



Chemically Modified Nanocellulose from Rice Husk: Synthesis and Characterisation

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Authors' contributions

Author MK designed, analyzed, interpreted and prepared the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

The synthesis of Cellulose nanocrystals by utilizing rice husk an agricultural waste material deserves to be an advanced approach. The present study evaluates the use of rice husk as an excellent source of cellulose to procure cellulose nanocrystals. A chemo-mechanical method was employed. Chemical processes involved alkali treatment, bleaching treatment and acid hydrolysis. Mechanical processes involved ultrasonication, cryocrushing and magnetic stirring. Cellulose nanocrystals have been characterised by scanning electron microscopy, field emission scanning electron microscopy, atomic force microscopy, X-ray diffraction, and Fourier transformed infrared spectra. The thermal stability of cellulose nanocrystals was investigated using thermogravimetric analysis. The result showed that synthesised cellulose nanocrystals were of the diameter 76.77nm.

Keywords: Cellulose nanocrystals; rice husk; agricultural waste; acid hydrolysis; ultrasonication.

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1. INTRODUCTION

Rice is a spectacularly diverse food crop and rice husk the hard protecting covering amenable for value addition is available in plenty. Rice husk consists of 43.3% cellulose, 32-33% sugars and 22% lignin [1]. Cellulose is an abundant [2] material and foremost component replenished continuously by photosynthesis [3] impart strength and stability to the plant cell wall [4]. Cellulose is present along with hemicelluloses, lignin, pectin and wax. In the natural plant cell wall, crystalline cellulose is embedded with these substances, which make it difficult to obtain pure cellulose [5]. Chemically, cellulose is a linear homopolysaccharide composed of β -D-glucopyranose units linked together by β -1-4-linkages. Each β -1-4 glucopyranose bears three hydroxyl groups where the primary OH group is located in the C-6 position, and the secondary OH group is located at the C-2 and C-3 position. It is demonstrated by the formation of healthy intermolecular and intramolecular hydrogen bonds [6,7] that play a significant role in determining the physical properties of cellulose. Currently, agricultural waste based cellulosic materials which are sustainable, green and environmental friendly [8] have been widely developed for the extraction of nanocellulose [9,10].

Nanocellulose a sustainable and pioneering material has remarkable properties viz. High surface area, high specific strength, modulus, nanoscale dimension, high crystallinity, optical properties and stiffness along with biodegradability, renewability low thermal expansion and low cost [11,12]. The uniqueness of nanocellulose particles depends on the properties of primary raw materials and methods used for their production [13].

On the basis of dimensions, functions and preparation methods nanocellulose can be classified into two categories cellulose nanocrystals [14] and cellulose nanofibres [15] with a diameter 100 nm or less in at least one dimension [16,17]. Nanocellulose is conventionally produced by reducing the crystalline fibres to nanosized particles by oxidation [18], electrospinning [19], refining and high pressure homogenization [20], steam explosion [21,22], cryocrushing [23], microfluidization [24], high intensity ultrasonication [25], enzymatic hydrolysis [26] etc. Among the different methods for preparing nano-cellulose acid hydrolysis is found to be the

most prominent and extensively used method [27-31] which disintegrate the disordered and amorphous segments of the cellulose releasing single and well-defined crystals with high degree of crystallinity [32-34]. In this study acid hydrolysis with sulphuric acid (surface charged sulfate esters were formed thereby promoting the dispersion of nanocrystals) proved to be a suitable method to isolate cellulose nanocrystals.

2. MATERIALS AND METHODS

2.1 Materials

Rice husk (raw material) was received from a local storage area. Sodium hydroxide was used for alkali treatment. Sodium chlorite for bleaching and sulphuric acid was used for acid hydrolysis. All chemicals used were of analytical grade. Distilled water was used throughout the experiments.

2.2 Methods

2.2.1 Extraction of nanocellulose

Nanocellulose was extracted by sequence of chemical treatments such as alkali treatment, bleaching treatment and acid hydrolysis.

2.2.2 Alkali treatment

The chopped, ground and sieved rice husk was subjected to alkali treatment with 4 wt % NaOH at 80°C for 1h. The insoluble rice husk residue was filtered and washed with distilled water. The alkali treatment was given to obtain purified cellulose by removing hemicelluloses and lignin.

2.2.3 Bleaching treatment

Following alkali treatment, the bleaching process was carried out with 5wt% sodium chlorite solution at 80°C for 2 h. The mixture was allowed to cool and was filtered using distilled water. The bleaching process was repeated four to five times.

2.2.4 Acid hydrolysis

The acid hydrolysis was conducted on the fibres at a temperature of 45°C for 1 h. The hydrolysed material was washed by centrifugation at 10,000 rpm at 10°C for 15 min. The centrifugation step was repeated several times until a constant pH in the range of 5-6 was reached. The resulting suspension was then sonicated for 1 h followed by magnetic stirring for three to four hours.

2.2.5 Cryocrushing

After acid hydrolysis the fibres were further cleaved by cryocrushing in which fibers were frozen using liquid nitrogen. When high shear forces were applied ice crystals exerted pressure on the cell walls. The cell walls got rupture and nanocrystals liberated.

2.3 Characterization

2.3.1 X-ray diffraction (XRD)

X-ray diffraction patterns were obtained using an X-ray diffractometer at room temperature with a monochromatic CuK α radiation source in the step-up scan mode with 2θ ranging from 10° to 50° with a step of 0.04 and scanning time of 5.0min. Each material in the form of milled powder was placed on the sample holder and leveled to obtain total and uniform X-ray exposure. The crystallinity index (CrI) was measured by Segal's method [35] with equation as follows:

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

Where I_{002} is the maximum intensity of the diffraction of the (002) lattice diffraction peak and I_{am} is the intensity value for the amorphous cellulose. The diffraction peak is located at a diffraction angle around $2\theta=22^\circ$ and intensity scattered by the amorphous region is measured at diffraction angle around ($2\theta=18^\circ$).

2.3.2 Fourier transform infrared (FTIR) spectroscopy

Fourier transform infrared spectra was recorded using a Perkin-Elmer FTIR spectrophotometer. Untreated, alkali treated, bleached, acid-hydrolysed rice husk fibres were analysed. Samples were finely ground and mixed with potassium bromide, KBr. The mixture was then compressed to pellet form. FTIR spectral analysis was performed within the wave number range of 400-4000 cm^{-1} .

2.3.3 Scanning electron microscopy (SEM)

The surface morphology of rice husk (cryocrushed and acid hydrolysed)fibres was analysed by scanning electron microscopy (SEM) on a JSM-6100 scanning microscope. Acid hydrolysed rice husk fibres were placed on the aluminium stub and incubated in the oven at 60°C . The sample was coated with gold using a

vacuum sputter coater. The thickness of the gold layer was ca. 0.01-0.1 μm . The accelerating voltage was 15 kV.

2.3.4 Atomic force microscopy (AFM)

Surface topography and morphology of the cryocrushed and acid hydrolysed samples were analysed using a NT-MDTs Solver PRO atomic force microscope (AFM). Silicon cantilevers with a typical resonant frequency of 240 kHz and spring constant of 11.8 N/m were used to acquire images in tapping mode at room temperature under ambient conditions. The scanning rate was around 1.5 Hz.

2.3.5 Field emission scanning electron microscopy (FESEM)

The surface morphology of cryocrushed and acid hydrolysed rice husk fibres was examined by field emission scanning electron microscopy (FESEM) with an accelerating voltage of 15-20 KV. Before examination, a fine layer of gold was sprayed on samples by an ion sputter coater with low deposition rate.

2.3.6 Thermogravimetric analysis (TGA)

TGA measurements were carried out using a SDT Q 600 thermogravimetric analyser to determine thermal stability of the different samples. The amount of sample used was ca.1 mg. All measurements were performed under a nitrogen atmosphere with a gas flow of 10 ml min^{-1} by heating the material from room temperature to 900°C at a heating rate of $10^\circ\text{C min}^{-1}$.

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction (XRD)

X-ray studies were performed to investigate the crystallinity of untreated, alkali treated, bleached and cryocrushed acid hydrolysed fibres of rice husk. Different peaks were obtained around $2\theta=16^\circ$, 22° and 35° but two main peaks at $2\theta=16^\circ$ and $2\theta=22^\circ$ corresponds to ordered crystalline arrangements due to formation of inter and intra molecular hydrogen bonding [31,36]. Crystallinity is expressed as the ratio of the diffraction from a crystalline region to the total diffraction of a sample [37]. The crystallinity index was determined for various samples and the results are summarized in Table 1. The progressive increase in crystallinity was mainly

due to the successive degradation and removal of lignin (a cementing material) and hemicellulose from the amorphous region during alkali treatment resulting in realignment of crystalline domains. During hydrolysis, penetration of hydronium ions into the amorphous regions of cellulose allocate hydrolytic cleavage of glycosidic bonds, which ultimately releases individual crystallites [38]. Alongwith this bleaching treatment efficiently removed the remaining amorphous components. Due to increase in the crystallinity of rice husk fibres the stiffness, rigidity and hence strength of fibres increases. From the literature it has been reported that the crystallinity of the sample fibres increases after acid treatment [39,40]. Diffraction pattern for (a) untreated, (b) alkali treated, (c) bleached and (d) acid hydrolysed rice husk has been demonstrated in Fig. 1.

Table 1. Crystallinity index (Crl) of rice husk fibres at different stages of treatments

Sample (rice husk)	Crl(%)
Untreated	30.7
Alkali treated	45.09
Bleached	60.6
Cellulose nanofibres	71.9

3.2 Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectra for (a) untreated (b) alkali treated (c) bleached and (d) acid hydrolysed rice husk has been shown in Fig. 2 (a,b,c,d) where cm^{-1} on X-axis is indicative of wavenumber and % T on Y-axis is transmittance. All the samples exhibit a broad absorption band in the region $3400\text{-}3440\text{ cm}^{-1}$ corresponding to the free O-H stretching vibration of the OH groups in cellulose molecules [36]. The peaks around $1635\text{-}1651\text{ cm}^{-1}$ are attributed to O-H bending of the water adsorption [7]. The peaks at $1155\text{-}1165\text{ cm}^{-1}$ ascribed to C-C ring stretching and C-O-C glycosidic ether linkages respectively [7]. The peaks at 1371 cm^{-1} , 1061 cm^{-1} and 897 cm^{-1} were attributed to the cellulose, the C-O stretching, O-H bending vibration and C-H deformation vibration. The peaks at $1034\text{-}1029\text{ cm}^{-1}$ is attributed to ether linkage (C-O-C) from lignin or hemicelluloses. The intensity of these peaks sharply weakened after acid hydrolysis because of the removal of hemicellulose material [41,42]. The peaks at $892\text{-}897\text{ cm}^{-1}$ in bleached and acid hydrolysed samples are assigned to C-O-C stretching vibration of pyranose ring and the glycosidic linkages between glucose units in cellulose respectively [43].

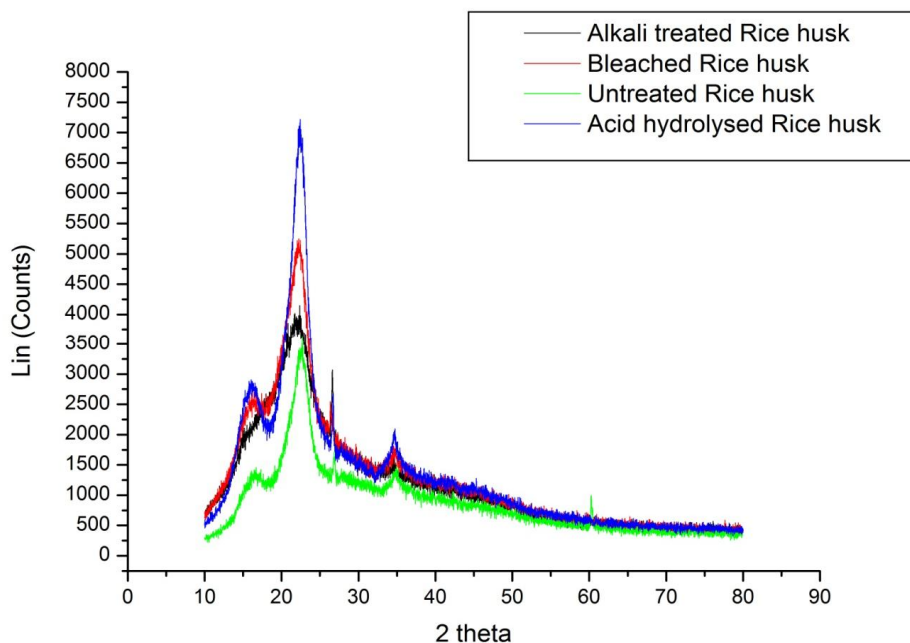


Fig. 1. X-RD pattern of Rice husk (with different treatments)

3.3 Scanning Electron Microscopy (SEM)

Fig. 3 depict the surface morphology of acid hydrolysed rice husk fibres using SEM analysis.

Illustratively it is apparent that acid treated rice husk is micro-sized fiber with 100 μm in size and irregular in shape due to removal of amorphous phase [44].

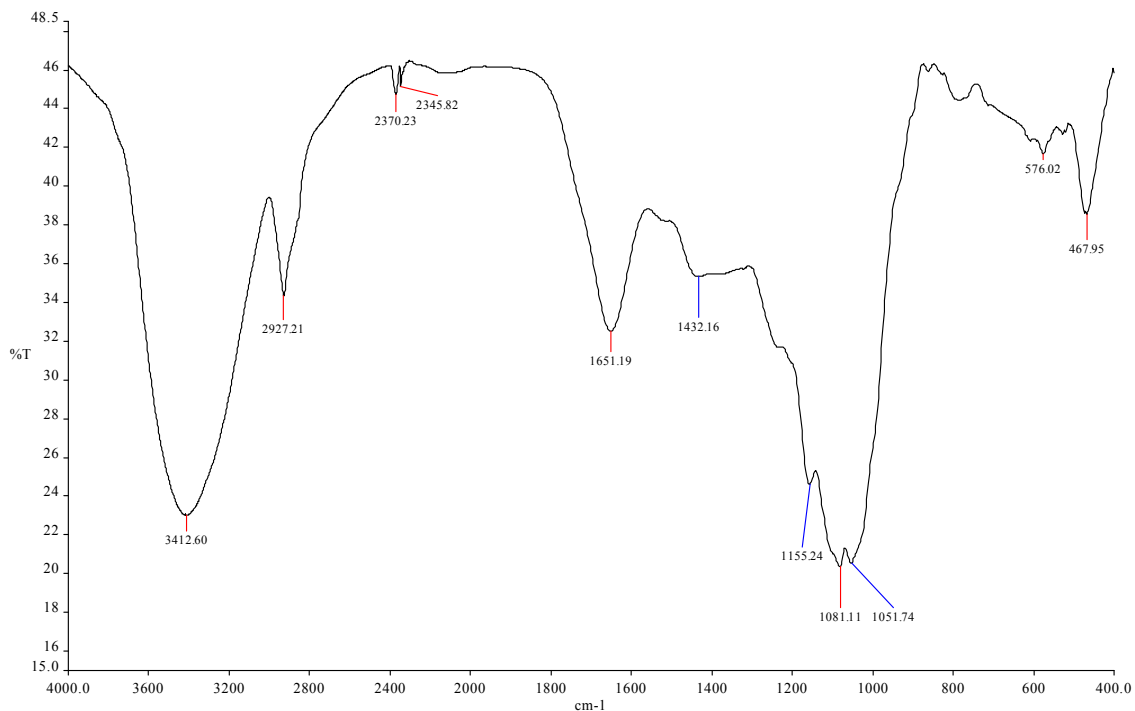


Fig.2 (a)

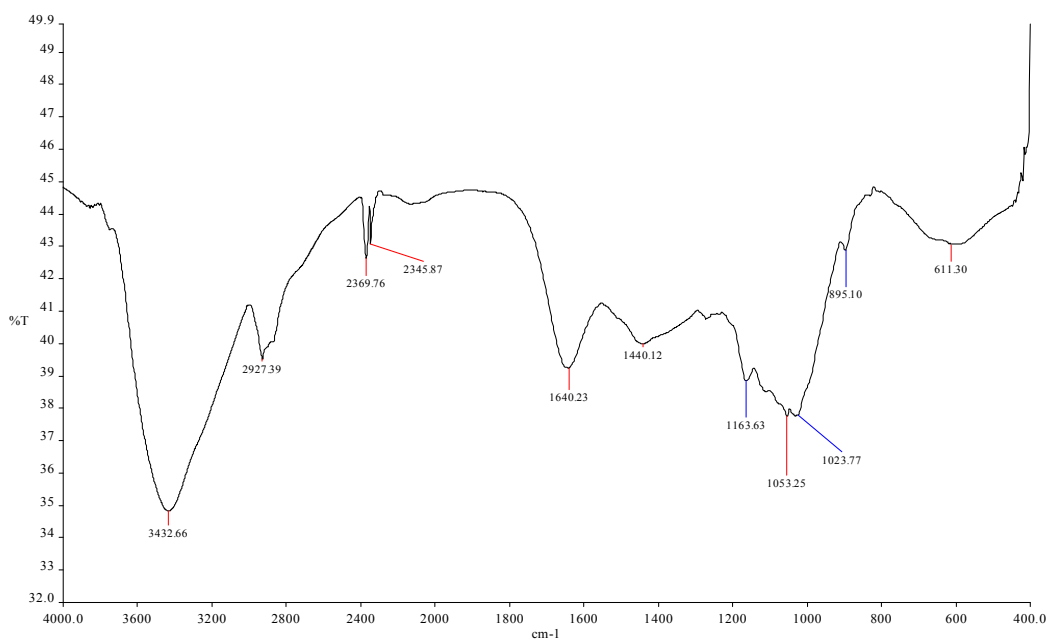


Fig. 2(b)

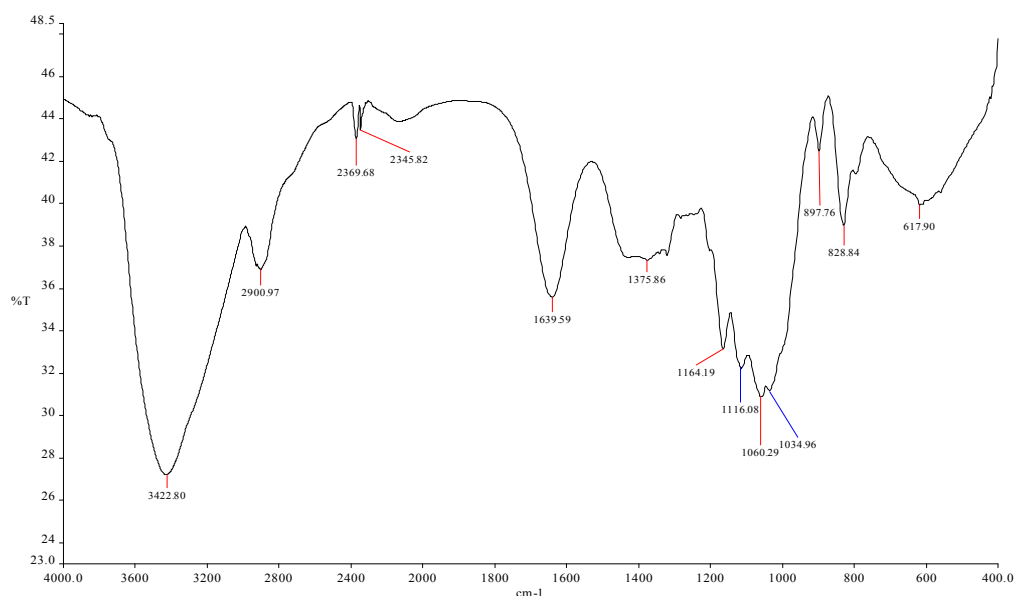


Fig. 2(c)

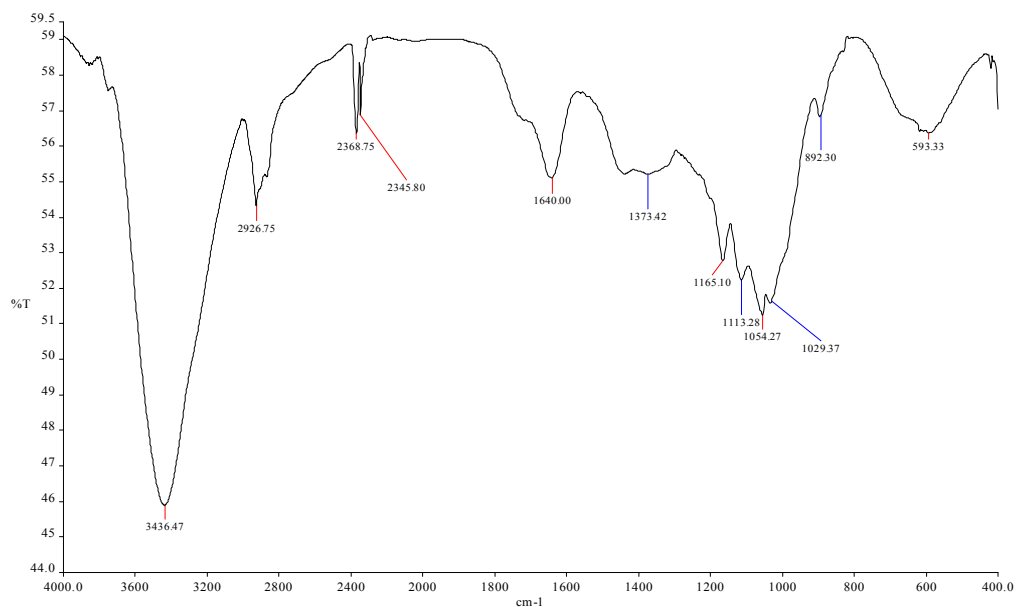


Fig. 2(d)

Fig. 2 (a- untreated,b- alkali treated, c- bleached, d- acid hydrolysed) FTIR Spectrum of Rice husk

3.4 Atomic Force Microscopy (AFM)

The morphological characteristics of nanocellulose have also been studied using Atomic Force Microscope (AFM). Image in Fig. 4 shows a three-dimensional view of the surface

structure of nanocellulose crystals having smooth surface and small particles size distribution as 76.77 nm under 60 min sonication. It is due to the removal of pectic polysaccharides which make clearly defined cellulose nanocrystals [45].

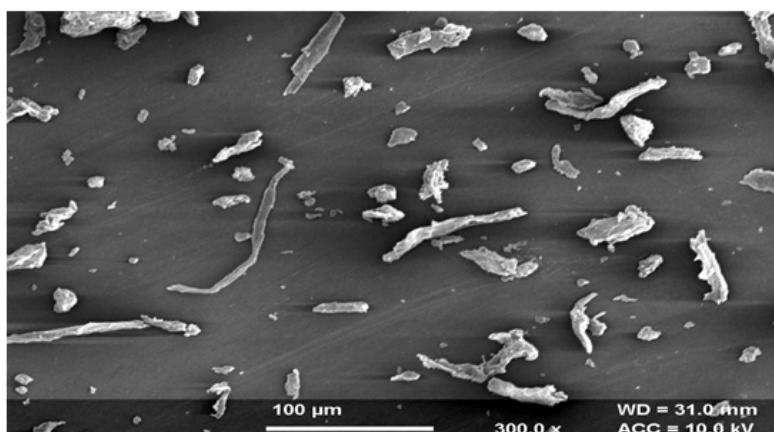


Fig. 3. SEM of cellulose nanocrystals of rice husk

3.5 Field Emission Scanning Electron Microscopy (FESEM)

FESEM micrographs in Fig. 5 confirms that acid hydrolysis alongwith magnetic stirring and cryocrushing has efficiently broken the inter fibrillar linkages between the rice husk fibers and subjected them to much smaller size.

3.6 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was computed to examine the degradation characteristics of rice husk fibres at different stages of treatment (untreated, alkali treated, bleached and acid hydrolysed). Due to variance in chemical structures among cellulose, hemicellulose and lignin they usually degrade at different temperatures. The thermal stability of

the material depends on the sample inbuilt characteristics as well as on the molecular interactions between different macromolecules. All the TGA curves show that an initial small weight loss occurred in the range of 35-150°C due to evaporation of moisture from these materials. The first degradation step corresponds to cellulose degradation processes such as dehydration, depolymerisation and decomposition of glycosyl units [44]. An incisive weight drop is observed at higher temperatures. In untreated and alkali treated fibres hemicelluloses, lignin and pectin alongwith cellulose remain present. These components have a lower decomposition temperature compared to cellulose [45] and their progressive removal after bleaching treatment improves the thermal stability of the rice husk.

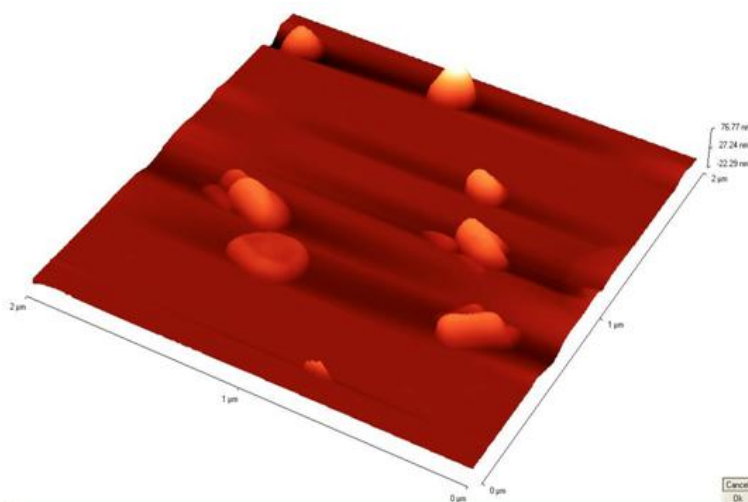


Fig. 4. AFM of cellulose nanocrystals of Rice husk

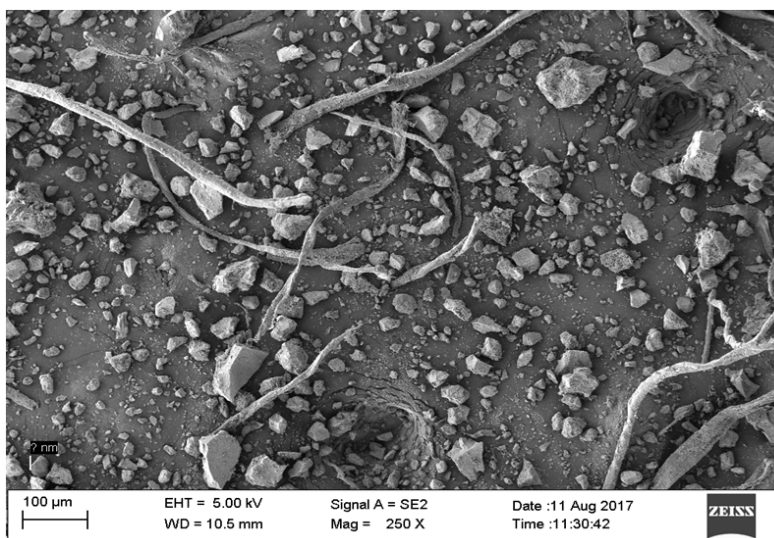


Fig. 5. FE-SEM of cellulose nanocrystals from rice husk

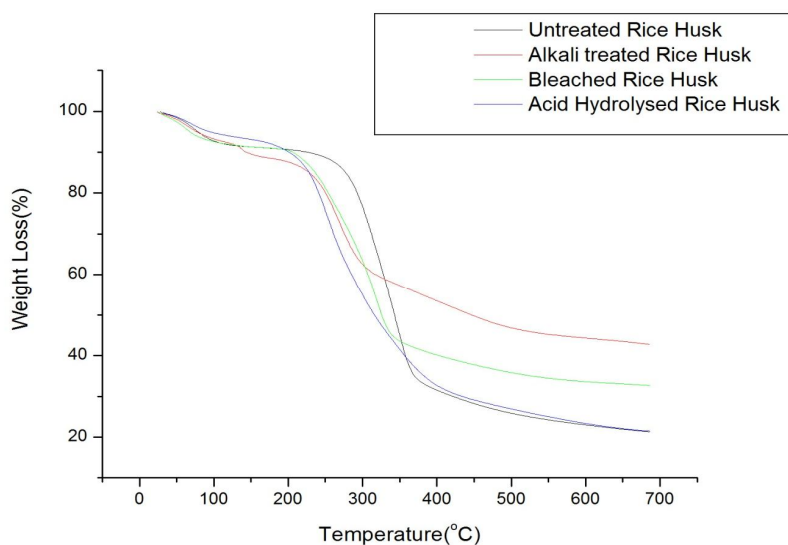


Fig. 6. TGA of Rice husk (with different treatments)

Fig. 6 indicates that the first degradation corresponding to evaporation of chemisorbed water occurred at a temperature close to 70°C. The second degradation occurred around 220-310°C, due to the thermal decomposition of hemicellulose and some of the lignin. Major decomposition occurred around 400-700°C where amount of residues in raw husk fibres were high and in the range of 30-40%. Other researchers have also investigated similar thermogram patterns indicating higher residue amount of rice husk fibres [46]. The weight fraction of fibres existing after heating above

400°C is due to the presence of carbon content [47].

4. CONCLUSIONS

The successful isolation of cellulose nanocrystals from rice husk provides an opportunity for efficient utilization of such a material which otherwise has become a threat to environment nowadays due to its burning, particularly in northern India. Chemical treatments efficiently eliminated unwanted noncellulosic materials and mechanical treatments assisted in

individualisation of cellulose fibres to nano dimension. Morphological investigation, infrared spectroscopy and X-ray diffraction analyses confirmed the removal of non-cellulosic materials. This study proves that chemo-mechanical approach is ingenious for the preparation of cellulose nanocrystals.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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