



Lignin from Micro to NAnosize

Principle investigator: Isabelle ZIEGLER-DEVIN, Laboratoire d'Etudes et de Recherche sur le Matériau Bois (LERMAB)

Collaborations: Laboratoire Lorrain de Chimie Moléculaire (L2CM, UMR 7053), Laboratoire Réactions et Génie des Procédés (LRGP, UMR 7274), Ecole Nationale Supérieure des Industries Chimiques (ENSIC), Institut Galien Paris-Saclay (UMR CNRS 8612), Laboratoire TIMR EA 4297 (UTC/ESCOM)

Thematic action concerned: WP3

Context —

The valorization of lignin is a particularly strategic and promising theme for the bioeconomy in the context of the lignocellulosic biorefinery of the future. To date, this biopolymer is very poorly valorized (only use for energy conversion) although, due to its chemical composition rich in phenolic units, it is a source of high value-added derivatives.

Objectives —

The objective of LIMINA project is to extract and produce nano-lignins from local forestry and agricultural waste by using eco-friendly processes (steam explosion, organosolv process and anti-solvent precipitation); then to valorize them in high potential applications: nanomaterials, biobased composite materials, pickering emulsions or substitution of non-biobased and non-biodegradable nanoparticles (TiO₂) in pharmaceuticals or cosmetic products.

Approaches —

The project is divided into several parts: Extraction and characterisation of macro-lignin, production of lignin nano-suspensions and finally valorization of lignin nanoparticles. Thus, the biomass is ground before undergoing a pre-treatment by steam explosion (thermal, chemical and mechanical pre-treatment of the material) and then an organosolv extraction (pressurised reactor) in order to obtain the macro-lignin (micrometric scale). Physico-chemical characterizations of this lignin are carried out (microscopy, NMR, SEC, FT-IR, laser diffraction granulometry) in order to optimize the pre-treatments. In a second step, macrolignins are solubilized in ethanol, an antisolvent precipitation (water) is then performed to produce lignin nanoparticles. The goal is to produce stable lignin nanosuspensions with a control on the size and the aspect of the particles according to different parameters of anti-solvent precipitation (addition rate, concentration and composition of the solutions, nature of lignin, use of stirring or shear forces). These suspensions are then characterized by transmission electron microscopy, dynamic light scattering and zeta potential measurements. Finally, multiple valorizations are being developed in the framework of collaborations with laboratories or industries (Institut Galien Paris-Saclay, LRGP, LIBIO).

Key results —

The main results are:

- Production of nano-suspensions with particle sizes between 50 and 200nm
- These nano-suspensions are stable over time (+30d)
- It is possible to recover solid nanoparticles by freeze-drying in the presence of cryoprotectors

Main conclusions including key points of discussion —

The production of lignin nanoparticles in a simple and environmentally friendly way is controlled and repeatable. These results are leading to an interest from the scientific community in the framework of high added value applications (Pickering, cosmetics, medicine or materials).

Perspectives —

The main research perspectives are:

- Determine the phenomena involved in nanoprecipitation by anti-solvent.
- Optimize the production parameters of nanolignins.
- To continue the development of the various valorizations of nanolignins.
- Increase the production scale

Valorization —

Scientific:

- Publication in process of reaction (to be submitted in Green Chemistry, IF 11.0).
- Accepted oral communication - ACS Spring 2023, Paper ID 3824081, 30/03/23, Indianapolis.
- Oral communication - LignoCOST seminar (01-02/02/23, Reims).
- Poster presentation - 11èmes Journées Scientifiques du GDR Sciences du bois (16-18/11/22, Nice).

Leveraging effect of the project —

Acquisition of new equipments in order to produce the different lignin nano-suspensions: peristaltic pump, ultra turrax homogenizer, syringe pump. Creation of an anti-solvent precipitation unit in a plexiglass box (protection against dust). Recruitment of 3 trainees (Engineer ENSTIB 3A and two students in L3).