A PFI mill can be used to predict biomechanical pulp strength properties

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ABSTRACT *Recently, we showed that a biomechanical pulping process in which aspen chips are pretreated with a white-rot fungus can give energy savings and can increase paper sheet strength. To optimize this process, we need more efficient ways to evaluate the fungal treatments. Here, we examine a method that consists of treating coarse refiner mechanical pulp, refining in a PFI mill, and testing for freeness. The relative abilily of a fungus to promote a decrease in pulp freeness in the PFI mill did not correlate with its ability to give energy savings in chip refining. However, it correlated with the ability to increase the strength properties of handsheets produced from the chips. We discuss (a) why the PFI mill method successfully predicts strength properties rather than energy savings, (b) the limitations of the method, and (c) the implications of these results for developing a method to predict energy savings.*

KEYWORDS
Biomechanical pulping
Biotechnology
Mechanical pulps
PFI mills
Physical properties
Screening
White-rot fungi

Chemical treatments are used in current chemimechanical (CMP) and chemithermomechanical (CTMP) pulping processes to increase sheet strength (*1–7*). These treatments function by removing or modifying lignin, or by increasing wood fiber swelling (*8, 9*). However, these treatments have disadvantages. They often increase the energy requirements, decrease the pulp yield, increase the sheet tear index less than burst and tensile indices, decrease important optical properties such as opacity, and produce dilute waste effluents that require costly waste treatment (*4*). The disadvantages of chemical treatments have prompted interest in evaluating the potential of biological treatments, based on lignin degradation by white-rotbasidiomycetes(*10–25*).

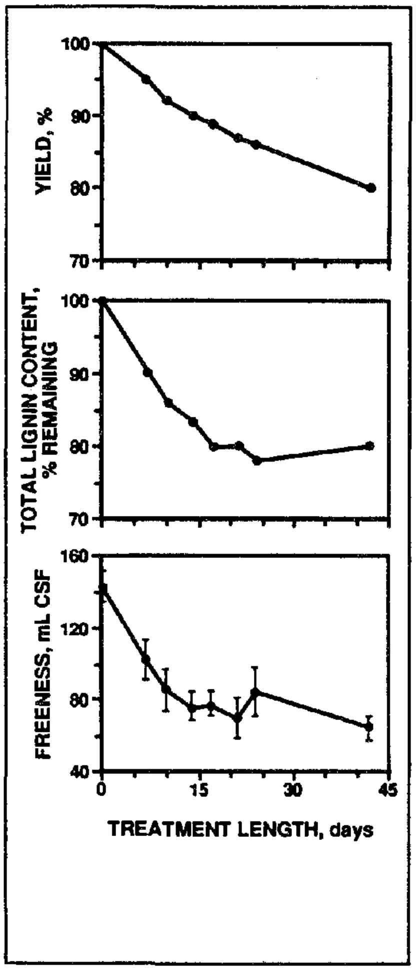
Recently, we demonstrated that aspen chips treated with certain fungi in a nonoptimized, high-yield, bench-scale biomechanical pulping process resulted in a substantial reduction in the energy required for chip fiberization (*19*). Treatments also were capable of markedly increasing burst, tensile, and tear strength indices without reducing opacity (*20, 21*). However, treatments giving maximal energy savings did not necessarily give maximal strength properties. Tests with *Phanerochaete chrysosporium* strain BKM-F-1767 indicated that a four-week treatment yielded both maximal energy savings (*19*) and strength properties (*20*).

Although promising, our model process requires optimization before it has the chance to become economically feasible. Unfortunately, the current methods used for process evaluation require that rather large batches of chips (> 3 kg) be treated and that labor-intensive, time-consuming chip pulping, pulp refining, and handsheet making and testing be carried out.

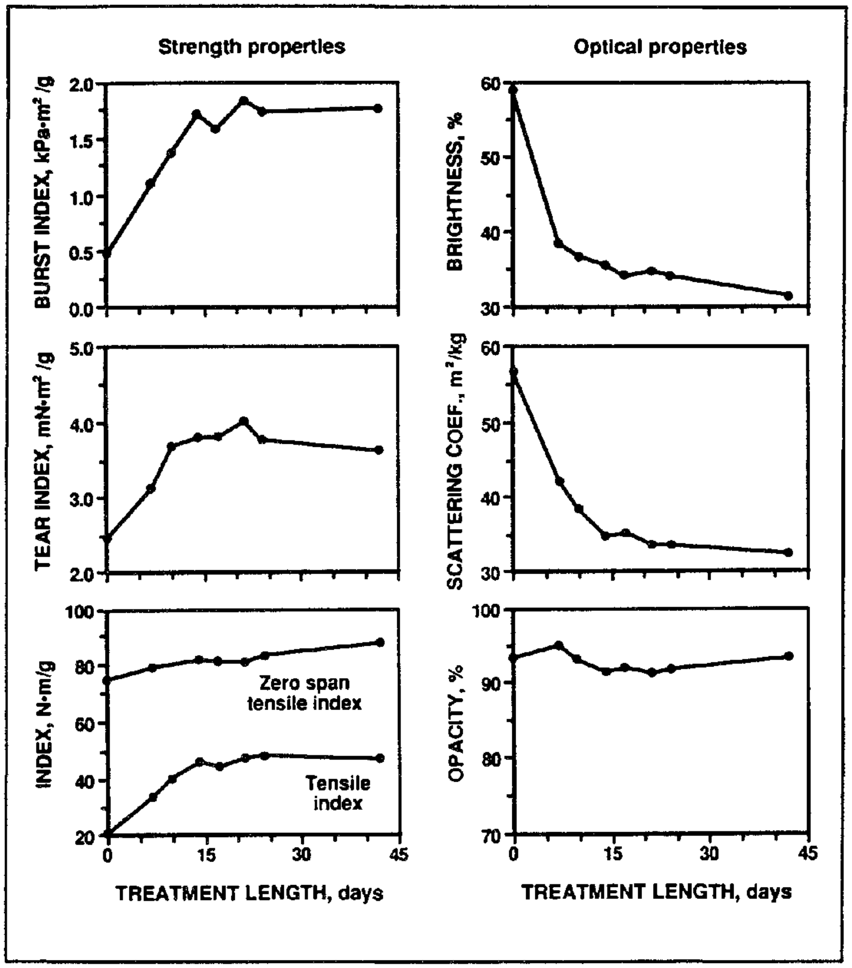
Effective optimization of this and related processes requires the development of more efficient evaluation methods. Methods based on changes in chip or pulp chemical composition have been unsuccessful so far (*19, 20*). Over the range of fungi and treatment conditions tested, little chip degradation (<5% weight loss) was required for maximal effects. Also, treatment efficacy did not correlate with weight loss or the bulk removal of lignin, hemicellulose, or cellulose. Consequently, we have chosen to evaluate methods based on mechanical refining techniques.

Others have used PFI mills for refining pulps to evaluate fungal treatments (*12, 16, 25*). The ability of a treatment to reduce the number of revolutions required to decrease the Canadian Standard Freeness (CSF) of pulp by a given amount has been interpreted as indicating the relative extent of energy savings (*12, 16, 25*). The effects on sheet strength have typically been estimated by generating a series of pulps of different

1. The effect of treatment duration on pulp yield, lignin content, and freeness



2. The effect of treatment duration on strength and optical properties of handsheets made from unbleached pulps (The freeness of each pulp is given in Fig. 1.)



freenesses in the mill, making handsheets, and testing the handsheets so that the strength properties could be estimated at a given freeness (*23*).

Our objective here is to determine the validity of using coarse refiner mechanical pulp and a PFI mill to predict the effectiveness of fungi intended for use in our model biomechanical pulping process. We tested stationary bioreactors, different treatment durations with a single fungus (*P. chrysosporium*), and the same treatment duration with eight different white-rot fungi. We determined the changes in pulp yield, total lignin content, CSF values resulting from PFI mill refining, and the strength and optical properties of 60-g/m^2 handsheets produced from the pulps. Finally, we compared the effects of treating pulp vs. treating chips.

Results and discussion

Effects of pulp treatment duration and fungal species

The effects of different treatment durations were tested using *P. chrysosporium* strain BKM-F-1767, stationary treatments similar to those previously used with chips (*20*). the same nutrient supplements (*20*), and coarse refiner mechanical pulp (320 mL CSF). Pulp yield, total lignin content, and the freeness resulting from 5000 revolutions in a PFI mill dropped to as low as 80%, 78%, and 49% (i.e., 144 mL CSF decreased to 70 mL CSF) of an untreated control after three weeks of treatment **(Fig. 1).**

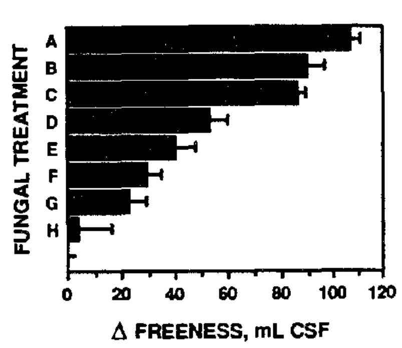
The relative changes in the properties of handsheets made from coarse pulps treated for three weeks (**Fig. 2,** unbleached pulps used) were essentially identical to those previously reported for pulp made from chips treated for four weeks in stationary tray bioreactors (*20*). Burst, tear, and tensile strength indices increased markedly, whereas zero span tensile index showed only marginal change (Fig. 2). Optical properties of brightness and scattering coefficient decreased markedly, whereas opacity remained essentially unchanged. As observed for the decrease in lignin content and freeness, maximal improvements in burst, tear, and tensile indices for sheets produced from unbleached pulps occurred at about three weeks (Fig. 2). However, the maximal optical changes occurred within two weeks.

The effect of three-week treatments by different species of white-rot fungi on the freeness of coarse pulp refined in the PFI mill was determined using the same fungi previously selected for chips (*19, 20*). The fungi differed widely in their ability to promote the extent of the decrease in freeness (ΔCSF) of the treated sample in a PFI

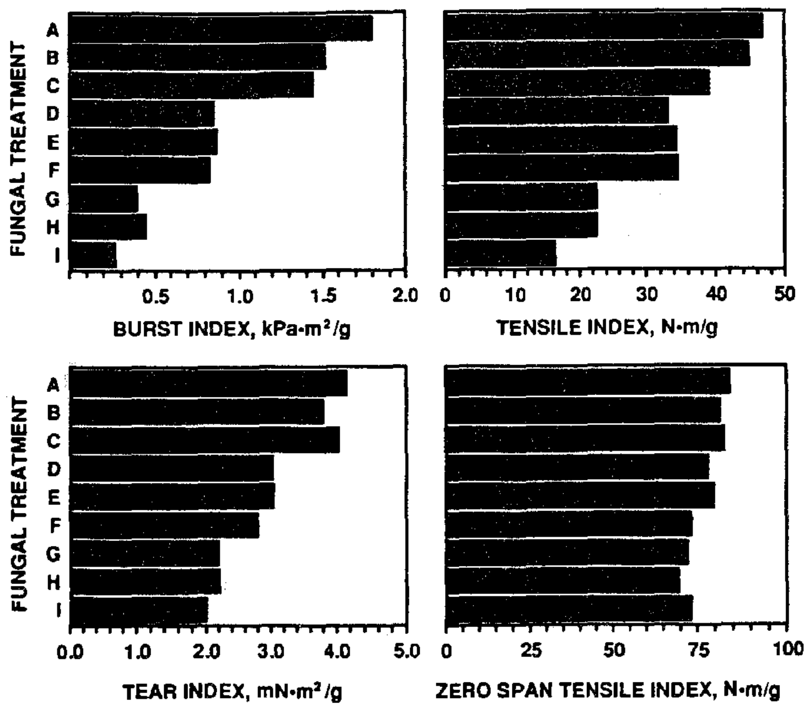
1. Fungal strains as labeled in figures

A	*P. tremellosa*
B	*P. subserialis*
C	*P. chrysosporium*
D	*D. squalens*
E	*P. medulla-panis*
F	*P. brevispora*
G	*P. mutabilis*
H	*T. versicolor*
I	None

3. Promotion of a decrease in pulp freeness resulting from three weeks of treatment with 8 different white-rot fungi. (Pulp samples that did not receive a fungal treatment showed a decrease in freeness from 320 mL to 144 mL.)



4. The effect of three-week treatment with eight different white-rot fungi on strength properties of pulps refined 5,000 revolutions in a PFI mill (The freeness of each pulp can be derived from Fig. 3.)



mill (5000 revolutions) compared to an untreated control (**Fig. 3** and **Table I**). Pulps treated with *Phlebia tremellosa, Phlebia subserialis,* and *P. chrysosporium* (strain BKM-F-1767) showed the greatest change in freeness. In contrast, pulp treated with *Trametes versicolor* showed little change in freeness.

The relative ability of a fungus to change the freeness of the PFI mill pulp was found to correspond closely with its ability to increase strength properties of handsheets made from the pulps. Burst index showed the greatest extent of change **(Fig. 4)** as well as a good correlation with change in freeness **(Fig. 5).** Similar correlations were obtained for both tear and tensile indices (data not shown). Unlike the relationship typically obtained during the mechanical refining of untreated chips, where tear index typically goes through a maximum value, the tear index of the fungal-treated pulps increased with both burst and tensile indices (Fig. 4). In contrast to the correlation with strength increase, the fungal-treated pulps showed no correlation between ΔCSF and the extent of loss of either pulp yield (dry weight) or total lignin content (Fig. 5).

Effects of PFI refining of fungal-treated pulps

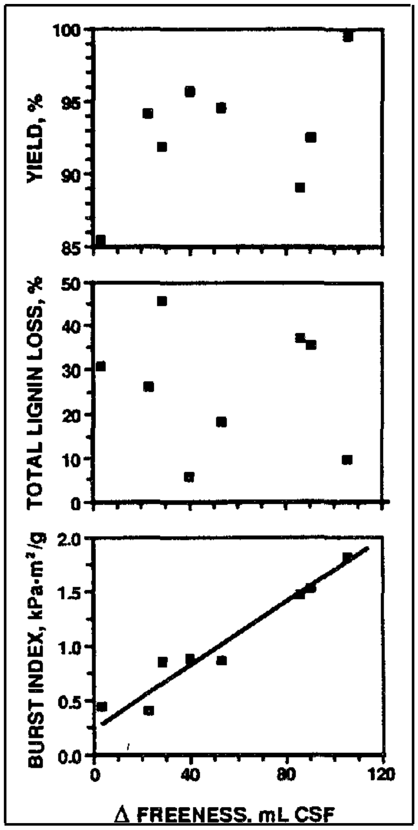
Next, we verified that the changes in strength properties of pulps refined in the PFI mill resulted from the fungal treatments themselves rather than from the different resulting freeness values. We did this by analyzing the effects of refining pulps to three different extents (between 2000 and 10,000 revolutions) in the PFI mill. As expected, the two fungal treatments tested and the extent of refining had a marked effect on the resulting freeness **(Fig. 6).** The fungal treatments had a much larger effect on burst, tear, and tensile indices than did the extent of refining. (The plot for burst index is shown in Fig. 6.) Further analysis showed that the change in freeness values resulting from different extents of refining (50 mL to 180 mL CSF) had much less influence than the fungus had on the burst index and had little effect on the resulting fiber length index **(Fig. 7).**

Taken together, these results indicate that the fungal treatments improved sheet strength by increasing interfiber bonding rather than by increasing the fiber length index. This also suggests that the change in freeness detected was due to modifications of the individual wood fibers that resulted in increased fiber flexibility or defibrillation upon refining.

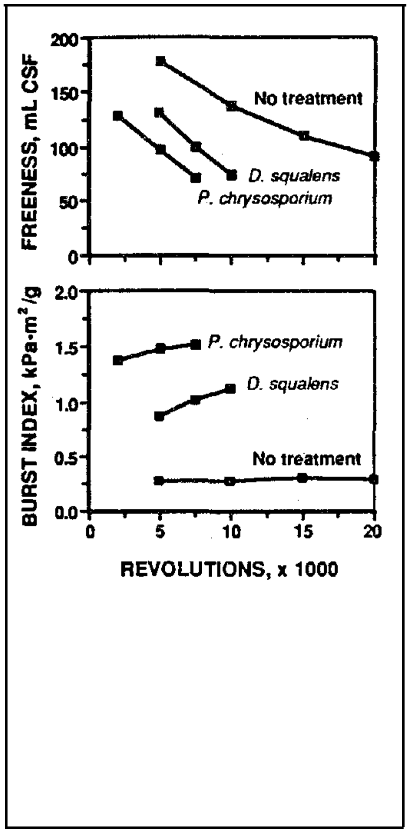
Correlation of PFI refining of pulp with mechanical disc refining of chips

We have shown that the change in

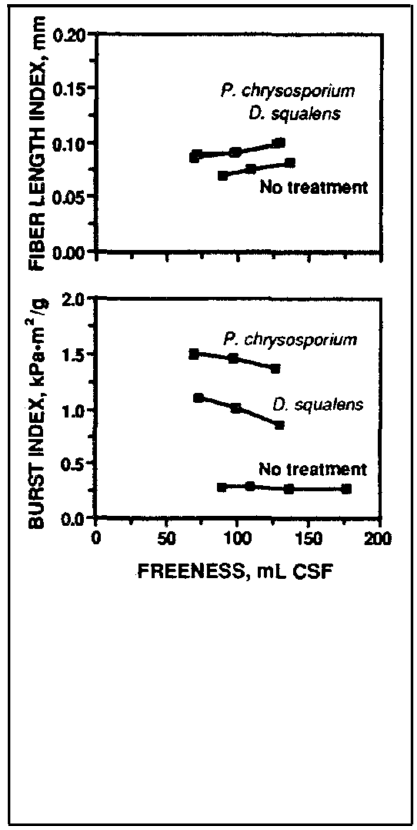
5. The change in freeness of pulp vs. yield, total lignin content, and handsheet burst index. (Data points indicate the 8 different white-rot fungi from Fig. 3.)



6. The effect of the number of revolutions in the PFI mill on the freeness of pulps and the burst index of handsheets



7. The effect of freeness of treated and untreated pulps on pulp fiber length index and handsheet burst index



freeness of pulp refined in the PFI mill accurately predicts relative strength properties of handsheets. We previously showed that chip treatments that give high relative energy savings do not necessarily result in high relative handsheet strength (*19*). Therefore, our next objective was to determine what the PFI mill refining of coarse pulp can predict for the biomechanical pulping of chips. Over the range of different white-rot fungal treatments tested, there was no correlation between the change in freeness values for pulps refined (for 5000 revolutions) in the PFI mill and energy savings for chips pulped in a mechanical disc refiner **(Fig. 8).** However, there was excellent correlation between change in freeness obtained in the PFI mill and increased burst index for handsheets produced from fungal-treated chips pulped to a 100 mL CSF in a mechanical refiner (Fig. 8).

Because we had produced a series of pulps with different freeness values from the treated chips, it was possible to extrapolate strength values at pulp freeness values identical to those obtained with treated pulps refined in the PFI mill. Good correlation was observed between the burst indices for handsheets produced from fungal-treated pulps and fungal-treated chips **(Fig. 9).** However, at equivalent freeness values, the burst indices were consistently higher for handsheets made from the pulps for which the chips were treated with fungus (Fig. 9). This difference was presumably due to fiber damage caused when producing the coarse pulp from untreated chips.

Screening limitations of the PFI mill

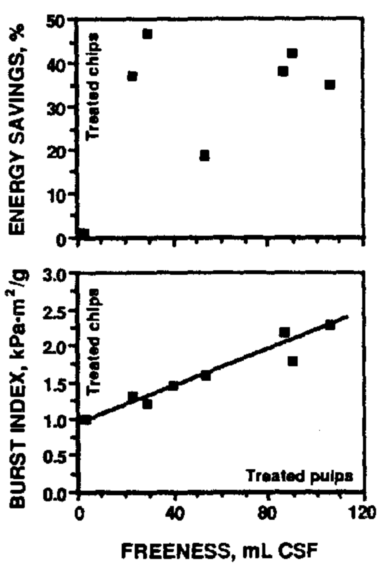
Because the PFI mill screening method showed promise for discriminating between treatments with different genera of white-rot fungi, we examined the method with more closely related fungal treatments. In these tests, we focused on (a) white-rot fungal species within the *Phanerochaete* genus as well as different strains of *P. chrysosporium* and (b) treatments with *P. chrysosporium* strain BKM-F-1767 receiving different relative levels of air exchange in either column or flask bioreactors.

Little difference in change in freeness resulted from the different fungal or aeration treatments **(Fig. 10).** In agreement with our earlier results, the strength properties fluctuated marginally around a similar mean value. An example is the plot of burst index shown in Fig. 10. This fluctuation was slight in spite of the fact that the pulp yields and lignin losses varied considerably between the treatments (data not shown).

Conclusions and further work

A screening method closely predicts the relative strength properties of handsheets made from fungal-treated chips pulped in a mechanical disc refiner, based on the changes in freeness derived from treating coarse pulps refined in a PFI mill. The method appears suitable for screening for the range of factors that markedly affect strength properties. However, it appears to lack the sensitivity required to discriminate among closely related treatments. Also, the meth-

8. Change in freeness of fungal-treated pulps vs. energy savings obtained during chip pulping and burst index of handsheets (Data points indicate the 8 different white-rot fungi shown in Fig. 3.)



od does not predict energy savings.

Initially, we were surprised that the PFI mill method predicted strength properties rather than energy savings: the method is based on detecting an inherent energy savings during mechanical refining. In retrospect, however, this result is consistent with the fact that fungal treatments in our process predominantly render energy savings during chip fiberization, which is a particularly energy-intensive step in mechanical pulping *(19)*.

Evidently, we simply lost the opportunity to measure fiberization energy by screening with pulps instead of chips. Also, it is logical that fungal treatments that promote a decrease in pulp freeness in a PFI mill also increase strength properties. Once first-stage mechanical pulps are produced, much of the energy required to refine them is needed to loosen interfibril bonds to increase fiber flexibility. These same fiber alterations increase interfiber bonding in handsheets and, hence, sheet strength properties. Our results simply indicate that the fungal treatments alter the individual wood fibers within both pulps and chips in a similar fashion.

We still wish to develop a method that predicts energy savings. Our observations suggest that this method should be based on the separation of fibers bound together in wood chips or particles when exposed to a mechanical pulping-like action. Two such methods that should be studied further include (a) the measurement of the amount of energy necessary to fiberize chips to a given extent in a wood defibrator (*16*) and (b) the initial stages of refining extremely coarse pulps in a PFI mill.

Experimental procedures

Fungi

The following fungal strains, maintained on potato dextrose agar (Difco Laboratories, Detroit) slants, were used (Table I):

- *Dichomitus squalens* (Karst.) Reid # TON-427
- *Phlebia* species, including *P. tremellosa,* (Schrad.:FR.) Nakas. *et* Burds. (syn. *Merulius tremellosus*) # RAB-25, *P. brevispora* Nakas. # HHB-7099, and *P. subserialis* (Bourd. et Galz.) Donk # RLG-6074
- *Perenniporia medulla-panis* (Jacq.: Fr.) Donk # RAB-82-5
- *Phanerochaete* species, including *P. chrysosporium* Burds. (syn. *Sporotrichum pulverulentum # BKM-F-1767, # HHB-6251, # K3, # ME-446, # SC-26, # 85118-22 (if not indicated otherwise, strain # BKM-F-1767 was used), P. flavido-alba* (Cooke) Rattan # 106-507, *P. rimosa* (syn. *hynoides*) (Cooke) Burds. # HHB-8704-Sp, and *P. laevis* (Pers.:Fr.) Erikss. *et* Ryv. # FP-101018-Sp
- *Pholiota mutabilis* (Schaeff.:Fr.) Kumm. # OKM-2994
- *Trametes* (syn. *Coriolus) versicolor* (L.:Fr.) Pilat # MAD-697.

Pulp, pulp treatments, and PFI mill refining

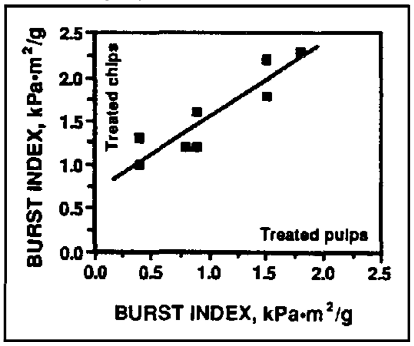
A large quantity of 320-mL-CSF refiner pulp was produced from fresh industrial-type 19-mm aspen chips (*Populus tremeloides* Michx.) in a Sprout Waldron Model D 2202 single, rotating, 305-mm disc mechanical refiner operating at atmospheric pressure. The pulp was stored at –20°C until use. The pulp mixture used for the treatments consisted of 30 g of o.d. pulp supplemented with 120 mL of a glucose-containing, chemically defined, liquid medium described previously (*20*), which was mixed in a 1-L Erlenmeyer flask and steam sterilized by autoclaving for 45 min at 120°C. After cooling, control flasks were set aside, and five test flasks were inoculated for each treatment using six 7-mm-diameter agar plugs taken from fungal-colonized potato dextrose agar plates (Difco Co.). Either the flasks were stoppered with cotton and used as bioreactors directly, or their contents were placed into heat-sterilized glass columns that were 250 mm by 50 mm (inside diameter). Air exchange was not facilitated except for the aeration experiment, in which water-saturated (humidified) air or oxygen was introduced either above the cultures in flasks or up through the culture mass in glass columns. Incubations were stationary for three weeks at 70% RH and 27.0 ±1°C for all fungi except *P. chrysosporium,* which was incubated at 39 ±1°c.

When the experiment was terminated, triplicate sets of treated pulp were frozen and stored at –20°C until refined. The remaining two cultures from each set were oven dried and used to determine pulp yields and compositional changes. Frozen samples were thawed and refined in a Norwegian Pulp and Paper Research Institute Model 109 PFI mill using 22.5 g of o.d. pulp at 10% consistency for 2,000–20,000 revolutions, following TAPPI procedure T248.

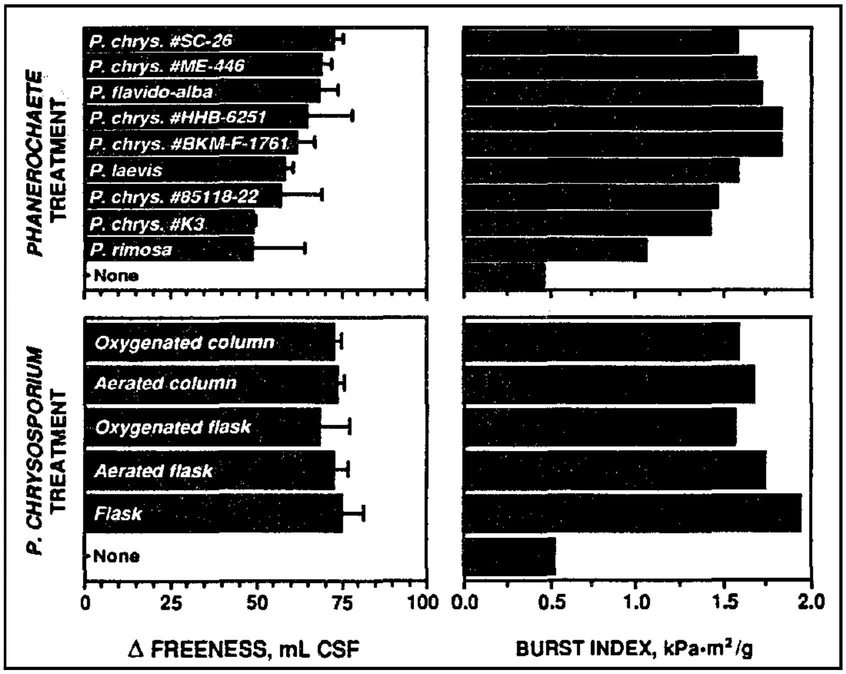
Chip treatments and mechanical disc refining

The fresh, standard, industrial-type 19-mm aspen chips (*Populus tremeloides* Michx.), the nutrient supplements, the inoculation method, aeration with humidified air, and stainless-steel wire trays in polypropylene bag bioreactors were as described previously (20). Stationary incubations were performed for four weeks using the same humidity and temperatures described for the pulps. Previously described methods were used for determining the following: chip yield due to fungal treatment (*20*), refiner mechanical pulping at atmospheric pressure (*20*), and energy savings (*19*).

9. The burst index of handsheets made from mechanical pulp compared to burst index of handsheets made from PFI-mill pulp. (Data points indicate the 8 different white-rot fungi shown in Fig. 3.)



10. The effect of closely related fungal treatments on change in freeness and burst index. Three-week pulp treatments were used with different *Phanerochaete* species and *P. chrysosporium* strains or with *P. chrysosporium* strain BKM-F-1767 receiving different relative levels of air exchange.



Paper production, paper testing, and chemical analysis

Whenever possible, test methods were used as described previously (21). Paper handsheets (60 g/m², T 205) were produced at the freeness values resulting from the indicated number of revolutions in the PFI mill. Handsheet properties tested included density, burst index (T 403), tensile index (T 404), tear index (T 414), zero span tensile index (T 233), opacity, brightness, and scattering coefficient (21). For treated chips that were refined to different freenesses for each treatment, handsheet properties were extrapolated for 100-mL-CSF pulps by regression from a series of three or four pulps (21). (Note: values for each pulp series gave the near linear relationships typically observed for nontreated chips.) The method for determining the total lignin content of pulp (Klason plus acid-soluble lignin) was that used previously (*21*). ❫

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