



# Lyon Polymer Materials Science

Master internships  
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## **Micro/nanoencapsulation of natural antimicrobial compounds for functional biodegradable polymer films**

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**Laboratories :** IMP and LAGEPP

Biodegradable polymers such as poly(butylene succinate-co-adipate) (PBSA), poly(butylene succinate) (PBS) and polylactic acid (PLA) are increasingly considered as attractive alternatives to conventional plastics for food packaging. Their bio-based or partially bio-based origin, their controlled biodegradability and their ability to be processed into films via melting process make them compatible with strategies aimed at reducing the environmental footprint of packaging materials. However, these polymers do not inherently exhibit antimicrobial properties which limits their ability to protect foods that are sensitive to microbial contamination.

To overcome this limitation, the incorporation of natural antimicrobial compounds, particularly essential oils, has been widely explored. These agents offer strong antimicrobial activity, a natural origin and a promising potential for active packaging applications. Nevertheless, their direct incorporation into biodegradable polymers remains challenging during melt processing: their volatility leads to significant losses, and their rapid migration within the film drastically reduces their long-term effectiveness. In addition, the presence of essential oils is known to induce plasticization, and reduce polymer crystallinity. These effects can alter the mechanical properties, degrade the barrier performance of the films, making it difficult to obtain functional and stable active materials.

To address these constraints, the current project focuses on the micro- or nanoencapsulation of the active agent prior to its incorporation into the polymer. Encapsulation protects the essential oil during melting process, limits its evaporation and enables a more controlled release. This strategy improves the distribution and stability of the active compound and allows the production of biodegradable films with more durable antimicrobial functionality.

The objective of this project is to study how encapsulation influences the preservation and release of the natural compound, as well as the antimicrobial performance of PBSA, PBS or PLA films, in direct comparison with the simple mixing approach used previously. This work aims to identify the key parameters governing the stability and effectiveness of the encapsulated antimicrobial systems.

## REcycling of PolyEthylene into long-chain Aldehyde Telechelics (REPEAT)

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Laboratories : CP2M and ICBMS

Polyethylene (PE) is the most abundant plastic (>130 MT produced yearly), used in many applications in packaging, materials and medical care, owing to their low cost, lightweight and exceptional properties.<sup>1</sup> Yet in 2024, the recycling rate of this polymer was limited to 29% in Europe, mainly attributed to the mechanical recycling of HDPE. Viewing PE as long-chain hydrocarbons, chemical recycling could complement simpler reprocessing of PE melts, de facto developing a waste-to-chemicals industry. In particular, focusing on preserving the polyethylene backbone without resorting to extensive deconstruction could be particularly sustainable; energy-wise. Chemical recycling of large quantities of waste polyethylene grades into value-added products is thus appealing but remains challenging due to the inherent inertness of the PE backbone.

Since PE is constituted exclusively of C-C and C-H bonds, its reactivity potential is rather low, which impedes its chemical recycling rate (without resorting to harsh treatments such as high-T°C pyrolysis). Initial approaches in chemical recycling of PE have been reported, focusing on milder catalytic pyrolysis<sup>2</sup>, hydrogenolysis<sup>3</sup> or ethenolysis<sup>4</sup> processes to obtain a mixture of gas/liquid/waxes as future sources of monomers, fuels or aromatic solvents. However, most of these methods still rely on high-T°C processes (>350°C), and require sensitive and sophisticated catalysts.

In this context, we propose to develop an original chemical upcycling strategy of polyethylene to aldehydes *via* oxidative or metathesis transformations (Figure 1). Dehydrogenated polyethylene derivatives will be first prepared through a ball-milling process (as a representative example of the use of mechanical stress, implementable at larger scales industrially), affording materials with olefin contents ranging from 0.1% to 4%, harnessing various catalysts.<sup>5</sup> Primarily, the controlled oxidative cleavage of these dehydrogenated intermediates will be investigated, leading to the formation of the aldehydes. These oxidation products will be further converted into the corresponding alcohols (preferred) and acids (over-oxidation), which can serve as precursors for the “re-”synthesis of polyethylene-like materials. This route was initially demonstrated on renewable substrates such as vegetable oils, as reported by N. Duguet and E. Métay<sup>6-7</sup> in ICBMS, emphasizing its strong potential for extension to partially unsaturated polymers, including dehydrogenated polyethylenes and ethylene-butadiene rubbers (EBR). Alternatively, the olefin metathesis of these substrates with vinylene carbonate will be investigated to obtain the corresponding telechelic long-chain aldehydes. These products could subsequently be transformed into polyethylene-like materials containing either ester or hydroxyl-ketone functionalities within the chain, *via* Tishchenko coupling or traditional benzoin condensation. All of the intermediates and eco-designed polyethylene-derived products will be characterized harnessing FT-IR, GPC-IR, and NMR analyses.

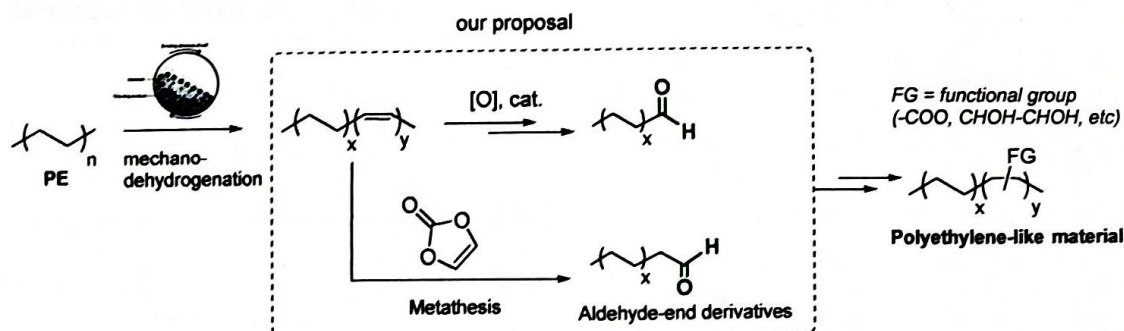


Figure 1: Our oxidation and metathesis approaches toward polyethylene-like materials.

<sup>1</sup> Mecking *et al.*, *Chem. Rev.* **2024**, 124, 5, 2327-2351

<sup>2</sup> Phan *et al.*, *Green Chem.* **2018**, 20, 1813-1823.

<sup>3</sup> Scott *et al.*, *Science* **2020**, 370, 437-441.

<sup>4</sup> Hartwig *et al.*, *Science* **2022**, 377, 1561-1566.

<sup>5</sup> Dr. Adamu Aminu Idris' PhD, defended @UCBL, 2023 – ED206 (supervision: Monteil & Raynaud), and submitted manuscript

<sup>6</sup> Duguet *et al.*, *ChemSusChem* **2021**, 14, 379-386.

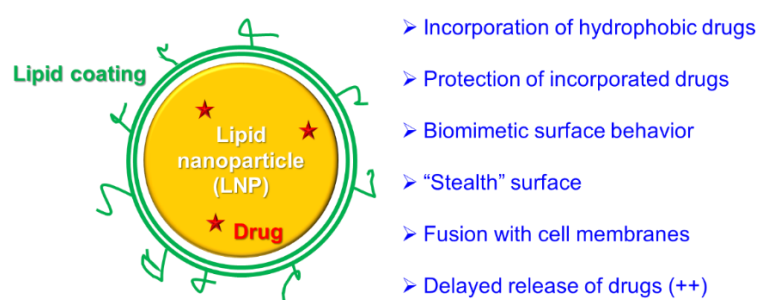
<sup>7</sup> Métay and Lemaire *et al.*, *ChemSusChem* **2018**, 11, 3431-3437.

## Physicochemical study of biomimetic lipid nanoparticles

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The goal of this master M2 SFRI project would be to characterize an innovative assembly based on a solid lipid nanoparticle carrier (NLC) core, coated with a biomimetic lipid membrane (Figure 1). NLC is expected to incorporate and protect hydrophobic drugs, and the lipid coating provides a biomimetic and a “stealth” surface (due to PEG polymer chains), as well as cell fusion, and delayed release properties to the resulting assembly. Currently, such assemblies are obtained by the lipid membrane reorganization of 120 nm-liposomes onto the 250 nm-NLCs’ surface, according to a physicochemical process previously optimized.



**Fig 1. Representation of the assembly investigated in the requested M2 SFRI project.**

The lipid modification of NLC cores was previously proven by an inversion of surface charge of anionic NLC by cationic membranes. Now, a more thorough colloidal characterization of these assemblies is necessary to add proofs of this lipid modification. To this end, it would be pertinent to achieve observations by confocal microscopy of assemblies with NLCs containing a fluorescent probe (DiD) different from one of lipid membrane (TopFluor PC®). Indeed, a co-localization of the both fluorescent signals would be a supplementary proof of the lipid formulation. In this context, stimulated emission depletion (STED) microscopy could also be investigated to create super-resolution images of these assemblies.

Moreover, a nanoparticle tracking analysis (NTA) would be useful to quantify the amount of NLC really modified by lipid membranes as a function of the “liposomes/NLCs” ratio, and the NLC size (from 100 to 300 nm). If difficulties are encountered to increase the NLC size, an optimization of their elaboration process could be carried out (e.g., by modifying the traditional surfactant agent by Kolliphor®RH40). The inversed system (anionic liposomes with cationic NLCs) will be also studied to examine the charge effect on these final systems.

Finally, a colloidal stability of these assemblies in comparison with NLCs (which show a limited stability in time) would be carried out as a function of time, temperature (4°C or 20 °C), and continuous phase (water, PBS, culture medium). This information would be very important for further biomedical applications of these assemblies.

## **Development and characterization of chitosan nanoparticles and filaments**

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Chitosan, a naturally derived polysaccharide, has attracted considerable attention in biomedical and pharmaceutical research due to its exceptional properties, including biocompatibility, biodegradability, pH sensitivity, and intrinsic antibacterial and antioxidant activities. These characteristics, coupled with its versatility, make chitosan an ideal candidate for developing nanoparticles and filaments with tailored functionalities.

This internship focuses on the development of chitosan-based systems, with the aim of producing and characterizing both chitosan nanoparticles and chitosan filaments. The student will first work on synthesizing chitosan nanoparticles using ionotropic gelation and optimizing key formulation parameters. The resulting nanoparticles will be characterized through dynamic light scattering to determine particle size and polydispersity, zeta potential measurements, and stability studies will be conducted. In parallel, the student will develop chitosan filaments followed by the evaluation of their mechanical properties, morphology, and behavior in aqueous environments. Depending on the results obtained, the possibility of stabilizing Pickering emulsions with the use of chitosan nanoparticles as stabilizing agents may be considered.

Overall, this research aligns with the objectives of the LPMS GI framework by advancing the functional properties of polymer-based materials through innovative design and processing of chitosan nanoparticles and filaments. The outcomes are expected to contribute significantly to the field of polymer chemistry and physicochemistry by providing scalable, reproducible methods for producing high-performance chitosan nanostructures with diverse functional applications.