Production of dissolving pulp from recovered paper using enzymes

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ABSTRACT

This preliminary study suggests that pulps with chemical properties similar to a dissolving pulp can be produced from once-dried, commercially-available, bleached hardwood kraft fiber and from high quality recovered paper rich in hardwood fiber. A sequence employing an initial cold alkali extraction, xylanase treatment, and a second cold alkali extraction can effectively remove hemicelluloses and produce pulps with high alpha-cellulose content and acceptable viscosity.

Application:

High quality recovered paper sources rich in hardwood content can provide a pulp with properties similar to a chemical cellulose.

MOST CHEMICAL CELLULOSE or dissolving pulp comes from wood using the prehydrolysis kraft or acid sulfite processes *(1)*. Due to increasing public interest in the recycling of paper products, recovered paper may be a potential new source of chemical cellulose. The following investigation evaluated the feasibility of deriving a chemical cellulose from once-dried, commercially-available, bleached hardwood kraft fiber and two sources of unprinted, bleached recovered waste using a combination of xylanase treatment and cold alkali extraction.

PREVIOUS WORK

Commercially available dissolving pulps, also termed chemical cellulose, have a high cellulose content of 90–98%, low hemicellulose content, very little residual lignin, low extractive and mineral content, high brightness, and a uniform molecular weight distribution *(1)*. The two major methods to produce pulps with these properties are the acid sulfite and the prehydrolysis kraft pulping processes. The sulfite dissolving pulp process uses a higher temperature and higher acidity compared with cooking conditions for producing paper products. The objective is to degrade lignin and hemicelluloses to enhance their removal by hot alkali extraction during the bleaching sequence. The prehydrolysis kraft process applies an acidic pretreatment before the alkaline pulping stage to degrade the hemicelluloses allowing easier removal during the cook. Cold alkali extraction in the subsequent bleaching sequence can remove residual hemicelluloses to obtain pulp grades for high quality end uses. In softwoods, cold alkali extraction of prehydrolysis kraft pulps can produce pulps with mannan and xylan levels of approximately 1%. Hardwood pulps can reach mannan and xylan levels of 0.5 and 1%, respectively. In contrast, the conventional kraft process stabilizes residual hemicelluloses against further alkali attack and therefore prevents the production of acceptable quality dissolving pulps through treatment in the bleach plant.

Cold alkali extraction of softwood kraft pulps has historically produced nitration grade pulps *(1)*. Such dissolving grade pulps were produced in Sweden and Australia as early as the 1930s and during World War II *(2)*. Other work *(1, 3)* has examined the conversion of bleached radiata pine kraft pulps and radiata pine bisulfite pulps to chemical cellulose. Both research groups reported that cold alkali extraction of the bleached pulps with 8–10% NaOH for 1 h at 20°C gave products with satisfactory chemical properties for nitration grade cellulose. This approach is not applicable to hardwood pulps due to the presence of a resistant xylan fraction that remains after alkaline extraction *(2, 4)*.

During the past several years, some studies have focused on removing or modifying hemicellulose in bleached chemical fiber with xylanases. Paice and Jurasek *(5)* investigated the removal of xylan from bleached hardwood sulfite pulp with a xylanase preparation from *Schizophyllum commune*. They reported a decrease from 4.0% to 3.6% pentosan after 24 h. The xylanase was not very effective in removing xylan from the bleached fiber. Senior *et al. (6)* performed a similar study using bleached kraft hardwood pulp and a xylanase from *Trichoderma harzianum*. They observed a 54% decrease in the xylan content of the pulp after 24 h, but xylan levels remained quite high in the final product—11.4%. The bleaching process apparently removed the more accessible hemicelluloses, or the modification of the hemicelluloses during pulping produced polysaccharides the enzyme did not recognize. Christov and Prior *(7, 8)* studied xylan removal from sulfite dissolving pulp using crude xylanase from *Aureobasidium pullulans* to provide a more complete removal of xylan. They observed a limit of 31% xylan removal with a minimum of 1.81% pentosans with high enzyme charges—1500 U/g. They attributed this limit to a lack of enzyme accessibility to xylan in the pulp.

Mora *et al. (9)* and Noe *et al. (10)* studied the modification of chemical fibers with xylanases. Mora *et al. (8)* suggested that xylans are more widely hydrolyzed in the fiber cell wall than

Sample no.	Sequence	Treatment levels Fiber	NaOH
1	-	-	-
2	+	-	-
3	-	+	-
4	+	+	-
5	-	-	+
6	+	-	+
7	-	+	+
8	+	+	+

Sequence: (-) XE Xylanase charge: 2500 U/g o.d. fiber; 24 h; (+) EXE where X is xylanase treatment, and E is extraction
Fiber: (-) Unrefined, all fiber fractions; (+) Refined, fibers > 100 mesh
NaOH: (-) 8%; (+) 10%

I. Factorial design at two levels for once-dried bleached hardwood kraft fiber

suggested by the hydrolysis rate based upon the release of soluble sugars. The sugar products do not account for random xylan hydrolysis in the fiber cell wall where the polysaccharide is physically retained through hydrogen bonding. Noe *et al. (10)* extended the study to determine if xylan modifications in the fiber cell wall by xylanases affected paper properties. They reported that bleached kraft birch wood pulp and bleached sulfite spruce pulp demonstrated enhanced beatability and fiber flexibility with a reduction of fiber intrinsic strength due to hydrolysis of xylan. These modifications occurred with only a slight yield loss. Roberts *et al. (11)* reported similar findings. Xylanase treatment of bleached kraft birch wood pulp resulted in a 25–30% decrease in burst strength and breaking length with a slight loss of 4% in zero span breaking length. Xylan removal was 20%. The low level of xylan removal was due to poor enzyme accessibility to xylan in fiber pores.

The objective of this research was to determine if a chemical cellulose can be produced from high quality recovered paper using a combination of xylanase treatment and cold alkali extraction. Fiber sources included once-dried, commercially-available bleached hardwood kraft pulp, unprinted white envelope clippings, and unprinted white ledger paper. The working hypothesis was that xylanase treatment may enhance the extractability of xylan from the fiber during cold alkali extraction to allow the production of a chemical cellulose. The targets for the chemical cellulose *(1)* included R_{10}—resistance to extraction by 10% NaOH—greater than 97%; xylan and mannan levels of approximately 2% and 1%, respectively; ash content of less than 0.1%; and a minimum cuene viscosity of 8.5 cp.

METHODS AND MATERIALS

Preparation of fiber

Bleached hardwood kraft pulp (Federal Paper Board Company, Inc., Riegelwood, NC) was obtained in air-dry sheets (7% moisture, 0.01% ash). The pulp was washed in distilled, deionized water, centrifuged, and fluffed. It was dried at 105°C according to the procedure of Bhat *et al. (12)*. The pulp therefore had characteristics similar to recycled fiber *(13, 14)*. Some pulp was beaten to 200-mL CSF (TAPPI T 200). Unprinted, white envelope clippings (4.5% ash) and unprinted, white ledger paper (11.0% ash) were disintegrated (TAPPI T 205). The beaten kraft hardwood fiber and two recovered papers were then fractionated with a Bauer-McNett classifier (TAPPI T 233) to remove fines or fillers. The fractions larger than 100 mesh were collected, mixed, centrifuged, and fluffed. Yields after fractionation and removal of the fines or filler for the hardwood, envelope, and ledger fiber sources were 60%, 77%, and 58%, respectively. Ash contents for the washed envelope and ledger fiber were 0.3% and 0.01%, respectively. The removal of fines served two purposes. It eliminated a variable that could cause a loss of R_{10} due to the damaged fiber particles, and it allowed increased availability of the xylanase to the fiber surfaces *(15)*.

Xylanase treatment

The xylanase preparation used in the study was Irgazyme 40S, a commercial blend of xylanases marketed for pulp bleaching (Ciba-Geigy, Greensboro, NC). Activity on oat spelt xylan was 7000 ± 300 U/mL *(16)*. The xylanase was charged on the basis of units of activity per gram o.d. fiber. The reaction conditions were 3% pulp consistency, pH 7.5, and 50°C. Reaction time varied according to the experimental design.

Cold alkali extraction

Cold alkali extraction was performed with either 8% or 10% NaOH at 20°C for 1 h. Pulp consistency was 3% to allow complete dispersion of the fiber in the caustic solution. Extracted pulp was washed with distilled water until the filtrate pH was neutral. The pulp was then washed with water adjusted to pH 3.0 with sulfuric acid to remove metal ions *(1, 2)*.

Carbohydrate analysis

Pulps were analyzed for glucose, xylose, and mannose by high pressure liquid chromatography after acid hydrolysis according to the method of Laver and Wilson *(17)*.

Alkali solubility at 25°C

Alkali solubility of the pulp samples was tested according to TAPPI T 235.

Cuene viscosity

Pulp viscosity was measured according to TAPPI T 230.

Ash content

The ash content was measured according to TAPPI T 211.

EXPERIMENTAL DESIGN FOR ONCE-DRIED BLEACHED HARDWOOD KRAFT FIBER

To investigate the feasibility of producing a chemical cellulose from once-dried, bleached hardwood kraft fiber, data were collected with a duplicated 2^3 factorial experimental design shown

Fiber + treatment	Glc, %	Xyl, %	Man, %	Visc., cp	R_{10}, %	S_{10}-S_{18}, %	S_{18}, %	Yield
Unrefined BHWK	83.3	16.5	0.2	19.7	91.0	3.3	5.7	100
+ Xylanase	84.9	14.9	0.2	12.9	88.7	4.6	6.7	96.7
+ 8% NaOH	89.9	9.8	0.4	23.0	98.0	0.9	1.1	87.5
+ 10% NaOH	89.3	10.4	0.3	21.3	98.5	0.4	1.1	88.7
Refined BHWK,>100 mesh	84.1	15.6	0.3	22.6	90.4	2.8	7.0	100
+ Xylanase	85.9	13.8	0.3	10.8	87.7	4.1	8.3	98.2
+ 8% NaOH	91.7	7.9	0.4	25.2	98.1	0.6	1.3	89.2
+10% NaOH	90.5	9.2	0.3	23.8	98.5	0.5	1.0	90.8

Glc: glycose; Xyl: xylose: Man: mannose
BHWK: Once -dried bleached hardwood kraft fiber
Xylanase: Xylanase treatment with 2500U/g o.d. fiber for 24 h
8% or 10% NaOH: Cold alkali extraction performed once on each extraction control sample

II. Chemical analysis of controls for once-dried hardwood kraft fiber

Sample No.	Glc,%	Xyl, %	Man, %	Visc., cp	R_{10}, %	S_{10}-S_{18}, %	S_{18}, %	Yield
1	92.8	6.8	0.4	17.1	98.1	0.8	1.2	84.2
2	97.0	2.5	0.5	4.3	96.1	2.5	1.4	75.2
3	94.9	4.7	0.4	11.2	97.7	1.0	1.4	84.3
4	97.4	2.1	0.5	4.0	95.4	3.1	1.6	78.2
5	91.6	8.1	0.3	16.0	98.8	0.4	0.8	82.9
6	97.8	1.8	0.4	3.8	98.0	0.9	1.2	71.2
7	93.6	6.1	0.3	14.0	98.7	0.4	1.0	84.5
8	98.2	1.5	0.3	3.6	98.2	0.9	1.0	72.5

See Table I for experimental details

III. Chemical analysis of treated samples of once-dried bleached hardwood kraft fiber

in **Table I** that evaluated the variables of treatment sequence, fiber refining, and alkali concentration *(18, 19)*. The two treatment sequences tested the effect of an alkali extraction stage before the xylanase treatment. The refining variable studied the effect of fibrillating the fiber to increase xylanase accessibility to the xylan. The two alkali concentrations bracketed those used in the dissolving pulp industry *(1)*. **Table II** shows that several controls were run besides the experimental design to characterize the effects of the extractions and xylanase treatments alone on pulp properties. The enzyme charge remained constant at 2500 U/g o.d. fiber. Reaction time was 24 h. The xylanase was initially applied in large excess to produce an observable effect. Subsequent experimental trials involved decreasing the enzyme charge and reaction time.

RESULTS FROM FACTORIAL DESIGN FOR HARDWOOD FIBER CARBOHYDRATE ANALYSIS

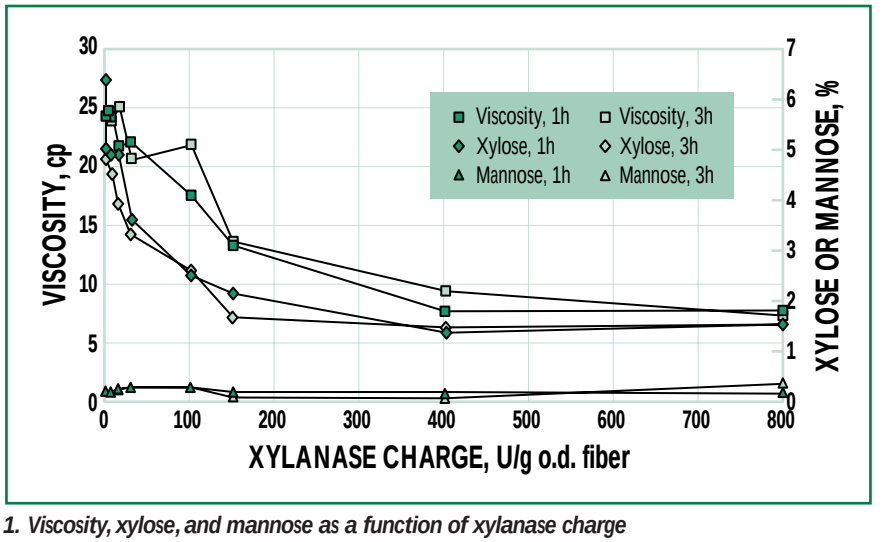
Tables II and III report xylose and mannose contents for the control samples and treated samples, respectively. It is obvious that target levels of xylose and mannose contents may be obtained with a sequence abbreviated EXE employing an initial cold alkali extraction (E), a xylanase treatment (X), and a final cold alkali extraction (E). The controls and samples treated with the sequence XE did not reach the targeted xylan level. A minimum xylose content of 4.7% was observed. Two interactions—a sequence by fiber type interaction and a sequence by NaOH concentration interaction—were significant at the 95% confidence level as **Table IV** shows. An extraction stage before the xylanase treatment decreased the xylose content 3.6% for refined and washed fiber. The unrefined fiber showed a decrease in xylose content of 5.3%. Although the refined and washed fiber produced lower xylose contents compared with the unrefined fiber, the fiber type variable was inconsequential. Including an extraction stage before the xylanase treatment using 10% NaOH resulted in a 5.5% decrease in xylose content. For the 8% NaOH, the decrease was 3.5%. An initial extraction stage before xylanase treatment therefore seems necessary for effective xylan removal. The level of 10% NaOH was more effective than 8% NaOH.

Viscosity

The xylanase treatment of the hardwood fiber resulted in severe viscosity reductions for the controls as **Table II** shows and all treated samples as **Table III** shows. Xylanase treatment of the controls resulted in reductions of pulp viscosity of up to 50%. Extraction before xylanase treatment resulted in a loss of viscosity of approximately 10 cp compared with the XE sequence as **Tables III and IV** show. The sequence EXE clearly produced unacceptable viscosity although producing acceptable carbohydrate results.

Average	Xyl, % 4.2 ± 0.01	Man, % 0.4 ± 0.0	Visc., cp 9.2±0.01	R_{10}, % 97.6 ± 0.04	S_{10}-S_{18}, % 1.2 ± 0.04	S_{18}, % 1.2 ± 0.01	Yield, % 79.1 ± 0.1
Main effects:							
Sequence	-4.4	0.1	-10.7	-1.4	1.2	0.2	-9.7
Fiber	-1.2	0.0	-2.1	-0.3	0.2	0.1	1.5
NaOH concentration	0.4	-0.1	0.2	-1.6	-1.2	-0.4	-2.7
Second order interactions:							
Sequence/fiber	0.9	0.0	1.9	0.0	0.1	-0.1	0.6
Sequence/NaOH conc.	-1.0	0.0	-0.7	0.7	-0.7	0.0	-2.2
Fiber/NaOH concentration	0.1	-0.1	1.0	0.3	-0.2	-0.1	-0.1
Std. error for effects:	0.03	0.01	0.01	0.08	0.07	0.03	0.2

IV. Estimated effects on chemical properties (±standard error) for the once-dried bleached hardwood kraft fiber



1. Viscosity, xylose, and mannose as a function of xylanase charge

Alkali solubility at 25°C

Tables II and III show that cold alkali extraction can produce fiber with R_{10} above 97%. **Table IV** shows that the variables of sequence and NaOH concentration were involved in a significant—95% confidence level—second order interaction. For the 10% NaOH samples, including the cold alkali extraction stage before the xylanases treatment resulted in a decrease of 0.65% in R_{10}. For the 8% NaOH, a decrease of 2.2% occurred. As expected, the 10% NaOH was therefore more efficient at extracting degraded carbohydrates. The EXE sequence required the 10% NaOH concentration to produce pulps with R_{10} above 98%. EXE samples using the 8% NaOH produced pulps with R_{10} below 97%. As expected, opposite trends occurred for the S_{18} and S_{10} - S_{18} values (S refers to solubility in NaOH.). The S_{18} values indicate carbohydrate chains with a degree of polymerization of less than 50 *(1)* that consist mainly of hemicelluloses. S_{10} - S_{18} values indicate the content of degraded cellulose with a degree of polymerization between approximately 50 and 150 *(1)*. The 10% NaOH reduced the S_{18} more effectively than 8% NaOH. This effect is consistent with the decrease in xylose content obtained with the 10% NaOH. S_{10} - S_{18} values for the EXE samples reflect the increase in degraded cellulose that may be responsible for the observed loss of viscosity. Note also that ash contents for each of the fiber samples was less than 0.01%.

Yield

Xylanase treatment combined with cold alkali extraction reduced the yield compared with the controls for all the treated samples. The average yield for the treated samples was 79%. Xylanase treatment before cold alkali extraction resulted in 3–6% decrease in yield over cold alkali extraction alone as **Tables II and III** show. Cold alkali extraction before xylanase treatment resulted in an additional loss of approximately 10% as **Tables III and IV** show. The 10% NaOH decreased the yield more than the 8% NaOH. It was more efficient at removing xylan.

RESULTS FOR VARYING THE ENZYME CONCENTRATION AND REACTION TIME

Using the findings above, a second series of samples was prepared using the EXE sequence, unrefined fiber, and 10% NaOH. Enzyme charges varied with reaction times of either 1 h or 3 h. A sequence of EE with 10% NaOH served as a control. The enzyme charges ranged from standard bleaching charges of approximately 1 U/g o.d. fiber up to 800 U/g o.d. fiber. Hopefully reducing the enzyme charge and reaction time could allow xylan removal with less cellulose damage.

Carbohydrate analysis and viscosity

Figure 1 shows xylose, mannose, and cuene viscosity levels. Xylanase charges of approximately 150 U/g o.d.

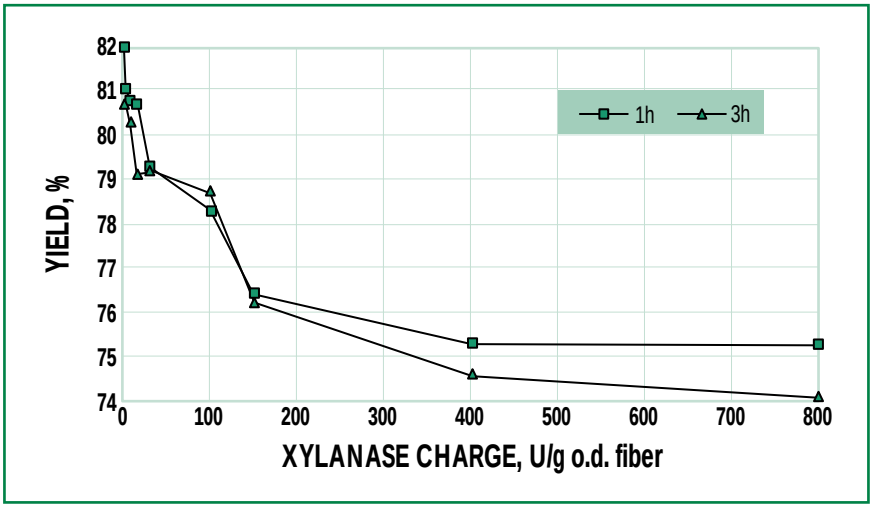
fiber resulted in xylose and mannose contents of approximately 2.0% and 0.3%, respectively, with cuene viscosities of approximately 13 cp. These values are within commercially acceptable limits. The xylanase treatment effectively enhanced the removal of xylan compared with the control at 6.4% xylose. There appeared to be little difference between the 1 h and 3 h reaction times.

Alkali solubility at 25°C

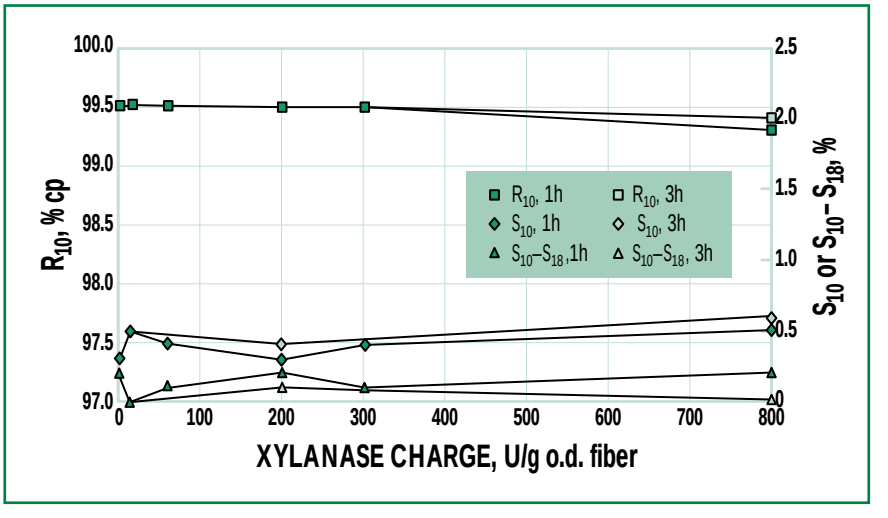
Figure 2 shows the various alkali solubilities as functions of xylanase charge and reaction time. The target values were easily obtained. It is apparent that very high R_{10} and low S_{18} and S_{10} - S_{18} values were obtainable with low enzyme charges and short reaction times. The treated pulp may therefore contain low amounts of degraded cellulose (degree of polymerization 50 –150) *(1)*.

Yield

Figure 3 shows yield for the various xylanase charges and the two reaction times. Extracting fiber twice with cold alkali but with no xylanase treatment resulted in a yield of 82%. Increasing the xylanase charge resulted in decreasing fiber yields. Yield appeared to level out at approximately 75% at the higher xylanase charges.



2. Alkali solubility as a function of xylanase charge



3. Fiber yield as a function of xylanase charge

EXPERIMENTAL DESIGN FOR UNPRINTED ENVELOPE CLIPPINGS AND WHITE LEDGER FIBER

Using the results obtained with the once-dried bleached hardwood kraft fiber, the feasibility of producing a chemical cellulose from unprinted white envelope clippings and unprinted white ledger paper was investigated with the 2^4 factorial design in **Table V**. It included the variables of fiber type, xylanase charge, xylanase reaction time, and NaOH concentration *(18)*. Several controls in **Table VI** were performed besides the experimental design to help characterize the effect of alkali extractions and xylanase treatments on the fiber.

RESULTS FOR THE FACTORIAL DESIGN FOR RECOVERED PAPER FIBER

Carbohydrate analysis

As **Table VI** shows, the recovered paper samples contained both hardwood and softwood fiber as indicated by the higher mannose content compared to the hardwood fiber in **Table II. Table VI** shows that successive cold alkali extractions of the recovered paper samples could not remove xylan effectively from the fiber. For envelope clippings, 2.4% xylose remained. The white ledger contained 3.8% xylose. The 10% NaOH was more effective at removing mannan compared with the 8% NaOH. The resulting mannose levels were between 1% and 2%.

Table VII reports the carbohydrate composition of the treated recovered paper samples. Samples 6, 12, 13, 14, and 16 had carbohydrate compositions that met target levels. Xylanase treatment be-tween the two extraction stages effectively lowered xylose levels to well below target values for all samples. Xylose content varied mostly with the fiber type as **Table VIII** shows. The white ledger appeared to have a more resistant xylan fraction than the envelope clippings. A significant interaction—95% confidence level—was observed be-tween reaction time and NaOH concentration. The effect of increasing the reaction time from 1 h to 3 h using 10% NaOH extraction increased the xylose content 0.43%. The reaction time increase for the 8% NaOH samples resulted in a decrease in xylose of 0.3%. The xylan

Fiber + treatment	Glc, %	Xyl, %	Man, %	Visc., cp	R_{10},%	S_{10}-S_{18}, %	S_{18}, %	Yield
Unrefined BHWK	84.3	12.2	3.5	12.3	86.2	2.3	11.6	100
+ Xylanase	86.0	10.4	3.6	12.3	86.3	3.0	10.7	95.6
+ 8% NaOH	94.8	2.5	2.7	13.5	98.3	-0.5	2.2	84.3
+ 10% NaOH	95.9	2.4	1.7	13.2	99.3	-0.1	0.8	80.7
Refined BHWK, >100 mesh	83.6	13.7	2.7	17.8	89.5	2.2	8.3	100
+ Xylanase	86.5	10.8	2.7	17.5	88.5	2.9	8.6	98.6
+ 8% NaOH	94.0	3.8	2.2	24.2	98.7	-0.4	1.7	85.5
+10% NaOH	95.2	3.8	1.1	23.7	99.4	-0.2	0.8	84.4

Envelope clippings and white ledger fiber washed through 100 mesh screen
Xylanase: Xylanase treatment with 200U/g o.d. fiber for 3 h
8% or 10% NaOH: Cold alkali extraction performed twice on each extraction control sample

VI Chemical analysis of controls for recovered paper fiber

Treatment levels Sample no.	Fiber	Xylanase	Reaction time	NaOH
1	-	-	-	-
2	+	-	-	-
3	-	+	-	-
4	+	+	-	-
5	-	-	+	-
6	+	-	+	-
7	-	+	+	-
8	+	+	+	-
9	-	-	-	+
10	+	-	-	+
11	-	+	-	+
12	+	+	-	+
13	-	-	+	+
14	+	-	+	+
15	-	+	+	+
16	+	+	+	+

Fiber: (-) Unprinted envelope clippings, > 100 mesh; (+) Unprinted white ledger, > 100 mesh
Xylanase: (-) 100 U/g o.d. fiber; (+) 200 U/g o.d. fiber
Reaction Time: (-) 1 h; (+)3 h
NaOH: (-) 8%; (+) 10%

V. Factorial design at two levels for envelope clippings and white ledger fiber

in the 8% NaOH extracted fiber was therefore effectively removed by xylanase. It appears that the remaining xylan in fiber extracted with 10% NaOH is resistant to cold alkali extraction and xylanase treatment. Mannose content appeared to vary mainly with the NaOH concentration. The 10% NaOH extraction was more effective at removing mannan. Levels approaching the target values were obtained.

KEYWORDS
Reclaimed fibers, xylanase, cold alkali extraction, dissolving pulps, waste papers, bleached pulps, hardwood pulps.

Viscosity

The average viscosity of the treated samples was 11.7 cp as **Tables VII** and **VII** show. The viscosities of the five samples noted above from **Table VII** exceeded the target viscosity. As with the hardwood fiber, cold alkali extraction before xylanase treatment resulted in a substantial decrease in viscosity compared with the extraction controls—approximately 13 cp and 24 cp for envelope clippings and white ledger, respectively. The envelope clippings showed a decrease of approximately 2-5 cp. A decrease of approximately 8-12 cp was observed for the white ledger. Four factors were involved in significant—95% confidence level—second order interactions. The behavior of the viscosity with the factor levels illustrates the sensitivity of the pulp degradation to the reaction conditions. Such results warrant further investigation into the purity of the enzyme preparations and to characterize further potential xylanases for such treatments of bleached kraft fiber.

Alkali solubility at 25°C

The target values for R_{10} were easily attained as **Table VII** shows. **Table VIII** shows that the NaOH concentration was the only variable dramatically affecting alkali solubility. The xylanase treatments showed no significant effect. This was true also for the solubility in 18% NaOH. S_{10} - S_{18} values for the treated samples were all negative. The two extraction stages despite xylanase treatment therefore removed most of the alkali soluble material from the fiber.

Yield

Tables VI and VII also report the yields for the controls and treatments, respectively. Yields use washed fiber as noted above. Xylanase treatment combined with the two cold alkali extraction stages decreased the yield by approximately 2-3% compared with the respective controls. The average yield for the treated fiber was 81.1%. The white ledger fiber resulted in a higher yield of approximately 2.3% compared with the envelope clippings as **Table VIII** shows. As expected, increasing the NaOH concentration from 8% to 10% also reduced the yield approximately 2.4%.

Sample No.	Glc,%	Xyl, %	Man, %	Visc., cp	R_{10}, %	S_{10}-S_{18}, %	S_{18}, %	Yield
1	96.3	1.1	2.6	9.8	98.1	-0.1	2.0	82.2
2	96.0	1.7	2.4	13.6	98.5	-0.3	1.8	83.6
3	96.2	1.1	2.7	10.3	98.0	-0.1	2.1	81.5
4	96.6	1.4	2.0	12.9	98.4	-0.2	1.8	83.3
5	96.9	0.9	2.1	10.8	98.2	-0.2	2.0	81.1
6	97.7	1.1	1.2	16.2	98.5	-0.2	1.7	83.3
7	96.8	0.9	2.3	9.9	98.0	0.0	2.0	80.9
8	97.0	1.2	1.8	14.3	98.5	-0.2	1.7	82.5
9	98.0	0.6	1.4	9.7	99.2	-0.1	0.9	78.6
10	97.0	1.4	1.7	13.6	99.3	-0.3	1.0	81.8
11	98.1	0.6	1.4	8.7	99.3	-0.1	0.8	78.9
12	98.0	1.0	1.1	11.6	99.3	-0.1	0.8	81.6
13	97.9	0.8	1.3	9.5	99.3	-0.1	0.8	78.0
14	97.1	1.6	1.3	15.8	99.4	-0.1	0.7	80.9
15	95.8	1.5	2.7	8.3	99.3	-0.1	0.8	78.5
16	97.9	1.4	0.8	12.5	99.3	-0.3	1.0	80.8

See Table V for experimental details

VII. Chemical analysis of treated recovered paper samples

Average:	Xyl, % 1.1 ± 0.05	Man, % 1.8 ± 0.1	Visc., cp 11.7 ± 0.06	R_{10}, % 98.8 ± 0.02	S_{10}-S_{18}, % -0.2 ± 0.03	S_{18}, % 1.4 ± 0.03	Yield, % 81.1 ± 0.06
Main effects:							
Fiber	0.38	-0.55	4.10	0.23	-0.11	-0.11	2.26
Enzyme charge	0.00	0.06	-1.32	-0.05	0.04	0.01	-0.19
Reaction time	0.08	-0.21	0.89	0.05	0.01	-0.06	-0.69
NaOH concentration	-0.09	-0.68	-1.02	1.03	0.01	-1.04	-2.41
Second order interactions:							
Fiber/enzyme charge	-0.18	-0.33	-0.66	0.00	-0.01	0.01	-0.16
Fiber/reaction time	-0.10	-0.29	0.89	0.00	0.01	-0.01	-0.01
Fiber/NaOH concentration	0.07	-0.05	0.14	-0.18	0.01	0.16	0.51
Enzyme charge/reaction time	0.16	0.31	-0.51	-0.03	-0.04	0.06	0.04
Enzyme charge/NaOH concentration	0.03	-0.04	-0.56	0.05	-0.04	-0.01	0.31
Reaction time/NaOH concentration	0.36	0.37	-0.25	0.00	-0.01	0.01	0.01
Standard error for effects:	0.09	0.20	0.12	0.03	0.05	0.05	0.12

See Table V for experimental details

VIII. Estimated effects on chemical properties (± standard error) for recovered paper fiber

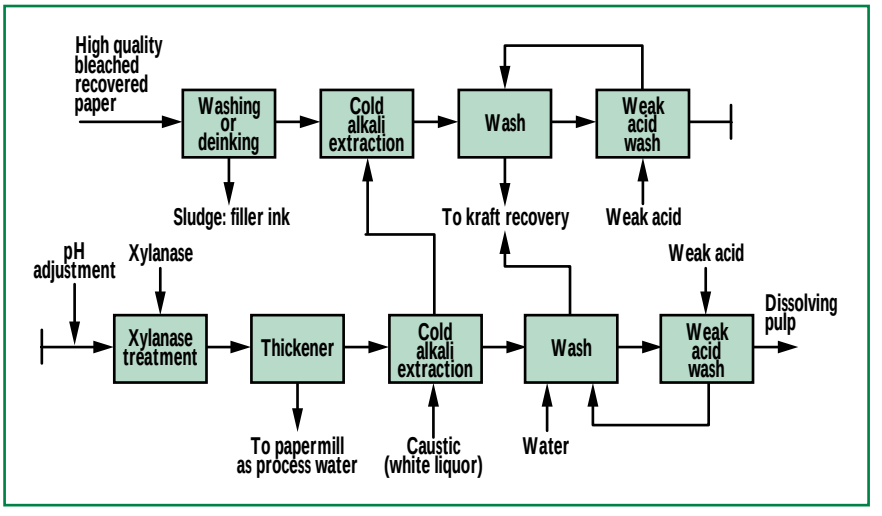
DISCUSSION

Experimental data

The two experimental designs and their respective controls for the hardwood fiber and the two recovered paper types indicated that a sequence involving cold alkali extraction, xylanase treatment, and a final cold alkali extraction can produce pulps with chemical compositions similar to that of a dissolving pulp. The xylanase stage was necessary to remove the xylan effectively. The initial cold alkali extraction enhanced the effect of the xylanase reaction. The swelling resulting from the initial extraction *(1)* may have allowed increased accessibility of the xylanase into the fiber cell wall. Xylanase accessibility to xylan therefore increased.

The dramatic decreases in viscosity observed for all three fiber types in both experimental designs may have been due to hydrolysis of cellulose by residual cellulase activity in the xylanase preparation. It is possible that the high xylanase charge resulted in a substantial amount of cellulase activity being applied to the fiber. In addition, swelling of the fibers from the initial extraction stage may have been a key factor. The swelling may have increased enzyme accessibility to crystalline and amorphous cellulose regions. Decreasing the xylanase charge and reaction time help to alleviate this problem. Effective xylanase charges ranged from 100–200 U/g o.d. fiber with a reaction time of 1 h depending on the fiber type.

Note also that the recovered paper sources included in this study were arbitrarily chosen to observe the effectiveness of the above procedure to purify their fiber. Future studies involving enzymatic purification of recovered paper should pay close attention to the fiber makeup of the recovered



4. Hypothetical dissolving pulp process flow sheet

paper depending on the proposed end product—cellulose acetate or cellulose xanthate. Fiber homogeneity—hardwood vs. softwood content—may affect end use of the chemical cellulose product. Evaluation of the pulp from the above process for conversion to cellulose acetate or cellulose xanthate is currently underway.

Hypothetical process

This study suggested that high quality recovered paper sources rich in bleached hardwood kraft fiber may be a potential feedstock for the production of dissolving pulp using such a biotechnical process. **Figure 4** shows a flow diagram for a hypothetical process. The main advantage of this proposed process is its negligible environmental impact. The process could be coupled to the kraft recovery system of a mill containing a recycling or deinking facility that uses high quality recovered paper rich in hardwood. Performing the cold alkali extraction sequences at medium fiber consistency of approximately 10–12% solids to reduce the effluent volume may be possible. Such a system would therefore be essentially effluent free. The waste streams from this process would most likely be suitable for the recovery system. An optimization study aimed at such a system would involve variables such as refining, caustic charge, reaction time, enzyme type, enzyme charge, fiber sources, pulp consistency, and pulp washing arrangements.

CONCLUSIONS

A pulp with properties similar to a chemical cellulose can be produced from high quality recovered paper sources rich in hardwood content with a sequence involving an initial cold alkali extraction, a xylanase treatment, and a final cold alkali extraction. A cold alkali extraction stage is necessary before xylanase treatment to enhance xylan removal effectively. Such a treatment may increase xylanase accessibility into the fiber cell wall. Pulp viscosity decreases because of the xylanase treatment. Optimization of xylanase charge and reaction time may be necessary to minimize fiber damage. TJ

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