Physico-Chemical Characterization of Microcrystalline Cellulose From A Wild Bamboo (Dendrocalamus Sahnii)

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ABSTRACT

*Microcrystalline cellulose was prepared from a wild bamboo (**Dendrocalamus sahnii**, Naithani & Bahadur) Species collected from Arunachal Pradesh, India, using nitric acid Pulping Process and X-Ray method was adopted for its microstructural characterization. Chemical study showed that although the removal of lignin and pentosan increased with the concentration of nitric acid but it effects the yield of pulp. The crystallinity percentage was found to be 28%. The average value of crystallite size in (002) plane direction was 8.2 nm (corresponding to 10 cellulose unit cells) and 18.8 nm (corresponding to 18 cellulose unit cells) in (040) plane direction. The crystallite size in (040) plane direction is larger than (002) plane direction by 10 nm (corresponding to 8 cellulose unit cells). From the present investigation, however, it can be attributed that the microcrystalline cellulose from this wild bamboo species is a suitable raw material for industrial use.*

INTRODUCTION

Microcrystalline cellulose is industrially important for making tablets, stable dispersion and emulsion in Pharmaceutical industry. It is also used in the preparation of food products and cosmetics as well as chromatographic separations. All forms of natural celluloses, alkali-celluloses, regenerated celluloses and even low-DS cellulose derivatives are used for the preparation of microcrystalline cellulose. Study on microcrystalline cellulose prepared from cultivated species of bamboo have been reported elsewhere (1). As there is no report of microcrystalline cellulose prepared from wild species of bamboo (**Dendrocalamus sahnii**) abundantly available in the hills of N.E. region of India, this paper presents the results obtained from the investigation undertaken on the preparation of microcrystalline cellulose from this wild species. Crystallinity and crystallite size of microcrystalline cellulose derived from this species are determined using X-ray diffraction method.

RAW MATERIAL

Wild bamboo (**Dendrocalamus Sahnii,** Naithani and Bahadur) collected from Arunachal Pradesh, India, was used as raw material for this study. For making pulp, the dried bamboo culms were first split, crushed and then cut into chips of (1/2)″ x (3/4)″ lengths.

PREPARATION OF HIGH ALPHA PULP

The chips were digested with nitric acid with occasional stirring in a round bottom flask of 5 litre capacity fitted with a condenser maintaining solid-liquid ratio 1:4. The treated chips were cooked at 160 $\pm$ 5°C temperature for two hours. The Chips after acid treatment were washed with water and the yield was determined. The acid treated chips were later digested with 2% sodium hydroxide w/w at 120°C for four hours. After completion of the digestion, the pulp was thoroughly washed with water and then bleached in six stages bleaching sequence.

PREPARATION OF MICROCRYSTALLINE CELLULOSE

The dried bleached pulp was treated with 0.1% Hydrochloric acid (v/v) at 3% consistancy for a period of 1/2 hour at 40-50°C and finally washed with distilled water to make acid free. Alpha-cellulose,

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Ash content, Pentosan content of the acid treated pulp were analysed.

X-RAY STUDY

Crystallinity (%) Determination

The degree of crystallinity (Kc) was determined from the ratio of the integrated crystalline scattering to the total scattering, both crystalline and amorphous and is given[2] :

$$Kc = \int_0^\infty s^2 r_c (s)ds / \int_0^\infty s^2 r (s)ds \qquad (E-1)$$

$$Crystallinity (\%) = KC \times 100\% \qquad (E-2)$$

Where : S is the magnitude of the reciprocal-lattice vector and is given by :

$$s = (2 \sin\theta)/\lambda$$

θ is one-half the angle of deviation of the diffracted rays from the incident x-rays.

λ is the x-ray wavelength.

I (s) is the intensity of coherent x-ray scattered from the specimen (both crystalline and amorphous).

Ic (s) is the intensity of coherent x-ray scattered from crystalline region.

Crystallite Size Determination

The mean crystallite size of a powder composed of relatively perfect crystalline particles can be determined with the familiar Scherrer equation :

$$L_{hkl} = K\lambda/\beta_0 \cos\theta \qquad (E-3)$$

Where L_{hkl} is the mean dimension of the

crystallites perpendicular to the plane (hkl).

β_0 is the integral breadth or breadth at half-maximum intensity of the pure reflection profile in radians.

K is a constant that is commonly assigned a value of unity.

Instrumental brodening was determined using the calibration curve method given elsewhere[3]. Both Cauchy and Gaussian methods were used for correcting the experimental reflection profiles for instrumental broadening as given below :

Cauchy profile

$$\beta = B - b$$

Gaussian profile

$$\beta^2 = B^2 - b^2$$

Where B = Breadth of experimental profile

b = Breadth of instrumental line broadening

β = Integral breadth of pure line profile

Diffraction data were obtained using computer controlled x-ray diffractometer (model JDX-11P3A, JEOL, JAPAN) attached with pulse-height analyser and scintillator counter with scintillator NaI (Ti) single crystal. The background correction and FWHM measurement were made from the X-ray pattern using Software PEAK SEARCH AND DATA DISPLAY: PATTERN respectively supplied with XRD Unit.

RESULTS AND DISCUSSION

Chemical analysis of bamboo chips is reported in **Table No. 1. Table No. 2** shows the effect of nitric acid concentration on the yield of pulp and its various chemical constituents. Bleaching conditions adopted for the nitric acid treated pulp are given in **Table-3.** Analysis of acid treated chips, unbleached and bleached pulp are recorded in **Table-4a, 4b** and **4c** respectively. Experimental results show that the removal of lignin and pentosan increases with the increasing of concentration of nitric acid. This however, effect the yield of pulp.

XRD pattern of the sample is shown in **Fig.1.** The crystallinity percentage for this microcrystalline cellulose was found to be 28%. The size of cellulose ..ystallites was calculated for quantitative comparision along (002) and (040) directions using equation

Table-1

Chemical Analysis of the Bamboo Chips

	Percent on O.D. basis
Alcohol/Benzene Solubility	05.12
1% NaOH Solubility	22.02
Lignin	23.95
Cross and Bevan Cellulose	62.51
Alpha Cellulose	44.59
Cold Water Solubility	10.95
Hot Water Solubility	14.51
Pentosan	18.96
Ash	02.07

Table-2

Analysis of chemical constituents of pulp cooked at different nitric acid concentration

Analysis	Percentage of acid (v/v) used for digestion 1	2	3	4
Alcohol/Benzene Solubility (%)	05.55	06.75	07.45	07.07
1% NaOH Solubility (%)	23.23	26.67	28.34	29.32
Cross and Bevan Cellulose (%)	64.15	64.41	66.11	70.34
Pentosan (%)	13.21	10.95	09.24	07.05
Ash (%)	02.13	02.02	01.82	01.83
Acid consumed(%)	08.54	08.56	09.29	08.77
Yield (%)	80.20	78.50	74.30	70.80

(E-3). Using cell dimensions [4] a = 8.35Å, b = 10.3 Å, c = 7.9 Å and α= 84°, the numbers of cellulose unit cells were calculated. Table-5 shows the cellulose crystallite dimensions in (002) and (040) plane directions. The Average value of crystallite size as determined using Cauchy and Gaussian corrections in (002) direction was 8.2 nm (corresponding to 10 cellulose unit cells) in (040) plane direction. However, it can be attributed that crystallite size in (040) plane direction is larger than (002) plane direction by 10.6 nm (corresponding to 8 cellulose unit cells). The results obtained from standard microcrystalline pulp cellulose [1] are also included in **Table-5** for direct comparision with the microcrystalline cellulose under investigation. As the data obtained from the present investigation are comparable to that of standard microcrystalline cellulose, this microcrystalline cellulose, however, can be a suitable raw material for industrial use.

CONCLUSION

The study showed that although the removal of lignin and pentosan increased with the concentration

Table-4a

Analysis of acid cooked bamboo chips

	% on O.D. basis
Alcohol/Benzene Solubility	08.85
1% NaOH Solubility	35.83
Lignin	12.56
Cross and Bevan Cellulose	74.45
α - Cellulose	60.63
Pentosan	05.34
Ash	01.24
Yield of the Chips	60.20

Table-4b

Analysis of unbleached pulp

	% on O.D. basis
Alcohol-Benzene Solubility	01.43
α - Cellulose	85.43
Pentosan	02.45
Permanganate No.	06.50
Yield	35.00

Table-4c

Analysis of Bleached pulp

	% on O.D. basis
α - Cellulose	94.690
β - Cellulose	03.010
Pentosan	03.080
Ash	00.075
Silica	00.018
Kappa No.	00.990
Yield	29.500

Table-3

Bleaching conditions for the preparation of pulp

Stage	1	2	3	4	5	6
Chemical	Cl	NaOH	NaOCl	NaOH	NaOCl	SO_2
Pulp Consistency (%)	3	4	4	4	4	4
Temperature (°C)	20	60	40	60	40	20
Time (hr)	1	1	2	1	2	0.5

Table-5

Crystallinity, Crystallite size and number of unit cells of microcrystalline cellulose (Dendrocalamus sahnii)

Sample	Crystall inity%	Crystallito size and number of unit cells											
		Basal plane 002				Basal plane 040				Average value			
		Cauchy correction		Gaussian correction		Cauchy correction		Gaussian correction		Basal plane 002		Basal plane 040	
		Size (nm)	No. of unit cells	Size (nm)	No. of unit cells	Size (nm)	No. of unit cells	Size (nm)	No. of unit cells	Size (nm)	No. of unit cells	Size (nm)	No. of unit cells
A*	28	8.0	10	8.4	11	19.6	19	17.9	17	8.2	10	18.8	18
B**	33	9.6	12	9.2	12	14.0	14	13.1	13	9.4	12	13.6	13

A* Sample under investigation.
B** Dendrocalamus hamiltonii (Ref. 1) as standard.

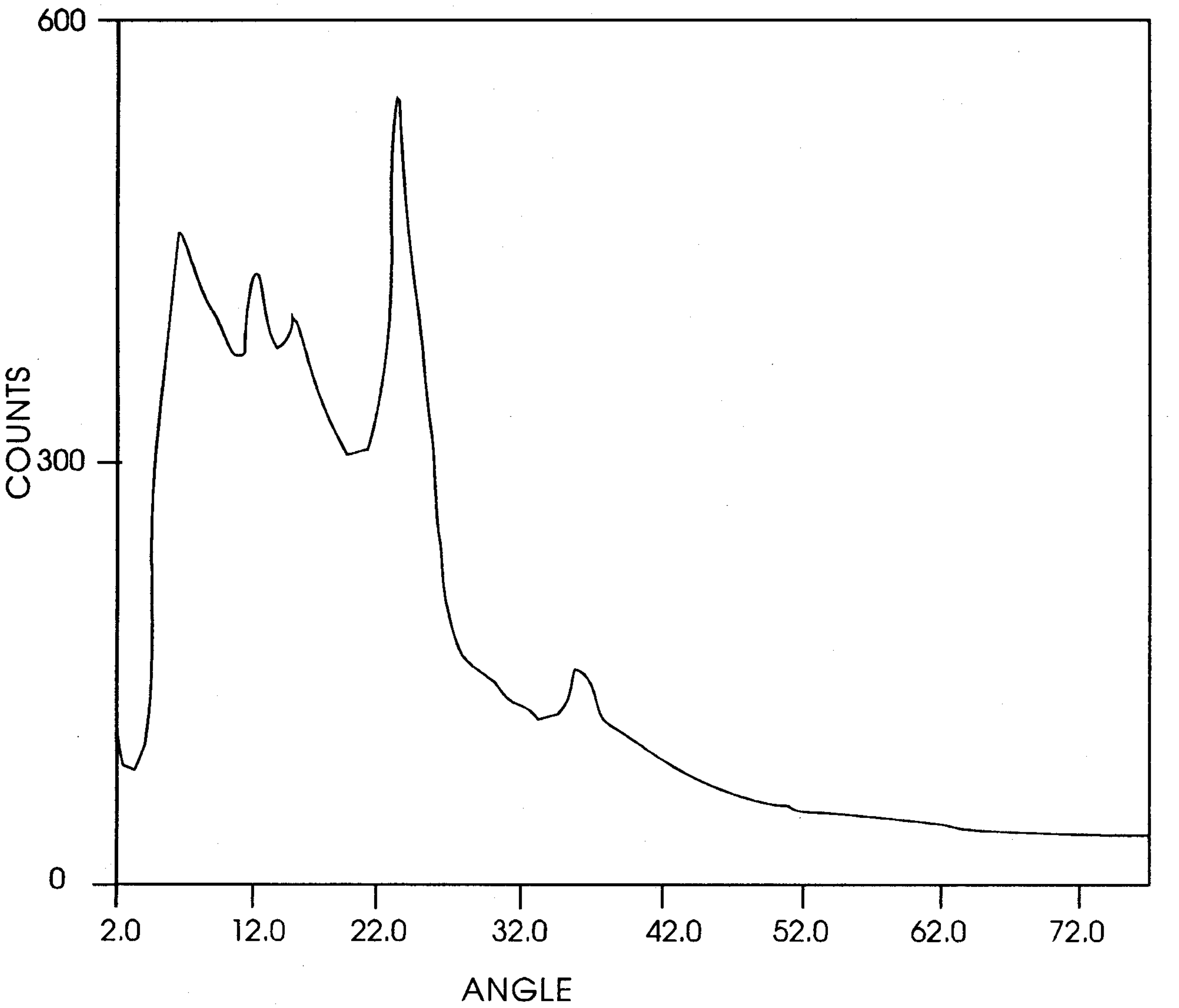

Fig.1 XRD Pattern of Microcrystalline Cellulose of Wild Bamboo Species (D. sahnii).

of nitric acid but it effects the yield. The crystallinity percentage was found to be 28%. The average value of crystallite size in (002) plane direction was found to be 8.2 nm (corresponding to 10 cellulose unit cells) and it was 18.8 nm (corresponding to 18 cellulose unit cells) in (040) plane direction. It can be concluded from the present investigation that microcrystalline callulose obtained from this bamboo species is found suitable for industrial use.

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