



International Symposium on Elaboration and Characterization of Multistructured Materials ECMM 2018

July 10th to 12th, 2018

Amphitheatre Pierre-Gilles de Gennes
Condorcet building
University Paris Diderot



Ambassade de France en Espagne

Introduction

The functional design of structured materials is the cause of many technological advances in the field of civil engineering, textiles and innovative coatings, energy, nano-materials, for a few examples. A new field of investigation includes the living world. It is built through self-assembly of biological macromolecules and inspires the development of biomimetic materials by processes involving mechanisms of supramolecular organic and inorganic self-assembly. 3d printing is also a new emerging axis of research.

This symposium aims to focus on elaboration as well as study at different scales of properties and structure of complex multi-component materials from polymers, colloids, composites, with some degree of self-organization, a high sensitivity to external fields and specific dynamical properties. Experimental and theoretical understanding is still a challenge for the scientific community, because these complex and disordered materials present strong interactions and often have multiple correlations of spatial and temporal scales, unlike simple liquids and solids.

Several topics are covered : elaboration and performing of multiscale structured materials, thermal and rheological characterization of materials, hydrogels, smart textiles; 3d printing, statistics and numerical analysis applied to the study of materials.

The objectives are to provide dynamic and fruitful discussion for all participants including young researchers and to stimulate ideas to start collaborative projects.

I wish you a pleasant stay in Paris.

Alain Ponton
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International Symposium on Elaboration and Characterization of Multistructured Materials ECMM 2018

University Paris Diderot-Paris 7/Condorcet Building
Amphitheatre Pierre Gilles de Gennes

■ Tuesday 10th of July

9:00-9:30	Welcome
9:30-9:45	Introduction
9:45-10:30	Cure kinetics by Differential Scanning Calorimetry (DSC), Artiaga Ramón (<i>University of A Coruña, Spain</i>)
10:30-11:00	Break
11:00-11:45	Thermoset curing through Joule effect, López Beceiro Jorge (<i>University of A Coruña, Spain</i>)
12:00-14:00	Lunch
14:00-14:45	Rheological study of silicone, Díaz-Díaz Ana (<i>University of A Coruña, Spain</i>)
14:45-15:30	Synthesis and study of new materials with barocaloric properties, Martínez Muíño Alba (<i>University of A Coruña, Spain</i>)
15:30-16:15	Thermal characterization of different filaments for 3D printers, Álvarez Robledo Marcos (<i>University of A Coruña, Spain</i>)

■ Wednesday 11th of July

9:45-10:30	Scaffolds for tissue engineering, Madeleine Djabourov (<i>University of São Paulo, Brazil</i>)
10:30-11:00	Break
11:00-11:45	Microrheology of hydrogels, Tetsuharu Narita (<i>École Supérieure de Physique et de Chimie Industrielles-ESPCI Paris, France</i>)
12:00-14:00	Lunch
14:00-14:45	Study of porcelains for 3D printing, Castro Garcia Socorro (<i>University of A Coruña, Spain</i>)
14:45-16:00	Discussion on double master

■ Thursday 12th of July

9:45-10:30 Some cases of statistical modelling in material science and tissue engineering, **Salvador Naya, Javier Tarrío-Saavedra** (*University of A Coruña, Spain*)

10:30-11:00 **Break**

11:00-11:45 Streaming flows, **Philippe Brunet** (*University Paris Diderot, France*)

12:00-14:00 **Lunch**

14:00-14:45 Heart Valves from Textiles: Potential and Remaining Issues, **Frédéric Heim** (*University of Haute Alsace, France*)

14:45-15:00 Conclusion of ECMM 2018

15:30-17:30 Master defense and jury (for involved people)

Cure kinetics by DSC

Ana-María Díaz-Díaz¹, Jorge López-Beceiro¹, Carlos Gracia-Fernández² and Ramón Artiaga^{1*}

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A kinetic model and the corresponding method to obtain the kinetic parameter values are presented in the context of a cure reaction observed by differential scanning calorimetry (DSC). The reaction is observed at several constant temperatures and also in linear heating experiments at several heating rates. The model can be classified into the Model Fitting class, with the important peculiarity that fitting of several DSC curves obtained in varied conditions is needed. On the other hand the model is of the reaction order type. However, resembling the classical Arrhenius expression, the dependence of reaction rate on temperature is not that of Arrhenius. DSC experiments were carried out in a TA Instruments Q2000 MDSC. An epoxy resin based on diglycidyl ether of bisphenol-A is cured with a mixture of hexahydromethylphthalic anhydride and methyltetrahydrophthalic anhydride. Sample mass was in the 4 to 11 mg range. In its derivative form, the model can fit DSC and DTG curves, where the transformation process is observed as a peak. Figure 1 illustrates how a mixture of a generalized logistic and its derivative accurately fit a heating DSC cure.

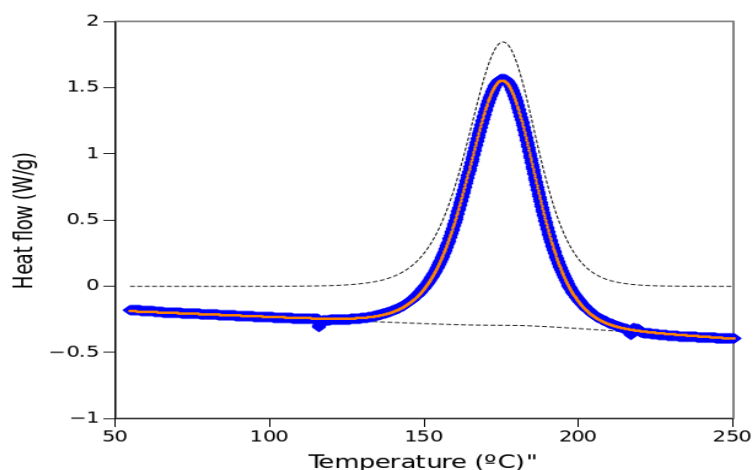


Figure 1: fitting of a DSC peak by a mixture of a generalized logistic function and its derivative

[1] López-Beceiro, J., Álvarez-García, A., Sebio-Puñal, T., Zaragoza-Fernández, S., Álvarez-García, B., Díaz-Díaz, A., Janeiro, J., Artiaga, R., 2016. BioResour. 11, 5870–5888.

[2] López-Beceiro, J., Fontenot, S.A., Gracia-Fernández, C., Artiaga, R., Chartoff, R., 2014. A logistic kinetic model for isothermal and nonisothermal cure reactions of thermosetting polymers. J. Appl. Polym. Sci. 131, 8428–8436.

Thermoset curing through Joule effect

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The curing of any thermoset material is usually performed using a heat source, that generally is a huge furnace. This furnace typically requires a great energy consumption. In this work, the main aim is the achievement of the curing of a thermoset material using the Joule effect reducing the amount of the required energy and getting a more homogeneous curing. Joule heating is the process by which the passage of an electric current through a conductor release heat. The amount of heat resulting from the Joule effect is proportional to the resistance, R , to the square of the current intensity, I , and to the time.

Some aeronautic composites with carbon fibers could be cured through the Joule effect [1,2]. In this case, the heat generated could be more or less homogeneous depending on the distribution of the fibers along the thermosetting matrix. Other possibility, consists of incorporating nano-sized conductive fillers [3,4], such as carbon black and carbon nanotubes (CNT). A good and homogeneous dispersion of these nano-sized fillers allows to get a homogeneous heating over the whole volume of the piece.

In the present work, CNTs will be used for getting a conductive thermosetting sample that will be cured by Joule effect heating. The curing reaction of the system will be monitored by measuring its viscoelastic properties. For doing this, a rheometer was connected to a DC electrical source which is controlled by a computer as function of the sample temperature (obtained from an infrared camera). The heating rate is controlled by adjusting the electric field through a PID control loop.



Figure 1: instrument layout

- [1] Sarles SA, Leo DJ. J Compos Mater., 42 (2008) 2551–2566.
- [2] Athanasopoulos N, Kostopoulos V. Compos Sci Technol. 72 (2012) 1273–1282.
- [3] Gungor S, Bakis CE. J Compos Mater. 49 (2015) 535–44.
- [4] Balberg I. Carbon. 40 (2002) 139–43.

Rheological study of silicone

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This work shows a characterization of two different podiatric silicones by comparing their viscoelastic properties according to the dynamic stresses they can be presumably subjected when used in podiatry orthotic applications. These results provide a deeper insight in the world of dynamic stresses developed in physical activity. Podiatric orthoses can be subjected to a set of static and dynamic shear and compressive forces..

Three kinds of rheological tests are performed: shear stress sweep, compression frequency sweep and shear frequency sweep, all the three with simultaneous control of the static force at three different levels. The static force represents a static load like that produced by the weight of a human body on a shoe insole. In a practical sense, dynamic stresses are related to physical activity and are needed to evaluate the frequency effect on the viscoelastic behavior of the material. It is considered that the dynamic stresses can be applied in compression and shear since, in practice, the way the stresses are applied in real life depends on the orthoses geometry and its exact location with respect to the foot and shoe. The effects of static and dynamic loads are individualized and compared to each other through the relations between the elastic constants for isotropic materials.

Three levels of static force were chosen in the range from zero to 90 kPa, which covers most of the mean peak plantar pressures developed during walking in the different regions of the foot under the conditions of barefoot and shod at different speeds [1]

This study present the rheological characterization to choose the right silicone for each podiatric application, taking into account the dynamic viscoelastic requirements associated to the physical activity of user. Accordingly, one soft and one hard silicones of common use in podiatry were tested. Each of the two silicones presents not only different moduli values, but also a different kind of dependence of the dynamic moduli with respect to the static load. In the case of the soft sample a linear trend is observed, but in the case of the hard one the dependence is of the power law type. Moreover, these samples exhibit very different Poisson's coefficient values for compression stresses lower than 20 kPa, and almost the same values for stresses above 40 kPa, as it is shown in Figure 1. That different dependence of the Poisson's ratio on the static load should also be taken into account for material selection in customized

podiatry applications, where static and dynamic loads are strongly dependent on the individual weight and activity. This work also prompts an effective methodology to evaluate the dynamic viscoelastic behavior of silicones [2].

In order to better understand the different behavior of both samples, the Poissons coefficient was also calculated from the same experimental data than the moduli. It is related with the other elastic constants through these expressions [3].

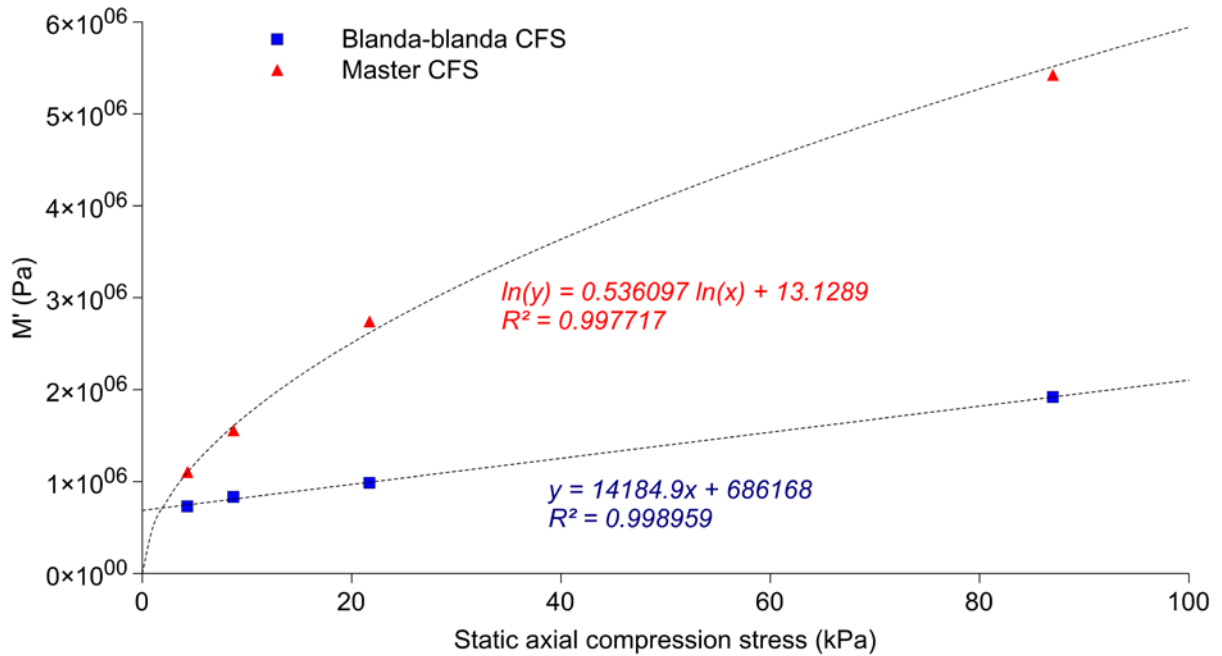


Figure 1: Longitudinal storage values obtained from the Blanda-blanda and Master samples in Compression frequency sweep tests at 1 Hz

[1] Burnfield, J.M., Few, C.D., Mohamed, O.S., Perry, J., Clin. Biomech 19 (2004) 78–84.

[2] Díaz-Díaz, A.M., Sánchez Silva, Bárbara, Tarrío-Saavedra, Javier, López-Beceiro, Jorge, Janeiro-Arocas Julia, Gracia-Fernández, Carlos, Artiaga Díaz, Ramón, JMBBM (2018) 66–71.

[3] Mavko, G., Mukerji, T., Dvorkin, J., “The Rock Physics Handbook: Tools for Seismic Analysis of Porous Media”, 2nd ed. Cambridge University Press (2009).

Synthesis and study of new materials with barocaloric properties

Alba Martinez Muiño

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The need of new refrigerants and coolants has led to the development of materials with elastocaloric, electrocaloric, magnetocaloric and barocaloric properties. But most of them need to be applied big external stimuli, making difficult their introduction on the common life. Nevertheless, recent studies show that hybrid perovskites can present giant barocaloric effects under low pressure.

This work describes the synthesis and structural characterization of DMAPbCl_3 and their homologous with Br doping: $\text{DMAPbCl}_{2.5}\text{Br}_{0.5}$, $\text{DMAPbCl}_{2.6}\text{Br}_{0.4}$, $\text{DMAPbCl}_{2.7}\text{Br}_{0.3}$, $\text{DMAPbCl}_{2.8}\text{Br}_{0.2}$ and $\text{DMAPbCl}_{2.9}\text{Br}_{0.1}$. All samples were characterized by P-XRD, SEM-EDX and TGA. P-DSC studies were also carried out at room pressure and 750 p.s.i. These studies showed barocaloric transitions around room temperature, and that the temperature transition can be tuned by changing the Br doping quantity.

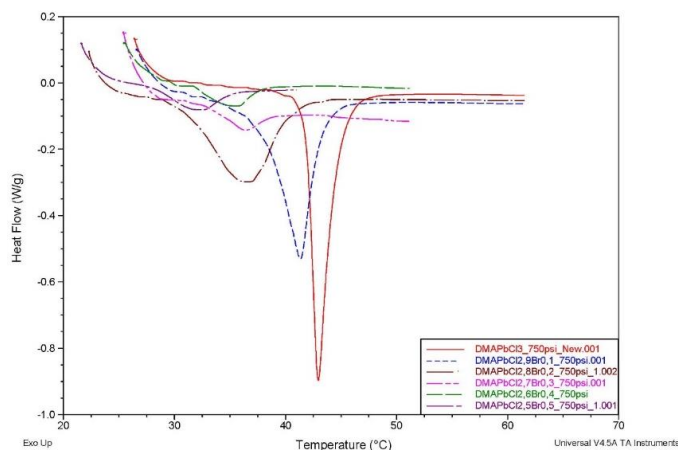


Figure 1: Heat flow vs Temperature plot of all samples at 750 p.s.i. $\text{DMAPbCl}_{2.5}\text{Br}_{0.5}$ (purple), $\text{DMAPbCl}_{2.6}\text{Br}_{0.4}$ (green), $\text{DMAPbCl}_{2.7}\text{Br}_{0.3}$ (fuchsia), $\text{DMAPbCl}_{2.8}\text{Br}_{0.2}$ (brown), $\text{DMAPbCl}_{2.9}\text{Br}_{0.1}$ (blue) and DMAPbCl_3 (red).

Characterization of different filaments for 3D printers

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² Université Paris Diderot

In the recent years a growing interest about new and automatized manufacturing techniques in industry has been taking place all over the developed world, specially a new method of fabrication of parts and objects called 3D printing, regardless of the material used during the production: polymers, metals, different kinds of composite... This advanced manufacturing method can imply a reduction of time and cost, but also a way to speed up the production in a continuous way, avoiding some defects and problems typically related with manual processes. 3D printing techniques have been during years, since their invention more than thirty years ago, a mainly experimental and limited field specially used with polymers, but nowadays this method has arrived to real industry and it's being employed for the commercial production of items not only made of polymer.

Among all the 3D printing variants it's necessary to underline the so called fusion deposition modeling (FDM), which is the most used in the industry due to its simply-to-use, low-cost and environment-friendly features, being the one that is going to be employed in this work.

Lot of factors are critical when we perform a printing, but the filament (feeding material for the fabrication of the parts) is specially relevant and it has been the subject of multiple studies. The composition, microstructure, mechanical, thermal and rheological properties of the employed filaments can be incredibly important factors in order to perform a successful printing and obtain good products.

Material analysis techniques are really useful to explore these critical features due to their capacity to study composition (XRD, laser spectroscopy), microstructure (optical and electronic microscopy), mechanical (simple tension tests), thermal (TGA, DSC...) and rheological features of the different filaments.

Because all of this, the purpose of this study is to make a thermal and rheologic characterization of different printing filaments, studying at the same time the effect of different printing parameters (print temperature and print speed for example) over the properties of the printed parts.

Scaffolds for tissue engineering

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In the new field of tissue engineering, polymers were designed to assist regeneration of three-dimensional tissues and organ structures and to comply with biological demands. Engineering of tissues or organs to treat patients appears as a promising therapeutic approach that combines biomaterials, cells and environmental factors, promoting tissue repair and/or functional restoration. Biomaterials called “scaffolds” play an important role as extracellular matrices to enable to create the correct microenvironment and promote *in vitro* tissue development. Scaffolds are the porous solid materials that provide a three-dimensional framework for selective cell penetration and facilitate the formation of the new tissue. Scaffolds are designed to have optimal pore sizes depending on the cells which are expected to grow inside: for fibroblasts and hepatocytes, it should be around 20 μm , for skin regeneration, between 20 and 150 μm or for bone regeneration in the range of 100–250 μm . In this presentation we report on scaffolds designed for skin regeneration.

We illustrate the relation between the chemical composition (choice of different biopolymers in solution) and their relative proportion, and the structure of freeze dried scaffolds allowing the growth of fibroblasts. Several thermal and chemical treatments are necessary to create the scaffolds starting from initial dilute solutions. These steps are designed to control the microstructure and the mechanical properties of the scaffolds. In addition to these constraints, cell attachment to the scaffolds is another critical factor in tissue regeneration together, with the control of biodegradation during the wound healing. Those properties will be discussed ^[1-4].

[1] Physical Gels from Biological and Synthetic Polymers, M. Djabourov, K. Nishinari and S.B. Ross Murphy, Cambridge University Press, 2013

[2] Disordered Pharmaceutical materials, J. Wiley, M. Descamps, Ed., 2016

[3] D. Lopez Angulo and P.J. Sobral, Materials Research 19 (2016) 839-845

[4] D. Lopez Angulo and P.J. Sobral Inter.J. Biol. Macromolecules 92 (2016) 645–653

Microrheology of hydrogels

Multiscale rheological characterizations by macro- and micro-rheology

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The dynamics of soft matters such as colloidal suspensions, emulsions, solutions and gels of synthetic/biological polymers, surfactants micelles, etc, extends across several orders of magnitude on the time ranging from microseconds to hours (or longer), reflecting the various relaxation processes at different length scales. At short length scales the dynamics are fast and high frequency rheology can provide insight into microstructure and interactions of the soft matters, which are of interest for fundamental and applied research. For the case of polymer solutions and gels, microscopic features such as single chain length and rigidity, architecture of associative/crosslinked chains, result in characteristic scaling regimes for the high frequency moduli.

The values of frequency corresponding to these local dynamics can be typically found between 10^3 to 10^6 rad/s. It is not easily accessible by conventional rotational shear rheometers which are usually limited to several hundred rad/s. And generally for such complex fluids time - temperature superposition is not suitable to extend the frequency range.

In the last decade novel optical rheological techniques which can allow us to access to the high frequency range have been available. In our laboratory we work on microrheology by dynamic light scattering. From the single or multiple dynamic light scattering signals coming from tracer particles dispersed in viscoelastic media, the mean square displacement of the particles are measured. By using the generalized Stokes-Einstein relation one can calculate high frequency viscoelastic moduli of the media (up to about 10^5 rad/s).

In this seminar I will talk about experimental details of DLS and DWS microrheology, then I will show you some examples of high frequency dynamics of polymer aqueous solutions and hydrogels:

- Zimm-Rouse mode of single chains in gels
- single chain rigidity of a giant polysaccharide in dilute solution
- percolation of chains by transient dynamic crosslinks

Study of porcelains for 3D printing

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Nowadays 3D printing is a very useful system to produce new forms and structures, which is used in more and more ambits. Inks are usually constituted of polymers, but the interest to print different materials is motivating the investigation and development of new systems and kinds of inks. Ceramics are among the most interesting materials to print to be used in several top fields, like biomedicine, electronics or even arts.



Figure 1: Porcelains printed in a DeltaWasp 20 40 Turbo2 3D-printer showing typical defects

This presentation deals with the modification of a ceramic material (in particular, a porcelain) to print in 3D. Printing ceramics comes with some difficulties. The biggest being the control of the physical properties of the material before, during and after the printing. The present study is focused on the variation of these properties and how it affects the printing process. The 3D printing system studied was extrusion, which works with the plasticity of clay when it is moistened. The principal problem of this method is the consistency of the pieces after the printing: they have to be flowing enough to be extruded but they have to keep the form after the process. If this property is not controlled, undesired defects appear, as observed in Fig. 1.

Some cases of statistical modelling in material science and tissue engineering

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² Cátedra UNESCO UPS Cuenca-Ecuador, Grupo de Investigación en Inteligencia Artificial y Tecnologías de Asistencia (GIATA), Cuenca, Ecuador.

³ Departamento de Matemática, Escuela Politécnica Nacional, Quito, Ecuador.

This work mainly deals with the application of statistical tools to study and model degradation due to hydrolysis and cells activity of bioscaffolds (biopolymers, collagen) used in tissue engineering.

On the one hand, the degradation due to biological action is measured. Thus, this study proposes a method based on image analysis, machine and statistical learning to model and estimate the osteocyte cells growth in type I collagen scaffolds for bone regeneration systems, and the collagen degradation degree due to cellular growth. The mass of collagen, subjected to the action of osteocyte cells growth and differentiation from stem cells, is measured each three days during two months, under human body conditions. In addition, optical microscopy is applied to obtain information about cellular growth, cellular differentiation and collagen degradation. The first contribution consist on the application of a supervised classification Random Forest algorithm to image texture features (structure tensor, entropy) for estimating the different regions of interest (ROI) in an image obtained by optical microscopy: extracellular matrix, collagen, and image background artifacts. Then, extracellular matrix and collagen ROI are defined by the extraction of features related to the progress of the cellular growth and collagen degradation (e.g. objects mean area, ellipse major axis, mode of intensity histogram, among others). Finally, these critical features are statistically modeled depending on time using nonparametric and parametric linear and nonlinear models such as those based on Gompertz and logistic functions. Relationship between osteocyte cellular growth and differentiation from stem cells, on the one hand, and collagen biodegradation, on the other hand, has been determined and modeled through the study of image objects circularity, area, and collagen mass loss. In addition, neural networks are also applied, combined or not with nonlinear regression models in order to predict the area of collagen and extracellular matrix for each time, i.e. to estimate the cellular mass growth and the degree of biodegradation. These set of imaging techniques, statistical tools and machine learning procedures allows to characterize and parameterize the type I collagen biodegradation when acting as a scaffold in bone regeneration tasks.

On the other hand, a statistical methodology is proposed for simulating the performance of different Poly(D,L-lactide-co-glycolide) copolymer formulations (PDLGA) in the human body, for identifying the more influencing variables on hydrolytic degradation and, thus, to estimate biopolymer degradation level. The PDLGA characteristic degradation trends, caused by hydrolysis processes, have been studied to define their future biomedical applications as dental scaffolds. For this purpose, the mass loss, pH, glass transition temperature (T_g) and

absorbed water mass of the different biopolymers have been obtained into a phosphate-buffered saline solution (PBS) with initial pH of 7.4, at 37°C (human body conditions). The mass loss has been defined as the variable that characterizes the biopolymer degradation level. Its dependence relationship with respect to time, pH and biopolymer formulation has been modeled using statistical learning tools such as generalized additive models (GAM) and nonlinear mixed-effects regression with logistic and asymptotic functions. GAM model provides information about the relevant variables and the parametric functions that relate mass loss, pH and time. Mixed effects are introduced to model and estimate the degradation properties, and to compare the PDLGA biopolymer populations. The degradation path for each polymer formulation has been estimated and compared with respect to the others in order to help to use the proper polymer for each specific medical application, performing selection criteria. It was found that the mass loss differences in PDLGA copolymers are strongly related with the way the pH decay versus time, related to carboxylic acid group formation. The results show that PDLGA polymers degradation degree, in terms of half-life and degradation rate, is increasing when acid termination is included, when DL-lactide molar ratio is reduced, decreasing the midpoint viscosity, or when glycolide is not included.

In addition, different techniques for data handling developed by the authors in the framework of interlaboratory studies and material science will be presented, with the companion of the corresponding software tools. Namely, the new functional extensions for h and k Mandel's statistics will be shown [1].

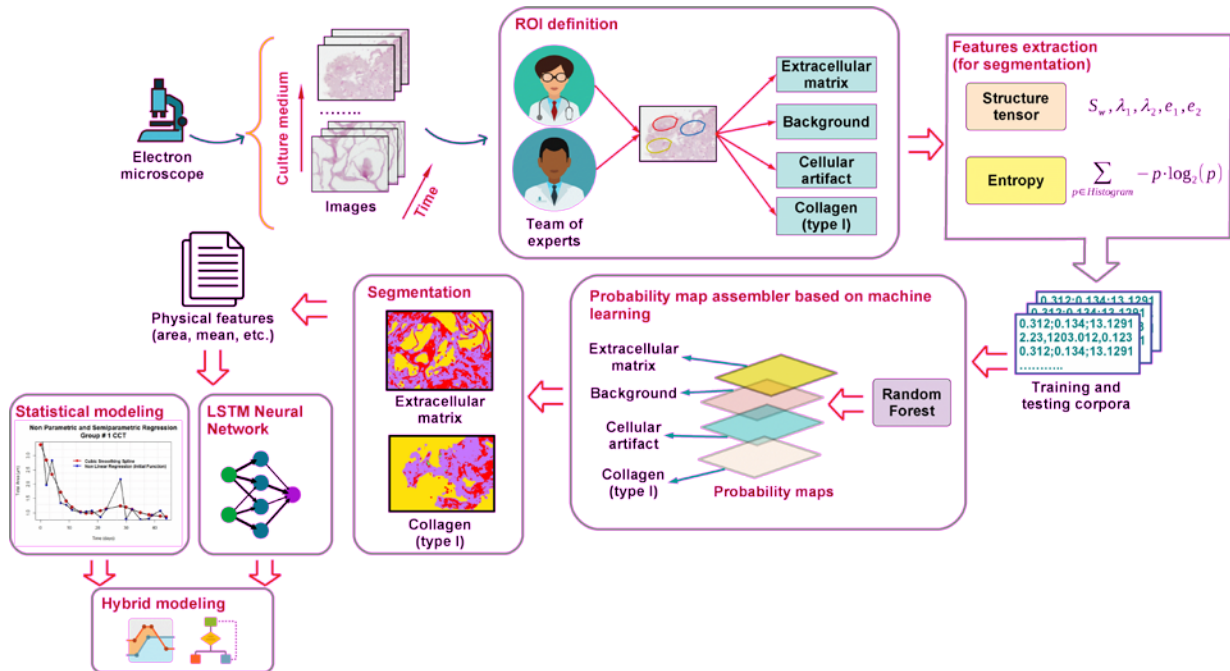
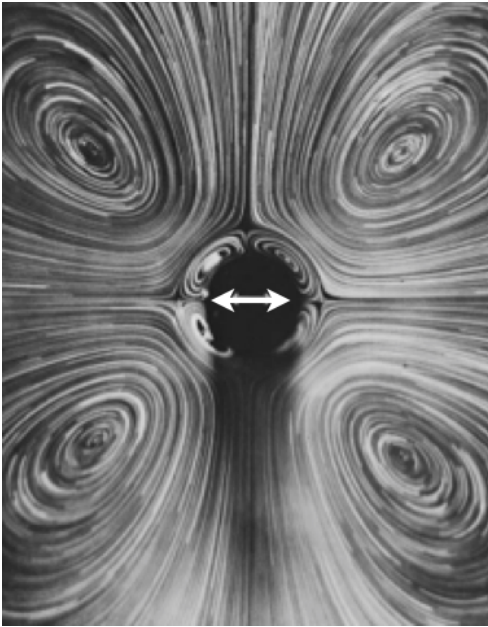


Figure 1: Proposal to model cellular growth and degradation degree of type I collagen

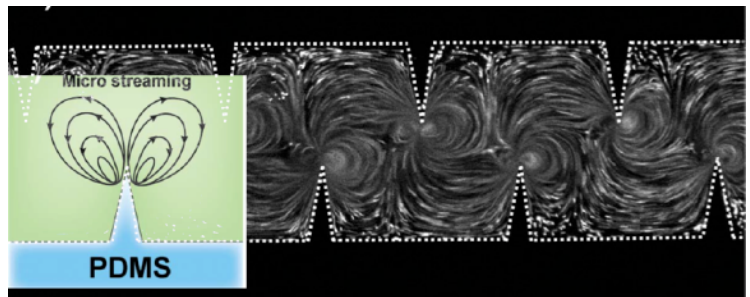
Streaming flow

Philippe Brunet

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Streaming flows denote steady time-independent flows occurring from a periodic forcing, generally mechanical vibrations or acoustic waves [1]. After reminding the historical discoveries, I will show some examples of such flows, in particular in microfluidic geometries where they are of great interest for mixing, pumping and particle sorting.



Left - Streaming flow generated by an oscillating cylinder immersed in a fluid (from Tatsuno, 1973 [2]).

Right - Streaming flow generated by a kHz acoustic wave, in a micro-channel with sharp pikes (from P.H. Huang et al. [3]).

[1] Sir J. Lighthill. Acoustic streaming. J. Sound Vib. (1978).

[2] M. Tatsuno. J. Phys. Soc. Jap. **35**, 915 (1973).

[3] P-H. Huang *et al.* Lab. Chip **13**, 3847 (2013).

Heart Valves from Textiles: Potential and Remaining Issues

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LMPT / ENSISA

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Abstract

Over 300.000 heart valves are replaced every year in western countries and valve therapy represents today one of the most common surgical procedures performed in the world. While open chest surgery remains the gold standard to replace a faulty valve, less invasive approaches have been developed over the last decade. Actually, the rapid developments and success in percutaneous vascular stents implantation over the last 2 decades to treat vessel stenosis has made this technique attractive today even for aortic valve replacement. The principle is to implant a stented valve prosthesis by going through the vascular network of the patients. With this new technique, patients are not exposed to the risks of surgery, and transcatheter aortic valve implantation (TAVI) has become highly suitable for an increasing elderly population and has become an accepted alternative technique to surgical valve replacement for over 100,000 patients worldwide. Despite minor issues related to the implantation of the device, this non-invasive technique is cost-effective and provides increased comfort to patients, relative to traditional surgical valve implantation. In a fast growing global market, where TAVI related survival rates depend highly on the initial patient's health, one can expect that more less-critical patients could be treated successfully with TAVI in the coming years. Currently, the valve material used in TAVI is biologic tissue, such as bovine or porcine pericardium. However, once assembled inside the metallic stent and crimped at low diameter for catheter insertion, studies have shown that the biological materials may become degraded. Textile polyester (PET) could be considered as an alternative material to replace TAVI biological valve leaflets. In particular, woven textile constructions have outstanding folding and resistance properties and as a result, these materials are easy to crimp and insert, even in low profile devices. Moreover, woven materials are discontinuous, mitigating the risk of a catastrophic rupture. Rupture propagation is isolated to the single filament. Recent works showed that woven textile materials could resist up to 200 million cycles *in vitro* under accelerated cyclic loading and 6 months *in vivo* successful implantations were reported with fabric valve prototypes implanted in juvenile sheep models. However, despite the high potential of the material, challenges remain before textile can be considered as a durable valve replacement solution.