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Isolation and characterization of bamboo fibres

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Abstract: Bamboo fibres were isolated from three Indian species of bamboo and their physical characteristics, dyeability with different reactive dyes and antimicrobial activity were evaluated. On dyeing the fibres with selected reactive dyes, excellent dye exhaustion and fixation above 90 per cent were obtained. Bleached bamboo fibres showed antimicrobial activity of over 90 per cent.

Key words: Bamboo fibre, delignification, dyeing, antimicrobial activity.

INTRODUCTION

Fibres isolated from bamboo are put to use in various commercial applications such as paper, food and bioenergy and their applications in reinforcement and composite board are being explored (Deshpande *et al.*, 2000; Scurlock *et al.*, 2000; Vu *et al.*, 2004; Ghavami, 2005). There is a need to evaluate bamboo fibres for the production of sanitary napkins, shoe insoles, fibre fills in pillows, bed cover, *etc.* However, information available on the fibres isolated from various species of bamboo is limited. Therefore, the present study was taken up to investigate the physical characteristics of fibres such as fibre length, diameter, moisture regain, X-ray crystallinity, dyeing and to study the antimicrobial properties of bleached and unbleached bamboo fibres.

MATERIALS AND METHODS

Ochlandra travancorica, *Bambusa tulda* and *Dendrocalamus hamiltonii* provided by the National Mission on Bamboo Applications (NMBA), DST-TIFAC, New Delhi were used for the study. One-year-old and two-year-old culms of *O. travancorica* and older culms of other species were studied. The reactive dyes (Table 1) supplied by Amtex Dye Chem Industries (India) were used.

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Isolation of fibres

Culms of all the three species of bamboo were chopped into chips and delignified by boiling with different concentrations of sodium hydroxide under a pressure of 1×10^5 Pa at 120°C for different durations. The material-to-liquor ratio was kept at 1:20. Final traces of alkali present in the fibre were removed by neutralizing with glacial acetic acid (pH 3-4) for 1 h. The bamboo strands were then thoroughly washed with water and dried at room temperature.

Delignified bamboo strands were bleached with 4 per cent (w/v) sodium chlorite at boil for different durations. The material-to-liquor ratio employed was 1:40. Formic acid was added to maintain pH 3-4. The fibres were then thoroughly washed with water and dried at room temperature.

Lignin content was calculated according to the following equation:

(A-B)/A * 100

Fibre yield was calculated as:

B/A * 100
where A is the weight of the untreated bamboo chips and B is the weight of the conditioned bamboo fibres after bleaching.

Physical properties of the fibres

Fibre dimensions
The length of bamboo fibres was measured manually by taking the reading of 50 samples. The diameter of the bamboo fibres was measured using an optical microscope (Leica), at a magnification of 100. Mean values were computed from 50 readings.

Moisture regain
Moisture regain was measured using a moisture analyzer (Sartorius MA 30). The samples were conditioned at 65 per cent RH and 24°C in a desiccator containing saturated sodium nitrite for more than 48 h prior to testing. Five readings were taken to compute the mean moisture regain.

X-ray crystallinity
X-ray diffractograms (scan range (2θ) = 10-35°,θ = diffraction angle, scan speed: 5 degree/min) of the samples were obtained with a PANalytical X-ray diffractometer having a X-ray tube producing monochromatic CuKα radiation. The powdered fibre samples were mounted onto sample stage to record the crystallinity index. Sample stage was mounted on horizontal axis and the diffracted beam optics and the detector

were mounted onto 2θ axis. The curves were then analyzed for crystallinity.

The % crystallinity was estimated using the equation:

% Crystallinity = (Crystalline Area/ Total Area) * 100

Fourier transform infra red (FTIR) spectroscopy

Fourier transform infra red (FTIR) spectra were recorded on a Perkin Elmer BX FTIR system using potassium bromide discs. A total of twenty scans for each sample were taken with a resolution of 4 cm^{-1}. The sample for FTIR was prepared as follows: KBr pellet was made by dispersing 1 mg of powdered fibre into 100 mg of KBr. Both fibre and KBr were dried before pellet preparation and the IR spectra were recorded immediately.

Dyeing studies

Fibres were prepared for dyeing by scouring them in a solution of 1 g/l of non-ionic detergent (Lissapol N) for 45 min at 80°C (liquor-to-fibre ratio 50:1). This was followed by rinsing in hot and cold water and then drying the fibres. Time and temperature profile for different dyeing processes were followed as specified by Bairagi *et al.* (2005) and the additions made to the various dyebaths are given in Table 2. Two per cent and four per cent shades were dyed. The percentage of dye exhaustion (E) achieved for each dyeing was determined spectrophotometrically, using a Pharmacia Biochrome 4060 spectrophotometer, according to the following equation:

$E\ (\%) = [1-(C_2 / C_1)] * 100$

where C_1 and C_2 are the concentrations of dye before and after dyeing respectively.

Dye fixation was measured by stripping the dyed samples with 50 per cent (w/w) DMF (liquor-to-fibre ratio 40: 1) for 15 min until all the unfixed dye was removed. Reflectance spectra of dyed and undyed samples were recorded and K/S (colour yield) values were calculated with Jaypak X_4000 spectrophotometer (illuminant D65, 10° observer). The percentage of dye fixation was then calculated according to the following equation:

$F\ (\%) = (K/S)_2 / (K/S)_1 * 100$

where $(K/S)_1$ and $(K/S)_2$ are the colour strengths of the sample before and after stripping respectively.

From the results of the dye exhaustion and fixation percentages, the total dye fixation (T) (*i.e.*, the percentage of dye which was chemically bound to the sample relative to the amount of dye applied to the sample) was calculated for all the dyeings using the

equation:

$T(\%) = \{E(\%) \times F(\%)\} / 100$

Antimicrobial activity testing

The bamboo fibres were analysed for their antimicrobial activity using two bacteria, *Staphylococcus aureus* as the gram positive bacterium and *Escherichia coli* as the gram negative bacterium. *S. aureus* (NCIMB-17) and *E. coli* (B27) were procured from the culture collection of Department of Biochemical Engineering and Biotechnology, IIT, Delhi, India. The bacterial culture was prepared by growing organisms in a flask containing 100 ml of 5 g/l peptone and 3 g/l beef extract with shaking at 37°C for 24 h. The bacterial culture of *E. coli* was found to have 3-4 * 10^8 colony forming units (CFU) and of *S. aureus* was found to have 3-4 * 10^7 CFU.

Fibre samples weighing 0.10 g were sterilized (120°C, 30 min, 1×10^5 Pa) placed in the liquid culture medium and 10 µl of bacterial culture (*E. coli* and *S. aureus*) was added. The liquid culture medium was prepared with 5 g/l peptone and 3 g/l of beef extract. The medium was incubated at 37°C for 24 h.

A solid culture medium containing 5 g/l peptone, 3 g/l beef extract and 20 g/l agar at pH 6.8 ± 0.1 was poured into petri dish and 100 µl of the microbial culture was spread over it. The agar plates were incubated for 16 h at 37°C. The microbial inhibition was determined by counting the colony forming units (CFU) and calculating the bacteria reduction percentage (BR %) after incubation using the equation:

$BR(\%) = [1-(A / B)] * 100$

where A is CFU/ml for the treated sample after 24 h incubation time and B is CFU/ml for the mother culture after 24 h incubation time.

Data Analysis

This was a preliminary exploratory study with limited samples. The data could not be subjected to statistical analysis.

RESULTS AND DISCUSSION

Isolation of fibres

On dissolution of lignin in sodium hydroxide, bamboo fibres were extracted with relative ease. From preliminary trials it was observed that maximum yield of fibres could be obtained at the following treatment conditions.

For *O. travancorica* (1- and 2-year-old):
Sodium hydroxide (w/v) = 1%; time = 2 h

For *B. tulda* and *D. hamiltonii*:
Sodium hydroxide (w/v) = 4%; time = 4 h

The lignin content and fibre yield of the bamboo fibres of different species are given in Table 3. It is evident that the lignin content in *B. tulda* and *D. hamiltonii* is more than in *O. travancorica*. This may be attributed to the increase in lignin content with the increase in age of bamboo. Fibre yield from *O. travancorica* is more than that from *B. tulda* and *D. hamiltonii*.

Delignification by alkali treatment resulted in change of colour of bamboo chips from brown to yellowish brown. Sodium chlorite bleaching of the fibre changed the colour to white. This has been attributed to the oxidative decomposition of the chromophore in lignin moiety to colourless substances (Subramaniam *et al.*, 2005).

The whiteness index of the bamboo fibres bleached for different time durations measured with spectrophotometer is given in Table 4. It was observed that whiteness index of bamboo fibres bleached for 120 min is more than fibres bleached for 90 and 60 min.

Physical properties

The physical characteristics of the bamboo fibres are given in Table 5. The length of isolated bamboo fibres was in the range of 3.7-5.0 mm with variation as high as 25 per cent. For *O. travancorica*, fibre length was about 5 mm and for *B. tulda* and *D. hamiltonii*, it was close to 3.7 mm. Diameter of isolated bamboo fibres was in the range of 9.0-10.5 μm with variation as high as 20 per cent.

Moisture regain of raw bamboo was in the range of 8.5-9.4 per cent and of bleached bamboo fibres, in the range of 9.0-9.6 per cent. This is an important parameter for application such as sanitary napkins, insoles, *etc.*

The X-ray diffractograms of the raw and bleached bamboo fibre samples recorded for the 2θ in the range of 10-35° revealed pronounced Bragg peaks around 15.7°, 15.9° and 22° indicating the presence of crystalline phase in the fibre samples. Sorption properties of fibers is governed by the crystallinity. Raw bamboo fibre was 48-66 per cent crystalline and bleached bamboo fibres were 60-69 per cent crystalline. The small increase in crystallinity may be attributed to the pure form of cellulose obtained by bleaching.

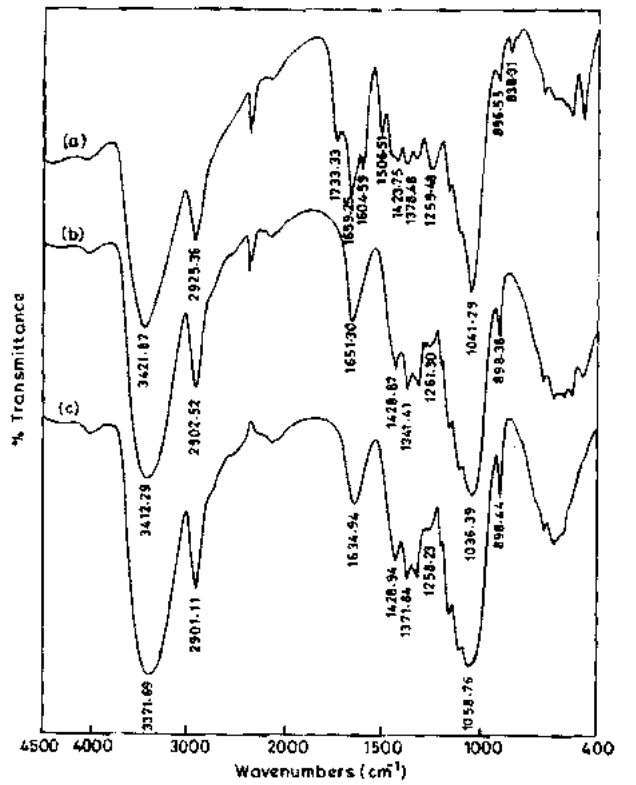

Figure 1. FTIR spectra of *O. travancorica* (2-year-old) (a) raw fibre (b) bleached fibre, 60 min (c) bleached fibre, 120 min.

FTIR analysis

FTIR spectra of raw and bleached bamboo fibres are given in Figure 1. The raw and bleached bamboo fibre samples of different species showed common absorption peaks at around 3410, 2900, 1600, 1370, 890 cm^{-1} which are similar as reported in other ligno-cellulosic fibres (Lawther *et al.*, 1996; Boeriu *et al.*, 2004; Subramaniam *et al.*, 2005).

Lignin in raw bamboo showed strong bands with peaks around 1260 cm^{-1} arising from guaiacyl ring (G ring), around 896 and 838 cm^{-1} arising from C-H out of plane vibration in position 2 and 6 of syringyl ring (S ring) (Boeriu *et al.*, 2004). These lignin bands either disappeared or were less intense in fibres bleached for 90 and 120

Table 1. Dyes used in this study

Dye	Commercial name	Type	λ_{max} (nm)
1	Reactive Blue R	Vinyl sulphone	600
2	Reactive Yellow HEXL	Homo-bifunctional	430
3	Reactive Navy Blue BFN	Hetero-bifunctional	600

Table 2. Dyebath additions

Dye	Additions	Quantity (g/l)
1	Glauber salt / Tri sodium phosphate	60 / 20
2	Sodium chloride / Soda ash	40 / 15
3	Glauber salt / Soda ash	60 / 20

Table 3. Lignin content and fibre yield of bamboo

Bamboo species	Sodium hydroxide (w/v) (%)	Time (h)	Lignin content (%)	Fibre yield (%)
O. travancorica (1-year-old)	1	2	44.80	55.19
O. travancorica (2-year-old)	1	2	45.36	54.64
B. tulda	4	4	52.35	47.74
D. hamiltonii	4	4	56	44

Table 4. Whiteness index of bleached bamboo fibre samples

Sample	WI (Hunter)
O. travancorica (1-year-old)	
Bleached, 60 min	57.15
Bleached, 90 min	62.74
Bleached, 120 min	63.96
O. travancorica (2-year-old)	
Bleached, 60 min	48.94
Bleached, 90 min	66.51
Bleached, 120 min	67.85
B. tulda	
Bleached, 60 min	66.04
Bleached, 90 min	67.42
Bleached, 120 min	66.62
D. hamiltonii	
Bleached, 60 min	66.07
Bleached, 90 min	68.62
Bleached, 120 min	68.95

min as can be seen from spectra in Figure 1 which may be because of the chemical treatment given to bamboo fibres.

Qualitative assessment of lignin in the bamboo fibres was done by staining the samples with an aqueous solution of Malachite Green. It was observed that the raw bamboo fibres developed deep-blue colour quite extensively, whereas samples treated with sodium chlorite for 90 and 120 min developed medium and light green colour, respectively. Reflectance spectra of Malachite Green stained bamboo fibres were recorded and K/S (colour yield) values were calculated (Table 6). It can be seen that the values of K/S decreased in the order of bleached fibre, 120 min < bleached fibre, 90 min < bleached fibre, 60 min < delignified bamboo < raw bamboo. This indicates

Table 5. Physical properties of bamboo fibres

Property	*O. travancorica* 1-year-old	*O. travancorica* 2-year-old	*B. tulda*	*D. hamiltonii*
Length (mm)	5 ± 1.12	5.16 ± 0.87	3.7 ± 1.5	3.75 ± 0.09
CV (%)	22	16.9	20	24.78
Diameter (μm)	10.26 ± 2.1	10.44 ± 1.53	9.19 ± 1.15	10.01 ± 1.02
CV (%)	20	14	12.55	10.19
Moisture regain (%)				
(raw)	9.368 ± 0.11	9.00 ± 0.38	8.554 ± 0.14	9.14 ± 0.46
(bleached)	9.56 ± 0.26	9.12 ± 0.29	9.212 ± 0.29	9.4 ± 0.2
Crystallinity (%)				
(raw)	51.41	58.52	66.32	48.21
(bleached)	61.25	60.35	66.25	69.03

Table 6. Qualitative assessment of lignin in bamboo fibre samples stained with Malachite Green

Sample	L*	K/S
O. travancorica (1-year-old)		
Raw	35.48	11.24
Delignified	27.21	7.32
Bleached, 60 min	53.13	1.75
Bleached, 90 min	63.01	0.94
Bleached, 120 min	63.99	0.92
O. travancorica (2-year-old)		
Raw	35.64	13.59
Delignified	26.52	8.73
Bleached, 60 min	55.52	3.93
Bleached, 90 min	64.01	1.54
Bleached, 120 min	64.59	1.02
B. tulda		
Raw	35.72	14.35
Delignified	27.26	9.48
Bleached, 60 min	56.08	4.68
Bleached, 90 min	66.01	2.11
Bleached, 120 min	65.52	2.03
D. hamiltonii		
Raw	36.12	15.12
Delignified	25.52	11.54
Bleached, 60 min	56.9	4.9
Bleached, 90 min	65.59	2.17
Bleached, 120 min	66.35	2.16

that the lignin content goes on decreasing as the duration of sodium chlorite treatment increases.

Moreover, results of measurement of L* values of these samples given in Table 6 also indicate that the shade goes on becoming lighter on removal of lignin which supplements K/S values. Malachite Green staining test supplements the FTIR spectroscopy observations.

Dyeing studies

On dyeing the bamboo fibres isolated from different species of bamboo with selected reactive dyes, 86-97 per cent of dye exhaustion and 72-96 per cent of total dye fixation were obtained as can be seen in Tables 7 and 8. Dye exhaustion and total dye fixation of fibres isolated from *B. tulda* and *D. hamiltonii* were found to be slightly lower

Table 7. Dyebath exhaustion (%) of bamboo fibres dyed with different reactive dyes

Fibre sample	Reactive Blue R		Reactive Yellow HEXL		Reactive Navy Blue BFN	
	2%	4%	2%	4%	2%	4%
O. travancorica (1-year-old)	92.00	92.00	96.25	95.75	97.25	96.37
O. travancorica (2-year-old)	92.00	93.00	97.00	95.37	97.25	96.62
B. tulda	86.00	89.50	95.50	95.25	97.25	96.62
D. hamiltonii	89.25	91.50	96.50	96.25	97.25	96.62

Table 8. Total dye fixation (%T) of bamboo fibres dyed with different reactive dyes

Sample	Reactive Blue R		Reactive Yellow HEXL		Reactive Navy Blue BFN	
	2%	4%	2%	4%	2%	4%
O. travancorica (1-year-old)	83.15	91.10	72.75	78.86	96.85	95.10
O. travancorica (2-year-old)	78.71	86.60	82.36	85.86	91.13	88.35
B. tulda	77.00	82.87	73.51	82.04	84.36	85.84
D. hamiltonii	79.10	81.83	72.15	84.21	90.00	88.25

Table 9. Antimicrobial activity of bamboo fibres

Sample	Bacteria reduction percentage (BR %)	
	Escherichia coli	*Staphylococcus aureus*
O. travancorica (1-year-old)		
Raw	26.6	59
Delignified	31.73	71
Bleached, 60 min	42.4	95.8
O. travancorica (2-year-old)		
Raw	27.2	44.33
Delignified	37.06	72.6
Bleached, 60 min	45.33	94.5
B. tulda		
Raw	18.66	44
Delignified	23.2	70.5
Bleached, 60 min	31.2	94.33
D. hamiltonii		
Raw	14.66	45.16
Delignified	22.4	75
Bleached, 60 min	31.7	92.5

than those from *O. travancorica* (1- and 2-year-old) which may be due to the more crystalline nature of the older bamboo.

Antimicrobial activity

The results of the antimicrobial activity of various forms of bamboo (raw, delignified and bleached fibres, 60 min) (Table 9) reveal that bamboo fibres bleached for 60 min had the maximum bacteria reduction in the range of 92-96 per cent against *S. aureus* and 32-45 per cent against *E. coli*. This may be due to the presence of trace amounts of chlorine in bleached fibres which is responsible for imparting the antimicrobial activity. This could be of importance in developing hygiene products and in clothing.

CONCLUSION

Fibres isolated from different Indian bamboos have length in the range of 3.7-5.0 mm and diameter in the range of 9.0-10.5 μm. Bleached fibres have moisture regain varying from 9.0-9.6 per cent which is slightly more than that of raw fibre. Fibres isolated from *B. tulda* and *D. hamiltonii* are more crystalline than those from *O. travancorica* (1- and 2-year-old). The FTIR analysis indicated that almost all the lignin in fibres produced from various species of bamboo was eliminated after bleaching with sodium chlorite. When dyed with reactive dyes, the bamboo fibres showed good exhaustion and total dye fixation of above 90 per cent. Fibres bleached for 60 min showed maximum bacteria reduction in the range of 92-96 per cent against *S. aureus*.

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