FTIR and XRD Studies of Extracted Cellulose and Methylcellulose from Wheat Straw

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(Received 20 Oct, 2017; Accepted 16 Dec, 2017; Published 20 Dec, 2017)

ABSTRACT: Many Straws have been utilized as crude materials for the preparation of cellulose and methylcellulose. The basic attention in blend finished from straw is that it obtainable good fiber for different industries with special properties, and that it is the foremost accessible source of untreated raw materials taken from agricultural waste. The present effort was to characterization of cellulose and methylcellulose prepared from wheat straw. The cellulose and methylcellulose prepared from wheat straw. The cellulose and methylcellulose received from wheat straw samples are illustrating with Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) methods. FTIR is the good techniques for studying changes by chemicals treatment. FTIR examination of cellulose samples showed downward moving in C-O-C stretching from 1151.15-1077.23cm-1 related to standard cellulose (SC). The cellulose samples showed upward moving of O-H stretching from 3945.56-3269.26cm-1 and originating moving in C-O stretching from 1458.28-1414.26cm-1 vibrations related to SC. The XRD analysis show that the cellulose and methylcellulose crystallinity index. Cellulose and cellulose derivative methylcellulose are utilized as plausible encompassing substance which has wide applications.

Keywords: Wheat Straw; Cellulose; Methylcellulose; FTIR and XRD.

INTRODUCTION: There is a developing enthusiasm for the utilization of non-wood fibers like as yearly plants and agricultural waste, as a crude material for cellulose and cellulose subordinates applications. Agricultural byproducts are the optional results of the vital harvests and described by low crude material cost and direct quality, for example: rice straw and wheat straw.1 Being rich in carbohydrates like cellulose, hemicelluloses, proteins, minerals, silica and fiery debris, their cell divider is very rich in cellulose filaments that were appropriate for examination of methylcellulose.2

Cellulose is a standout amongst the most old hand polymers. Nowadays, cellulose is known to have high potential applications in various fields. It is practical, biodegradable and can be derivatized to yield diverse significant items.3 Cellulose talk to an enormous potential feedstock for different business and has made a considerable measure of research intrigue.4 Cellulose is a polysaccharide, extensive with hundreds or thousands of glucose particles, β-(1→4)-connected D-glucopyranosyl units with three hydroxyl groups, which having shape complex between inter- and intratomic hydrogen bonds.5 Cellulose is composed into fibrils, which are encompassed by a framework of lignin and hemicellulose.5 Cellulose can’t be broken up in similar way to solvents and does not soften prior to the thermal degradation. Various activities have been made to be familiar with realistic solvents for cellulose.6 Wheat straw extracted cellulose which is unsolvable in organic solvents and water. In this manner, cellulose is often changed over into its derivatives methylcellulose.

Methylcellulose is a cellulose derivative that can be set up from the reaction of soluble base cellulose with dimethyl sulphate or iodomethane.7,8 For example, it may be used as thickener, drugs in pharmaceutical industry, bond and as an administrator for adjusting water thickness in the petrochemical business for considerable oil recuperation which empower business.9,10,11,12 Right when this polymer is set up with a level of substitution in the region of 1.4 and 2.0, hot or frosty water may be used to make courses of action or scatterings in low focus.13,14 Methylcellulose demonstrates changes generally in an extension of thermal dependability and water dissolvability related with the development of the level of substitution, which upgrade the business genuine nature of the polymer.15 Considering these points of view, in this work methylcellulose extricated from wheat straw cellulose was depict in association with crystallinity and warm properties.16,17 At this end, the characterization of cellulose and methylcellulose which are extracted and synthesized from wheat straw was done by FTIR and
XRD. The created range was contrasted with Standard Cellulose (SC) and Standard Methylcellulose (SM). In this work it is suggested that for amplifying the likelihood by the utilization of agricultural waste, can be used in the generation of cellulose derivatives.

MATERIALS AND METHODS:

Sites of Sampling: Wheat straw was taken from local agricultural area of Himachal Pradesh of four different districts Bilaspur (BLP), Hamirpur (HMR), Kangra (KNG) and Mandi (MND), India. These samples taken from Himachal Pradesh by the following reasons like very less use other than animal feeding, burning from Himachal Pradesh by the following reasons

Methylcellulose in gram of holocellulose taken for test.

Where

Percentage of alpha cellulose content

Extraction of Cellulose and Synthesis of Methylcellulose: Cellulose was extracted from wheat straw of different sampling sites as Bilaspur (CBLP), Hamirpur (CHMR), Kangra (CKNG) and Mandi (CMND) by TAPPI method, T-203-cm-99. 5gm holocellulose was prepared from oven dry dust which is obtained from wheat straw. It was treated with 30ml of 17.5 percent NaOH at 20ºC. After standing for 5mins duration with 10ml portions with steady shaking, the sample mixture is macerated with flattened glass rod. After 30mins, 75ml of uncontaminated water was added at 20ºC with stirring and then the materials was acceptable to place for 30mins 100ml of pure water at 20ºC was added again and the contents were kept for 30mins more in contact with alkali. The remains was filtered and then soaked in 8.3% NaOH for few mins and drained by suction. The residue was rinse with 250ml of pure water and saturated in 2N acetic acid for 5mins In conclusion mixture was rinse with 400ml of pure water and dehydrated in oven at 105±1ºC. The alpha-cellulose content was determined on O.D. basis as:

\[
\text{Percentage of alpha cellulose content} = \frac{W \times 100}{W}
\]

Where: \(w\) = weight in gram of residue and \(W\) = weight in gram of holocellulose taken for test.

Methylcelluloses [Bilaspur (MBLP), Hamirpur (MMHR), Kangra (MKNG) and Mandi (MMND)] were synthesized from different extracted cellulose [CBLP, CHMR, CKNG and CMND] by methylation process. 5gm α-cellulose oven dry weight was mercerized in 100 grams 40% sodium hydroxide solution for 1hr at the room temperature. Upon the filtration, the mercerized pulp, 150ml 2-propanol, and 50ml iodomethane were added into a flask. The methylation reaction lasted for 22hrs at 60ºC. This process was continual. Finally, Methylcellulose samples were saving in a refrigerator at 4ºC.

Characterization of Extracted Celluloses and Synthesized Methylcelluloses:

FTIR: A Shimadzu IR Prestige-21 FTIR was applying to trace the IR range of the dehydrated powered samples. The powdered sample was mix with material of KBr. For all spectrums, scans were collect with a 4cm⁻¹ determination.

XRD: XRD design was obtained with a Shimadzu Diffractometer XRD 6000 with Kα Cu rays of 4° to 70° and a Ni sieve. These diffraction models are proposed to classify structural changes in the being crystal and imprecise segment after processing the cellulose and methylcellulose calculated.

The FTIR and XRD spectrum of standard cellulose (SC) and standard methylcellulose (SM) are compared with extracted cellulose and synthesized methylcellulose in these studies.

RESULTS AND DISCUSSION: FTIR spectra of Standard Cellulose & Methylcellulose (SC and SM) and extracted cellulose samples (CBLP, CHMR, CKNG and CMND) & synthesized methylcellulose samples (MBLP, MHMR, MKNG and MMND) from wheat straw showed the major differences in cellulose and methylcellulose samples in terms of functional groups at the region from 4000 to 400 cm⁻¹ as shown in Figures 1 & 2.

Comparison the spectra of Standard Cellulose with extracted Celluloses: FTIR spectra of the extracted celluloses from wheat straw compared with that of SC as shown in Figure 1. This showed strong absorption at 3946-3269 cm⁻¹ which is ascribed to O-H stretching vibration. This absorption band is self-possessed of two vibrations positioned at 3302.11-3269.24 cm⁻¹ ascribed to intermolecular hydrogen bonds and 2933.26-2931.13 cm⁻¹ ascribed to intra-molecular hydrogen bonds. The vibration at 1427.31 cm⁻¹ showed in figure 1(a) SC is due to the –CH₂ group. This absorption band moved to 1427.27 cm⁻¹, 1414.26 cm⁻¹, 1427.23 cm⁻¹ and 1428.38 cm⁻¹ in the Figure 1(b) to Figure 1(e) of CBLP, CHMR, CKNG and CMND showed different bands which are ascribed to the –CH₂ groups. The vibration at 1163 cm⁻¹ is assigned for C=O–C stretching which appears almost simultaneously with the vibration located at 894cm⁻¹ ascribed to β–linkage that present in the structure of cellulose.
(Viera, R. G. P. et al., 2007). The vibration located at 1149.24 cm\(^{-1}\) on the figure 1(a) assigned to the antisymmetric bridge C–O–C stretching vibration. The vibration band shifted to 1149.20 cm\(^{-1}\), 1151.18 cm\(^{-1}\) and 1149.32 cm\(^{-1}\) spectrum in all samples CBLP, CHMR, CKNG and CMND respectively in Figure 1(b) to 1(e).

From the FTIR, most of the bands of the extracted cellulose are equivalent with that of the SC. This indicate that the cellulose was successfully extracted from the sample of Wheat Straw from different four district such as Bilaspur, Hamirpur, Kangra and Mandi, and that sufficient removal of lignin and hemi-celluloses was done from the used raw materials. FTIR spectrum which indicates that the most of the lignin was removed. FTIR spectra of four samples of Cellulose which are extracted from wheat straw showed the similar pattern as that of SC.

**Comparison the spectra of Standard Methylcellulose with Synthesized Methylcelluloses:** Comparing the spectrum of SM with the spectra of methylcelluloses which are synthesized from different extracted celluloses. The major changes observed were band intensity decline approximately 3500 cm\(^{-1}\) attributed to stretching the O-H bond (hydroxyl groups) of Methylcellulose, which was to some extent substituted by methyl groups during the methylation reaction, and the increased intensity of the bands between 2835-2900 cm\(^{-1}\) which are attributed to the stretching of C-H aliphatic.

The spectrum of SM observed were band intensity decline 3496.55 cm\(^{-1}\) in figure 2(a) attributed to stretching the O-H bond, which was to some extent substituted by methyl groups during the methylation reaction, and the increased intensity of the bands 2893.49 cm\(^{-1}\) in figure 2(a) which are attributed to the stretching of C-H aliphatic. The spectrum of MBLP, MHMR, MKNG and MMND observed were band intensity decline 3489.52, 3478.35, 3504.63 and 3520.67 cm\(^{-1}\) respectively in figure 2(b-e) attributed to stretching the O-H bond which was some extent substituted by methyl group during the methylation reaction, and the increased intensity of the bands 2895.47, 2898.30, 2891.56 and 2892.60 cm\(^{-1}\) respectively in figure 2(b-e) which are attributed to the stretching of C-H aliphatic.

SM spectra usually present bands at 1459.54, 1368.26, 1334.55 and 947.55 cm\(^{-1}\) attributed to C-H stretching of CH\(_2\) and CH\(_3\) groups, more easily identified in the spectrum of SM (Figure 2(a)). The presence of an intense band around at 1144.55 cm\(^{-1}\) in SM indicates the presence of C-O-C bonds, characteristic of cellulose ethers. MBLP spectra usually present bands at 1451.54, 1369.56, 1335.56 and 947.57 cm\(^{-1}\) attributed to C-H stretching of CH\(_2\) and CH\(_3\) groups, more easily identified in the spectrum of MBLP (Figure 2(b)). The presence of an intense band around at 1081.53 cm\(^{-1}\) in Figure 1(b) indicates the presence of C-O-C bonds, characteristic of cellulose ethers. MHMR spectra usually present bands at 1455.37, 1370.38, 1314.39 and 946.39 cm\(^{-1}\) attributed to C-H stretching of CH\(_2\) and

Figure 1: FTIR spectra of (a) Standard Cellulose and extracted celluloses from Wheat Straw of (b) Bilaspur district (CBLP), (c) Hamirpur district (CHMR), (d) Kangra district (CKNG) and (e) Mandi district (CMND).
CH₃ groups, more easily identified in the spectrum of MBLP (Figure 2(c)). The presence of an intense band around at 1146.37 cm⁻¹ in figure 2(c) indicates the presence of C-O-C bonds, characteristic of cellulose ethers. MKNG spectra usually present bands at 1456.62, 1368.64, 1335.63 and 947.63 cm⁻¹ attributed to C-H stretching of CH₂ and CH₃ groups, more easily identified in the spectrum of MKNG (Figure 2(d)).

The presence of an intense band around at 1079.60 cm⁻¹ in figure 2(d) indicates the presence of C-O-C bonds, characteristic of cellulose ethers. MMND spectra usually present bands at 1454.66, 1368.65, 1335.67 and 946.67 cm⁻¹ attributed to C-H stretching of CH₂ and CH₃ groups, more easily identified in the spectrum of MMND (Figure 2(e)). The presence of an intense band around at 1148.64 cm⁻¹ in figure 2(e) indicates the presence of C-O-C bonds, characteristic of cellulose ethers. For this purpose the spectra were normalized in relation to the band at 1110 cm⁻¹ which is attributed to stretching the C-O-C bond of an anhydroglucoside ring.²¹

X-ray diffraction analysis: The XRD results of SC, SM, extracted cellulose (CBLP, CHMR, CKNG and CMND) and extracted methylcellulose (MBLP, MHMR, MKNG, MMND) of wheat straw from four different districts of Himachal Pradesh was shown in Figures 3-4.

XRD spectra of Standard Cellulose and extracted Celluloses: Figure 3 noticeable the XRD intend of Standard Cellulose (SC), cellulose extracted from wheat straw of four different districts such as CBLP, CHMR, CKNG and CMND obtained after chemical action. The strongest cellulose peak, at 2θ = 22.8°, originates from the cellulose crystalline plane 002 and the peak at 2θ = 16.3° corresponds to the cellulose (101) crystallographic planes.²²,²³ SC illustrates four major reflection peaks. The SC exhibits a peak at curve 1, the peaks at 2θ = 15.22°, 17.12°, 18.06° and 23.06° for the (101), (101̅), (110), and (200) planes are characteristic of the cellulose crystalline form. The CBLP exhibits a peak at curve 2, the peaks at 2θ = 15.28°, 17.38°, 18.22° and 23.24° for the (101), (101̅), (110), and (200) planes. The CHMR exhibits a peak at curve 3, the peaks at 2θ = 15.1°, 17.24°, 18.14° and 23.16° for the (101), (101̅), (110), and (200) planes. The CKNG exhibits a peak at curve 4, the peaks at 2θ = 15.18°, 17.22°, 18.04° and 22.98° for the (101), (101̅), (110), and (200) planes. The CMND exhibits a peak at curve 5, the peaks at 2θ = 15.26°, 17.28°, 18.20° and 23.32° for the (101), (101̅), (110), and (200) planes. Cellulose samples CBLP, CHMR, CKNG and CMND has a very similar diffraction pattern to the SC. The crystallinity index (C.I.) was estimated from the peaks of the (101), (101̅), (110), and (200) planes using the Segal method.²⁴ The C.I. values of the SC, extracted cellulose samples CBLP, CHMR, CKNG and CMND were 44.9%, 52.4%, 54.09%, 51.25% and 56%, respectively showed in table 1.
straw had a greater crystallinity than the SC due to the chemical treatment.

Table 1: Crystallinity Index of Standard and Extracted Celluloses.

<table>
<thead>
<tr>
<th>Samples</th>
<th>SC</th>
<th>CBLP</th>
<th>CHMR</th>
<th>CKNG</th>
<th>CMND</th>
</tr>
</thead>
<tbody>
<tr>
<td>C.I. %</td>
<td>44.9</td>
<td>52.4</td>
<td>54.09</td>
<td>51.25</td>
<td>56</td>
</tr>
</tbody>
</table>

Figure 3: XRD spectra of Standard and Extracted Celluloses.

XRD spectra of Standard and Synthesized Methylcelluloses: XDR patterns for standard and synthesized methylcelluloses are shown in Figure 4. The observed diffraction peaks for all samples can be attributed to crystalline scattering and the diffuse background to disordered regions. The SM spectrum 20 peaks at 8.38° and 20.34° respectively. The spectrum of extracted methylcellulose sample from wheat straw cellulose 20 peaks for MBLP at 8.52° and 20.38°; MHMR at 8.76° and 20.42°; MKNG at 8.5° and 19.98°; MMND at 8.56° and 20.24°. Comparing the XRD patterns of methylcellulose with cellulose spectrum one observes that the peak around 8° is not present in the diffractogram of cellulose, which according 25 is an evidence of cellulose modification. The position of this peak indicates an increase in the interplanar distance compared to the original cellulose diffractogram, due to generation of disorder when cellulose is modified. The projection of the substituting methyl groups is associated with an increase in the inter-fibrillar distance. The maximum peak approximately 20° or slightly greater than 20° presents in the all methylcellulose samples is known as van der Waals forces, which appears for all polymers and corresponds to the polymeric chain packing due to the van der Waals forces. Whereas the maximum around 10°, which is known as light of low van der Walls, which occurs for some amorphous polymers due to the existence of regions with aggregates of segments of parallel chains. 25 The patterns for all samples of methylcellulose are very similar. Crystallinity index % of all samples are 34.22% of SM, 37.68% of MBLP, 30.19% of MHMR, 29.80% of MKNG and 38.96% of MMND shown in table 2. The changes in the crystallinity of SM samples as compared with extracted methylcellulose are due to mercerization performed in the methylation process. Mercerization promotes an expansion of the cellulosic fibers, increasing the portion of less ordered material and reducing the crystalline portion. These crystallinity indexes were the highest found in the literature for methylcellulose samples synthesized from alternative sources of cellulose. For the methylcellulose prepared from sugarcane bagasse and mango seed, crystallinity was between 61% and 52%, respectively. 26 The results showed that obtained methylcellulose samples from wheat straw with lower crystallinity as compared to other extracted methylcellulose from different plant residues.

Table 2: Crystallinity Index of Standard and Synthesized Methylcelluloses.

<table>
<thead>
<tr>
<th>Samples</th>
<th>SM</th>
<th>MBLP</th>
<th>MHMR</th>
<th>MKNG</th>
<th>MMND</th>
</tr>
</thead>
<tbody>
<tr>
<td>C.I. %</td>
<td>34.22</td>
<td>37.68</td>
<td>30.19</td>
<td>29.80</td>
<td>38.96</td>
</tr>
</tbody>
</table>

Figure 4: XRD spectra of standard and Synthesized Methylcelluloses.

CONCLUSION: Agricultural material which are waste contain extensive amounts of cellulosic constituents could be changed over to esteem included items. During the present study, cellulose which has many industrial and medical applications was extracted efficiently from four different sampling sites. The results accessible in this work shown that synthesized methylcelluloses presents properties similar to commercial methylcellulose, which emphasize the importance of the wheat straw extracted cellulose as raw material for producing cellulose derivative methyl-
cellulose. Through the FTIR spectra, a specific absorption region could be observed between 4000–400 cm$^{-1}$ attributed to the vibration. FTIR spectra of all extracted celluloses compared with SC and have shown similar patterns. Similarly synthesized methylcellulose samples compared with SM showed similar pattern. The spectrum of MBLP, MHMR, MKNG and MMND has shown intensity bands at 2895.47, 2898.30, 2891.56 and 2892.60 cm$^{-1}$ respectively which are attributed to the stretching of C–H aliphatic shown similar pattern with SM at 2893.49. XRD analysis validate that extracted cellulose is crystalline and has some amorphous region. The C.I. values of the SC and extracted celluloses CBLP, CHMR, CKNG and CMND were 44.9%, 52.4%, 54.09%, 51.25% and 56%, respectively. The extracted celluloses from wheat straw had a greater crystallinity than the SC due to the chemical treatment. XRD diffractogram has shown two peaks at 8º and 20º in four synthesized methylcelluloses (MBLP, MHMR, MKNG and MMND) which are same as well as SM.

REFERENCES:


