# BOOK OF ABSTRACTS

FUR4Sustain TRAINING SCHOOL

**POLYMER CHARACTERIZATION:**

# HOW TO REVEAL THE SECRETS OF POLYMERS FOR BETTER EXPLOITING THEIR POTENTIALS

**14 > 17th JUNE'21**

# AGENDA

## 14TH JUNE

**10:00 - 10:30**
**Welcome participants** and introduction of the training school

**10:30 - 12:30**
**Prof. Marco Sangermano**
(Politecnico di Torino)
Monitoring of UV-Curing process

**12:30 - 15:00 Lunch break**

**15:00 - 17:00**
**Prof. Nicolas Sbirrazzuoli**
(Université Côte d'Azur)
Advanced thermal analysis of polymers and biopolymers

## 15TH JUNE

**09:30 - 12:00**
**Dr. Laurent Heux**
(CNRS CERMAV Grenoble)
NMR as a tool for investigating polymer dynamics and structural features

**12:00 - 14:30 Lunch break**

**14:30 - 17:00**
**Dr. Jean-Luc Putaux**
(CNRS CERMAV Grenoble)
Electron microscopy of polymer systems

## 16TH JUNE

**09:30 - 12:00**
**Prof. Noelle Billon**
(Mines Paris Tech/PSL Université)
Good uses in the mechanical characterization of new polymers; Application to "FURAN based -packaging"

**12:00 - 14:30 Lunch break**

**14:30 - 17:00**
**Dr. Sylvain Caillol**
(CNRS/ENSCM Montpellier)
From natural phenols to polymers: Structure/reactivity/property relationships

## 17TH JUNE

**09:00 - 11:00**
**Dr. Séverine Boyer**
(Mines Paris Tech/PSL Université)

**11:00 - 12:00**
**Dr. Nathanael Guigo**
(Université Côte d'Azur)
Monitoring fast organization processes in polymers

**12:00 - 12:30**
**Concluding remarks**

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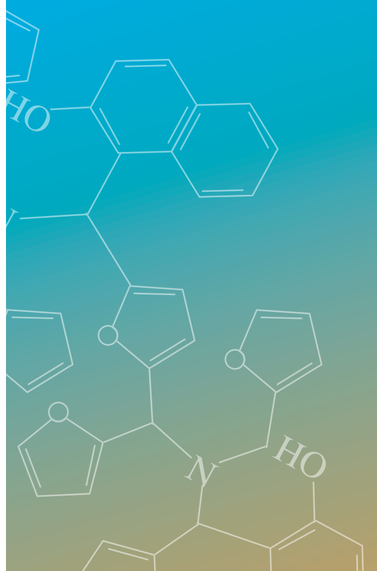



## PROF. MARCO SANGERMANO

POLITECNICO DI TORINO
Dipartimento di Scienza Applicata e Tecnologia
Torino, Italy


Marco Sangermano is a Professor of polymer science and technology at the Politecnico di Torino. His research activity is focused on photopolymerization and applications in the fabrication of advanced hybrid materials, nanostructured polymeric coatings, composites, gas sensors, membranes, 3D printing objects, tissue engineering. In2006 he received the prestigious Alexander von Humboldt grant and he spent a period of 6 months at IPF in Dresden, Germany. In 2016, he received the IBM University Award for the development of polymeric gas sensors for the fabrication of an electronic nose. Recently his research is focused in the development of new monomers from biorenewable resources to be used in the production of more environmentally-friendly materials by UV-Curing. He currently collaborates with numerous academic and the R&D of many corporate partners worldwide. He is co-author of over 280 papers and several patents.


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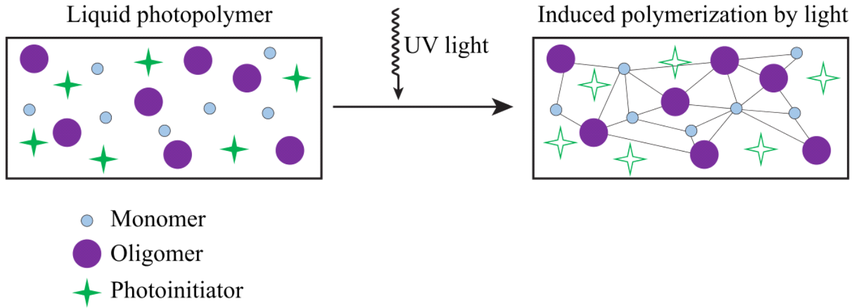
## MONITORING UV-CURING PROCESS

Radiation curing technologies are expanding rapidly on an industrial scale, as shown by the increase on monomer production in the last 20 years. These new technologies use light beams to start photochemical and chemical reactions in organic materials leading to the formation of a new polymeric material whose final uses may be encountered in various industrial areas.

Some of the most significant applications were originally related to the UV curing of coatings in the wood-finishing industry and, then, extended to the surface treatment of a large variety of substrates (plastics, metals, wires, pipes, vinyl flooring), to the coatings of optical fibers, to adhesives, to dental materials and composites. They continue to develop and provide several economic advantages over the usual thermal operation: rapid through cure, low energy requirements, room-temperature treatment, non-polluting and solvent free formulations, and low costs. On the other hand, new applications are nowadays emerging in the graphic arts and in the coatings industry. Another promising area, opened by the commercial development of various powerful lasers, is concerned with the applications of laser induced process in monomeric and polymeric materials to photoimaging, microelectronics, holographic or information recording and storage.

One of the common features of all UV-curable systems is the rapidity at which the polymerization takes place under intense illumination, usually within few seconds. Therefore, it is difficult to accurately follow the kinetics of such ultrafast reactions, which is a prerequisite for a better understanding and control of the curing process. Moreover, evaluation of the kinetic parameters (rate of polymerization, kinetic chain length, propagation and termination rate constant) is essential in order to compare the reactivity of different photosensitive resins and assess the performance of novel photoinitiators and monomers.

The aim of this lecture is to introduce the attendee to the UV-Curing process and describing the most important technique to follow the curing kinetics: Photo-DSC, Real-Time FT-IR analysis and Photorheology.

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## PROF. NICOLAS SBIRRAZZUOLI

PROFESSOR AT UNIVERSITY CÔTE D'AZUR
Leader Eco-friendly Materials and Polymers Team (MAPEC)
Institute of Chemistry of Nice, ICN UMR CNRS 7272
University Côte d'Azur
Parc Valrose, Nice cedex 02, France.

Prof. Dr Nicolas SBIRRAZZUOLI (University Professor of Exceptional Class, ORCID: 0000-0002-6031-5448, citations ~ 10k,) has his expertise in development of synthetic, biobased and non-toxic polymers and composites, resulting from the recovery of plant biomass, co-products of industry and biorefineries. He worked on application of thermal analysis, calorimetry, dynamic mechanical analysis and rheometry to a variety of polymeric materials. He contributed to develop "Advanced isoconversional kinetic analysis" a new approach for kinetic analysis of thermally stimulated reactions in the condensed phase and developed kinetic and mechanistic studies on complex polymerizations, crystallization and relaxation of polymers. He developed a new method to calculate the Hoffman-Lauritzen parameters and for simulation of crystallization processes.

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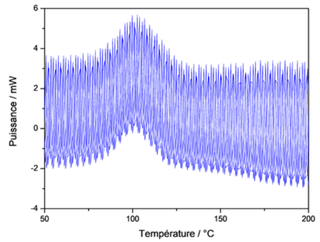
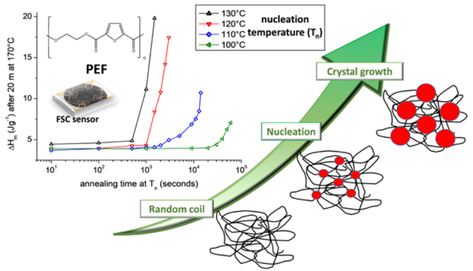
# ADVANCED THERMAL ANALYSIS OF POLYMERS AND BIOPOLYMERS

The course focuses on recent advances in thermal analysis. After a brief description of several techniques, specific applications and methods allowing a more powerful exploitation of the experimental data will be presented. The techniques presented will be Differential Scanning Calorimetry (DSC), Temperature Modulated DSC, Fast Scanning Calorimetry (FSC), Thermogravimetric analysis (TGA), Coupling TGA – MS, TGA – FTIR, TGA – GC/MS, TGA – microGC, FTIR with temperature control, Dynamic Mechanical Analysis (DMA). Applications will concern polymerizations, polymer crystallization, glass transition, physical ageing, kinetics (model-fitting methods and model-free kinetics). The last part of the course will be dedicated to the interpretation and physical meaning of isoconversional kinetic parameters for crosslinking, crystallization and glass transition of polymers.

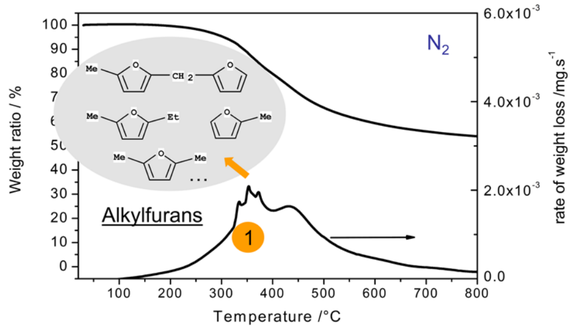
Temperature Modulated DSC for epoxy crosslinking



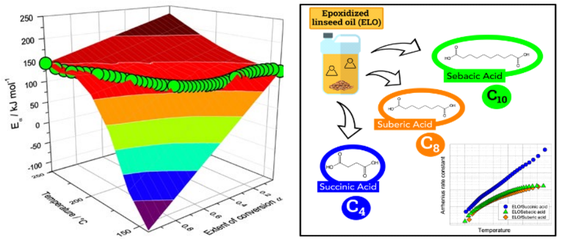

Fast Scanning Calorimetry (FSC) applied to PEF nucleation



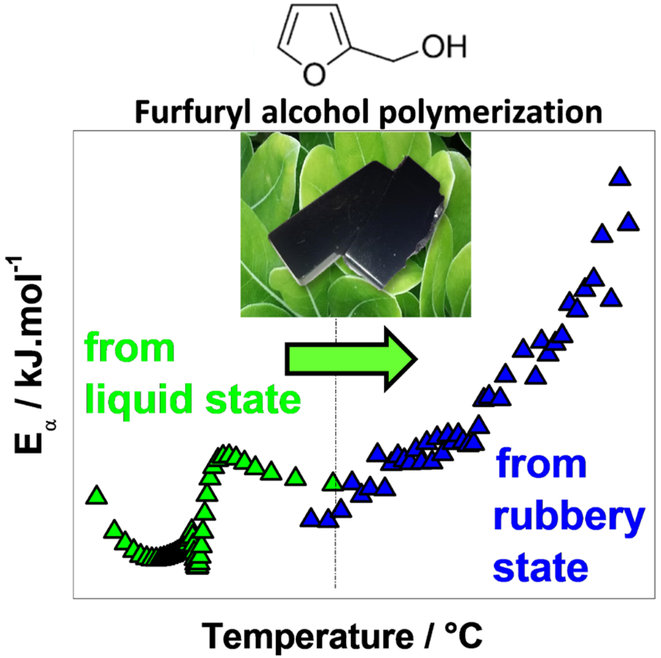

TGA-GC/MS of biobased thermoset degradation




Isoconversional Kinetic Analysis for polymerization of ELO with biobased dicarboxylic acids



Crosslinking of Furfuryl alcohol/Maleic anhydride in the liquid state and in the solid state

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CENTRE DE RECHERCHES SUR LES MACROMOLÉCULES VÉGÉTALES – CNRS
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J.-L. Putaux is a senior scientist at CERMAV where he studies the morphology, structure and polymorphism of crystalline polysaccharides by transmission electron microscopy and wide-angle X-ray diffraction.


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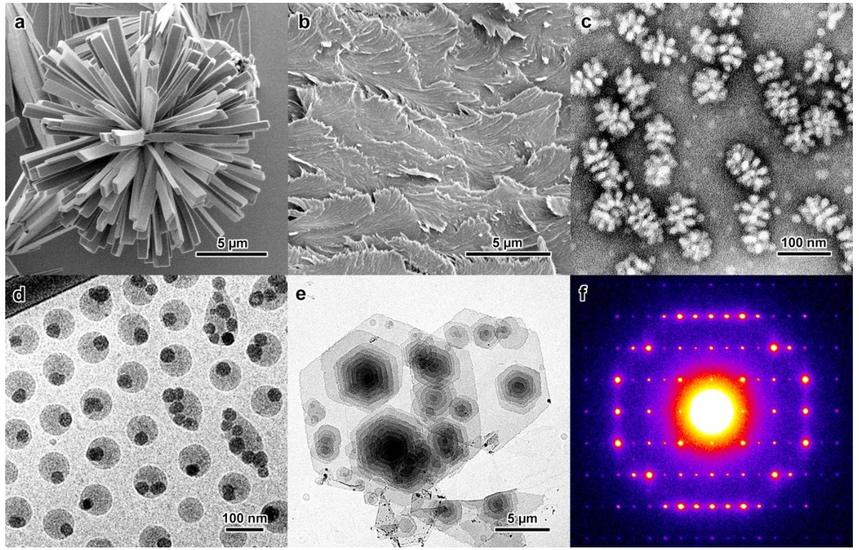
# ELECTRON MICROSCOPY OF POLYMER SYSTEMS

Electron microscopy techniques are crucial to characterize the morphology and structure of macromolecular and polymer systems at various lengths-cales, from colloidal nanoparticles to nanocomposite materials. Scanning electron microscopy (SEM) is generally used to characterize the surface topography of large / bulk samples and fractured materials, while transmission electron microscopy (TEM) allows visualizing the projected volume of small / ultrathin specimens.

One of the main limitations in SEM is the non-conductivity of organic samples that, in most cases, can simply be solved by coating their surface with a thin layer of metal. However, in the recent years, thanks to significant technological development, different approaches have been implemented, such as low-voltage, low-pressure or low-temperature observation.

The limiting factors of TEM are the poor intrinsic contrast of the small organic objects and their very high sensitivity to the electron beam that rapidly generates radiation damage and induce sample degradation, particularly in the case of crystalline materials. As a consequence, the specimens must often be observed under low-dose illumination and / or at low temperature in order to limit / slow down the damage. Specific staining techniques involving heavy atoms can also be applied to increase the contrast, while colloidal suspensions can be observed using fast-freezing and cryo-TEM procedures. In addition, electron diffraction can be used to characterize the local molecular structure of crystalline specimens.

After an introduction explaining why and how electrons can be used to characterize systems at the nanometric scale, the important information about the specificities of both scanning and transmission electron microscopies and related equipment will be given. Using several images from specific examples, the main sample preparation methods and their constraints will be detailed. The strategies to observe radiation-sensitive materials and analyze the contrasts in the images, both qualitatively and quantitatively, will be presented.




a) Recrystallized amylose (SEM);

b) fractured film of cellulose nanocrystals (SEM);

c) polystyrene dendrigrafts (TEM, negative staining);

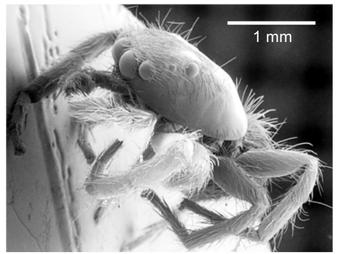
d) silica beads encapsulated in poly(butyl acrylate) (cryo-TEM);

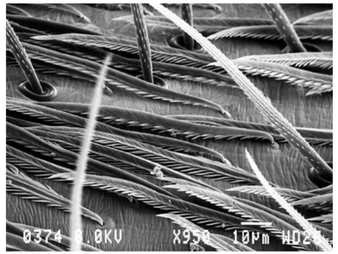
e) crystals of amylose complexed with butanediol (TEM);

f) electron diffraction pattern of an amylose-butanediol single crystal (images: CERMAV).

## SEM : secondary electron images

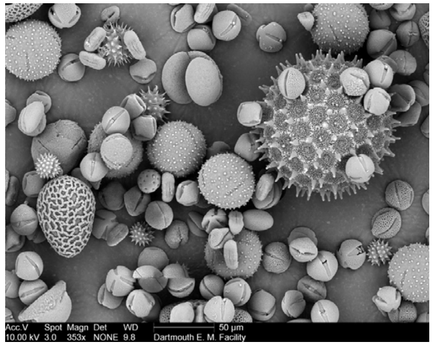
Spider / side detector *(CERMAV)*




Pollens / axial detector

*(Darthmouth College image gallery)*



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## NOELLE BILLON

CENTRE FOR MATERIAL FORMING (CEMEF – MINES PARIS TECH),
Sophia Antipolis, France

Noëlle BILLON is Professor at Mines Paristech -"Ecole des Mines de Paris", which is one of the leading engineer school in France.

She developed her research at Centre for Material Forming (CEMEF) in Sophia Antipolis since 1983. First fields of interest were crystallisation during processing and development of physical approaches to assess mechanical behaviour of polymer in relationship with microstructure.

Her approaches relied on theoretical (development of physically based macroscopic models) and on promoting relevant for polymers and accurate experimental technics and protocols.

She created and led the team "Physics and Mechanics of Industrial Polymers". The objective she gave to that team was to understand, characterize and model the mechanical behaviour of polymers and composites from their solid to their rubbery states. Relationship with process conditions and microstructure evolution was one of the main concerns.

Injection stretch-blow moulding, thermoforming and film stretching process were an important field of application since 1995. The team acquire a high expertise concerning Strain Induced Crystallisation of Polyester such as PET, PLA and more recently PEF.

**NOELLE BILLON**

Centre for Material Forming (CEMEF –Mines Paris Tech), Sophia Antipolis, France

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# GOOD USES IN THE MECHANICAL CHARACTERISATION OF NEW POLYMERS; APPLICATION TO PACKAGING

Illustrated the interest of adapting mechanical characterisation to specific properties of polymers in the specific case of the characterisation of stretching range of PEF compared to that of PET.

The behaviour of polymer materials is not a simple intrinsic property of the molecule and must account for microstructure. It is ruled by:

- **the ability to activate molecular relaxations in the loading conditions (e.g., temperature, plasticization),**
- **the ability to promote activation at given stress, which is constrained by the microstructure through interactions between not linked segments and extension of chains,**
- **the number of occurrences at given time, which are related to the strain rate and loading path.**

Consequently, the protocols, the strategy and the stretching range can be analysed in the frame of so-called Time Temperature Superposition Principle (TTP) and loading conditions reduced to the knowing of one parameter: the equivalent strain rate at reference temperature. This approach enables to find stretching conditions of PEF and to accurately compared stretch ability of PEF to that of PET.

Main concepts can be found in following literature:


- Gorlier E et al. (2001). Experimental and theoretical study of uniaxial deformation of amorphous poly(ethylene terephthalate) above glass transition temperature. Plastics, Rubber and Composites, 30/2 : 48-55
- Gorlier E et al. (2001) Strain-induced crystallisation in bulk amorphous PET under uni-axial loading. Polymer, 42/23 : 9541-9549
- Erner A et al. (2005) Thermal and friction effects during plug assisted thermoforming; Experimental approaches. Int. J. of Forming Processes, 5/8: 265-280
- Tan C W et al. (2008) Modelling of the injection stretch blow moulding of PET containers via a pressure-volume-time (PV-t) thermodynamic relationship. Int. J. Mater. Form. Suppl 1 : 799-802
- Billon N (2012). New constitutive Modelling for time-dependant mechanical behaviour of polymers close to glass transition: Fundamentals and experimental validation. J Appl Polym Sci, 125:4390-4401
- Billon N et al. (2014) Stretch blow moulding of PET; structure development and constitutive model. Int. J. of Mater. Forming, 7/3: 1–10
- Billon N et al. (2015). Propriétés et comportement mécanique des polymères thermoplastiques. In : Techniques de l'Ingénieur, Editions T.I., AM3115 - 26 p
- Maurel-Pantel A et al. (2015). A thermo-mechanical large deformation constitutive model for polymers based on material network description: Application to a semi-crystalline polyamide 66. Int J Plast. 67:102-126
- Gehring F et al. (2016). Modeling of time dependent mechanical behavior of polymers: Comparison between amorphous and semicrystalline polyethylene terephthalate. J Appl Polym Sci, 133(35):43837-43853
- Candau N et al. (2016). On the use of a four-cameras stereo vision system to characterize large 3D deformation in elastomers, Polym Test, 56:314-320
- Federico CE et al. (2018). Large strain/time dependent mechanical behaviour of PMMAs of different chain architectures. Application of time-temperature superposition principle. Polymer, 139:177 – 187
- Fabre V et al. (2018). Time-Temperature-Water content equivalence on dynamic mechanical response of polyamide 6,6. Polymer, 137: 22-29
- Forestier E et al. (2020). Understanding of strain-induced crystallization developments scenarios for polyesters: Comparison of poly(ethylene furanoate), PEF, and poly(ethylene terephthalate), PET. Polymer, 203:122755
- Federico CE et al. (2020). Modelling strain rate and temperature dependent mechanical response of PMMAs at large deformation from below to above Tg. Polymer, 202:122710
- Girard M et al. (2021). Effects of annealing prior to stretching on strain induced crystallization of polyethylene terephthalate. Polymer, 230:124078


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## SYLVAIN CAILLOL

INSTITUT CHARLES GERHARDT, MONTPELLIER

Sylvain Caillol is Research Director at CNRS. He graduated engineer from the National Graduate School of Chemistry of Montpellier in 1998 and then received his M. Sc. Degree in Chemistry from the University of Montpellier. He received his PhD degree in 2001 from the University of Bordeaux. Subsequently he joined Rhodia Company. Later, promoted Department Manager, he headed the Polymer Research Department in the Research Center of Aubervilliers. In 2007 he joined the CNRS at the Institute Charles Gerhardt of the University of Montpellier where he started a new research topic dedicated to Green Chemistry and Biobased Polymers.

He is co-author of more than 200 articles, patents and book chapters. He is Chairman of Oleochemistry division of European Federation of Lipids. He won the Green Materials Prize in 2018 and 2020 and was nominated Pioneering Investigator by the Royal Society of Chemistry in 2019.

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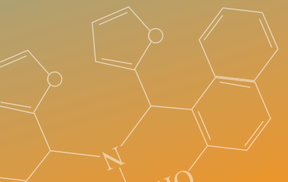
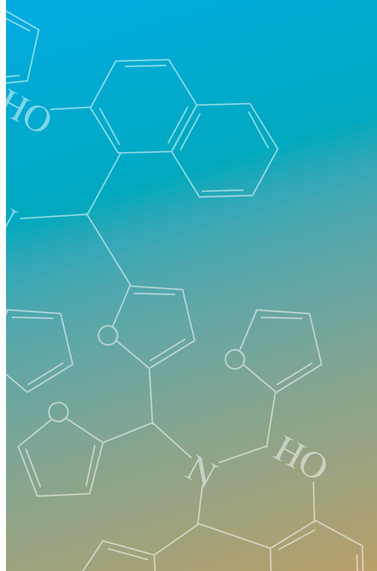


## FROM NATURAL PHENOLS TO POLYMERS: STRUCTURE/REACTIVITY/PROPERTY RELATIONSHIPS

Recent years have witnessed an increasing demand on renewable resource-derived polymers owing to increasing environmental concern and restricted availability of petrochemical re-sources. Thus, a great deal of attention was paid to renewable resources-derived polymers and to thermosetting materials especially, since they are crosslinked polymers and thus cannot be recycled. Also, most of thermosetting materials contain aromatic monomers, able to confer high mechanical and thermal properties to the network. Therefore, the access to biobased, non-harmful, and available aromatic monomers is one of the main challenges of the years to come. The choice of phenol-based reactants is crucial and depends strongly on various parameters such as availability in large volume, potential toxicity, reactivity and expected properties. Indeed, the impact assessment is of tantamount importance for the substitution of fossil resources. Hence, we have previously studied tanins, lignin-derived vanillin or cardanol. Recently, we studied an interesting natural phenol, eugenol (4-allyl-2-methoxyphenol), which is a major component of clove oil or could be obtained from lignin. It is an aromatic renewable resource with potential to replace some petroleum-based aromatic monomers. We interestingly synthesized a new platform of eugenol-derived monomers and developed a series of various polymers therefrom.

Various aromatic building blocks bearing polymerizable functions were synthesized: epoxy, amine, acid, carbonate, alcohol, (meth)acrylates... These bio-based aromatic monomers can potentially lead to numerous polymers. The substitution of bisphenol A was studied in epoxy thermosets. Materials were prepared from the biobased epoxy monomers obtained from eugenol. Their thermo-mechanical properties were investigated and the effect of the monomer structure was discussed. High Tg phenol-free phenolic thermosets have been synthesized. Phenol and formaldehyde free phenolic thermosets were also prepared with high thermal stability. We also interestingly synthesized a new platform of eugenol-derived methacrylates and studied for the first time their reactivity in radical aqueous emulsion (and miniemulsion) polymerization.

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## NATHANAËL GUIGO

ECO-FRIENDLY MATERIALS AND POLYMERS TEAM (MAPEC)
Institute of Chemistry of Nice, ICN UMR CNRS 7272
University Côte d'Azur
Parc Valrose, Nice cedex 02, France.

Nathanael Guigo, aged 40, is an associate professor recruited in 2010 at Université Côte d'Azur. He is specialized in physical chemistry of polymers with a peculiar interest on sustainable polymeric materials (e.g. preparation and characterization of biobased polymers) within a circular approach (e.g. valorization of co-products from biorefineries like humins or lignins). He has worked on biobased furanic polymers. His scientific expertise relies to the structure/property relations in polymers such as re-organization during processing, physical ageing, glass transition behaviour, cross-linking, crystallization, thermal degradation, etc.. He has published more than 60 publications in internationally recognized journals and one awarded patent (ORCID 0000-0002-0858-4093).

N. Guigo has actively participated in EU projects always in close connection with industry. He did his PhD in the frame of the Ecobinders Project (FP6) on eco-friendly solutions for binders in automotive and construction area. He worked as a Postdoctoral fellow within a FEDER project NAFI on preparing sustainable ski-boards. From 2010, N. Guigo has set up and managed the BIOFUR EU project (IAPP scheme in FP7) followed by the HUGS projects (EID-ITN in H2020). N. Guigo is currently the Vice-Chair of the COST Action FUR4SUSTAIN (CA18220).

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# MONITORING FAST ORGANIZATION PROCESSES IN POLYMERS

The organizational processes in polymers are connected with their properties and on how these properties can be tuned for reaching desired performances. These reorganizations at macromolecular scale involve thermodynamic and kinetic aspects that can be monitored by thermo-analytical techniques. Some of these processes are very fast and consequently they are not adequately investigated with regular analytical devices like Differential Scanning Calorimetry. The emergence of chips calorimeters like Fast Scanning Calorimetry allows to reach very fast cooling and heating rates.

This lecture aims at providing three examples on how fast organization processes in polymers can be investigated via the correlation between different techniques and approaches.

- **Fast crystallization of polytetrafluoroethylene (PTFE)**

First, Polytetrafluoroethylene (PTFE) is a well-known smart polymer that has non-adhesive, anti-frictional and insulating properties and has thus found a wide range of applications in coatings, sealings, solid lubricant and so on. This fluoropolymer presents ordered microstructures which lead to outstanding mechanical properties at macroscopic scale. Compared to other semi-crystalline polymers, PTFE demonstrates a very fast crystallization process on cooling.

This study explores for the first time the nonisothermal PTFE ultra-fast crystallization under tremendously fast cooling rates (up to 800 000 K.s-1) achieved by using Fast Scanning Calorimetry (FSC) and Ultra-Fast Scanning Calorimetry (UFSC). Both FSC and UFSC data show that it is impossible to bypass the crystallization and so to reach a metastable glassy state even for the fastest cooling rate employed (800 000 K.s-1). The crystals formed in such conditions are slightly less stable than those produced under slower cooling rates as reflected by a shift of the melting peak to lower temperature. SEM observations confirms that only axialite crystals forms under fast cooling while concentric arrangement of ribbon-like crystals are obtained at slow cooling rate.

- **Nucleation and self-nucleation in Poly(ethylene 2,5-furandicarboxylate)**

Poly(ethylene 2,5-furandicarboxylate) (PEF) is a new emergent 100 % bio-based polyester derived from copolymerization of both bio-derived 2,5-furandicarboxylic acid (FDCA) and ethylene glycol (EG). It has a chemical structure analogue to petrochemical based Poly(ethylene terephtalate) (PET). PEF is currently receiving increasing attention due to its excellent barrier and thermal properties. FSC measurements were carried out in order to highlight nuclei formation during both cooling from the melt (non-isothermal conditions) and annealing at either low- or high- temperatures (isothermal conditions). Investigations on nucleation at high temperatures below polymer melting point were conducted to get knowledge about polymer self-nucleation behavior. Annealing in temperature range close to the glass transition resulted in significantly longer onset time of nucleation. On the other hand, annealing close to the melting allowed to determine a narrow temperature range for self-nucleation which can positively impact the overall crystallization rate.

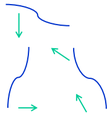
- **Atypical gelation on heating of gelatin solutions**

Gelatin is a polypeptide based material made from collagen. It has a wide range of applications in the areas of pharmaceutics, food, and cosmetics. The typical sol–gel conversion of aqueous gelatin solution occurs on cooling. Such gelation on cooling can be avoided either by dramatically decreasing the gel concentration or increasing the cooling rate or both so that a metastable supercooled solution is formed. The present study for the first time explores the process of gelation on heating of supercooled solutions. This resulting process bears analogy with the cold crystallization observed in polymers.

## Conclusion

- ❑ Gelation of gelatin solution on cooling can be avoided and the resulting supercooled solutions turn into gels on heating
- ❑ Atypical process of gelation has positive temperature dependence and kinetic features depend on concentration

**Diluted solutions**



Diffusion constrains continously increases with crosslinking → *E* increases

**Concentrated solutions**

Diffusion constrains is already high; formation of stable nuclei is more problematic → *E* decreases

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Funded by the Horizon 2020 Framework Programme of the European Union

Cost Action CA18220