IMPROVING ASPEN KRAFT PULP BY A NOVEL, LOW-TECHNOLOGY FUNGAL PRETREATMENT¹

Yi-ru Chen

Research Assistant

and

Elmer L. Schmidt

Professor

Department of Forest Products College of Natural Resources University of Minnesota St. Paul, MN 55108

(Received January 1994)

ABSTRACT

This study investigated the biopulping agent *Phanerochaete chrysosporium* with a new process that required neither wood sterilization nor pure culture incubation conditions. Aspen (*Populus tremuloides* Michaux) chip bales with three treatments were studied. Each bale was kraft cooked after 8.5 weeks of pretreatment. The effects of fungal inoculation and foil-wrapping on pulp and paper strength properties were evaluated. Fungal pretreatment caused significantly faster response to beating as lower freeness was noted. Foil-wrapping retarded the loss of moisture within the bale, and as a result, prolonged fungal activities, resulting in substantial increases in burst strength. Tear was slightly increased, but there was no increase in tensile strength. In some bales, brightness of unbleached pulp was reduced. This study has shown that substantial improvements in certain paper properties and potential beating energy savings could be achieved through this compression/baling technique. Optimization of this system has the potential to provide a practical method of chip pretreatment for the pulp and paper industry.

Keywords: Aspen, Phanerochaete chrysosporium, biopulping, paper properties.

INTRODUCTION

The kraft pulping process is the most widely used pulping method, representing over 70% of the world's annual pulp production (Eriksson and Kirk 1985). The advantages of kraft pulping include more flexibility with regard to wood species, shorter cooking times, and higher paper strength properties; but the kraft process consumes large volumes of various chemicals for pulping and bleaching, and improvements in the kraft system are needed.

Biopulping, generally considered the use of lignin-degrading fungi to pretreat wood chips prior to pulping, has been studied since the 1950s in the context of the mechanical pulping process (Eriksson 1985). It has been found that certain enzymes (ligninase or lignin peroxidase) released from wood decay fungi (mainly white-rot fungi) may alter or degrade lignin by breaking carboncarbon bonds of phenolic elements resulting in oxidative ring cleavage, and therefore making lignin molecules more susceptible to chemicals in the papermaking processes (Kirk and Chang 1975; Pilon et al. 1982). Currently, the concept of biopulping has been extensively explored together with chemical and mechanical pulping processes and has been found to provide encouraging benefits such as energy savings,

¹ This paper is published as Paper No. 21,132 of the contribution series of the Minnesota Agricultural Experiment Station based on research conducted under Project 43-80, supported by McIntire-Stennis funds.

Wood and Fiber Science, 27(2), 1995, pp. 198–204 © 1995 by the Society of Wood Science and Technology

reductions in chemical consumption, paper property improvements, and decreases in the productions of undesirable wastes (Eriksson 1990; Leatham et al. 1990; Oriaran et al. 1991; Pilon et al. 1982; Reid 1991; Setliff et al. 1990; Trotter 1990).

Currently, laboratory investigations on biopulping have been conducted under controlled situations, with the need for initial wood sterilization, a noncontamination or pure culture incubation environment, and optimal fungal growing conditions often using nutrient supplements and aeration. All such factors represent obstacles to an industrial scale fermentation application (Reid 1989). Furthermore, relatively little information on kraft pulp of fungus-treated wood is available. Since the kraft pulp process accounts for a large portion of pulping methods, the effects of fungal pretreatment of chips on kraft pulp and papermaking properties need to be explored.

This study investigated kraft pulp and papermaking properties of Phanerochaete chrysosporium-degraded aspen chips from compressed dewatered bales based on earlier studies of wood chip fuel (Lin 1991; Lin and Schmidt 1991, 1992; Steklenski et al. 1989). It was found that the compression/baling process altered wood moisture content, affected living cells in the wood, promoted quicker heating of chips, and tended to alter the natural microflora in the bales. The growth of Phanerochaete chrysosporium was noted in the nonsterilized chips without fungal inoculation and without pure culture incubation conditions during compression/baling studies (Lin 1991; Lin and Schmidt 1991, 1992). Additionally, this approach was expected to provide processing energy reductions, based on previous studies of fungal treatment of wood chips. The objective of this study was to determine if such a compression/baling system, which did not require wood sterilization nor pure culture incubation conditions, would provide kraft paper property improvements after fungal pretreatment. Specific gravities of the chips were determined after incubation. Five pulp and paper properties were also evaluated after kraft pulping.

MATERIALS AND METHODS

Quaking aspen (*Populus tremuloides*, Michaux) logs from north-central Minnesota were cut into 1,250-mm (4-foot)-long bolts with diameters ranging from 115–150 mm (4–6 in.). They were hand debarked and chipped in a drum chipper, which produced chips of $38 \times$ 30×5 mm average size. The chips were then fed by a vibratory feeder into an hydraulic ram press and pressed at 34.5 MPa (5,000 psi) for 5 min and then strapped to make bales (333 \times 333 \times 420 mm) with a bulk density of 360 kg/m³. The details of bale manufacture processes have been described previously (Lin 1991).

Prior to bale-making, 200 ml (106 spores per ml) of an aqueous conidial dispersion containing a mixture of two P. chrysosporium strains (ATCC 24725 and a wild isolate that was obtained earlier from aspen chips) were sprayed onto chips as they were loaded into the press. Four kinds of bales were evaluated in this study: 1. a compression-dewatered bale with foilwrapped sides to retard drying during incubation but without fungal inoculation (CNF); 2. a compression-dewatered bale with fungal inoculation and foil-wrapping (CIF); 3. a compression-dewatered bale with fungal inoculation but without foil-wrapping (CI); and 4. a control set of chips (which were air-dried to minimize fungal growth) of the same initial weight as went into each bale (NC). All bales were stored at 25 C and 50% relative humidity for 8.5 weeks prior to pulping and paper handsheet evaluations.

Specific gravity of chips in each bale was determined after storage using a water displacement method and oven-dry weight addressed by Lin and Schmidt (1991). The specific gravity of the chips from each bale was obtained from the average of 9 replicates of 0.1-kg samples.

After the bale incubation period, chips were screened to discard fines, oversized wood chips, and knots prior to the kraft pulping process. One-half kg of wood chips (oven-dry weight basis) were kraft cooked in a mini-mill laboratory digester (M/K Systems Inc) at 12% ef-

TABLE 1. Summary of specific gravity (S.G.) from each bale type.¹

Bale type	\$.G. ²	% Loss ³
NC	0.361ª	_
CIF	0.333abc	7.76
CI	0.350 ^{ab}	3.05
CNF	0.316 ^c	12.47

¹ The values shown are the averages obtained from 9 replicates.

² The same letters within a column indicate the homogeneous groups determined by the Tukey honest significant difference paired comparisons ($\alpha = 0.05$). ³ Percent loss refers to the comparison of treated bales to controls.

NC: Noncompressed control set; CIF: Compression-dewatered bale with fungal inoculation and foil-wrapping; CNF: Compression-dewatered bale with foil-wrapping but without fungal inoculation; CI: Compression-dewatered bale with fungal inoculation but without foil-wrapping.

fective alkali concentration and 25% sulfidity for 2 h following the cooking conditions addressed by Oriaran et al. (1991). In order to provide consistent cooking conditions for each batch, cooking times were slightly modified to obtain the same H factor of 1778. Four replicate cooks were prepared for each of the four chip bales.

After kraft cooking, each batch of pulp was rinsed and disintegrated in a standard disintegrator for 2 min. After beating intervals of 0, 5, 10, 15, 20, 25, and 30 min in a Valley beater, samples were taken for freeness testing and handsheet formation according to TAPPI standards T 227 om-85 and T 220 om-88, respectively (TAPPI 1989). Handsheets were conditioned at 25 C and 50% relative humidity for 24 h and then evaluated for burst, tensile, and tear strength properties and brightness according to TAPPI standards T 403 om-85, T 404 om-87, T 414 om-88 and T 452 om-87, respectively (TAPPI 1989).

Second-order polynomials were used to graph the relationship of handsheet strength values and beating time. Ninety-five percent confidence intervals were used for comparisons.

RESULTS AND DISCUSSION

A white fluffy-type mycelial growth was noted throughout most of the inoculated bale with foil (CIF) at the conclusion of incubation. Scattered pockets of similar fungal growth were

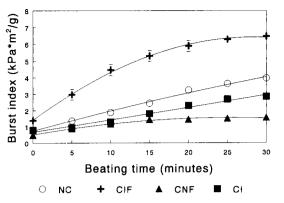


FIG. 1. Burst index as a function of beating time for each bale type. NC: Noncompressed control set; CIF: Compression-dewatered bale with fungal inoculation and foil-wrapping; CNF: Compression-dewatered bale with foilwrapping but without fungal inoculation; CI: Compression-dewatered bale with fungal inoculation but without foil-wrapping.

noted for the noninoculated bale (CNF) and the inoculated bale without foil (CI). Control chips (NC) had no obvious fungal growth visible. Representative mycelial fragments placed onto culture media confirmed *P. chrysosporium* as the fungus observed.

Chips used in this study were from a homogeneous source. After incubation, chips were screened prior to kraft pulping. No obvious difference in unacceptable portions such as fines and oversized wood chips was noted.

Specific gravity

The specific gravities of the chip bales were estimated to determine wood substance losses during pretreatment. Wood substance loss was considered to be due primarily to the deterioration caused by microorganisms within the chip bales. The specific gravity of the control, noncompressed chips (NC), was 0.361. Table 1 reveals that only the foil-wrapped bale without fungal inoculation (CNF) showed significantly lower specific gravity. The reason for the substantially lower specific gravity in CNF might be the presence of a wild type of *P. chrysosporium* that was more aggressive than the strains inoculated into the bales. Also, other

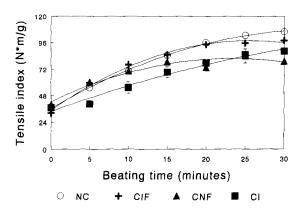


FIG. 2. Tensile index as a function of beating time for each bale type.

microorganisms in addition to *P. chrysosporium* may have been active within the noninoculated chip bales.

Handsheet properties

Burst factor and tensile strength of paper are related primarily to fiber flexibility, which influences interfiber bonding established within the paper mat (Byrd et al. 1965). Tear strength, on the other hand, is known to be more dependent on fiber length except during the early stages of beating and is governed mainly by the intrinsic strength of the fibers (Byrd et al. 1965; Labosky and Ifju 1981).

In this study, paper strength properties of handsheets were compared to controls (NC) after a 30-min beating interval (Table 2). Only the handsheets made from the foil-wrapped bale with fungal inoculation (CIF) showed significantly higher burst strength than the control (Fig. 1). On the other hand, the handsheets

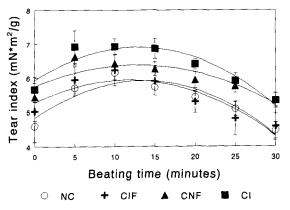


FIG. 3. Tear index as a function of beating time for each bale type.

obtained from the bale with fungal inoculation but without foil-wrapping (CI) and the foilwrapped bale without fungal inoculation (CNF) showed lower burst index with the CNF bale having the greatest decrease (61%). CNF also had the lowest specific gravity (Table 1) indicating the highest degree of fungal decomposition. Foil-wrapping and fungal inoculation resulted in substantial benefits in burst index as high as 81% at 20 min into beating (Table 2).

Although the handsheets prepared from the foil-wrapped bale with fungal inoculation (CIF) had the slightly higher tensile strength early in beating, the increase in tensile strength reported from laboratory studies of biomechanical pulp was not noted (a slight decrease noted in fungal pretreated material at 30-min beating—Table 3 and Fig. 2). The bales without foil or with foil and natural microflora (nonin-

				Time				
Bale type	0 min	5 min	10 min	15 min	20 min	25 min	30 min ²	% Change ³
NC	0.79	1.36	1.88	2.45	3.26	3.64	3.98 ^c	
CIF	1.38	2.98	4.47	5.31	5.90	6.29	6.48 ^d	+62.8
CI	0.79	0.94	1.29	1.81	2.31	2.70	2.86 ^b	-28.1
CNF	0.49	0.89	1.18	1.44	1.45	1.51	1.56 ^a	-60.8

TABLE 2. Summary of burst index from each bale type.¹

¹ The values (kPa·m²/g) shown are the averages obtained from 15 replicates out of 3 cooks.

² The same letters within a column indicate the homogeneous groups determined by the Tukey honest significant difference paired comparisons ($\alpha \approx 0.05$). ³ Percent change refers to the comparison of treated bales to controls after a 30-min beating interval.

				Time				
Bale type	0 min	5 min	10 min	15 min	20 min	25 min	30 min ²	- % Change ³
NC	36.67	55.15	70.34	84.67	96.33	102.84	106.13 ^d	
CIF	32.18	59.70	76.30	85.25	94.48	95.82	98.11°	-7.6
CI	37.09	40.37	55.22	69.37	77.82	85.17	88.47 ^b	-20.0
CNF	39.95	60.39	70.30	78.88	73.69	84.08	84.08 ^a	-20.8

 TABLE 3.
 Summary of tensile index from each bale type.¹

¹ The values (N+m/g) shown are the averages obtained from 15 replicates out of 3 cooks

² The same letters within a column indicate the homogeneous groups determined by the Tukey honest significant difference paired comparisons ($\alpha = 0.05$). ³ Percent change refers to the comparison of treated bales to controls after a 30-min beating interval.

oculated) provided pulps with substantially reduced tensile strength at the end of beating.

Tear properties are generally lower in handsheets obtained from fungal-degraded wood (Hunt et al. 1978; Oriaran et al. 1990, 1991; Reid 1991). In this study, the handsheets prepared from the inoculated bale without foilwrapping (CI) and the foil-wrapped bale without fungal inoculation (CNF) showed increased tear strength. Samples from the bale with fungal inoculation and foil-wrapping (CIF) had a similar tear factor to those from the control (NC) (Table 4 and Fig. 3). The absence of detrimental effects on tear strength is promising and suggests that the tear reduction associated with fungal pretreatment is obviated in the compression/baling system approach.

Brightness of paper is known to decrease in pulps prepared from decayed wood. Hunt et al. (1978) studied the effects of natural decay on aspen kraft pulp and found that higher levels of bleaching chemicals and longer bleaching times were needed to attain a desirable pulp brightness. As reported by Pilon et al. (1982), during the early stage of the pulping, phenolic units of lignin were oxidized to quinones, which darkened the pulps and were not readily removed, resulting in reduced brightness. The laboratory fungal pretreatment of wood chips has reportedly decreased the brightness of the pulps in most studies, and increased bleaching requirements are seen as one drawback of biopulping (Oriaran et al. 1990, 1991; Reid 1991; Setliff et al. 1990).

Brightness of the handsheets from the bale with fungal inoculation but without foil (CI) was not statistically different from that of the control (NC) ($\alpha = 0.05$) either before or after beating (30 min), while brightness of the handsheets made from the bales with foil regardless of fungal inoculation (CIF and CNF) was significantly lower (Table 5 and Fig. 4). The presence of foil to extend the fungal activity by reducing drying rate was detrimental to brightness, although the 20% reduction noted is relatively low compared to those found in other studies. For example, brightness reduction was 58% for aspen pulps after 30 days of fungal pretreatment (Oriaran et al. 1990).

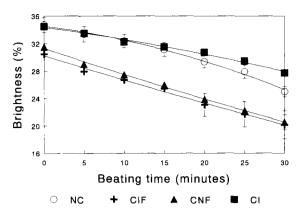
Beating involves hydration of fibers and increases their plasticity. Fibers with a low lignin and high hemicellulose content would reach a

TABLE 4. Summary of tear index from each bale type.¹

	Time								
Bale type	0 min	5 min	10 min	15 min	20 min	25 min	30 min ²	- % Change ³	
NC	4.58	5.71	6.15	5.73	5.44	5.09	4.45 ^a		
CIF	5.01	5.94	6.23	5.90	5.30	4.79	4.57 ^a	+2.7	
CI	5.66	6.92	6.92	6.86	6.40	5,91	5.34 ^b	+20.0	
CNF	5.43	6.62	6.42	6.26	5.94	5.76	5.36 ^b	+20.4	

¹ The values (mN·m²/g) shown are the averages obtained from 15 replicates out of 3 cooks.

² The same letters within a column indicate the homogeneous groups determined by the Tukey honest significant difference paired comparisons ($\alpha = 0.05$). ³ Percent change refers to the comparison of treated bales to controls after a 30-min beating interval.



 F_{IG} . 4. Brightness as a function of beating time for each bale type.

given freeness level faster since they tend to hydrate more readily (Byrd et al. 1965). Oriaran et al. (1990, 1991) reported that hardwood kraft pulps obtained from fungal-incubated wood chips required less beating time to reach a given freeness than pulps obtained from sound wood chips. This was also noted for chips from bales of fungal pretreatment in this

TABLE 5. Summary of brightness from each bale type.¹

	Ti	me	
Bale type	0 min ²	30 min ²	% Change
NC	34.71ª	24.98 ^a	
CIF	30.49 ^b	20.16 ^b	-19.3
CI	34.37 ^a	27.71ª	+10.9
CNF	31.54 ^b	20.64 ^b	-17.4

 1 The values (%) shown are the averages obtained from 9 replicates out of 3 cooks. 2 The same letters within a column indicate the homogeneous groups de-

termined by the Tukey honest significant difference paired comparisons ($\alpha = 0.05$).

³ Percent change refers to the comparison of treated bales to controls after a 30-min beating interval.

TABLE 6. Summary of freeness from each bale type.¹

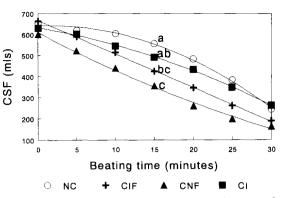


FIG. 5. Canadian Standard Freeness as a function of beating time for each bale type. [The same letters at a beating time indicate the homogeneous groups determined by the Tukey honest significant different paired comparisons ($\alpha = 0.05$).]

study (Table 6 and Fig. 5). For example, the CIF bale material took 40% less time to reach a CSF value of 400 than the NC control pulps. These results indicated that less beating time or energy was required for CNF and CIF to reach a desirable freeness.

CONCLUSIONS

This fungal pretreatment system combining foil-wrapping and compression-dewatering baling technique has shown initial potential to provide a more feasible fermentation technology for biopulping as substantial improvements in certain paper properties (such as significant burst increase and some increases in tear) were noted. The decreased freeness implied that less beating time (or energy) was required for a desirable freeness after fungal pretreatment. Relatively minor reduction in

			Time				
Bale type	0 min ²	5 min	10 min	15 min ²	20 min	25 min	30 min ²
NC	653.0ª	620.7	604.7	556.7ª	483.7	382.3	243.0ª
CIF	663.3 ^a	589.7	514.3	424.7 ^{bc}	345.3	261.0	189.0 ^t
CI	630.0 ^a	602.0	545.3	491.3 ^{ab}	432.7	347.3	261.7ª
CNF	600.3 ^a	522.7	439.7	355.0 ^c	259.7	198.3	163.7 ^t

¹ The values (ml) shown are the averages obtained from 3 replicates out of 3 cooks.

² The same letters within a column indicate the homogeneous groups determined by the Tukey honest significant difference paired comparisons ($\alpha = 0.05$).

nonbleached brightness did occur in some bales when fungal pretreatment was effected by this novel low-technology system. Conditions created in such inoculated bales are in general uniform and conducive to the development of *P. chrysosporium* without the need to sterilize wood or maintain pure culture or highly regulated incubation conditions. Nutrient supplements are not required but may increase benefits.

REFERENCES

- BYRD, V. L., E. L. ELLWOOD, R. G. HITCHINGS, AND A. C. BAREFOOT. 1965. Wood characteristics and kraft paper properties of four selected loblolly pines. II. Wood chemical constituents and their relationship to fiber morphology. Forest Prod. J. 15(8):313–320.
- ERIKSSON, K-E. L. 1985. Swedish developments in biotechnology related to the pulp and paper industry. TAP-PI 68(7):46-55.
- -----, AND T. K. KIRK. 1985. Biopulping, biobleaching, and treatment of kraft bleaching effluents with whiterot fungi. Pages 271–294 *in* M. Moo-Young, ed. Comprehensive biotechnology IV. Pergamon Press, New York, NY.
- HUNT, K., J. T. BASHAM, AND J. A. KEMPERMAN. 1978. Kraft-pulping evaluation of decayed trembling aspen (*Populus tremuloids*) from Ontario. Canadian J. Forest Res. 8:181–187.
- KIRK, T. K., AND H.-M. CHANG. 1975. Decomposition of lignin by white-rot fungi. II. Characterization of heavily degraded lignin from decayed spruce. Holzforschung. 29:56–64.
- LABOSKY, P., JR., AND G. IFJU. 1981. A study of loblolly pine growth increments—Part IV. Paper-making properties. Wood Fiber 13(2):120–137.

- LEATHAM, G. F., G. C. MEYERS, T. H. WEGNER, AND R. A. BLANCHETTE. 1990. Biomechanical pulping of aspen chips: paper strength and optical properties resulting from different fungal treatments. TAPPI 73(3):249–255.
- LIN, Y.-L. 1991. Storage characteristics of compressiondewatered aspen fuel chip bales. Ph.D. thesis, University of Minnesota, St. Paul, MN. 134 pp.
- , AND E. L. SCHMIDT. 1991. Effects of compression on parenchyma cell viability, initial heating, and microflora of aspen fuel chips. Wood Fiber Sci. 23(2):253– 259.
- -----, AND ------. 1992. Changes in specific gravity and pH of compression-dewatered aspen fuel chip bales during storage. Forest Prod. J. 42(4):67-69.
- ORIARAN, T. P., P. JR. LABOSKY, AND P. R. BLANKENHORN. 1990. Kraft pulp and papermaking properties of *Pha-nerochaete chrysosporium*-degraded aspen. TAPPI 73(7): 147-152.
- -----, ----, AND -----. 1991. Kraft pulp and papermaking properties of *Phanerochaete chrysosporium* degraded red oak. Wood Fiber Sci. 23(3):316-327.
- PILON, L., M. C. BARBE, M. DESROCHERS, AND L. JURASEK. 1982. Fungal treatment of mechanical pulps—Its effect on paper properties. Biotechnol. Bioeng. 24:2063–2076.
- REID, IAN D. 1989. Solid-state fermentations for biological delignification. Enzyme Microb. Technol. 11:786– 803.
- ——. 1991. Biological pulping in paper manufacture. Tirtech. 9:262–265.
- SETLIFF, E. C., R. MARTON, S. G. GRANZOW, AND K. L. ERIKSSON. 1990. Biomechanical pulping with white-rot fungi. TAPPI 73(8):141-147.
- STEKLENSKI, P. G., E. L. SCHMIDT, AND J. G. HAYGREEN. 1989. Properties of compression dewatered and baled aspen wood chip fuels. Forest Prod. J. 39(2):8–13.
- TAPPI. 1989. Paper and paperboard testing T400-T547. TAPPI test methods, vol. 1. Tappi Press, Atlanta, GA.
- TROTTER, P. C. 1990. Biotechnology in the pulp and paper industry: A review. Part 1: Tree improvement, pulping and bleaching, and dissolving pulp applications. TAPPI 73(4):198-204.