SOCIEDAD ESPAÑOLA
DE CERAMICA Y VIDRIO

Workshop for Young Researchers in Ceramics and Glasses for Medical Applications, YouCGMed

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Book of Abstracts

Workshop for Young Researchers in Ceramics and Glasses for Medical Applications, You-CGMed

10th – 11th October 2019, Sociedad Española de Cerámica y Vidrio, Madrid-Spain

We gratefully acknowledge the support of our sponsors.

Welcome to the YouCGMed Workshop

Dear Delegates,

Since ancient times, humankind has used materials for its wellbeing and improvement in the quality of life. However, in the last decades of the 20th century, the field of biomaterials has grown rapidly. Ceramics and glasses have played a relevant role: from the first generation of bioinert ceramics, to the following generation of bioactive glasses up to the third generation of bioresorbable calcium phosphates. The appearance of nanotechnology, tissue engineering and additive manufacturing has led to a new generation of biomaterials, permitting ceramics and glasses to be developed in multiple dimensions and for many applications. Nowadays, ceramics and glasses are developed for clinical diagnosis, treatment of illness, drug delivery, 3D printing of scaffolds and bioprinting.

Now we, the young researchers, have the opportunity to further the legacy of knowledge and technology that our mentors left to us and join forces for the development of new generations of ceramics and glasses with medical applications.

This workshop has been organized to promote dialogue and the exchange of ideas, with the aim of building new collaboration networks in the field of ceramics and glasses with medical applications.

Dr Maria Canillas

Chairwoman of the YouCGMed

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Programme

THURSDAY, 10th OCTOBER	
9:00 – 9:30	REGISTRATION
9:30 – 10:00	OPENING CEREMONY
Session I: Bioglasses	
10:00 – 10:30	Plenary Speaker: Julian Jones, *Bouncy bioglass for regenerative medicine*
10:30 – 11:30	Session I
11:30 – 12:00	Coffee Break – Posters exhibition
Session II: Diagnosis	
12:00 – 12:45	Session II
12:45 – 13:45	Flash Poster Session
13:45 – 15:30	Lunch – Posters exhibition
Session III: Processing	
15:30 – 16:00	Plenary Speaker: Helene Reverone-Cabotte, *Zirconia-based ceramics for structural biomedical applications*
16:00 – 17:15	Session III
21:00	Cocktail ABC-SKY Serrano

FRIDAY, 11th OCTOBER	
Session IV: Ceramics	
9:30 – 11:00	Session IV
11:00 – 11:30	Coffee Break – Posters exhibition
11:30 – 12:00	Plenary Speaker: María Pau Ginebra, *Bioinspired strategies for bone regeneration*
12:00 – 12:45	Session IV
12:45	Closing Ceremony

THURSDAY 10th OCTOBER

9:00 - 9:30 am- REGISTRATION

9:30 – 10:00 **Opening ceremony**

10:00 – 10:30 **PLENARY SPEAKER - Bouncy bioglass for regenerative medicine** by *Professor Julian Jones (Imperial College London, UK)*

Session 1: 10:30 – 11:30

Cell biology evaluation of laser sintered HAp and 45S5 bioactive glass coatings on micro-textured zirconia surfaces using MC3T3-E1 cells (primary osteoblasts)

- J. Mesquita-Guimarães *(Centre for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro)*

Comparison between chitosan coated Bioglass 45S5 and apatite-wollastonite scaffolds for tissue engineering applications

- Djurdja Vukajlovic *(School of Engineering, Newcastle University)*

Properties of plasma sprayed bioactive glass coatings: role and control of the microstructure

- Beatriz Garrido *(Ciencia Department of Materials Science and Physical Chemistry)*

Molecular Dynamic Simulations of Bioactivity Changes Associated with the Incorporation Copper in Silicate-based Glasses for Tissue Engineering Purposes

- Mitra Soorani (*Department of Materials, Loughborough University)*

11:30 – 12:00 COFFEE BREAK – POSTERS EXHIBITION

Session 2: 12:00 – 12:45

Preparation of ceramic targets based on mixed metal oxides with magnetics and radiological properties for the production of nanoparticles by laser ablation.

- Eduardo José Félix Ruiz *(Physics of condensed matter, Science faculty of Cádiz University)*

Synthesis of B_4C powder via dynamic thermochemical method

- Hamza Boussebha *(Sakarya University Research, Development and Application Center)*

The evaluation of photophysical properties of advanced biomaterials directed on potential biomarkers of the urogenital tract cancer diseases

- Aneta Lewkowicz *(Faculty of Mathematics, Physics and Informatics, University of Gdańsk)*

Flash Poster Session: 12:45 – 13:45

High efficient strategy for the production of controlled hydroxyapatite/sericin nanocomposites

- Anabela Veiga (*Process Engineering, Environment, Biotechnology & Energy, Dep. of Chemical Engineering, Faculty of Engineering of Porto, Univ. of Porto)*

Modelling and Interpretation of Adsorption Isotherm of paranitrophenol on diatomite-based composite

- Nedjma Khelifa (*National School of Sciences of the Sea and Coastal Development BP19, Bois des cars, DelyIbrahim, Algiers, Algeria)*

Evaluation and pre-clinical trials for the development of biomaterials and cell therapies for bone regeneration in non-critical defects

- A.C. Sousa *(Departamento de Clínicas Veterinárias, Instituto de Ciências Biomédicas de Abel Salazar Universidade do Porto)*

Bioceramic blocks with graded composition for bone defects

- Helena Pereira (*Center for MicroElectroMechanical Systems, University of Minho)*

Structure, electronic, and elastic properties of Magnesium silicide Mg_2Si

- Brahim Bahloul (*Department of Exact Sciences, Ecole Normale Supérieure de Bou-Saada)*

Dissolution studies of magnesium-containing silicate glasses for bone regeneration

- Marcela Arango-Ospina (I*nstitute of Biomaterials, University of Erlangen-Nuremberg)*

Laser irradiation of zirconia surfaces: effects on microstructure and mechanical performance

- Diana Faria (*Center for Micro-Electro Mechanical Systems, University of Minho)*

Biocompatibility assessment of PEO/Sol-Gel coatings deposited on AZ31B Magnesium alloy substrates for biomedical applications

- Juan Pablo Fernández Hernán (*Área de Ciencia e Ingeniería de los Materiales, Escuela Superior de Ciencias Experimentales y Tecnología, Universidad Rey Juan Carlos)*

Tailoring hydroxyapatite bio-ceramic powder properties for selective laser sintering/melting shaping

- Pedro Navarrete-Segado (*Laboratoire de Génie Chimique, Université de Toulouse)*

3D bioprinting of cements by Laser Induced Forward Transfer (LIFT) for biomedical applications

- C. Muñoz-García *(Centro Láser UPM, Universidad Politécnica de Madrid)*

Bioactivity study of plasma sprayed 45S5 bioactive glass coatings from solid and liquid feedstocks

- Eugeni Cañas (*Instituto de Tecnología Cerámica (ITC), Universitat Jaume I)*

Study of mechanical properties and degradation study of porous magnesium alloy based bone scaffolds

- Adithya Garimella *(Mechanical Engineering, Manipal University Jaipur, Dehmi kalan)*

Elaboration and metallurgical characterization of the metal/metal multimaterials interface realized by thermal spraying

- Alya Harichane (*Département de métallurgie Université Djilali Bounaama Khemis Miliana Algérie)*

Screening of the synthesis route on the structural, magnetic and magnetocaloric properties of La0.6Ca0.2Ba0.2MnO3 manganite: A comparison between solid-solid state process and a combination polyol process and Spark Plasma Sintering

- Haithem Ben Khalifa (*Physics, sfax tunisia)*

EPD of Halloysite clay nanotubes/gelatin composites for drug deliver

- Behnam Abdollahi (*Faculty of New Sciences and Technologies, Department of Life Science Engineering, University of Tehran)*

Doping β-TCP ceramics to improve their densification and mechanical properties in use of resorbable bone substitutes

- Nicolas. Somers *(Laboratoire des Matériaux Céramiques et Procédés Associés)*

13:45 – 15:30 LUNCH AND POSTERS EXHIBITION

15:30 – 16:00 **PLENARY SPEAKER - Zirconia-based ceramics for structural biomedical applications** by *Helene Reverone-Cabotte (Centre National de la Recherche Scientifique, France)*

Session 3: 16:00 – 17:15

Novel SOL-GEL coating for local treatment of fungal infections associated with biomaterials

- Beatriz Toirac *(Materials Science and Engineering Department, Carlos III University of Madrid)*

Enhancement of mechanical properties of Ceria-Calcia stabilized Zirconia by alumina reinforcement

- Daniela Tovar Vargas *(Department of Materials Science and Metallurgical Engineering, Universitat Politécnica de Catalunya)*

Microstructural study on the interface of ceramic-metallic multi-material obtained by direct selective sintering

- Eren Ozmen *(Université de Toulouse)*

The influence of microstructure on tribological properties of stoichiometric lithium disilicate glass-ceramics

- Crislaine da Cruz *(Department of Physics, State University of Ponta Grossa)*

Fused deposition modelling method to obtain PLA/HA 3D scaffolds using a novel colloidal feedstock

- Ferrandez-Montero *(Instituto de Cerámica y Vidrio, CSIC)*

21:00 COCKTAIL ABC-SKY SERRANO

FRIDAY 11th OCTOBER

Session 4: 9:30 – 11:00

Biohybrid composite materials containing β-TCP/SrFO/ZnFO for osteochondral tissue regeneration

- Gerardo Asensio *(Instituto de Ciencia y tecnología de Polímeros, CSIC)*

3D Strontium Folate electrospun nanofibrous scaffolds for bone regeneration in craniofacial defects

- Marcela Martín del Campo *(Institute of Polymer Science and Technology, CSIC)*

Synthesis of customized bioceramic/bioglasses based scaffolds for bone tissue engineering by selective laser melting

- Nikhil Kamboj *(Tallinn University of Technology)*

Impact of ice-templating combined with indirect 3D printing on phase composition of hydroxyapatite scaffolds

- Lucie Pejchalova *(Brno University of Technology, Faculty of chemistry)*

From bio-waste to bone substitute: Biomimetic chitosan/hydroxyapatite scaffolds

- Antonia Ressler *(Faculty of Chemical Engineering and Technology, University of Zagreb)*

Gentamicin Sulfate Release from Polylactic Acid Fiber Reinforced Calcium Phosphate Cements. In vitro assessment against Staphylococcus Aureus

- Daniel Moreno *(Grupo de Investigaciones Pirometalúrgicas y de Materiales Universidad de Antioquia UdeA)*

11:00 – 11.30 COFFEE BREAK – POSTERS EXHIBITION

11:30 – 12.00 **PLENARY SPEAKER - Bioinspired strategies for bone regeneration** by *Professor María Pau Ginebra (Universitat Politècnica de Catalunya, Spain)*

Session IV: 12:00 – 12:45

Development of new biomedical devices based on polylactic acid and marine ceramics by 3D printing

- S. Pérez-Davila *(Grupo Novos Materiais, Dpto. Física Aplicada Univ. de Vigo)*

Evaluation of Direct Light Processing for the fabrication of bioactive ceramic scaffolds

- Claudia Paredes *(Departamento de Ingeniería Mecánica, Energética y de los Materiales, Escuela de Ingenierías Industriales, Universidad de Extremadura)*

Synthesis and physicochemical evaluation of b-type carbonated hydroxiapatite for bone replacement applications

- Hamilton Copete López *(Instituto de Cerámica y Vidrio, CSIC)*

12:45 CLOSING CEREMONY

Type of Presentation: Oral

Properties of plasma sprayed bioactive glass coatings: role and control of the microstructure

Beatriz Garrido[a]* Sergi Dosta[a] and Irene Garcia Cano[a]

[a]*Ciencia Department of Materials Science and Physical Chemistry, CPT (Thermal Spray Centre), c/Martí i Franqués 1, 08028, Barcelona, Spain*

The microstructure of plasma sprayed bioactive glass coatings were studied using crushed 45S5 bioactive glass powder. It is widely accepted that plasma sprayed coating microstructure is highly affected by the characteristics of the powder and the parameters set on the spraying process.

Once the 45S5 coatings were obtained, their microstructure was analyzed through scanning electron microscopy and X-ray diffraction. Additionally, mechanical properties such as coating adhesion to the substrate was evaluated for the as-sprayed coatings. Coatings have been in vitro tested to evaluate their response when culturing human osteoblasts and to assess their bioactivity by immersion in Hank's solution.

Finally, controlled cooling with carbon dioxide during spraying was also carry out in order to study the effect of this procedure on the final properties of the coating. For this analysis, characterization of splats was also considered as a test for knowing the spreading and flow behaviour of single particles.

Thus, a comparative study of the features of the coatings between atmospheric plasma spraying and controlled cooling during plasma spraying will be explored and discusse.

Type of Presentation: Oral

Comparison between chitosan coated Bioglass 45S5 and apatite-wollastonite scaffolds for tissue engineering applications

Djurdja Vukajlovic[a]* Julie Parker,a Oana Bretcanu[a] Katarina Novakovic[a]

[a]*School of Engineering, Newcastle University, NE1 7RU, Newcastle upon Tyne, United Kingdom*

A broad range of bioglass and glass-ceramic scaffolds exhibit advantageous characteristics including biocompatibility, biodegradability and antibacterial properties. Furthermore, they have the ability to bond to human bone, which makes them suitable for orthopaedic implants. Following the discovery of Bioglass 45S5 (BG) in 1969, many other bioglass and glass-ceramic scaffolds with improved mechanical properties and slow degradation rate were developed. However, until now, inadequate mechanical properties of these scaffolds, and in particular their brittleness, are their biggest drawbacks. In this work, seeking to overcome these limitations, BG and apatite-wollastonite (AW) scaffolds were coated with chitosan-based hydrogels. Chitosan was introduced as an organic part of the composite in order to improve the mechanical properties and mimic the composition of human bone. As chitosan is derived from chitin, which is the second most abundant natural polymer and a waste material in the seafood industry, its utilisation is also environmentally beneficial. Herein, highly porous BG and AW scaffolds were made by using the sponge replication method. Different heat treatments for producing scaffolds were evaluated and the most appropriate in terms of sinterability were chosen for further testing. The BG and AW scaffolds were then immersed in hydrogel solutions for different times and polymerized at 37°C. Scanning electron microscopy images of coated scaffolds showed that the coating reduces the porosity, as pores were partially covered by the hydrogel. However, the scaffolds still maintained an open porosity which is suitable for blood flow and cell ingrowth. Compressive strength values of AW scaffolds before and after coating were higher than those of BG scaffolds. However, the compressive strength reached only the lower limit for cancellous bone. BG scaffolds showed a decrease in strength after coating with the hydrogel. Conversely, the strength of AW scaffolds improved after coating. This behaviour can be explained if we consider the biomaterial's reactivity. As BG is very reactive, it gradually dissolves in the hydrogel making the coated scaffolds weaker than the uncoated ones. On the other hand, AW is less reactive than BG and the AW scaffolds increased their strength after hydrogel coating. The hydrogel acts as a glue, binding the glass-ceramic structure and improving its strength. Therefore, AW scaffolds will be further investigated for applications in tissue engineering.

Type of Presentation: Oral

Cell biology evaluation of laser sintered HAp and 45S5 bioactive glass coatings on micro-textured zirconia surfaces using MC3T3-E1 cells (primary osteoblasts)

J. Mesquita-Guimarães[a,b], R. Detsch[c], A. C. Souza[b], B. Henriques[b,d,e], F.S. Silva[b], A.R. Boccaccini[c], O. Carvalho[b]

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[e] *School of Dentistry (DODT), Postgraduate Program in Dentistry (PPGO), Federal University of Santa Catarina (UFSC), Campus Trindade, 88040-900, Florianópolis/SC, Brazil*

Laser surface texturing has been used as a topographic technique to create specific patterns on surfaces. Laser surface modification is a process associated with a coating technique that can be used to functionalize a surface with bioactive properties, stimulating cell differentiation and adhesion. This study focuses on the cell biology study of laser sintered coatings of hydroxyapatite and 45S5 bioactive glass (BG) on zirconia textured surfaces using MC3T3-E1 cells. For this purpose, zirconia surfaces were micro-textured via laser and then coated with hydroxyapatite and 45S5 bioactive glass via dip coating. Afterwards, the bioactive coatings were sintered using conventional sintering, and other groups of samples were laser sintered. The cell biology characterisation was done by determining the cell viability by performing live/dead analysis using fluorescence stains and by SEM observations for a qualitative analysis of cell adhesion. The in vitro results have shown that the functionalized laser sintered coatings do not present significant differences on cell viability when compared to the conventionally sintered ones, indicating that laser sintering of hydroxyapatite and 45S5 BG coatings is a faster and promising coating technique.

Type of Presentation: Oral

Molecular Dynamic Simulations of Bioactivity Changes Associated with the Incorporation Copper in Silicate-based Glasses for Tissue Engineering Purposes

Mitra Soorani, Jamieson K. J. Christie, and Elisa Mele

[a]*Department of Materials, Loughborough University, Epinal way, LE113TU, Leicestershire, UK*

The active surface of silicate-based bioactive glasses (SiBGs) has been widely used for dierent medical applications due to their strong active response in contact with a physiological medium [1]. The present project focuses on copper-containing silicate-based glasses (CuSi-BGs) for use in soft tissue engineering. Previously, the addition of copper ions into the basic BGs composition has been only studied experimentally to investigate their effect on biological tissue [2] [3] [4]. To the best of our knowledge, this is the first time the effect of copper on bioactive behaviour of SiBGs is studied via Molecular Dynamic (MD) simulation. Therefore, the aim of the project is to precisely describe the effect of copper incorporation into the SiBGs via classical MD simulation to establish a better understanding of the microscopic, structural basis of the bioactive behaviour with relation to the composition. The simulations are carried out on a series of SiO_2-Na_2O-CaO-Cu_2O-P_2O_5 compositions. The amounts of Cu_2O are increased to siubstitute with Na_2O. The DL POLY [5] and Atomic Molecular Massively Parallel Simulator (LAMMPS) [6] were separately used to carry out a classical MD simulation. The empirical fitting of the interatomic force fields was performed using General Utility Lattice Program (GULP) [7] code to reproduce copper aluminate $AlCuO_2$, Cu_2O, coppersilicate $CuSiO_3$, and $Na_2Si_3O_8$ to drive the Buckingham potential the potential parameters for both CuO and Cu_2O interactions. The simulations resemble the melt-quenching method. The potentials are with full charge potentials. The polarization is included via the shell-model. The partial pair distribution functions, coordination number and its distribution as analysis tools are used to study the effects of copper integration on the local environment of cations and network connectivity. Open Visualization Tool (OVITO) [8] and Visual Molecular Dynamic (VMD) [9] visualization tools are also used to localize the modifiers around the glass former cations and the structural properties of the glass.

In the next step of this project, we will focus on the fabrication and characterization of the CuSiBGs for the development of dermal scaffolds by the use of the results from the initial part. Keywords: bioactive glass; bioactivity; molecular dynamic; simulation; copper; structure properties; tissue engineering; scaffolds.

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[2] Rath, S.N., Brandl, A., Hiller1, D., Hoppe A., Gbureck, U., Horch, R.E., Boccaccini, A.R., and Kneser1, U., Bioactive Copper-Doped Glass Scaffolds Can Stimulate Endothelial Cells

in Co-Culture in Combination with Mesenchymal Stem Cells. PLoS One., 9(12), p.1-24, 3 Dec 2014.
[3] Baino F., Potestio I., and Vitale-Brovarone C.,Production and Physicochemical Characterization of Cu-Doped Silicate Bioceramic Scaffolds. Materials., 11(9), p.1-15, 24 Aug 2018.

Type of Presentation: Oral

The evaluation of photophysical properties of advanced biomaterials directed on potential biomarkers of the urogenital tract cancer diseases

Aneta Lewkowicz[a], Piotr Bojarski[a], Robert Bogdanowicz[b], Kuba Karczewski[b], Leszek Kułak[d], Anna Synak[a], Michał Mońka[a], Wiktoria Struck-Lewicka[e], Renata Wawrzyniak[e], Michał Markuszewski[e]

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The soft conditions of the sol-gel technique and the good compatibility organic molecules: 1,8-diazafluoren-9-one (DFO) allow to design and develop organic-inorganic hybrids materials to address as a luminescent probe of amino acids(potential biomarkers of the urogenital tract cancer diseases). Nowadays, for searching of potential biomarkers in biological samples, complementary analytical techniques are used like: gas chromatography (GC) and liquid chromatography (LC) coupled with different types of detectors. According to the literature, there are some biomarkers currently used in cancer diagnosis like PSA (prostate cancer antigen) in case of prostate cancer, but their efficiency as indicators of detection of cancer cell are mostly unsatisfactory [1,2]. A new way on enhancing of biomarker's signal is studied using originally developed biomaterials, wherein the potential biomarkers are incorporated. The developed biomaterials are formed during the originally procedure involving the sol-gel process [3]. Spectroscopic and structural properties of 1,8-diazafluoren-9-one in titanium dioxide thin films are studied. Particular interest was paid to the possible role of the matrix- titanium dioxide as a basic environment for DFO. The photophysical properties of DFO in the titanium dioxide thin films were identified by a variety of spectroscopic methods including: stationary absorption and emission, the fluorescence intensity decay profiles, fluorescence microscopy, spectroscopic ellipsometry, atomic force microscopy. The photophysical parameters were determined using the steady state and time-resolved spectroscopy. The signal intensity as well as life time of luminescent were depended on concentration of each potential biomarker in urine sample. Spectroscopic analysis in the range of aggregation process influencing the concentration of active biomolecules will give primary information about signal enhancement from proposed potential biomarkers. The study of photophysical properties of originally designed biomaterials would allow for obtaining of relevant information about the role of proposed biomarkers of urogenital tract cancer. Figure1.Representative fluorescence micrograph for 9-(1,8-diazafluoren-9-ylidene)amino-1,8-diazafluorenone in titanium dioxide thin films.

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and GC-MS reveals metabolite changes in prostate cancer: a pilot study. Journal of Phartmaceutical and biomedical Analyst 2015;111:351-361
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Type of Presentation: Oral

Preparation of ceramic targets based on mixed metal oxides with magnetics and radiological properties for the production of nanoparticles by laser ablation.

[a] Eduardo José Félix Ruiz, [b] Sara Román Sánchez, a Rocío Litrán Ramos, c Miguel Ángel Rodríguez, a Oscar Bomatí Miguel

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[b]*Electroceramica, Instituto de Cerámica Vidrio (CSIC), C/Kelsen 5. 28049 Campus de Cantoblanco, Madrid, Spain.*
[c]*Ceramica, Instituto de Cerámica Vidrio (CSIC), C/Kelsen 5. 28049 Campus de Cantoblanco, Madrid, Spain.*

One of the main problems in the breast cancer diagnosis is the early detection of the tumors. Therefore, there is a great interest in development of new multimodal contrast agents to help the early diagnosis. In this work, our approach has been the synthesis of new multimodal contrast agents as nanoparticle colloid dispersions, obtained from binary and ternary mixed metal oxides systems. In laser ablation in liquid, the laser beam hits the surface of a bulk material producing nanoparticles from the sublimated material, theoretically with the same composition. For this purpose, it has been studied and developed thermal treatment of the oxide mixture in order to synthetize the corresponding binary and/or ternary phases during the sintering thermal schedule. Reaction-sintering process has been used to prepare bulk materials with the appropriate phase compositions and densities to be used as precursors in laser ablation in liquid. In order to characterize our targets, it has been used XRD and SEM to identify the phase, morphology and porosity of the surface. Also, we observed the ablation energy threshold of the materials in order to know the minimum energy to sublimate the surface. The characterization of the nanoparticles was carried out by TEM, DLS, XRD and SAED to obtain the morphology, sizes of the nanoparticles and colloid dispersions, phase identification and crystalline information.

Type of Presentation: Oral

Synthesis of B_4C powder via dynamic thermochemical method

Sinan Bakan[a]; Hamza Boussebha[a*]; Betül Özdemir[a]; Ali O. Kurt[ab]

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[b]*Dep. of Materials Science, Sakarya University, Esentepe Kampüsü, 54187, Sakarya, Turkey*

Boron carbide B_4C proved itself as a ceramic of a big importance due to its great hardness. Furthermore, its physical and chemical properties, enable the use of its nanoparticles as a potential agent in the boron neutron capture therapy. Because of the effect of free carbon on the mechanical properties, as well as the chemical characteristics, a high-quality powder, with absence of free carbon, has always been desired. In addition, the extremely high synthesis temperature which exceeds 1800°C, and the further milling necessary to reduce the powder size, result an excess of energy, which proportionally increase the powder price. In this study, a reaction temperature of 1500°C for 1h was sufficient to synthesize submicron boron carbide powders from a mixture of boron oxide B_2O_3 and Carbon, via dynamic carbothermal reduction (DCR) method. XRD results showed a lower free carbon content, while SEM analysis demonstrated a reduced particle size of the as-synthesized powders.

Type of Presentation: Oral

Fused deposition modelling method to obtain PLA/HA 3D scaffolds using a novel colloidal feedstock

A. Ferrandez-Montero[1 *], P. Ortega[1], A.J.Sanchez-Herencia[1], Rainer Detsch[2], Aldo R. Boccaccini[2], B. Ferrari[1]

[1] *Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain*
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The additive manufacturing (AM) techniques are arising as the most appropriated techniques to produce customized 3D pieces with complex structures and geometries. This group of techniques has been applied in different industries but biomedicine has gained a special attention because patient-customized products are often required. Well-known AM techniques for processing of ceramics inks are binder jetting, inkjet technology, stereolithography (SLA) and robocasting. All of these methods allow printing of green samples with complex structures with a good control on composition and microstructure, like scaffolds for biomedical application. Among AM techniques, the fused deposition modelling (FDM) is one of the most simple and inexpensive techniques, which allows high printing speeds using thermoplastics as structurers.

On the other hand, hydroxyapatite (HA) is a bioceramic similar in composition to the mineral component of human bones. Several authors review its exceptional properties such as no toxicity, biocompatibility, bioactivity, osteoconductivity and good osteointegration properties. This work is aimed to produce available HA modified granules feedstock to process PLA/HA scaffolds by FDM, following a patented procedure (nº: 201830503). The thermoplastic behavior of the HA feedstock as well as the particles packing and dispersion depend on the processing additives used and mixed with HA particles through colloidal processing. The stability and dispersion of the ceramic particles is provided by their surface modification with polyelectrolytes or surfactants which also allow enhancing the interfacial interaction between HA and thermoplastic additives. This dispersion is studied by rheology as a function of the thermoplastics agent selected, PLA (Poly-L-lactic acid or Poly-L, D-lactic acid) and PEG (Polyethylenglicol). Both are thermoplastic polymers with exceptional biocompatible properties. Regarding to the hybrid feedstock homogeneity, it is possible to increase the ceramic load as high as 78wt.% without compromising the thermal stability of the polymer. Ceramic particles/Polymers composition and developed links during the thermal-shaping are studied in terms of zeta potential, ATR-FTIR, and DSC. This processing procedure leads to PLA/HA porous scaffolds suitable for the biomedical industry without modifying their biocompatibility.

Type of Presentation: Oral

Novel SOL-GEL coating for local treatment of fungal infections associated with biomaterials

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The need for innovation in developing metallic joint replacements increases every year due to the massive longevity. Among the improvements that take an active interest is adding additional functions to implants such as osseointegration or antimicrobial properties.

Even though joint prostheses lead to an improvement in the patients' quality of life, as any invasive medical procedure, complications can occasionally arise. One of the most dangerous consequences are Prosthetic Joint Infections (PJI), with important implications for the patient health and high costs for Public Health. Fungi infections are a concern for the orthopedic community due to their recent increased incidence and their resistance to antifungals. To treat them, antimicrobial agents are usually administered orally/parenterally. However, the administered dose does not completely reach the implant area, without being able to increase the dose due to the risk of patient intoxication.

Accordingly, the proposed alternative in this research is the local release of the drug by incorporating an antifungal in a sol-gel coating. The coatings can be designed with controlled biodegradability properties over time to achieve local treatment.

In this manner, in this research, new biodegradable coatings were designed, synthesized and characterized with the incorporation of antifungals and were applied on a biomedical powder metallurgical titanium (TiPM) substrate.

Coatings were prepared using as silicon precursors γ-methacryloxypropyltrimethoxysilane (MAPTMS) and tetramethoxysilane (TMOS). Two different fungicides (fluconazole and anidulafungin) were separately introduced in different concentrations. Coatings were deposited on TiPM substrates by dip-coating technique. After coating the pieces, there were subjected to a drying process. The surface, chemical stability and hydrophilic features and the electrochemical and microbiological behavior of the coatings were studied.

After the drying step, obtained coatings showed homogeneous and continuous surfaces without presence of imperfections. The antifungals introduction to the sol-gel synthesis did not affect the obtained thicknesses (around 10 μm) nor the hydrophilic nature of coatings. The thermal characterization showed that the antifungal agents interfere in the network formation, obtaining more mass loss as the fungicides' concentration increases due to the less formation of siloxane bonds. For the electrochemical study, samples were exposed to physiological conditions similar to the human body. After 24 hours of exposure to the

physiological medium, most specimens were completely covered or almost completely covered by the sol-gel coatings without revealing the metallic substrate (TiPM). However, the increase of antifungal concentration leaded to a greater deterioration of the coatings. The biofilm formation was analyzed in coatings containing fluconazole, resulting that the antifungal concentration positively influenced the decrease in the biofilm development.

This work concludes that new biodegradable organic-inorganic hybrid sol-gel coatings loaded with antifungals were synthesized. In addition, coatings loaded with fluconazole have antifungal properties to coat titanium-based joint prostheses.

Type of Presentation: Oral

The influence of microstructure on tribological properties of stoichiometric lithium disilicate glass-ceramics

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Friction, lubrication and wear are present in the bucal environment due to the relative movement between teeth in the presence of saliva production. Teeth wear occurs naturally and depends on several factors. However, wear can be increased by the introduction of foreign bodies in the bucal environment. Glass-ceramics are used as dental materials since 1972. Mica-based glass-ceramics were the first machinable glass-ceramics used for odontological purpose. Other glass-ceramics such as those based on lithium disilicate, apatite, leucite and diopiside were developded. Among these, lithium disilicate glass-ceramics (LS2) presented hardness and translucency similar to natural teeth. The greatest advantages of LS2 are the good aesthetic and the high fracture toughness that enables three-teeth bridges prostheses. Previous works have shown how LS2 tribological properties changed with environment and surface finish. There are no systematic investigation about the relationship between its tribological properties and its microstructure. Looking at this literature gap, this study investigates the microstructure influence on the tribological properties of LS2 for odontological applications. LS2 glass was prepared by melting and it was crystallized using a double stage heat treatment for nucleation and crystal growth. Glass-ceramics with different crystallized volume fractions and crystal sizes were prepared. The crystalline volume fraction varied from glass to a 98 % crystallized sample for samples with 8 mm crystal size. For samples with 64 ± 1 % of crystalline volume fraction, the crystal size varied from 8 to 34 mm. The tribological properties were investigated using a pin-on-disc tribometer with an alumina sphere, applied load of 5 N and sliding speed of 5 cm/s at normal atmosphere conditions. The coefficient of friction (COF) was measured continuously up to 10.000 sliding turns. The surface profiles of the worn tracks were measured using a UNAT instrumented indenter. Scanning electron microscopy was used to analyze the surface morphology. The specific wear rate of each sample was calculated from the profile measurements. The first results show that microstructure have no influence in COF. On the other hand, the specific wear rate increased with crystal size and decreased with crystalline volume fraction. For all samples, the wear regime was severe, except for the sample with 98 % of crystalline volume fraction and 8 mm of crystal size, who presented a mild wear regime. The main wear mechanism was due to fatigue wear, except for the sample with 98 % of crystalline volume fraction, who presented abrasive wear.

Type of Presentation: Oral

Enhancement of mechanical properties of Ceria-Calcia stabilized Zirconia by alumina reinforcement

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Zirconia ceramics stabilized using 10 mol % CeO_2 and 1 mol % CaO were tested with the addition of small amounts of α-alumina. The elaboration process of five different compositions was done by wet mixing of powders using 0, 2.5, 5, 10 and 15 wt. % alumina, followed by pressing and sintering. A smaller grain size of zirconia was obtained with a well-dispersed alumina phase. This resulted in an increase in hardness and mechanical strength, measure by ball in three ball bending. The hydrothermal degradation resistance was also improved by the addition of alumina, finding only up to 6% of monoclinic volume phase after 60 hours.

Type of Presentation: Oral

Microstructural study on the interface of ceramic-metallic multi-material obtained by direct selective sintering

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The present study focuses on the microstructural analysis of interfacial ceramic-metallic zone of bi material pieces printed by direct laser beam sintering method with a commercial 3D Systems machine. During this work, firstly metallic parts were obtained by selective laser sintering of aluminum-silicon (ca. 12% Si) alloy powder. Then, fully yttria-stabilized zirconia powder (ZrO_2-$8Y_2O_3$) was used to directly print the ceramic part on top of the metallic one. The laser absorbency of YSZ powder at the Nd:YAG laser wavelength was increased up to 60% by adjunction of small amount of graphite powder to the ceramic powder blend, what enables us to manufacture YSZ parts with relative density of 96.5 %. Investigations of the micro-structure (by XRD) and chemical composition (by microprobe analysis) layer by layer from the metallic to the ceramic materials were conducted to assess the inter-diffusion of joining materials, the melting-sintering mechanisms and the phase transformations. SEM and TEM observations of this interfacial zone allowed describing the grains changes in size, crystallographic characteristics and composition. The study shows an integrated interfacial zone in which unchanged metallic phases are surrounded by ceramic ones but also reduction of the grain size as well as elemental diffusion of aluminum and yttrium occur.

Type of Presentation: Oral

From bio-waste to bone substitute: Biomimetic chitosan/hydroxyapatite scaffolds

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Human bone consists 65-70% of inorganic phase (calcium phosphates and trace elements), 20-25% of organic phase (primarily collagen) and 5-8% of water [1]. Biological hydroxyapatite in its structure contain various trace elements, such as CO_3^{2-}, Mg^{2+}, Na^{+}, K^{+}, Zn^{2+}, Sr^{2+}, which have crucial role in bone growth. Incorporation of mentioned ions in synthetic HAp crystal lattice can affect its crystallinity, morphology, lattice parameters, thermal stability, solubility and phase composition, which can significantly improve biological properties of HAp bioceramics [2]. The aim of the present work was comparative study of physiochemical and biological properties of obtained chitosan/hydroxyapatite scaffolds, where hydoxyapatie was obtained from three different biogenic sources. As a source of calcium ions biogenic calcium oxide, derived from hen eggshells, sea shells and cuttlefish bone was used.

The synthesized powders have been characterized by elemental analysis, Fourier transform infrared spectrometry, X-ray diffraction, and Rietveld refinement studies. Highly porous chitosan/hydroxyapatite composite structures have been prepared by freeze-gelation technique, while morphology of scaffolds was imaged by scanning electron microscopy (SEM). The as-prepared powders were composed of calcium deficient hydroxyapatite and amorphous calcium phosphate. Heat treated powders were composed of hydroxyapatie, α-tricalcium phosphate and β-tricalcium phosphate. In as-prepared powders Sr^{2+}, Mg^{2+} and Na^{+} ions were detected as result of using biogenic precursor of Ca^{2+} ions. SEM observations of cross section and surface area of prepared hydroxyapatite/chitosan scaffolds have shown highly porous structure with very well interconnected pores and homogeneously dispersed hydroyapatite particles. The MTT assay of scaffolds with hydroxyapatite obtained from different biogenic sources has shown no toxicity and the live dead staining has confirmed good viability, as well as proliferation of seeded cells by the culture time.

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Type of Presentation: Oral

Evaluation of Direct Light Processing for the fabrication of bioactive ceramic scaffolds

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For the past recent years, the use of different Additive Manufacturing (AM) techniques to fabricate porous substrates has been widely studied. Developing AM scaffolds from materials with bonelike properties that are capable to interact with the tissues and actively induce bone regeneration has permitted overcoming the main issues of currently available procedures for repairing large bone lesions, namely: the limited amount of material and need of secondary surgical sites in the case of autografts, risk of immunogenic response and disease transmission from donor in the case of allografts, and fixation problems of current bioinert prostheses.

In this work, the suitability of the additive manufacturing technique, digital light processing (DLP) is studied in order to obtain bioactive scaffolds for bone regeneration. To this end, ceramic suspensions were made from acrylic resins filled with β-tricalcium phosphate (TCP) powder, and porous structures consisting of a tetragonal three-dimensional mesh of interpenetrating struts with squared section were obtained and characterized, both microstructurally and mechanically. The influence of the variation of the strut / pore size were be evaluated in terms of mechanical properties under compression stresses.

The results of this study provide valuable insight into the mechanical behavior of scaffolds for bone tissue engineering applications fabricated with DLP, and pave the way for future work aimed at optimizing the fabrication and mechanical performance of complex structures.

Type of Presentation: Oral

Gentamicin Sulfate Release from Polylactic Acid Fiber Reinforced Calcium Phosphate Cements. In vitro assessment against Staphylococcus Aureus

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The properties of calcium phosphate cements allow it to be used as a drug carrier to treat bone affections. However, one of the major drawbacks is the burst release that may occur, limiting the release time, thus different strategies have been investigated to overcome this issue. In this work Calcium phosphate cement, derived from alpha-Tricalcium phosphate (α-TCP), was reinforced with L-polylactic acid (PLLA) fibers and loaded with gentamicin sulfate (GS) in order to improve the mechanical behavior and to present a more sustained GS release with effective growth inhibition of Staphylococcus aureus (S. aureus).

PLLA was added as 5% in volume of the samples and GS was added in 10 or 20 mg/g of α-TCP. Samples were prepared at a liquid to powder ratio of 0.44 ml/g. As controls, two cements were prepared, one with α-TCP and water and a second one with α-TCP, water and PLLA fibers. Physicochemical characterization of the different cements prepared was performed and effect of the GS concentration and PLLA fibers incorporation on the release kinetics of the antibiotic in PBS at 37°C was determined by UV-VIS absorption. Also the effectiveness of the GS release was in vitro assed against S. aureus by measurement of the inhibition halo in agar plate and confocal microscopy of samples cultured with the bacteria for 16 h.

Results showed that fibers and GS does not change the mechanical strength, nevertheless conditions where PLLA fibers were added increased the energy absorbed before rupture. In addition, GS slightly affects the precipitation of calcium deficient hydroxyapatite crystals. It was noticed that PLLA fibers addition has a positive effect as it slows down the GS release kinetics and that such release is effective to avoid the bacteria colonization of the samples. In the control conditions where no antibiotic is added, the S. aureus highly adhered and covered the samples.

Type of Presentation: Oral

Biohybrid composite materials containing β-TCP/SrFO/ZnFO for osteochondral tissue regeneration

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Text: Musculoskeletal degeneration commonly results in the development of injuries such us osteoarthritis and arthritis rheumatoid, eventually leading to progressive articular destruction especially at subchondral level. To provide an effective regenerative therapy for its treatment, it is necessary to design a composite material that recreates the hierarchical microstructure of the osteochondral interphase, which is highly calcified at the subchondral bone and rich in collagen and glycosaminoglycans at the articular surface1. Hence, we present the fabrication of a hybrid scaffold as tissue replacement consisting of a 3D porous support made of PLGA, PEGDMA and β-TCP as a ceramic component that mimics the bone layer, and provided as well with a photopolymerized HAMA hydrogel on the top that mimics the cartilage region. Besides, folic acid derivatives (SrFO and $ZnFO)_2$ are included in the formulation as they have been reported as non-protein bone growth factors.

SrFO and ZnFO were synthesized by hydrothermal reaction between folic acid and the corresponding metal halide. Biomimetic scaffolds were fabricated by a two steps manufacturing process: first, the 3D support was elaborated by cryopolymerization of PLGA and PEGDMA in the presence of SrFO and β-TCP finely grinded. Then, the biomimetic hydrogel was elaborated by deposition and infiltration of a HAMA solution on the scaffold followed by irradiation with a UV light lamp. Composition, microstructure and in vitro behaviour of scaffolds were determined by ESEM-EDS and ICP/UV-VIS analysis. Biological role of SrFO and ZnFO on hMSC was evaluated in terms of Ca and GACs deposition and ALP kinase expression. Biocompatibility of the scaffolds was studied with Alamar blue assay. hCs and hOB cells were micro-seeded in the scaffolds and stained with Vibrant and Höesch staining respectively to examine the colonization capacity. Scaffolds were fixed with formaldehyde, embedded in paraffin and subsequently sliced with a microtome sectioning equipment .The sliced samples were stained with H&E and by using anti-CD44 and D2-40 primary antibodies, for podoplanin and CD44 protein immunohistochemical detection.

Synthesis of folic acid derivatives and their coordination mode was confirmed by ATR-FTIR. SrFo and ZnFO exhibited potential to induce hMSC differentiation by the deposition of Ca and GACs as well as an up regulation of ALP expression in the early stages of bone formation. 3D supports presented interconnected pores and a uniform distribution of β-TCP. ESEM analyze revealed different swelling capacity for each zone of the scaffold evidencing its ability to host different cell populations. In vitro studies showed a sustained release of SrFO and ZnFO over 21 days in biologically active concentrations without citotoxicity. Gradient composition obtained allowed hCs and hOB co-culture to selectively colonize the scaffold after 10 days, which was corroborated with the histochemical staining. SrFO and ZnFO were successfully synthesized and exhibited promising properties as bone growth factors. Hierarchical scaffolds fabricated showed gradient composition and microstructure with the capacity of host selectively different cell populations through the structure.

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Type of Presentation: Oral

Synthesis and physicochemical evaluation of b-type carbonated hydroxiapatite for bone replacement applications

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The carbonate content in biological bone is about 2-8 wt% according both function in body and age of the individual, the carbonate ion occupies phosphate ion sites in apatite lattice of stoichiometric Hydroxyapatite (HA) (Ca/P ratio = 1,67), this substitution is classified as B-Type. Therefore, synthesizing calcium phosphates whose composition is similar mineral phase of the bone becomes a great alternative for bone substitution applications. In this work, hydroxyapatite synthesis was developed through the inverse method with different carbonation levels by controlling carbonate/phosphate ratio (C/P) of precursor solution with PO_4^{3-} and CO_3^{2-} ions, which is drop by drop added to Ca^{2+} ions solution, controlling parameters: inert atmosphere (to avoid carbonation by carbon dioxide gas in the atmosphere), temperature, pH, maturation time of the formed precipitate and concentration of the reactants (Ca, PO_4^{3-} and CO_3^{2-}). The materials obtained were characterized by Infrared Spectroscopy (FTIR), X Ray Diffraction (X), Thermogravimetric Analysis (TGA-DTA) and carbon element analysis, finding that the synthesized phase is Hydroxyapatite, which presents the antisymmetric vibration (v_3) and flexion (v_2) characteristics of the B-type substitution. Mechanical characterization was performed in terms of uniaxial compression (ASTM C1424) and degradation studies were performed in an acidic medium, the results found that the mechanical behavior and degradation of Carbonated Hydroxyapatite makes it suitable for designing bone substitutes. The next steps of investigation are to develop macropore scaffolds for cell colonization and infiltration of gentamicin charged chitosan to provide mechanical stability and prevent biofilm growth of osteomyelitis promoter bacteria (*Staphylococcus Aureus*).

Keywords: Carbonated Hydroxyapatite, Synthesis, Mechanical Behavior, Degradation

Type of Presentation: Oral

Impact of ice-templating combined with indirect 3D printing on phase composition of hydroxyapatite scaffolds

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Hydroxyapatite is one of the most frequently used materials in regenerative medicine of bone tissue. This material provides important properties such as bioactivity and biocompatibility. However, a pore size of at least 300 μm together with sufficient micro and nano-porosity is necessary for the integration of bone cells. Bioceramic scaffolds prepared from hydroxyapatite by combination of indirect 3D printing and ice-templating provide good mechanical properties and multiscale porosity. Ice-templating utilises controlled freezing of ceramic suspension to form lamellar structure which is caused by the growth of ice crystals in the direction of temperature gradient. In the second step of the process, ice crystals are removed by lyophilisation to prevent damage of the lamellar structure. Addition of 3D mesh forms a net of macro channels with defined size within the whole scaffold. Final scaffolds, containing the 3D mesh, are sintered to burn out the mesh and reinforce the structure. There are many aspects affecting resulting structure, including freezing schedule, suspension concentration, used additives, etc.
Porosity, mechanical stability, and phase composition of scaffolds was measured. Resulting scaffolds have tailored ratio between porosity and mechanical stability necessary for bone replacements. Changes in the phase composition of ice-templated scaffolds were also observed after sintering. Various compositions contain hydroxyapatite, β-TCP or their mixture were observed. Sintering temperature, mixing and freezing conditions are crucial parameters for the final phase composition, which could also be beneficial in tailoring scaffold's biological properties.

Type of Presentation: Oral

3D Strontium Folate electrospun nanofibrous scaffolds for bone regeneration in craniofacial defects

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Craniofacial defects generally cause significant negative impacts on the quality of life and self-esteem of those individuals with musculoskeletal dysfunctionalities. Advances in regenerative medicine have arrived, giving new hope to patients that can benefit from new tissue engineering therapies based on the supportive action of biomaterials together with the synergic action of osteo-inductive molecules and recruited stem cells that can be driven to the process of bone regeneration. As the same time, many polymers or polymer-based composites are contemplated as an alternative for bone fixation due to their biocompatibility, high strength-to-weight ratio, radiolucency, biofunctionality, and nontoxicity of degradation by-products. Polylactid Acid (PLA) and their copolymers have been approved for human clinical uses, PLA has already been used for craniofacial fracture. In the same way, another group of polymers, polyhydroxyalkanoates, represented by 3- hydroxybutyric acid (PHB) and its copolymers, gained a fixed place in the biomedical field, due to their biocompatibility, biodegradability, and physical and mechanical properties. PHB scaffolds are highly compatible with osteoblast and can induce ectopic bone formation and it was speculated that PHB patches or PHB in form of composites could be an interesting examination object in the treatment of bony defects. Moreover, nowadays, electrospinning technique has been paid particular attention because of its capability to make nanofibers with morphological structures similar to those in natural extra cellular matrix, furthermore, it is able to process various polymer materials, and the resulting electrospun nanofibers are cost-effective and favorable for a variety of cellular functions. In another hand, strontium is an element currently known to stimulate osteoblasts promoting osteogenesis both in vivo and in vitro, while conversely, down-modulates function in osteoclast preventing bone resorption. The Sr-based-systems seem to be a useful alternative for the regeneration of bone tissue in complicated defects, for example, the Strontium Folate (SrFO), is a recently developed non-protein based bone-promoting agent with interest in medical and pharmaceutical fields due to its improved features in comparison to current therapies for bone diseases. Hence, it is important to develop innovative strategies for making biologically and clinically relevant 3D electrospun nanofibrous scaffolds with desired morphological/structural properties capable of including osteoinductive molecules in order to regenerate bone tissue of craniofacial defects. The aim of the current study was to evaluate the effect of PLA-PHB/SrFO 3D composites on the osteogenesis process on cranial defects.

Type of Presentation: Oral

Synthesis of customized bioceramic/bioglasses based scaffolds for bone tissue engineering by selective laser melting

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The repair and regeneration of load-bearing defects by bioceramic/bioglasses scaffolds remain a significant clinical challenge resulting from disease or trauma. The needs for repair and regeneration are one of the key assets for the synthesis of the scaffolds for load-bearing repair applications. The criteria include (1) porous structure, (2) geometry, (3) biodegradability, (4) biomolecules delivery, (5) and mechanical competence, which is generally seen as a problem.

Herein, we developed silicon-bioceramic/bioglasses based composite scaffolds with high strength via selective laser melting which can be easily tailored with shape and size to the diseased or injured area avoiding the binder addition, post-processing stages with single step technology and no geometric constraints. As a result, the scaffold with a circular pore size of 400 um and porosity of approximately 35.2% exhibited a high compressive strength of 110 MPa (cortical bone defects). Moreover, finite element simulation results were in good agreement with the experimental results. The as obtained scaffolds significantly expresses osteogenic genes (RUNX2, OSAD and OCN) and moreover the inflammatory factors were also studied onto the scaffolds. Additionally, local delivery of Vancomycin and bone morphogenetic proteins (BMPs) were also studied on the scaffolds and promising results were observed. These findings demonstrate that novel composite scaffolds obtained by selective laser melting can pave the way for treating dental and maxillofacial defects too for large load-bearing repair applications.

Type of Presentation: Oral

Development of new biomedical devices based on polylactic acid and marine ceramics by 3D printing

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Currently, 3D printing techniques are in full swing as they facilitate the rapid manufacture of prototypes of medical devices, getting into personalized medicine. In the particular case of biomedical engineering, three are the main application and research axes: biomodels for the preparation of surgical interventions, design and manufacture of customized biomedical devices and porous scaffolds for regenerative medicine.

Among the 3D printing biomaterials, polylactic acid is widely used for clinical applications since it is biodegradable, bioabsorbable and biocompatible. On the other hand, compounds based on calcium phosphate (CaP) are highly interesting for applications in the field of biomedicine, in particular, for the regeneration of bone tissue, favouring bioactivity and osseointegration.

In this work, a new bioceramic and biocompatible filament is developed from several compositions based on polylactic acid and F-enriched hydroxyapatite of marine origin. The physico-chemical properties of both the filament and the 3D printed devices were studied by scanning electron microscopy, x ray diffraction, Raman and IR spectroscopy. The analyses show that the bioceramic particles are uniformely distributed in the polymer matrix and their incorporation can be controlled depending on the selected mixture.

As well, being the PLA a thermolabile polymer of very low thermal resistance (60-70ºC), a detailed investigation on the effects of different sterilization methodologies on their properties were carried out. 3D printed samples have been submitted to conventional methods of clinical sterilization, such as the autoclaving and the gamma radiation, and to the innovative one by supercritical CO2 (conditions of pressure and temperature of CO_2 above its critical point). A biological evaluation was also carried out, for assessing that possible modifications of the material properties do not induce changes in the biological response that could compromise the biocompatibility of future biomedical products.

Type of Presentation: Poster

Evaluation and pre-clinical trials for the development of biomaterials and cell therapies for bone regeneration in non-critical defects.

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In recent years, the average life expectancy of the European population has increased and so as the number of diseases and bone fractures [1,2]. Therefore, the development of synthetic bone substitutes has arisen as a major research interest in the need to find an alternative to autologous bone grafts [3]. Using an ovine model, in the present pre-clinical study, we present a synthetic bone graft (Bonelike®) in combination with a cellular system of human dental pulp stem cells (hDPSCs) as an alternative for the regeneration of non-critical defects.
Before the pre-clinical tests, the in vitro biological behavior of Bonelike® (250 μm <Φ <500 μm) samples was evaluated in terms of cell adhesion to the material, cell viability/proliferation, and ability to support cell differentiation (osteointegration of the biomaterial). The in vivo tests are performed in 12 healthy skeletally-mature Merino sheep (Ovis aries) to evaluate the biomaterial' behavior in the presence of hDPSCs, in non-critical defects, and implantation times of 30, 60, and 120 days.
Results showed that Bonelike® and hDPSCs treated defects showed improved bone regeneration compared to the defects treated with Bonelike® alone. Also, it was observed

that the biomaterial matrix was reabsorbed and gradually replaced by new bone during the healing period. We therefore propose Bonelike® and hDPSCs as an efficient binomial strategy that promotes bone growth and vascularization in non-critical bone defects.

Type of Presentation: Poster

Study of mechanical properties and degradation study of porous magnesium alloy based bone scaffolds

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Commonly used metallic biomaterials, although are suitable as load bearing implants, they pose serious limitations in terms of 'stress shielding', non-degradability and high density. Magnesium (Mg) and its alloys, in this regard have shown promise, as Mg has similar mechanical properties when compared with a natural bone. Magnesium is biocompatible, a key feature of a bone implant and it is biodegradable, which can potentially avoid the revision surgery on the patient. But the disadvantage of Mg based implant is its high rate of degradation. Structurally, human bone has extracellular matrix with a porous microstructure, reinforced with a nano-bioceramic. So, to bio-mimic this extracellular porous matrix, Mg alloy based bioactive nanocomposites were developed in this study. Nano-fluorcanasite (n-FC), a novel bioactive ceramic has been used both as nano-reinforcement and in enhancing bone tissue regeneration. The porosity in Mg alloy nanocomposite is anticipated to act as supply channel for nutrients to newly formed bone tissue and also to remove waste products from it, in addition to reducing the weight of the implant. This study investigated the effect of incorporating selective metallic elements and nano-bioceramic towards controlling the rate of degradation of the implant. These Mg alloy based nanocomposite implants were prepared using powder metallurgy route in which carbamide was used as a space holder particle. The fabricated samples were evaluated for mechanical properties such as compressive strength and Young's modulus. The results from our study established satisfactory and tunable mechanical properties of the samples. Degradation rate after immersion in simulated body fluid (SBF) was measured at regular intervals by weight loss approach. The study revealed that the rate of degradation of magnesium alloy based nanocomposite samples could be systematically controlled as opposed to their unmodified counterpart. Overall, It could be concluded that these Mg alloy based nano-composite biomaterials have outstanding potential that can be used as a degradable implant materials for hard tissue replacements.

Type of Presentation: Poster

Elaboration and metallurgical characterization of the metal/metal multimaterials interface realized by thermal spraying

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Thermal spraying is one of the methods used for both combating wear and increase corrosion protection in several engineering field. Despite of its wide spread industrial use, little is known about thermal fatigue cycles consequence on physico-chemical and mechanical behavior of the interface. In this work, we have studied the formation of metal/metal multimaterials using the thermal spraying technique. A Cr–Ni stainless steel (55E) was deposited on two aluminum alloys (AG3 and AU4G) which are commonly used in the aeronautical industry. In order to improve adherence, a thin Ni–Al bond coat (75E) was deposited in aluminum alloys before thermal spraying. Physico-chemical and mechanical characterizations of the interface, using Scanning Electronic Microscopy (SEM), Vickers indentation and ultrasonic techniques were achieved and the influence of temperature, thermal fatigue cycles and the post treatment on adhesion properties was highlight.

Keywords: aluminum alloys; Thermal spraying; multimaterial; thermal fatigue; adhesion.

Type of Presentation: Poster

High efficient strategy for the production of controlled hydroxyapatite/sericin nanocomposites

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Hydroxyapatite (HAp) is the main constituent of the inorganic component of hard tissues, which makes synthetic HAp widely used in bone tissue engineering. Special attention has been given to nano-HAp because of its similarity in composition with bone. However, the use of HAp has limitations due to its low toughness and poor mechanical resistance, which limits the clinical applications of this material [1]. HAp/Protein composites can accommodate a broad spectrum of functional requirements, such as strength, degradability and bioactivity [2]. In this context, sericin (SS) has shown an improvement in cell proliferation when used as an organic matrix [3]. Its combination with HAp may thus promote bone formation mechanisms. The reported approaches used to obtain HAp/SS nanocomposites are based on alternate soaking and precipitation, being the last the simplest route for the synthesis of nano-HAp. Meso-oscillatory flow reactors (meso-OFR) provide precise control of mixing, having proved to result into significant enhancement in particle mixing in multiphase systems [4], making them good candidates to promote ideal conditions for the controllability of HAp/SS properties. In the present work, synthesis of HAp/SS nanocomposites was achieved through a precipitation process in a stirred tank batch reactor (ST) and in a meso-OFR. Different concentrations of sericin were studied. Monitoring of the experiments was done by measuring the pH of the reaction medium over time. The particles obtained were evaluated by their purity, crystallinity, size and morphology. In the pH profiles of the ST different stages are observed, which may suggest the presence of intermediate calcium phosphate phases. Regarding the meso-OFR, it is not possible to distinguish different stages. The formation of the HAp/SS nanocomposites is therefore achieved in less time, being approximately four times faster. FTIR analysis displayed the presence of most of the bands attributed to HAp and SS. The formation of nano-HAp with poor crystallinity was confirmed by XRD, especially when SS concentration increased, which may be advantageous in the interaction with bone cells, in an in vivo scenario. SEM and TEM images show that the particles obtained in both reactors are similar in size (nm) and morphology, adopting a rod- and plate-like shape. Furthermore, an increase in SS concentration was associated with the formation of more plate-shaped particles, with increased size. This works makes important contributions in the understanding of the biomineralization process of HAp in the presence of SS.

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Type of Presentation: Poster

EPD of Halloysite clay nanotubes/gelatin composites for drug deliver

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This work describes the incorporation of halloysite clay nanotubes (HNT), as nanocarriers of strontium renalate (SrR), into gelatin (GN) films to tailor a controlled release system of an anabolic bone-forming agent to stimulate bone growth. It has been demonstrated that thermal gelling of the Fish Gelatine de Lapi can be used to improve films shaping on cooling by using electrophoretic deposition (EPD). Then suspensions composed by HNT/GN (1/2 wt/wt) were formulated. Zeta potential determination was used to fit the preparation conditions of HNTs–SrR in terms of electrostatic loading, as well as the NHT-SrR stabilization in aqueous GN suspensions by adding Polyethilenimnine (PEI) as dispersing agent. EPD kinetics was determined for suspensions prepared with and without SrR, by adjusting the bath temperature to force the crosslinking of the gel structure during the cathodic deposition. The microstructure of the prepared films was studied by comparing the drying conditions, as well as the SrR release.

Type of Presentation: Poster

Structure, electronic, and elastic properties of Magnesium silicide Mg_2Si

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The crystal structure, electronic, and elastic properties of antifluorite Mg2Si (Magnesium silicide) have been studied by first-principles calculations within DFT. These properties are critically discussed to evaluate the development of modern Mg based biocomposites and alloys for biomedical applications. Magnesium silicide is used in biomedical applications such as bone fixation, cardiovascular stents, hip joints, screws/pins, and dental implants.

The optimized structure has been obtained from the GGA total-energy minimization, where we used the Hartwigzen–Goedecker–Hutter scheme to generate the norm-conserving nonlocal pseudopotentials, in order to study the effect of the exchange-correlation energy in the bulk structure. Indeed, in the beginning, we have calculated the band energy, Debye temperature, elastic constants and related quantities such as the sound velocities, Young's modulus, anisotropy factor and Poisson ratio. The structural parameters such as the lattice constants a0, bulk modulus and corresponding pressure derivatives have been computed and were found to be in good agreement with the existing experimental and theoretical data. The formation energies (ΔH) have been calculated according to the reaction equations as following:

$2MgH_2+Si \rightarrow Mg_2Si +2H_2$
$2\ Mg + Si \rightarrow Mg_2Si$

The electronic structures of Mg_2Si presented an indirect band gap (Γ – X) at pressure (P= 0, 5, 10 GPa). This band gap decrease with increasing pressure.

Type of Presentation: Poster

3D bioprinting of cements by Laser Induced Forward Transfer (LIFT) for biomedical applications

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The main objective of this work is to adapt Laser Induced Forward Transfer (LIFT), a well-known laser direct writing technique for material transfer, to cure cements (calcium aluminates base) onto acceptor substrate.

LIFT is a promising technique in different biomedical fields due to it is a fast, clean and non-contact direct-write technique that allows the deposition of small volumes of a wide range of materials in a very precise and controlled way. This technique has been used to print in a wide range of viscosities, from solid phase to Newtonian and non-Newtonian fluids.

In this work, we present first results using this approach to transfer cements of great interest in biomaterials applications. The calcium aluminate cements are used in dentistry for endodontics.

A layer of synthetized cement with thickness in the order of 100 µm is applied over a glass substrate using a blade coater. In the glass with the deposited cement paste is set a controlled gap over the acceptor substrate. A solid-state pulsed laser emitting at 532 nm is focused on the glass/cement interface producing a droplet of paste that it is transferred to the acceptor substrate.

The process parameters (cement paste thickness, gap and laser parameters -spot size, pulse energy and overlapping of pulses) are modified and the morphology of the voxels is studied using confocal microscopy.

Keywords: Laser Direct Write, Laser Induced Forward Transfer, cement paste.

Type of Presentation: Poster

Laser irradiation of zirconia surfaces: effects on microstructure and mechanical performance

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Zirconia (ZrO_2) is a ceramic biomaterial that has been increasingly used as a metal substitute for biomedical applications including implants (e.g. dental implants, hip and knee prostheses) and prosthesis structures (e.g. dental crowns) due to its biocompatibility, mechanical properties and wear resistance [1]. As a bioinert material, ZrO_2 has been the subject of several studies, showing a significant improvement in its properties upon surface modification [2]. Several techniques can be used to perform the surface modification of zirconia-based materials [2]. Among them, laser technology has demonstrated to be a viable alternative to overcome the conventional existent methods, since it is versatile and environmentally friendly. Laser, as a direct energy source, acts over ZrO_2 by removing content from its surface and altering the material property through changes in its configuration [3]. However, it has been reported that when using laser to modify the surface of ceramics, microcracks and residual stresses are introduced, due to locally induced thermal mismatches, besides the surfaces typically presenting a blackening effect [4]. In light of the mounting concerns expressed over the use of laser to modify zirconia surfaces, a systematic and comprehensive evaluation of its effects on zirconia seems to be inevitably required. In this sense, the purpose of this study is to full characterize the surface of zirconia irradiated by laser through a systematic and comprehensive evaluation of the microstructural and mechanical changes. For this purpose, green ZrO_2 compacts were pressed at 200 MPa for 30 s and sintered at 1500ºC for 2h. After that, a Nd:YAG laser (SISMA, Italy) with a maximum working power of 40 W and a maximum spot size of 2 mm was employed in shot mode for welding. Different shot modes were explored and in each of them the laser parameters like power, duration of pulse, frequency of repetition and spot size were maintained constant. Each irradiation pulse created a circular black pit surrounded by a circular raised rim with a sunken depression at the center, such that an assembly of irradiated black pits covering the whole ZrO_2 surfaces were created. The surface of the samples was characterized using Scanning Electron Microscopy and X-ray diffractometry. Additionally, the aging resistance and the hardness of the samples were also investigated.

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Type of Presentation: Poster

Bioactivity study of plasma sprayed 45S5 bioactive glass coatings from solid and liquid feedstocks

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The use of plasma spraying to obtain high performance coatings is increasing. A wide range of both metallic and ceramic materials can be deposited with this method. During the last decades, there has been an evolution in the type of feedstock (solid or liquid) used for deposit the coating with this technique. Traditionally, materials were injected into the plasma jet in powder form, but nowadays the use of particle suspensions and precursor solutions is increasing. This evolution is due to the possibility of working with smaller particle size distributions and the properties of the resulting coatings, which are better than those shown by the coatings obtained from powder feedstocks.

The present research deals with the bioactivity study of 45S5 bioactive glass coatings deposited by plasma spraying from different feedstocks. Coatings were obtained from powders, particle suspensions and solutions of glass precursors, all of them with a composition close to the 45S5 bioactive glass. The spraying parameters used to deposit each feedstock were optimised in previous works of the authors. Once deposited, coatings bioactivity was assessed by immersing them in Simulated Body Fluid (SBF) for 1, 7 and 14 days. After each time, the pH of the SBF was measured and the samples were removed from the SBF and gently rinsed with distilled water. Then, the nucleation and growth of the hydroxycarbonate apatite layer (HCA) onto the coating surface was monitored by X–ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM) and energy–dispersive X–ray microanalysis (EDX). Furthermore, cell tests were carried out with some of coatings, using MG–63 osteoblasts–like cells. The plates containing the coatings with cells were incubated inside at 37 ºC in humidified atmosphere of 10% CO2 in air for 24 hours.

Results showed that, although all coatings developed a layer of HCA onto their surface, the nucleation rate was different in each coating, being this rate affected by the microstructure of each coating which in turn is function of the type of feedstock employed. Furthermore, related to cell tests, there was a good contact, adhesion and growth of cells onto coatings surfaces proving an adequate interaction between the deposited material and the cells.

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Type of Presentation: Poster

Screening of the synthesis route on the structural, magnetic and magnetocaloric properties of La0.6Ca0.2Ba0.2MnO3 manganite: A comparison between solid-solid state process and a combination polyol process and Spark Plasma Sintering

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La0.6Ca0.2Ba0.2MnO3 ceramics are prepared by an original route, combining soft chemistry and Spark Plasma Sintering, within a few minutes at 700 °C and by the solid-state reaction at high temperatureswith an annealing temperature of 1200 °C. We have studied the leverage of the powder synthesismethod on the structural, morphological, magnetic and magnetocaloric properties of the samples. X-ray diffraction analysis using Rietveld refinement revealed that our materials crystallize in the rhombohedral system with R3-c space group for the sample prepared by the Polyol-Spark Plasma Sintering method and in the orthorhombic structure with Pbnm space group for the sample synthesized by the solid-state reaction. Magnetization measurements versus temperature under magnetic applied field of 0.05 T show a paramagnetic-ferromagnetic phase transition for both samples. The Arrott plots reveal that ours materials undergo a second-order phase transition. The maximum values of the magnetic entropy change (-ΔSmax M) under the magnetic field change of 5 T are 2.4 and 4.7 J/kg K for La0.6Ca0.2Ba0.2MnO3 synthesized by using solid-state reaction and Polyol-Spark Plasma Sintering methods respectively. The highest value of the relative cooling power RCP is found to be 244 J/kg for the Polyol-Spark Plasma Sintering sample under 5 T. These results are interesting enough and suggest that the Polyol-Spark Plasma Sintering synthesis method is a feasible route to prepare high quality perovskite material for magnetic cooling application.

Type of Presentation: Poster

Bioceramic blocks with graded composition for bone defects

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The design of innovative solution for the treatment of bone diseases should be inspired on the structure and function of biological systems. Bone is a hierarchical and complex tissue with an amazing ability to regenerate itself, though, when the size of the fracture is critical surgical interventions are needed. Autografts and other bone substitutes, including allografts, xenografts or synthetic biomaterials are commonly used to induce bone regeneration. Autografts are still the gold standard treatment, however, their availability is limited [1]. Calcium-phosphates such as Hydroxyapatite (HA) and Beta–tricalcium-phosphate (β-TCP) are found in natural bone and have been used in bone substitutes to stimulate bone regeneration[2, 3]. These biomaterials present several advantages, specially its biocompatibility and biodegrability avoiding problems like transmission of diseases which is a risk in xenograft and allografts. Calcium-phosphates present different biodegradabily rates and completely resorbs within 6 to 12 months, being gradually replaced by new osseous tissue [4, 5]. By combining different calcium phosphates it is possible to achieve an optimum balance between the least soluble phase (HA) and the most soluble phase (β-TCP) aiming to obtain controlled dissolution and resorption [3].

The main objective of the present work is to produce a block with gradation in composition of different calcium-phosphates namely HA and β-TCP. This compositional gradation allows to orientate the dissolution rate in different regions, allowing to customize the bone substitute according to the host bone defect and location.

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Type of Presentation: Poster

Biocompatibility assessment of PEO/Sol-Gel coatings deposited on AZ31B Magnesium alloy substrates for biomedical applications.

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Magnesium alloys are interesting as biomaterials because of their properties. Magnesium has the lowest value of elastic modulus of all structural metals, closer to that of bone tissue. It presents a low-density value and a weight/resistance ratio better than aluminum and steel. Furthermore, magnesium is a bioresorbable material. For these reasons, magnesium and magnesium alloys are feasible candidates to be used as biomaterials in implants and equipment for bone fracture treatments. However, the main drawback of magnesium is its low corrosion resistance, which could lead to a decrement in the mechanical integrity of implanted parts and accumulation of evolved hydrogen that could lead to necrosis of surrounding tissues and finally the failure of the implant.

Different strategies are used in order to improve magnesium resistance to corrosion processes. Among these strategies, we have focused on coatings to develop an effective method to isolate the metal substrate from the aggressive medium, achieving a reduction of evolved hydrogen derivated from the magnesium corrosion process and increasing the service life of the implant. These coatings consist of monolayer and multilayer plasma electrolytic oxidation and Sol-gel coatings.

It is very important to assess the biocompatibility of these coatings, in order to obtain good cellular viability and subsequent osseointegration of the implant.
In this study, six different conditions were evaluated: control substrate, sol-gel monolayer coating, sol-gel with graphene nanoplatelets monolayer coating, plasma electrolytic monolayer coating, plasma electrolytic coating and a sol-gel multilayer coating, and finally plasma electrolytic coating and a sol-gel with graphene nanoplatelets multilayer coating.

C2C12 mouse myoblasts were used to assess the biocompatibility of the different coatings developed. C2C12 cultures were deposited on the different coatings and DMEM medium was used as a growth medium for cells. Photographs were taken every 24 hours using fluorescence microscopy to assess the cellular viability and adhesion on the surface of the different coatings. Alamar Blue test was also carried out to assess the metabolic activity of cultures deposited on the different coatings.

Coated samples show improved cell viability and adhesion on the surface of the assessed material, among these, multilayer coatings show the best response, been plasma

electrolytic oxidation and sol-gel multilayer coating which shows the best behavior in contrast to control samples.

Type of Presentation: Poster

Dissolution studies of magnesium-containing silicate glasses for bone regeneration

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Bioactive glasses are known to be promising biomaterials for applications in bone regeneration due to the interactions with body fluids during the dissolution process. The incorporation of therapeutic ions in the glass compositions is meant to enhance osteogenic and angiogenic properties of the material. Magnesium is a therapeutic ion known for promoting cell adhesion and stimulating bone formation [1]. ICIE16 bioactive glass is a melt-derived silicate glass originally designed by Elgayar et al [2], the main advantage of this composition is the larger sintering window compared to the well-known 45S5 bioactive glass while keeping a relatively high apatite formation rate (bioactivity). In this study, undoped and doped ICIE16 glass were produced via melting and the release of therapeutic ions during dissolution in different media (tris(hydroxymethyl)aminomethane (TRIS) buffer and simulated body fluid (SBF)) was evaluated with ICP-OES. Additionally, experiments about the in vitro bioactivity of the glasses were carried out in SBF and characterized using Scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The study demonstrated the great potential of ion doped and undoped ICIE16 bioactive glasses for regenerative applications.

[1] Hoppe A., et al., A review of the biological response to ionic dissolution products from bioactive glasses and glass-ceramics, Biomaterials 2011, 32(11) 2757-2774.
[2] Elgayar, I., Aliev, A. E., Boccaccini, A. R., & Hill, R. G. (2005). Structural analysis of bioactive glasses. Journal of Non-Crystalline Solids, 351(2), 173-183.

Type of Presentation: Poster

Modelling and Interpretation of Adsorption Isotherm of paranitrphenol on diatomite-based composite

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The main objective of this work is to study the mathematical modeling of the adsorption of paranitrphenol (PNP) in aqueous solution on diatomite-based composite. This composite consisting of diatomite (Dia) and activated carbon (C) was prepared using a mixture of diatomaceous earth and glucose. Adsorption results were processed using the Langmuir, Elovich, Freundlich, Temkin, Hill and De Boer equations to estimate the nature of the adsorption and to determine various equilibrium parameters. : the maximum adsorption capacity, the adsorption energy, the interaction energy, the adsorbate-adsorbent balance constants and the possible interactions between adsorbed molecules, thus to estimate the nature of the adsorption. It is verified that the application of the Temkin and Langmuir relations is satisfactory for the compound under study, the results obtained using the other models show that there would be no complex formation or interaction between the adsorbed molecules.

Key words: modeling, models, adsorption isotherms, paranitrophenol, diatomite, composite material.

Type of Presentation: Poster

Tailoring hydroxyapatite bio-ceramic powder properties for selective laser sintering/melting shaping

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Text: Stoichiometric hydroxyapatite (HAP) bio-ceramic is used as a model for the inorganic component of bones and teeth, thanks to the ability of its surface to provide nucleating sites for precipitation of apatite crystals in culture medium and in body fluids. Powder bed fusion (PBF), also known as selective laser sintering/melting (SLS/M) as additive manufacturing (AM) technique, allows the shaping of bio-ceramic parts with complex structures and ideal properties, like bioactivity and biocompatibility, through the accurate densification of a bio-ceramic powder bed. It would make possible the production of specific patient-matched implants leading to better performance and more cost-effective treatments. [1] However, a deeper study of the properties of the bioceramic raw materials is crucial when using the SLS/M technique. Specific optical and morphological properties of the powder bed are still required to produce a suitable heat/matter exchange between the particles to trigger the material densification. [2] The improvement of the laser radiation absorption by mean of absorption additives (also called, energy transferring vectors) and the adaptation of the powder morphology (particle size distribution and shape) are crucial to avoid the formation of cracks during SLS/M process. [3] [4]

In this communication, a description of the whole process for the production of a powder bed is discussed. Starting from the precipitation of the stoichiometric HAP powder, going through the adaptation of the powder properties by mean of wet grinding, spray drying, and additive mixing steps and finishing with the first tries of shaping stoichiometric HAP bio-ceramic parts in a SLS/M equipment. The powder bed here described would be suitable for its use on a SLS/M apparatus equipped with a continuous Nd: YAG laser (λ = 1.06μm).

The flowability of the HAP powder produced, required for its use in a SLS/M process, was analyzed by measurement of the compressibility as well as the angle of repose of the powder. Commercial graphite (TIMREX KS44) was used as absorption additive improving the laser-material interaction of the powder bed, reaching a value higher than a 30% of absorption for a 2.5% mass of graphite. Particle size distribution and shape of the agglomerates were evaluated by granulometric analysis and scanning electron microscopy (Figure 1 and 2). It can be observed that a smaller particle size distribution is obtained through the wet grinding process required to improve the performance of the following spray drying step. Finally, the effect of using a combination of dispersing agent/binder during the multistep process on the HAP micro-spherical agglomerates size and structure was clarified.

REFERENCES

[1] Hwa, Lim Chin, Srithar Rajoo, Alias Mohd Noor, Norhayati Ahmad, and M. B. Uday. Current Opinion in Solid State and Materials Science 21, no. 6 (December 1, 2017): 323–47.
[2] Tolochko, Nikolay K., Yurii V. Khlopkov, Sergei E. Mozzharov, Michail B. Ignatiev, Tahar Laoui, and Victor I. Titov. Rapid Prototyping Journal 6, no. 3 (September 2000): 155–61.
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[4] Colin, C., Bartout, J.-D. & Shaker, E. OSSEOMATRIX PATENT - WO2014154901A1.

Type of Presentation: Poster

Doping β-TCP ceramics to improve their densification and mechanical properties in use of resorbable bone substitutes

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2 LMCPA - Laboratoire des Matériaux Céramiques et Procédés Associés
Boulevard Charles de Gaulle, 59600 Maubeuge, France
3 GIS TECHCERA – Groupement Intérêt Scientifique Transfrontalier sur les céramiques (BE -FR)

β-tricalcium phosphate (β-TCP, β-Ca3(PO4)2) is one of the most attractive biomaterials for bone repair since it shows an excellent biological compatibility, osteoconductivity, and resorbability. It can be used to produce bone implants serving as temporary supports for bone regeneration. Even if it is already used in the market under granules or preforms for bone filling, there are still some issues with β-TCP porous scaffolds. As their mechanical properties are weak, they do not allow an use in large bone defects or in load-bearing areas. The major drawback in the manufacturing of these scaffolds is the difficulty to densify the β-TCP. Indeed, the tricalcium phosphate presents a phase transition to α-TCP at 1150°C which occurs with a large lattice expansion causing microcracks and reducing shrinkage during sintering with as consequence a sintering temperature limitation. Thus, this phase transformation is a drawback for the densification and so the mechanical properties of the material.
A possible solution is to dope the β-TCP with cationic dopants that replace the calcium within the β-TCP lattice and increase its thermal stability. Indeed, cationic substitution can allow reaching higher relative density value and higher mechanical properties. Moreover, dopants can also improve biological properties of β-TCP as bone implant.
In this work, in order to prevent the phase transformation and increase its sintering temperature, the β-TCP is synthetized by coprecipitation of Ca(NO3)2 and (NH4)2HPO4 solutions under controlled temperature and pH. The cationic dopants are added into the reagent solutions to optimize the substitution. Complete characterizations are conducted to evaluate the influence of these dopants on the thermal stability and biological properties of β-TCP powders. In addition, conventional and microwave sintering are compared in terms of microstructural and mechanical properties

Type of Presentation: Poster

New aproach to tailoring fused deposition modelling method to obtain HA-based 3D scaffolds.

A. Eguiluz [1], A. Ferrandez-Montero[1], P. Ortega[1], A.J.Sanchez-Herencia[1], B. Ferrari[1]

This work is aim to develop an additive manufacturing (AM) method to process porous hydroxyapatite-based scaffolds with high potential in the field of medicine. Over recent years, additive manufacturing (AM) techniques have gained a special attention in order to process complex structures and patient-customized pieces for biomedical application. One of the most known technique is Fused Deposition Modeling (FDM), which highlight as an inexpensive method, simple and fast. However, FDM present the disadvantage of being limited to process thermoplastic materials, therefore it is restricted to polymer feedstock or polymer-based composites with very low amount of inorganic load dispersed in the matrix. In recent works (Patent Nº201830503), a new route to process composite materials with a high ceramic content or 100% ceramics has been developed. This novel process combines the use of FDM technique with the feedstock processing by colloidal techniques.

In this work, different Hydroxyapatite-based (HA) materials have been developed, using polylactic acid (PLA) and polyethilenglycol (PEG) as processing agents that provide the thermoplasticity necessary to these materials for FDM technique requirements. We propose to process HA-based materials through the mixture of a suspension of HA particles with a solution of PLA. Aqueous suspensions of HA particles were prepared preserving the chemical stability of their surface by modifier adsorption. The good dispersion of the particles in the polymeric medium has been guaranteed by the study of rheology in dissolution. Additionally, the extrusion and 3D printing parameters have been determined by melting rheology. Finally, the thermal sintering process has been developed by TDA-TA and DSC to obtain 100% HA.

Development of new biomedical devices based on polylactic acid and marine ceramics by 3D printing

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Dept. Applied Physics
Universidad de Vigo, Galicia, Spain

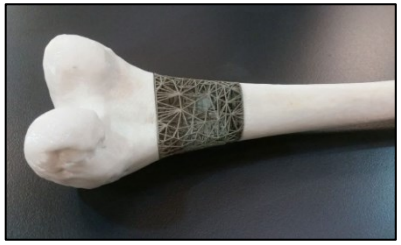
Universidade Vigo

Current 3D Printing Applications

Biomedical applications

Biomodels /surgical guides

Scaffolds for regenerative medicine

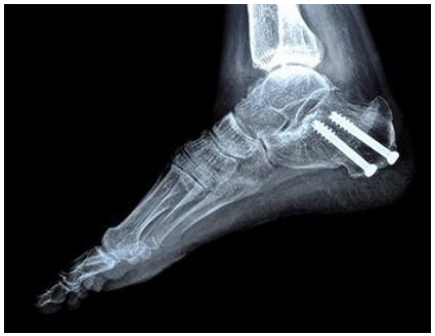


Medical devices & prosthetics

A need for personalisation and rapid manufacture

Biomaterials for 3D printing

Polylactic acid (PLA)



Biodegradability, bioabsorbility & biocompatibility

Widely used in clinic: sutures, screws…

Thermolabile polymer → Choose the correct sterilization method

Combinated with…

Calcium Phosphate (CaP)

Source: Shark teeth (fishing discards)

Bone tissue regeneration (similar composition) → **Bioactivity**
Osseointegration

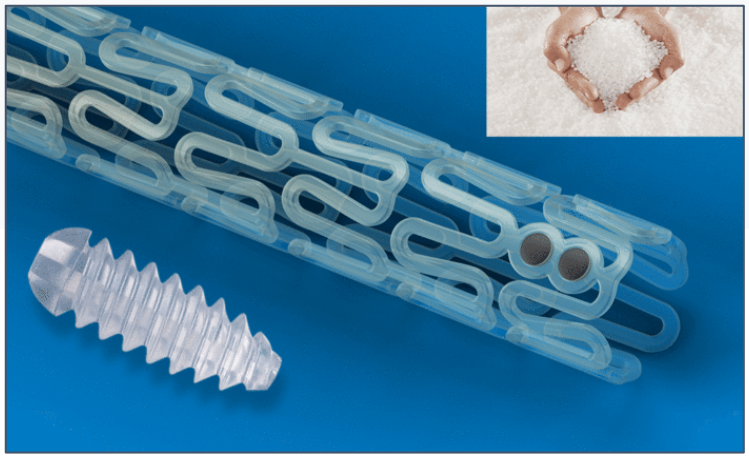
López-Álvarez, M.; Pérez-Davila, S.; Rodríguez-Valencia, C.; González, P. y Serra, J. (2016). The improved biological response of shark tooth bioapatites in a comparative in vitro study with synthetic and bovine bone grafts. Biomedical Materials 11 (3): 035011, 13 pp.

Objectives

- Manufacture of filaments with different bioceramic-polymer proportions
- Validate its use for 3D printing
- Detail the effects of different sterilization methodologies of PLA in:

  -The physicochemical properties

  -The biological response



Materials and methods: Filament manufacturing & 3D printing


PLA pellet


-80ºC


Grinding


Sieving


granulated PLA


Shark teeth

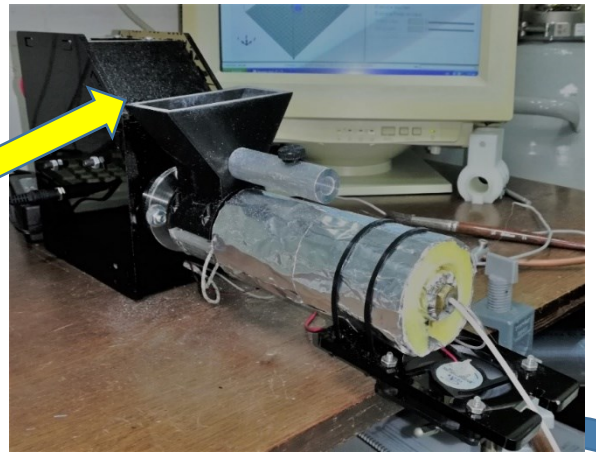

Grinding


Pyrolized

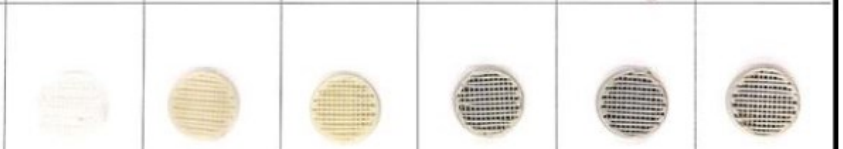

Sieving


granulated Bioceramics

Extruder

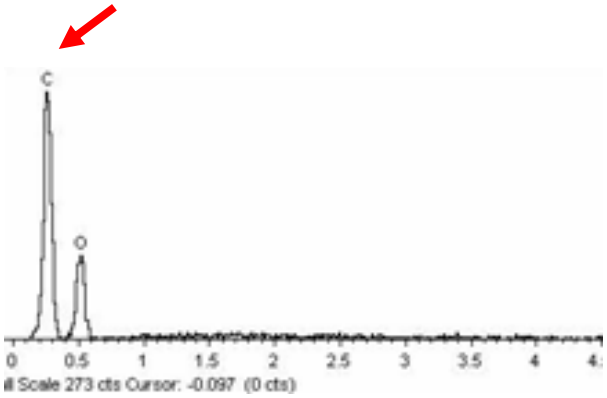


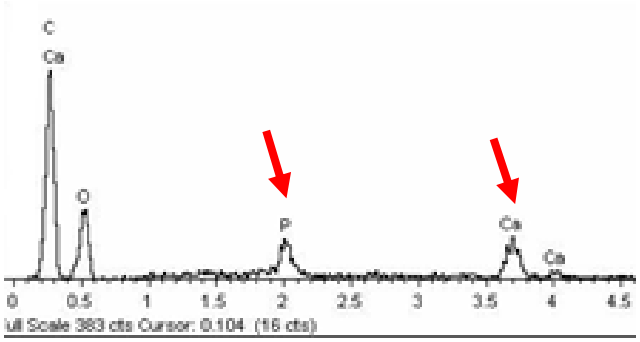

3D Printer (FDM)

% Bioceramic	0%	3%	5%	20%	30%	40%
Filament						
3D Printing						

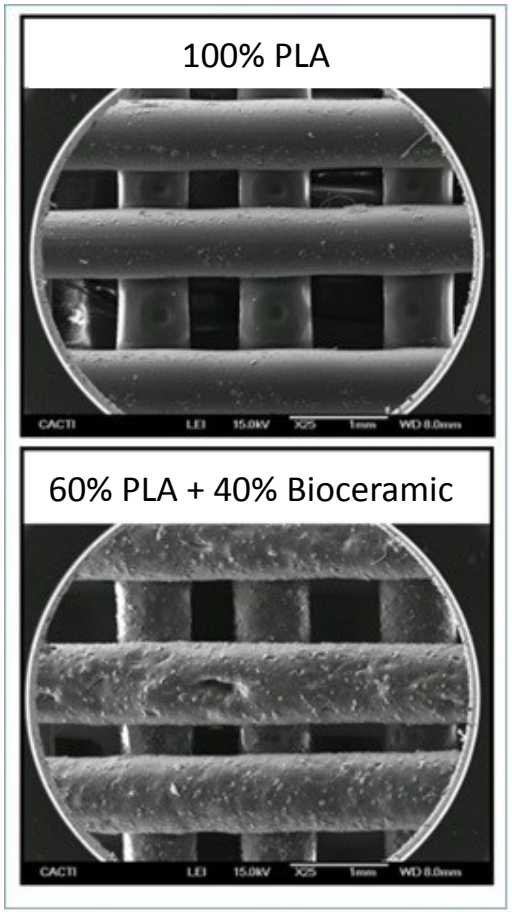
Results: Physical-chemical characterization

EDS

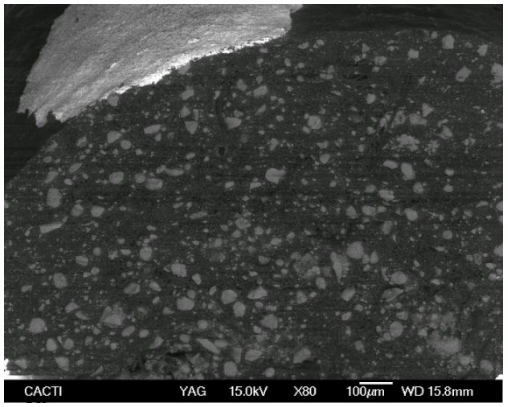




Presence P and Ca



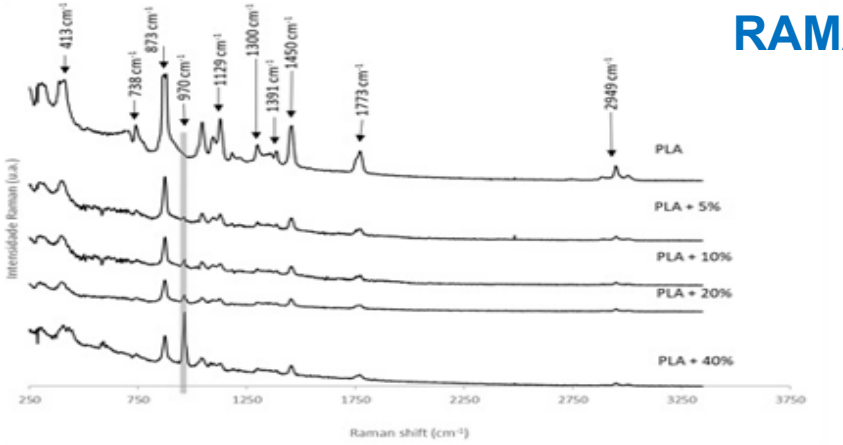
SEM

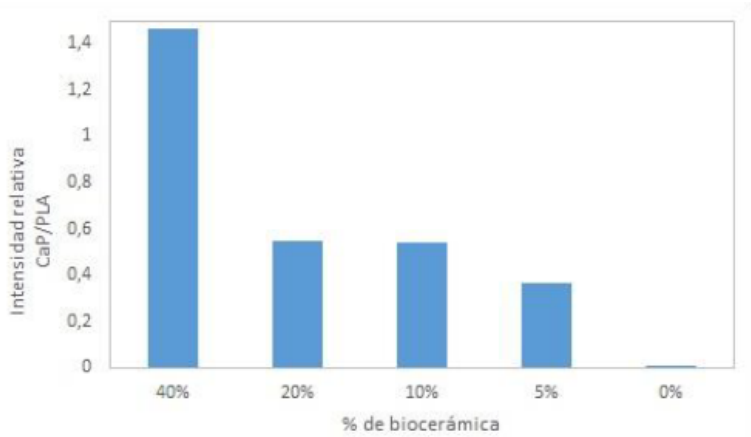


Bioceramics are distributed through the polymer matrix
Roughness increases

Results: Physical-chemical characterization

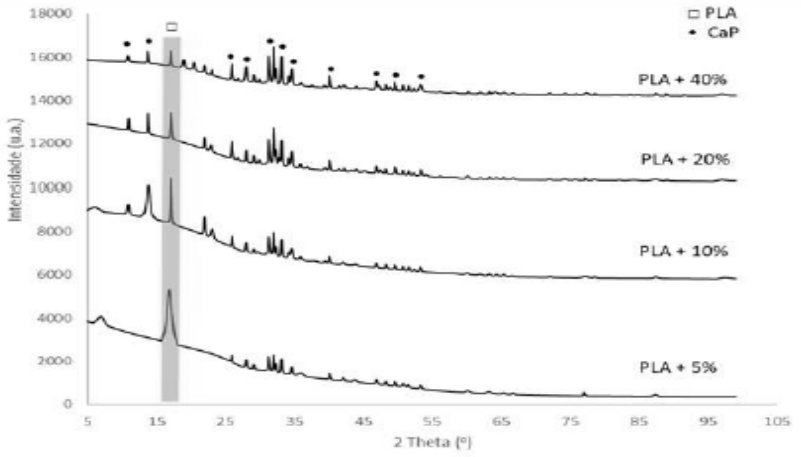
RAMAN





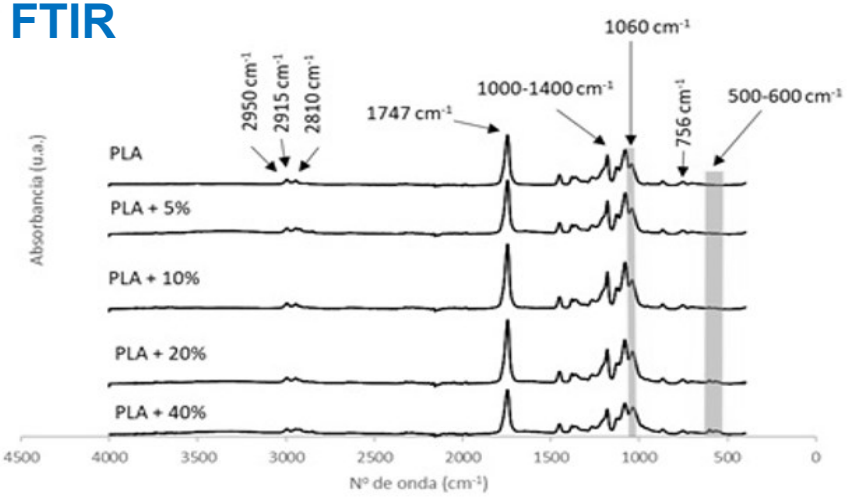
Increase in the relationship CaP/PLA with the increase in the % of the bioceramic

XRD



Decrease in the PLA peak as the percentage of bioceramic increased

FTIR

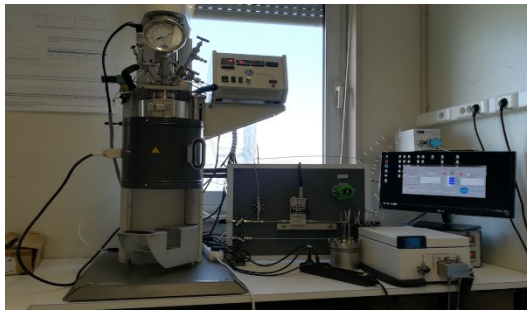


Increase of phosphate groups by increasing the bioceramic percentage

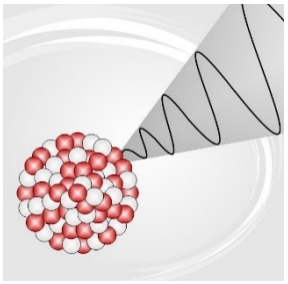
Materials and methods: Sterilization

3D Printing PLA disc (5x1mm)

Supercritical CO_2 sterilization



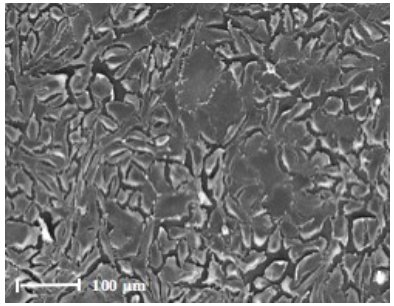
Gamma radiation



Autoclave

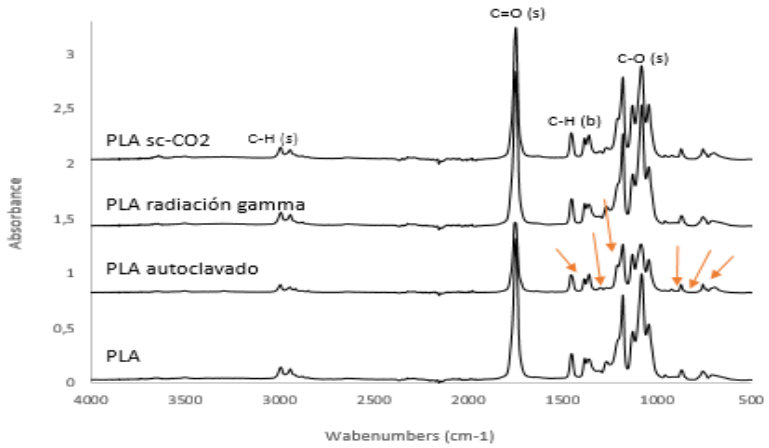
Physicoquemical characterization

Biological tests



Results: Sterilization

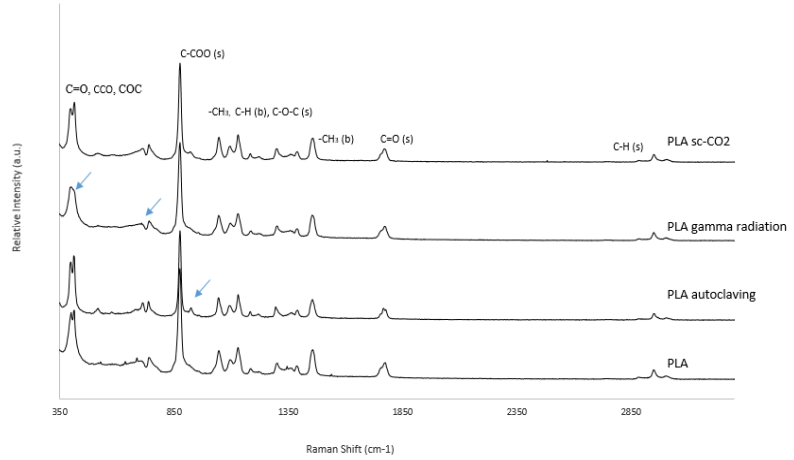
FTIR



Similarity to PLA control (%)	
PLA autoclaving	90.04 %
PLA gamma radiation	**99.84 %**
PLA sc-CO2	97.58 %

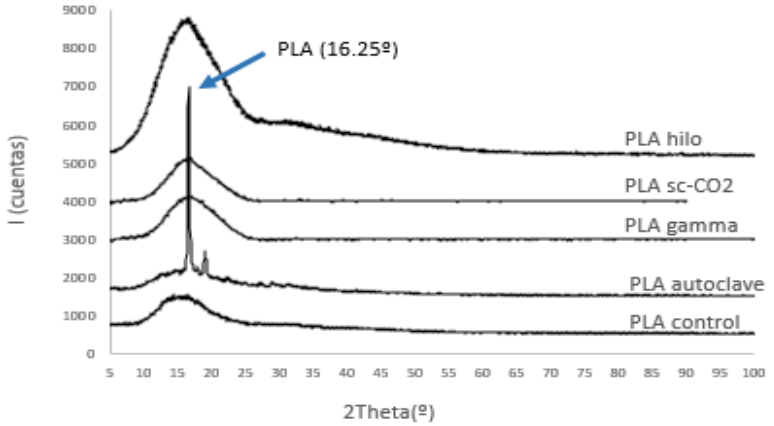
Gamma sterilization produces fewer changes

RAMAN



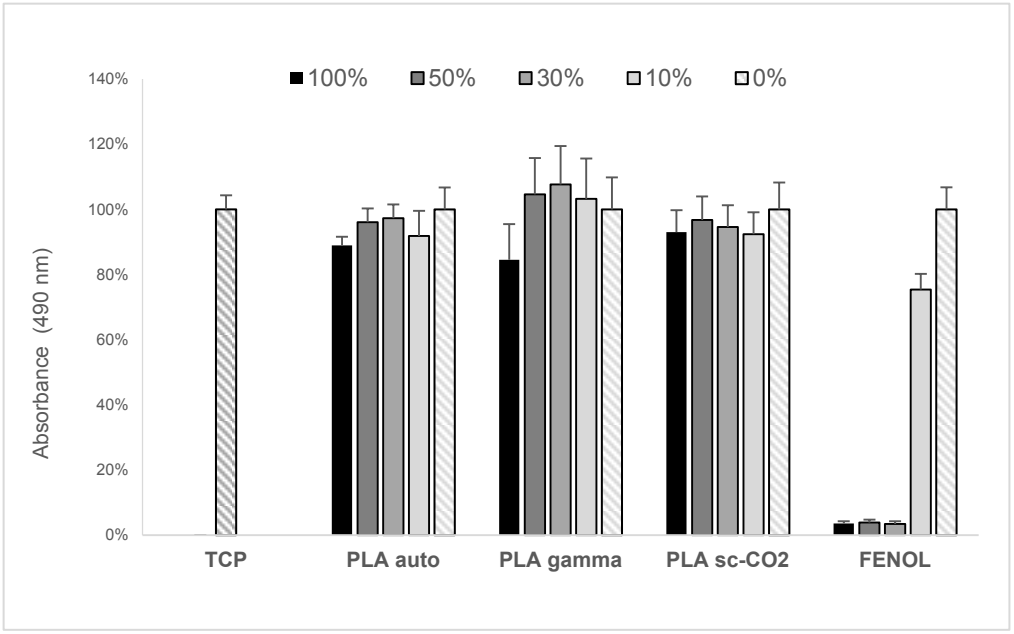
Autoclave sterilization and gamma radiation cause minor changes in some of the Raman bands

XRD



Significant changes in autoclaving process

Results: Sterilization & citotoxicity



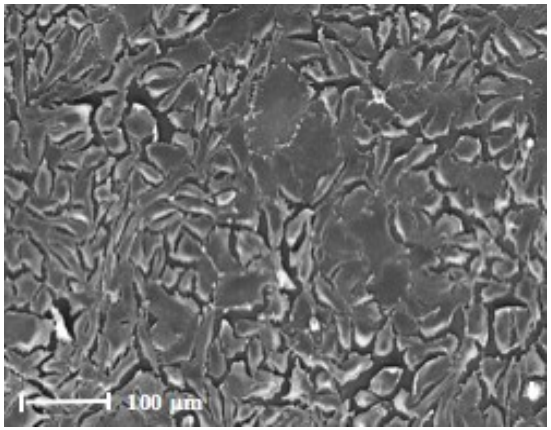
Citotoxicity test

There are no significant differences in any of the concentrations in the 3 sterilizations respect the control

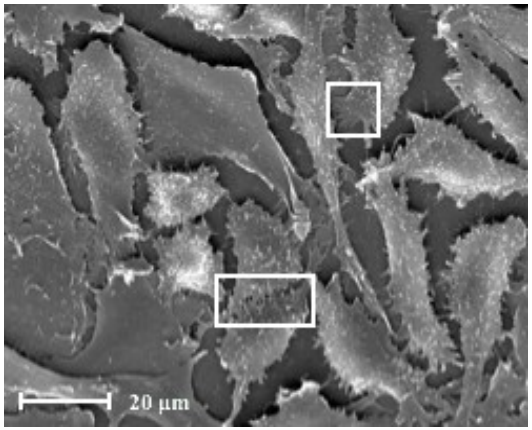
NO CITOTOXICITY

SEM (supercritical CO2)

200 x



1000 x



Fibroblasts L929:
healthy morphology

Connections between cells and with the material

Conclusions

- Development of a new filament based on PLA and CaP (marine origin) suitable for use in 3D-FDM printing
- Autoclave sterilization produces significant changes in physicochemical properties
- No cytotoxicity behaviour its observed for the 3 sterilizations

Thank you for your attention

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