

CHARACTERISATION OF NANOCRYSTALLINE CELLULOSE WITH WOOD MACERATION TECHNIQUE BY MILLINGTONIA HORTENSIS L.F

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Abstract- Wood fibres are cellulosic material that are extracted from trees and from various other parts of plants that are used to make materials including papers and pulps, etc. Wood maceration process is carried out to separate fibers from the wood core samples which is then viewed under fluorescent electron microscope. Cellulose is extracted from nanocellulose which has various range of useful properties like high viscosity, stiffness, non-toxic nature, lightweight, electrically conductive. The present studies that the nanocellulose is extracted from the wood samples taken from the *Millingtonia hortensis* first the sample are made free from lignin and hemicellulose content and followed by alkali and acid treatment the final sample are characterised using FT-IR and X-Ray diffraction.

Keywords: Wood- Cellulose-fibers-Nanocellulose-Maceration-*Millingtonia hortensis*.

I.INTRODUCTION

Wood fiber is mainly used for the production of medium density fiberboards (MDF). They are used as building material and insulation material in order to regulate the temperature and climate, as well as to provide noise protection. When differences in fibre densities are considered, wood pulp fibres have strength and modulus properties that compare favourably with glass fibres. Softwood pulp fibres with fibre aspect ratios near 100 are easily dispersed into high-density polyethylene or isotactic polypropylene with the help of carboxylic dispersing agents to form mixtures containing 50 weight-percent wood pulp that can be injection moulded. The mechanical properties of the moulded specimens were similar for all types of pulp, including Kraft (bleached and unbleached), mechanical and chemical-mechanical pulps, waste pulps, and reclaimed newspapers (Woodhams *et al.*, 1984). Cellulose is extracted from the wood fibers.

Cellulose is the main component of many plants and there is 50% of the cellulose present in the wood of the plant. Wood fiber has a high level of total porosity, a high level of air-filled porosity, and a low level of readily available water. It also has a higher oxygen diffusion rate than peat.

Nanomaterials can be extracted from cellulose because of its hierarchical structure. Various methods are used to extract nanoparticles from cellulose, resulting in particles with varying crystallinity, surface chemistries, and mechanical properties. Different types of nanocellulose have distinct properties that influence their applicability and functionality; for example, certain types of nanocellulose are better suited for specific applications than others. High Young's modulus/tensile strength (150 GPa/10 GPa for CNCs), a range of aspect ratios that can be accessed depending on particle type, and potential compatibility with other materials such as polymer, protein, and living cells are among the unique properties of nanocellulose. Furthermore, the chemical and material processing options for nanocellulose are extremely versatile, opening up a wide range of structural and functional possibilities. Nano-gels have properties such as swelling, chemical functionality, and degradation, which can be controlled by filling pores in nanogels. (Vaishali Thakur *et al.*, 2021).

Nanocellulose-based nanocomposite materials always have unique properties such as high mechanical strength, high thermal properties, lightweight, and transparency (Pachiyaphanthong *et al.*, 2018). Poly lactic acid (PLA) is one of the main attracting polymeric materials for nanocomposite materials due to its renewable properties (Eduardo Robles *et al.*, 2015).

II.MATERIALS AND METHODS

MACERATION TECHNIQUE

The wood samples are collected from a tree of *Millingtonia hortensis* in PSG College of Arts & Science, Coimbatore. Collected wood samples submerged in 37% of formaldehyde. Prior to start the maceration process, samples were drained for formaldehyde solution to avoid more evaporation of fumes. Three concentrations of nitric acid viz. 40%, 50% and 60% were used. Wood core samples were taken in test tubes, dipped them completely in nitric acid solution and kept in a water bath at 70°C. Maceration process completes in 5–6 hrs with separation of white coloured fibers. Test tubes containing macerated fibers were removed from the water bath and allowed to cool at room temperature.

After cooling, nitric acid was drained and macerated fibers were washed thrice with distilled water and filtered using Whatman Grade 1 filter paper for separation of fibers. Precautions for well drain of formaldehyde solution is necessary prior to immersion in nitric acid and after immersion also as the fumes contains nitrogen oxide, carbon dioxide and nitric acid (when formaldehyde preserved samples immersed in nitric acid, the exothermic reaction occurs due to oxidation of formaldehyde). For slide preparation fibers were stained with 20% safranin solution and again washed with distilled water for destaining of excess safranin. Placed some

amount of fiber suspension on a standard glass slide with the help of ink/medicine dropper and allowed for air drying. Mounting was done in Canada balsam using a cover glass. Use of glycerol enhances visibility of fiber (Mahesh *et al.*, 2015).

EXTRACTION OF NANOCELLULOSE

Extraction of nanocellulose involves of three main procedures,

- a. Lignin removal.
- b. Alkali treatment.
- c. Acid hydrolysis.

Lignin removal and alkali treatment

Firstly, the cellulose is extracted by removal of lignin and hemicellulose and followed by alkali treatment using sodium hydroxide (4%). The bleaching process was carried out with the addition of acetic acid, sodium chlorite, and distilled water for 4 h at 60°C.

Acid hydrolysis

The mixture was cooled and filtered using excess distilled water. Later, the dried filtrate was treated in 10 mol-1 L H₂SO₄ for 40 minutes under continuous stirring. The above mixture was washed with distilled water at 10,000 rpm 10 °C for 10 minutes until the pH was between 5-6. The resulting suspension was sonicated for 30 minutes and freeze-dried to obtain solid nanocellulose (Divya Natraj *et al.*, 2021).

Finally extracted nanocellulose is characterised using various analysis such as Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD).

III. RESULTS AND DISCUSSION

Maceration results

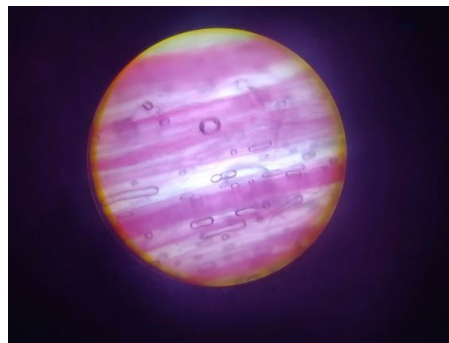


Fig.1 Macerated fibers of *Millingtonia hortensis* after treating in Nitric acid treatment, viewed under Fluorescent Electron Microscope

The maceration done using 50% of nitric acid is found to be effective than other concentration like 40% and 60%, because the fibers (Fig.1) get split well without any damage it is also being less time-consuming process and cost effective. Mahesh *et al.*, 2015 also said that maceration done using 50% concentration is found to be effective Nitric acid acts as an easy and fast resolving agent to break down the middle lamella for separating the cells. Boiled nitric acid separates organs/cells much faster. Results reveal that 50% nitric acid is not only convenient for maceration in hot condition but dissolves other extractives also. This protocol helps to reduce the time and chemicals' cost.

The characterisation of nanocellulose is done using following ways

1. X-Ray diffraction.
2. FT-IR.

X-Ray-diffraction

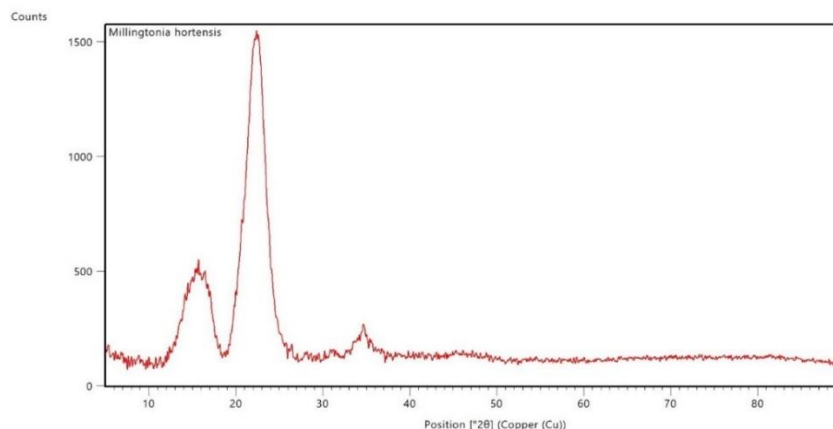


Fig.2 X-Ray diffractometer of nanocellulose extracted from *Millingtonia hortensis*

The X-Ray diffractometer of extracted nanocellulose shows high peaks and the crystallinity index also shows high range. Gond *et al.*, 2021 has observed that the treated and untreated nanocellulose of sugarcane bagasse shows the X-ray diffraction patterns in terms of intensity versus 2θ also the highest value of intensity, crystallinity index and degree of crystallinity shows between 22 and 24° of the 2θ with different intensities. Also in this study shows the same kind of pattern of intensity ranges from 22 and 24° of the 2θ (Fig.No:2).

FT-IR Analysis

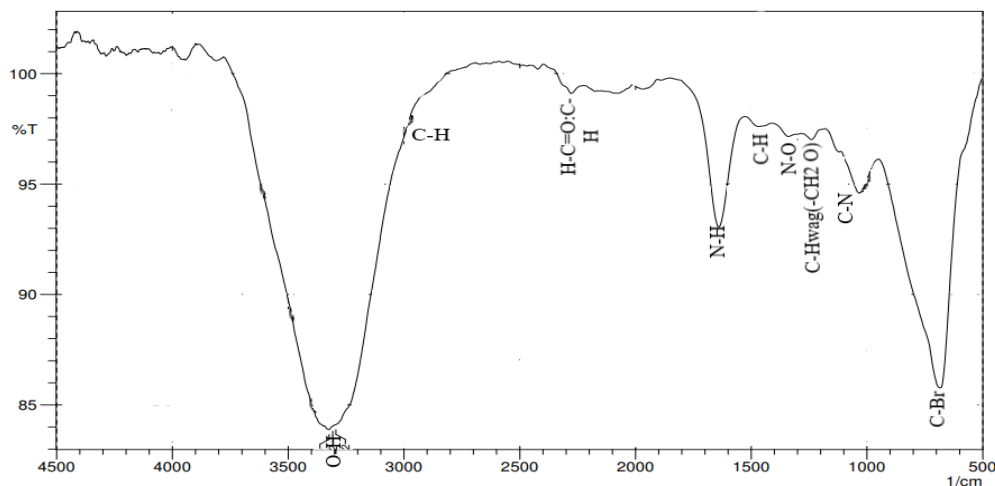


Fig: 3 FT-IR Analysis of extracted nanocellulose from *Millingtonia hortensis*

Gond *et al.*, 2021 FT-IR analysis was carried out to study the change in the chemical structure by recognizing the functional group present in the fibers after treatments and mechanical grinding. There are two absorption zones that are predominated: low-wavelength region (560 - 1731 cm^{-1}) and area of high-wavelength (2912 - 3342 cm^{-1}). In this study the highest peak bond represents the $-\text{OH}$ bonds ranges 3323.35 cm^{-1} and the lowest peak ranges 682.8 (Fig:3)

IV. ACKNOWLEDGEMENT

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