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ORIGINAL RESEARCH PAPER

Structural and Morphological Characterization of Nanocellulose Extracted from Cotton Straw Residue

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ABSTRACT

This study is focused on the synthesis and characterization of cotton straw residue. Nanocellulose was synthesized by chemical method and followed by ultrasonication and cryocrushing. The results of the present study show that the cotton straw residue consists of lignin (27%), hemicellulose (15%), cellulose (32%), and ash content (2.3%). Nanocellulose was characterized by FTIR, XRD, FESEM, TEM, DSC, TGA, and AFM. Two aromatic rings were observed at wavelength 1650.47cm^{-1} and 1436.53 cm^{-1} which indicates that there is a presence of cellulose in the prepared sample which was characterized by FTIR. The structural analysis shows that the material was amorphous and the nanocellulose crystallinity is 23 %. The morphological analysis using FESEM indicates even elongated fiber with a smooth surface and it contains pore in the nanocellulose of cotton residue. TEM analysis indicates that nanocellulose has an irregular shape with a circular rod-like structure of different sizes. The enthalpy of nanocellulose changes at 168.48℃ due to endothermic transition. TGA results show that the nanocellulose is degraded in the temperature range 300-355℃ and low thermal stability was observed during the experiment. AFM result shows the needle shape particle (root square mean roughness = 0.1738nm) and the size of nanocellulose was observed 7.1 nm.

Keywords: *Nanocellulose, Cotton Fiber, Acid Hydrolysis, Cryocrushing, Nanocellulose characterization, Agriculture waste.*

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INTRODUCTION

Cotton is one of the most important commercial cash crops in India which is produced in more than 12 states and Gujarat is the leading cotton-producing state in India. Major cotton-producing nations are China, USA, Pakistan, India, Uzbekistan, Australia, Egypt, Argentina, Greece, etc. India holds 1st rank in the refinement of cotton fiber [1]. Cotton crop residue has many other applications such as fuel for cooking food and fodder for animals [2], pulp and paper [3], energy production [4], heavy metal removal [5], removal of organic matter [6], etc. A few researchers have used effectively cotton crop residue in order to upgrade soil nourishment to further develop crop usefulness by utilizing rice-based harvest practices [7].

Cotton straw consists of constituents such as lignin, cellulose, and hemicellulose. Cellulose is a straight-chain polysaccharides unit made up of numerous glucose monosaccharide units which are linked with β(1-4) linkage of the D-glucose unit with a hydrogen bond[8]. Cellulose could be extracted from a diversity of sources such as firewood, bast filaments, greenswards, bud fiber, seed strings, invertebrates, microorganisms, etc. These sources are mainly categorized into industrial and agricultural waste [9]. Hemicellulose has

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distinct monosaccharide units of glucose, galactose, mannose, xylose, and arabinose which are isomers of each other[10]. Lignin which is a complex structure derived from wood and an integral part of the secondary cell wall of the plant acts as gluing the cellulose units together. It helps in water transportation, provides mechanical strength, and is insoluble in water[11-12]. Hemicellulose is allied with hydrogen bonds and lignin with a covalent bond [13]. Lignin, cellulose, and hemicellulose are interlinked which shows that the fiber is arranged in a crystalline and disorderly manner as shown in Fig. 1.

Various extraction methods of nanocellulose such as mechanical (High-pressure homogenization [14], chemical methods,hammer milling [15], Cryocrushing, high shear homogenization[16-17], high-speed blending [18],micro-fluidization [19], grinding [20] high-intensity ultrasonication [21], and alive enzymes [22] are reported in the literature.

In Northern states, mainly Haryana, after harvesting cotton crops, a lot of residues which include stalks, locules, cotton bolls, leaves, and roots are left in the fields. In the absence of adequate sustainable management practices, a huge quantity of residue is being burnt on-farm to clear the field for sowing the next rabi crops (mainly wheat and mustard). Crop residue burning has become a major environmental challenge that causes health issues due to the emission of CO_2, CO, CH_4, NO_X, and SO_X which is contributing to global warming[23].

This research paper mainly emphasizes the utilization of cotton crop straw for nanocellulose extraction by the chemical method followed by ultrasonication and Cryocrushing. It also focuses on how we overcome the cotton crop residue burning problem to minimize global warming and the best utilization of cotton crop residue.

Novelty of research

In the absence of an adequate sustainable management plan for the reuse of agro-waste, the farmers burn their crop residue on the farm in huge quantities. This burning of the crop has many environmental problems caused by the emission of various gases which results in global warming and climate change. Due to the facts mentioned above, the present study was conducted in order to handle crop residue by extracting cellulose from the agricultural biomass.

MATERIALS AND METHODS

Materials

The cotton straw (Bt-cotton) was collected, cut into small pieces, and washed with tap water. The raw material was washed and dried in sunlight. After drying, the same was grinded and sieved from a 80µm size sieve. The raw powder sample was oven-dried and kept in PVC bottles for further treatment. AR grade chemicals were used in the whole experimentation.

Estimation of Lignocellulosic constituents

Cellulose, hemicellulose, lignin, and ash content were evaluated according to Goering and Van Soest method(1991) [24-25].

Synthesis of Nanocellulose

The powder raw sample was soaked into 4%(w/v) sodium hydroxide (NaOH)and stirred at 80°C for 2 hr. pH level was increased by adding NaOH, and to maintain their pH, the sample was washed with double distilled water. At last, strained excess of cotton straw was collected and alkaline treatment refine hemicellulose and lignin.

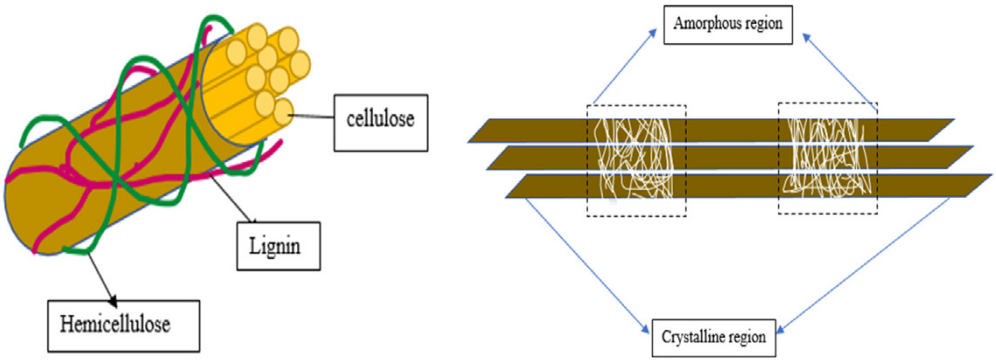

Fig. 1. The binding pattern and structure of lignocellulosic biomass

In bleaching treatment, the filtrate of the previous experiment was treated at 80°C by subsequently adding sodium hypochlorite (NaOCl) 80% (w/v) solution until the bleached white fiber was attained and this process was repeated 4 to 5 times. The sample was cooled, filtered, and washed with distilled water till the pH of the sample become neutral. A white color bleached cellulose fiber was collected and dried at 40-50°C in a hot oven for 10-12 hours. This action liberates lignin and other impurities from the processed sample. To extract nanocellulose fiber, acid hydrolysis was given to cellulose fiber to disrupt the severe arrangement of cellulosic constituents. A 40%(w/v) H_2SO_4 was added to the bleached cellulose fiber and was heated at 40°C for 1 hour. To obtain nanocellulose fiber, the sample was washed with distilled water 4 to 5 times to remove excess acid and maintain the pH neutral. In the sonication process, water was separated from the sample of nanocellulose fiber at 60°C for 1 hour and then further centrifuged the sample at 4000 rpm. After centrifugation, the Cryocrushing process was accomplished to increase the further surface area of nanocellulose fiber in the nanometer. The Liquid Nitrogen (LN_2) was added which solidify the sample and then immediately crushed the material with mortar and pestle. The sample was dried and stored in a PVC bottle for further characterization. Fig. 2 showed the extraction process of nanocellulose.

Characterization of cotton straw nanocellulose
Fourier Transform Infrared Spectroscopy(FTIR)

FTIR is an analytical technique that is used by a material analyst to know a substance's chemical composition. Perkin Elmer spectrums, BX-II (FTIR) spectrophotometer were used. Spectra were recorded of raw material, alkali-treated, bleached and acid hydrolyzed material.

The different processed samples were mixed with KBr, mechanically grinded, and formed a film. Spectrum was obtained in the range of 4400-500 cm^{-1}.

Structural property(XRD)

X-ray diffraction (XRD) was performed on a Rigaku Miniflex-II diffractometer at room temperature with a copper anti cathode operated at 40 kV and 40 mA to check whether the structure of samples is crystalline or amorphous. Diffraction was executed on power samples spread smoothly on a neutral quartz glass sample holder and expose k-alpha radiation source and scanned in the range of 10° to 80° at 2 theta angles. The Segal method was used to calculate the crystallinity index (CrI) [26][26].

$$CrI = \frac{I_{(002)} - I_{(AM)}}{I_{(002)}} \times 100$$

Where: (002) is the extreme crystallinity value and (am) is for the amorphous part of the sample. Higher intensity is diffracted at an angle 2θ = 22° and low-intensity peak is scattered at 2θ =18° angle.

Microscopic study

Field Emission Scanning electron microscope (7610F plus/JEOL) was used to analyze the shape, dimension surface morphology of the sample. The sample was assessed at two stages before and after the chemical treatment. The voltage was accelerated at 30 kV and the sample was coated with sputter coater before treatment. TALOS HR-TEM (Thermofisher) at 200 kV was used to analyze TEM for particle size and shape and their morphology.

Differential Scanning Calorimeter (DSC)

Three mg of sample were placed in the platinum container and heated from 0 to 400°C at 5°C/min in a helium atmosphere. Thermo-analysis was performed on Q-10, TA Instruments Waters.

Thermal Gravimetric Analysis (TGA)

To know the thermal degradation, the sample was analyzed by using SDT Q 600 thermogravimetric equipment. The temperature range for analysis was 25°C-900°C at a heating rate of 10°C/min under a nitrogen atmosphere (10ml/min).

Atomic Force Microscopy (AFM)

The sample was treated with methanol/ethanol mix for dissolving. Bruker multimode 8 AFM was used for AFM analysis.

RESULT AND DISCUSSIONS

Mechanism of lignocellulosic biomass

The raw cotton straw has 27% lignin, 32% cellulose, 15% hemicellulose, and 2.3 % ash content (shown in Table 1). For nanocellulose extraction, the raw material was chemically treated. The alkaline treatment(Sodium hydroxide) removes a certain amount of lignin and hemicellulose

Fig. 2. Extraction of nanocellulose

Table 1. Lignocellulosic biomass content in Percent (%)

Crop Residue	Lignin	Cellulose	Hemicellulose	Ash content	References
Cotton stalk	29.4	40.10	13.60	-	[27]
Corn straw	07.3	42.04	41.02	-	[29]
Cotton stalk	30.9	-	-	1.8	[15]
Rice straw	19.64	38.6	19.7	18.67	[30],[31]
Wheat straw	18.8	52.4	18.2	3.7	[32]
Bagasse	20-30	32-48	19-24	1.5-5	[33]
Cotton straw	27.0	32.0	15.0	2.3	Present study

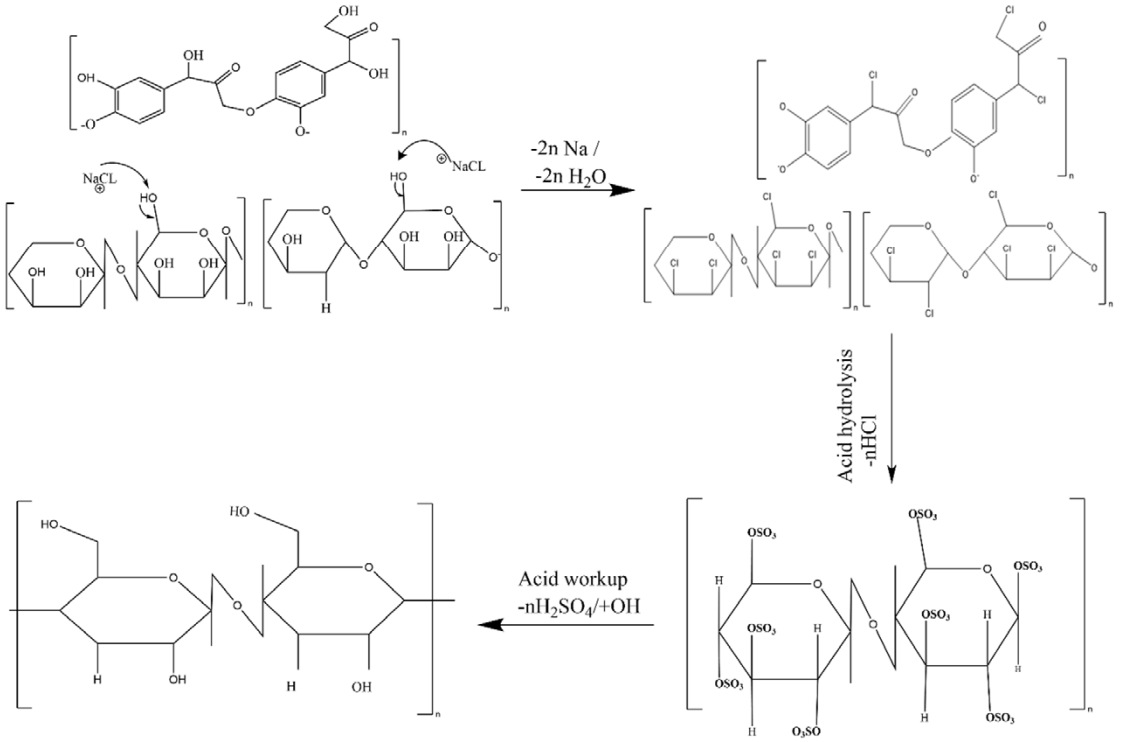

Fig. 3. Mechanism of Nanocellulose Extraction

depending on treatment time and concentration. When sodium hydroxide is treated with raw cotton straw, sodium hydroxide break down into sodium ions and hydroxide ion then they attach with lignin, cellulose, and hemicellulose and sodium ions are left into solution. Now, fiber had an effective surface to treat with sodium chlorite (Bleaching agent). The material was treated with sodium chlorite for the extraction of pure cellulose. Hydroxyl compounds are replaced with chlorite ions and form white smooth surface material. Again the material is neutralized with distilled water to overcome the bleaching process. At last, when the extracted material is treated with 40% H_2SO_4 then, the material starts to degrade and the Chlorite molecule was replaced with OSO_3^{2-} the material is turned into yellow color due to acid concentration. When we did acid workup then all OSO_3^{2-} were replaced with hydroxide ion and pure cellulosic material is formed. Acid hydrolysis follows the reaction given below:

$$H_2SO_4 \rightarrow H^+ + HSO_4^-$$

$$HSO_4^- \rightarrow H^+ + SO_4^{2-} \; or \; OSO_3^{2-}$$

$$OSO_3^{2-} + H_2O \rightarrow OH + H_2SO_4$$

Here, H_2SO_4 is acidic but when we treated it with cellulose material then it loses H^+ ion become basic by forming OSO_3^{2-} as a product. Now, OSO_3^{2-} is replaced with OH^- ion when we neutralize the solution with distilled water (shown in Fig. 3).

Characterization of Nanocellulose

The extracted material was characterized by FTIR (Fourier Transform Infrared Spectroscopy) for chemical composition, XRD (X-ray Diffraction) for the crystallinity of material, and FESEM (Field Emission Scanning Electron microscopy) for surface morphology analysis, TGA(Thermogravimetric analysis) to measure the thermal stability, DSC (Differential scanning calorimetry) to measure the energy absorbed and released by a sample.TEM (Transmission Electron Microscopy) for size and structural morphology.

Fourier Transform Infrared-Spectroscopy

Five spectra were examined for raw material (C1), alkali treatment (C2), bleaching treatment (C3), acid treatment (C4), and nanocellulose(C5)

Table 2. FTIR Reading of Cotton Straw Material

Frequency	Functional group	Raw Material (C1)	Alkali Treatment (C2)	Bleaching (C3)	Acid Treatment (C4)	Nanocellulose (C5)	Reference
4000-3000	OH stretching	3422.19 3857.28	3419.43	3863.31 3742.71 3432.99	3385.50	3857.28 3421.42	[34]
3000-2500	C-H	2928.64	2922.61	2928.64		2901.82	[2]
2000-1650	C=O	1737.85	1728.81	1725.80	1725.80	1734.83	[35]
1670-1600	C=C	1632.92	1638.41	1641.43		1650.47	[36]
1600-1300	N-O	1436.53	1433.52 1352.16	1430.50		1436.53 1319.02	[37]
1400-1000	O-H bonding	1243.69 1108.09 1053.37	1231.63 1162.33 1111.11 1053.79	1165.34 1053.78	1288.88 1168.36 1050.84	1165.34 1114.12 1057.88	[38]
1000-650	β-glycosidic linkage	900.18 822.87 657.76	891.14 665.16	827.87 668.17	885.12 851.97	894.13 668.17	[39]

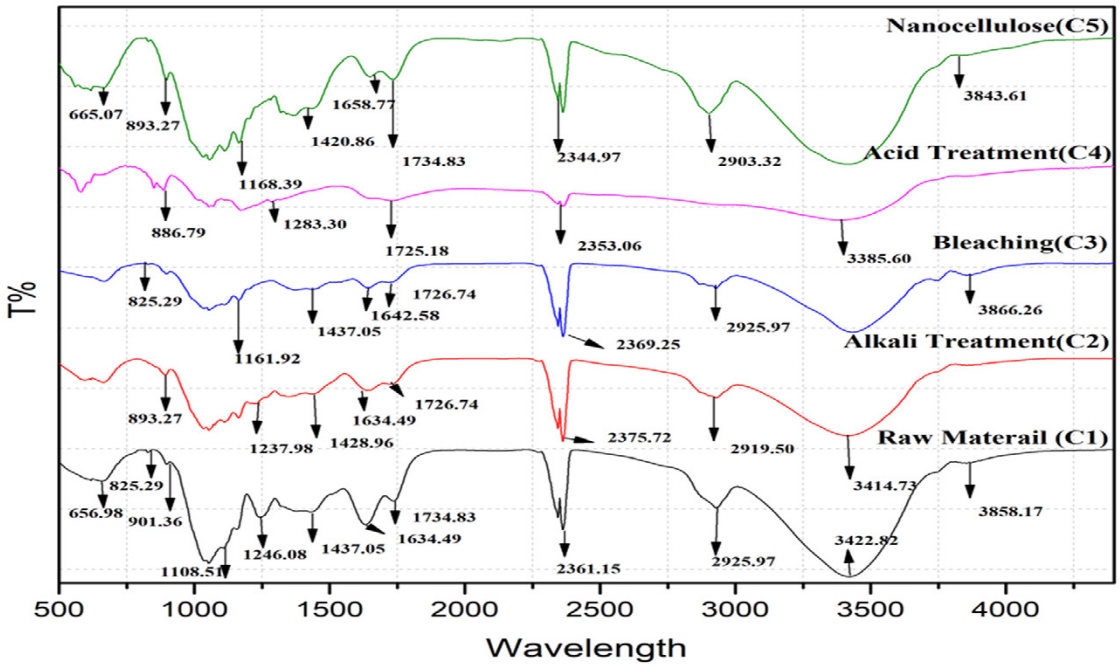

Fig. 4. FTIR analysis of processed samples

common peaks were obtained at different wavelengths as shown in Table 2. The wavelength of 1436.53cm^{-1} indicates the presence of cellulose (shown in Table 2 and Fig. 4) which shows approximately the value recorded in the extraction process of cotton straw nanocellulose analysis from 500 to 4400 cm^{-1} wavelength.

X-ray Diffraction

(XRD) is a fast methodical procedure used to generate a diffraction pattern whether the material is crystalline or amorphous. The lower peak defines that the crystal is arranged in random order and the higher peak shows the desired crystal orientation. To characterize the crystallinity, the sample was scanned at 0°to 80°with 2 theta angle. Cotton straw recorded the high-intensity diffraction peak approx.at diffraction angle 2θ=22° and scattered at amorphous part of sample approx at 2θ=18°. Similar results of diffraction peak 2θ=20,22, 15.1˚ (110), 16.9˚ (110), and 23.0˚ (200) are recorded in nanocellulose which was

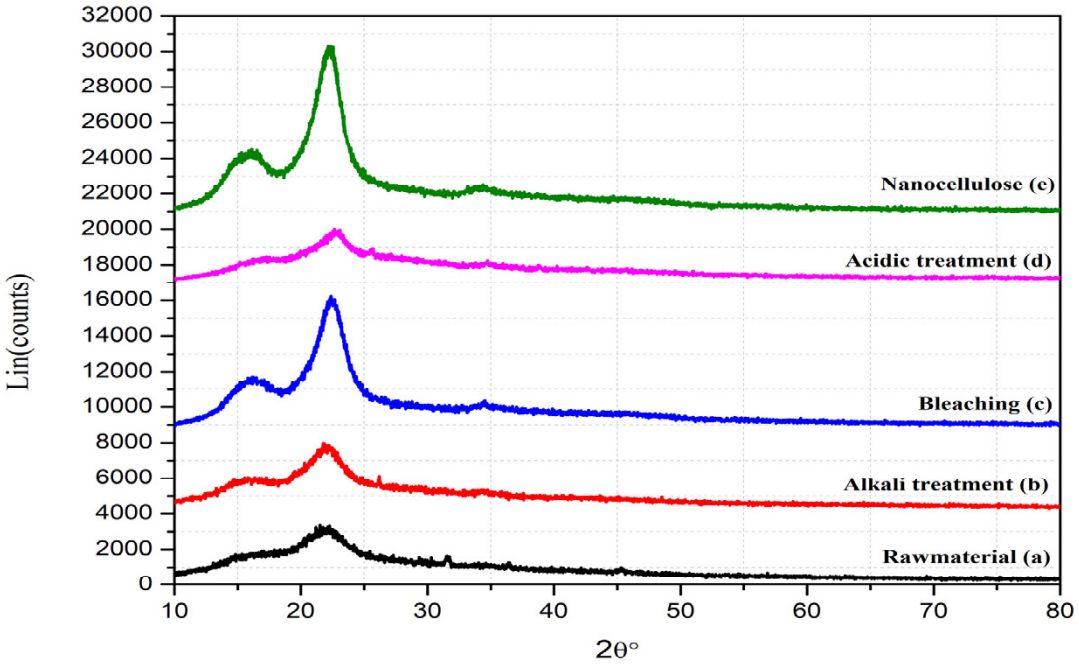

Fig. 5. The XRD pattern of a processed sample

Table 3. CrI value of cotton straw material

Sample	CrI(%)
Raw material	43%
Alkali treatment	26%
Bleaching fibers	31%
Acid hydrolysis	6%
Nanocellulose	23%

extracted from cotton cellulose with high-pressure homogenization[39-40]. In XRD data, raw material and alkali treatment material are amorphous which indicates the reflection or diffraction is not a particular place. Bleached fiber shows crystallinity at high-intensity peaks which resulted in the material being arranged in a specific order. Again Crystallinity is disappeared when we treated the material with 40 %(w/v) H_2SO_4.So, nanocellulose has a slight amorphous structure due to the arrangement of particles in random order and this was shown by low intensity and broad peaks while high-intensity peak shows the crystallinity of the material. CrI value was calculated with the equation such as:

$$CrI = \frac{I_{(002)} - I_{(AM)}}{I_{(002)}} \times 100$$

Whereas, I(*Cr*) measure for crystallinity of material and I(am) for the amorphous nature of the material. The results of CrI value and crystallinity structure are shown in (Table 3 and Fig. 5).

Field Emission Scanning Electron Microscopy

FESEM helps to analyze the shape, dimensional structure and recorded the surface morphology of cotton straw residue at 2 different stages Fig. 6(a) raw material and 6(b) nanocellulose extracted by acid hydrolysis with 40% (w/v) H_2SO_4 at 40°C for 1-hour FESEM records even and elongated fiber in the raw material of cotton waste. Fig 6(a) shows the raw material is recorded as a rough surface by using 10kV energy for FESEM analysis and Fig. 6(b) was recorded the surface is smooth and pores are present on the surface. In Tables 4(a) and 4(b) surface morphology was calculated by using Image J software of raw material in the standard form at

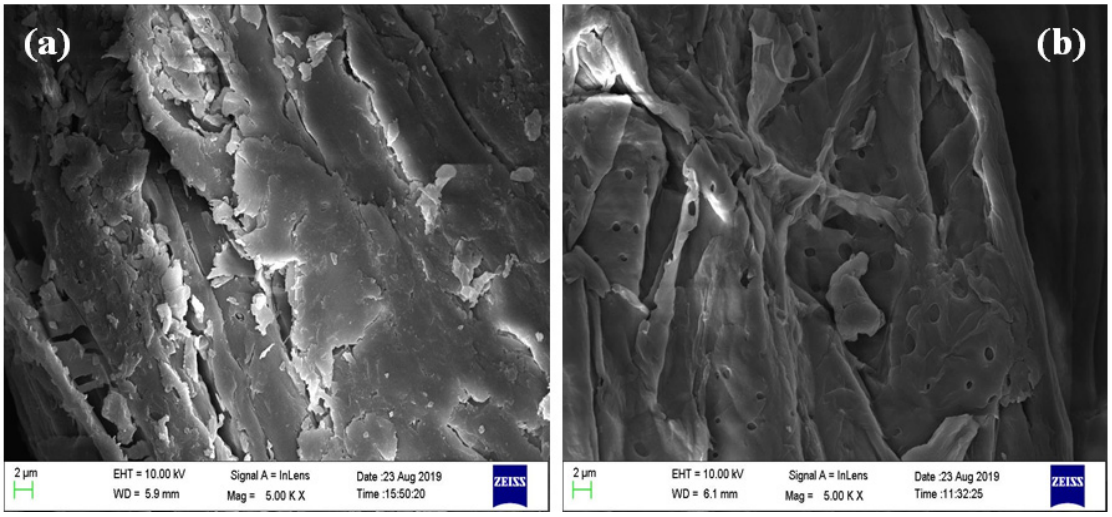

Fig. 6. (a) Raw material, (b) Nanocellulose

Table 4(a). FESEM image analysis of raw material

	Label	Area	Std.Dev.	Perimeter	Angle	Circularity	Integrated Density	Median
1		113846.154	41.834	2000	-3.18	0.358	21677379.03	208.444
2		187692.308	11.988	3330.05	29.982	0.213	13938051.22	70.5
3		166153.846	17.591	2928.901	-127.304	0.243	23221994.16	131.729
4		193846.154	24.123	3417.601	-103.134	0.209	22633087.3	109.364
5		224615.385	42.618	3993.841	0	0.177	30929230.77	133
6		193846.154	6.716	3446.291	-3.691	0.205	11177866	55.871
7		286153.846	11.294	5110.472	27.121	0.138	11776654.04	40.06
8	Mean	195164.835	22.309	3461.022	-25.744	0.22	19336323.22	106.996
9	SD	52694.112	14.664	952.328	63.063	0.069	7288043.893	57.751
10	Min	113846.154	6.716	2000	-127.304	0.138	11177866	40.06
11	Max	286153.846	42.618	5110.472	29.982	0.358	30929230.77	208.444

Table 4(b). FESEM image analysis of nanocellulose

	Label	Area	Std.Dev.	Perimeter	Angle	Circularity	Integrated Density	Median
1		236923.077	110.474	2000	-3.18	0.744	44892307.69	255
2		483076.923	14.491	4320.969	-119.197	0.325	33129230.77	69
3		203076.923	26.17	1775.041	0	0.81	21553846.15	114
4		335384.615	32.111	2892.962	122.471	0.504	28366153.85	101
5		483076.923	22.474	4217.181	-178.493	0.341	28707692.31	55
6		415384.615	19.987	3672.769	-25.017	0.387	37043076.92	90
7		587692.308	20.652	5114.083	-40.601	0.282	51430769.23	79
8	Mean	392087.912	35.194	3427.572	-34.86	0.485	35017582.42	109
9	SD	140462.545	33.643	1250.381	95.608	0.212	10327147.15	67.308
10	Min	203076.923	14.491	1775.041	-178.493	0.282	21553846.15	55
11	Max	587692.308	110.474	5114.083	122.471	0.81	51430769.23	255

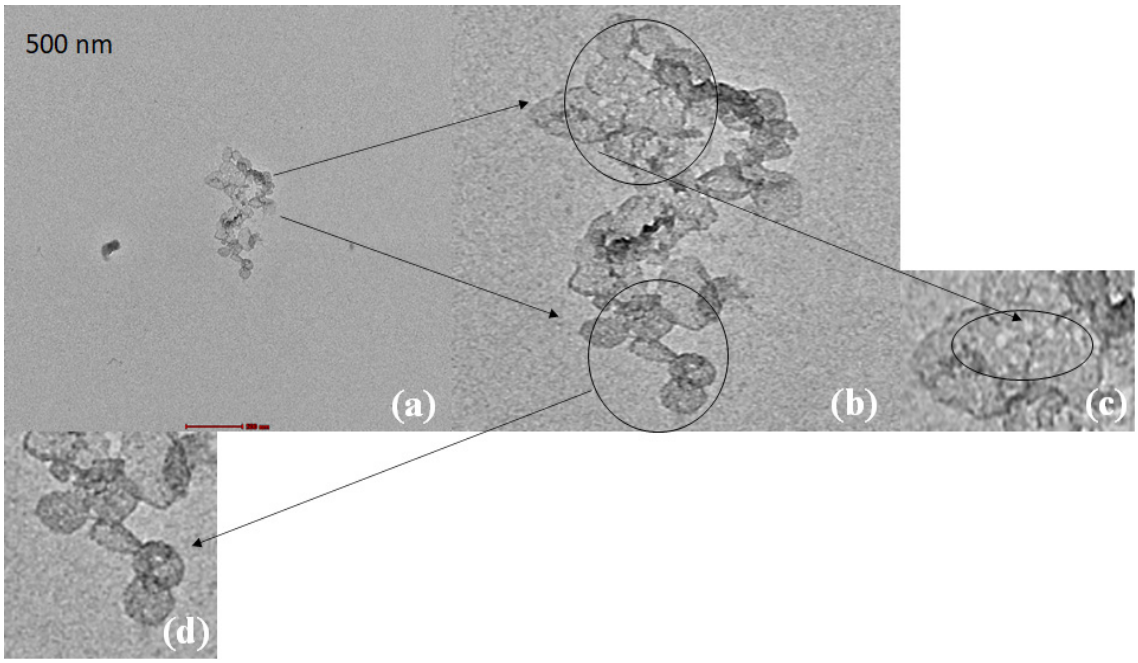

Fig. 7. Transmission Electron Microscopy analysis of the cotton straw nanocellulose.

magnification (5.00 kX) is 2000nm with electron high tension value at 20kV [42]. The mean area of raw material is 195164.835 and its mean integrated density is 19336323.22 (is the multiply of the area and mean gray value). If the Circularity value is 1 that indicates the material is perfectly circular, but here the value was recorded less than 1 which means the particle is in an irregular shape.

Transmission Electron Microscopy

To know the morphology, size, compositional structure, and texture of the material and the sample was magnified at 500 nm.TEM image of cotton straw nanocellulose pores was present on the surface as shown in Fig. 6(b). Crystals were arranged regularly.

TEM micrograph (a) exhibits polycrystalline material because of grain particles arrangement that shows irregular shape structure and small size particles. The particles are oriented in a different direction and arranged in random order. In the image (b) the particle is connected in a random order this is a magnified image to know the structure particle orientation. There are some pores at the surface which we already recognized before in the FESEM image and these pores are found all over the material so, both porous structure and hollow structure are highly visible in the image (c). The structure of particles has irregular shapes with circular rod-like structures of different sizes.

Differential Scanning Calorimetry

It is used to measure the enthalpy variation or the behavior of the material as a function of temperature. The equipment measures the heat flow produced in a sample when it is heated, cooled, or held isothermally at a constant temperature. Nanocellulose was analyzed by using a platinum container and a 3g sample at 0-400°C with a heating rate of 10°C/min in Q-1, TA Instrument held in a helium atmosphere. Cotton straw nanocellulose showed a crystalline structure with enthalpy (411.33J/g) at 168.48°C and melting peak due to endothermic transition (shown in Fig. 8) and the similar result of Rosselle fiber has also been observed at 277.32-289.57°C [43].

Thermogravimetric Analysis

TGA used for the analysis of mean weight loss of the material is recorded as a function of weight and temperature, when the substance is heated at the final stage during the ignition then the weight of the substance is decreased with increasing the temperature. The thermal stability of nanocellulose is lower than lignocellulosic biomass such as

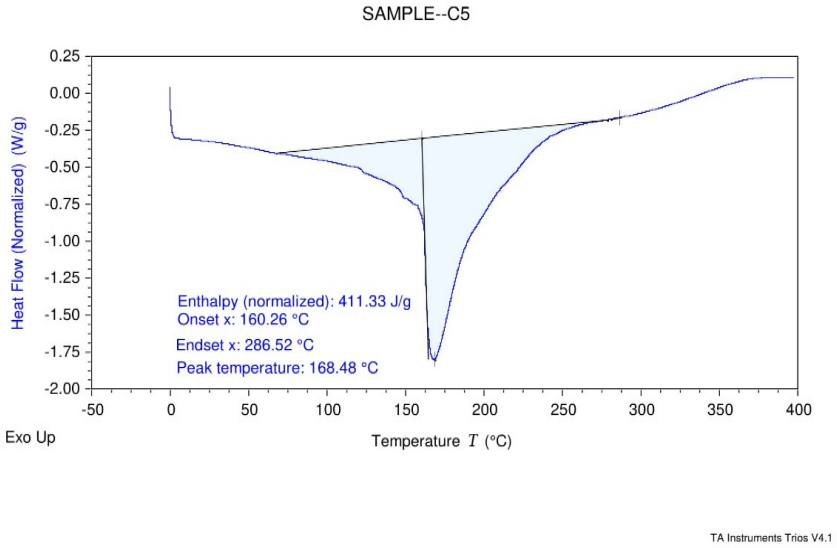

Fig. 8. Differential Scanning Calorimetry of nanocellulose

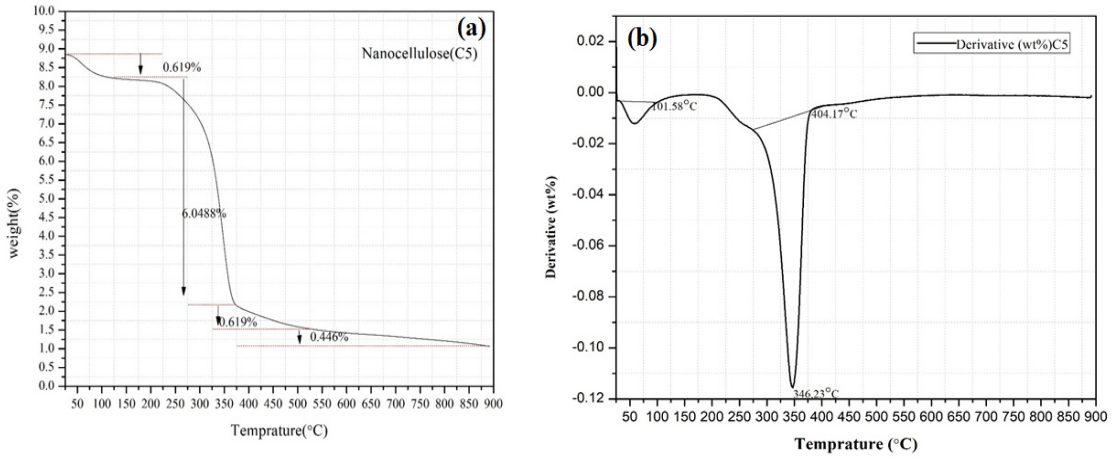

Fig.9. (a) Thermogravimetric analysis (b) Differential Thermogravimetric analysis

lignin, hemicellulose is eliminated after chemical treatment[36]. Four stages of thermal degradation were observed during the experiment (Fig. 9(a) and (b)). The weight loss (0.619%) was noticed at 25-150°C in the initial stage of the experiment. This may be due to the evaporation of moisture content. No degradation was observed between 150-190°C and a similar result has also been observed in lignocellulose biomass [44]. In the second stage during depolymerization, the weight loss (6.0488%) was noticed at the temperature range 200-375°C. At the third stage, samples were degraded from 375- 525°C with a weight loss of (0.619%). Finally, a rapid degradation with weight loss of (0.446%) was noticed after 525°C. In Fig. 9(b) DTG curve was recorded the nanocellulose has major thermal stability at 346.23°C and similar results have also been observed in jackfruit peel [45].

Atomic Force Microscopy

The surface was characterized by atomic force microscopy (AFM) at ambient temperature. Silicon tip with a diameter of 10 nm was used to scan the surface in tapping mode. Cotton straw nanocellulose is uniformly distributed, due to the treatment of 40% acidic solution [46]. The resolution for AFM images was 256× 256 pixels. After imaging, the surface was flattened and analyzed

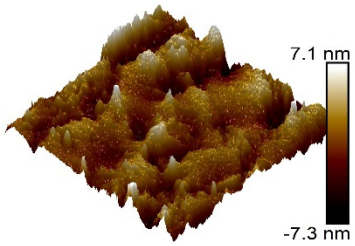

Fig .10. AFM image of nanocellulose

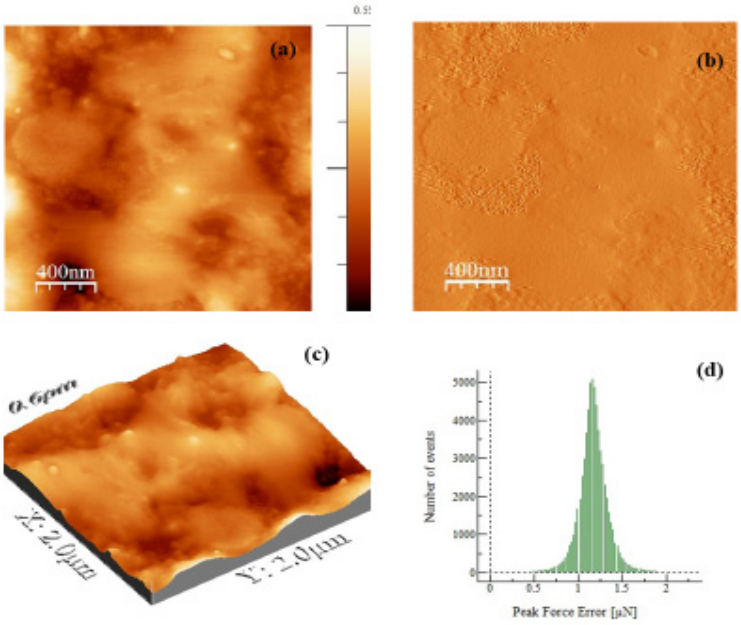

Fig .10. (a) Topographic image, (b) Phase image, (c) Three-dimensional structure,(d) Roughness surface variation

using WSxM software [47] to obtain the real height and phase image. The average height (1.2003 nm) of nanocellulose was calculated from the software. This indicates the ultra-smoothness of the surface. The root means square roughness(0.1738 nm) of the nanocellulose surface was calculated. This small value of roughness confirms the smoothness of the nanocellulose surface [48]. The size (7.1 nm)of nanocellulose was confirmed from AFM shown in Fig. 10. The detail of the topographic image, phase image, three-dimensional structure, and roughness surface variation is shown in Fig. 10(a-d).

CONCLUSION

The following conclusion has been made from the present study

1. Nanocellulose is extracted successfully with chemical method followed by ultrasonication and cryocrushing which helps the material to become nanosized with 23% crystallinity.
2. 1436.53cm^{-1} shows the presence of cellulose content extracted from cotton straw which has smooth surface morphology.
3. Particles of nanocellulose are irregular in shape with a circular rod-like structure.
4. Maximum weight loss of nanocellulose is found at 300-355°C.
5. Due to the removal of lignin, hemicellulose the thermal stability of crystalline material is decreased.
6. The size of nanocellulose was obtained at 7.1 nm.

CONFLICTS OF INTEREST

There are no conflicts to declare.

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