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RESEARCH ARTICLE

Extraction andCharacterization of Cellulose from Agricultural Residues: Wheat Straw and Sugarcane Bagasse

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Abstract

Wheat straw (SW) and Sugarcane bagasse (SCB) are agricultural as well as industrial wastes rich in lignocellulosic components that can be extracted easily and used as renewable source of energy. The main aim of this present work was to explore the alternative source of cellulose extraction using a simple, fast and ecofriendly conditions. The process involves NaOH degradation, acid hydrolysis and bleaching using hydrogen peroxide as bleaching agent. The extracted compound was analyzed by X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) techniques. The XRD peaks obtained were specific to cellulose I_β which is crystalline allomorph with monoclinic structure. The crystallite size of cellulose obtained from sugarcane bagasse was 10.51 nm which is larger than the size of cellulose obtained from wheat straw i.e 4.04 nm. Cellulose from sugarcane bagasse showed crystallinity index of 51.84 % whereas from wheat straw only 17.94 % was observed. The yield was slightly higher in wheat straw than sugarcane bagasse. FTIR analysis ofcellulose extracted fromsugarcane bagasse showed characteristic peak at 3255.84 cm^{-1} which is shifted to 3340 cm^{-1} in case of cellulose obtained from wheat straw. This peak is due to the vibration of –OH group in the cellulose extracted from both of the materials. However, cellulose obtained from bothmaterials showed the vibration of C-O-C bond at 1033 cm^{-1}.

Keywords: Cellulose, Wheat straw, Sugarcane bagasse, XRD, FTIR.

Introduction

Cellulose is the world's most abundant and important renewable natural organic polymer produced on the surface of Earth. In 1839, the French chemist AnselmePayen first used the term 'cellulose' for a white powder which he had isolated from plant matter. Later in 1920, German organic chemist Hermann Staudinger proposed the chemical structure of cellulose [1]. Cellulose is one of the major component of all plant materials including wheat straws and sugarcane bagasse [2].

Chemically, cellulose is a polymer consisting of a linear chain of anhydroglucose monomer units connected by glucosidic linkages at C-1 and C-4 positions. The presence of three – OH groups of varying acidity or reactivity confirms the structure of cellulose. Among these three –OH groups two are at secondary position on C-2 and C-3 while the third one is at primary position on C-6. These groups aids in the development of strong intermolecular and intramolecular hydrogen bonds because of which cellulose forms very tightly packed crystallites [3]. The molecular orientation and hydrogen bonding network in crystalline domains can differ greatly, generating four cellulose polymorphs, namely, cellulose I, cellulose II, cellulose III, and cellulose IV. The polymorph of cellulose I (cellulose I_α and cellulose I_β) has been predicted to exist in native cellulose. Cellulose I_α has a triclinic unit cell whereas cellulose I_β has a monoclinic unit cell. Both these chains of cellulose I_α and cellulose I_β possess parallel configurations [4].

Lignocellulosic materials derived from agricultural residues such as wheat straw, sugarcane bagasse, rice straw, and other plant parts provide an abundant source of renewable energy for bioconversion as well as raw materials for a variety of industries including paper and pulp, food, pharmaceuticals, and biofuels [5]. Sugarcane Bagasse (SCB) is a waste product produced in enormous quantities in the sugar and alcohol industries after the complete removal of sugarcane juice. It is primarily utilized as a fuel to power the sugar mill. However, the residual bagasse is still a threat to the environment [6]. Sugarcane bagasse (SCB) is generally composed of about 45% cellulose, 26% hemicelluloses and 24% lignin with other minor nonstructural components such as ash and extractives [7]. Similarly, wheat straw is the residual plant part obtained after the complete removal of wheat grain. The average yield of straw is 1.3 to 1.4 kg per kg of wheat grain, resulting in a significant amount of excess straw [5]. Wheat straw consists of about 33%-40% cellulose, 20-25% hemicellulose and 15-20% lignin [8]. Using agricultural leftovers in biomaterials now has potential to release the possibilities of these underutilized recyclable sources while also providing a non-food market for the agricultural industry [9].

The main aim of the present work was to explore the alternative source of cellulose from above mentioned agricultural residues using a simple, fast and ecofriendly extraction conditions for both of the material and characterize and compare their physicochemical properties using Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD).

Materials and methods

Materials

The local and cheap materials were used for this project. Sugarcane bagasse was collected from Dudhpati, Bhaktapur, Nepal and the wheat straw was collected from Chhapkaiya, Birgunj, Nepal. Sugarcane bagasse was collected from a local vendor after complete juice removalwhile wheat straw was collected from a local farmer. Both of these samples were dried in sunlight for a month and cut into small pieces of about an inch after complete dryness and then grinded. These materials are easily available and biodegradable.

Chemicals and reagents

All the reagent and apparatus used in experiments were LR/AR grade. Reagents used such as sodium hydroxide (NaOH), sulphuric acid (H_2SO_4), nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) were manufactured by Thermo Fischer Scientific India Pvt. Ltd. Mumbai.

Extraction of cellulose

The extraction process was simple, fast, and ecofriendly adapted from Hasan and Sauodi [10]. About 25 g of powdered material was washed with water several times and then filtered. The residue was dried in sunlight for a couple of days. Then the dried powdered material was put in a plastic container and 500 mL of 1M HNO_3 was added to it bycontinuous stirring with glass rod followed by storage in same for 24 hrs. After complete storage time, the material was subjected to filtration using muslin clothand the residue obtained from the filtration was washed with distilled water for several times. It was then dried in hot air oven (Toshniwal Instruments, India) at 100 °C for complete dryness.

The dried acid treated material was then treated with 500 mL of 1M NaOH by continuous stirring and left over for 24 hrs. It was then filtered with muslin cloth to remove impurities like silica and lignin. The residue was washed with distilled water and then treated with 6M NaOH for 6 hrs. The solution was filtered again with muslin cloth and filtrate was collected. Thus, obtained filtrate was treated with 5N H_2SO_4 in drop wise fashion so that the pH of the resulting solution was in the range of 5-6. The pH of the solution was measured using pH meter. At this level the cellulose begun to precipitate. For complete precipitation and sedimentation, the solution was left for another 24 hrs. After the completion of required time,the precipitate of cellulose was filtered using Whatmann Filter paper No. 42 and the residue was collected leaving behind the filtrate.

Thus, obtained residue was dried in sun until complete dryness. The completely dried residue was

then bleached using H_2O_2 solution and left for 24 hrs. Thus, obtained cellulose was dried, crushed and grinded in mortar and pestle. It was then stored in a sample bottle and used for characterization.

Characterization

Yield of cellulose

After completion of the cellulose extraction the yield of the extracted cellulose was determined using a simple formula [11].

$$\%\ yield\ of\ cellulose = \frac{weight\ of\ extracted\ cellulose}{weight\ of\ material\ taken} \times 100$$

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) has become more widely employed in the modern period to study the chemical structure and functional groups of lignocellulosic substances [1]. FTIR spectrum of the extracted cellulose samples was recorded on IRAffinity-1S instrument with a maximum resolution of 0.5 cm^{-1}. The samples were recorded in the transmittance mode in the range of 4500-500 cm^{-1}.

X-ray Diffraction (XRD)

X-ray Diffraction (XRD) is a practical, non-destructive technique for determining the chemical composition and crystalline structure of natural and artificial materials [12]. Structures, phases, preferred crystal orientations (texture), and other structural factors such as crystallinity, average grain size, strain, and crystal defects are all revealed by XRD [13]. The structural analysis of the samples was evaluated by X-ray diffraction (XRD) using Bruker D2 Phaser X-ray diffractometer with Cu- Kα radiation source (λ = 1.54060Å) operating at 40 kV and 30 mA. The XRD patterns were obtained over the angular range of 2θ = 10° to 80°.

The crystallite size of the samples was calculated using Debye-Scherer equation which is given as [13]

$$CI\ (\%) = \frac{Total\ area\ of\ spectrum - area\ of\ amorphous\ spectrum}{Total\ area\ of\ spectrum} \times 100$$

Where, d is crystallite size of the sample, K is Scherer constant, λ is the wavelength of incident X-rays, β is Full Width of Half Maximum (FWHM) of XRD peaks and θ is the diffraction angles corresponding to the planes.

The crystallinity index (CI) of the extracted samples was calculated using the following formula outlined by Ruland[14].

$$CI\ (\%) = \frac{Total\ area\ of\ spectrum - area\ of\ amorphous\ spectrum}{Total\ area\ of\ spectrum} \times 100$$

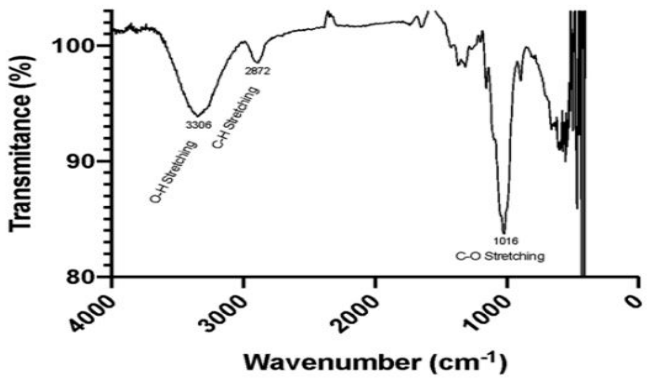
Results and Discussion

Percentage yield of extracted cellulose

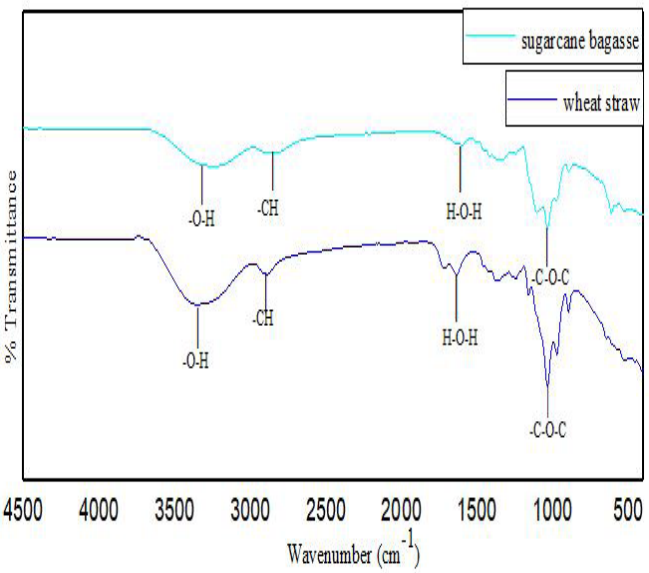
25 g of sugarcane bagasse and 25 g of wheat straw was used to extract the cellulose by the process of acid pretreatment, NaOH degradation and acid hydrolysis. The amount of cellulose extracted was 5.22 g from SCB and 5.48 g from Wheat Straw. Cellulose yield was calculated to be 20.89 % from sugarcane bagasse and 21.92% from wheat straw by the formula mentioned in % yield of cellulose part of characterization above. Thus, more amount of cellulose can be obtained from wheat straw than sugarcane bagasse.

FTIR Analysis of extracted cellulose

FTIR analysis is used for the identification of functional groups through absorption by functional groups present in the sample. The FTIR spectrum of commercial cellulose and extracted cellulose is given in figure 1. FTIR analysis of commercial cellulose showed absorption peak at 3306 cm^{-1} which was due to –O-H band and absorption peak at 2872 cm^{-1} due to –C-H stretching vibration. The peak at 1016 cm^{-1} was due to C-O-C bonds in 1,4-glycosidic linkage[15]. Cellulose from SCB showed absorption peaks at 3255.84 cm^{-1} attributed to –O-H stretching vibration whereas in that of wheat straw shifted to 3340 cm^{-1}. The –C-H stretching vibration of cellulose from SCB and WS showed absorption peaks at 2901 cm^{-1} and 2916 cm^{-1}. Both the cellulose extracted from SCB and WS showed absorption peak of –C-O-C bond vibration at 1033 cm^{-1}.



(a)



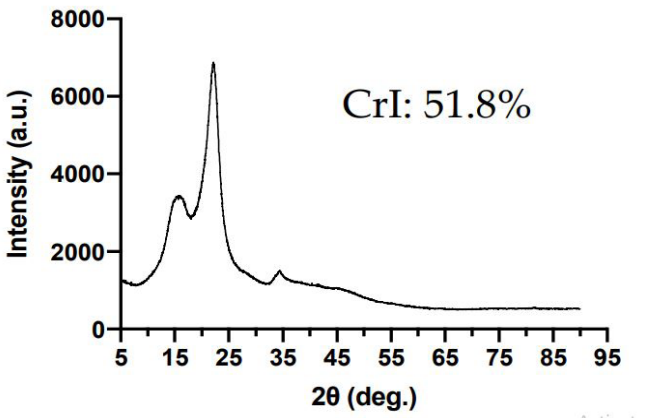
(b)

***Figure 1**: FTIR analysis of (a) commercial cellulose (b) extracted cellulose*

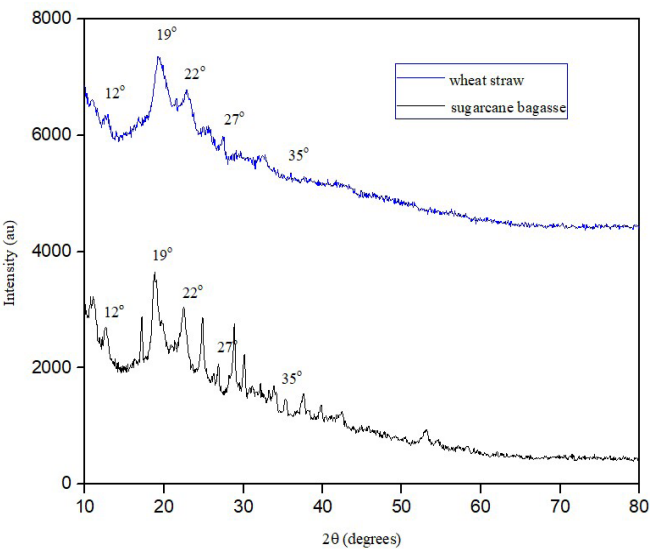
The absorption peak at 972 cm^{-1} and 894 cm^{-1} was associated with cellulosic β-1-glycosidic linkage. The presence of absorption peak at 1732 cm^{-1} in FTIR spectra of WS showed incomplete degradation of hemicellulose. However, in FTIR spectra of extracted cellulose from SCB, it was completely removed. Both the FTIR spectra of cellulose from SCB and WS showed absence of peak at 1502 cm^{-1} which attributed to removal of lignin. The bands at 972.12 cm^{-1} and 894.97 cm^{-1} was associated with cellulosic β-1-glycosidic linkage. The spectral band observed at 1419.61 cm^{-1} and 894.91 cm^{-1} indicated significant cellulose I content [16].

XRD analysis of extracted cellulose

To investigate the crystalline configuration of extracted cellulose, the native samples were characterized by means of X-Ray Diffraction. The X-Ray diffractogram of commercial cellulose and extracted cellulose is shown in figure 2. The main sharp peaks in the diffractogram was 16° and 22°. The peak at around 35° showedcrystalline behavior of typical cellulose Ithat is prominent in sugarcane bagasse than wheat straw[15].Different peaks were obtained for different samples of cellulose. The four diffraction peaks obtained for cellulose from sugarcane bagasse were12°, 19°, 22° and 27° which were specific to Cellulose I_β crystalline allomorph having monoclinic structure [17].



(a)



(b)

***Figure 2**: XRD analysis of (a) commercial cellulose (b) extracted cellulose*

The cellulose obtained from wheat straw showed 2θ values of 12°, 22°, 27° and 35° showing presence of typical cellulose I. The crystallinity index of commercial cellulose was calculated to be 51.8%.

The crystallinity index of cellulose extracted from SCB was calculated to be 51.84% whereas that of WS was only about 17.94%[18,19].The approximate crystallite size of extracted cellulose from SCB was found to be 10.51 nm which is found to be greater than cellulose obtainedfrom wheat straw that was found to be only 4.047 nmusing Debye-Scherer's equation [20].

Conclusion

Manipulating agricultural residues regarding them as renewable resources of energy conceits a cleaner and better environment. Such waste materials are rich in lingo-cellulosic components that can be used to manufacture practicable products. In this study, widely applicable cellulose was extracted from sugarcane bagasse and wheat straw efficiently. Cellulose was extracted using simple, economically viable method of extraction. The yield of cellulose was slightly more when wheat straw was used rather than sugarcane bagasse. XRD and FTIR analysis of obtained sample showed presence of peaks at the respective planes, characteristic absorption peaks elucidating the presence of significant amount of cellulose I_{β}. The approximate crystallite size calculated from XRD peaks showed the extracted particles to be in nanometer range although particle size of cellulose from SCB were larger than cellulose extracted from WS. Furthermore, the measurement of crystallinity index of extracted samples showed that cellulose obtained from SCB was more crystalline in nature than that extracted from WS.Thus extracted cellulose have promising applications in paper bag manufacturing, biomedical fields, as alternative for water treatment processes and many more. Besides these applications, one of the benefits of this extraction method that is difficult to be ignored is its assistance in solving problem of unmanaged agricultural residues and their effect in environment.

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(a) *(b)*

***Figure 3**: Extracted cellulose (a) Sugarcane bagasse (b) Wheat straw*

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