Complutense University of Madrid
Knowledge Transfer Office (OTC)

Production and characterization of nanocellulose products

Brief description

Nanocelluloses are biomaterials derived from cellulose, with at least one of their dimensions in the nanometer scale (<100 nanometers). Thanks to their nanometric structure and cellulose-based nature, they exhibit exceptional properties (Figure 1), including high mechanical strength, large specific surface area, good thermal stability, and high chemical reactivity.

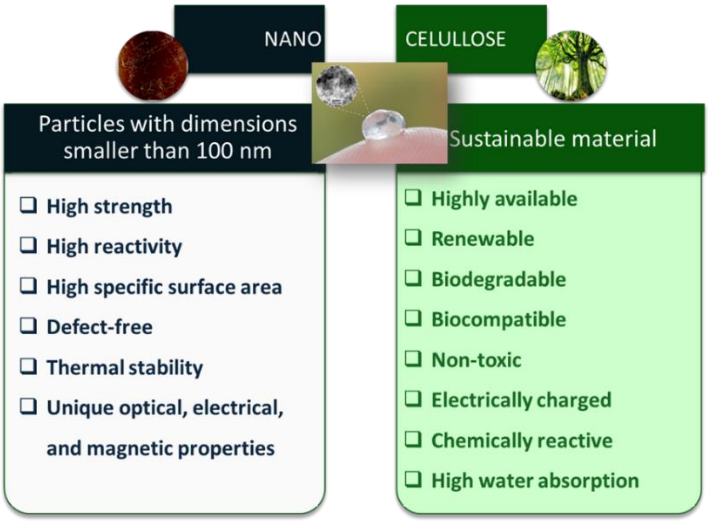

Figure 1. *Properties of nanocellulose*

How does it work?

The term *nanocellulose* encompasses various nano-structured cellulose materials: micro/nanofibrillated cellulose (MFC/NFC), cellulose nanocrystals (CNC), bacterial cellulose (BC), and hairy nanocellulose (Figure 2).

- **Microfibrillated cellulose:** Obtained through intensive mechanical refining and disintegration treatments.
- **Nanofibrillated cellulose:** Produced by mechanical fibrillation using high-pressure homogenization, often preceded by enzymatic, chemical (such as TEMPO-mediated oxidation), or mechanical (refining) pretreatments.
- **Cellulose nanocrystals:** Obtained via acid hydrolysis, typically using sulfuric acid, which removes the amorphous regions of cellulose while leaving the crystalline domains intact.
- **Bacterial cellulose:** A biopolymer produced by bacteria of the *Komagataeibacter* genus, such as *Komagataeibacter xylinus*, through an extracellular biosynthesis process. BC can be synthesized in agitated cultures (forming cellulose aggregates) or static cultures (forming membranes).
- **Hairy nanocellulose:** Sterically stabilized cellulose nanocrystals, produced through periodate oxidation followed by thermal treatment at 80 °C.

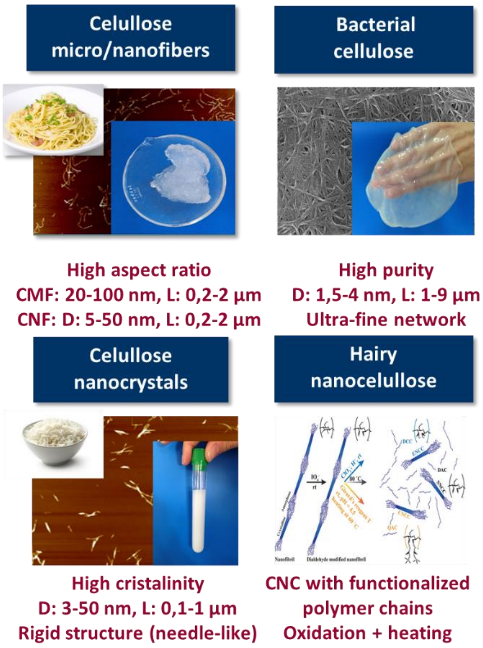

Figure 2. *Types of nanocellulose*

What problem does it solve?

Nanocelluloses serve as reinforcing agents, rheological modifiers, and biomaterials with numerous applications across various industrial sectors. For the optimal implementation of cellulose-based materials, it is necessary to develop ***fit-for-use* nanoproducts tailored to the specific requirements of each application**. To achieve this, nanocelluloses are characterized using the following methods:

- **Microscopy techniques:** Scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atomic force microscopy (AFM) are used to determine the dimensions of nanocellulose materials.
- **Anionic or cationic charge:** Zeta potential and cationic demand are measured (especially when CMF/CNF or CNC has been chemically modified).
- **Transmittance:** This parameter is related to the transparency of cellulose nanomaterials. The presence of impurities and aggregates leads to light absorption, reducing transmittance. Therefore, transmittance provides insight into the degree of nanofibrillation and helps monitor the production process.
- **Nanofibrillation yield:** Indicates the percentage of nanofibrillated material present in the suspension. It is determined by centrifuging a diluted CNF suspension to separate the nanofibrillated material (supernatant) from the non- or partially-fibrillated material (sediment).
- **Polymerization degree (PD):** Refers to the number of repeating glucose monomer units in the cellulose polymer chain. It is determined using the limiting viscosity method with cupriethylenediamine (CED) as the solvent, according to ISO 5351. Once the limiting viscosity of the diluted CNF suspension is known, the PD can be calculated using empirical equations that correlate the two parameters.
- **Carboxyl and aldehyde groups:** When fibers undergo oxidation pretreatment (e.g., TEMPO-mediated or sodium periodate oxidation), it is important to determine the amount of carboxyl and aldehyde groups present in the oxidized pulp. The degree of oxidation significantly impacts the subsequent homogenization process and the final properties of the CNFs.
- **Gel point:** Used to estimate the aspect ratio of nanofibers (length/diameter) and is defined as the lowest volumetric concentration at which all the fibers in the suspension are interconnected, forming a self-supporting network.
- **Degree of dispersion of nanocellulose**: Calculated based on the gel point and must be optimized for each specific application.
- **Other techniques:** X-ray diffraction (XRD), provides information about the crystallinity, which is particularly important in the case of CNC and Fourier Transform Infrared Spectroscopy (FTIR), used to identify functional groups, which is essential when nanocellulose is functionalized.

What future products will it develop?

Nanocellulose applications span both **high-volume industrial sectors and high value-added markets**. Nanocellulose is primarily used as **reinforcing nanofibers**, for example, in the production of paper and cardboard, construction materials, and polymer-based materials.

They also function as **rheological modifiers** in applications such as paints and coatings, cosmetics, food products, and 3D printing inks. In addition, emerging applications are being developed in areas such as water treatment (as **bioflocculants and bioadsorbents**), biomedicine (in **tissue engineering**), **controlled drug and fertilizer delivery systems**, and **aerogels** for thermal and acoustic insulation, among others.

Competitive advantages compared to other research

The *fit-for-use approach* allows quality to be tailored to each specific application. Competitive advantages can be achieved by adjusting critical parameters for various uses and optimizing production processes.

- It has been shown that low-cost, minimally fibrillated nanocelluloses can be used effectively in many applications.
- Transparency can be controlled based on particle size and degree of fibrillation for CNF, or by the number of cultivation days in the case of BC.
- Aspect ratio control enables optimization of strength and barrier properties.
- Degree of surface functionalization enhances compatibility with polymer matrices and/or increases hydrophobicity. This allows the production of lighter, more sustainable, high-performance materials that can compete with other alternatives such as carbon fibers, synthetic nanoparticles, or conventional additives.
- Purity and biocompatibility make nanocellulose suitable for biomedical applications.

Where has it been developed?

The **Cellulose, Paper, and Advanced Water Treatment Group** has a broad and well-established background in the field of research related to the production, characterization, and application of nanocelluloses. Over the past years, the group has actively participated in numerous nationally competitive research projects (CTQ2012-36868, CTQ2013-48090, CTQ2017-85654, PID2020-113850RB-C21, PCD2021-120964-C21, PID2023-147456OB-C22) and has developed several doctoral theses, reflecting its leadership and innovative capacity in this area. This research activity is complemented by close collaboration with other research centers and companies in the sector, enabling effective knowledge transfer toward high value-added industrial applications.

And moreover…

Nanocellulose hydrogels can be used to produce other structures, such as aerogels through lyophilization, expanding the potential applications of nanocellulose across numerous industrial sectors.

Researcher in charge

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