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Synthesis and characterization of cellulose nanocrystals/PVA based bionanocomposite

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ABSTRACT

Sugarcane bagasse based cellulose nanocrystals (CNCs) and poly (vinyl alcohol) (PVA) were used to fabricate biodegradable porous foam bionanocomposites for application in biomedical applications by thermally- induced phase separation (TIPS) (freeze drying) technique. Aqueous suspension of cellulose nanocrystals and poly (vinyl alcohol) (PVA) solution in water were mixed to prepare (20/80) (v/v) composition of these polymers. For the comparative study 3D porous PVA foam was also prepared. Samples were characterized by FE-SEM, FTIR, X-ray diffraction analysis, porosity and pore size by ImageJ software. FE-SEM showed the surface morphology of pores distribution of the foam samples. The porosity of 3D porous PVA foam and 3D porous CNCs/PVA bionanocomposite was calculated as 90.8% and 89.0% respectively. The functional groups and crystallinity of the samples were determined by FTIR and X-ray diffraction spectra, respectively. The diameter of CNCs was in the range from 29 nm to 65 nm and the average diameter was calculated as 43 ± 3 nm by image analysis using ImageJ software. Thus produced porous composite in the present study might find potential applications to be used in biomedical applications. Copyright © 2013 VBRI press.

Keywords: Sugarcane bagasse; cellulose nanocrystals; PVA; bionanocomposite; freeze drying.





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Introduction

Nanotechnology has rapidly become an multidisciplinary field and is one of the growing research areas in synthesis of cellulose-based nanoparticles (CNPs), or nanowhiskers (CNWs), or Nanocrystals (CNCs) from bioresources, especially from annual plants and residues of agricultural crops such as corn grain, sugarcane bagasse, corn cobs, wheat stems, and seed coats using top-down technologies [1].

Sugarcane bagasse is an abundant agro-industrial by product worldwide and is used in many different applications [2-4]. Sugarcane bagasse consists of cellulose 43.8%, hemicellulose 28.6%, lignin 23.5%, ash 1.3%, and other components 2.8% [5, 6]. Chemically purified cellulose from sugarcane bagasse, is a homopolysaccharide composed of β -1-4 glucopyranose unit, shows semi-crystalline behavior, is insoluble in water and common organic solvents but extensively used as cellulose suspension solution in water. The properties of cellulose including low density, good mechanical properties, biodegradability, and availability from renewable resources have become increasingly important and have contributed to a rising interest in this material [7]. Cellulose nanocrystals are expected to show high stiffness because the Young's modulus of the cellulose crystal is as high as 134 GPa [8].

Polyvinyl alcohol (PVA) is well known biologically friendly synthetic polymer with properties such as water soluble, semicrystalline, fully biodegradable, non-toxic, and biocompatible, high optical clarity, and therefore, it finds use in a broad spectrum of biomedical applications [9-12]. The hydroxyl groups on partially hydrolyzed polyvinyl alcohol (PVA) are expected to interact with the hydrophilic surfaces of the cellulose nanocrystals leading to strong hydrogen bonding and this is the point of processing parameters [13].

In this work, our aim is to increase the potential of sugarcane bagasse cellulose as biomaterial with biocompatibility and high mechanical properties and synthetic polymer (PVA) for designing hybrid polymer bionanocomposite by thermally-induced phase separation (freeze drying) process and evaluate the material aspects of CNCs/PVA composites for biomedical application including morphology, structure, and crystallinity. Therefore, in this work, the preparation and characterization by FE-SEM, FTIR, X-ray diffraction of porous bionanocomposite of cellulose nanocrystals (CNCs) and poly (vinyl alcohol) blend are described.

Experimental

Materials

Sugarcane Bagasse was provided by a local sugar factory (Saharanpur, Uttar Pradesh, India). After drying in sunlight, it was ground and sieved under 30 mesh screens. Bagasse was dried in oven at 105°C for 3 h and stored at room

Chemical purification of cellulose

Chemical purification of sugarcane bagasse was performed according to the methods of [14-16]. Sugarcane bagasse (5.0 g) was first dewaxed in a Soxhlet apparatus with a 2:1 (v/v) mixture of benzene and methanol for 5-6 h. Lignin in the bagasse was then removed by using acidified sodium chlorite solution at 75°C for 1 h while repeating the process 3-4 times until the product became white. After this delignification process, product was treated in 2 wt% potassium hydroxide at 85-90°C for 2 h to remove hemicelluloses with residual starch, and pectin. For highly chemically purified cellulose, product was further treated with acidified sodium chlorite solution at 75°C for 1 h and then treated with 5 wt% potassium hydroxide at 85-90°C for 2 h. After the whole chemical treatments, the product was filtered and rinsed with distilled water until filtrate pH was neutral. Now, product was dried in oven at 105°C for 6 h and stored in air tight polybags.

Isolation of cellulose nanocrystals

Aqueous suspension of cellulose nanocrystals was prepared by hydrolysis of chemically purified cellulose by 60% (w/w) sulfuric acid for 5 h at 50°C under strong agitation. The hydrolysis was quenched by adding 10-fold excess water to the reaction mixture. The resulting mixture was cooled to room temperature and dialyzed against deionized water until neutrality. The suspension was then sonicated for 10 min in an ice bath to avoid overheating. Thus generated suspension was stored in refrigerator at 4°C [17].

Preparation of the porous bionanocomposites

3D CNCs/PVA composite was prepared by the following procedure. Typically, 1 wt% aqueous suspension of cellulose nanocrystals was added to an 8 wt% PVA solution with composition (20/80) (v/v) ratio. After adding this aqueous suspension of CNCs, the solution was stirred for 5 h followed by 15 min of sonication in order to eliminate the bubbles from the solution. Following this process, composite solution was frozen at -40°C for 5 h. The solvent was then extracted by a freeze drying using Lyodel (Delvac Pumps Pvt. Ltd, India) for 3 days. After freeze drying composite was stored in desiccators until further characterization was done.

Measurements and characterization

Surface morphology of the samples was studied with the help of field emission scanning electron microscopy (FE-SEM). SEM studies were made on FEI Quanta 200 F microscope. The sample were mounted on Au coated stubs and observed at 10-15 kV.

The porosity of freeze dried porous composite was evaluated by using a gravimetric measurement. The porosity (%) was calculated by the following formula.

Porosity (%) = $[1-(d_{app}/d_p)] \ge 100$

where, d_{app} is the apparent density of porous composite and d_p is the density of non- porous composites.

Chemical structure of CNC/PVA blend porous composite was determined by FTIR spectroscopy using a Nicolet spectrophotometer. Samples were freeze dried at -40°C for 2-3 days, mixed with KBr in a ratio of 1:200 (w/w) and pressed under vaccum to form pellets. FTIR of the samples were recorded in transmittance mode. The physical structure of the samples were evaluated by X-ray diffraction (XRD) pattern using a Bruker AXS D8 Advance diffractometer with a scanning rate of 5°C per min with CuK α radiation source ($\gamma = 1.54060$ Å) operating at 40kV and 30 mA. The patterns were obtained within a 5-90° at 20 scale.

Results and discussion

Physical appearance

Fig. 1 (A, B, & C) shows the physical appearances of sugarcane bagasse agro-waste as raw material, chemically purified cellulose, and chemically purified cellulose in distilled water. As it shows, the chemically purified cellulose is white in color while sugarcane bagasse is grayish and is insoluble in water and only soluble in selected organic solvents and combinations of solvents which limit its application ability. However, its use as reinforcement opens the application range in potential applications particularly in biomedical area.



Fig. 1. Physical appearance of (a) sugarcane bagasse (b) chemically purified cellulose (c) chemically purified cellulose in water.



Fig. 2. (A) Physical appearance of CNCs suspension, (B) SEM image of agglomerated CNCs.

Field emission scanning electron microscopy (FE-SEM)

SEM results in **Fig. 2** (B) presented an agglomerated CNCs at 50000x magnification (scale bar = 1.0 μ m) and the diameter of cellulose nanocrystals was in the range from 29 nm to 65 nm and the average diameter was calculated as 43± 3 nm by image analysis using ImageJ software. The field emission scanning electron microscopy images showed porous morphology of the fabricated 3D PVA porous structure as surface view (magnification-100x) showing surface morphology which showed highly porous structure and opening of the pore capillaries and cross section (magnification-5x) showing the formation of 'channel-like' fine structures of the pores as shown in **Fig. 3** (B).



Fig. 3. SEM images of PVA (8 wt %) porous structure (magnification – 100x), (A) Surface view, (B, C) Cross-section view at different magnifications (50x and 1000x).



Fig. 4. SEM images of CNCs/PVA (20/80) (v/v) bionanocomposite (surface view) (A) Magnification = 100x, (B) Magnification = 500x.



Fig. 5. SEM images of CNCs/PVA (20/80) (v/v) bionanocomposite (cross-section view) (A) Magnification = 100x, (B) Magnification = 500x, (C) Magnification = 1000x.

At higher magnification (as shown in **Fig. 3**(**C**)) some capillary type pores through 'channel-like' pores, which are likely to be used for diffusion of fluid, were observed. The aspects of surface morphology of CNCs/PVA bionanocomposite at two different magnifications (100x, 500x) presented etched surface of pores because of the interaction of hydroxyl groups of PVA polymer matrix with hydrophilic surface of cellulose nanocrystals (as shown in **Fig. 4** (A & B)). As shown in **Fig. 5** (A, B, & C), SEM results of cross-section of CNCs/PVA porous bionanocomposite showed 'channel-like' pattern with composite walls of CNCs and PVA blend which are supposed to give higher strength than that of PVA as a homopolymer.

Porosity

The porosities of freeze dried PVA foam and CNCs/PVA bionanocomposites were evaluated by using a gravimetric measurement. In brief, the freeze dried structures were cut into square with side by 2 cm. After measuring the thickness, the volume of the scaffolds could be determined. The mass of the scaffolds was also measured for determination of the apparent density of the porous structures (d_{app}) and density (d_p) of non-porous structures with average value of three porous structure for particular sample. The porosity (%) of PVA foam and CNCs/PVA bionanocomposite were calculated as 90.8 (%) and 89.0 (%) respectively. These values are shown in **Table 1**.

Table 1. Crystallinity and porosity percentage of the samples.

Sample	Crystallinity (%)	Porosity (%)
Cellulose nanocrystals (CNCs)	68.3	
PVA foam structure	49.2	90.8
CNCs/PVA composite	59.0	89.0

FTIR spectroscopy

FTIR spectra of sugarcane bagasse (SCB) as raw material and chemically purified cellulose (CPC) from sugarcane bagasse showed the major differences in relation to the functional groups at the region from 2000 to 400 cm⁻¹ as shown in **Fig. 6**.



Fig. 6. FTIR spectra of sugarcane bagasse, chemically purified cellulose and cellulose nanocrystals.

The main absorption bands in spectra of sugarcane bagasse corresponding to different functional groups and linkages are 1730 cm⁻¹ for unconjugated stretching of acetyl or carboxylic acid (xylans), 1634 cm⁻¹ for C=O stretching with aromatic ring, 1620-1595 cm⁻¹ and 1512 cm⁻¹ for C=C stretching for aromatic ring (lignin), 1429 cm⁻¹ for CH₂ bending, 1376 cm⁻¹ for C-H deformation, 1335 cm⁻¹ for O-H in plane bending (cellulose), 1250 cm⁻¹ for C-O

stretching of ether linkage, 1166 cm⁻¹ for C-O-C asymmetric bridge stretching (cellulose), 1062 cm⁻¹ for C-O symmetric stretching of primary alcohol, 904 cm⁻¹ for β -Glucosidic linkages between the sugar units, 670 cm⁻¹ for C-OH out-of-plane bending (cellulose). But after the chemical treatment for removing the lignin, the absorption bands at 1730, 1620, 1595 and 1512 cm⁻¹ are corresponding to the functional groups present in the lignin, not observed on the spectrum of CPC and two other absorption bands which must be kept in mind and emphasized, are at 1512 and 1250 cm⁻¹. The band at 1512 cm⁻¹ is not present while the band at 1250 cm⁻¹ is drastically reduced on the CPC FTIR spectrum which indicates that the most of the lignin was removed [18, 19]. FTIR spectra of CNCs showed the similar pattern as that of CPC but having strong and sharp band intensity of the functional groups with high crystallinity as compared to CPC.

Fig. 7 shows the FTIR spectra of CNCs, PVA (PVA-85-89), and CNCs/PVA bionanocomposite. For PVA (PVA-85-89), the large band observed between 3600 and 3200 cm⁻¹ are linked to the stretching O-H from the intramolecular and intermolecular hydrogen bonds, the vibrational band observed between 2840 and 3000 cm⁻¹ refers to the stretching C-H from alkyl groups and the peaks between 1750-1620 cm⁻¹ are due to the stretching C=O and C-O from acetate group remaining from polyvinyl acetate, peaks for C-O-C at 1150-1085 cm⁻¹ and for C-O at 1141 cm⁻¹ (crystallinity), bending vibration related to CH₂ groups at 1461-1417 cm⁻¹ [**20**].



Fig. 7. FTIR spectra of cellulose nanocrystals (CNC), neat polyvinyl alcohol (PVA), and cellulose nanocrystals/polyvinyl alcohol (CNC/PVA) porous bionanocomposites.

In FTIR spectra of CNCs/PVA porous bionanocomposite, mixed major intense peaks of PVA (PVA-85-89) and CNCs (as discussed above) were observed, which confirms the physical blending of both polymers. On composite formation, the peak intensity increases. This may be due to low intermolecular hydrogen bonding due to aggregation of cellulose nanocrystals into PVA matrix and hence, the homogenous interfacial bonding was decreased.

X-ray diffraction analysis

The XRD results of CNCs, PVA, and CNCs/PVA porous composite was shown in **Fig. 8**.



Fig. 8. X-ray diffraction pattern of cellulose nanocrystals, polyvinyl alcohol, and CNCs/PVA porous composite.

XRD profile of CNCs/PVA exhibited the characteristic peaks of both CNCs and PVA, basically the diffractogram of CNCs showed peaks around $2\theta = 16.5$ 22.5, and 33 degree assigned to plane (110), (200), and (004) planes, which are believed to represent the typical cellulose-I structure [**16**, **21**, **22**, **23**, **24**]. The diffractogram of PVA showed three main peaks characteristic of crystallinity of polymer at 19.4° (strong), 22.5° (medium), and 40.3° (weak) [**25**]. The CNCs/PVA porous composite exhibited a crystalline structure with a peak around $2\theta=20^\circ$, which contribute to crystalline segment of PVA and cellulose and other composite peaks are weaker than that of individual peaks of polymers [**26**].

Crystallinity percentage of CNCs, PVA, and CNCS/PVA composite was calculated according to amorphous subtraction method [27] and were observed as 68.3%, 49.2%, and 59.0%, respectively. High crystallinity percentage of CNCs showed the indication of effective hydrolysis which produces crystalline cellulose with removal of amorphous cellulose of chemically purified cellulose. Crystallinity percentage of CNCs/PVA composite decreased because of CNCs strongly interacted with the hydroxyl groups of PVA and the twisting together into a confused mass between them caused steric effect and destroyed the highly ordered arrangement of PVA, and in turn showed the decrease of its degree of crystallinity. However, intensity of CNCs-related peaks decreased because of lower content of CNCs in composition of the composite. The crystallinity percentage of the samples is summarized in Table 1.

Conclusion

The main aim of the above study was to produce a 3D porous PVA foam and 3D CNCs/PVA porous bionanocomposite by adding sugarcane bagasse based CNCs into PVA polymer matrix using thermally-induced process. phase separation (freeze drying) The characterization of PVA foam and CNCs/PVA porous composite was done by FE-SEM, FTIR, and XRD. FE-SEM showed the surface morphology of pores distribution of the porous samples. FTIR demonstrate the structural changes and intermolecular interaction of the components. The porous 3D PVA foam and 3D porous CNCs/PVA bionanocomposite showed high porosity such as 90.8% and 89.0% respectively. The crystallinity percentage of CNCs, PVA, and CNCs/PVA porous bionanocomposite were

calculated as 68.3%, 49.2%, and 59.0% respectively. The porous composite obtained in present study has a premise to be used in biomedical applications.

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