A BIOPULPING FUNGUS IN COMPRESSION-BALED, NONSTERILE GREEN PINE CHIPS ENHANCING KRAFT AND REFINER PULPING

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ABSTRACT

The present study indicated that inoculation of compression-baled, nonsterile jack pine chips with *Ceriporiopsis subvermispora* led to a 20% reduction in the total kraft pulping time necessary for achieving pulp and paper properties comparable to those from controls. The resulting pulp from the control and *C. subvermispora*-treated chips responded to hydrogen peroxide bleaching similarly; the final brightness values were statistically identical, although the biokraft pulps consumed less hydrogen peroxide. Refiner pulps from these baled chips led to significant increases in paper burst, tensile, and tear strength.

Keywords: Compression-baling, jack pine, *Ceriporiopsis subvermispora*, biokraft pulp, biomechanical pulp, hydrogen peroxide bleaching.

INTRODUCTION

The lignin-degrading capability observed in white-rot fungi bears a relationship to chemical pulping processes, which dissolve or modify lignin leading to the separation of wood fibers for papermaking. In fact, the concept of treating pulpwood with fungi prior to conventional pulping processes, which is referred to as biopulping, has been extensively explored in attempts to reduce energy and chemical consumption associated with current pulping processes. Savings in energy and chemical intake as the result of biopulping will not only have economic benefits but also improve environmental sustainability of pulping and papermaking processes. However, before biopulping can be practically employed, several obstacles have to be overcome. First, an incubation time of several weeks is currently needed for any notable benefits on a laboratory scale. Another drawback is sterilization or heating of wood since most explored fungi (e.g., *Ceriporiopsis subvermispora* (Pil). Gil. et Ryv.) cannot compete well against other naturally occurring microorganisms. Biopulping studies in laboratories are usually carried out in highly controlled environments with various nutrient supplements for small volumes of wood chips. Extensive researches have been undertaken to facilitate fungal or enzymatic activities, but no feasible practices have been reported. It is also known that the effects of fungal pretreatments depend greatly on the fungal species and strains as well as

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inoculation and incubation conditions. Therefore, no common approach for successful biopulping has been identified. The only large-scale approach tested to date requires steaming of chips, addition of nutrients, and forced filtered air flow through chip piles (Brennan 1998; Scott et al. 1997). Nevertheless, the benefits of biopulping observed to date merit more research for possible eventual commercialization.

The majority of biopulping studies have been undertaken on mechanical pulping. The most important benefit of biomechanical pulping is the reduction in electrical energy during mechanical defiberization and/or refining, which is believed to result from lignin modification allowing fibers to be separated more readily (Akhtar 1994; Kashino et al. 1993; Setliff et al. 1990). In addition, it is found that fungal pretreatment prior to mechanical pulping also brings about considerable improvements in subsequent paper strength properties such as burst, tensile, and tear indices, although decreases in brightness and light-scattering coefficient are also encountered and opacity of paper is usually not affected (Akhtar 1994; Akhtar et al. 1992, 1993; Kashino et al. 1993; Pilon et al. 1982; Setliff et al. 1990).

Relatively little information on biochemical pulping is available. Oriaran and colleagues (1990, 1991) reported that under the same kraft pulping conditions, kappa number was decreased when hardwood chips were pretreated with *Phanerochaete chrysosporium* Burds for 20 or more days. The enhanced penetration of cooking liquor caused by the removal of wood mass during fungal incubation could explain these differences. Biokraft pulping studies also revealed that the improved strength properties (e.g., tensile) and faster response to beating could be obtained that corresponded to the increased fiber flexibility indicated by water retention value. As observed for biomechanical pulps, biokraft pulp did not show effects on handsheet opacity while brightness was reduced (Chen and Schmidt 1995; Oriaran et al. 1990, 1991). The reduction in kappa number on sulfite pulp as the result of fungal pretreatment has also been reported. On the other hand, such fungal treatment prior to sulfite pulping causes decreases in paper strength properties (Messner et al. 1992; Scott et al. 1995).

The compression-baling process, which was first investigated for the storage of aspen fuel chips, lowers the moisture content of wood chips, promotes a temperature rise, reduces the numbers of viable parenchyma cells, and tends to alter the distribution of natural microorganisms in the chip bales (Lin and Schmidt 1991; Steklenski et al. 1989). These factors are thought to contribute to the establishment of *P. chrysosporium,* a biopulping fungus, in hardwood chip bales. Early studies on the inoculation of aspen (*Populus tremuloides,* Michx.) with *P. chrysosporium* using this compression-baling technique revealed that increases in burst and tensile indices could be obtained for both refiner (Schmidt et al. 1994) and kraft pulps (Chen and Schmidt 1995). In addition, substantial reduction in beating time was documented for the biokraft pulp.

The purpose of this study was to determine whether refiner and kraft pulping benefits could be realized using the biopulping fungus *C. subvermispora* in the compression-baling system with nonsterile jack pine (*Pinus banksiana* Lamb.) chips. This fungus has been used successfully to improve biomechanical softwood pulps in a number of laboratory studies.

MATERIALS AND METHODS

Bale manufacturing and storage conditions

Jack pine logs (150–200 mm in diameter) from the University of Minnesota Forestry Experimental Station, Cloquet, Minnesota, were cut into 1,830-mm-long bolts. The bolts were hand debarked and chipped using a drum chipper and then screened. The freshly screened chips were placed into plastic bags to minimize moisture losses prior to compression and baling. About 0.11 m^3 chips were compressed for 3 min at 20.7 MPa (3,000 psi) to provide a bale (333 mm × 333 mm × 420 mm) with a density of 368 kg/m^3. This represents a 54%

reduction in total volume compared to the same weight of noncompressed chips (Lin 1991). For *C. subvermispora* (FP-L-14807-Sp) treated bales, previously sterilized chips overgrown with *C. subvermispora* were added (at approximately 3% on a wet basis) as chips were being fed into the press and then compressed under the same conditions as the control chips. The details of the bale manufacturing were described previously (Lin 1991). For each treatment, four bales were made. The inoculated bales were loosely wrapped with foil to reduce surface drying, which would more nearly simulate conditions found in bale stacks. The bales were kept indoors under ambient temperature (20–25°C) and relative humidity conditions for 45 days. The compressed control chips were immediately air-dried to prevent microbial activity.

Mechanical refining

For *C. subvermispora*-treated chips, two bales with similar visible hyphal development internally were chosen for pulping studies. Six chips were taken from each of six areas (upper, center, lower) within each bale and plated onto agar plates to confirm that the fungus noted by eye was in fact the one inoculated. The chips from the two bales were blended before being used. Prior to refining, the whole chips were soaked overnight to soften the wood and to facilitate the fibrillation process. The wood chips were fibrillated in 600-gram batches using a 300-mm Sprout-Waldron rotating single disk refiner (Muncy, PA) at atmospheric pressure using C-2976 stainless steel plates. The chips were hand-fed into the refiner using a vibratory feeder. After the initial pass, the chips were left in hot water for approximately 20 min to reduce any latency in the fiber bundles. Subsequent passes were put through the plates at 4–5% consistency at roughly 30°C. The hot water is thought to increase fiber flexibility, enabling attainment of the desired CSF level while retaining average fiber length. The samples were refined using the following series of gap settings: 1.02, 0.51, 0.20, 0.10, 0.05, and 0.025 mm. The chips were run through at the 0.025-mm gap setting until the CSF value reached 100 ± 5 ml to allow for direct comparison of strength properties. The pulp was dewatered in between passes in a 200-thread per inch muslin bag to enhance retention of fines. Two refiner runs from each treatment were done.

TABLE 1. *Kraft pulping conditions for jack pine chips.*

Minutes to temperature	45
Pulping temperature (C)	170
Wood chips charge (grams, oven-dry basis)	700
Effective alkaline (%)	16
Sulfidity (%)	24
Liquor to wood ratio	4.5:1

The refiner plates were cleaned before pulping a new sample to prevent contamination from the previous bale. Before the first pass, at least fifty grams of excess chips from the sample to be refined were passed through the refiner and discarded. This was done to reduce variations in chip refining during the first pass.

Kraft pulping

Seven hundred grams (based on oven-dry weight) of either control jack pine (without any fungal development) or fungal-treated chips from bales were individually cooked in a laboratory digester (M/K Systems Inc., Danvers, MA) according to the kraft pulping conditions described by Hunt and Benoit (1979) in Table 1. However, H factors were modified to reach a target kappa number (ca. 35 for control) and various H factors were applied for wood chips subjected to fungal pretreatment to produce paper strength properties comparable to the controls. By doing so, savings in kraft pulping time as a result of fungal inoculation could be assessed. For both treatments, four replicate cooks were performed, and the resulting pulp and paper properties from three of the most comparable kraft runs were calculated and averaged for comparison.

After kraft pulping, pulp was washed and screened. Screened pulp yields and reject percentages were determined based on the oven-

dry weight of initially charged wood chips. Pulp was evaluated for kappa number (TAPPI Test Method T236 cm-85) and fiber length using a Kajaani FS-200 fiber analyzer (Kajaani Electronics Ltd., Norcross, GA). Pulp was then beaten in a valley beater (Valley Iron Works Co., Appleton, WI) and pulp samples were withdrawn at intervals of 0, 5, 15, 25, 30, and 35 min for CSF determination (TAPPI Test Methods T200 om-85 and T227 om-85).

Handsheet formation and testing

Handsheets of 60 g/m^2 were prepared according to TAPPI Test Method T205 om-88 and conditioned at 21°C and 50% RH for 24 h before testing. For refiner pulps with CSF 100 ± 5 ml, five handsheets without formation defects were tested for strength (burst, tensile, and tear) properties. For kraft samples, five handsheets from each beating interval were tested for thickness, strength properties, brightness, opacity, and light-scattering coefficient according to TAPPI Test Method T220 om-88. The properties of the handsheets made from pulps with similar CSF were compared among different treatments.

Hydrogen peroxide bleaching

Kraft pulps from the noninoculated and *C. subvermispora*-inoculated chips that resulted in comparable handsheet strength properties were bleached with hydrogen peroxide. This was done to study differences in the response of these two types of pulps to bleaching when subjected to different degrees of kraft pulping. If fungal pretreatment yields handsheet properties similar to the control and results in comparable or better response to hydrogen peroxide bleaching, the savings in kraft pulping time can be justified.

The bleaching conditions are summarized in Table 2. Unbeaten pulp was placed into a plastic bag. The prepared bleaching liquor was introduced into the bag and mixed well with pulp. Starting pH was taken at this point. The plastic bag was then placed in a water bath with a temperature of 90°C for 60 min. Final pH was determined at the end of bleaching and the residual concentration of H_2O_2 was titrated using the iodometric procedure. The pulp was then diluted, washed, and neutralized with 4N sulfuric acid to pH 6.5. The pulp was kept at ambient temperature for at least 1 h for neutralization to complete. Five handsheets were made and tested for brightness.

TABLE 2. *Hydrogen peroxide bleaching conditions.*

Pulp (grams, oven-dry basis)	9
Consistency (%)	15
Temperature (C)	90
Time (minutes)	60
Hydrogen peroxide concentration (%, based on oven-dry pulp)	2
Sodium hydroxide (%, based on oven-dry pulp)	1
Magnesium sulfate (%, based on oven-dry pulp)	0.2
Sodium silicate (%, based on oven-dry pulp)	3

RESULTS AND DISCUSSION

Refiner pulp

The enhancement of softwood mechanical pulps by *C. subvermispora* pretreatment has been well documented in laboratory scale trials. Energy consumption for reaching a given freeness can be greatly reduced and paper strength properties can also be improved (Akhtar et al. 1992; Setliff et al. 1990). However, those studies were carried out with heated or sterilized wood samples under controlled incubation conditions that represented challenges for industrial application. In this study, jack pine chips were inoculated with *C. subvermispora* using the compression-baling system without heating of chips for decontamination and nutrient supplementation. Figure 1 reveals that such pretreatment led to significant increases in burst, tensile, and tear indices with burst index having the greatest percentage improvement (140% increase). It appeared that *C. subvermispora* was able to develop efficiently in the chip bales resulting in positive effects on refiner pulps.

Kraft pulp

Pulp and fiber properties.—The presence of lignin in pulp has adverse effects on pulp and

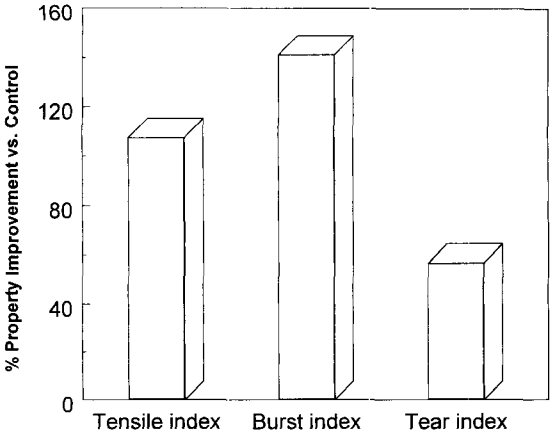

FIG. 1. Handsheet strength properties improvements due to *C. subvermispora* inoculation.

TABLE 3. *Summary of kraft pulping results from the untreated and* C. subvermispora*-treated (cs) jack pine chips using the respective H factor.*

	Control (H1600)[1]	Control (H2000)[2,3]	Cs (H1400)[2,3]
Screened yield (%)	39.4	43.0 a	42.8 a
Rejects (%)	1.4	1.0 a	0.7 a
Kappa number (ml)	43.8	31.5 a	37.2 b
Fiber length (mm)		3.0 a	2.9 a
Coarseness (mg/100m)		16.0 a	14.6 a

[1] The values represent one kraft pulping run.
[2] The first four values represent the averages of three replicates (three kraft pulping runs) while the last two are obtained from the averages of two replicates.
[3] The same letters within a row indicate the homogeneous group determined by Tukey pairwise comparisons ($\alpha = 0.05$).

paper properties. Pulps with too high a lignin content require more beating energy to reach a given freeness and show poor interfiber bonding. This, consequently, produces paper of low density (high bulk) and reduced strength properties (Robinson 1980). Moreover, pulps with higher lignin content require more costly bleaching chemicals to reach a given brightness. Therefore, it is desired to obtain the pulp with the lowest kappa number without degrading holocellulose during pulping. With conventional kraft pulping processes, softwood chips are normally cooked to a kappa number of 20–35 yielding pulps of bleachable grade (Biermann 1993). In this study, several preliminary kraft pulping trials were undertaken, and it was found that an H factor of 2,000 gave rise to such target kappa number for the control (noninoculated) jack pine chips. When the cooking time of control chips was reduced (e.g., an H factor of 1,600), the resulting kappa number (a value of 44) was too high (Table 3). Although only a single cook was made at this stage of experiment, it indicated that the H factor should be raised to produce pulp with a lower kappa number.

The purpose of this kraft pulping study was to investigate whether fungal pretreatment could lead to a reduced cooking time while maintaining acceptable pulp and paper properties. Therefore, the H factor was decreased, i.e., cooking time was shortened, for *C. subvermispora*-treated jack pine chips, and the resulting handsheet properties were compared to those from the control. After several preliminary kraft pulping trials using H factors ranging from 2,000 to 1,200, it was decided to explore the properties of the handsheets prepared from *C. subvermispora*-inoculated chips cooked using an H factor 1,400. When the H factor was lowered below 1,400, the resulting kappa number was significantly higher (e.g., 43.6 at H factor 1,200), which impaired beating response and strength properties. It has been reported that under identical kraft or sulfite pulping conditions, kappa number is greatly decreased as the result of degradation and/or modification of lignin by the inoculated white-rot fungi (Fischer et al. 1994; Messner et al. 1992; Oriaran et al. 1990, 1991; Scott et al. 1995). This was not noticed in this experiment. When the H factor was equal to that of controls (2,000), similar properties of pulp were noted, but fungal-treated chips had a lower screened yield (40.0 vs. 44.6%). However, Table 3 shows that when the fungal-treated chips were cooked using an H factor 1,400, which corresponded to an approximately 20% reduction in total cooking time, no substantial differences in screened yield, rejects, and pulp properties (fiber length and coarseness) from the control chips were noticed. On the other hand, kappa number of such pulp was 18% higher than the control sample which was kraft

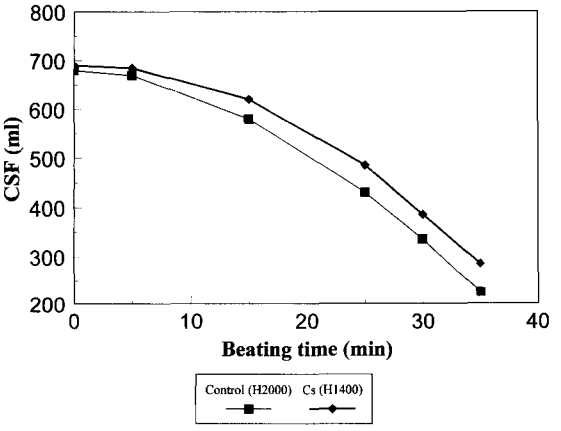

FIG. 2. CSF development during the course of beating.

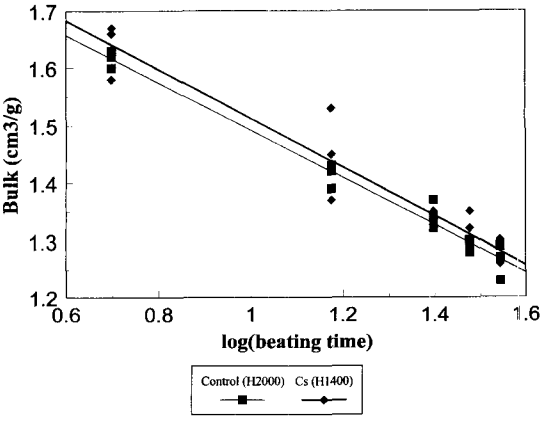

FIG. 3. Bulk as a function of the logarithm of beating time.

pulped using an H factor of 2,000. Nevertheless, this higher kappa number did not have adverse effects on beating responses and paper properties.

Beating response.—It has been reported that fungal pretreatment leads to a reduction in both beating time and energy required to reach a given freeness for chemical and mechanical pulps (Akhtar 1994; Chen and Schmidt 1995; Kashino et al. 1993; Oriaran et al. 1990, 1991). The reduction is thought to be attributed to the enhanced swelling and flexibility of fibers as well as the higher holocellulose content of pulp (Oriaran et al. 1990, 1991; Sachs et al. 1989). In this study, a lower H factor, that is, a shorter kraft pulping time, was used for the fungal-treated wood, which resulted in higher kappa number as seen in Table 3. However, Fig. 2 shows that at a given beating time there was no significant difference in the freeness levels of both pulps, which indicated that the higher lignin content as a result of shortening cooking schedules employed on *C. subvermispora*-inoculated chips did not impair beating responses.

When the bulk values of the handsheets from various pulps are plotted against the logarithm of the beating time, parallel straight lines will result, and the slope of the lines solely depends on the particular beating conditions used (Clark 1985). Therefore, this relationship can also be employed to evaluate the behavior of pulps during beating. Figure 3 shows that this equivalent slope relationship held, which meant that the beating conditions were constant throughout the study. Figure 3 also indicates that the bulk values of the handsheets from the respective wood chips at any given beating time were not significantly different. This implies that there was no difference in fiber flexibility between these two treatments that corresponded to what was observed for freeness profiles.

Handsheet properties.—It has been documented that at comparable freeness (CSF) levels, fungal pretreatment of hardwood chips led to an increase in the paper strength properties under the same kraft pulping conditions (Chen and Schmidt 1995; Oriaran et al. 1990, 1991). On the other hand, Messner and coworkers (1992) reported that pretreatment of birch chips with white-rot fungi selective for lignin degradation resulted in slight decreases in the tensile and tear strength of the handsheets obtained from magnesium-based sulfite pulping. Neither situation was observed in this study.

At the end of beating, which yielded a freeness level of approximately 300 ml, the tested handsheet properties, with the exception of brightness, were not significantly different between the noninoculated and *C. subvermispora*-inoculated wood samples subjected to the respective kraft pulping (Table 4). Some re-

TABLE 4. *Average handsheet properties obtained from untreated and* C. subvermispora*-treated jack pine chips kraft cooked using the respective H factors at similar CSF levels.*[1,2]

	Control (H2000)	*C. subvermispora* (H1400)
CSF, ml	300	330
Burst index, kPa·m²/g	6.41 a	6.77 a
Tear index, mN·m²/g	8.40 a	7.74 a
Tensile index, N·m/g	90.36 a	97.16 a
Zero-span index, N·m/g	147.38 a	146.88 a
Brightness, %	17.21 a	16.19 b
Opacity, %	89.32 a	89.73 a
Scattering Coefficient, m²/kg	16.70 a	16.79 a

[1] The values represent the averages of 15 handsheets from 3 kraft pulping batches.
[2] The same letters within a row indicate the homogeneous group determined by Tukey (HSD) pairwise comparisons ($\alpha = 0.05$).

ports have noted a slight increase in the tear index and brightness of the paper of sulfite-pulped chips after treatment of *C. subvermispora* (Fischer et al. 1994; Messner and Srebotnik 1994). On the other hand, the reduction of the tear index and brightness due to fungal pretreatment has also been reported (Oriaran et al. 1990, 1991; Messner et al. 1992). The decrease in brightness is thought to be due to the formation of conjugated carbonyl groups, e.g., quinones, from the oxidation of phenolic compounds during incubation and pulping (Pilon et al. 1982; Samuelson et al. 1980). The observed brightness decrease, although statistically significant ($P < 0.001$), was very minor (6% of control chip value) compared to those reported elsewhere for kraft pulp obtained from fungal-treated hardwood chips (Oriaran et al. 1990, 1991). Zero-span tensile strength was not altered as the result of *C. subvermispora* pretreatment implying that neither deterioration of cellulose fibrils nor increase in individual fiber strength took place as reported by others (Pilon et al. 1982; Oriaran et al. 1990). Other biopulping studies showed a reduction in the bulk and light-scattering coefficient of the handsheets prepared from fungal-treated wood chips (Pilon et al. 1982; Setliff et al. 1990). However, in this study, no decrease in the bulk or light-scattering coefficient was observed. This indicated no increase in fiber flexibility or fiber bonding, which is consistent with the same burst and tensile strength of the control and *C. subvermispora*-treated chips under the respective kraft pulping time intervals. The disagreement could be attributed to the reduction in cooking time for *C. subvermispora*-inoculated chips, which offset the swelling effects of alkali. As reported by others for biomechanical pulp (Akhtar 1994), no difference in opacity was noted.

In summary, the inoculation of jack pine chips with *C. subvermispora* by this compression-baling technique engendered a 20% reduction in total kraft pulping time while it produced comparable pulp and fiber properties related to the noninoculated compressed chips. The resulting paper properties were also not different from those of the control. The 20% reduction in total kraft pulping time indicated that substantial energy savings could be achieved when the jack pine chips are inoculated with this fungus and formed into compression bales prior to the pulping process with neither steaming nor nutrient supplementation. This was significant as studies on steam-sterilized chips inoculated by *C. subvermispora* with nutrient additions have shown that decreases in cooking time ranging from 5 to 24% for calcium- and magnesium-based sulfite pulping, respectively, could be achieved for a given kappa number (Messner et al. 1998; Scott et al. 1995).

Hydrogen peroxide bleaching

As discussed previously, as a result of shortening kraft pulping time by 20%, jack pine chips subjected to *C. subvermispora* led to a higher kappa number than did the control wood. The brightness of the handsheets prepared from fungal treated chips was also slightly reduced (by 1 point) (see Table 4). The purpose of this study was to evaluate, under these conditions, whether the fungal pretreatment resulted in any differences in the responses of pulp to hydrogen peroxide bleaching.

Table 5 shows that the final pH values from both bleaching liquors were above 10.5, which

TABLE 5. *Bleaching performance of jack pine kraft pulps.*[1,2]

	pH		H_2O_2 concentration (% O.D. pulp)[3]		Handsheet brightness[4]	
	Initial	Final	Initial	Final	Initial	Final
Control	11.53	10.55	2.0	0.179 a	22.20 aA	33.08 aB
Cs	11.66	10.52	2.0	0.256 b	21.12 aA	34.22 aB

[1] *Control*: the pulp obtained from the control chips. *Cs*: the pulp obtained from *C. subvermispora*-treated chips.
[2] The same letters within each column and the same capital letter within each row indicate the homogeneous group determined by Tukey (HSD) pairwise comparisons ($\alpha = 0.05$).
[3] The values shown are means of 2 replicates.
[4] The values are the average obtained from 5 replicates.

indicated that the bleaching time could be prolonged or the NaOH charge could be reduced to yield a final pH of about 9. With the same bleaching conditions, the pulp prepared from *C. subvermispora*-treated chips consumed 4% less hydrogen peroxide (a higher residual H_2O_2 concentration, Table 5).

Hydrogen peroxide bleaching gave rise to an 11- to 13-point increase in brightness for the control and fungal-treated wood (Table 5). There was no significant difference in the resulting brightness between these two samples. The brightness gain was smaller than that reported for softwood kraft pulp (Moore 1995; Troughton and Sarot 1992). Nevertheless, this study showed that, despite the slightly higher kappa number, kraft pulp of jack pine chips subjected to *C. subvermispora* responded to hydrogen peroxide bleaching the same way as did the noninoculated control pulp. The results corresponded to other reports that mechanical and sulfite pulps obtained from fungal-treated wood could be bleached to a comparable brightness level as the control at the same hydrogen peroxide charge (Kashino et al. 1993; Scott et al. 1995; Sykes 1993).

CONCLUSIONS

This study showed that *C. subvermispora* was able to engender biopulping benefits under this compression-baling system. The resulting refiner pulp produced handsheets with higher strength properties. Kraft pulping time could be reduced by 20% while maintaining comparable pulp and handsheet properties comparable to controls. These results from the compression-baling biopulping are in contrast to those in other systems where this fungus is reportedly unable to develop efficiently on nonsterile chips (Kirk et al. 1993; Sykes 1994). Therefore, steaming or sterilization of chips has thought to be required for the positive effects of biopulping to occur (Setliff et al. 1990; Sykes 1994; Wall et al. 1993). As mentioned earlier, this compression-baling technique used nonsterilized wood chips without nutrient additions. Nevertheless, the conditions that occurred naturally inside the chip bales were favorable for the biopulping benefits created by *C. subvermispora* growth. Moreover, compression-baling of wood chips reduces the storage volume by 54% (Lin 1991), which could be very advantageous when the chips are to be exported. With further studies it should be feasible to produce larger-sized, inoculated bales that could yield improved pulps due to fungal action during storage.

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