IMT Mines Albi-Carmaux
École Mines-Télécom

IMT Mines Alès
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PhD thesis proposal at IMT Mines Albi and IMT Mines Alès

Development of biopolymer foams by supercritical CO_2 assisted extrusion

The development of new polymers and bio-based composites should gradually overcome fossil resources and offer new materials with improved properties such as biodegradability, safety, lightness, ... Extrusion assisted by a supercritical fluid, such as CO_2, has proved over the last decade to be a technology of choice for the development of these new materials. The RAPSODEE laboratory (UMR IMT Mines Albi-CNRS) has made it a specialty and collaborates on this research topic with the C2MA laboratory (IMT Mines Alès).

As part of this thesis, we propose to develop a continuous process for the elaboration of micro- (and/or nano-) structured porous biopolymer materials. The work will aim to study a continuous process to develop innovative cellular biopolymers to meet the challenge of density reduction and homogeneous decrease in pore sizes in cellular polymeric structure. To achieve these porous materials, the extrusion technique coupled with supercritical CO_2 injection is a relevant solution. Indeed, depending on the operating conditions and the solubility of CO_2 in the (bio)-polymer used, its injection into the extruder will modify the rheological properties of the polymer and allows to modulate the extrusion conditions. Moreover, supercritical CO_2 play the role of physical foaming agent during the pressure drop that undergoes the polymer during at the exit of the extruder die.

This research work presents a strong experimental component and consists in the development of biopolymer foams of controlled shapes and porosity. Indeed, it is essential to extend the scope of the process to the manufacture of foams of various shapes and at different scales. The nucleation and the growth of the bubbles making it possible to create the porous structure will be studied through the operating conditions (i.e. the nature of the polymers and their mixtures, the temperatures of the polymer and the extruder die, the geometry of the die, the content of CO_2, the addition of charges and/or micro- / nano-metric reinforcements,...). Particular attention will be given to the study of the microstructure of the resulted foams in connection with their functional properties. Several analytical techniques will be used, among others: pycnometry (H_2O and He), environmental scanning electron microscopy (ESEM), oscillatory and capillary rheometry, thermal characterization (DSC, TGA), study of crystallinity, study of thermo-mechanical properties of the foams.

This PhD work will therefore better understand the phenomenology of porosity formation during the supercritical fluid assisted extrusion process, which is still largely misunderstood, and whose control and monitoring remains problematic. Particular attention will be given to the control of open or closed porosity, this structural parameter conditioning indeed many properties of foams: thermal conductivity, damping, resilience,... Moreover, this work could lead to new applications such as for example galenic forms allowing a control of the gastric retention time or in the biomedical field by proposing materials whose porous structure allows the vascular recolonization (scaffolds). This process is finally likely to improve the grinding ability of the polymers thus manufactured, hence opening a door to easier subsequent shaping or better recyclability of all types of polymers (well beyond the biopolymers that make the focus of this study).

The experimental data will serve as a basis for modeling the process. Indeed, a first model has already been developed in previous works to represent the phenomena of nucleation and growth within the polymer during its passage through the die. This thesis may also be an opportunity to contribute to the improvement of this model.

The study will benefit on the complementarity and experience of the two laboratories in Albi and Alès on these topics [1-3]. The PhD student will be based in Alès during the 1st year of the thesis and in Albi during the 2nd and 3rd years of the thesis. Short internships in each laboratory are also expected. The doctoral school will be MEGeP ED-468 (http://www.ed-megep.fr/)

[1] Le Moigne, N., Sauceau, M., Chauvet, M., Bénézet, J.-C., Fages, J. Microcellular foaming of (nano)biocomposites by continuous extrusion assisted by supercritical CO_2 (2018) ACS Symposium Series, 1304, pp. 171-188.

[2] Hijazi, N., Le Moigne, N., Rodier, E., Sauceau, M., Vincent, T., Benezet, J.-C., Fages, J. (2018) Biocomposite films based on poly(lactic acid) and chitosan nanoparticles: Elaboration, microstructural and thermal characterization (2018) Polymer Engineering and Science, 59:E350–E360.

[3] Le Moigne, N., Sauceau, M., Benyakhlef, M., Jemai, R., Benezet, J.-C., Rodier, E., Lopez-Cuesta, J.-M., Fages, J. Foaming of poly(3-hydroxybutyrate-co-3-hydroxyvalerate)/organo-clays nano-biocomposites by a continuous supercritical CO_2 assisted extrusion process (2014) European Polymer Journal, 61 (1), pp. 157-171.

Thesis supervisors: Prof. Jacques FAGES (Albi) and Dr. Jean-Charles BENEZET (Alès)
Co-supervisors: Drs. Martial SAUCEAU and Romain SESCOUSSE (Albi) and Dr. Nicolas LE MOIGNE (Alès)
Salary: € 21,611 gross annual (i.e. € 1,800 gross monthly). Possibility of teaching hours capped at 64h per year.

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Profile: Engineer (or Master II) in Materials Science and Engineering with good knowledge in polymers and processing. He (she) must have demonstrated during the course of his (her) study an aptitude for experimental research. He (she) must be autonomous, rigor, curious with excellent communication skills (oral and written, in French and English) and teamwork.

Send CV and cover letter to the first 2 email addresses above.