Experiment Proposal

Experiment number GP2024006

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Experiment title	Morphological characterisation and compositional analysis of SiCa based Bioglasses using SEM-EDX

MRF Instrument	**SEM with correlative AFM**	**Days requested:** 3
Access Route	Direct Access	**Previous GP Number:** -
Science Areas	Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract

The main objective of the study is the production of bioglass nanoparticles with bioactive, osteoconductive and antimicrobial functions to be used as coatings for titanium grafts. These bioactive glasses will have composition within the ternary system silicon, calcium, phosphorus, formulations already proven to promote the formation of hydroxyapatite, and stimulate rapid regeneration and new bone tissue formation.

In order to correctly characterize the synthesized nanoparticles a thorough morphological characterization (SEM, EDX) is of fundamental importance in order to identify the correct synthesis routes to have a spherical geometry of the nanoparticles produced. At the same time, the nanoparticles created will have to maintain a stoichiometric level of the final elements similar, if not identical to those of the starting formulations or in any case such as to achieve the doping level necessary to activate the sought osteogenetic properties.

Publications -

ISIS neutron and muon source	**E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
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Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SEM with correlative AFM**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	BioactiveGlasses nanoparticles	-	-
Formula	SiCa / SiCaSr / SiCaZn / SiCaGa	-	-
Forms	Solid		
Volume	1 cc		
Weight	10 mg		
Container or substrate	Sample holder	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300K - K	-	-
Pressure Range	1 atm / High vacuum - mbar	-	-
Magnetic field range	None - T	-	-
Standard equipment	-	-	-
Special equipment	none	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

1. Background and Context

The research activities that will be carried out are part of a broader project which specifically aims to validate an innovative platform that allows the creation of a patient-specific path, which includes the different phases (assessment, treatment, and rehabilitation) which articulates the execution of a surgical procedure optimized for osteotomy (in the specific case, tibial osteotomy). This platform must prove capable of quantifying functional needs, minimizing the invasiveness of the operation and monitoring the rehabilitation process, thus drastically reducing recovery times and complications associated with the operation itself. As part of this activity, medical titanium grafts with a trabecular structure will be created, which can be completely integrated into the patient's bone structure. This result cannot be obtained with current technological knowledge, as current prostheses allow only superficial osseointegration, without involving the presence of vascularization and subsequent formation of bone tissue within the metal structure. These will be subsequently coated to increase osteointegration by bioactive glass nanoparticles, the latter produced using the sol-gel technique, with which particles of regular and nanometric shape can be created, but with a low percentage of dopant elements, or by grinding from melt, which usually gives rise to irregularly shaped micrometer-sized particles.

The main objective of the study is the production of bioglass nanoparticles with bioactive, osteoconductive and antimicrobial functions to be used as coatings for titanium grafts[1]. These bioactive glasses will have composition within the ternary system silicon, calcium, phosphorus, formulations already proven to promote the formation of hydroxyapatite, and stimulate rapid regeneration and new bone tissue formation. New formulations will be proposed with appropriately selected doping elements to confer improved characteristics in terms of osteoconduction and bacteriostatic properties, according to the project requests. Of primary importance will be the fabrication of spherical nanoparticles with high dimensional homogeneity. A methodology for the coating of bioactive grafts will also be developed. The creation of these nanoparticles requires an extensive characterization campaign of the bioglasses created both to control their morphology and to quantitatively define the quantity of dopants included. For this reason, the possibility of using electron microscopy (SEM) systems coupled with Energy Dispersive X-ray spectroscopy (EDX) microanalysis is of fundamental importance.

The advantage of the proposed approach is due on the one hand to the use of highly doped glasses with formulations capable of increasing osteogenesis (through the use of dopants such as strontium, potassium, magnesium and manganese), but above all in the obtaining of nanoparticles with a size and shape that allow uniform coverage of the surface of the titanium graft, speeding up the processes of osteointegration and osteogenesis.

Therefore, a correct morphological characterization is of fundamental importance in order to identify the correct synthesis routes to have a spherical geometry of the nanoparticles produced. At the same time, the nanoparticles created will have to maintain a stoichiometric level of the final elements similar, if not identical to those of the starting formulations or in any case such as to achieve the doping level necessary to activate the sought osteogenetic properties.

2. Proposed experiment

From what has been said, the need to have an elemental microanalysis coupled with microscopy images and therefore a SEM-EDX system is of paramount importance for the characterization of the prepared nanoparticles[3]. For this reason, we propose an experiment taking advantage of the capabilities of the “SEM with correlative AFM” instrument at the Tor Vergata Unit of IM@IT. The instrument allows a concurrent measurement of the morphology (using SEM) and elemental analysis (using EDX) that will allow to extract and assess the following information: morphology and shape of the nanoparticles, size of the nanoparticles, elemental composition of the nanoparticles. This set of information will be needed for a series of bioactive glass nanoparticles samples with different doping levels and synthesis methodologies, and the general composition SiCa / SiCaSr / SiCaZn / SiCaGa. The comparison of the results from a given set of samples will allow to identify the correct synthesis routes to have a spherical geometry of the nanoparticles produced and the right stoichiometry.

3. Justification of experimental time requested

Considering the need to acquire both SEM images and elemental maps with EDX from a set of 6 different samples, we request 3 days of instrument time on the “SEM with correlative AFM” instrument, to be used as follows: about half a day per sample, to obtain a good statistics and accurate results of the elemental composition, for a total of 6 samples.

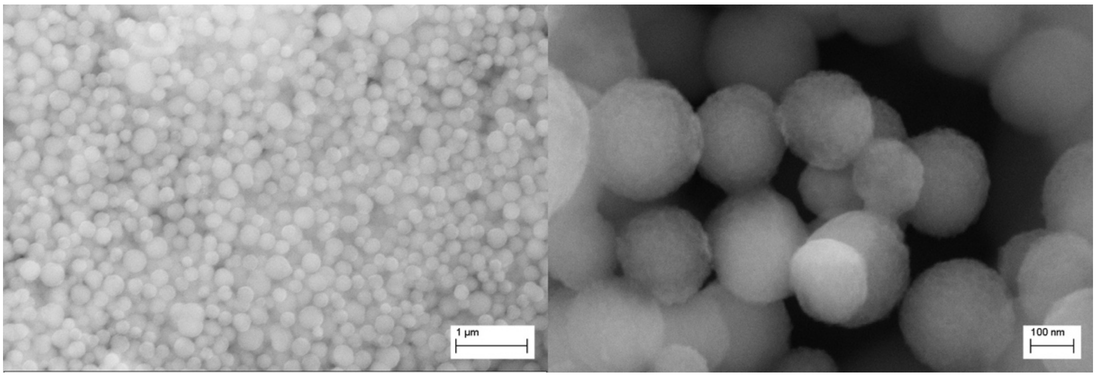

Figure 1. SEM micrographs of the bioglass nanoparticle sample with nominal composition 85% Si : 15% Ca at two different magnifications.

4. References

1. S.L. Greasley “Controlling particle size in the Stöber process and incorporation of calcium” J. Colloid Interface Sci. 469 (2016)

2. Rainer et al. “Fabrication of bioactive glass–ceramic foams mimicking human bone portions for regenerative medicine” Acta Biomaterialia 4 (2008) 362–369

3. G.M. Luz, J.F. Mano, Preparation and characterization of bioactive glass nanoparticles prepared by sol–gel for biomedical applications, Nanotechnology. 22 (2011) 494014.