most active. Due to the frequent eruptions, tons of volcanic ash are produced annually; only in the mid of March 2021 over 250 tons of ashes were collected, but where does all this material go? There are two solutions: the landfill or aggregate recovery plants).

The aim of this study is to find a more sustainable third way to transform Etna's ash into a resource for Sicily by the use of these biocomposites for producing pots for agriculture applications with a double function: being biodegradable and able to release ashes in the soil with a high fertilizing action. At this purpose, Etna ashes were collected, sieved and added into a poly(butylene succinate-co-adipate) (PBSA) biodegradable polymeric matrix. Different ash amounts were embedded into the matrix from 10 up to 40 wt.% by extrusion compounding. The granules obtained were then injection molded for the subsequent thermomechanical characterization, studying the effect of the ash on the injection molding processability. Disintegration tests and phytotoxicity tests were also carried out for selecting the best formulation as promising solution to produce injection molded objects to be used in agriculture applications.

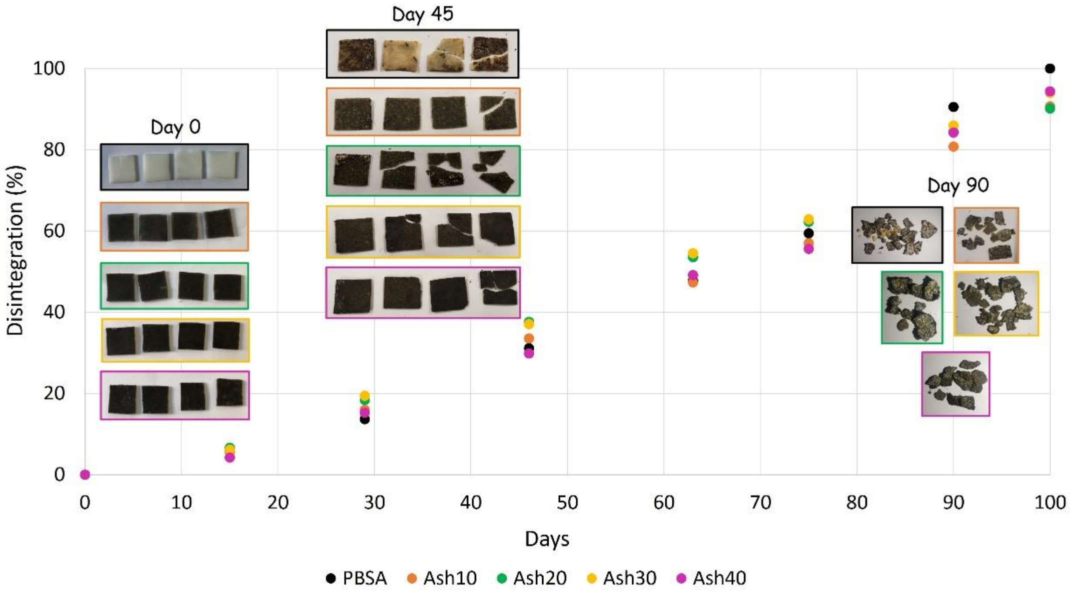

Figure 1. Disintegration results ad photographs of samples once the composting under laboratory conditions at day 0, day 45 and day 90.

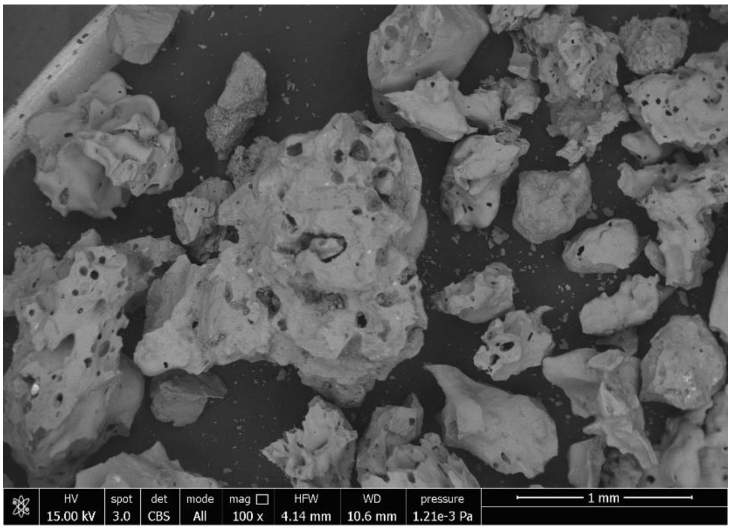

Figure 2. Etna's ashes SEM micrograph.

S4.10 CARBON MINERALIZATION OF ALKALINE WASTES: TOWARDS A CIRCULAR ECONOMY APPROACH

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The crucial need to reduce carbon dioxide emissions pushed the development of new technologies, such as mineralization, in which CO_2 reacts with alkaline compounds, including minerals and solid waste, and converts into solid inorganic carbonates.

In this research, the potential of two industrial wastes towards carbon mineralization was investigated. Electric Arc Furnace (EAF) slag and Cement By-Pass Dust (CBD), in the form of powders, were submitted to a slurry carbonation process (Fig. 1a). In a future perspective, the mineralized powders could be reused as supplementary cementitious materials (SCMs) or fine aggregates, thus contributing not only to reduce the CO_2 levels, but even to decarbonize the construction industry, and boost the circular approach.

While EAF slag originates during the production of steel from metal scraps, CBD is generated during cement production: here, as the by-pass is used to extract alkali, sulphates and chlorides originated from the kiln fuel, these salts remain in the waste besides basic compounds. Slurry carbonation was implemented in an open system, under mild conditions: room temperature, atmospheric pressure, short reaction time. A minimum of three replicates assessed the reproducibility of the tests. The efficiency of the process was calculated in terms of CO_2 uptake, considering the content of CO_2 before and after the carbonation. Three CO_2 quantification methods were used: thermogravimetric analysis, furnace decomposition and acid digestion.

The study confirmed the effectiveness of both industrial wastes to provide CO_2 mineralization, while the three quantification methods showed comparable results (Fig. 1b). Worth of mention is the CBD efficiency, providing ~ 23% CO_2 uptake and highlighting its great potential to contribute to CO_2 permanent storage. Furthermore, during the same process, it was possible to separate ~ 60% of the salts included into CBD, boosting its possible employment as SCM or aggregates in the cement industry.

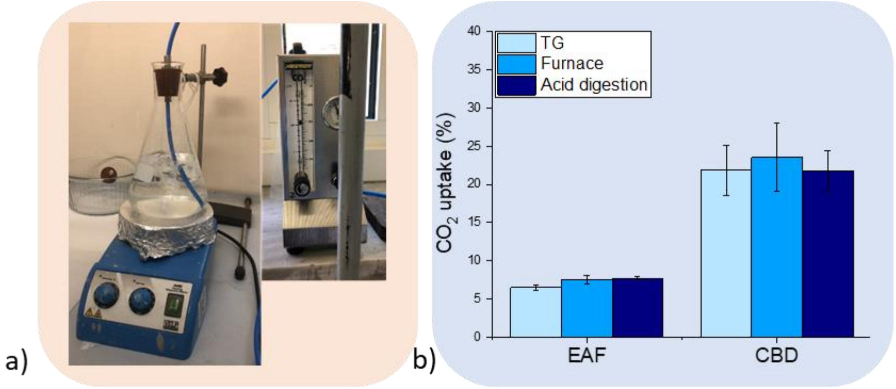

Fig. 22. a) Set-up of the slurry carbonation technology; b) CO_2 uptake results related to EAF and CBD waste, quantified by three different methods: thermogravimetry (TG), furnace decomposition and acid digestion.

S4.11 INTEGRATING 2D MATERIALS INTO MESOPOROUS FILMS, AN ADVANCED FUNCTIONAL PLATFORM

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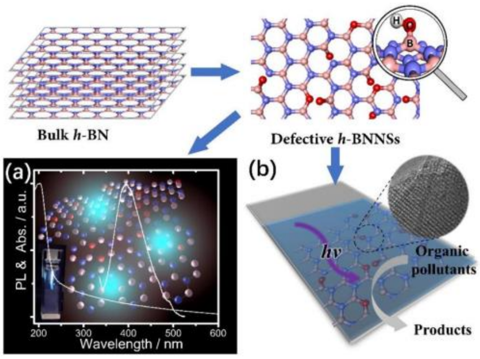
2D layered materials, which have a layered structure similar to graphene, have attracted much attention because they have disclosed revolutionary new properties. The fabrication of advanced multifunctional materials whose properties are governed by the nanoscale requires the ability of mastering complex systems and related synthesis processes. The new generation zero-dimensional materials, such as carbon dots, provide the necessary functional properties that however need to be integrated into nanosystems of different scales. Two-dimensional materials such as graphene, boron nitride, WS2 and SnSe2, have been used to obtain heterostructures with highly controlled properties upon integration in mesoporous ordered titania films. Supramolecular self-assembly techniques, governed by weak forces during the evaporation of the solvent, allow the integration of nanomaterials into complex structures. Mesostructured materials are an example of such self-assembled materials that can host 2D materials without the disruption of the mesophase. The fabrication of devices capable of performing functions at different scales or through synergy due to the formation of nano-heterostructures can be, therefore, achieved through a pre-programmed self-organization. The 2D materials-mesoporous films heterostructures have shown to increase the photocatalytic performances of the system.



S4.12 DEODORIZATION OF POST-CONSUMER MIXED PLASTICS: DEGASSING AND MELT COMPOUNDING WITH ZEOLITES AS VOC ADSORBENTS

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One of the major challenges for the up-grading of mechanically recycled polymers, to make them fit to re-use for the same or new applications, concerns the removal of malodorous substances embedded inside the materials. This study focused on the deodorization of two types of post-consumer plastic waste: PW and Fil-S. Even if they come from completely different waste collection and recycling processes, they show a quite similar composition, being constituted mainly of polyethylene and a minor content of polypropylene. Two deodorization techniques were implemented on these post-consumer mixed plastics. In particular, a new promising strategy, i.e. the melt compounding of the recycled materials with zeolites, was compared with the more traditional extrusion with degassing, which is already applied at industrial scale for VOC removal.

Based on the results obtained in this study, the degassing technology wasn't able to reduce, in a satisfactory extent, the unpleasant smells of both the recycled plastics, despite the optimization of the residence time inside the extruder and the use of water, as stripping agent. This suggested the need for alternative and/or complementary deodorization strategies.

The melt compounding of the recycled materials with two micrometric zeolites (zeolite 13X and Z310) resulted more effective in the off-odors removal respect to degassing. In particular, at zeolites' amount of 4wt% the highest reduction (-45%) in the Average Odor Intensity (AOI) was measured for both PW/Z310 and Fil-S/13X systems, compared with the corresponding recycled matrices. Finally, by combining degassing and melt compounding with zeolite, the best result was obtained for the composite Fil-S/13X, whose AOI value resulted quite close (+22%) to the one of the corresponding virgin benchmark.

S4.13 INTEGRATING 2D MATERIALS INTO MESOPOROUS FILMS, AN ADVANCED FUNCTIONAL PLATFORM

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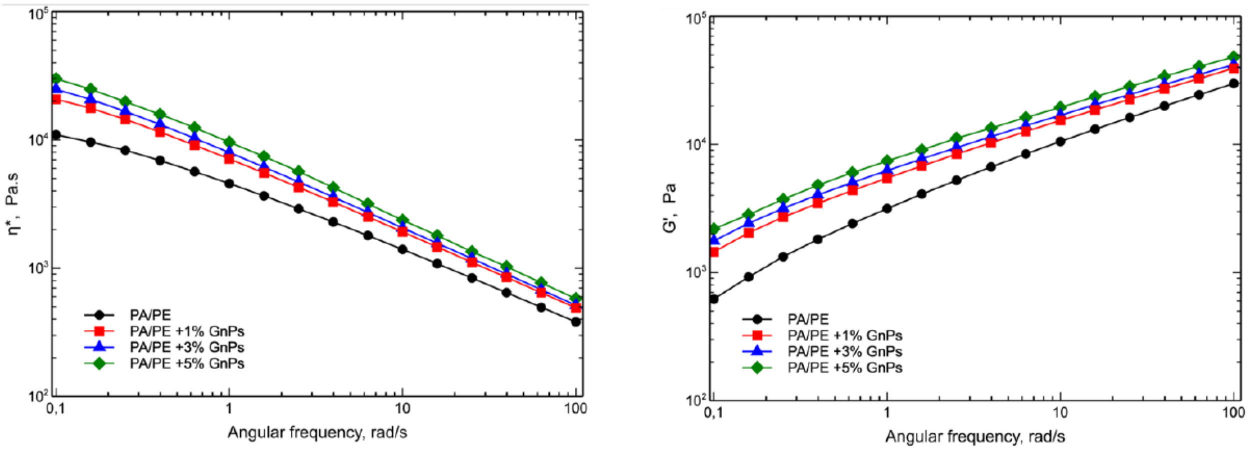
Recycling or reusing the plastic packaging is essential to prevent the unstoppable increase in the release of polymeric materials into the environment and thus minimize environmental pollution. However, barrier food packaging films are very difficult to recycle since they are multilayer films composed by at least two different materials. In this content, aim of this work was to evaluate the use of a nanofiller for the mechanical recycling of multilayer barrier plastic bags used for food vacuum packaging.

Polyamide (PA)/polyethylene (PE) bilayer commercial packaging was ground and then melt mixed with different amount of graphene nanoplatelets (GnPs). Moreover, nanocomposites were obtained by using plastic bags subjected to accelerated weathering or to the contact with a food simulant.

The materials were fully characterized through morphological analysis; rheological characterization; tensile tests and thermal analysis. All the specimens used for the characterizations were prepared by compression molding in a hydraulic laboratory press.



a) ***b)***

Figure 1. a) Complex viscosity (η^*) as a function of angular frequency; b) Storage modulus (G') curves as a function of angular.

The results clearly show that GnPs influences the rheological response of nanocomposites (see Figure 1a). In particular, it is observed that the complex viscosity increases as the amount of GnP increases at low frequencies, with slightly more pronounced non-

Newtonian behavior at high frequencies compared to the PA/PE matrix. The viscoelastic behavior of the three nanocomposites is also affected by the presence of GnPs. The modulus values are slightly higher, with a slight pseudo-solid behavior at lower angular frequencies (see Figure 1b).

Mechanical test results show that the addition of GnPs causes an increase in elastic modulus and tensile strength, but a slight decrease in elongation at break values due to better interfacial adhesion between the phases. Finally, results on the effect of GnPs on post-consumer packaging showed that the resulting nanocomposites can be a viable way to recycle these products.

S4.14 EVALUATION OF THE ENVIRONMENTAL IMPACT OF PLASTIC SPRAYERS THROUGH LIFE CYCLE ASSESSMENT: AN INDUSTRIAL CASE STUDY

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This Life Cycle Assessment aims to evaluate the environmental impact of trigger sprayers which are used for the distribution of liquid detergents for household cleaning operations. A trigger sprayer is an object designed to release a specific amount of the liquid present in the container. The functional unit of the process is one trigger sprayer. Primary data regarding core processes and the distribution of the sprayer were directly provided by the producer. Data referred to the year 2019. Background data were taken from the Ecoinvent v. 3.5 database. The environmental impact assessment has been evaluated using the EPD 2018 method (v1.04) and included 8 impact categories.

From the results of the study three main contributors to the overall environmental impact were identified: few heavier plastic components of the sprayer, the electricity consumption for the production process and the end-of-life stage of the sprayer and related packaging. The substitution of polyoxymethylene with polypropylene reduced the impact according to the water scarcity category of 35 %. The use of recycled PP material instead of the virgin one for the production of three components allowed a modest reduction in the total contributions for each impact category: from 1 to 18 % (global warming potential -5 %). The use of renewable resources allowed a strong decrease in the total contribution for each impact category: from 11 to 54 % (global warming potential -16 %). The combination of the three proposed variants led to a carbon footprint of 0.0771 kg CO_2 eq, with a reduction of around 23 % with respect to the analysed industrial process. Moreover, to further improve the environmental performance of this product a twofold strategy should be followed: from one side the implementation of technologies able to produce the same components using only recycled material and on the other side the enhancement of a sorting and recycling supply chain able to provide high-quality recycled polymers.

S4.15 POLYMER COMPOSITES INCORPORATING PERMANT MAGNETS FROM WASTE ELECTRICAL AND ELECTRONIC EQUIPMENT

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Composite materials produced by embedding ferro-and ferrimagnetic particles in a nonmagnetic matrix are also known as bonded magnets. With respect to traditional bulk magnetic materials (metals, alloys, or ferrites), they have a significant advantage thanks to their higher electrical resistivity, lower density, chemical stability, and processability. They can be used to fabricate magnetic and electric devices, including electrical machines, microwave inductors and antennas.

In this work we report on the study of the influence of the content of magnetic particles obtained from waste electrical and electronic equipment (WEEE) on the mechanical, thermal and magnetic properties of the bonded magnets. The Neodymium Iron Boron (NdFeB) permanent magnets employed were taken from obsolete hard drives, demagnetized, and ground to particles under inert atmosphere down to a maximum size of50 micron. Incorporation of the so obtained particles into a thermoplastic flexible polymer matrix (polybutylenadipate-co-terephthalate -PBAT) was then carried out in a twin-screw extruder. Specimens containing30 and 50% wt of particles were finally obtained using composite pellets by injection molding in the form of model 5A specimens(UNI EN ISO 527)(Figure 1). Another set of samples was obtained by fuse deposition modelling (FDM) 3D printing either on a traditional support or directly onto a permanent magnet.

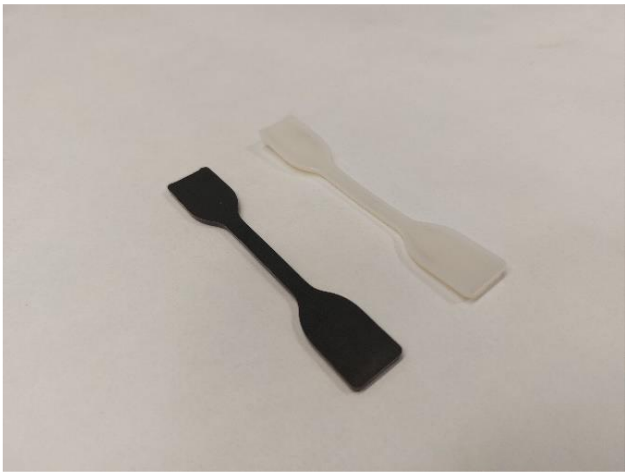

Figure 1. Injection molded plastomagnet (on the left) and the same type of specimen with the pristine polymer (on the right).

The results of the effect of the particles content and of the fabrication method on the magnetic properties of the sample will be discussed.

Aknowledgments

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SESSIONE 5-C		
Codice	**Titolo**	**Corresponding Author**
S5-C.1	RECYCLING OF PET PACKAGING IN CEMENT MORTARS: TOWARDS CLOSED-LOOP PROCESS	Giusy Santomasi
S5-C.2	ASSESSMENT OF DIFFERENT PHOTOCATALYTIC FILLERS IN CEMENT MATRIX	Angela Malara
S5-C.3	COMPARISON BETWEEN THE USE OF ALKALI-ACTIVATED, LIME-BASED AND CEMENTITIOUS MATRICES FOR FRCM APPLICATIONS	Jacopo Donnini
S5-C.4	VALORIZATION OF WOOD ASHES IN GEOPOLYMER – BASED MORTARS	Assunta Campanile
S5-C.5	CIRCULAR SOLUTION FOR SHELL WASTE AS CEMENT SUBSTITUTE IN CONSTRUCTION MATERIALS: STRATEGY FOR THE IMPROVEMENT OF MECHANICAL PERFORMANCE	Chiara Giosuè
S5-C.6	VALORIZATION OF A SECONDARY STREAM OF RECYCLED CARBON FIBRES IN CONCRETE APPLICATION: COMPATIBILITY, PERFORMANCE, AND COMPOUNDING OPTIMIZATION	Matteo Sambucci
S5-C.7	EFFECTIVENESS OF SODIUM BICARBONATE TREATMENT ON THE MECHANICAL PERFORMANCES OF SISAL FIBER REINFORCED GEOPOLYMERIC COMPOSITES	Carmelo Sanfilippo
S5-C.8	APPLICATION OF CELLULOSE AND CHITIN NANOSTRUCTURED SPONGES FOR CONTAMINATED SEDIMENTS REMEDIATION	Milvia Elena Di Clemente
S5-C.9	PHOSPHATE TREATMENTS IMPROVE THE PHYSICAL-MECHANICAL PROPERTIES OF CEMENTITIOUS SUBSTRATES	Enrico Sassoni
S5-C.10	A FUZZY MODEL FOR STUDYING DEGRADATION KINETICS OF GENNA MARIA NURAGIC BUILDING	Marta Cappai
S5-C.11	DEVELOPMENT AND PERFORMANCE OF A SUSTAINABLE ANTIMICROBIAL NANOCOMPOSITE TREATMENT FOR POROUS STONES PROTECTION	Paola Scarfato
S5-C.12	SMART PROPERTIES OF ALKALI-ACTIVATED SLAG-BASED MORTARS WITH TITANIUM DIOXIDE	Denny Cofetti
S5-C.13	LOW-CARBON PERVIOUS CONCRETE MADE WITH ALKALI-ACTIVATED BINDER AND RECYCLED SAND AND GRAVEL: THE ROLE OF THE AGGREGATE SIZE	Luigi Coppola

S5-C.1 RECYCLING OF PET PACKAGING IN CEMENT MORTARS: TOWARDS CLOSED-LOOP PROCESS

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Polyethylene terephthalate (PET) is a thermoplastic polymer belonging to polyesters and is one of the most widely used plastics in the packaging industry. The largest use of PET is for beverage bottles, but a wide quantity of PET in sheet form is used for transparent rigid food packaging in thermoformed trays. Rather than recycling bottles is a proven process, for PET-trays it is still challenging. Indeed, PET-tray feedstock, characterized by high heterogeneity with multiple packages and different compositions (mono-material or multi-layer), is not designed for recycling and contains food residues.

This research focuses on the recycling of rigid PET packaging. In particular, the studied recycling pathway concerns mechanical recycling for producing rPET pellets and alternative reuse of industrial scraps as replacement of the conventional aggregate in cement mortars.

Firstly, the study aimed to develop a deep understanding of the impurities present in the sorted PET trays and their impact on the properties of the obtained rPET. Therefore, contaminants were identified, and processes were studied to either avoid their presence or to remove them during the recycling process. The experimental phase involved a pilot-scale mechanical recycling process with the production of rPET pellets, which were injection-molded into plaques for thermal and optical analysis. Thereafter, different types of conglomerates were studied in fresh (rheology) and hardened states (mechanical, thermal, and microstructural properties) to show the peculiar characteristics of each composite.

It is possible to conclude that new applications for rPET were identified in both studied recycling pathways.

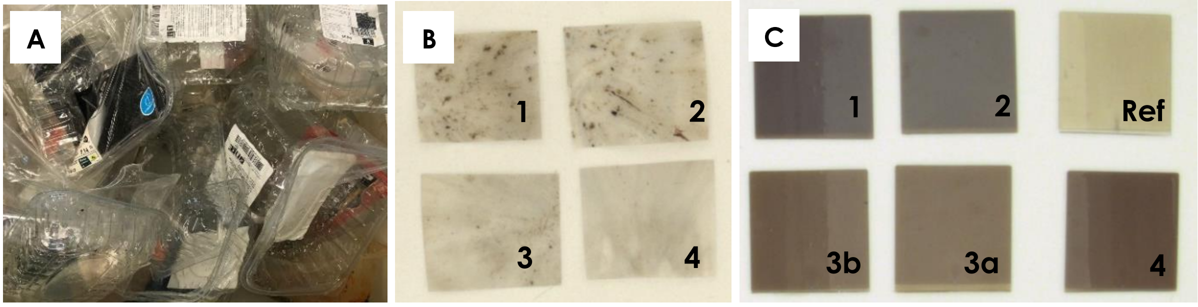

Figure 1. A) PET trays waste samples B) Compression-molded foils produced for optical evaluation (impurities presence examination and color analysis) before extrusion process C) Injection-molded plaques produced from extruded pellets of rPET

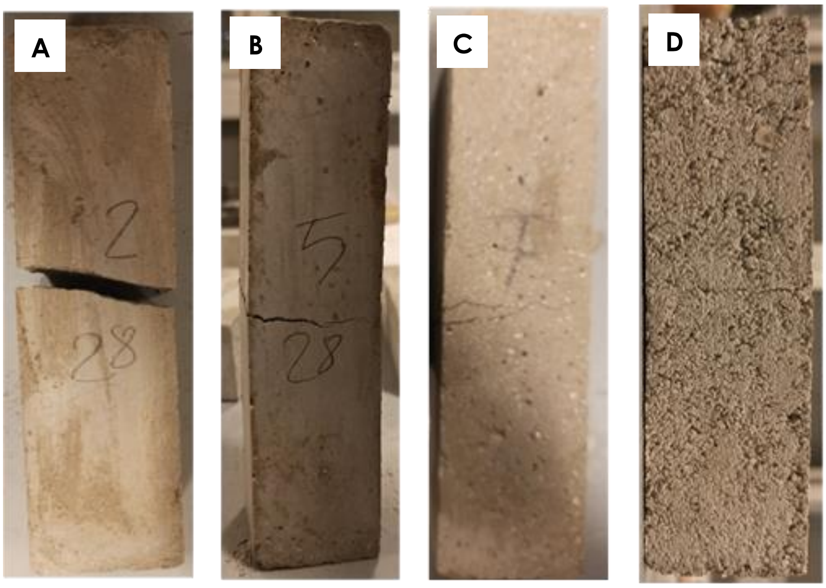

Figure 2. Flexural cracks in conglomerate samples C) SAND1 sample, D) SP50 sample, E) PET2 sample, F) PET1 sample.

S5-C.2 ASSESSMENT OF DIFFERENT PHOTOCATALYTIC FILLERS IN CEMENT MATRIX

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The application of photocatalysts in concrete technology is a well-established concept that potentially could take the environmental benefits. Photocatalytic cement is a type of cement that has been modified with photocatalytic materials. When exposed to light, the photocatalytic materials in the cement react with water vapor and pollutants in the air to break down organic and inorganic compounds. The main application of photocatalytic cement is in building materials, particularly for paving stones, tiles, and concrete. By incorporating photocatalytic fillers into these materials, it is possible to create surfaces that can help to clean the air by breaking down pollutants such as nitrogen oxides, volatile organic compounds (VOCs), and other harmful substances.

Typically, titanium dioxide (TiO_2) is the most extensively utilized photocatalyst in building materials because of its properties like a greater photocatalytic activity than other metal oxide photocatalyst and compatibility with standard building materials like cement.

In this work materials alternative to titanium oxide were incorporated in cement matrix to investigate their photocatalytic activity. The fillers investigated are microporous materials ETS-4 and ETS-10 (Engelhard Titanium Silicalite), that present a structure similar to that of inorganic microporous zeolites, composed of tetrahedral SiO_4^{4-} and octahedral TiO_6^{8-} units. ETS-4 ($Na_9Si_{12}Ti_5O_{38}(OH)\cdot 12H_2O$) and ETS-10 ($M_2TiSi_5O_{13}$ H_2O with M = Na, K) have both a microporous structure but differ from each other in the Si/Ti ratio and in the pore size.

The photocatalytic activity of ETS incorporated in the cement was measured in situ. The benefits of adopting in situ measuring devices, such as automated monitoring systems, are as follows: I) data are collected under ambient circumstances; ii) in situ data collecting is beneficial for exploratory or diagnostic research; and iii) real-time data acquisition enables maintenance planning.

S5-C.3 COMPARISON BETWEEN THE USE OF ALKALI-ACTIVATED, LIME-BASED AND CEMENTITIOUS MATRICES FOR FRCM APPLICATIONS

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The use of Fabric-Reinforced Cementitious Matrix (FRCM) systems for the reinforcement of existing masonry and concrete structures is nowadays a well-established technique. Despite the term 'cementitious', these systems also provide for the possibility of using cement-free matrices, such as lime-based or alkali-activated mortars. In this study, the possibility of using different alkali-activated matrices, based on fly ash or metakaolin precursors, for FRCM applications, was investigated. The mechanical performances of FRCM made with alkali-activated mortars have been compared with those of two commercial FRCM systems, made with lime- or cement-based mortars. All mortars have been initially characterized in terms of workability, dynamic elastic modulus, flexural and compressive strength. Then, direct tensile tests have been performed on a total of 48 FRCM coupons, reinforced with a bidirectional grid made of basalt or AR glass fibers. Finally, 18 shear bond tests on masonry bricks have been also carried out to evaluate the compatibility of developed alkali-activated mortars with masonry substrates.

Experimental results showed that the tensile resistance of alkali-activated FRCM is comparable (sometimes higher) than that of lime or cement-based FRCM commercial systems. Bond tests also showed that alkali-activated mortars exhibit excellent adhesion to masonry substrates, since failure always occurred without involving the interface between the mortar and the substrate. These preliminary tests showed that it is certainly of interest to deepen the possibility of using this type of matrices, with a lower environmental impact, for FRCM applications.

Figure 23. FRCM systems: tensile test setup.

S5-C.4 VALORIZATION OF WOOD ASHES IN GEOPOLYMER – BASED MORTARS

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Current global biomass ash production is estimated at about 170 Mt/year [1]. In particular, wood ash results from the combustion of wood resources used for energy production and heating [2] and is classified as biomass ash. Unfortunately, still today, most wood ash (about 70%) is disposed in landfills [3]. With this in mind, the present research activity focuses on the reuse of wood ash as a sustainable raw material for the production of geopolymer mortars. Beechwood ashes (BWA) was selected for this research and the XRD spectrum was reported in Figure 1, that reveals the presence of three crystalline phases, Calcium Carbonate, Potassium Calcium Carbonate and Calcium Oxide.

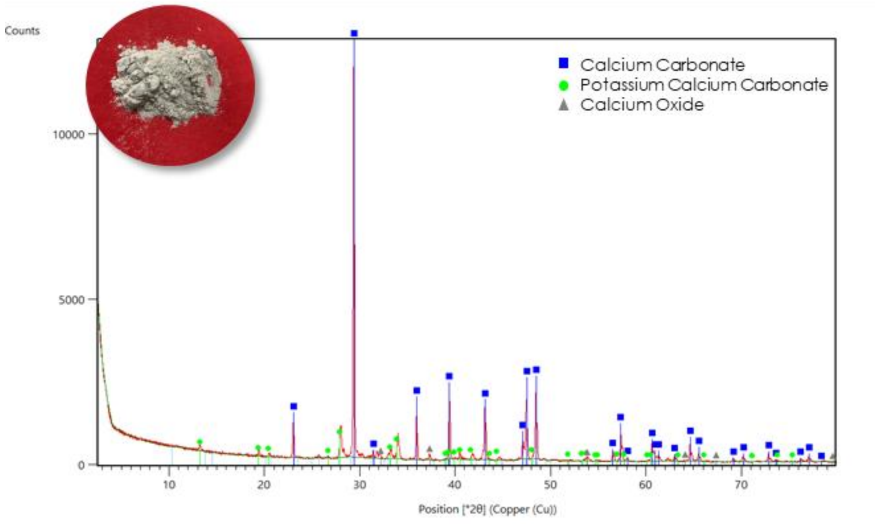

Figure 1. XRD spectrum of Beechwood Ash (ashes picture in the inset)

BWA, Fly Ash (FA) and/or Ground-Granulated Blast-Furnace Slag (GGBFS) were used as raw materials; Na_2SiO_3 solution (R = 3.3) and NaOH solution (10 M) were used as alkaline activators. Different mixtures were prepared (following the diagram in the Figure 2) by replacing FA and/or GGBFS with BWA (10 – 70 wt%).

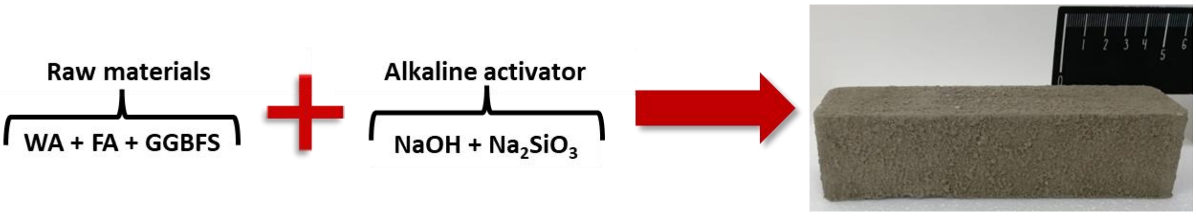

Figure 2. Synthesis procedure

A solid – to – liquid ration of 0.45, alkaline activator silica modulus (SiO_2/Na_2O) of 0.9 were keep constants for all mixtures. Furthermore, the effect of curing temperature (25 and 60 °C), relative humidity (75 and 100%) and curing time (1 to 28 days) was also investigated. Preliminary results in terms of consolidation and mechanical properties confirm the suitability of wood ashes as partial replacement in geopolymer mortars pushing towards a circular and sustainable management of this waste category.

Acknowledgements

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S5-C.5 CIRCULAR SOLUTION FOR SHELL WASTE AS CEMENT SUBSTITUTE IN CONSTRUCTION MATERIALS: STRATEGY FOR THE IMPROVEMENT OF MECHANICAL PERFORMANCE

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Cementitious materials are the most common and effective building materials. While guaranteeing excellent performance, the production has adverse effect on environment in terms of greenhouse gasses emission. This motivation drives scientists and researchers to found sustainable raw materials for its production. This research investigates the use of shell, a waste from food industry, as cement substitute and, it investigates the best treatment and mix design to prepare cementitious mortar. Shell waste was characterized in terms of physical and chemical constitution and then it was grinded until passing at 150 μm, to be used as filler in substitution to cement, at 10, 20 and 30wt%, in mortar with 0.5 water to binder (w/b) ratio. Results were also compared to a reference mortar (0% substitution), with the same w/b ratio.

The fresh state and hardened properties such as density, Elastic dynamic modulus (Ed), compressive strength (Rc), were tested. The mechanical results in terms of Rc showed that the properties were decreased up to 50% when 30% of shell waste filler was used. To mitigate this loss of performance, 3 different approaches were adopted: i) pretreatment of shell waste, ii) reduction of w/b ratio, and, iii) preparation of ternary mixes with blast furnace slag as pozzolanic material and shell waste as partial substitute of cement.

40% of reduction of Rc was reached when a pre-treatment able to remove the organic part of the shell is guarantee. With the reduction of w/b ratio from 0.5 to 0.4, a superplasticizer was added to keep same the workability, and the decrease of Rc was 30% when 30% of binder was substituted. However, when 10% of filler waste was substituted, in case of both approaches negligible reduction of Rc was registered.

The addition of slag in a mixture sand:cement:shell:slag of 3:0.7:0.1:0.2 wt% also permitted a recovery of Rc when compared to 30% filler waste substitute, with a reduction of Rc of about 25% when compared to the reference mortar.

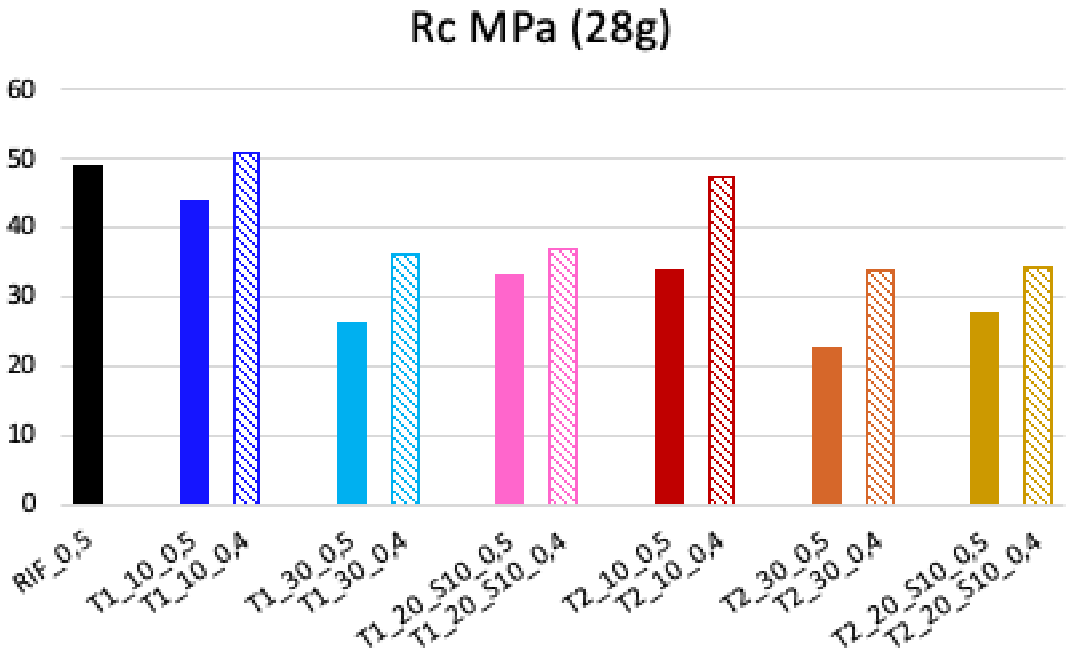

Figure 24 Compressive strength of the different mortar: T1 and T2 represent different pre-treatment of shell waste, and S represents slag.

S5-C.6 VALORIZATION OF A SECONDARY STREAM OF RECYCLED CARBON FIBRES IN CONCRETE APPLICATION: COMPATIBILITY, PERFORMANCE, AND COMPOUNDING OPTIMIZATION

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Downcycling synthetic fibre waste for reinforced concrete in the construction sector can provide mutual benefits for both industries due to not only alleviating the strain on the environment and socio-economic impact but also enhancing the properties of the cementitious material. Incorporating carbon fibres to develop fibre-reinforced concrete (FRC) is an attractive route in enhancing some engineering performance for better applicability of the material, including mechanical strength, post-cracking behaviour, shrinkage mitigation, and thermal resistance. In the framework of eco-sustainable design of construction materials, this work dealt with the viability of engineering cementitious mixtures with scrap carbon fibres (sCF) deriving from an industrial thermal recycling processing of waste carbon-fibre composites. Due to the agglomerate-like structure of the recycled fraction, the main criticality that emerged in the manufacturing stage was to ensure adequate dispersion of the reinforcement in the matrix. Therefore, in this work, a de-agglomeration treatment of the fibres by nanoclay slurry was developed. Nanoclay-based functionalization aimed to ensure a more homogeneous distribution of the reinforcement while providing pozzolanic activity for concrete improving its microstructural characteristics. In the present research, different contents of sCF were implemented (from 0.25 w/w% to 1 w/w%) with respect to the amount of cement binder, studying FRC mix designs with and without nanoceramic treatment. The influence of the reinforcing fibres as well as the compatibilizing effect of nanoclay were investigated by a multi-methodological experimental analysis including, rheological tests, mechanical characterization, and microstructural assessment. The graphical abstract in Figure 1 illustrates the main phases of the research activity proposed in this study.

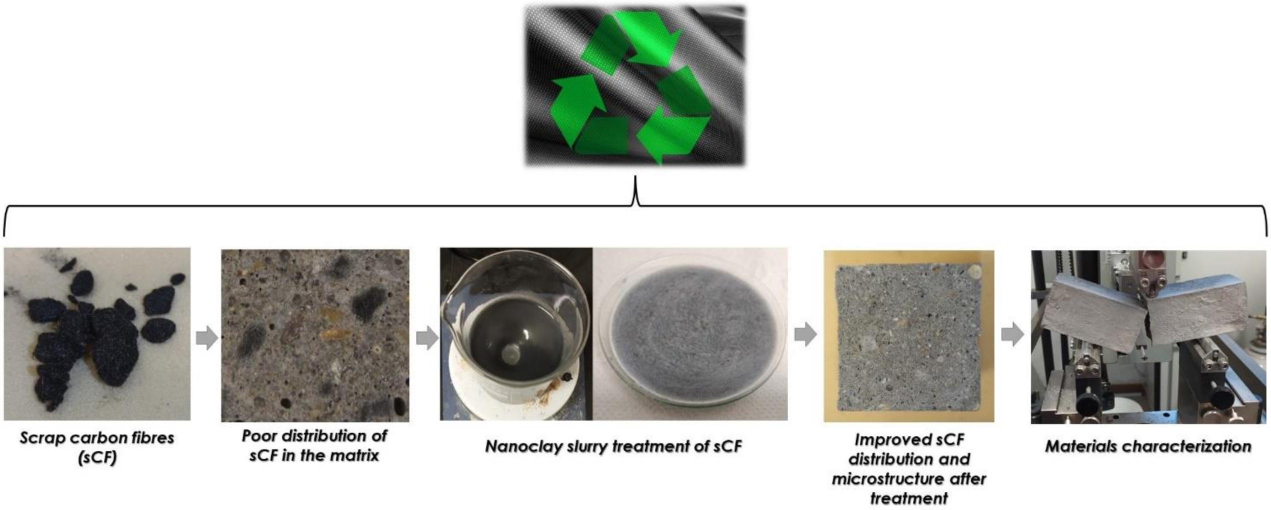

Figure 25. Graphical abstract illustrating the research workflow of the present study.

S5-C.7 EFFECTIVENESS OF SODIUM BICARBONATE TREATMENT ON THE MECHANICAL PERFORMANCES OF SISAL FIBER REINFORCED GEOPOLYMERIC COMPOSITES

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The present work aims to investigate the effectiveness of an eco-friendly and cheap fiber treatment based on sodium bicarbonate ($NaHCO_3$) aqueous solutions in the improvement of the mechanical performances of sisal fiber reinforced geopolymers. All the specimens were made by using metakaolin as a precursor, which was activated using an alkaline solution of KOH 7M and a powder of potassium silicate (K2O• nSiO2). A river sand, with a maximum nominal diameter of 2 mm, was also used as aggregate.

The effect of untreated short sisal fibers (2.5 cm length) as reinforcement was evaluated by performing compression, three-point bending and splitting tensile tests (UNI-EN-1015) on fiber reinforced geopolymer composites. In particular, three fiber weight contents were investigated (i.e., 0.5%, 1%, 2%) showing that the best mechanical performances are obtained for composites reinforced with 2%wt sisal fibers.

Furthermore, sisal fibers were soaked for 5 days at room temperature in sodium bicarbonate aqueous solutions at different weight concentrations (i.e., 2.5%, 5% and 10%). The mechanical characterization of 2%wt sisal fiber reinforced samples evidenced the existence of a growing trend of the composites maximum strength with the weight content of sodium bicarbonate (Figure 2). Furthermore, chemical, and morphological tests were carried out to investigate deeper the efficiency of the sodium bicarbonate treatment. In particular, functional group modifications on fiber surfaces were evaluated by Fourier transform infrared (FTIR) analysis, while the failure surfaces of sisal fiber reinforced composites were observed by scanning electron microscope (SEM). Overall, these tests confirmed that the proposed treatment represents a valid and effective alternative to traditional treatments.

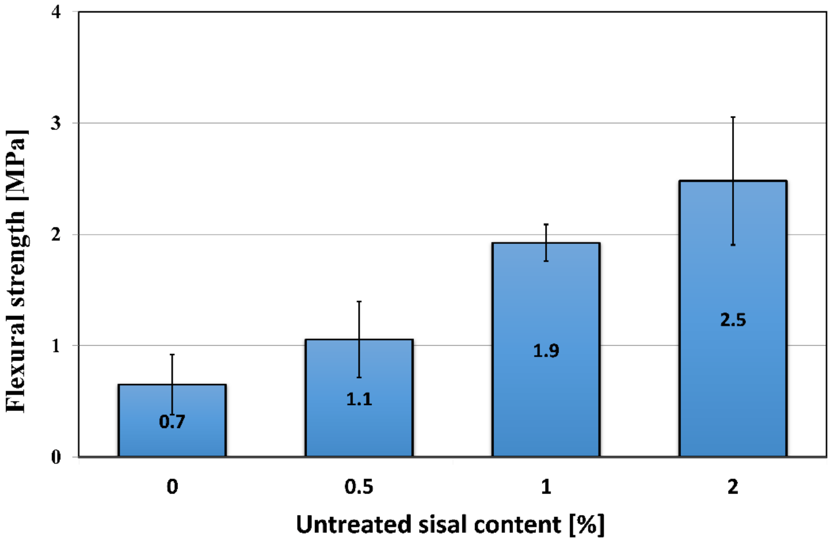

Figure 1. Flexural strength of composites at varying the weight content of untreated sisal fibers.

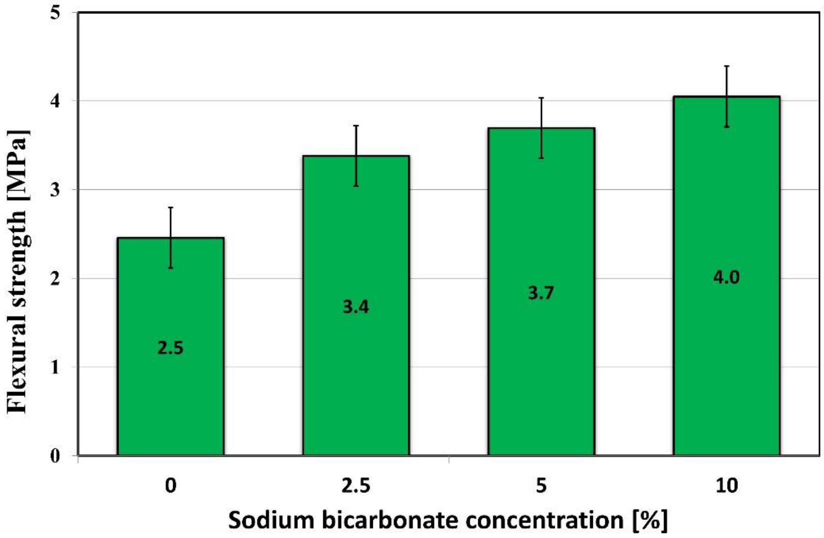

Figure 2. Flexural strength of 2%wt sisal fiber reinforced composites at varying the weight concentration of the sodium bicarbonate solution.

S5-C.8 APPLICATION OF CELLULOSE AND CHITIN NANOSTRUCTURED SPONGES FOR CONTAMINATED SEDIMENTS REMEDIATION

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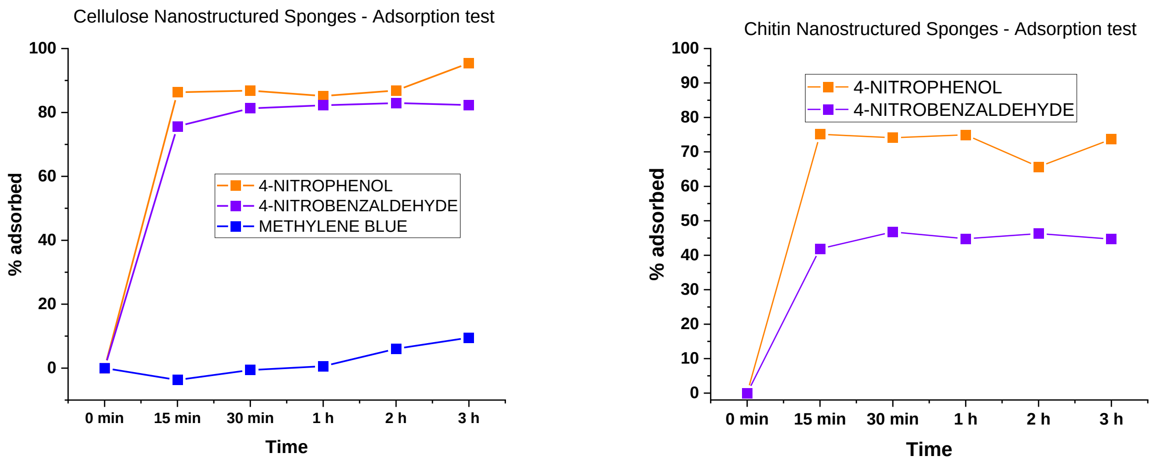
The presence of persistent pollutants in the environment is a serious concern. Recently, nanomaterials are gaining ground in the remediation sector, especially if they have natural substances or waste as precursors. In this context, cellulose and chitin sponges nanostructured meet sustainability goals.

This study's objective is to develop sustainable materials for the remediation of contaminated marine sediments. The materials' synthesis provides: precursor oxidation with 2,2,6,6-tetramethylpiperidinyloxyl (TEMPO), acidification and branching with branched polyethyleneimine (bPEI), and freeze overnight. A Soxhlet extractor with ethanol as solvent was used to obtain an aerogel.

The synthesized materials were characterized by FTIR analyses to observe the chemical structure. Furthermore, materials were observed by SEM to identify the morphological characteristics of each one.

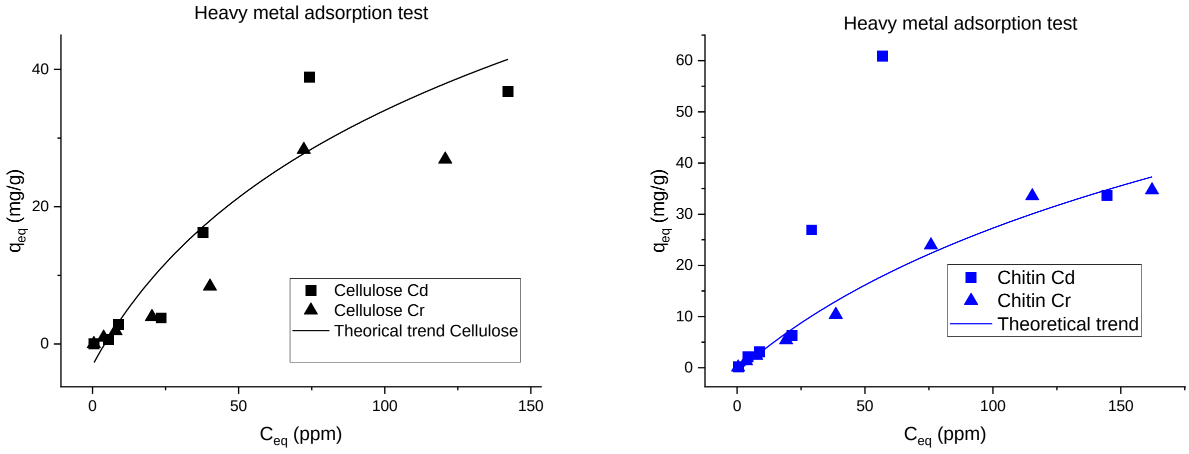
Cellulose and chitin nanostructured sponges are tested for the adsorption of organic and heavy metal. The Figure 1 shows adsorption kinetics of three organic molecule (4-nitrophenol, 4-nitrobenzaldehyde and Methylene blue); instead, the Figure 2 shows adsorption kinetics of two heavy metal (Cadmium and Chromium).

The results suggest that the materials have similar structures and similar morphology. In addition, the adsorption tests have shown promising results. Therefore, this experimental study will significantly contribute to paving the way toward the use of cellulose and chitin nanostructured sponges in the remediation of contaminated sites.



a) b)

Figure 1. Organic compounds adsorption test for cellulose (a) and chitin (b) nanostructured sponges.



a) b)

Figure 2. Heavy metal adsorption isotherm for cellulose (a) and chitin (b) nanostructured sponges.

S5-C.9 PHOSPHATE TREATMENTS IMPROVE THE PHYSICAL MECHANICAL PROPERTIES OF CEMENTITIOUS SUBSTRATES

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In recent years, phosphate treatments have been investigated to preserve marble, limestone and lime mortars used in cultural heritage. The idea is to form new calcium phosphates (CaP) as the reaction product between the substrate and an aqueous solution of a phosphate precursor. Thanks to the low water solubility of CaPs, the substrate can be protected from dissolution in rain. Inspired from this, here we investigated the possibility of improving the surface properties of cementitious substrates by phosphate treatments, to extend the service life of new buildings and preserve historic concrete buildings.

Three precursors were considered, i.e. diammonium hydrogen phosphate (DAP), dipotassium hydrogen phosphate (DPP) and disodium hydrogen phosphate (DSP). First, the new phases expected when Portland cement is treated with aqueous solutions of the three precursors were predicted by thermodynamic modeling, to discard treatments that lead to undesired soluble phases. Then, corresponding experimental tests were carried out to determine the composition (XRD) and the morphology (SEM-EDS) of the new CaPs (Fig. 1). Improvements in resistance to abrasion, indentation and staining were assessed, together with the permanence of the beneficial effects after prolonged immersion in water. For the most promising precursor (DPP), the influence of its concentration (from 0.1 M to 2 M) and the cement age when the treatment is applied (from 1 d to 28 d) was investigated.

The results of the study indicate that all the three precursors provide substantial improvements in physical-mechanical properties of cementitious substrates. In the most promising conditions (7 d of cement curing before treatment with 1 M DPP and 1 mM $CaCl_2$ for 24 h), improvements in resistance to abrasion and staining up to ~80% were reached. To ascertain that the beneficial properties are retained after repeated wetting-drying cycles and no undesired efflorescence is formed, long-term tests are currently in progress.

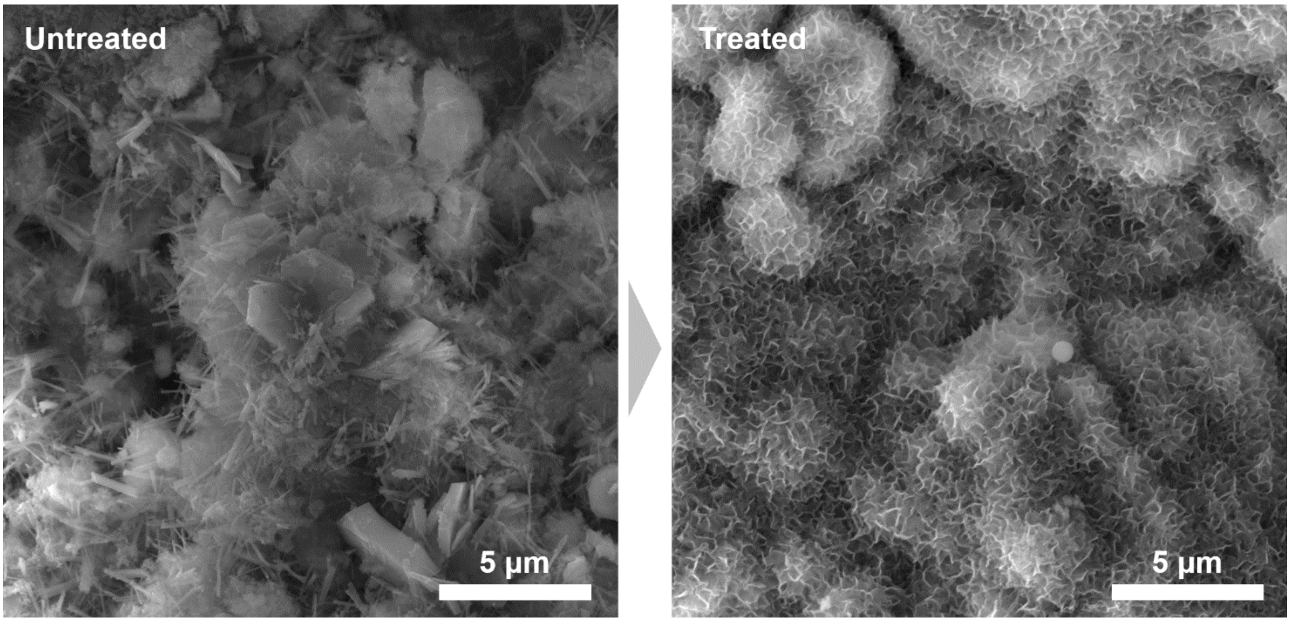

Figure 26. SEM image of a cement powder before (left) and after (right) treatment with a DAP solution.

S5-C.10 A FUZZY MODEL FOR STUDYING DEGRADATION KINETICS OF GENNA MARIA NURAGIC BUILDING

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The Nuragic Sardinian civilization (ca. 1600–1020 BC) represents one of the most important Bronze Age cultures of the Western Mediterranean area. Today, the built heritage of this period can be found in the megalithic tombs, temples, villages and Nuraghi. These, with an estimated number ranging between 7.000 and 10.000, represent the distinctive prehistorical buildings and constitute a prominent feature of Sardinia's landscape.

At present, a large number of Nuraghi are exposed to a series of pathogenic factors which substantially influence degradation kinetics (DGRK). Currently, only effective conservation and protection strategies could slow down the degradation process.

The behaviour of structures and materials over time, the incompleteness of materials characterisation, the complexity of relationships and the randomness of future environmental and cultural conditions are all remarkable obstacles to the preservation of these structures.

However, according to holistic approaches, some conceptual tools are now available to develop predictive models on the complex 'life' of an ancient building.

In the present work, a new fuzzy model has been formalised in order to estimate DGRK of the Genna Maria site. It considers environmental parameters, properties of materials, weathering phenomena, anthropic action such as numerical and linguistic variables derived from instrumental measurements as well as direct observations.

The modelling procedure has been carried out by considering the variables month per month. The results highlight that DGRK accelerates and slows down in different parts of the year. For April, May and December, model calculated a relevant degradation risk, while a lower possibility to have a significant decay has been recorded for August, September and October.

Overall, the proposed model represents a tool that can usefully and explicitly encode uncertain knowledge and empirical experience and thus provide support for future decisions on site conservation.

S5-C.11 DEVELOPMENT AND PERFORMANCE OF A SUSTAINABLE ANTIMICROBIAL NANOCOMPOSITE TREATMENT FOR POROUS STONES PROTECTION

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Conservation of building materials is one of the greatest challenges of contemporary research. Among the many natural building materials used throughout history, tuff stone is one of the most vulnerable because of its porous nature, which promotes water penetration into its structure and microorganism growth on its surface. Therefore, reducing the tuff hydrophilicity is an essential tool to counteract the chemical-physical and biological phenomena that cause its deterioration.

To obtain this goal, many water-repellent polymer protection systems were developed, mostly based on silicone, siloxane and acrylic resins and fluorinated polymers. All of them are effective hydrophobizing agents since they strongly reduce the water permeability of the substrates, but often show limited performances when additional requirements should be satisfied, e.g., surface consolidation, transparency, sustainability, or antimicrobial activity. Hence, new high performing, multifunctional and durable treatments are still required.

In this work, we report the results of a preliminary experimental study on an innovative, multifunctional surface treatment for tuff protection, able to ensure at the same time water-repellency, consolidating function and long-lasting antimicrobial activity, in a water- based environmentally friendly formulation. For this purpose, a commercial protective/consolidant oligosiloxanic resin was used as the base for designing new treatment systems incorporating alternatively a wide range biocide, a natural layered nanoclay and their combination. In the latter case, part of the biocide was intercalated into the nanoclay interlayer, which acts as carrier and offers a potential for a delayed release of the biocide. The effectiveness of the treatments was evaluated by applying them on tuff substrates that were then submitted to water capillary absorption tests, static contact angle measurement, abrasion tests, colorimetric analysis and preliminary microbiological tests.

S5-C.12 SMART PROPERTIES OF ALKALI-ACTIVATED SLAG-BASED MORTARS WITH TITANIUM DIOXIDE

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The use of titanium dioxide nanoparticles in cement-based materials is a well-known solution to obtain self-cleaning capability of mortars, improving the quality of urban surfaces and road tunnels. However, the effect of such additions in low-carbon binders has not yet been studied. The purpose of this work is to investigate the influence of the addition of TiO_2 *nanoparticles on the rheological, physico-mechanical and photocatalytic properties of different alkali-activated slag-based mortars. The self-cleaning capability of* TiO_2*-containing composites were evaluated by means of accelerated dye degradation tests and 24-months outdoor exposure tests in comparison with traditional mortars based on white Portland cement.*

Experimental results show that the variation of the rheological properties of mixtures due to the addition of TiO_2 *is negligible while it can be noted a general reduction on the setting times by increasing the amount of the titanium dioxide content. The use of* TiO_2 *seems to have a minor effect on the elasto-mechanical properties of slag-based mortars at early and long ages. Only alkali-activated materials with low alkali content evidence an increase in compressive strength from 9 to 14% with respect to the same mortars manufactured without nanoparticles.*

The beneficial effect of TiO_2 *addition in self-cleaning capability is evident both in cementitious and alkali-activated mortars under accelerated tests. In particular, regardless of UV light exposure time and dye nature, the color variation increases almost linearly by increasing the nanoparticles content as it can be seen in Figure 1. Finally, the self-cleaning ability has been evaluated by measuring the color variation during the exposure of mortar samples to the industrial environment for 2 years. Results evidenced the efficiency of the titanium dioxide on the reduction of the color variation promoted by the polluted environment.*

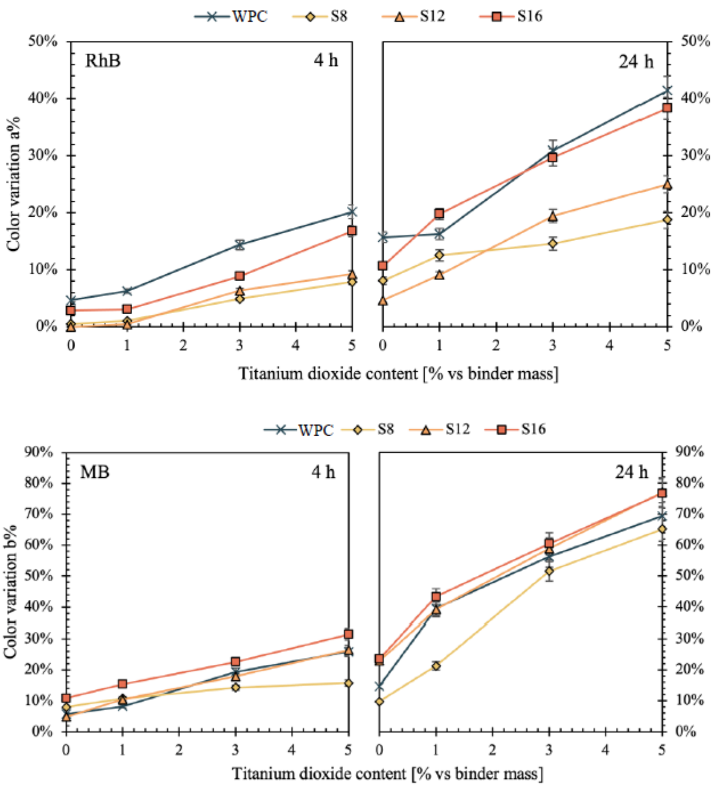

Figure 1. Color variations of samples sprayed with Rhodamine B (RhB, on the top) and Methylen Blue (MB, on the bottom) as a function of titanium dioxide content after 4h (left) and 24h (right). WPC is the reference cement-based mortar, letter S refers to the alkali-activated slag-based materials and it is followed by a number that indicates the activator-to-slag ratio.

S5-C.13 LOW-CARBON PERVIOUS CONCRETE MADE WITH ALKALI-ACTIVATED BINDER AND RECYCLED SAND AND GRAVEL: THE ROLE OF THE AGGREGATE SIZE

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The uncontrolled urban development of the last century caused a high land consumption and strong non-renewable natural raw materials utilization. In order to solve the problems generated by soil sealing, caused by a strong urbanization, the building sector has developed a pervious concrete (also called "no-fines concrete") manufactured with Portland cement and natural aggregates. Although this mixture mitigates the effects of soil sealing, the production of a Portland-based pervious concrete has a strong environmental impact.

The purpose of this research is to investigate an alkali activated slag-based pervious concrete (AASPC) manufactured with tunnel muck (TM) as recycled aggregate instead of natural sand and gravel and to evaluate the relationship between aggregate size and physico-mechanical properties of no-fines concrete.

Six different single-sized recycled aggregates from tunneling works (drilling and blasting technique) were used to produce six different AAS pervious concretes that were characterized in terms of compressive strength, porosity and water permeability under constant and variable flow.

Experimental results evidenced that the average size of aggregates strongly influences the open and total porosity of the materials, thus determining very different compressive strengths (from about 6 MPa for concrete with 16-22 mm gravel to 20 MPa for concrete made with 1-2 mm sand) and water permeability (Figure 1a). Finally, the environmental impact of these mixtures (energy requirements, CO_2 emissions and natural raw material consumption) is strongly reduced in comparison to traditional Portland-based no-fines concrete at equal strength class (Figure 1b).

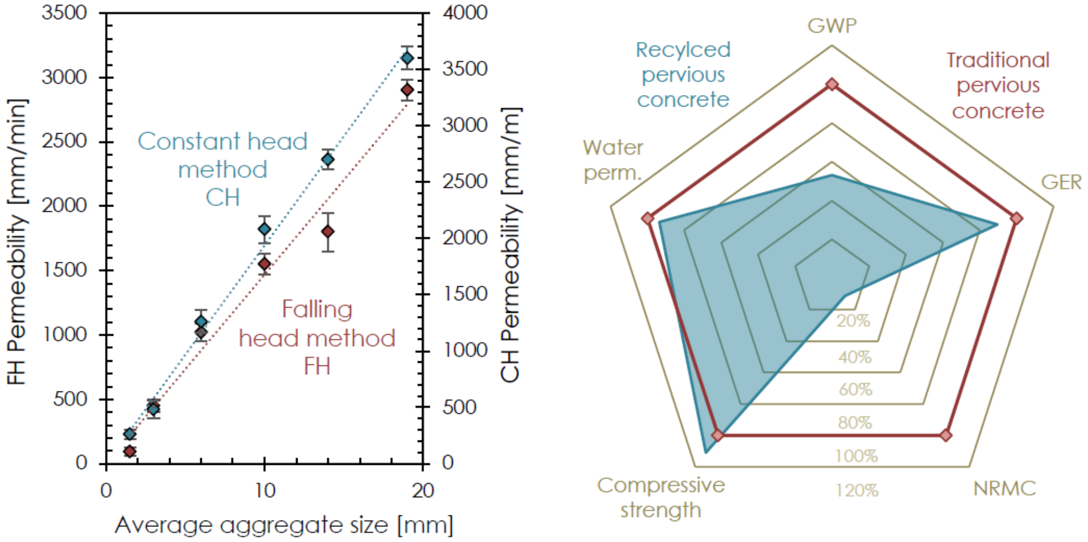

Figure 1a) Water permeability coefficient of different pervious concretes as a function of aggregate size (left). b) Environmental parameters (GWP: Global Warming Potential, GER: Gross Energy Requirement, NRMC: Natural Raw Materials Consumption) and main performance (water permeability and compressive strength) of recycled pervious concrete normalized with respect to a traditional Portland cement-based pervious concrete (right).

Codice	Sessione	Titolo	Corresponding Author
P1	1	COMPARISON OF DIFFERENT ELECTROCHEMICAL SURFACE TREATMENTS ON ALUMINUM ALLOY 7075 TO ENHANCE THE ADHESION BETWEEN METAL SUBSTRATES AND THERMOPLASTIC BASED COMPOSITES	Dionisio Badagliacco
P2	1	ANISOTROPIC HEAT TRANSFER IN BRICK-AND-MORTAR GNP/EPOXY NANOCOMPOSITES	Fabrizia Cilento
P3	1	EFFECT OF FILLER CONTENT ON VISCOELASTIC BEHAVIOUR OF EPOXY VITRIMER NANOCOMPOSITES	Alfonso Martone
P4	1	TUNABLE PECTIN/COLLAGEN-BASED BIOINKS FOR 3D MODELS OF NEURAL TISSUES	Gloria Romero
P5	1	AN INNOVATIVE 3D PRINTED BIOCATALYZER FOR THE BEVERAGES TREATMENT	Erika Lunetta
P6	1	FILTRATION AND ADSORPTIVE PROPERTIES OF BIODEGRADABLE ELECTROSPUN POLYACTIC ACID AND CELLULOSE ACETATE FILTERS WITH BIOMASS-DERIVED ACTIVATED CARBON	Natalia Czerwinska
P7	1	NOVEL PBI/GO COMPOSITES FOR POTENTIAL USE IN PROTON EXCHANGE MEMBRANE ELECTROCHEMICAL DEVICES	Mattero Di Virgilio
P8	1	EVALUATION OF PERFORMANCES OF FLAX FIBER REINFORCED EPOXY COMPOSITES DURING SALT SPRAY FOG-DRY AGING CYCLES	Vincenzo Fiore
P9	1	DESIGN AND OPTIMIZATION OF INNOVATIVE BIOMEDICAL IMPLANTS BY ADDITIVE MANUFACTURING TECHNOLOGIES	Valerio Papa
P10	1	DEVELOPMENT AND CHARACTERIZATION OF CHITOSAN HYBRID COATINGS BY SOL-GEL METHOD	Rosa Taurino
P11	1	SYNTHESIS AND CHARACTERISATION OF FLUORINE-FREE MIL-100(Fe) FOR HYDROGEN, CARBON DIOXIDE AND WATER VAPOR STORAGE	Muhtadi Idrees
P12	1	DEVELOPMENT OF ECOSUSTAINABLE ELECTROSPUN FIBERS FOR THE ABSORPTION OF HEAVY METALS	Marianna Messina
P13	1	THE THERMOMECHANICAL BEHAVIOR OF BIO-BASED EPOXY RESIN AND DAYLIGHT-CURABLE BLENDS FOR 3D PRINTING THROUGH THE USE OF LIQUID CRYSTAL DISPLAY (LCD) TECHNOLOGY	Claudio Tosto
P14	1	REFRACTIVE INDEX MODELLING OF TRANSPARENT 3D PRINTABLE PHOTOCURABLE RESINS FOR MICRO-OPTOFLUIDIC DEVICES: A REGRESSION APPROACH	Lorena Saitta
P15	2	NICKEL EXSOLVED PEROVSKITE CATALYST FOR ETHANOL AUTOTHERMAL REFORMING	Patrizia Frontera
P16	2	3D PRINTING OF SELF-MONITORING PIEZORESISTIVE HIGH-PERFORMANCE NANOCOMPOSITES	Lorenzo Paleari
P17	2	BIOPOLYMERS AS MATERIALS FOR THE REALIZATION OF TRANSIENT MECHANOELECTRICAL DEFORMATION SENSORS	Antonino Pollicino
P18	2	HYBRID COATINGS IMPROVE THE OXYGEN BARRIER OF POLYMERIC SUBSTRATES	Filippo Ghisoni
P19	3	THE FUSED DEPOSTION MODELLING (FDM) AND THE RHEOLOGICAL PROPERTIES OF POLYLACTIDE (PLA)-BASED FILAMENTS	Antonella Patti
P20	3	2.5 SURFACES VIA FOCUSED ION BEAM PATTERNING AND NANOINDENTATION IMPRINT	Daniele De Felicis
P21	3	DEVELOPMENT AND VALIDATION OF A NEAR-FIELD DIRECT-WRITING ELECTROSPINNING SYSTEM FOR TISSUE ENGINEERING APPLICATIONS	Laura Armenio
P22	3	AUSTENITE-MARTENSITE SURFACE TRANSFORMATION ON AISI 316L INDUCED BY LOW-ENERGY HIGH-CURRENT ELECTRON BEAM TREATMENT	Andrea Lucchini Huspek
P23	4	3D PRINTED GREEN COMPOSITES BASED ON POLYMERIC MATRICES AND ANCHOVY FISHBONE WASTES	Roberto Scaffaro
P24	4	EVALUATION OF MICRO FIBRILLATED CELLULOSE (MFC)/POLY (LACTIC ACID) (PLA) BIOCOMPOSITES FOR FILM BLOWING PROCESS	Luigi Botta
P25	4	SUSTAINABLE CONCRETE FROM RECYCLED ORGANIC WASTE MATERIALS AS AGGREGATE REPLACEMENT	Nicolas Bertoldo
P26	4	UPCYCLING OF POLYESTER FIBER AND GARMENT-TEXTILE WASTE	Claudia Marano
P27	4	MONOPOLYMER BLENDS OF VIRGIN AND RECYCLED BIODEGRADABLE POLYMERS TO PROMOTE THE DECARBONIZATION OF PLASTICS.	Francesco Paolo La Mantia
P28	4	NEW SUSTAINABLE COMPOSITES BY USING BIO-BASED MATERIALS	Erika Alessia Di Liberto
P29	4	GREENING EARTHEN PLASTERS: A STUDY ON BIO-SOURCED POLYMERS AS STABILIZERS	Giorgio Pia
P30	4	INDUSTRIAL APPLICATIONS OF BIODEGRADABLE POLYMERS AND PAPER FOR THE PACKAGING SECTOR	Marco Morreale
P31	4	RECYCLING OF COMPACT DISCS: A CLEAN SOLUTION	Maria Chiara Mistretta
P32	4	BIODEGRADATION BEHAVIOR OF POLY (LACTIC ACID) BASED COMPOSITES FILLED WITH SHORT NATURAL FIBERS	Libera Vitiello
P33	4	EFFECT OF GNP CONTENT ON THE PHOTOOXIDATIVE BEHAVIOR OF PP-BASED NANOCOMPOSITES	Manuela Ceraulo
P34	5-C	MICROSTRUCTURE AND SUSTAINABILITY OF CONCRETE CONTAINING RECLAIMED ASPHALT PAVEMENT AS NATURAL AGGREGATE REPLACEMENT	Maria Chiara Bignozzi
P35	5-C	MANUFACTURING AND DURABILITY OF ALKALI-ACTIVATED COMPOSITES WITH BAMBOO FIBRES	Stefania Manzi
P36	5-C	EFFECT OF COATING ON CHLORIDE PENETRATION IN CONCRETE	Britta Schallock
P37	5-C	STUDY OF THE PASSIVATION OF STEEL IN CONCRETE MADE WITH RAP AGGREGATE	Andrea Filippi
P38	5-C	EVALUATION OF ALKALI-SILICA REACTIVITY IN EXISTING RC TRANSPORT INFRASTRUCTURES	Barbara Liguori

P1 COMPARISON OF DIFFERENT ELECTROCHEMICAL SURFACE TREATMENTS ON ALUMINUM ALLOY 7075 TO ENHANCE THE ADHESION BETWEEN METAL SUBSTRATES AND THERMOPLASTIC BASED COMPOSITES

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Nowadays, one challenge in aerospace industry is to find an anodizing process able to substitute chromic acid anodizing, due to its carcinogenic and environmental threats. This process allows to achieve an optimal best corrosion resistance of the metal substrate as well as the full mechanical strength of the adhesive joint. In this study, anodization in tartaric-sulfuric acid (TSA) and sulfuric-phosphoric acid (PSA) solutions (usually employed separately by AIRBUS) were mixed to create a two-step anodizing process that gives both corrosion protection and mechanical resistance to the substrate. Specifically, fiber metal laminates (FMLs) were made from anodized aluminum alloy 7075 and carbon fabric reinforced polyether ether ketone (PEEK) composite substrates. The metal substrates were first anodized in TSA for 10 min at 14V and 37°C and then in PSA for 10 min at 18V and 28°C. In this way, it was possible to obtain a anodic porous layer on the surface having pores of different diameters through the thickness. This shape was useful in improving both corrosion protection and mechanical resistance of the FML. For comparison, TSA and PSA anodizing treatments were performed, as well as a mechanical abrasion treatment with sandpapers (MT). Finally, a post immersion treatment of TSA samples in a NaOH 0.1M solution (TSA-NaOH) was carried out to evaluate the effect of a pore-widening step on the anodic layer with. Electrochemical impedance spectroscopy in a 3.5%wt of NaCl solution were performed to study the corrosion resistance of treated aluminum substrates. A mechanical characterization of FMLs was conducted by evaluating their fracture toughness and interlaminar shear strength through double cantilever beam (ASTM D5528) and short beam shear (ISO 14130) tests, respectively. Overall, the proposed anodizing process allowed to obtain FMLs having the mechanical resistance of PSA samples and about the corrosion resistance of TSA samples, thus evidencing its effectiveness as more sustainable alternative to current harmful treatments.

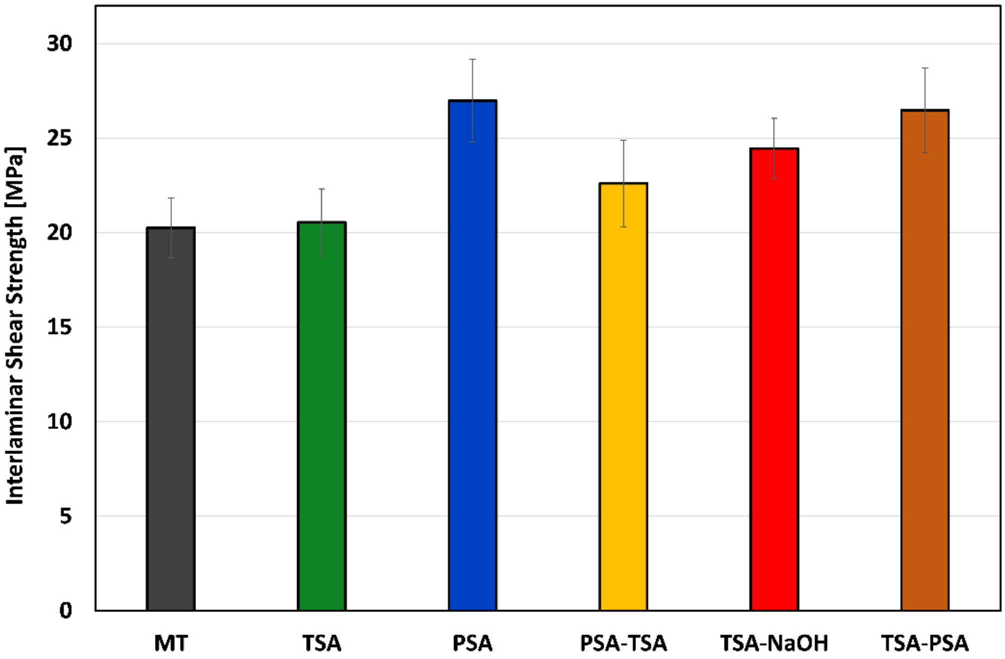

Figure 1. Interlaminar shear strength by short beam shear test varying the anodization process on aluminum alloy substrate of fiber metal laminates.

P2 ANISOTROPIC HEAT TRANSFER IN BRICK-AND-MORTAR GNP/EPOXY NANOCOMPOSITES

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Lightweight multifunctional materials are increasingly in demand in the automotive and aerospace industries. Biomimetic brick and mortar (B/M) thin films could fill the gap thanks to their outstanding multifunctional properties. The well-aligned nanoarchitecture combined with the high loading of lamellar nanoparticles (up to 90 vol%) allows the production of nanocomposites whose properties are similar to those of the brick. The use of highly conductive nanofiller, such as graphene and 2D analogues, should enable good mechanical performances combined with remarkable thermal and electrical conductivities and gas/liquid barrier properties. In this work, the authors fabricated graphite nanoplatelets (GNPs)/epoxy thin films (70 μm) with a brick-and-mortar (BM) architecture, following a top-down approach based on a spray deposition process. Samples with different filler content (from 60 to 90 vol%) have been produced and the thermal behaviour of the films has been investigated. Both in-plane and cross-plane thermal diffusivities, in the temperature range between 25°C and 200°C, have been assessed, by using light flash analysis (LFA). Thermal conductivities vis-a-vis filler content have been computed. An increase of the in-plane conductivity with filler content up to 140 W/mK at 90 vol% of GNPs is found. Whereas a decrease in the cross-plane conductivity is shown with filler content.

The inner architecture of the materials ensures a high anisotropy of thermal conductivity, which increases with filler content up to 180 at 25°C for films loaded with 90 vol% of GNPs, making these materials optimal candidates for thermal management as heat spreaders.

P3 VISCOELASTIC CHARACTERIZATION OF REFORMABLE EPOXY VITRIMERS COMPOSITES

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Increasing environmental awareness and industrial competitiveness encourage the use and development of repairable and recyclable structural materials to reduce polymer waste and extend their service life. Recently, the development of thermoreversible cross-linked networks has been introduced as a viable alternative to produce composite components that can be recycled and reformed as thermoplastic materials.

Introducing a thermoreversible covalent bond in the cross-linked backbone allows the topological reshuffling of polymeric networks and makes it possible to rework and reform the cross-linked material. One possible mechanism that enables the vitrimeric behaviour in epoxy resins is based on transesterification exchange reactions between esters and beta-hydroxyls formed by reacting epoxy precursors with suitable acids/anhydrides. Incorporating a catalyst activates the transesterification reaction and induces topological variations, stress relaxation and flow in the cross-linked networks, even though the total number of cross-links does not change. During transesterification, the network's connectivity is altered via exchange reactions, inducing stress relaxation and plastic flow at elevated temperatures without depolymerisation.

In the present work, a commercial epoxy system (araldite® LY 3508 and aradur® 917-1 by Huntsman Corporation) suitable for CFRP manufacturing was selected for its modification as a vitrimer. Furthermore, $ZnAc_2$ was selected as a transesterification catalyst, and two formulations at different ratio between epoxy precursors and curing agent have been prepared and modified by adding the catalyst.

Vitrimer carbon fiber reinforced composites were produced using hand lay-up and vacuum consolidation. The thermal properties of vitrimeric matrix have been investigated by DSC and DMA tests. The thermal reshaping capability of vitrimer-CFRP has been investigated by reprocessing CFRP coupons.

P4 TUNABLE PECTIN/COLLAGEN-BASED BIOINKS FOR 3D MODELS OF NEURAL TISSUES

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Due to the complexity of the central nervous system and the various pathologies involving this area, many studies are focused on the development of new models to simulate the mechanical, chemical, and physical properties of the nervous tissue. New materials are being explored to develop a suitable hydrogel as a bioink able to embed cells and with the same viscoelastic properties as target tissues, taking advantage of blending, a common choice to create composite materials with the required properties, and 3D printing technology to create complex structures.

In this work (funded by Fondazione Cariplo, G.A. 2019-4615. DOI: 10.3389/fbioe.2022.1032542), pectin, an anionic polysaccharide from plant cells, was chosen as a support material due to its capability of crosslinking with $CaCO_3$ by internal gelation to get homogeneous networks. Pectin also presents several bioactive and conductive properties that could be applied to neural tissue engineering. Due to the absence of cell-adhesion sites in pectin, collagen was added to the pectin-based gel. Moreover, this study introduced a new 3D printing approach in which pH is fundamental to determine the viscoelastic properties of the bioink and printing parameters, giving predictable information about the crosslinking speed, printing time, and the final printed structure. Finally, different models of neural cells were embedded in the bioink, obtaining a scaffold that can maintain cell viability and proliferation, without additives or post-printing treatments (Figure 1).

Figure 27. Pectin/collagen-based printed scaffold with cells.

The control of pH over viscoelastic properties and printing parameters makes this new bioink an ideal candidate for clinical applications, such as filling up defects by tuning the crosslinking kinetics. Furthermore, our work opens the doors to new cell delivery therapies.

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P5 AN INNOVATIVE 3D PRINTED BIOCATALYZER FOR THE BEVERAGES TREATMENT

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Beverages, such as fruit juices and wine, contain suspended particles that influence their turbidity and flavor [1]. Hence, they must be clarified, preserving their sensory characteristics. Traditional methods, based on clarifying agents (e.g., gelatin, bentonite), remove the molecules responsible for turbidity through physical processes as filtration or centrifugation [2]. However, clarification also removes some essential components of the color and aroma of beverages. A promising alternative involves enzymes, which promote the hydrolysis of the haze precursors, improving and maintaining the qualitative characteristics of the beverages during bottle storage [3]. Nevertheless, the instable nature of the free enzymes under certain conditions (e.g., pH, temperature, natural inhibitors in food matrices) and their disposability are the main drawbacks [4]. To overcome these criticisms, the enzymes can be immobilized on appropriate solid supports [5], commonly beads. The treatment occurs in fluidized-bed reactors (FBR), which avoid the decantation of the immobilized enzymes in the reactor bottom, and aid the mass transfer and the free circulation of the particles [6].

In this work, supports with innovative geometry and higher exposed surface area were proposed as promising alternatives to improve the continuous enzymatic treatment. They consisted in 3D-printed polylactic acid, submitted to alkaline hydrolysis and chitosan coating to facilitate covalent immobilization of the selected enzyme through the functionalization with natural crosslinkers. After optimization of the printing parameters, the thermal and mechanical properties of the supports were investigated. Scanning electron microscopy and contact angle measurements were performed to evaluate the efficacy of the chitosan deposition. The catalytic activities of free and immobilized enzymes were compared through a kinetic assay. The turbidity of the treated beverage was tested after experiments in FBR.

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P6 FILTRATION AND ADSORPTIVE PROPERTIES OF BIODEGRADABLE ELECTROSPUN POLYACTIC ACID AND CELLULOSE ACETATE FILTERS WITH BIOMASS-DERIVED ACTIVATED CARBON

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Air filters are crucial components of a building ventilation system and kitchen hoods that contribute to improving indoor air quality. Activated carbon, thanks to its high specific surface area trap odor particles (Volatile Organic Compounds) and particulate matter (PM2 and PM10) produced during cooking.

Electrospinning, which is a very simple and low-cost method of synthesis of nanofibers, guarantees excellent filtering performance of the obtained membranes. Compared with conventional air filter media like glass fibers and melt-blown fibers, the electrospinning membranes due to the smaller pores are more efficient for capture various pollutants.

In this paper, eco-friendly PLA (polylactic acid) and Cellulose acetate (CA) with activated carbon (AC) sandwich-like filters were prepared via electrospinning to obtain a high-quality factor (QF) fibrous mat for aerosol particle filtration and VOC adsorption. For this purpose, special attention was paid to the biodegradable and sustainable materials used to produce fine nanofibers. Two different methods of functionalization of the fibers with AC have been used. These methods are air spraying and electro-spraying. Several configurations of the final membranes have been investigated. Various process parameters such as spinning concentration, activated carbon loading, spinning volume of the membranes, voltage and flow rate were tested in terms of air filtration performance and fiber morphology. Physio-chemical properties and morphology of obtained filters were characterized by TG, SEM, FTIR, N2 adsorption-desorption isotherm analysis. Filtering efficiency and adsorption properties were evaluated in real-scale room by measuring the filter penetration of new-synthesized and commercial filters, against neutralized aerosol particles (2% NaCl) and VOC (Methyl Ethyl Ketone). Regeneration methods were studied. Our results indicate that proposed hybrid membranes, would be promising materials for highly efficient and sustainable air filters for home appliance systems.

P7 NOVEL PBI/GO COMPOSITES FOR POTENTIAL USE IN PROTON EXCHANGE MEMBRANE ELECTROCHEMICAL DEVICES

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The urgency of developing innovative electrolytes for proton exchange membrane electrochemical devices, able to outperform Nafion by working at temperatures between 80 and 120 °C, is raising. This work proposes the design of a simple and reproducible method to combine polybenzimidazole (PBI) and graphene oxide (GO) in composite membranes. Five PBI:GO (X:Y) mass ratios are studied, corresponding to GO mass contents one order of magnitude higher than those typically reported in literature. The main aim is to determine if the properties of PBI and GO can be successfully blended in the produced self-assembled membranes.

The preparation of the PBI/GO composite membranes (Figure 1) relies on the magnetic mixing (1000 rpm, 1 hour, room temperature) of 2 wt% PBI-in-dimethyl sulfoxide (DMSO) and 1 wt% GO-in-DMSO solutions, up to the obtainment of homogenous slurries. The slurries are then casted onto Petri dishes and oven-dried at 90 °C for a minimum of 3 hours. The self-assembled membranes are recovered as intact products by simple detachment from the support.

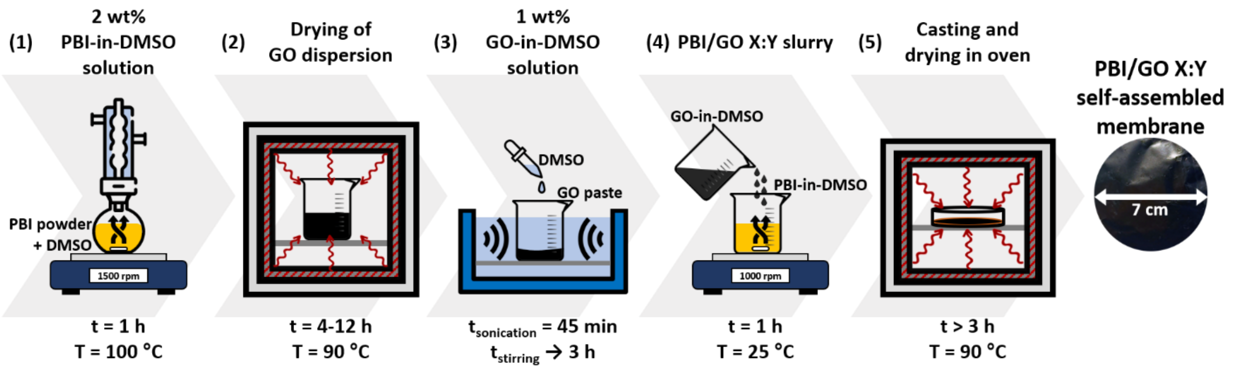

Figure 28. Preparation procedure of the PBI/GO X:Y composite membranes.

The designed preparation procedure is regarded as reliable due to the homogeneity of all the samples, likely related to an adequate reciprocal dispersion of the constituents, as indicated by SEM inspection. XRD patterns suggest an alternated stacked framework via mutual π-π interactions between the benzimidazole rings of PBI and the aromatic domains of GO. The presence of PBI enhances the thermal stability of the composites with respect to pure GO below 130 °C. Higher tensile strengths and Young's moduli are recorded for the composites with respect to pure PBI, at the cost of lower maximum strains. Higher IEC values *than virgin GO and Nafion are measured at larger GO mass contents. Proton conductivities extrapolated from EIS tests are promising, especially at 100 °C, thanks to the positive influence of the hydrophilic oxygenated functionalities of GO.*

P8 EVALUATION OF PERFORMANCES OF FLAX FIBER REINFORCED EPOXY COMPOSITES DURING SALT SPRAY FOG-DRY AGING CYCLES

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In the last decades, natural fiber reinforced composites (NFRCs) have attracted ever growing interest since they represent a promising solution to combine satisfying mechanical performances along with environmental sustainability. However, some drawbacks related to heterogeneity of natural fibers, their weak interfacial adhesion with polymeric matrices and their significant moisture absorption, limit the widespread diffusion of these kind of materials mainly for applications in wet or humid environments. In such a context, the aim of this work is to evaluate how three alternated wet-dry aging cycles can alter the physical and mechanical performances of flax fiber reinforced epoxy composites (FFRC$_s$), manufactured via vacuum assisted resin infusion process. In particular, the maximum aging time was 12 weeks since each wet/dry cycle has a duration of 4 weeks: i.e., 10 days of wetting followed by 18 days of drying. In more detail, the wet phase was performed by exposing the composite laminates to a salt-spay fog (5wt% NaCl solution) at 35°C and 100% R.H. using a corrosion chamber (Ascott CC1000iP) according to ASTM B 117 standard, while the dry phase was carried out by keeping them under controlled conditions (i.e., 22°C and 50% R.H). Three-point bending tests were carried out for each wet-dry cycle after 0, 5, 10 and 18 days of drying through a universal testing machine (Zwick-Roell Z005), according to ASTM D790 standard.

Physical properties such as water absorption capacity, density and voids content were evaluated by monitoring the weight of the composite at regular intervals of time during both phases. The water uptake during the first wet-dry cycle of FFRC materials is reported in Figure 1. On the other hand, Figure 2 evidences the flexural strength recovery shown by the investigated composites during the first dry phase.

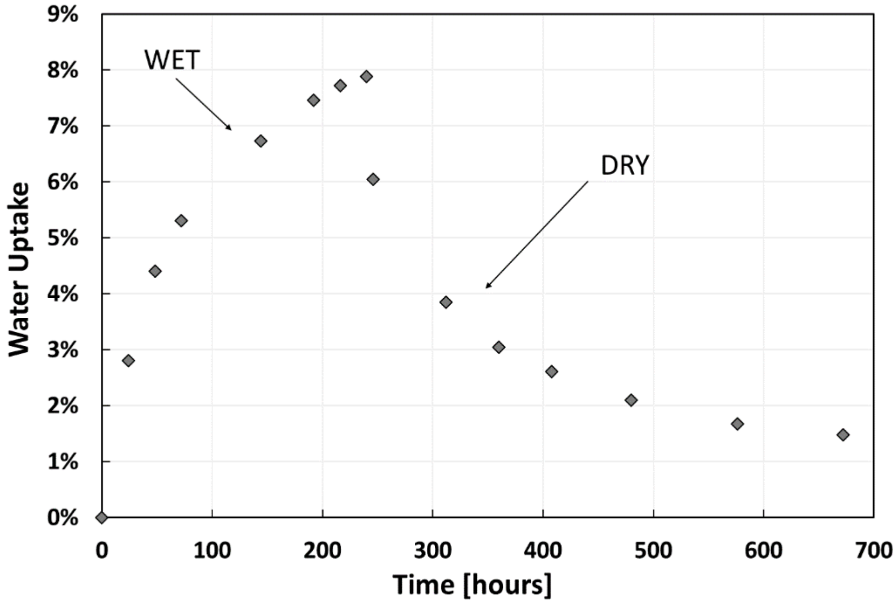

Figure1. Water uptake of FFRC$_s$ during the first wet-dry cycle.

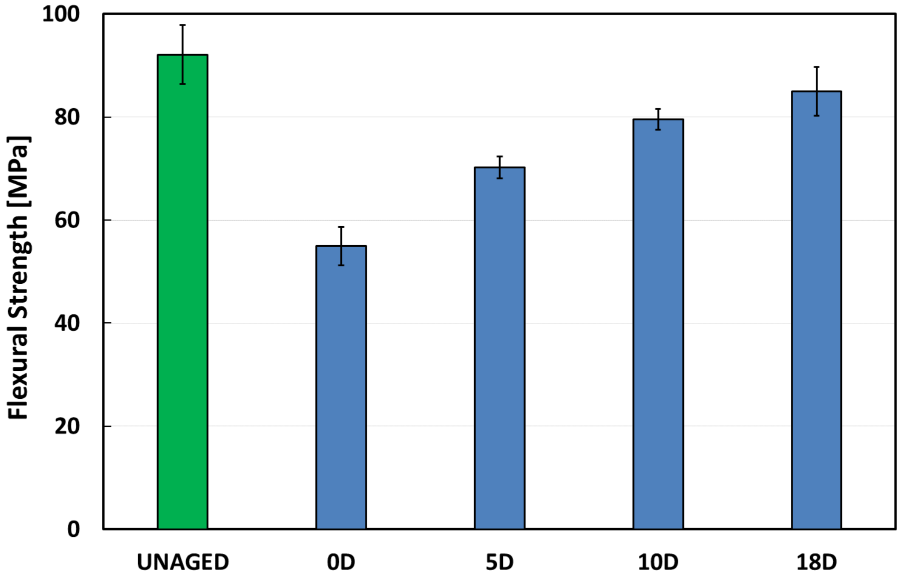

Figure 2. Flexural strength of unaged and aged FFRC$_s$.

P9 DESIGN AND OPTIMIZATION OF INNOVATIVE BIOMEDICAL IMPLANTS BY ADDITIVE MANUFACTURING TECHNOLOGIES

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The introduction of additive manufacturing offers great potential in terms of production technology (Gibson et al; 2014); for example, in regenerative medicine, it makes it possible to create individual medical devices with complex geometries and personalised designs. The processable materials are several (Shah A et al; 2017), starting with metals such as titanium, then moving on to polymers. The aim of this work is the optimisation of the main process parameters of various thermoplastic polymers such as polymethyl methacrylate (PMMA) (El-Ghani et al; 2018), polylactic acid (PLA), polycaprolactone (PCL) and polyetheretherketone (PEEK) by means of Fused Deposition Modelling (FDM) technology. Starting with the modelling of the test samples using Autodesk Inventor 2023 CAD software, the process instructions were then set in special slicing software (Ultimaker Cura). At the first step, main process parameters (i.e. nozzle temperature, bed temperature, print speed, acceleration, jerk and flow) were optimized in terms of the printed material reproducibility through weight and size measurements, and aesthetic quality of the product through observation under a digital and scanning electron microscope. The thermal properties of the used filaments and the printed systems were investigated by differential scanning calorimetry (DSC) measurements, in order to study the influence of the printing process on the thermal behaviour. Furthermore, the influence of the selected material, nozzle, and deposition paths of the fused material (e.g. Line pattern, Grid, Gyroid, Concentric, Octet, Triangle, Zig-Zag) on the mechanical properties was studied by tensile, compression and bending tests. Once this phase was completed, the optimal parameters were set for the production of devices for craniomaxillofacial applications such as prostheses and fixation systems.

Acknowledgements

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P10 DEVELOPMENT AND CHARACTERIZATION OF CHITOSAN HYBRID COATINGS BY SOL-GEL METHOD

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This paper describes the preparation and characterization of hybrid coatings constituted by SiO_2, TiO_2 and chitosan in varying compositions on aluminium, and glass substrates by sol–gel method. The optimized sol-gel solutions were deposited using by dip-coating and spray coating and the thermal treatment conditions (temperature) were optimized. In particular, condensation reactions necessary to allow the formation of the metal oxide networks were performed by storing the films in an open-air oven at 100°C and 80 °C for 30 minutes. Moreover, the functionalization of hybrid has been carried out with vinyltrimethoxysilane (VTMS).

The films were investigated by scanning electron microscopy (SEM), Fourier transformed infrared (FTIR) spectroscopy, hydrolytic degradation and corrosion resistance.

The process yields transparent chitosan hybrid coatings with good adhesion on glass substrates. An increase in roughness values was noted after the application of SiO_2-TiO_2/chitosan hybrid coatings.

The obtained results suggest that the functionalization of the hybrid coatings was affected through in situ hydrolysis–condensation reaction of vinyltrimethoxysilane (VTMS) in the reaction medium.

Furthermore, the composition of the coatings as well as their chitosan content affect the final chemical-physical properties. A possible application of the present hybrid coatings could be in packaging industries.

P11 SYNTHESIS AND CHARACTERISATION OF FLUORINE-FREE MIL-100(Fe) FOR HYDROGEN, CARBON DIOXIDE AND WATER VAPOR STORAGE

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Metal-organic frameworks due to their extremely large surface area and pore volume, and tunable surface features, can be considered competitive solutions for gas storage applications, e.g. hydrogen, carbon dioxide and water vapor. In this study, MIL-100(Fe) was synthesized though a HF-free hydrothermal route in a Teflon-lined autoclave at 160 °C for 12h using Iron Nitrate Nonahydrates as precursors. MIL-100(Fe) is composed of a trimeric iron coordinated by a benzene -1,3,5- tricarboxylic linker. The storage capacities towards hydrogen, carbon dioxide, and water vapor have been investigated. The excess gravimetric capacity for hydrogen and carbon dioxide were quantified by a High-pressure volumetric gas sorption analyzer (isorb). Water vapor adsorption is measured by Dynamic vapor sorption DVS analysis. The sample was characterized by powder X-ray diffraction (XRD), N_2 *adsorption-desorption isotherms and FT-IR analysis. XRD and FTIR data showed an agreement with literature data [1] , thus confirming the synthesis production of the desired MOF. The Nitrogen adsorption isotherms show that MIL-100(Fe) MOF has a BET surface area of* $1199\ m^2/g$*. The hydrogen adsorption in MIL-100(Fe) MOF is dominated by weak Van der Waal forces. As reference, hydrogen excess gravimetric capacity at 278K and 40 bar is 0.25 wt%. Adsorption- desorption isotherms of carbon dioxide showed a reversibility of the physisorption process. MIL-100(Fe) can store 32.64 wt% at 278 K and 10 bar and this value decreased to 23.86 at 303K and 10 bar. Similarly, the material showed promising water vapor sorption/desorption capacity. These preliminary results make the MIL-100(Fe) an interesting candidate for gas sorption applications. Further studies are ongoing for better assessing the materials storage capacity in different conditions.*

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P12 DEVELOPMENT OF ECOSUSTAINABLE ELECTROSPUN FIBERS FOR THE ABSORPTION OF HEAVY METALS

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Toxic heavy metal contamination of the aquatic ecosystem is a significant problem at a global scale [1,2]. This leads to irreversible accumulation of the heavy metals in the various tissues of sea animals, thus endangering the entire aquatic biota [3,4]. The conventional methods currently employed to adsorb these contaminants are precipitation, membrane filtrations and adsorption [5,6]. Nowadays nanotechnologies and nanomaterial sciences have a focus on fabricating nanomaterials that can be used in various water purification applications. Electrospinning is one of the most important techniques utilized in this field [7]. Electrospun fibers have promising properties, such as an interconnected porous structure, light weight, high porosity, and large surface area [7]. The present work is aimed at obtaining polylactic acid (PLA) electrospun fibers, covered with alginate or chitosan, as promising heavy metals absorbers. As references, alginate and chitosan beads and films were produced, by ionic reticulation and solvent casting techniques, respectively. Alginate derived from brown algae is a highly popular material for the biosorption of heavy metals due to its low cost, and high affinity to metals via gelation. Indeed, it is characterised by the presence of carboxy and hydroxy groups which can crosslink with cations. Thus, due to the negatively charged carboxyl groups, it can electrostatically adsorb heavy metal ions by chelation [8]. Similarly, the chitosan, a low-cost natural polysaccharide produced from the deacetylation of chitin, has been used in many studies for the heavy metal ions adsorption [9]. The produced beads were observed at optical microscope, whereas the films and fibers at scanning electron microscope (SEM). Swelling and solubility tests were carried out for the beads and films, respectively. *The successful coating deposition and the wettability of the PLA nanofiber were evaluated by observation at SEM and contact angles measurements, respectively.*

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P13 THE THERMOMECHANICAL BEHAVIOR OF BIO-BASED EPOXY RESIN AND DAYLIGHT-CURABLE BLENDS FOR 3D PRINTING THROUGH THE USE OF LIQUID CRYSTAL DISPLAY (LCD) TECHNOLOGY

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Bio-based epoxy resins are widely utilized in various applications such as adhesives, coatings, composites, and electrical components, offering comparable performance characteristics to conventional epoxy resins, including high strength, durability, and chemical resistance [1].The utilization of bio-based materials in 3D printing has been receiving increasing attention as a means of minimizing the environmental impact of this technology.In general, it can be observed that the majority of formulations developed for stereolithography (SLA) and digital light processing (DLP) still predominantly rely on non-renewable resources and entail significant costs. While there is growing interest in incorporating biomass materials into these formulations, only a few noteworthy examples have been reported in recent literature, demonstrating considerable efforts in achieving this [2].This study aimed to explore the potential of blending a bio-based epoxy resin with a commercial daylight-curable resin [3] at various weight percentages to enhance the thermomechanical properties of 3D-printed parts while adhering to the working principle of LCD (Liquid Crystal Display) printers.The different formulations were initially assessed in terms of their thermomechanical properties both before and after post-treatments like photo- and thermal-curing. This facilitated a comparison of the various blends based on their mechanical strength, glass transition temperature, and other pertinent properties.Upon identifying the optimal formulation, 3D-printed samples were produced using LCD printing technology. Thermo-calorimetric and morphological tests were then conducted to evaluate the thermal stability and microstructure of the printed parts.Overall, the findings of this study indicate that blending bio-based epoxy resins with commercial resins can lead to enhanced properties in additive manufactured parts. This approach has the potential to promote sustainability in 3D printing by reducing the consumption of non-renewable resources, while still meeting the performance standards required for numerous applications.

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P14 REFRACTIVE INDEX MODELLING OF TRANSPARENT 3D PRINTABLE PHOTOCURABLE RESINS FOR MICRO-OPTOFLUIDIC DEVICES: A REGRESSION APPROACH

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In this work, a quadratic polynomial regression model showing good performance (R^2 equal to 80%) was developed for refractive index value estimation of transparent 3D printable photocurable resins usable for micro-optofluidic (MoF) devices which found applications within the field of the Lab-on-a-Chip (LOC). The regression model was empirically developed by correlating experimental optical transmission measurements (dependent variable) to known refractive index values (independent variable) of photocurable resins that found application in optics, hence obtaining a related regression equation. Thus, in this experimental work is proposed, for the first time, an innovative, simple and cost-effective experimental setup for running transmission measurements which is suitable for 3D printed parts having smooth surfaces (i.e., between 0.4 and 2 μm). Once the regression model was determined, it was further exploited to estimate the unknown refractive index value of novel photocurable resins used in vat photopolymerization (VP) 3D printing techniques to fabricate micro-optofluidic devices with a direct 3D printing approach, i.e., without using any additional assembly process. In the end, by running a wide experimental campaign it was proved how the knowledge of the estimated parameter permitted to compare and interpret empirical optical data collected by mean of microfluidic devices made of more traditional materials, such as Poly(dimethylsiloxane) (PDMS), up to novel 3D printed resins which are certified for biological and biomedical applications.

Aknowledgements
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P15 NICKEL EXSOLVED PEROVSKITE CATALYST FOR ETHANOL AUTOTHERMAL REFORMING

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The autothermal reforming (ATR) of ethanol is a process in which ethanol is converted into hydrogen and carbon monoxide via a series of chemical reactions, which has significant environmental advantages. For the autothermal reforming of ethanol, various catalysts based on precious (Rh, Pt, Ru) and non-precious metals can be utilized (Ni, Co). Due to its ability to rupture C–C bonds, the nickel active catalyst is the most frequently studied in reforming reactions, as it meets the economic and industrial application criteria. To increase the efficiency and selectivity of the ATR of ethanol process, scientists are continuously developing and refining new strategies for the formulation of catalyst materials with improved performance and cost-effectiveness. The ex-solution of active metal prior to incorporation into the lattice of the crystalline oxide is a more advanced and efficient method for introducing metallic nanoparticles on ceramic support, as perovskite oxide. Some catalyst is incorporated as cations within the host lattice during the synthesis process under oxidizing conditions, and the metallic catalyst exsolves as nanoparticles upon reduction.

To reduce coke deposition, this research has been devoted to the development and testing of an ex-solved $Sr_2FeMo_{0.6}Ni_{0.4}O_{6-\sigma}$ *catalyst containing ruthenium. It has been discovered that the ruthenium/nickel ex-solved perovskite catalyst has excellent activity due to its high surface area and strong interaction between the nickel and perovskite structure, as well as outstanding stability due to the loading of ruthenium, which prevents coke formation by promoting the water-gas shift reaction.*

P16 3D PRINTING OF SELF-MONITORING PIEZORESISTIVE HIGH-PERFORMANCE NANOCOMPOSITES

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High-performance polymers are a class of polymer materials characterised by high mechanical, thermal and chemical properties, which make them crucial in demanding applications such as the medical, aerospace or naval sectors. In these applications, the demanding conditions give rise to the need for a high degree of reliability during a component's operating life. Structural health-monitoring (SHM), thus, represents a useful tool to ensure reliability by continuously monitoring the strain applied to a structure. 3D-printing is a new manufacturing paradigm suitable for a low number of customized and complex components, such as those needed in high-performance applications, but 3D-printed parts are inherently less reliable due to their inhomogeneity, anisotropy and non-standardized process. Therefore, the application of SHM on 3D-printed components is especially valuable. A novel way to perform SHM without external sensors is the use of self-monitoring materials, which are structural materials capable of detecting their own deformation and integrate more functions into a single smart component. The piezoresistive effect in an electrically conductive composite can be used to grant self-monitoring performances, as it involves the variation of electrical resistivity with an applied deformation.

In this work, piezoresistive nanocomposites based on a high-performance polymer, Polyether-ether-ketone (PEEK), with 3, 5 and 10 wt% of carbon nanotubes have been produced. Extruded filaments and 3D-printed parts showed exciting self-monitoring performances with Gauge Factors up to 14.5.

The mechanical, thermal and rheological properties of the material were investigated, and a model of the complex electrical conduction mechanism in a 3D-printed part was proposed. 3D-printed parts showed promising reliability as self-monitoring components in cyclic loading as well, demonstrating the possibility of employing these multifunctional materials in high-performance applications.

P17 BIOPOLYMERS AS MATERIALS FOR THE REALIZATION OF TRANSIENT MECHANOELECTRICAL DEFORMATION SENSORS

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The use of electronics in everyday life is constantly growing. This continuous multiplication of devices risks having a very negative impact on the environment. Suffice it to say that 7.3kg of e-waste per capita were produced in 2019, and that this quantity is expected to double by 2050. For this reason, the development of eco-friendly green electronic devices represents a hotly discussed research topic.

Polymers are being considered as a promising class of materials for these new green electronics. In the last decade among polymers there is a growing interest in implementing biodegradable polymeric materials in what is called "transient electronics". Transient technology is an emerging field that requires materials, devices, and systems to be capable of disappearing with minimal or non-traceable remains over a period of stable operation.

Our research group is engaged in the study of biodegradable polymers that can be used in the realization of transducers or sensors by exploiting the piezoionic behavior of their composites. Among biodegradable polymers we have studied bacterial cellulose (BC), polyhydroxyalkanoates (such as polyhydroxybutyrate), pullulan, xanthan gum that are produced in nature by numerous microorganisms, including through bacterial fermentation of sugars or lipids. Thus the use of such materials in electronics involves a reduced environmental impact deriving both from their biodegradability (and in some cases from their solubility in water) and from their biosynthesis which ensures the possibility of recycling waste products deriving for example from the food industry.

In this communication we report on the results obtained by impregnating biopolymer membranes with Ionic Liquids and covering the composite with PEDOT:PSS as conducting electrodes. Such devices generate an electrical signal when deformed. The SC current generating properties of the composites as mechanoelectrical deformation sensors have been experimentally investigated and compared.

P18 HYBRID COATINGS IMPROVE THE OXYGEN BARRIER OF POLYMERIC SUBSTRATES

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Introduction

State-of-the-art food packaging systems are largely based on multi-layer, multi-material design, limiting the end-of-life recycling process and ultimately leading to resource waste. Demand for sustainable solutions pushes the research for innovative packaging design based on reducing barrier layer complexity and mass. The present work aims at developing a new class of hybrid mixed oxides coatings with improved oxygen barrier performance on polyethylene terephthalate (PET) and biaxially oriented polypropylene (BOPP).

Material and Methods

Hybrid coatings of silica and alumina have been prepared via sol-gel and deposited by rod coater on corona-treated substrates. The network organic phase was reinforced by introducing poly(vinyl alcohol) (PVA). Coating morphology has been analyzed by SEM and EDS, and the barrier properties were investigated by measuring the oxygen transmission rate (OTR) and the water vapor transmission rate (WVTR). Lastly, the UV-Vis transmittance has been measured to evaluate the optical transparency.

Results

The morphological characterization showed that adding PVA was essential for preparing defects-free coatings. EDS analysis confirmed the presence of a dense coating presenting both silica and alumina. Coated samples showed a significant decrease in the OTR with a barrier improvement factor (BIF) between 2 and 13, while the WVTR exhibited no significant changes (Figure 1).

Discussion and conclusions

The typical brittle nature of dense ceramic coatings has been reduced due to the presence of PVA, allowing for improved barrier properties compared to the pristine films. Alternative alkoxides will be exploited during sol-gel synthesis to obtain comparable WVTR improvement. This work highlights the potential of mixed oxides coatings deposited on commercial substrates as a sustainable alternative to multi-layer solutions for oxygen-sensitive food.

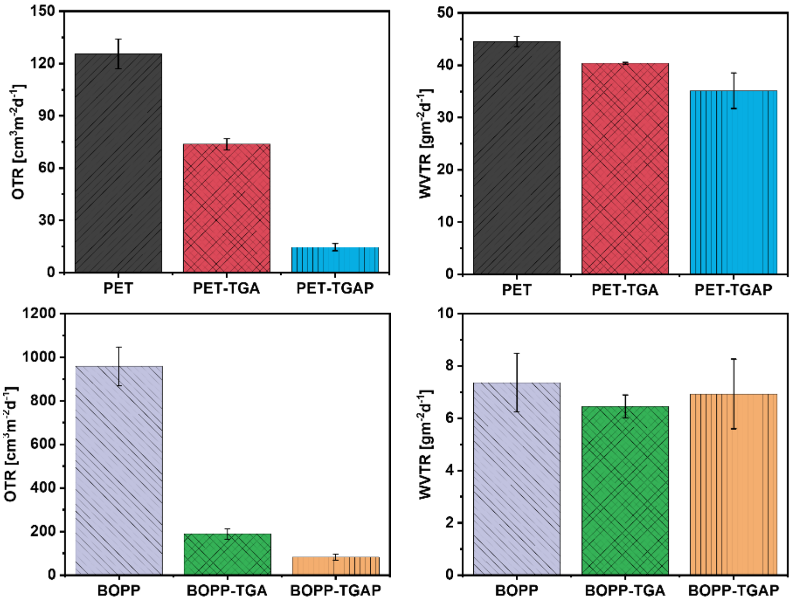

Figure 29. Comparison of the barrier properties of pristine polymers with coated counterparts.

P19 THE FUSED DEPOSTION MODELLING (FDM) AND THE RHEOLOGICAL PROPERTIES OF POLYLACTIDE (PLA)-BASED FILAMENTS

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In the last 30 years, additive manufacturing (AM) technology has emerged as a new digital manufacturing technology that integrates machinery, computer, numerical control, and materials. By means of computer aided design (CAD), this process permits the realization of 3D objects with detailed and accurate geometry. Fused Deposition Modelling (FDM) is a popular 3D printing technology that involves melting a thermoplastic filament and depositing it layer-by-layer on a heated support to create 3D parts.

*Because of its biodegradability, excellent mechanical properties, and low crystallization rate, polylactide acid (PLA) has been widely used for conventional additive manufacturing. The purpose of this study was to collect data for predicting the printability of three different poly(lactide) acid (PLA)-based filaments, two commercially available and one processed in a lab-scale extruder. The samples were characterized using dynamic rheological testing via frequency sweep at five different temperatures (130, 150, 170, 190, and 210°C). Figure 1 shows the storage (G') and loss (G") moduli as a function of frequency (ω) for each material. The rheological measurements were reworked to calculate the characteristic relaxation times of polymer chains, activation energy, zero shear viscosity, and shear thinning index. All the filaments were processed in a 3D printer at three different nozzle temperatures. Macroscopic shaping defects in 3D objects were verified in correspondence of specific values of polymer relaxation time (~10^{-2} s), zero-shear viscosity (10^3 Pa*s), and molecular weight (~10^5 g/mol). Then, specific values of material viscosity and printing speed resulted in a discontinuous flow of molten polymer from the duct.*

In conclusion, the polymer relaxation time and zero-shear viscosity were important factors during melt deposition on the platform to reduce deformation and aesthetic defects, whereas viscosity was the main parameter to favour polymer flowability during extrusion.

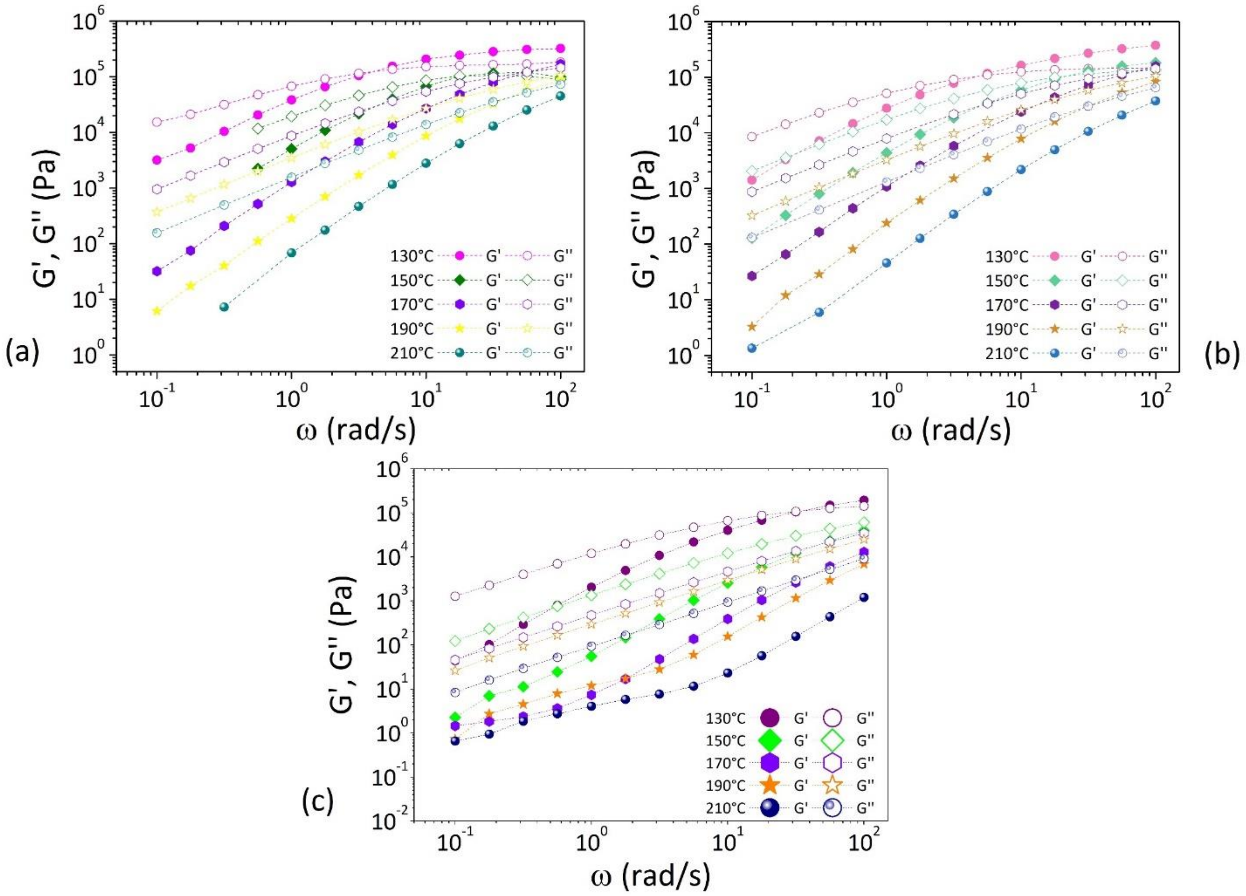

Figure 30. Storage (G') and Loss (G'') modulus as a function of frequency at different testing temperatures of three considered filaments: (a) first commercially available filament; (b) second commercially available filament (c) filament realized in lab.

P20 2.5 SURFACES VIA FOCUSED ION BEAM PATTERNING AND NANOINDENTATION IMPRINT

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Realizing complex micro- and nano-scale 2.5 patterns with high accuracy requires lithographic techniques. Notably, ion lithography reduces typical electron and photolithography limitations such as small Z (depth), surface sample preparation (photoresist), mask use, and costs. However, this technique has some drawbacks because it's time-consuming, the patterned area is limited to a few thousand square microns, and the beam drift produces stitching mismatch after tens of minutes. These limitations can be reduced or even eliminated (on some materials) thanks to a proper milling strategy and beam control. This work presents a possible solution based on a multiparametric scripting software developed to obtain complex 3D patterns over large areas starting "simply" from its CAD (Figure 1). The developed protocol allows minimization of the redeposition and obtains accurate and scalable milling routines. A hemisphere and a pyramid shape are milled and compared to the shapes obtained using conventional techniques to show the method's applicability. Results show that the process produces complex shapes while overcoming the detrimental effect of traditional raster/serpentine FIB strategies, such as redeposition. Lastly, a fish-net structure with a pitch of ~200 nm and a series of truncated cones with sub-micrometrical details are realized to show the potential impact of this new method. Results show that a spatial resolution of less than 100 nm is achievable with the help of this method. The developed methodology could also speed up the fabrication of 2.5D surfaces by nanoimprint lithography. The present work achieved this by creating a complex multi-scale pattern on top of a nanoindentation tip to benefit from novel high-speed mapping capabilities (time reduction) and full-control capabilities from FIB machining protocols.

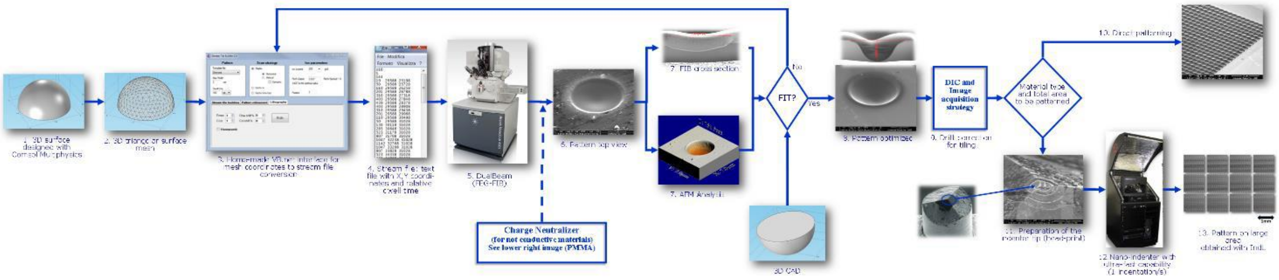

Figure 1. Complete workflow of the developed methodology for 2.5D shape-optimized during ion lithography and nanoimprint lithography by FIB machining.

P21 DEVELOPMENT AND VALIDATION OF A NEAR-FIELD DIRECT-WRITING ELECTROSPINNING SYSTEM FOR TISSUE ENGINEERING APPLICATIONS

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Introduction

Near-field direct-writing electrospinning (NFES) can reach higher accuracy than fused deposition modelling but its use to process natural polymers for tissue engineering is still limited.

This study aims at developing a versatile NFES system to fabricate nature-derived polymeric scaffolds.

Methods*A NFES setup was assembled from 3D printers components and validated with Gelatin B (GelB) and Bovine Serum Albumin (BSA) 10:1 solution in 70% v/v acetic acid, depositing fibers in parallel arrays (20x20 mm^2) or circles (Ø=26 mm) and optimizing XY speed, electric field and, only for parallel arrays, fiber spacing.*

Then multilayered parallel fiber samples (10x10 mm^2) were created with Methacrylated Gelatin and BSA in 70% v/v acetic acid varying the total process iterations (300, 600, 900) and photo-crosslinked.

Morphology of single fibers and multilayered samples was evaluated by SEM and ImageJ software was used to analyze images measuring mean fiber diameter and alignment degree.

Results

The NFES setup allowed a high-fidelity collection of various patterns with an easy XY motion trajectories programming. 22% w/v GelB/BSA concentration led to continuous fiber deposition in various environmental conditions. In both linear and curved trajectories, control over fiber deposition and straightness was reached at 8000 mm/min speed except for some coils observed in confined regions and submicrometric fibers were reproducibly obtained at an electric field of 1.5 kV/mm. In parallel arrays, fiber spacing up to 500 μm provided submicrometric straight fibers deposition.

In multilayered samples, the highest alignment was obtained at 300 iterations and 83% of fiber diameters were suitable for anisotropic tissues regeneration.

Conclusions

A custom-assembled NFES system allowed to easily and precisely deposit oriented fibers with a reproducible morphology comparable to extracellular matrix and to design with nature-derived polymers multilayered biocompatible architectures.

P22 AUSTENITE-MARTENSITE SURFACE TRANSFORMATION ON AISI 316L INDUCED BY LOW-ENERGY HIGH-CURRENT ELECTRON BEAM TREATMENT

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Low-Energy (1-6 J/cm$_2$), High-Current (up to 25 kA) Electron Beam (LEHCEB) sources have emerged in the last decades as a promising option for surface modification of materials [1]. This technology relies on the generation of pulsed (0.01-0.2 Hz), wide-area (50 cm$_2$) electron beams in a plasma filled diode (PFD) from an explosive emission cathode (EEC). Extremely short pulses (2-4 μs) allow to induce thermal gradients at the surface of electrically conductive materials in the order of 10$_8$-10$_9$ K/s, causing melting and even evaporation phenomena followed by fast solidification. As a consequence, modifications of thin surface layers (0.1-10 μm), unattainable through conventional surface treatments, are obtained [1]. Moreover, LEHCEB provides other advantages such as improved hardness, increased corrosion resistance and removal of contaminants [2].

In the present work, LEHCEB was employed to induce microstructural modifications of AISI 316L samples with the aim of forming a thin surface martensitic layer, characterized by higher mechanical properties. AISI 316L is a stainless steel of austenitic type which is widely applied, thanks to its corrosion resistance and its good mechanical properties. The effect of electron accelerating voltage (25 and 30 kV) and number of pulses (5 and 25 pulses) was investigated. Experimental parameters were selected according to computational simulations by Comsol Multiphysics, which allowed to estimate the thermal gradients in the material and the thickness of the modified layer.

Samples were characterized by means of Scanning Electron Microscopy, Energy Dispersive X-Ray Spectroscopy, Electron Backscatter Diffraction, X-Ray Diffraction and Vickers Microhardness. The formation of a thin surface layer (1-5 μm) with martensitic structure was assessed. Experimental results agreed with computational simulations data.

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P23 3D PRINTED GREEN COMPOSITES BASED ON POLYMERIC MATRICES AND ANCHOVY FISHBONE WASTES

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Global use of fish products has recorded a huge increase over the last years. Consequently, also the amount of fish wastes produced and grossly discarded in markets has undergone a dramatic increase causing environmental and hygiene issue. Use of these scraps for the production of materials with higher added value would promote the implementation of circular bioeconomy, reducing waste production and solving environmental and hygiene issue related to them. Combining biopolymers with animal waste could be an effective strategy in the view of producing food packaging decreasing the amount of bioplastic needed and, consequently, the cost of the final products. In this work, anchovy fishbone (EE), obtained by local market, were ground into powder and added to a biopolymeric matrix (Mater-Bi®) in order to produce a 3D-printed fish crate.
Anchovy fishbone flour (EE) was obtained by anchovy scraps collected directly at "Ballarò" market (Palermo, Italy) following the process reported in Scheme 1.

Scheme 1. Schematic representation of anchovy fishbone flour preparation.

MB/EE10 composite filament was able to be 3D printed for FDM into flat (Figure 1a) or geometrically complex products (Figure 1b). The printing process proceeded smoothly and no clogging of the nozzle was observed.

Figure 1. Specimen 3D printed (a) and product with complex geometry printed with MB/EE10.

EE particles appear well embedded and uniformly dispersed in the polymeric matrices (Figure 2a). Moreover, good adhesion between layers was achieved for samples printed with raster angle of ± 45° (Figure 2b).

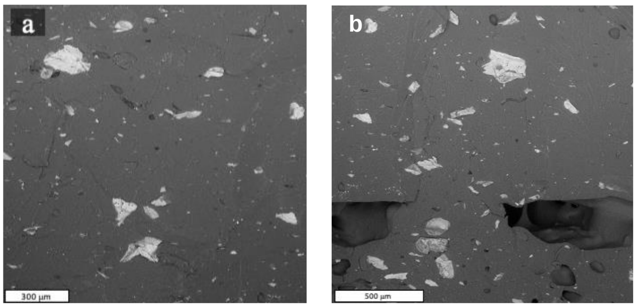

Figure 2. Cryo-fractured cross-section of 3D-printed MB/EE10.

The addition of 10% of EE to MB lead to a decrease in E and TS but the ductility of the composite displayed a strong increase if compared with pure matrices, with EB value going from 55.6 to 258%, reasonably due to the presence of the oily phase in the filler that act as plasticizer (Figure 3).

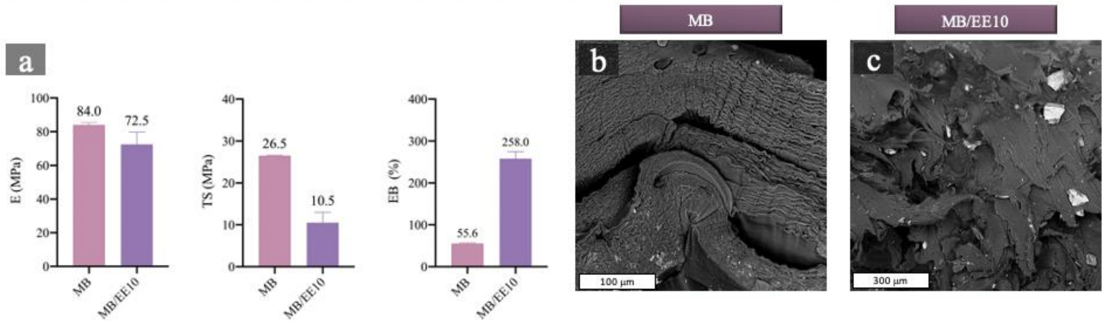

Figure 3. Elastic modulus (E), tensile strength (TS), and elongation at break (EB) of 3D-printed MB and MB/EE10 (a) and relative SEM micrograph of their fractured cross-section (b, c).

P24 EVALUATION OF MICRO FIBRILLATED CELLULOSE (MFC)/POLY (LACTIC ACID) (PLA) BIOCOMPOSITES FOR FILM BLOWING PROCESS

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Poly(lactic acid) (PLA) has gained significant interest for use in a wide range of commodity applications due to its versatility, good heat stability during processing, and high tensile strength. However, its limited flexibility, impact resistance, and low barrier properties have restricted its application. To address this, a common sustainable technique involves improving the final performance of PLA through various strategies, such as developing natural fiber-reinforced biocomposites. Specifically, cellulose has been found to improve the barrier and mechanical properties of thermoplastic biopolymer films, and there has been particular interest in microfibrillated cellulose (MFC).

This work investigates the processability of biocomposites based on PLA filled with MFC to assess their suitability for the film blowing process. The biocomposites were produced using a semi-industrial twin-screw extruder with the addition of a biobased plasticizer as a dispersing aid. The first part of the study focused on evaluating the influence of the filler on the rheological behavior of the melts in non-isothermal elongational flow to identify the processing behavior of the composites. Additionally, the morphology and mechanical properties of melt-spun biocomposite fibers with different draw ratios were evaluated. Based on the results, a suitable filler concentration was determined for producing films through a film blowing operation. The obtained blown films were characterized to evaluate their thermal, optical, barrier, and mechanical properties. The films exhibited excellent optical properties that were preserved in the presence of the filler. This was due to the good dispersion of the filler, which was maintained even after the reprocessing of the composite during the film blowing operation, as shown in Figure 1. Moreover, the addition of MFC resulted in an improvement in the barrier properties of the biocomposites compared to their respective plasticized matrices.

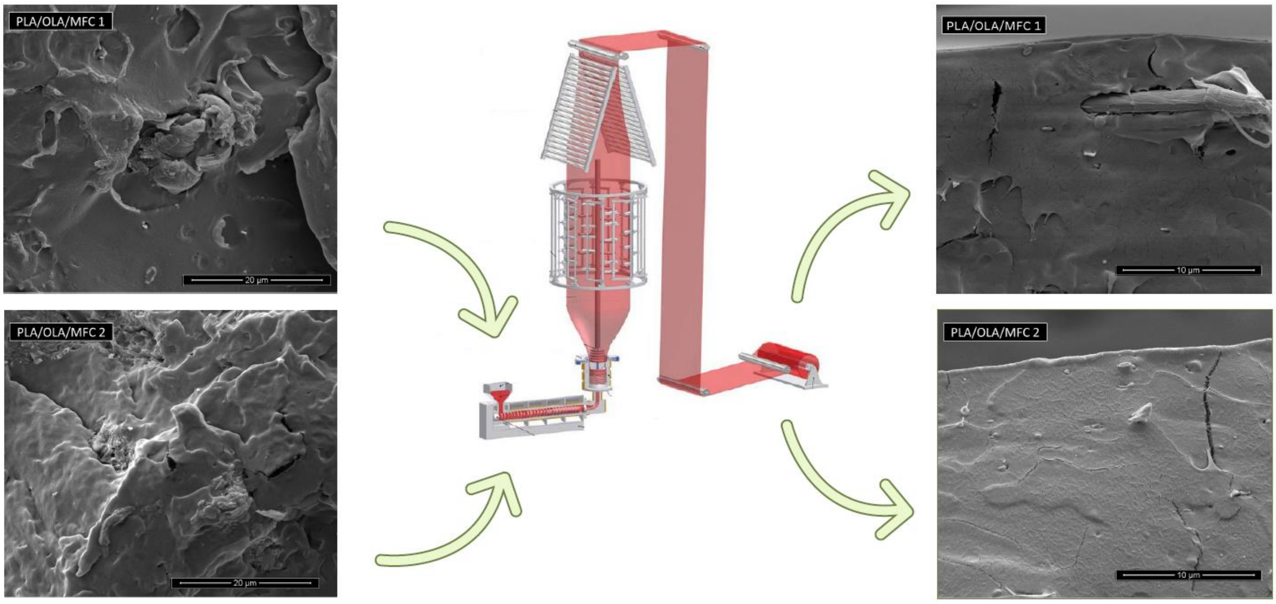

Figure 31. Comparison of the morphology between extruded bicomposite samples and their respective blown films.

P25 SUSTAINABLE CONCRETE FROM RECYCLED ORGANIC WASTE MATERIALS AS AGGREGATE REPLACEMENT

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The disposal of higher volumes of organic waste has become a challenging issue globally. While using organic waste materials instead of sand for aggregate does not directly reduce the carbon dioxide emissions from concrete production it does reduce the strain on finite sand resources across the world. This project investigated the viability of replacing aggregates in concrete with various different organic waste materials, namely rice husk ash, wood ash, corn cob granules, and wheat straw. Concrete mixes were produced replacing 10% of the sand aggregate in the mix. Concrete fresh properties were studied using the slump test, and mechanical properties were measured using the compressive strength and flexural strength tests. Finally, ground theory case studies along with a life cycle assessment (LCA) comparison between the organic waste replaced concrete and pure concrete were conducted. The result suggests that rice husk ash and wood ash are the most suitable organic waste products for use as aggregate replacers considering mechanical properties are reduced but close to that of pure concrete (Figure 1). Because of the comparably low strength of the concrete sample made using wheat straw and corn cob granules, there might be limited use of those organic waste in concrete otherwise advanced treatment that would improve concrete performance. Furthermore, it is expected a potential reduction of the environmental impact of the concrete alternatives with organic waste, compared to traditional concrete.

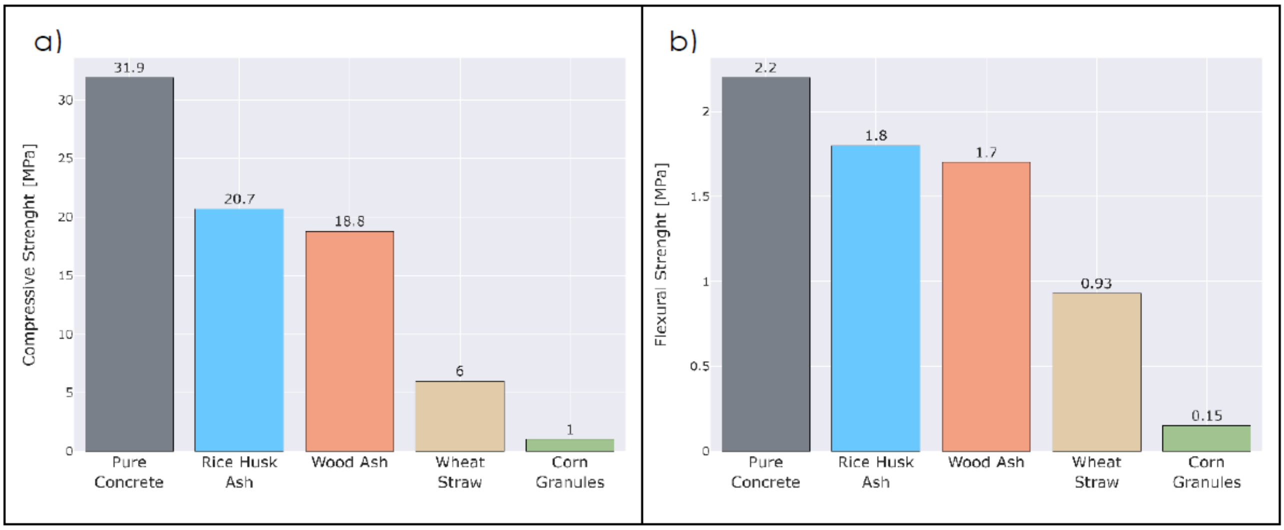

Figure 1. a) Average 14 days Compressive Strength and b) average 14 days Flexural Strength of the Concrete Samples.

P26 UPCYCLING OF POLYESTER FIBER AND GARMENT TEXTILE WASTE

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This research is part of the project MATE.RIA, aimed at extending the life of post-consumer garment-textiles and identifying a new market for polyethylene terephthalate (PET) waste produced during fiber manufacturing. Here we investigate the possibility of developing innovative products exploiting the thermal insulation capability of textiles for architectural applications. The materials considered in this work are the polyester fiber waste from the production line and the end-of-life garment-textiles, provided by the project partners FranaPolifibre and HUMANA, respectively.

Gel Permeation Chromatography, Differential Scanning Calorimetry, and uniaxial tensile tests were used to preliminarily determine structural, thermal, and mechanical properties. The materials were then used to produce prototype of thermoregulatory panels, which were obtained by applying a layer of PET to one or both fabric surfaces: the fabric acts as a thermal insulator while the PET layer provides rigidity.

Two techniques were used to apply PET layers on a fabric: textile dip coating in PET solution and coupling with cast film. Both processes required the preparation of a PET solution. Hexafluoro isopropanol (HFIP) has been selected as solvent due to its compatibility with the textiles.

A solution of 250 g/L was utilized for dip coating. Textiles varying in percentages of cotton and polyester were dip-coated in a solution on their "Right side", "Wrong side", or on both surfaces. Cast films were obtained by 200 g/L of PET/HFIP. Dried membranes that ranged in thickness from 157 to 190 μm were coupled with the fabrics after wetting the membrane surface with additional HFIP.

The thermal regulation capability of the panel could be modified through Phase Change Materials (PCMs) inclusion. Composites' morphological and porosity characteristics have been examined via optical microscopy analysis. Incorporating PCMs suitable for architectural applications into the composite is under investigation.

Acknowledgements

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P27 MONOPOLYMER BLENDS OF VIRGIN AND RECYCLED BIODEGRADABLE POLYMERS TO PROMOTE THE DECARBONIZATION OF PLASTICS

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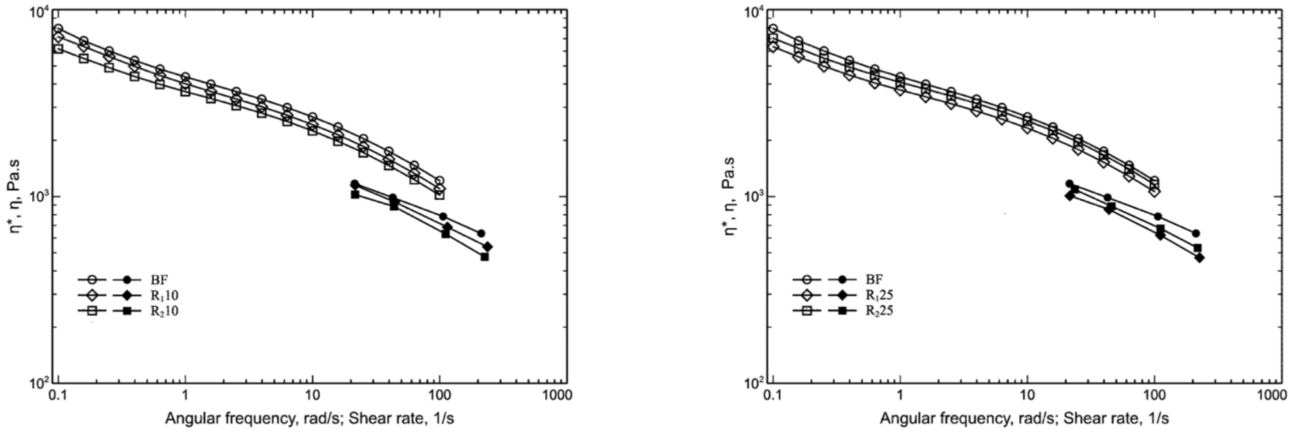
One of the solutions for decarbonizing plastics is to increase recycling rates. To date, the recycling of conventional polymers has been fairly well studied, while the recycling of biodegradable polymers has not been as well investigated.
In this work, monopolymer blends of the same virgin and recycled polymer were prepared. Specifically, a sample of commercially available biodegradable blend (Bio-Flex® F2110) *were reprocessed in a single-screw extruder until two extrusion cycles were completed. These samples were ground and reprocessed with 75% and 90% of the same virgin polymer.*

The rheological characterization was performed at 180 °C using both an ARES G2 (TA Instruments, USA) plate–plate rotational rheometer and a Rheologic 1000 capillary viscometer from CEAST (CEAST, Italy).

Tensile measurements were carried out with an Instron Model 3365 universal testing machine on rectangular specimens (10 × 90 × 0.4mm) cut from plates prepared by compression molding at 180 °C using a Carver hydraulic laboratory press.



a) **b)**

Figure 32. Flow curve of virgin BF sample and reprocessed blends: (a) BF, $R_1$10 and $R_2$10; (b) BF, $R_1$25 and $R_2$25.

The results obtained showed that, in the case of the 10 % blend, see **Figure 1a**, the viscosity decreases as the amount of reprocessed material and the number of reprocessing steps increase in the monopoly blend, while the opposite occurs in the case of the 25 % blend, see **Figure 1b**, where the viscosity decreases less as the amount of reprocessed material and the number of reprocessing steps increase, due to the formation of branched structures.

Tensile results (data not shown for brevity) demonstrated the great recyclability of this polymer system in monopolymer blends, as the values remain almost constant compared with virgin material. In addition, the behavior of these monopolymer blends was predicted very well by a proposed additive model (no reported for brevity).

Reference

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P28 NEW SUSTAINABLE COMPOSITES BY USING BIO-BASED MATERIALS

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As petroleum resources become depleted and growing global environmental concerns about climate change and environmental pollution, challenges ahead include the development of new recycling technologies while simultaneously creating new sustainable composite materials made from bio-based and renewable materials including vegetable and food wastes. As known, sustainable composites from renewable materials, waste and biopolymer matrices are of great importance, as they can potentially reduce environmental impacts and implementing an effective circular economy. However, the *overall properties of such biocomposites are still far from the high-performance conventional composites, although some of these new eco-friendly materials could be considered suitable candidates for new generation multifunctional packaging.*

In this work, biopolymer films, based on chitosan and cellulose and containing galactaric acid derivatives, have been formulated by solvent casting and characterised by spectroscopy (FTIR and UV-visible), mechanical (DMA) and morphological analysis. All obtained results highlights a good compatibility between the chitosan and cellulose, and additionally, the presence of galactaric acid derivatives promotes the interactions and/or reactions between the chitosan and cellulose macromolecules. Further, the photooxidative resistance of the new sustainable composite films has been evaluated subjecting thin films to UV exposure and the progress of degradation has been followed monitoring the development of oxygen-containing groups in time. Finally, all results highlight the possibility to consider these sustainable composites for the formulation of new generation multifunctional packaging materials.

P29 GREENING EARTHEN PLASTERS: A STUDY ON BIO-SOURCED POLYMERS AS STABILIZERS

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Earthen material is a common construction material that has been used for centuries. Today, it is estimated that around 50% of the world's population lives in buildings made from earthen material. The widespread availability of this material combined with its low embodied energy makes it an attractive option for sustainable construction. However, the use of stabilizers (such as cement or acrylic resins) to improve its durability and mechanical performance can have a high environmental impact, reduce its recyclability and compromise its hygrothermal properties.

In the past, earthen material was stabilized using fibers, agricultural waste and biopolymers, but little research has been conducted on their properties and suitability for other techniques due to the challenges of standardizing such systems.

At this moment, an alternative, as compromise, is represented by bio-sourced polymers which allow to reduce carbon footprint and CO2 emissions.

In this work, earthen materials for plasters have been prepared in the lab and stabilised by using two different types of bio-sourced polymers (alkyd emulsion and urethane alkyd dispersion). Experimental tests for measuring mechanical, heat and mass transfer properties have been carried out on six different mix-designs.

A comparison between results obtained for earthen materials as received and stabilised by using bio-sourced polymers have been reported. No particular improvements have been noted in system treated with alkyd emulsion, while those fabricated by using urethane alkyd dispersion show similar thermal conductivity values but relevant enhancements in mechanical properties (about three times the value measured for as received samples) and water resistance (lower capillary rise and lower capillary coefficient).

P30 INDUSTRIAL APPLICATIONS OF BIODEGRADABLE POLYMERS AND PAPER FOR THE PACKAGING SECTOR

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Films for industrial packaging applications (and with special concern to the tissue and food sectors) are usually made of non-biodegradable polymers and are manufactured via film blowing process (thus requiring the use of polymers with suitable rheological properties). On the other hand, stricter legislation requirements substantially ask to replace traditional polymers with biodegradable and/or compostable ones, and to increase the use of other *biodegradable materials such as paper (due to ever-rising request by the GDO). In this context, there is very limited information about the properties of transparent biodegradable polymers to be used for film blowing, and similar considerations apply to thin paper sheets to be coupled with polymer films.*

Therefore, we studied the rheological and mechanical properties of biodegradable polymers for film blowing, as well as the mechanical properties of paper and of the laminated films for the packaging sector. The investigated materials included several MaterBi® grades, as well poly-(lactic acid) (PLA), polybutylene adipate terephthalate (PBAT), PLA/PBAT and PLA/biodegradable copolyester blends, paper sheets with 35-60 g/m2 grammage, and compostable adhesives. Polymer films were prepared via laboratory equipments as well as industrial ones, paper films were studied as received, polymer/paper multilayer films and laminates were manufactured in a special line at Joeplast. Characterization included rheological, tensile, tear, permeability and optical tests, in order to choose the most suitable polymers and paper samples, and the optimum multilayer system.

The investigated materials showed suitable properties for application in the tissue packaging sector; moreover, the results from the research activity were developed from a technological-industrial point of view, obtaining a biodegradable and compostable MOCA compliant final product, with partial transparency and adequate processability.

Figure 33. Example of obtainable product

Acknowledgements

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P31 RECYCLING OF COMPACT DISCS: A CLEAN SOLUTION

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The aim of this work is to set up a technique for the recycling of polycarbonate contained in electronic devices such as Compact Disc (CD) and Digital Versatile Disc (DVD), which is considered as green as possible.

As yet there are no techniques on an industrial scale that propose the recovery of the of the polymers from which these devices are made. In our approach, we propose a technique for the recovery of polycarbonate that is as "clean as possible" and at the same time the most accessible from an economic point of view. In fact, the removal of the non-polymeric layers is easily obtained through a thermo-mechanical treatment in a basic solution by ammonia.

We treated in an ultrasonic cleaner bath post consumer CD immersed in a solution of water and ammonia with different concentration of ammonia (0.25, 0.5 and 0.75) for different temperatures (20, 30 and 50°C) , power of the ultrasonic treatment and times.

The polycarbonate discs obtained was subjected to two extrusion cycles in order to simulate the recycling steps. The temperature profile was 280–280–285–290 ◦C. In order to verify the

effect of the humidity and therefore of the hydrolytic chain scission of the polycarbonate, the extrusion tests were performed on wet and dry (130 °C for 12 h) polymer.
A mechanical characterization and rheological characterization were carried out on the reprocessed polymer in order to evaluate the performance following recycling operations. The results obtained showed that in the dried polymer the properties remained good up to two extrusions. On the contrary, in the wet polymer the hydrolytic degradation phenomena have reduced the molecular weight and consequently all properties.

P32 BIODEGRADATION BEHAVIOR OF POLY (LACTIC ACID) BASED COMPOSITES FILLED WITH SHORT NATURAL FIBERS

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Although the use and consumption of plastics have significantly improved human quality of life, their massive use is having a huge environmental impact and it is now urgent to switch to sustainable alternatives. Biopolymers represent one of the most interesting solutions. These "green" plastics have the inherent advantage of being produced from renewable resources and, more important, they can be biodegradable or compostable. Contrary to common perception, biodegradation of such materials in the natural environment can be a very slow process, with obvious consequences in terms of their actual environmental sustainability. The challenge is shortening the degradation time as much as possible while preserving the plastic properties. In the present study, this ambitious goal is pursued by exploiting the inherent pro-degradation ability of natural fibres. To reach this goal, biopolymeric matrices susceptible to hydrolysis and hygroscopic vegetal fibres were combined into green composites. The hemicellulose fraction of the fibres acts as a water

vehicle that triggers the hydrolysis of the matrix. The shortened polymer chains can be hence mineralized by microorganisms. To exploit the full potential of the pro-degradative effect of the fibres, the optimal material-related parameters to speed up biodegradation were investigated and the properties of selected biocomposites were assessed. Polylactic acid (PLA) based composites with different volumetric percentage of natural fibres were prepared using hot processing techniques. Degradation tests were performed in water and compost, and the degradation rate was estimated by evaluating physical and chemical changes in the samples. The relevant increase in matrix hydrolytic degradation rate recorded in green composites confirms that natural fibres can be exploited to accelerate the degradation of hydrolysable polymeric matrices.

P33 EFFECT OF GNP CONTENT ON THE PHOTOOXIDATIVE BEHAVIOR OF PP-BASED NANOCOMPOSITES

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In this work the photo-oxidative behavior of neat PP and PP/GnP nanocomposite samples was studied. The influence of the amount of incorporated GnP on photo-oxidative behavior was evaluated. The photo-oxidation has been followed by monitoring the change of the rheological, mechanical and spectroscopic properties upon artificial exposure to UV-B light. The photo-oxidative behavior of polymer nanocomposites depends not only on the photo-oxidation behavior of the matrix, but also on the chemical composition and on the physical proper-ties of the nanoparticles and on the possible interactions between the two phases.
The materials used are: Polypropylene, Capilene®E50E, supplied by Carmel Olefins Ltd. GnP: Graphene nanoplatelets, trade name xGnP®, Grade C, supplied by XG Sciences Inc.
Nanocomposite samples were prepared with a GnP concentration of 0.5, 1 and 2 wt/wt% (PP0.5; PP1; PP2) using a co-rotating twin-screw extruder.
All the samples were exposed to accelerated weathering in a Q-UV chamber containing UVB-313 lamps up to 144 hours.

The rheological characterization was performed using a plate-plate rotational rheometer (ARES-G2, TA Instruments), operating at 210 °C on samples obtained by compression molding.

Mechanical tests were carried out by using a Universal Testing Machine (Instron) on rectangular shaped specimens (10×90 mm).

ATR-FTIR analyses were performed on irradiated samples using an FTIR Spectrum One Instrument (Perkin-Elmer).

The results of all the tests performed on the samples subjected to accelerated weathering revealed that the presence of GnP reduced the rate of photo-oxidation of the PP matrix. In particular, the photo-stabilizing action improved with increasing the GnP content. Indeed, EB as a function of the irradiation time decreased more slowly with increasing the GnP content. Furthermore, the rates of formation of photo-oxidation products decreased on increasing the GnP content in the samples.

The photo-stabilizing action of the GnP can be attributed to two main reasons, i.e. i) the ability of the GnP to absorb the ultraviolet energy; ii) the radical scavenging efficiency of GnP.

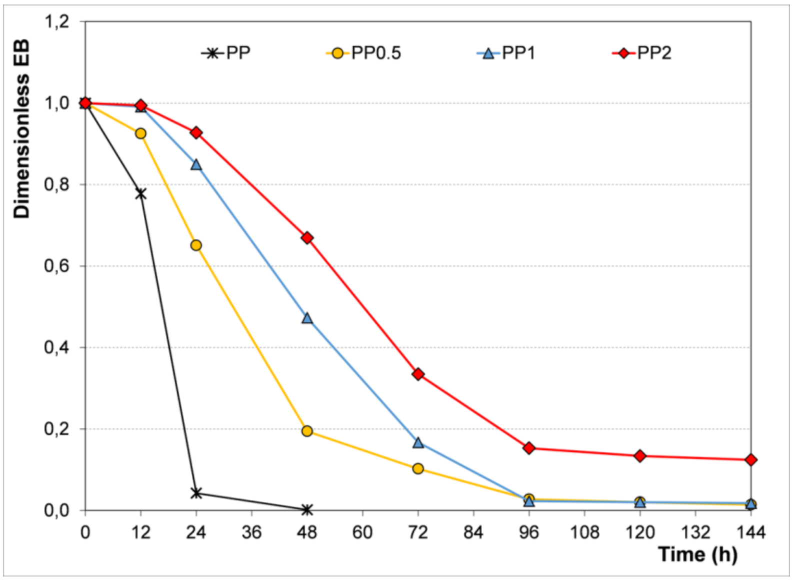

Fig 1: Dimensionless elongation at break (EB) as a function of the irradiation time.

The other results, not reported here, indicated the fast photo-oxidation of the PP, while the nanocomposite samples showed a very slow rise of the same absorption peak as a function of the irradiation time. The elongation at break as a function of the irradiation time decreased more slowly with increasing the GnP content. In particular, the presence of GnP increased both the photo-oxidation induction time and the EB-half time. The photo-stabilizing action of the GnP can be attributed to two main reasons, the ability of the GnP to absorb the ultraviolet energy that, then, is no more available for the formation of radicals and consequently for the propagation of the oxidation reactions; the radical scavenging efficiency of GnP.

P34 MICROSTRUCTURE AND SUSTAINABILITY OF CONCRETE CONTAINING RECLAIMED ASPHALT PAVEMENT AS NATURAL AGGREGATE REPLACEMENT

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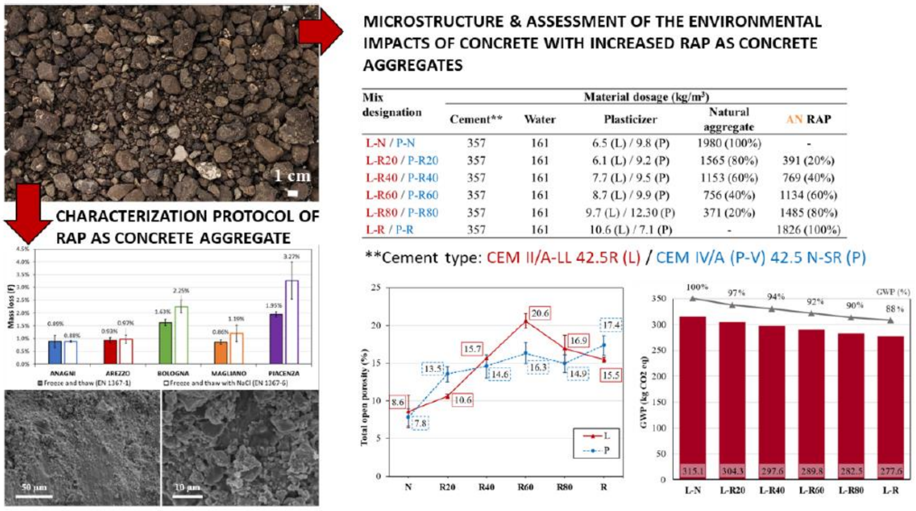
One possible route to reduce carbon dioxide (CO2) emissions of concrete, one of the most used construction materials worldwide, is to replace natural aggregate with recycled ones. Among the others, the performance of Reclaimed Asphalt Pavement (RAP), a solid waste produced by the demolition and milling of existing asphalt pavements during the road maintenance, can be assessed as concrete aggregate. To face these challenges, this study aims at reporting some results obtained in the project "Sustainable concrete made with recycled asphalt pavement (RAPCON)" (2020–2023), funded by Cariplo Foundation under the 2019 call "Circular Economy for a sustainable future" (Figure 1). A characterization protocol of RAP as concrete aggregate was developed starting from the requirements of EN 12620. Then, the microstructural properties of concrete with increasing amount of RAP were investigated.



CHARACTERIZATION PROTOCOL OF RAP AS CONCRETE AGGREGATE

MICROSTRUCTURE & ASSESSMENT OF THE ENVIRONMENTAL IMPACTS OF CONCRETE WITH INCREASED RAP AS CONCRETE AGGREGATES

Mix designation	**Material dosage (kg/m³)**				
	Cement**	**Water**	**Plasticizer**	**Natural aggregate**	**AN RAP**
L-N / P-N	357	161	6.5 (L) / 9.8 (P)	1980 (100%)	-
L-R20 / P-R20	357	161	6.1 (L) / 9.2 (P)	1565 (80%)	391 (20%)
L-R40 / P-R40	357	161	7.7 (L) / 9.5 (P)	1153 (60%)	769 (40%)
L-R60 / P-R60	357	161	8.7 (L) / 9.9 (P)	756 (40%)	1134 (60%)
L-R80 / P-R80	357	161	9.7 (L) / 12.30 (P)	371 (20%)	1485 (80%)
L-R / P-R	357	161	10.6 (L) / 7.1 (P)	-	1826 (100%)

**Cement type: CEM II/A-LL 42.5R (L) / CEM IV/A (P-V) 42.5 N-SR (P)

Figure 1. Graphical abstract reporting the rational of the study.

The assessment of the environmental impacts was also carried out applying the Life Cycle Assessment (LCA). Finally, the durability performances of a scaled-up concrete formulation with 40 wt% RAP replacement, produced in industrial environment (CTI, Imola, Italy), were investigated. It was found that due to the variability of RAP, a complete characterization is fundamental in view of its use in concrete mixes, in order to produce durable concrete and to enlarge the diffusion of circular economy. Increasing the amount of RAP in concrete promotes an increase in open porosity and, accordingly, the 40 wt% replacement was selected as the more promising content to optimise the microstructure of the hardened material. Such a replacement induced a reduction of 6% for the global warming potential (GWP) impact category, thus indicating that RAP as recycled concrete aggregate can increase the sustainability of concrete.

P34 MICROSTRUCTURE AND SUSTAINABILITY OF CONCRETE CONTAINING RECLAIMED ASPHALT PAVEMENT AS NATURAL AGGREGATE REPLACEMENT

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Alkali-activated materials (AAM) represent a new class of materials that has rapidly grown in interest in the last twenty years in order to reduce the CO2 emissions for cement and ceramic materials productions. They are based on alkali-activation of precursors able to consolidate at room or slightly higher temperatures. The possibility of using waste-based powders such as for example coal fly ashes is one of the main advantages of AAM, thus promoting a circular economy approach. Even if characterized by a lower environmental impact, AAM suffer from some drawbacks, mainly the extreme brittleness and the low dimensional stability. One of the main solutions to overcome these problems can be the use of fibres, such as steel, basalt, or polypropylene ones. Moreover, the addition of natural fibres with the scope to obtain a similar improvement could provide a huge environmental benefit. The overall effect of bamboo fibres on the properties of mortars formulated with fly ashes activated by alkaline solutions has been here investigated. The length of the fibres was varied in order to obtain the best properties of the composite materials. A reference

mortar without fibres and with the same liquid/binder and binder/sand ratio has been prepared for comparison. No heat curing has been used in all the mortars.
The consistency at the fresh state, as well as the microstructural, physical and mechanical properties at the hardened state of the obtained materials have been studied. Finally, the durability of the investigated composite materials has been investigated by means of both freeze-thaw and temperature-humidity cycles. Obtained results show a good resistance to temperature-humidity changes and freeze-thaw stresses of the reinforced mortars, thus fostering the use of natural fibres to improve the performance of low carbon footprint building materials such as alkali-activated composites in the civil engineering applications.

Figure 1. Image of a specimen of AAM reinforced with bamboo fibres after the flexural strength test

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Figure 2. Image of AAM during freeze-thaw tests

P35 EFFECT OF COATING ON CHLORIDE PENETRATION IN CONCRETE

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Reinforced concrete structures worldwide are suffering rebar corrosion causing problems to the structural safety. Especially chlorides induced corrosion is particularly dangerous, since it leads to unpredictable localized attacks, generally resulting in a fast consumption of the steel cross section. Decreasing the penetration of chlorides into concrete, may delay the corrosion initiation and so prolongate the structural service life. An efficient and feasible system to reach this purpose is the use of concrete surface coatings.

Laboratory tests were carried out to compare the effectiveness of different coatings on the penetration of chlorides. 12 concrete samples were prepared; 4 were kept uncoated as control specimens (UN) and 8 were coated by 3 different commercially available coatings: a hydrophobic pore-lining treatment (HYDRO), a polymer-modified cementitious coating (CEM) and an acrylic film forming coating (PAINT).

Unidirectional diffusion test according to EN 12390-11:2015 and chloride's migration test based on ASTM C1202-19 were performed. Figure1 shows, as an example, the results of the unidirectional diffusion test for two uncoated samples and two samples coated by the hydrophobic pore-lining treatment. The chloride diffusion coefficients were of about $25\cdot10^{-12}$ m^2/s and $1\cdot10^{-12}$ m^2/s for the uncoated and coated samples respectively. Figure 2 shows the results obtained from the chloride's migration test performed on all the coating. The uncoated samples showed a high chloride ion penetrability (according to ASTM C1202-19), while all the coated samples exhibited a moderate, low and very low chloride ion penetrability respectively for the film forming coating, the hydrophobic pore-lining treatment and the cementitious coating.

All the coatings analysed proved to be effective in reducing the penetration of chlorides in concrete and thus in increasing the durability of reinforced concrete structures.

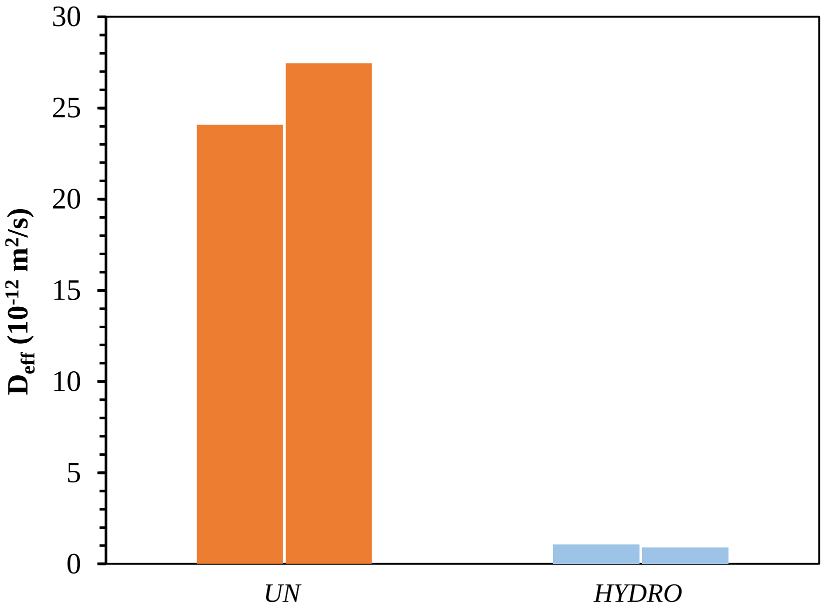

Figure 34. Apparent diffusion coefficient determined with the unidirectional diffusion test for uncoated samples (UN) and coated samples (HYDRO).

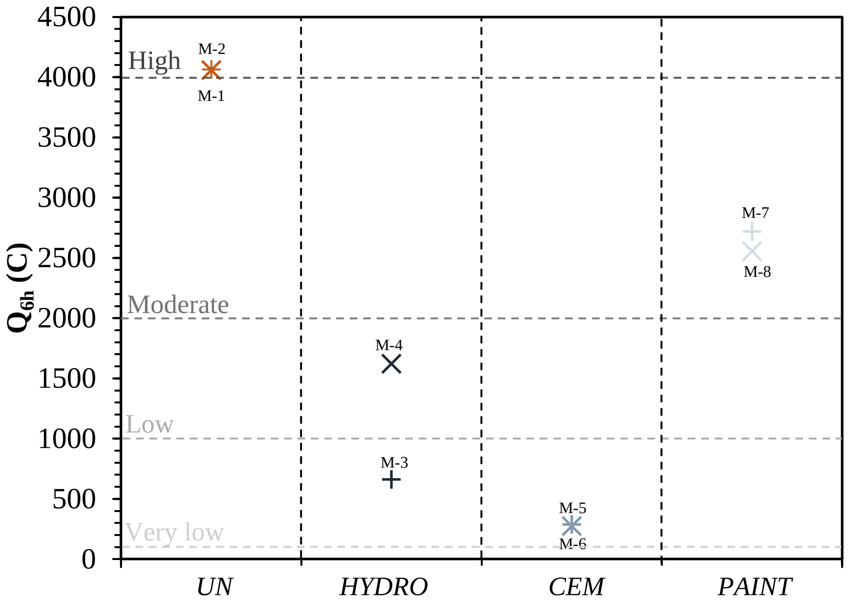

Figure 2. Results of the chlorides migration test: Charge passed in 6h and indication of chloride ion penetrability according to ASTM C1202-19.

P36 STUDY OF THE PASSIVATION OF STEEL IN CONCRETE MADE WITH RAP AGGREGATE

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RAP is a material coming from road maintenance, and it could be contaminated with chlorides (e.g., de-icing salts). Hence, it is important to study the corrosion behavior of steel in contact with concrete made with RAP aggregate, focusing on the achievement of passivity conditions of carbon steel rebars.

Cylindrical reinforced specimens were cast with two types of cement (CEM II/A-LL 42.5R and CEM IV/A (P-V) 42.5N-SR, respectively marked with L and P), two w/c ratios (0.45 and 0.65, signed with a and b) and RAP replacements to natural aggregates of 0% (N), 60% (R60), 100% (R). Immediately after casting, rebar potential was monitored while the corrosion rate was measured through LPR. After about 10 days of monitoring in laboratory conditions, the samples were demoulded, wrapped in foil and kept at T=20°C.
The corrosion current density was used to identify the passivity condition of rebars, with a threshold value of $i \leq 1 mA/m^2$.
During the first days, values of $i \geq 1 mA/m^2$ and very low potentials were detected, over the time i drops as the potential rises. In figure 1 the monitoring of these parameters through time is shown. Values for all specimens are listed in table 1 (for the sample L-b-N continuous monitoring was not possible, the first value recorded after the suspension is reported)
The time required to reach passivation conditions changed as a function of the RAP content (figure 2).
For natural aggregates (N), it is observed that the time required to reach the passivity condition is about 6÷8 days (except for L-b-N sample, for the reason mentioned before).
Moving on 100% RAP, the difference in terms of time does not seem to be so marked, in order to achieve passivity conditions, rebars needed between 9÷11days. The worst situation is represented by 60% RAP where it generally takes more than twice as long to reach the threshold limit, indeed, times about 21÷23 days were recorded.
The time required for each specimen to reach passivation is given in table 1.

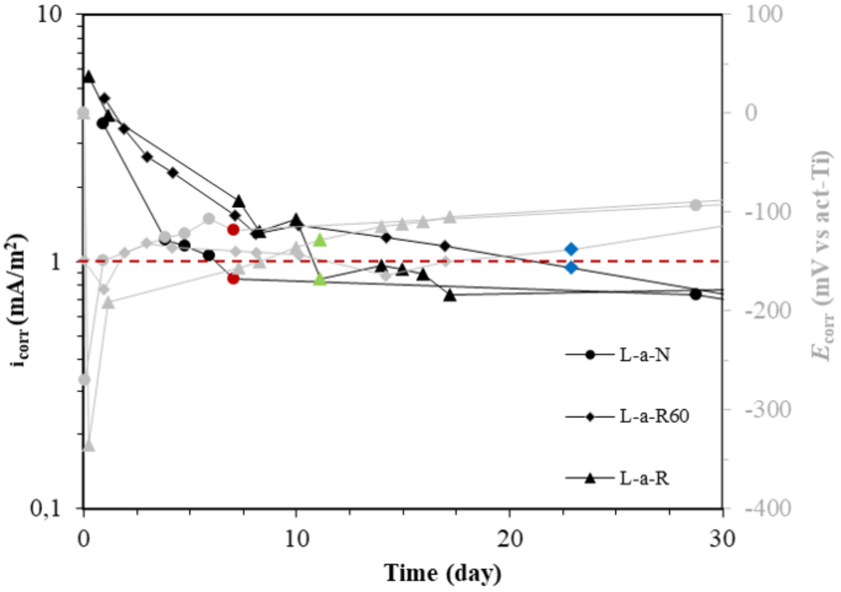

Figure 1 Potential and current density of steel for specimens with CEM II/A Il-LL 42.5R and w/c = 0.45. Colored symbols indicate achievement of passivation

Table 1 potential and current density values for all specimens

SPECIMEN	***E (mV vs act-Ti)***	***i (mA/m²)***	***time (days)***
L-a-N	*-119*	*0.85*	*7*
L-a-R60	*-138*	*0.94*	*23*
L-a-R	*-129*	*0.85*	*11*
L-b-N	*-128*	*0.93*	*29*
L-b-R60	*-148*	*0.86*	*23*
L-b-R	*-131*	*0.70*	*11*
P-a-N	*-98*	*1.00*	*8*
P-a-R60	*-129*	*0.92*	*21*
P-a-R	*-117*	*1.08*	*9*
P-b-N	*-114*	*1.00*	*7*
P-b-R60	*-143*	*0.97*	*21*
P-b-R	*-117*	*1.06*	*9*

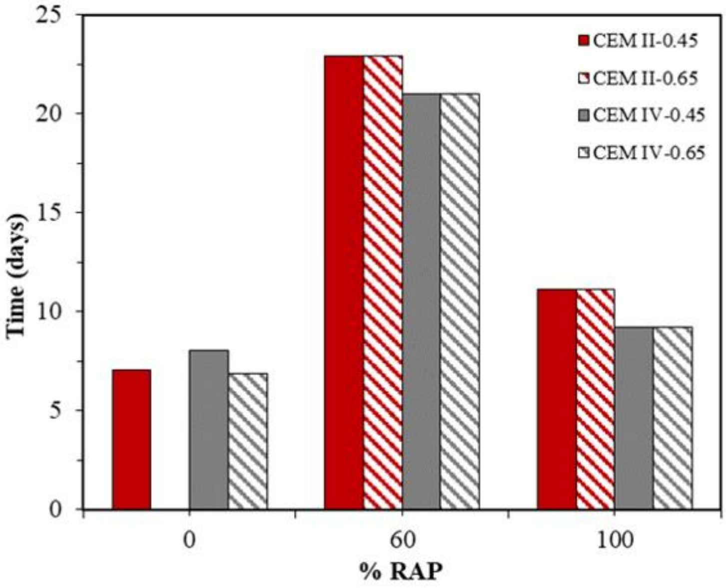

Figure 2 Time required to reach a corrosion current density i ≤ 1mA/m²

P38 EVALUATION OF ALKALI-SILICA REACTIVITY IN EXISTING RC TRANSPORT INFRASTRUCTURES

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Alkali-silica reaction (ASR) is a chemical reaction that occurs between the alkalis present in cement paste and the reactive amorphous silica commonly found in aggregates used to make concrete[1]. ASR can produce significant damage to concrete structures over time, including cracking, expansion which affect its strength, stiffness, serviceability, safety, durability, and overall stability. The experimental program herein presented was carried out in collaboration with Autostrade per Italia (Direction 7 – Pescara section) and aimed to assess the extent and magnitude of the damage produced by ASR in existing transport infrastructures. To this scope, different test methods and diagnostic methodologies currently available have been applied.

Figure 35. Concrete cores extracted from different bridges after ACPT exhibiting pop-up and gel formation.

Concrete cores were taken from existing RC bridges, especially from the piles, both from potentially intact and damaged areas. The cores were investigated petrographically, by means of X-ray diffraction analysis (XRD) and in conjunction with chemical analyses, carbonatation test and the accelerated concrete prism expansion test (ACPT) at 60 °C and RH > 95% according to RILEM AAR-4.1.The experimental outcomes, especially the residual expansion measured after 56 weeks both in longitudinal and radial directions, confirmed the potential reactivity of the aggregates in the concrete belonging to the existing RC *structures. After the expansion tests, visual analysis (Figure 1) showed typical phenomena of aggregate pop-out and silica gel formation; in most of the cases, the expansion of the concrete cores had exceeded the limits established from the actual regulations valid for new concrete structures (0,04% for UNI 11604:2015; 0,10% for RILEM AAR-0 and ASTM C1567-22; 0,20% for DD 249:1999).*

References

[1] Swamy, R. N. (Ed.). (1992). The alkali-silica reaction in concrete (No. 18002). Glasgow: Blackie.