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Synthesis of Cellulose Nano-Crystals from Coconut (Cocos Nucifera) Coir Pith

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Abstract: ***Experiments on isolation of cellulose nano-crystals (CNC) from coconut coir pith were performed. Sodium hydroxide pretreatment (NaOH), Hydrogen peroxide (H_2O_2) delignification and sulphuric acid (H_2SO_4) acid hydrolysis were applied for the isolation of CNC from coir pith. The CNC suspension thus obtained was dialyzed, sonicated and lyophilized to get CNC powder. The lyophilized powder was examined to study the physiochemical properties of CNC using Fourier transform infrared spectroscopy (FT-IR), Scanning electron microscopy (SEM), Transmission Electron Microscopy (TEM) and X-ray powder diffraction (XRD). FT-IR studies confirmed the presence of cellulose in the isolated crystals by the molecular conformations of cellulose. From the SEM and TEM images, it was observed that the isolated CNC is in the form of fibrils or rod like structures with an average width of 12-20 nm and average length of 112-308 nm. XRD studies confirm the crystalline nature of the sample and the crystallinity index of CNC was found to be 75.17 %. Coir pith has high lignin content when compared to other agro waste materials. Hence it is suggested to carry out effective delignification process for the isolation of CNC. The morphological and structural characteristics of CNC obtained from coir pith proved that it could be used to produce nano-composite materials.***

Keywords**: *Coir Pith, Cellulose, Acid Hydrolysis, Nanocrystals.***

I. INTRODUCTION

Coconut coir pith is an industrial by product obtained during the separation of coconut coir fiber from coconut husk in coir processing units. Coir pith is a non-fibrous, fluffy and light weight corky material. It is estimated that 1.6 tons of coir pith is generated during the extraction of coir fiber from 10000 coconut husks and reported that 10 lakh tons of coir pith is produced annually in India [36]. Even though it is produced in large quantities, its effective utilization for applications such as organic manure, adsorbents etc., is limited due to its physical and chemical characteristics. Disposal of coir pith is not cost effective due to its low particle density (0.8 g/cc) and leaching of tannins during the rainy seasons leads to severe ground water contamination. As per the survey reports, technology for value addition of coir pith and new products development from coir pith is helpful to maintain economic sustainability of coir industries [37]. But so far, not many high value products have been developed using coir pith. Production of cellulose nano-crystals (CNC) using coir pith as raw material is in line with waste to wealth concept. This is the first attempt to use coir pith as raw material for the synthesis of CNC. CNC finds many applications due to its high aspect ratio, large specific surface area, environmental benefits and low cost apart from its unique mechanical [1] and optical properties [2, 3]. It has wide applications in the production of biofilms, nanocomposite materials, biomedical devices, implants and textiles. It is also used in adhesives, coatings, cosmetics, cements, and foams [4-10] Many researchers reported their results on isolation of cellulose nanocrystals from different biomasses such as cotton, sisal fibers, saw dust, corn straw, garlic skin [11-15] etc. CNC obtained from the different natural fibers have different diameters ranging from 2 to 20 nm with the length from 100 nm to 500 nm based on the natural fibers used as raw material [16]. Acid hydrolysis is one of the methods applied widely by the researchers on wide variety of biomass sources for CNC production. Sulphuric, hydrochloric and phosphoric acids were used for this purpose [17-19]. In this research work, experiments were performed to isolate the CNC from sodium hydroxide and hydrogen peroxide pretreated coir pith by sulphuric acid hydrolysis process. The resultant CNC suspension was dialyzed, sonicated and lyophilized to get CNC powder. Fourier-transform infrared spectroscopy (FT-IR), X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques were applied to characterize the isolated CNC from coir pith

II. MATERIALS AND METHODS

A. Raw Materials

Coir pith was obtained from the Coir Board, Regional Extension Center, Thanjavur, Tamil Nadu, India. Sodium hydroxide (NaOH) pellets (98.5% assay) for alkaline treatment and sulphuric acid (H_2SO_4, 98%) for acid hydrolysis were purchased from Fisher Scientific. Glacial acetic Acid (CH_3COOH; 99.5%) was purchased from Sigma-Aldrich, Hydrogen peroxide (30% w/w H_2O_2) was purchased from Rankem Chemicals to be used as bleaching agent.

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B. Processing of Coir pith

Coir pith was cleaned using distilled water to remove dust and other impurities. Weighed quantity of coir pith was dried in a hot air oven for 2 h at 105°C to remove moisture content.

Drying was repeated by checking the mass of the samples for every 1 h by placing it in the oven until the mass of the sample remains unchanged. [20]. Dried coir pith was sieved using 20-30 mesh sieves to maintain the size uniformity of the coir pith. It was used for further experimental studies and isolation of CNC.

C. Analysis of Coir pith

The cellulose, hemicellulose and lignin present in the coir pith were evaluated using the Technical association of pulp and paper industry (TAPPI) protocols: T 429 cm-10, T 223 cm-01 and T 222 om-02. All the experiments were repeated for three times and the average results were calculated with an experimental error less than 5%.

D. Separation of cellulose

5 g of coir pith was measured and soaked for 4 h using toluene and ethanol (2:1 ratio) as solvents for purification. It was then filtered and wrapped in a filter paper to absorb excess solvent. Dewaxing was done by boiling it in distilled water at 90°C for 3 h in an oil bath heater. The pith was dried in a hot air oven maintained at 60°C for 8 h and used for alkali treatment. The dried coir pith was repeatedly washed with 1 M NaOH solution. For each wash, the dewaxed coir pith was soaked in 1 M of NaOH solution and heated at 70°C for 1 h with constant stirring. The filtrate was discarded and the solid part was repeatedly washed using distilled water for about 7 times until the water was clear. Following the alkali treatment, it was treated with 30% w/w hydrogen peroxide solution for delignification. 60 mL of 30% hydrogen peroxide and 1 mL of 99.5% assay acetic acid to 40 mL of distilled water was mixed and the alkali pretreated coir pith was added to it. The temperature of the mixture was maintained at 60°C using a hot water bath and stirred constantly for 1 h. The coir pith was then separated by filtration and washed with distilled water repeatedly until the coir pith turned off white in color [18, 20-23].

E. Acid Hydrolysis

The cellulose from coir pith was refrigerated overnight by placing it in 100 mL of water. 50 mL of sulphuric acid was added dropwise to the refrigerated cellulose kept in an ice bath. The temperature was maintained below 20°C for 60 min. The aqueous suspension of hydrolyzed product was centrifuged at 8000 rpm for 1 h to remove excess acid and decanting the supernatant until the pH of the filtrate is neutral. The cellulose was then dialyzed against water for 4 days replacing the water daily using cellulose membrane with MWCO 14000. The resulting suspension was then sonicated for 2 h at 40°C at 24 kHz and lyophilized for 72 h. The resulting CNC powder was stored in glass vials at dry condition for further characterization studies [18, 22, 23].

F. Characterization of Cellulose nano-crystals

The structural properties, molecular conformations, hydrogen bonding pattern and presence of functional groups in the CNC were evaluated using FT-IR spectrum within the wave number range of 400 - 4000 cm^{-1}. FT-IR spectrophotometer (Spectrum 100, Perkin Elmer, USA) was used for recording the spectrum at ambient conditions. Samples were analyzed by grinding with KBr.

The samples were analyzed by X-ray powder diffraction system (Rigaku Ultima III, USA) to determine the crystallinity index of the CNC. The freeze dried cellulose nano crystal samples were compressed to obtain total and uniform X-ray exposure. Monochromatic Cu target (Kα) radiation source (λ=1.540600 nm) with a 2Θ angle ranging from 20° to 80° with a step of 0.010^0 and scanning time of 1 s at room temperature was used [13]. Crystallinity index (CrI), was determined using the equation 1 [24].

$$CrI\ (\%) = (I_{002} - I_{am} / I_{002}) \times 100 \quad (1)$$

Where, CrI is the relative degree of crystallinity, known as Crystallinity Index, I_{002} represents the diffraction intensity of both crystalline and amorphous materials (at $2\theta = 22.5^0$), and I_{am} represents the diffraction intensity for amorphous cellulose (at $2\theta = 18^0$ for cellulose I). Study of surface morphology and the microstructures of the cellulose nanocrystals was carried out using Scanning Electron Microscope (JSM 6701F, JEOL, Japan) at accelerating voltage from 5-10 kV. Before analysis, the samples were incubated in the oven at 60^0C and sputtered with gold. Transmission electron microscope (JSM 2100F JEOL, Japan) was used for the determination of morphological characteristics of CNC. The samples for TEM were made by dispersing 0.5 mg of CNC in 10 ml of water. Then the solution was subjected to 5 h of sonication. Images were captured and the dimensions of CNC were determined using 'ImageJ' software

III. RESULTS AND DISCUSSION

A. Chemical analysis of Coir pith

The chemical composition of the coir pith sample used in this study was found and presented in table 1. It was found that the lignin content is high when compared to cellulose and hemicellulose. This is in contrary to other agro residue lignocellulosic biomasses like coconut fiber [25], sugarcane bagasse [26], rice straw [27], wheat straw [28], elephant grass [29] etc., which contains less lignin than cellulose. Presence of high lignin in the coir pith suggests for the effective unit operations and unit processes for the delignification in the isolation of CNC from coir pith.

B. Fourier-transform infrared spectroscopy analysis

FT-IR spectroscopy gives information about chemical structure, functional groups identification, molecular conformation and hydrogen bonding patterns of the sample [25]. FT-IR spectra of raw coir pith (Fig. 1) and the CNC (Fig. 2) were taken to compare the presence of functional groups and other chemical moieties.

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Table- I: Chemical composition of coir pith

Components	% w/w[a]
α-cellulose	28.24±0.03
hemicellulose	14.91±0.16
Lignin	41.60±0.32
Extractives and ash	15.25±0.18

[a] Mean± standard error

From FT-IR spectral analysis of the CNC sample, the dominant peaks are observed at 3375 cm^{-1}, 2905 cm^{-1}, 1641.19 cm^{-1}.

The peak intensity at 3375 cm^{-1} and 2905 cm^{-1} is due to the O-H symmetric and aliphatic C-H stretching vibration of alkyl groups respectively [21]. The peak at 1641.19 cm^{-1} is resulted from the bending vibration of water absorbed by hydrophilic groups. Presence of small quantities of non-cellulosic components is confirmed by the sharp peak at 3375 cm^{-1}. The presence of shoulder at around 1704 cm^{-1} in the spectrum may be due to the presence of carboxyl groups in the *p*-coumaric, ferulic and uronic acids, that were identified in hemicellulose associated with lignin [29]. The weak peaks observed at 1427 cm^{-1} and 1369 cm^{-1} are due to the aromatic skeletal vibrations of syringyl units and phenolic groups in small quantities [31]. FT-IR analysis confirms that the acid hydrolysis process does not collapse the cellulose macromolecules and no new functional groups were added. By comparing the spectra of raw coir pith and CNC, it is confirmed that the cellulose content is high in CNC than the raw coir pith [32].

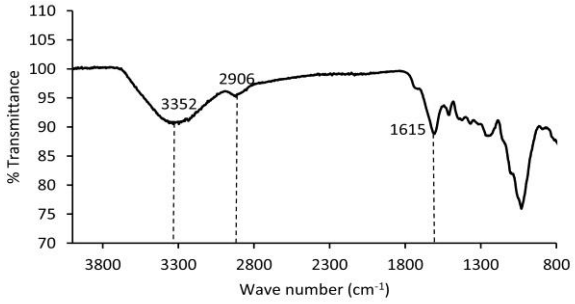

Fig. 1. FT-IR spectra of raw coir pith

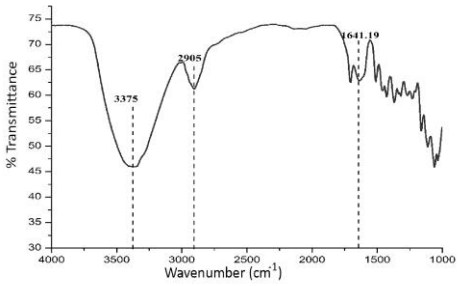

Fig. 2. FT-IR spectra of CNC

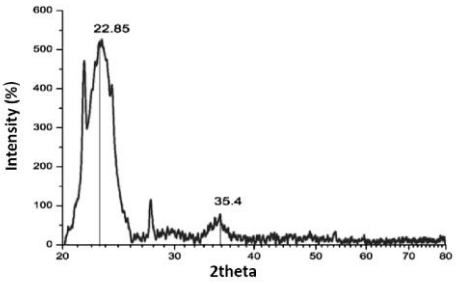

Fig. 3. X-ray diffraction patterns of CNC

C. X-ray Diffraction analysis

Major compounds present in any biomass are cellulose, hemicellulose and lignin. Cellulose molecules are present in crystalline form whereas hemicellulose and lignin are present in amorphous form [32]. Hence the measure of crystallinity of the isolated CNC is accepted as an important characterization technique to confirm the crystalline nature of CNC. XRD pattern of the isolated CNC is given in the Fig. 3.

XRD shows distinct peak at diffraction angle (2θ) =22.85°, which is a characteristic peak of cellulose I crystals corresponding to (200) and (004) lattice planes of cellulose I [33]. The low intense peak at around 35.1° indicates the amorphous arrangement of typical cellulose structure [21]. The high intensity of the peak indicates the crystalline structure of CNC sample. The crystallinity index (CrI) calculated using the equation 1 is 75.17%. . High crystallinity index is required to increase the stiffness and rigidity and strength, conferring a higher resistance to cracks, thus enabling the production of nanocomposites with improved mechanical properties with these CNC [35]. As per the reports, the crystallinity index differs with the source of biomass used for the isolation of CNC (Table II).

D. Scanning Electron Microscopy analysis

The FE-SEM studies show that the CNC are similar in shape and size (Fig. 4a). The SEM image of CNC indicates the crystal shape is mostly in rod or whisker fibrillary with thickness in the range of 12-20 nm thickness (Fig. 4b). Upon closer observation, the ribbon forms are wider with the width ranging from 112-308 nm (Fig. 4c). Based on the 100 nm measurement from SEM image, the fine particles with the width ranging from 30 nm–80 nm (Fig. 4d) are observed. The reduction in size is due to the removal of lignin and

Table- II: Crystallinity index of the CNC obtained from different biomass sources

Sources	Nano cellulose Crystallinity Index	Reference
Bagasse	89%	[16]
Rice husk	79%	[22]
Garlic skin	63%	[15]
Saw dust	80.95%	[13]
Elephant grass	72%	[29]
Cotton linter	90.45%	[35]
Coconut Coir pith	75.17%	Present study

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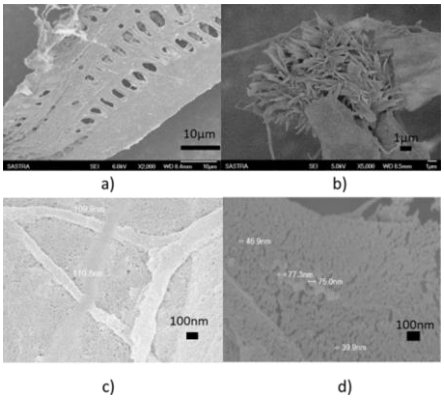

Fig. 4. FESEM image of CNC shown at different scale a) 10 µm; b) 1 µm; c) and d) 100 nm

hemicellulose from the cellulose by acid hydrolysis and further purification of cellulose suspension.

E. Transmission Electron Microscopy analysis

TEM was used to study the morphology of CNC. The acid hydrolysis process digests the amorphous portion of the cellulosic fibers, while leaving the crystalline region intact and eventually reducing the size of the fiber to nanometer scale. The CNC tend to agglomerate, probably due to surface ionic charge which had the crystallites stacked together as a result of the processes. Fig. 5a shows the FE-TEM micrographs of CNCs, at 1 nm scale, small lines known as fringes were observed. The distance between the fringes (100 fringes) was found to be 0.22-0.29 nm. These observations indicated that the CNC are crystalline in nature. High crystallinity was expected to increase the stiffness and rigidity, and therefore strength, conferring a higher resistance to cracks, thus enabling the production of nanocomposites with improved mechanical properties with these nanostructures [35]. From the Selected Area Electron Diffraction (SAED) pattern (Fig. 5b) of the sample, it is noted that the d-spacing of the sample is same as the fringe distance in TEM image. The FE-TEM image reveals the presence of different size particles mixtures i.e., fibers, CNC with non-hydrolyzed cellulose and long CNC in the sample. The dimension of CNCs determined by TEM is in the range of 12-20 nm.

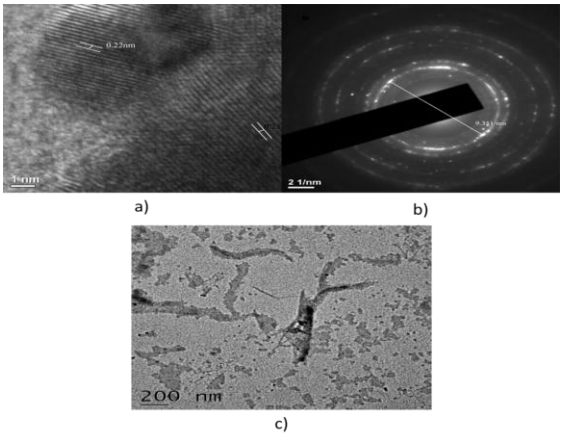

Fig. 5. FETEM image of CNC a) Image of CNC shows the fringe's at 1nm; b) SAED pattern; c) Isolated rod-shaped CNC

IV. CONCLUSION

Coconut coir pith which is an agro biomass and coir industry waste is used for CNC isolation using acid hydrolysis method. From the composition analysis, it is observed that the coir pith has more lignin than cellulose. Hence effective pretreatment and delignification process is suggested to increase the efficiency of further processes. FT-IR results have confirmed the presence of cellulose. SEM and TEM images have shown that the isolated CNC are fibrils or rod like structures with average width of 12-20 nm and average length of 112-308 nm. XRD results have shown high crystallinity of the CNC of about 75.17 %. These high crystalline fibrils of CNC can be further used as reinforcement agents for the manufacture of nanocomposites for different applications. From the results, it is concluded that coir pith could be used as a raw material for the isolation of CNC. Further, techno-economic feasibility studies have to be conducted for the isolation of CNC from the coir pith. Since the demand for CNC is high due to its wide applications in various fields of engineering and technology, this could be the potential raw material and also additional source of income for coir industries.

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