

Technology Offer

## Extraction of nanocellulose from cellulose pulp in reactive eutectic media

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*A novel green chemistry process to prepare nanocellulose in efficient, safe and sustainable manner based on the use of eutectic, highly concentrated ionic media is offered.*

### Background

Nanocellulose is a general term for nanoscale materials originated from cellulose, the most abundant biopolymer on earth. Relatively recently large attention to nanocellulose grew over the scientific and industrial communities, due to the unique combination of abundancy and sustainability of the precursors and desirable properties of nanocelluloses, tailored for impressive amount of applications.

Plants produce cellulose via photosynthesis by binding carbon dioxide, thus creating biopolymer with neutral/negative carbon footprint that can be harvested instead of being synthesized. This natural polymer is biocompatible and biodegradable.

Cellulose is the polymer responsible for the impressive mechanical strength of tree, and this property is reflected in its nanostructured counterpart to much larger extend, making nanocellulose stronger than steel per weight and stronger than Kevlar, while simultaneously being lightweight. Practically that opens for nanocellulose applications striking from construction sector as reinforcing and rheology modifying additive, all the way to food, biomedicine and cosmetics applications. Nanostructured particles of cellulose are able to form dense transparent films that completely seal gas penetration at much lower thickness than traditional synthetic polymers, and pave the way for environmentally friendly packaging.

Nanocellulose is currently isolated from macroscale bleached pulp fibers by energy-demanding mechanical methods that damage the fibers negatively affecting on mechanical properties. Furthermore, mechanical treatment requires extreme diluting of cellulose colloids to make them processable in homogenizing devices, and produce nanocellulose with broad distribution in fibril diameter and length, making it less suitable for most of the applications.

To decrease the energy requirements, one of the most applied processes use a chemical oxidation with TEMPO. This nitroxyl radical loosens the nanofibrils and makes it easier to mechanically separate them, though native structure of the nanoparticles degrades. Nanocellulose is also isolated from bleached pulp with the assistance of mineral acids, most often H<sub>2</sub>SO<sub>4</sub> and HCl, followed by mechanical methods. Chemical nanocellulose extraction methods pose issues for disposal procedures and requires thorough washing of the products.

Both approaches considerably oxidize cellulose and result in production of anionically modified nanocellulose affecting for instance its thermal stability and hindering applications in construction materials and electronics. Often nanocellulose is modified with positive functionalities as prerequisite for improved adhesion to negatively charged surfaces, like that of paper, or to improve sorption of anionic species for wastewater treatment. As done post-synthetically, those procedures considerably add on preparation time and price.

### Technology

This technology aims at meeting the requirements of green chemistry to prepare nanocellulose in efficient, safe and sustainable manner with minimal impact on the environment. With the proposed method, nanocellulose is extracted from lignocellulosic biomass and functionalized with cationic groups in a one-step procedure, minimally affecting the native structure and properties of the biopolymer.

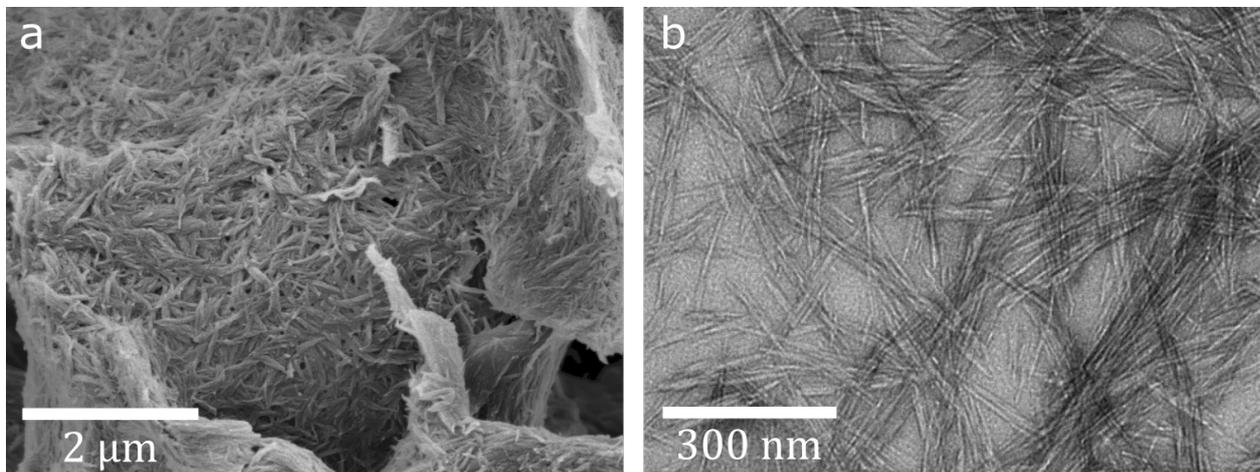
This technology is based on the use of a eutectic, a highly concentrated ionic media similar to that found in living cells under extreme cold or dry conditions. Abundant in the hydrogen bonding, the eutectic media is suitable for the treatment of the cellulose due to its plentiful hydroxyl groups. The increasing interest in technologies based on those media originates from the flexibility to adjust their properties to different biorefinery needs by simply varying the composition of the eutectic media. The simplicity of preparation by simple mixing of the components, combined with the use of components with low or no toxicity at low melting temperature makes eutectic media the optimal condition to process natural carbohydrate polymers in a sustainable fashion. A clear advantage for the biomass treatment is its high tolerance to the presence of water; in fact, some amounts of water generally improve the performance.

By means of the present technology it is possible to extract cationic nanocellulose by short-time heating at approximately 140°C the cellulose feedstock in a eutectic media comprising ammonium formate (a low melting salt composed of two of the cheapest mass chemicals, ammonia and formic acid) and an organic acid chosen from naturally occurring organic acids or those obtained from biomass hydrolysis. The cationic nanocellulose obtained is a nanocrystalline cellulose (cellulose nanocrystals, CNC) of 10-20 nm diameter and up to 200 nm length (see figure).

The relatively mild operation conditions and the possibility to use natural organic acids obtained from biomass hydrolysis confer sustainable character to the process and safety of use.

### Advantages

- Obtain cationically modified nanocellulose in a one-step procedure
- Obtain nanocellulose of 10-20 nm diameter and up to 200 nm length
- Low temperature operating procedure and use of non-toxic and sustainable materials
- Use of cheap and readily available materials



**Figure:** (a) Scanning electron microscopy (SEM) and (b) Transmission electron microscopy (TEM) image of cationic nanocellulose extracted from microcrystalline cellulose.

### Patent Information

PCT priority patent application filed in August 2020.

### Literature

E. E. Jaekel, S. Filonenko, J. A. Sirviö, M. Antonietti: "One-step method for the preparation of cationic nanocellulose in reactive eutectic media"; Green Chem., 2021, 23, 2317

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