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Isolation and Characterization of Cellulosic Nano Fibrils from Teak Wood

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Abstract

In this study, we solely focused on the isolation of nano cellulose from the teak wood by adopting pulping and acid hydrolysis. Top down method is carried out in the extraction of Cellulosic nano fibrils. The removal of non-cellulosic constituents is monitored by FTIR-ATR and UV-Vis DRS Spectroscopic techniques throughout the isolation process. The acidic hydrolysis is applied to extract the nano fibrils and the crystallinity index increased with the treatment is identified using XRD analyses of the fibrils. The morphological studies of CNFs were done by employing SEM and TEM to ensure the size of the nano fibrils obtained from Teak wood.

Keywords: *Wood fibre, Cellulose, Pulping, Acid Hydrolysis, Nano Cellulosic fibrils, Characterization.*

1 Introduction

The need of finding eco-friendly material to the environment has pushed the scientific community to discover the novel bio materials. Cellulose, the major constituent of all plant materials is most desirable for producing nano based bio materials. Cellulose fibre reinforced polymer composites have received much attention because of their versatile properties. It has lightened up the way for bio fibres and bio composites. Cellulose is an abundant and naturally occurring polymer that can be obtained from numerous resources and cellulose micro fibrils are the basic structural unit of all plants [1]. Cellulose is an organic compound with the formula $(C_6H_{10}O_5)_n$, consisting of a linear chain of several hundreds to over ten thousand β-glucose units. Its structure is organized into fibrils and these are surrounded by lignin and hemicelluloses [2]. The properties of cellulose including good mechanical properties low density and bio degradability depend on the type of cellulose present [3]. There are several types of celluloses (I, II, III, IV & V) and type I shows the best mechanical properties. It is learnt that different cellulosic resources were used as sources for the generation of cellulose nano particles and several methods have been deployed to obtain highly purified nano fibrils from ligno cellulosic fibres. Different raw materials used for production of cellulose nano fibres are soy hulls, pineapple, cassava bagasse, rice husk etc., [4]. Depending on the source of raw material and the disintegration process, nano fibrils with wide range of dimensionality are isolated. In this work, we have extracted cellulosic nano fibrils from the teak wood fibres. The cellulose fibre grade is different for different species fibre grade is different for different species and it decides the strength of the tree [5]. From the previous research studies, we have learnt that the teak wood resources are not employed as precursors for the extraction of cellulose nano particles. The morphology and properties of cellulosic nano fibrils were evaluated using FTIR – ATR, UV-Vis DRS, XRD, SEM and TEM Techniques.

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2 Experimental

2.1 Materials

The sawn teak wood fibre/dust was collected, sorted and cleaned. The teak wood chips were further ground and the fraction passing through 60 mesh (less than 0.250 mm) was selected for the extraction of cellulose.

2.2 Methods

Further the wood fibre is delignified with the analytical grade reagents. Teak wood fibre was cooked with acetone and double distilled water at 1:50 fibre to liquor ratio at 120^{o}C. The washed fibre is further treated with Sodium Hydroxide and the pH was further adjusted by acetic acid. The fibre was again bleached with Sodium chlorite. The mixture was allowed to boil for 5h at 120^{o}C to remove the lignin completely and hemicellulose partially. The residue was subsequently washed with double distilled water. Bleaching was repeated twice to know the efficiency of the bleaching. The fibre was washed several times after bleaching. The obtained cellulose was further hydrolyzed with H_2So_4 at 60^{o}C for an hour. The obtained cellulose nano fibril was washed carefully to remove the residual chemicals and reagents. The washed fibrils as suspension were sonicated using Ultrasonic Sonicator for 10 minutes in an ice bath to avoid overheating.

3 Characterization of Cellulose Nano fibrils

3.1 FTIR-ATR Spectroscopic technique

The FTIR-ATR spectroscopic technique was deployed to learn the removal of lignin and the non-cellulosic, unappealing substance from Cellulose Nano Fibrils. The technique was highly useful in each and every step of extraction. Perkin Elmer FTIR Spectrum Two Spectrometer with ATR accessory and of resolution of 4cm^{-1}at 16 scans, housed in Sophisticated Analytical Instrumentation Facility, St. Peter's Institute of Higher Education and Research is used to carry out this study.

3.2 XRD Analysis

X-ray diffractometer at Alagappa University, equipped with Cu K_ radiation at 40 kV and 30 mA to investigate the XRD spectra of the cellulosic sample.

3.3 SEM Analysis

The surface of wood fibres and the Cellulose Nano Fibrils was captured using Carl Zeiss MA15 / EVO 18 Scanning Electron Microscope at Crystal Growth Centre, Anna University, Chennai.

4 Results and Discussions

The alkali treatment removes a certain amount of lignin, hemicellulose, wax and oils covering the external surface of the fibre cell wall, depolymerises the native cellulose structure, defibrillates the external cellulose microfibrils and exposes short length crystallites. In FTIR spectroscopy, among far and near, the middle Infrared (IR) region i.e., 4000 - 450cm^{-1}, is most commonly employed, as it covers most of the vibrational transitions. The mid IR region is further divided two regions as high frequency and low frequency region. The finger print region between 1450 and 900 cm^{-1}, mostly deformation and few stretching vibrations of the functional groups of the material are characterized in this region, is highly used in the structural determination of a molecule. In Fig.1, the FTIR spectra of ground Teak wood powder, bleached teakwood dry pulp and dry Cellulose Nano Fibrils are given and the removal of lignin and the changes in the physical properties of the CNFs are studied. The disappearance of spectral bands around, 2850 cm^{-1} , 1730 cm^{-1} , 1510 cm^{-1} , 1466 cm^{-1}, 1230-1250 cm^{-1} and 831 cm^{-1} after cooking and bleaching of Lignocellulosic fibres and the appearance of new bands after the cooking and bleaching processes at 1200 cm^{-1} , 1050 cm^{-1} and 560 cm^{-1} are clearly visualized by using FTIR spectra. Lignin, a glue binds the cellulosic fibers together, the elimination of lignin leads cellulose to vibrate freely and upraise of new bands are found in the spectra of cellulose fibres and papers obtained from FTIR technique. After pulp bleaching, intensity or optical density of the cellulosic fibres increases with the absence of lignin and hemicellulose. Hence the appearance and the disappearance of distinctive peaks in cellulose fibers were well explained by FTIR spectroscopy.

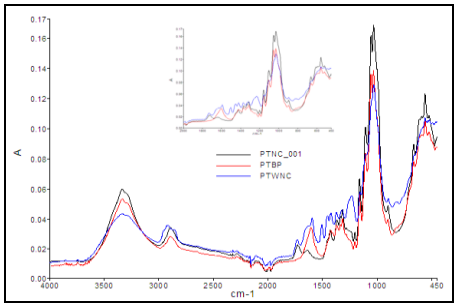

Fig. 1 FTIR spectra of ground Teak wood powder, bleached teakwood dry pulp and dry Cellulose Nano Fibrils in the region of 4000-450cm^{-1}. Inset spectra in the region of 1800 – 450 cm^{-1}.

On removing the noncellulosic constituents of the fibres by chemical treatment, the degree of crystallinity and crystallinity index will change. The fibre constitutes crystalline and amorphous regions. The degree of crystallinity, i.e., the amount of crystalline cellulose present in a cellulosic fibre cannot be exactly defined, as neither the crystalline portions are perfect crystals nor the noncrystalline portion completely disordered. Alkalization of plant fibres changes the surface topography of the fibres and their crystallographic structure.
The effect of various treatments on the crystallinity of the fibres was also calculated. The crystallinity index of the fibre can be calculated using the given formula.

$$I_c = \left[\frac{I_{2\,0\,0} - I_{am}}{I_{2\,0\,0}}\right] \times 100$$

From the peak intensity of the variously treated fibres that were observed, it has been found that acid hydrolysis changes the fibre diameter as well as the crystallinity.

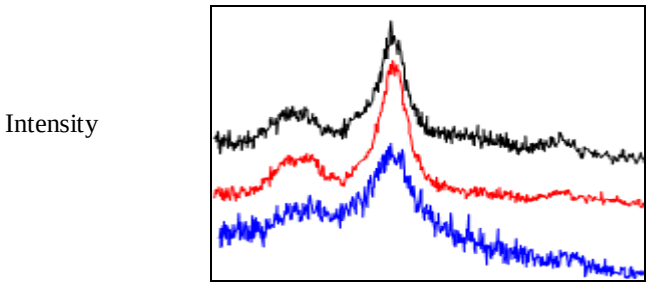

Fig. 2 XRD Pattern of ground Teak wood powder, bleached teakwood dry pulp and dry Cellulose Nano Fibrils.

The SEM micrographs of the original ground teak wood fibre (a), the pure cellulose after the removal of lignin and hemicellulose (b) and finally the nano cellulose fibrils produced by acid hydrolysis of the pure cellulose (c). The diameter of the original teak wood fibre was much bigger and each fiber appears to be composed of several microfibrils. Each elementary fiber possesses a compact structure

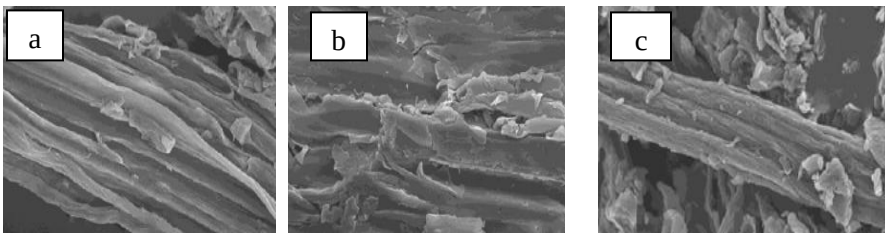

Fig. 3 SEM images of (a) Ground Teak wood powder, (b) Bleached teakwood dry pulp and (c) Dry Cellulose Nano Fibrils.

Conclusions

The maximum yield of Cellulose Nano Fibres is obtained from Teak wood precursor, due to its rich content of cellulose. The Cellulose Nano Fibrils were isolated successfully and characterized to learn the physical and chemical properties of it. This nano fibrils has great potential as reinforcement material and nano composites.

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