IIT MADRAS
Indian Institute of Technology Madras

Technology Transfer Office
TTO - IPM Cell

Industrial Consultancy & Sponsored Research (IC&SR)

METHODS FOR PREPARING NANOFIBRILLATED CELLULOSE (NFC) AND NANO CRYSTALLINE CELLULOSE (NCC) FROM CELLULOSE PULP

IITM Technology Available for Licensing

Problem Statement

- Cellulose nanomaterials are in high demand because of their specific properties such as **sustainability**, biodegradability, **bio-compatibility** for human and animal use, **pseudo-plasticity** (thixotropic), **high aspect ratio** and **scaffolding** ability.
- However, **production cost of NFC and NCC is high** due to challenges such as **high energy** consumption, use of large quantities of **hazardous chemicals** such as strong acids and post-processing **treatment of waste**.
- There is a need for **developing a simple low energy and green process** that uses less harmful, water soluble, easily recoverable and recyclable chemicals requiring simplified purification and concentration processes.

Intellectual Property

- IITM IDF Ref.**1654**
- **IN 529915 Patent Granted PCT?**

TRL (Technology Readiness Level)

TRL 4 Technology Validated in Lab

Technology Category/ Market

Category- Micro & Nano Technologies
Industry Classification:

- **NIC (2008)- 3830-** Materials recovery**; 17011-** Manufacture of pulp; **28291-** Manufacture of machinery for making paper pulp, paper, paperboard and articles of paper board

- **Applications-** biomedical applications, water purification, air filter, Nano-catalysts, biocatalysts, and flexible electronics

Market drivers:
The global nano-cellulose market size was valued at USD 351.5 million in 2022 and is projected to grow at a CAGR of 20.1% from 2023 to 2030.

Research Lab

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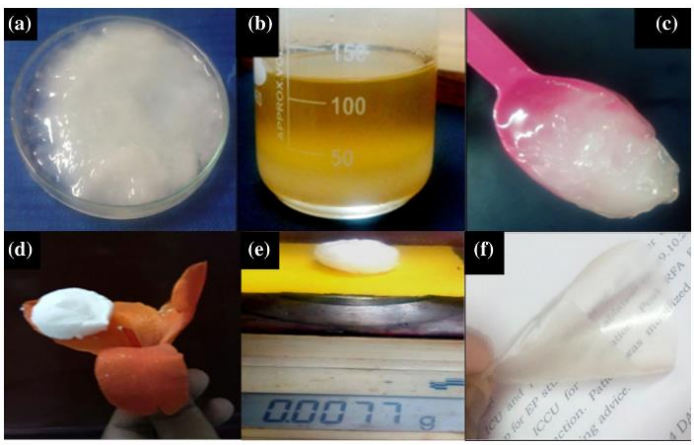

Figure: Photographs of **(a)** NFC obtained after treatment in hot glycerol after centrifugal separation and water rinsing **(b)** NCC that settled after heat treatment in 1 M H2SO4 in glycerol, **(c)** NCC dispersion before freeze drying, **(d)**, **(e)** NCC aerogel obtained after freeze drying, and **(f)** transparent flexible film of NCC

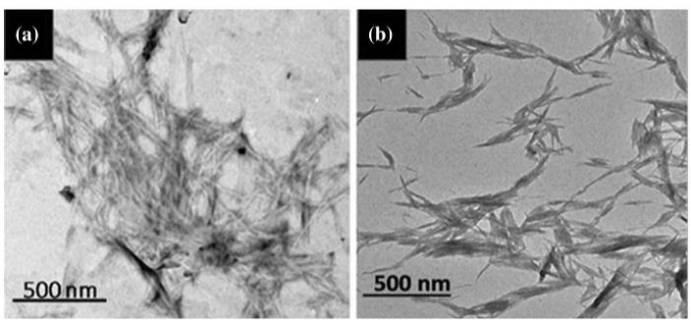

Figure: TEM micrographs of **(a)** NFC from cotton, **(b)** NCC from cotton

Figure: Photographs showing the flocculation of NCC dispersion with time

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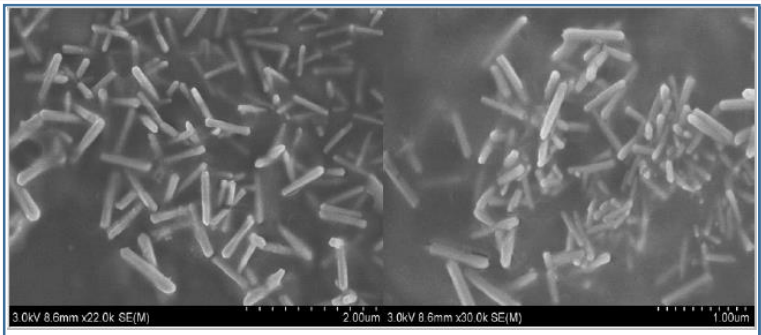

Figure: Illustrates the SEM image of NCC from cotton after HNO_3 / glycerol treatment

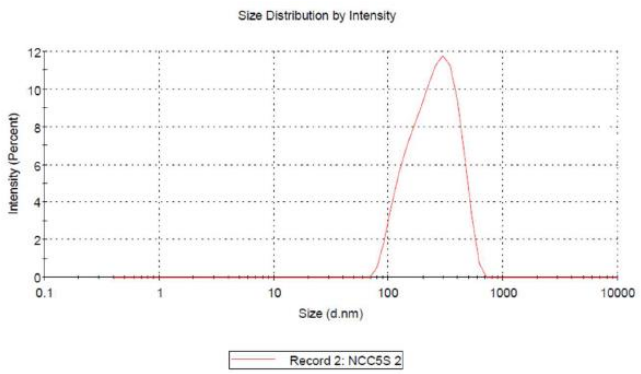

Figure: Illustrates the particle size distribution of NCC from cotton - Average size 265.6 nm

Technology

The method involves a pretreatement stage for removal of lignin and hemicellulose for cellulose sources including corn stover, sugarcane bagasse, banana fiber, rice / wheat straw, waste paper, wood chips, saw dust unless it is cotton or MCC or bleached pulp.

Oven-dried cellulose material such as cotton / microcrystalline cellulose (MCC) along with pretreated (bleached) cellulose pulp is heated in glycerol at 160ºC for 4 hours. The settled residue is obtained after cooling and washed with water. Finally the residue is centrifuged to obtain NFC

The cellulose pulp and cotton can also be treated with H_2SO_4 before heating with glycerol. The process of glycerol treatment is repeated several times followed by washing. Finally the centrifugation process is repeated 6-7 times with double distilled water to yield NCC in the turbid supernatant and NFC as the residue in the final cycle of centrifugation.

The cellulose pulp can also be heated in a mixture of 8 ml H_2SO_4 and 150 g Glycerol. The residue is washed and ground mildly and centrifuged repeatedly till the supernatant turns turbid and passes through a 25 µm sieve/mesh. Films that are transparent and very flexible are obtained by drying NCC dispersions in a petridish at 50º C overnight

Key Features / Value Proposition

- The direct heating in glycerol is a green process and does not require the use of large quantity of water to remove all of the unused solvent and other products. The excess glycerol is recovered and reused for the next cycles.
- NFC prepared from cotton and other pretreated cellulose sources with very little lignin by this method can be used directly in food and diverse bio-compatible applications as glycerol is a FDA approved food additive. Whereas, conventional processes that use harmful chemicals have limited applications.
- The versatile technology enables production of nano-cellulose as NFC dispersion, NCC residue and NCC fiilm using a single green process.
- The Crystallinity Index (CI) comparison of the obtained NCC/NFC with cellulose raw material show that the technology yields NCC/NFC of very high crystallinity.
- The technology can work with a variety of easily available raw materials without requiring any additives. Whereas, conventional technologies require additives that increase costs and require further processing.

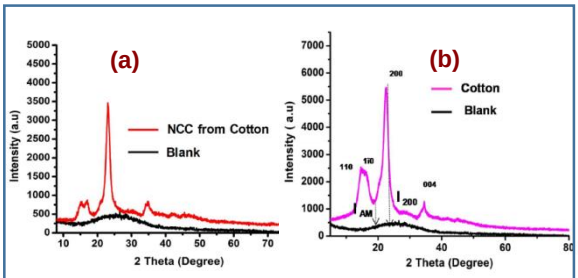

Figure: The Crystalliinity Index (CI) of **(a)** NCC and NFC are much higher than the **(b)** raw materials, because almost all the amorphous regions were dissolved during the reaction while the crystalline regions are retained as they are more resistant to the reagent attack.

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