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# PROJECT REPORT FORM

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٨, PROJECT NO. 2278 COOPERATOR Institute of Paper Chemistr 1 REPORT NO. April 30, 1962 DATE <u> 1997 </u> NOTE BOOK-81 PAGE τo SIGNED M M. G. Karnik Research Officer Cellulose and Paper Branch Forest Research Institute Dehra Dun, U.P., India 1 or A

## HEMICELLULOSES AND DISSOLVING PULP FROM INDIAN BAMBOO (DENDROCALAMUS STRICTUS)

#### ABSTRACT

A dissolving pulp was prepared from Indian bamboo, <u>Dendrocalamus</u> <u>strictus</u>, using a modified alkaline cook. While the silica content and ash in general were somewhat higher than in dissolving pulps from wood, they were much lower than is usual in such pulps from the grass family. A limited evaluation for the viscose process carried out by courtesy of the American Viscose Corporation showed that, except for filtration, the bamboo pulp presented no undue difficulties in viscose processing and is potentially useful. In a preliminary composition study 40.80% of the weight of a chlorite holocellulose was removed by alkaline extractions but only half of this was recovered. Their sugar composition and extractibility indicate that they probably belong predominantly in the methylglucuronoarabinoxylan group. There are also small amounts of galactans and mannans present, however, the structural relationship was not investigated. X-ray diffractograms and intrinsic viscosity measurements were made.

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#### INTRODUCTION

Bamboo constitutes a large potential source of cellulose in tropical and subtropical regions of the world. The usefulness of this member of the huge Graminae family is enhanced by its very rapid growth. In fact, it forms one of the main fibrous materials for pulp and paper manufacture in India. It is natural, therefore, that bamboo should be one of the first sources considered when the question of discolving pulp manufacture in India is raised.

In 1961, by arrangement with the Indian government and the International Co-operation Administration, Washington, D.C., Mr. Karnik who has been very active in the investigation of indigenous Indian fibers at the Forest Research Institute, Dehra Dun, India spent eight months at The Institute of Paper Chemistry, Appleton, Wisconsin. In this co-operative study he expanded his preliminary investigations on bamboo hemicelluloses and dissolving pulps (<u>1-3</u>). The present paper outlines the results of this work.

<u>Dendrocalamus strictus</u> is the genus most commonly used for pulping in India. Several discussions of its properties, management and utilization in paper are available (4-6).

There has been a good deal of study of possible processes for preparing banboo dissolving pulps, which has been reviewed by Maffziger, <u>et al.</u>, in his paper on the subject (7). Only Karnik seems to have used <u>D</u> strictus; it may not be safe to assume that all banboos behave alike, although Thoria (8) points out similarity. In his work with dissolving pulps (8, 9) be does not specify the kind of bamboo used. In the present paper the <u>D</u>, strictus pulp has been evaluated as reyon and found promising.

A similar situation exists with the subject of bamboo hemicelluloses. It has been known since 1928 (10) that the isolated bamboo hemicelluloses are in the xylan group. Recently Karnik (3) has further studied those from <u>D</u>. <u>strictus</u> in a preliminary study, while Matsuzaki, <u>et al</u>. (11) have described those from a Japanese bamboo [Moso bamboo, which is <u>Phyllostachys</u> <u>edulis Riv. (5)</u>]. In the present paper, Karnik's study has been made more quantitative. The values for uronic anhydride content are higher than those of Matsuzaki, but in both cases the main hemicellulose of bamboo would seem to be consistent with the structure of 4-0-methylglucuronoarabinoxylan, but the only basis for this so far is analogy with the wood hemicelluloses.

# A. INVESTIGATIONS OF HEMICELLULOSES

The culms were collected from the Demonstration Area of the New Forest Estate, Dehra Dun, U. P., India. The bamboo was three years old, yellowish green in color and in sound condition. For the separation of hemicelluloses, the bamboo was split and prepared as airdry shavings. The shavings were then ground in the Wiley mill, passing a no. 3 screen of 0.120 inch diameter openings. The ground material was then air dried. Table I shows the analysis of these shavings.

# TABLE I

## CHEMICAL ANALYSIS OF BAMBOO SHAVINGS

	%, o.d. material
Ash ( <u>21</u> ) at 425°C.	1.90
700°C.	1.74
Silica ( <u>22</u> )	0.48
Nitrogen ( <u>23</u> )	0.63
Alcohol-benzene soluble ( <u>24</u> )	6.50
Ethyl ether soluble ( <u>25</u> )	2.10
Cold water soluble ( <u>26</u> )	9.60
Hot water soluble ( <u>26</u> )	11.20
1% NaOH soluble ( <u>27</u> )	29.70
Klason lignin ( <u>28</u> )	26.70 <sup>4</sup>
Soluble lignin <sup>b</sup> (absorbance per 1 g./l.) at 230 µ	0.61
at 280 µ	0.91
Uronic anhydride ( <u>18</u> )	4.24
Methoxy1 ( <u>19</u> )	4.70
Acetyl ( <u>29</u> )	2.02
Pentosans ( <u>30</u> )	17.50
Total sugars (as glucose) ( <u>16</u> ) (Somogyi)	57.80

<sup>a</sup> Specimen pre-extracted with alcohol-benzene., Lignin corrected for ash.

<sup>b</sup> The filtrate from the Klason lignin determination was made up to a definite volume and the absorbance was determined spectrophotometrically. The filtrates were run against a sulfuric acid solution of comparable concentration to that of the sample of 230  $\mu$  and 280  $\mu$ . The value reported represents the absorbance of the solution from a 1-gram sample made up to one liter.

Purification was carried out by Soxhlet extractions, first with a mixture of ethanol:benzene (1:2) for 8 hours, then with 95% ethanol for 7 hours. Finally a 5-hour extraction was carried out with distilled water at a consistency of 2% at 23°C. using stainless steel containers and stirring frequently. The distilled water had a pH of 6.1 and the aqueous extract had a pH of 7.1. The extracted material was washed thoroughly with distilled water on a Buchner funnel and air dried. The air-dried, purified material was then chlorited for 5 hours according to the method of Wise, <u>et al. (12</u>). The holocellulose obtained was slightly yellowish in color, while retaining its fibrous structure. The percentage of extractives and chlorite holocellulose are reported in Table II. The chlorite holocellulose was analyzed for its chemical composition and the results are given in Table III.

#### TABLE II

## EXTRACTIVES AND CHLORITE HOLOCELLULOSE<sup>a</sup>

Extractives with benzene:ethanol	4.70
Extractives with 95% ethanol	2.22
Extractives with cold water	3.48
Chlorite holocellulose	65.99

<sup>a</sup> %, on basis o.d. bamboo shavings.

# TABLE III

# CHEMICAL ANALYSIS OF CHLORITE HOLOCELLULOSE<sup>a</sup>

Ash at 600°C. ( <u>21</u> )	2.07
Silica ( <u>22</u> )	0.58
Lignin [Klason] ( <u>28</u> )	0.70 <sup>b</sup>
Soluble <sup>C</sup> 230 µ	2.29
absorbance	
per 1 g./1. 280 µ	1.23
Alpha-cellulose ( <u>31</u> )	66.30 [Not corrected for ash or lignin]
Beta-cellulose ( <u>31</u> )	19.00
Gamma-cellulose ( <u>31</u> )	10.10
Methoxyl ( <u>19</u> )	1.42
Uronic anhydride ( <u>18</u> )	5.00
Polyoses [chromatography] ( <u>16</u> )	
Galactan	1.1
Glucan	63.9
Mannan	0.5
Araban	2.0
Xylan	23.9

<sup>a</sup> On basis o.d. material
<sup>b</sup> Specimen pre-extracted with alcohol-benzene. Lignin corrected for ash.
<sup>c</sup> See footnote Table I.

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## Extraction of the chlorite holocellulose

Duplicate 400 gram samples of the holocellulose were extracted with alkali under nitrogen (13). The samples were contained in sealed flasks and rotated at 1 r.p.m. at a controlled temperature of 23°C. The holocellulose was treated at 2% consistency for 24 hours with 3.4N potassium hydroxide (16.7% w/w), followed by a 24-hour extraction with 1.95N potassium hydroxide (10.0% w/w). The next extractions were at 5% consistency, once, for 2 hours and subsequently for 22 hours with alkaline potassium metaborate (223 gram potassium hydroxide and 120 gram boric acid per liter) followed finally at 1% consistency by one 24-hour extraction with 3.4N sodium hydroxide (12.0% w/w) and a 24-hour extraction with 1.9N sodium hydroxide (7.1% w/w). In each of these treatments the cellulose residues were always filtered on a Buchner type fritted-glass funnel in order to remove the extracted hemicelluloses. The cellulose residues were washed with a 5% potassium hydroxide solution or a 3% sodium hydroxide solution respectively after the second treatments with the individual extractant, then washed, neutralized with 10% acetic acid, washed free from acid, and air dried. Small samples of the cellulosic residues were removed for moisture determinations.

To precipitate any acid-insoluble hemicelluloses, the filtrates from the extractions were immediately acidified with glacial acetic acid while cooling the flask, then allowed to stand overnight or longer. From those extracts which yielded precipitates the acid-insoluble materials were separated. To these supernatant liquids and the remaining clear extracts 95% ethanol was added in a sufficient quantity to attain a final ethanol concentration of 70% by volume. After standing until clear, usually requiring 1 to 2 days, the precipitate which had formed in all cases was separated. These precipitates were washed with 80, 85, 90, and 95% ethanol, then solvent exchanged with hot absolute ethanol, methanol, and finally ethyl ether. All the hemicellulosic extracts were dried in a vacuum desiccator, over phosphorus pentoxide. The supernatant liquors containing the alcohol-soluble hemicelluloses were concentrated in a large flash evaporator. The concentrates obtained were then dialyzed by repeatedly concentrating the dialyzates. Finally the concentrates were freeze dried and the hemicelluloses recovered. Percentage losses of chlorite holocelluloses during alkaline extractions are given in Table IV. The yields of different hemicellulose extracts based on holocellulose and on bamboo shavings are recorded in Table V. The analytical results of the final residue are given in Table VI.

#### TABLE IV

## REMOVAL OF NONCELLULOSIC MATERIALS FROM CHLORITE HOLOCELLULOSE DURING SEQUENTIAL ALKALINE EXTRACTIONS<sup>a</sup>

	Removal, %	Yield Residual Material, %
Potassium hydroxide [Two extractions]	36.69	63.31
Aklaline potassium metaborate [Two extractions]	1.47	61.84
Sodium hydroxide [Two extractions]	2.64	59.20
Total -	40.80	

<sup>a</sup> Based on o.d. chlorite holocellulose.

# TABLE V

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# FRACTIONATION OF HOLOCELLULOSE RECOVERY OF EXTRACTS AND RESIDUAL FIBER

	On Basis Holo- cellulose, %	On Basis Bamboo Shavings, %
Potassium hydroxide extract		
Acid-insoluble (after 1st KOH treatment)	6.27	4.14
Acid-soluble, 70% ethanol insoluble	12.40	9.16
Acid-soluble, 70% ethanol soluble	0.63	0.41
Acid-insoluble (after 2nd KOH treatment)	0.20	0.12
Potassium metaborate extract		
Alcohol insoluble	0.02	0.01
Sodium hydroxide extract		
Acid-insoluble (after 1st NaOH treatment)	1.03	0.68
Acid-soluble, 70% ethanol insoluble ( " )	0.12	0.08
Acid-insoluble, (after 2nd NaOH treatment)	) 0.12	0.08
<u>Total</u> , recovered	20.79	14.68
Total, removed	40.80	26.93
Unaccounted, by difference	20.01	12.25
Residual fiber	59.20	39.07

#### TABLE VI

# CHEMICAL ANALYSIS OF FINAL RESIDUE<sup>a</sup>

Uronic anhydride	2.17
Alpha-cellulose	96.70
Beta-cellulose	1.60
Gamma-cellulose	0.30
Sugars (chromatography)	
Galactose	0.50
Glucose	95.80
Mannose	0.50
Arabinose	0.30
Xylose	1.30

<sup>a</sup> Yields % on basis of o.d. residue.

#### Characterization of the hemicellulose extracts.

(a) <u>Viscosity</u>: Cupriethylenediamine of 1<u>M</u> concentration was used as solvent for the viscosity determinations. The measurements were made with an Ubbelohde viscometer. The degree of polymerization was calculated by the formula D.P. = 470 [T]. This constant is the average of four determinations on highly purified methylglucuronoxylans, glucuronoarabinoxylan and arabinoxylan of spruce and elm recently reported by Swenson (<u>14</u>). The assumption of a linear relationship is not quite certain, nor is the value of the factor precisely known, but the above value is probably more correct than one based on cellulose. For comparison, Goring (<u>15</u>) has reported values averaging about 580 for similar hemicelluloses.

(b) <u>Specific rotation</u>: The optical rotations were determined in 10% sodium hydroxide solutions at concentrations of around 2%. The readings were taken in a 1 dm. tube at a temperature between 25 and 30°C., using a Bausch & Lomb saccharimeter. The hemicellulose solutions were decolorized by the addition of Norit A and filtered over a pad of Celite and Fibra-Flo through a sintered-glass Buchner-type funnel. Because of the peptization of Norit A, the solutions which remained cloudy had to be centrifuged at 20,000 g for 30 minutes.

(c) <u>Chromatographic qualitative and quantitative estimation of</u> <u>sugars</u>: The Forest Products Laboratory method (<u>16</u>) was followed. Whatman No. 1 filter paper for chromatography was used. The developing agent consisted of a mixture of ethyl acetate:pyridine:water in the proportions 8:2:1 by volume. The sugars were detected in the qualitative tests with the spray reagent of Timell <u>et al</u>. (<u>17</u>) and in the quantitative estimations with aniline hydrogen phthalate. A Beckman DU spectrophotometer was used for the optical density measurements.

The results of the measurements of the viscosity and specific rotation; and sugars estimations are recorded in Table VII.

## TABLE VII

# VISCOSITY, DEGREE OF POLYMERIZATION, SPECIFIC ROTATION, AND COMPOSITION OF HEMICELLULOSE EXTRACTS

Extract:	Limiting Viscosity Number []]	Degree of Polymerization	Specific Rotation	Composition of Extract, % <sup>a</sup>
Potassium hydroxide: Acid-insoluble (after lst KOH extraction)	0.95	446	$[\alpha]_{\rm D}^{26} = -64.7$	Uronic anhydride 6.43 Methoxyl 0.70 Glucan <sup>b</sup> Mannan 1.6 Galactan <sup>b</sup> Araban 13.5 Xylan 79.7
Acid-soluble, 70% ethanol insoluble	0.69	324	$\left[\alpha\right]_{\rm D}^{29} = -26.5$	Uronic anhydride 9.84 Methoxyl 3.56 Glucan 0.8 Mannan 1.6 Galactan 0.6 Araban 6.9 Xylan 78.8
Acid insoluble	0.48	225		
(after 2nd KOH extraction)				
<u>Sodium hydroxide:</u> Acid-insoluble	0.82	385	$\left[\alpha\right]_{D}^{29} = -8.7$	
(after 1st NaOH extraction)				
Acid-insoluble, 70% ethanol insoluble	0.66	310		
Acid-insoluble	0.67	315		
(after 2nd NaOH extraction)				

<sup>a</sup> On basis o.d. extract.

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<sup>b</sup> Qualitative analysis with Timell's spray reagent (<u>17</u>) showed traces of this sugar.

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(d) <u>Determination of the uronic anhydride and methoxyl group</u> <u>content</u>: The uronic acids were determined by decomposition with 12% hydrochloric acid and gravimetrically estimating the amount of carbon dioxide formed (<u>18</u>). A modified Zeisel method was used for the determination of the methoxyl groups whereby the alkyl iodide is collected in an acetic acid solution of potassium acetate to which bromine is added (<u>19</u>).

(e) <u>X-ray diffraction data</u>: X-ray diffractograms of the hemicellulosic extracts and the cellulosic materials were taken by means of a Norelco para focused diffractometer, with a wide angle goniometer. Nickelfiltered copper x-ray radiation ( $\lambda = 1.5418$  A) was used. The tube was run at 35 kv. and 20 ma. using 1/4° divergence and scattering slits, and a 0.006 inch receiving slit. A powder technique was employed. The x-ray diffraction data are recorded in Table VIII. The index of molecular order is calculated at the indicated values of 20 by the method of Gillespie, <u>et al.</u> (<u>20</u>). Figures 1 and 2 show the x-ray diagrams of some of the extracts.

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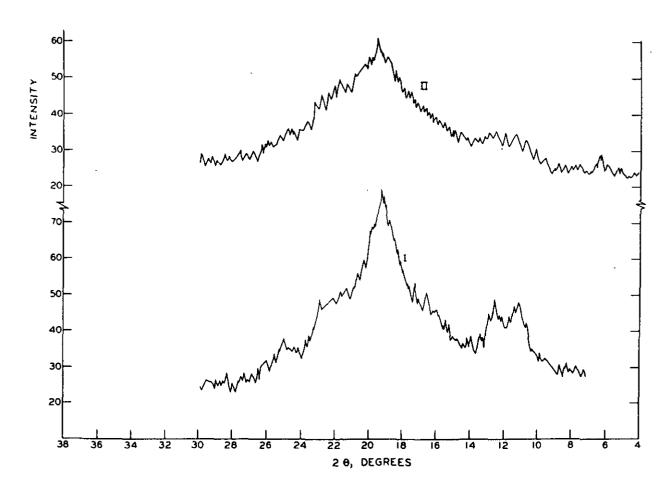
# TABLE VIII

# X-RAY DIFFRACTION DATA

	20 Degree	Relative Intensity	Width at Half Height, Degree	d, Å.	Index of Molecular Order, %
Hemicellulose extract, acid- insoluble, after lst KOH					
extraction:	19.2 11.0	55.5 41.5	5.56	4.62 8.03	55 
	12.6	43.0	4.30	7.00	
Hemicellulose extract, acid- soluble, in 70% ethanol-					
insoluble	19.6	43.8	4.38	4.53	52
Hemicellulose extract, acid- insoluble, after first NaOH extraction	20.2	71.0	7.10	4.4	62
Hemicellulose extract, acid- soluble, in 70% ethanol					
insoluble (from above)	20.4	77.0	7.7	4.4	38
Chlorite Holocellulose	23.0 15.4	69.0 53.5	6.90 5.35	3.86 5.70	82
Final residue	22.0 20.4 12.4	44.3 42.0 17.5	4.44 4.20 1.75	4.00 4.35 7.10	77 

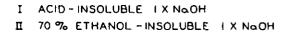
I ACID - INSOLUBLE I X KOH

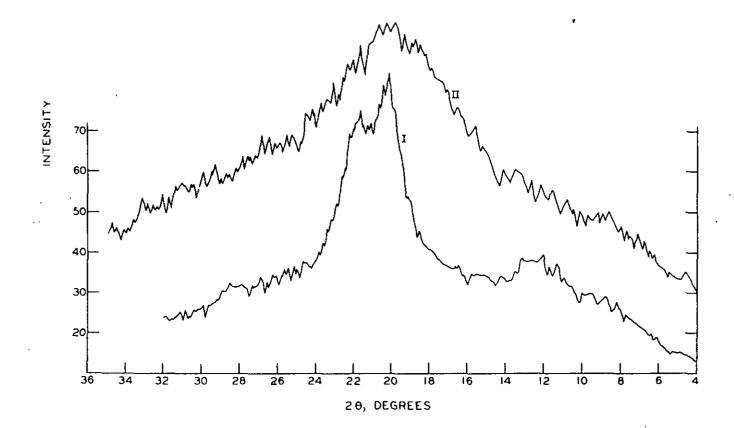
1 70 % FTHANOL - INSOLUBLE I X KOH



Fi	gure	1
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X-Ray Diffractogram of Bamboo Hemicelluloses Extracted with 3.4<u>y</u> Petassium Hydroxide (<u>D. strictus</u>)







X-Ray Diffractogram of Bamboo Hemicelluloses Extracted with 3.4<u>N</u> Sodium Hydroxide (<u>D. strictus</u>)

(f) <u>Electron microscopy</u>: The hemicellulosic extracts were also investigated by electron microscopy. A dry sample was sprinkled on a collodion grid. Then the greater part was blown off leaving only the small particles on the grid. Each of the grids was shadowed with palladium at an angle of 30° and viewed in an R.C.A. electron microscope, Type EMU - 3F, operated at 50 kv. at the magnifications of 1650x, 2340x, and 11,500x. Figures 3 and 4 show typical electron micrographs of the potassium hydroxide-extracted materials.

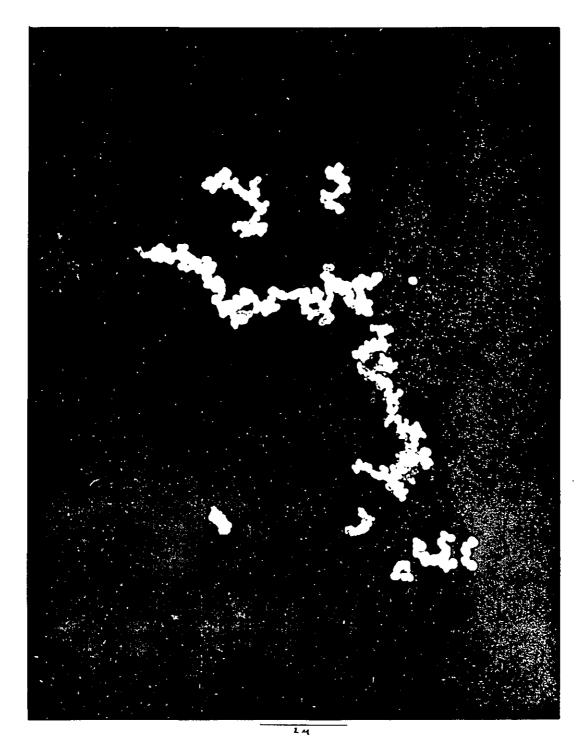


Figure 3

Electron Micrograph of Bamboo Hemicellulose Extract (After first KOH-extraction) <u>D. strictus</u>

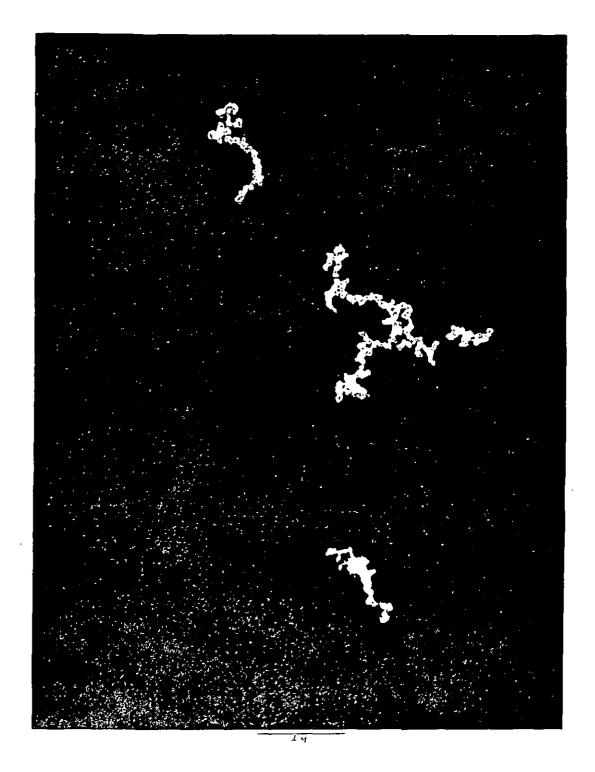


Figure 4

Electron Micrograph of Bamboo Hemicellulose Extract (After first KOH-extraction) <u>D. strictus</u> In conclusion it can be said that the potassium hydroxide extractions of the holocellulose removed 90% of the hemicelluloses, whereas only 3.6%, and 6.4% were removed by the borate and sodium hydroxide extractions respectively, however, only about 51% of the removed extracts were actually isolated. The composition of the acid-insoluble and the acid-soluble potassium hydroxide-extracted hemicelluloses varies somewhat. The former has a higher portion of arabans, a xylan:uronic anhydride ratio of 12.5:1 and the methoxyl group content of 0.70%. The latter shows less araban, a ratio of 8:1 and a higher methoxyl group content of 1.38%. It can be stated by analogy with hardwood hemicelluloses that both these fractions are glucuronoarabinoxylans; however more work would be required to verify this statement and to gain ultimate knowledge of the structure of these extracts. The acid-insoluble extract has a higher D.P. and is of higher molecular order, as shown by the x-ray diffractograms.

As expected from the low mannose content of the holocellulose, the borate extract was negligible. In fact, the small amount of material extracted by sodium hydroxide may be identical or very similar to the potassium hydroxide extract except for accessibility.

#### B. EVALUATION OF THE DISSOLVING PULPS

#### Experimental

In preparing the dissolving pulp, the culms were cut into chips about 2.5 cm. in length. These chips then were screened to remove material passing a 4-mesh screen. They were then prehydrolyzed with six parts water in which the temperature was raised from room temperature to 162°C. over 1-1/2 hours, then held at 162°C. for two hours. The pH at the end of the treatment was 4.0. The hydrolyzed chips were then given a sulfate cook, using 24% of the chip weight as total alkali and a sulfidity of 33%. The chip-liquor ratio was 1 to 4 and the cooking conditions were 6.5 hours at 162°C., including 1-1/2 hours to reach cooking temperature.

For bleaching, the unbleached pulp was treated at 3% consistency at 35°C. with 4% chlorine (based upon the o.d. weight of the unbleached pulp) for 15 minutes. After this period, the pH of the stock was raised to about 10 by the addition of caustic soda and the bleaching was continued for an additional 45 minutes. After this stage the pulp was thoroughly washed with water and then subjected to an alkali treatment with sodium hydroxide (2% on o.d. weight of the pulp) in 3% consistency at 50°C. for one hour. After washing the pulp was bleached with a sodium hypochlorite solution containing 1.5% available chlorine (on the o.d. weight of the unbleached pulp) at 5% consistency. The pulp was again washed with water and finally bleached with sodium chlorite and acetic acid solution containing 0.5% available chlorine. The pulp was washed and made into sheets.

Sheeted pulp, 8 inch by 8 inch in size (1600 gram total) were prepared. This quantity was insufficient for a complete evaluation. Evaluation was done, as far as the amount of sheets permitted, through the courtesy of the American Viscose Corporation. A commerical film grade pulp was used as a control. Viscose and spinning specifications are itemized in Table IX. The following discussion of the results is taken from the report of the American Viscose Corporation.

# TABLE IX

#### VISCOSE AND SPINNING SPECIFICATIONS

1. Viscose Specifications 18% NaOH-hemi free Preparation Steep 1 hr. at 25°C. Press ratio 2.8 1 hr. at 30°C. Shred Alkali cellulose ageing 20 hr. at 36°C. C-pulp 20 hr. at 26°C. B-pulp Xanthation 85 min. at 29°C. Mixing 2 hr. at 16°C. Viscose ripening 20 hr. at 18°C. Composition % Cellulose 8.0 % NaOH 6.5 % CS2, based on cellulose 28 Spinning viscosity (ball fall sec.) 40 Spinning salt test 4.5 2. Spinning Specifications Spinneret 40 holes - 0.0025 in hole diameter <sup>% н</sup>2<sup>so</sup>4 Spin bath composition - 9.0 % ZnSO<sub>4</sub> - 3.0 % Na<sub>2</sub>SO<sub>4</sub> -16.0 45°C. Spin bath temperature Immersion length 10 inches Godet stretch 40% Spinning speed 72 meters per minute Denier/filament 125/40

# Viscose

The pulp analysis for the bamboo pulp (B-pulp) was received from the supplier and is compared with the control (C-pulp) analysis in Table X. Major differences here are the alpha-cellulose concentration and the inorganic impurities, especially the silica.

## TABLE X

# PULP ANALYSIS

Pulp	B-	C
Alpha-cellulose, %	90.1	93.0
Beta-cellulose, %		3.0
Gamma-cellulose, %		3.5
Pentosans, %	3.52	1.0
D.P.	1026	1030
Ash , p.p.m.	1100	, 600
Fe , p.p.m.	48	20
Mn , p.p.m.	< 2	
Co , p.p.m.	< 0.3	
Si , p.p.m.	280	20

Visually, the B-pulp appeared as white as the commerical C-pulp. However, B-pulp was approximately one-half the density of the C-pulp. Twice the number of sheets were required of B-pulp to yield the same weight as C-pulp. During <u>steeping</u>, the B-pulp floated, because of the low pulp density.

It was further noted that the B-pulp steep liquor darkened considerably more than did the C-pulp liquor.

<u>Shredding</u> of both pulps was poor because of an unavoidable shredder condition. The recently plastic-coated shredder blades and bed plate are apparently too smooth to macerate the pulp effectively. Hard, unopened crumbs were still present. This may have contributed to the poor filterability discussed later.

<u>Alkali cellulose ageing</u> for 20 hours at 36°C. for the first trial with B-pulp yielded a low viscosity (16 sec.). The temperature was adjusted to 26°C. and viscosity was in the desired vicinity of 40 seconds.

<u>Xanthation</u> was normal, however B-pulp crumbs were darker in color than the crumbs of the C-pulp xanthate.

<u>Mixing</u> too was normal, however, the dark color of the B-pulp xanthate crumbs persisted throughout the mixing process.

<u>Viscose ripening</u> intensified the dark brown color of the B-pulp solution. There was no haze or muddiness associated with either viscose.

<u>Viscose compositions</u> were consistent. The cellulose and sodium hydroxide concentrations are recorded in Table XI.

The change in viscosity and salt test with time at 18°C. is recorded in Table XII for the second viscose trial. The data indicate that B-pulp ripens at a slower rate than does C-pulp.

## TABLE XI

## VISCOSE COMPOSITION

Viscose	Cellulose, %	NaOH, %
B-pulp -1	7.71	6.38
-2	7.82	6.40
C-pulp -1	7.83	6.40
-2	7.73	6.37

TABLE XII

#### VISCOSE

## VISCOSITY AND SALT TEST

Ripening Time of Viscose, Hr.	Viscosi Falling Ba B-2		Salt B-2	Test C-2
0	43	44	8.2	6.1
21	36	39	6.0	4.3
26	36	39	5.4	3.9
45	36	39	3.9	2.8
50	36	38	3.7	2,6

<u>Filterability</u> of all viscoses were too poor to be measured on the constant-rate-of-delivery tester. The B-pulp viscoses developed 100 pounds per square inch back pressure within 15 minutes. C-pulp viscoses lasted for 45 minutes. This test is dependent on maintaining less than 100 pounds per square inch pressure for 2 hours. Ordinarily the C-pulp gives good filterability. The poor shredding of the pulps, as noted earlier, may have contributed to this condition.

A rough comparison of the poorer filterability of B-pulp could be noted by its plugging of the standard-plate filter, which did not occur with the commercial C-pulp. This filtration was carried out at constant pressure.

A comparison of the particle count in the viscose is given in Table XIII. The results confirm the filtration experience. The count was obtained by means of the Coulter counter.

<u>Spinning</u> proceeded well. During spinning for 6 hours, no obvious differences between the viscoses were observed. Tensile properties of the yarns spun are given in Table XIV. Although tenacities are equivalent, the lower extensibilites for the B-pulp indicate the yarn may have been overstretched. The lower ratio of wet to condition elongation for B-pulp indicates a condition of excessive strain.

# TABLE XIII

# PARTICLE COUNT IN VISCOSE

Particle Diameter (µ)	No. o: 4	f Particles 8	in T 16	housands <sup>a</sup> 32
<b>B - 1</b> .	553	124	48	5
в-2	445	76	24	2
C-1	214	43	7	0.7
C-2	185	37	4	0.3

<sup>a</sup> Number of particles greater than indicated micron size.

#### TABLE XIV

## TENSILE PROPERTIES OF YARNS SPUN

No.	Denier	Tenacity g./Denier (Conditioned)	Elongation, % (Conditioned)	Tenacity, g./Denier (Wet)	Elongation, % (Wet)
<b>B-1</b>	122	1.94	13.8	0.89	12.2
B-2	134	1.96	16.0	1.02	19.0
C-1	126	. 1.97	15.1	1.00	16.0
C-2	136	2.18	17.1	1.10	20.3

# Conclusions

B-pulp is potentially commercial.

Viscose processing presented no undue difficulties other than filtration. Viscose filtration data show that improvement is necessary. Spinning was normal. Yarn elongations indicate that spinning conditions were not the best and/or the pulp might possibly have had too high a content of low D.P. material.

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