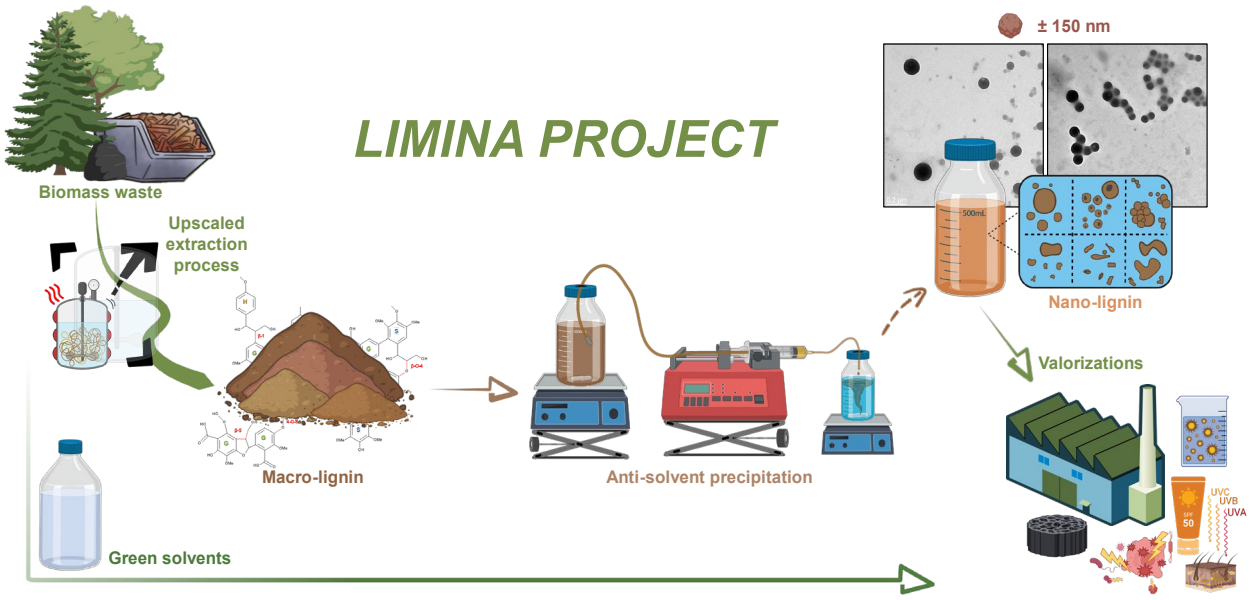
LIMINA



LIgnin from MIcro to NAnosize

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Collaborations : Institut Galien Paris-Saclay (UMR CNRS 8612), Laboratoire TIMR EA 4297 (UTC/ESCOM), Holding Textile Hermès, Universidad de Concepción (Chili), Mahatma Gandhi University (Inde), Universiti Sains Malaysia (Malaisie)

Thematic action concerned *WP3* *Transversal*

Context —
The valorization of lignin biopolymer is an essential theme in the development of lignocellulosic biorefineries within the bioeconomy. Despite its interesting chemical structure, rich in phenolic units, lignin is still considered a waste product from pulping processes (kraft) and is mainly reserved for energy uses.

Objectives —
The aim of the LIMINA project was to extract and then produce lignin nanoparticles from local forest and agricultural waste using innovative, environmentally-friendly processes (steam explosion, organosolv process and anti-solvent precipitation); then to valorize this polymer with enhanced properties in high-potential applications: nanomaterials, properties for bio-based plastics, Pickering emulsions or substitution of non-bio-based, poorly biodegradable nanoparticles in medical and cosmetics products.

Approaches **—**
The project was structured around several axes: firstly, the study of 3 types of biomass (beech, spruce, wheat straw) and 3 different pre-treatments (steam explosion, organosolv, deep eutectic solvents) highlighted the importance of the chemical structure of the extracted lignin (analyzed by NMR, SEC, FT-IR) in its nano-precipitation mechanism by anti-solvent. Next, analysis of precipitation parameters (addition rate, solution concentration, anti-solvent composition and temperature) led to the production of stable suspensions of nano-lignins with controlled properties (size, shape and reactivity characterized by DLS and TEM microscopy). Finally, these suspensions were incorporated into various products such as sunscreens, PVA and Pickering emulsions, notably to improve their anti-UV properties (transmittance analysis by UV-Vis spectrometry).

Key results **—** Key results include

- Development of an innovative, environmentally-friendly process, using only water, ethanol and heat, for the production of nano-lignins from local biomass waste.
- Identification of the impact of biomass type (harwoods, softwoods, herbaceous) and macro-lignin extraction process on the properties of the produced nanoparticles.
- Study and control of production parameters for nano-suspensions stable over time (+90d) with particle sizes ranging from 50 to 200nm.
- Valorization of these nano-lignin suspensions for packaging applications, sun creams and Pickering emulsions, with very interesting results, particularly in terms of UV resistance.
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Main conclusions including key points of discussion **—**
The LIMINA project is breaking new ground in lignin recovery. This complete study, from tree to nanoparticle, offers an innovative perspective on lignin, viewing it as a first-rate resource rather than a mere waste product. This study paves the way for the controlled and repeatable production of renewable and biodegradable nanoparticles, generating increased interest in high-value applications such as medicine, cosmetics and materials.

Perspectives **—** Future work aims to publish the various results (total of 4 publications), master the drying of nanoparticles in order to preserve the properties of the particles between liquid suspension and solid powder, and start the NANOLIGNOV (NANOLIGnin for inNOVative materials) project in order to better develop the valorization of lignin nanoparticles while reinforcing current collaborations.

Valorization —
Conferences: poster presentation at the 11th Journées Scientifiques du GDR Sciences du bois (November 16 - 18, Nice) / oral presentation at the LignoCOST meeting (January 31 - February 2, 2023, Reims) / oral presentation at the ACS Fall 2023 (August 13 - 17, 2023, San Francisco) / oral presentation at the ACS Spring 2024 (March 17 - 24, New Orleans). Publications : Girard, V., Chapuis, H., Brosse, N., Canilho, N., Marchal-Heussler, L., & Ziegler-Devin I. (2024). Lignin nanoparticles: Contribution of biomass types and fractionation for an eco-friendly production. Preprint : hal-04232814 / Girard, V., Chapuis, H., Brosse, N., Canilho, N., Parant, S., Marchal-Heussler, L., & Ziegler-Devin I. (2024). Size and chemical structure impact of lignin 1 biopolymer over performances of next-generation 2 sunscreens. Preprint : hal-04503643 Broadcast: participation in a podcast on LERMAB with the objectives of the LIMINA project.

Leveraging effect of the project —
Introduction of a new research theme in LERMAB's *Procédés de Valorisation du Bois et des Déchets* team, focusing on lignin and leading to the new NANOLIGNOV doctoral project (starting in October 2024 as part of a MESR thesis). Equipment purchase including a peristaltic pump, syringe pump, ultra turrax and freeze-dryer to produce and valorize lignin nanoparticles.