Improving softwood mechanical pulp properties with Ophiostoma piliferum

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ABSTRACT

Earlier reports showed that chip treatment with a nonstaining fungus, Ophiostoma piliferum, *reduced pulp extractive contents. Producers using the fungus have seen increases in paper strength properties. In the present work, reductions in extractive content of loblolly pine* (Pinus taeda) *wood meal by 1-, 3-, and 5-week treatments were similar to those reported earlier. Chips from the same wood were refined with a Sprout-Waldron atmospheric refiner to various CSF levels. Resultant pulps were used to make handsheets. Pulp and handsheet properties were evaluated using TAPPI Test Methods. Tensile strength increased marginally, while tear strength increased significantly. Treated chips yielded pulps with increased fiber lengths and decreased fines contents.*

During a pilot-scale trial to examine fungal treatment effects, southern pine chips were treated with O. piliferum *and aged for 5 weeks. Control chips were frozen during this time period. Chips were refined in a pressurized double-disc refiner, while an atmospheric double-disc refiner was used for secondary refining. TAPPI standard handsheets produced from* O. piliferum*–treated chips exhibited tensile and tear strengths greater than controls. Refining energy consumption decreased with fungal treatment. Increased fiber lengths and decreased fines contents were not evident in the pilot-scale pulps. Increased strength properties may result from greater intrinsic fiber strength and increased bonding.*

Application:

This paper confirms that chip treatment with Ophiostoma piliferum *removes extractives and offers an explanation for increased paper strength observed following fungal treatment.*

THE FUNGUS *OPHIOSTOMA PILIFERUM* (*O. piliferum*) is used to control pitch problems in paper production. Natural pitch is defined as low-molecular-weight oleophilic materials extracted from wood chips by neutral, nonpolar, organic solvents. Pitch contains triglycerides, fatty acids, diterpenoid resin acids, sterols, waxes, and other compounds, some of which are not well characterized *(1)*.

Cartapip 97® (Cp) is a commercially available albino strain of *O. piliferum* (Sandoz Chemicals Corp.) that does not stain wood as do most blue-stain fungi *(2)*. In addition to controlling pitch problems and preventing blue stain, Cp chip treatment yields products with improved strength and runnability characteristics *(3)*. The objectives of this work were to document any strength changes in mechanical pulps produced from loblolly pine *(Pinus taeda)* chips treated with Cp and explore probable causes.

MATERIALS AND METHODS

Wood source

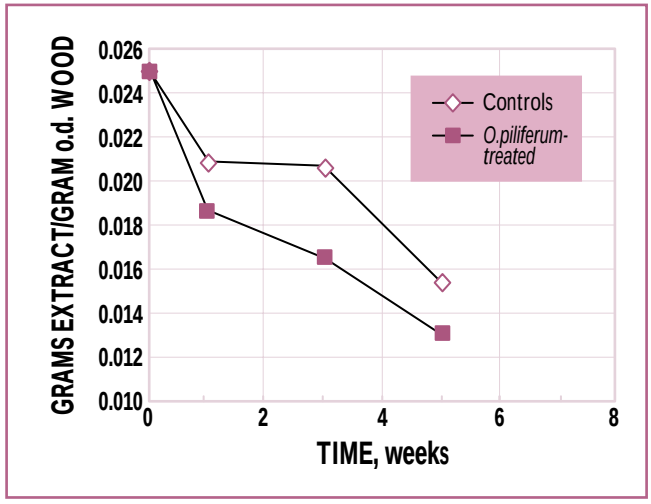
Laboratory scale. Three half-sib, loblolly pine trees were obtained in southeastern Georgia to use as the wood source for this project. Trees were cut into boards; the boards were sawn into blocks and then uniformly cut into chips by hand with a band saw. To increase uniformity, care was taken to remove knots and associated compression wood. Chips were stored frozen until used. All experiments were conducted with random mixtures of nonsterile chips from all trees, including early wood, late wood, juvenile, and mature wood.

Pilot scale. Approximately 3 tons of southern pine chips were obtained from a local mill for the pilot-scale trial. Half of the chips were placed in a freezer to be used as nonaged, nontreated controls. The other half of the chips were treated as explained later. As in the laboratory-scale work, nonsterile chips were used for the pilot-scale trial.

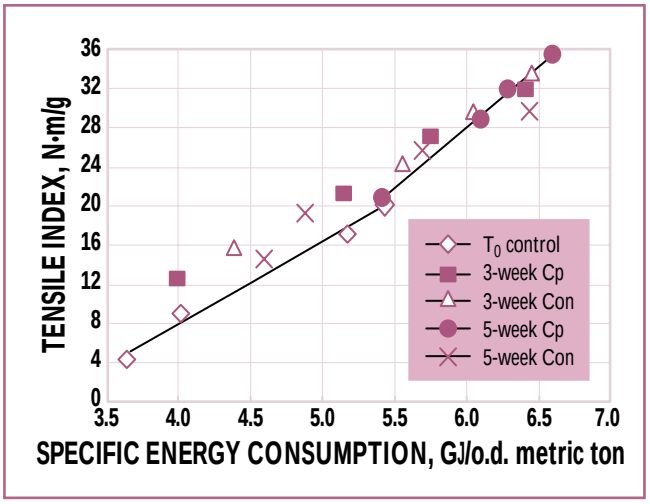
Inoculation and fungal growth period

Laboratory scale. Cp was grown at the Institute of Paper Science and Technology (IPST) in shake flasks. Fungal suspensions were centrifuged after 36 hours. Pellets from centrifugation were homogenized and diluted before being pipetted into plastic bags containing about 1200 g (wet weight) of wood. Chips were inoculated with 1.61 x 10^7 fungal cells for every 100 g of chips (wet weight) and incubated at 25°C for 1-, 3-, and 5-week periods. Nontreated controls were also incubated and aged for the same time periods. This was necessary to verify that dominant fungal growth present was wild-type *O. piliferum*. Significant growth of *O. piliferum* wild types occurred on all aged controls.

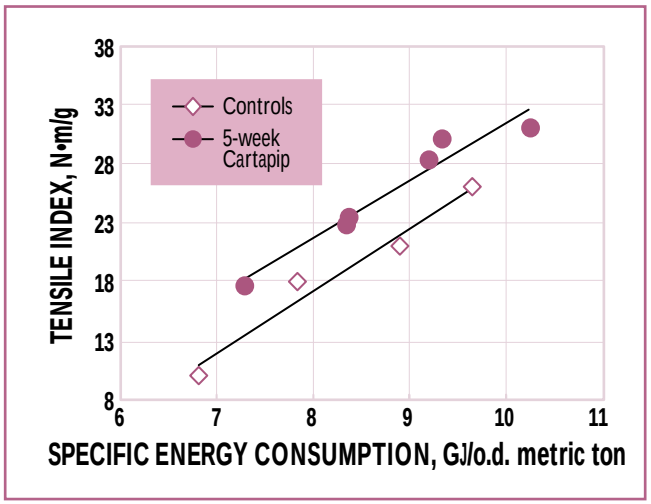
Pilot scale. Cp frozen product was diluted and sprayed with a garden sprayer onto the wood chips as each 55-gal drum was opened. Each drum of chips was treated with 5.61 x 10^{11} fungal cells of Cp. The fungus was allowed to grow for 5 weeks on



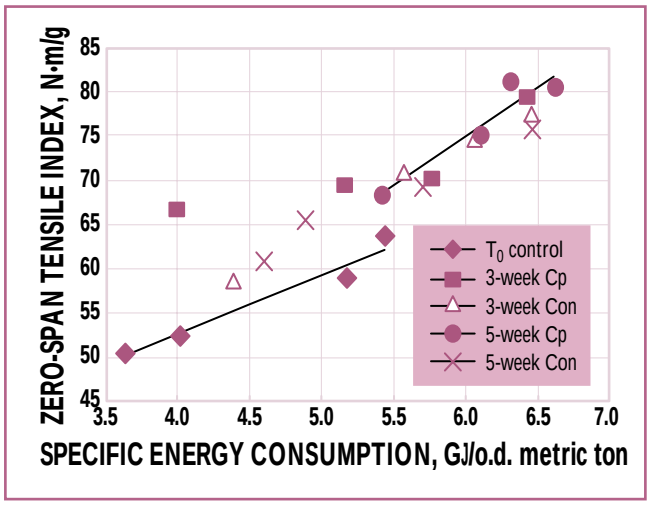
1. Extractive content vs. O. piliferum *residence time in wood*



2. Change in tensile index with increasing specific energy consumption for laboratory-scale experiments. (Cp in the legend refers to Cp treatments. Con in the legend refers to wild-type nontreated controls.)



3. Change in tensile index with increasing specific energy consumption for pilot- scale experiments



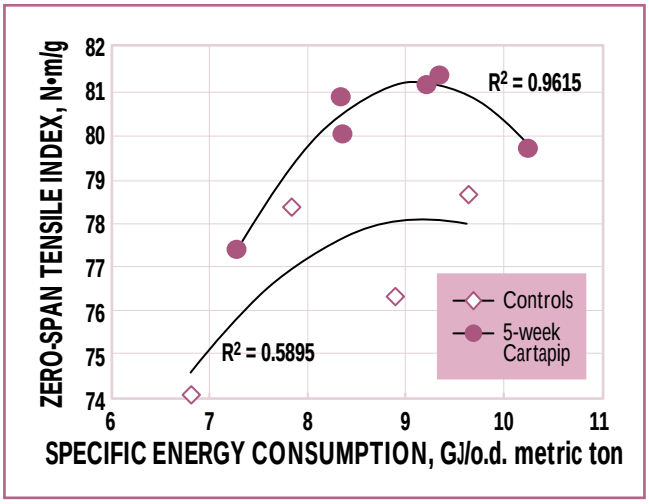
4. Zero-span tensile/specific-energy consumption relationship for laboratory-scale work. (Cp in the legend refers to Cp treatments. Con in the legend refers to wild-type nontreated controls.)

the wood chips in a small room, where temperature during the incubation period ranged from 18°C to 26°C. Aged controls were not included in this trial because previous analyses of aged controls, had produced effects similar to the Cp-treated chips. This wild-type control was necessary for the laboratory study to show that Cp produces a greater effect than the wild type without staining the wood, thereby creating additional cost.

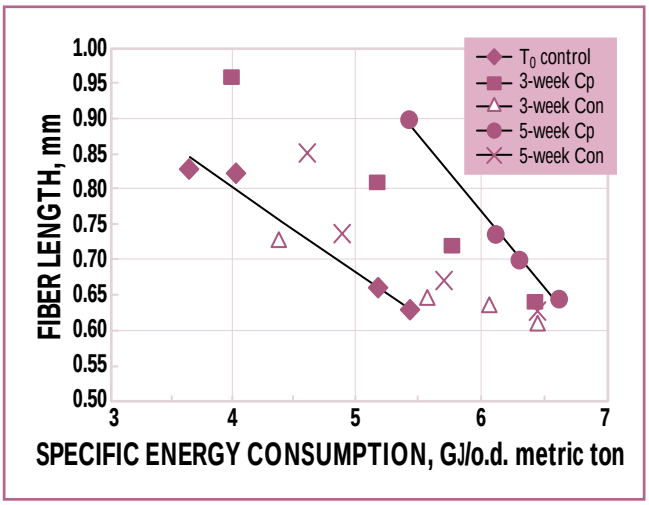
Refining

Laboratory scale. A Sprout-Waldron atmospheric refiner equipped with 12-in., D2B505-patterned, 440C stainless steel plates was used to refine the fungal-treated and non-treated chips. Motor load was measured with a Hall Effect power transducer and recorded with a reporting integrator. Chips were refined with peripheral water flowing into the refiner casing. Consecutive passes were carried out at 20% consistency. Refining was executed in 5–7 refining passes. Pulp was retained for latency removal, and handsheet production ranged in freeness from 250 to 30 mL CSF.

Pilot scale. Primary refining was carried out in an Andritz Sprout-Bauer Model 418 pressurized double-disc refiner at 2.76 bar for 2 min. Secondary refining took place in an Andritz Sprout-Bauer Model 401 atmospheric double-disc refiner. Refiner plate 36104 was used for



5. Zero-span tensile/specific-energy consumption relationship for pilot-scale work



6. Effect of increasing energy consumption on fiber length. (Cp in the legend refers to Cp treatments. Con in the legend refers to wild-type nontreated controls.)

each refining pass at 1200 rpm.

Dichloromethane extractions and handsheet production

Laboratory scale. Wood meal samples from chips used for refining were extracted with dichloromethane (dcm) according to TAPPI test method T 204 after grinding to 10-mesh size in a Wiley mill. Freeness testing, handsheet production, and physical- and optical-property testing were carried out according to TAPPI test methods. Fines contents and fiber lengths were estimated from Bauer-McNett classification *(4)*.

Pilot scale. Freeness testing, handsheet production, and physical- and optical-property testing were carried out according to TAPPI test methods. Fines contents and fiber lengths were estimated from Bauer-McNett classification *(4)*.

Data analysis

Multiple regression analyses were used to determine the contributions of independent variables to the dependent variables of interest for this study. Coefficients of the multiple regression were tested for significance at the 95% confidence level. Multiple regression analysis was performed with *O. piliferum* treatment, incubation time, and specific energy consumption (SEC) designated as independent variables. Freeness, fines content, tensile, density, tear, zero-span, fiber length, and scattering coefficient were used as dependent variables.

For the statistical analysis of the pilot-scale trial, only specific energy consumption and *O. piliferum* treatment were used as independent variables. Incubation time was not varied in this experiment.

The impact of individual independent variables was evaluated using simple linear regression. The slope from each regression equation was compared with those from other treatments by testing their homogeneity.

RESULTS AND DISCUSSION

Extractive removal

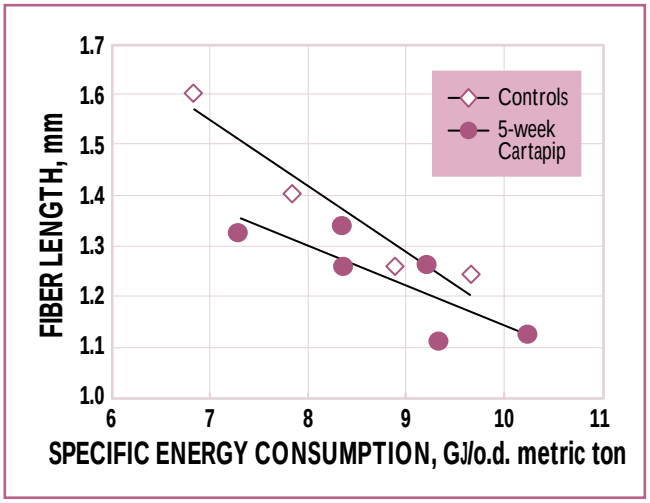
In the lab, extractive content after 1- and 5-week fungal treatment was 25% and 45.8% lower, respectively, than the extractives content of nonaged, nontreated control chips. The Cp also reduced the extractive content 7% and 16.5% more than wild- type fungi on nontreated, nonsterile wood chips aged for 1- and 5-week time periods, respectively **(Fig. 1)**.

Analyses of the wild-type fungi revealed that *O. piliferum* was the primary fungus present on the samples. The lower extractive levels in the Cp-treated chips show that the Cp is a rapid colonizer that out-competes wild-type fungi for extractive material in the wood.

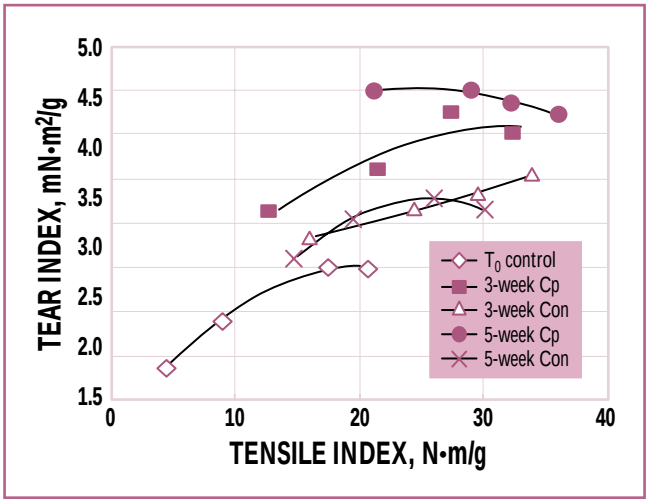
Pulp and handsheet properties

Results of the multiple regression analyses are presented first in this section and then used along with graphical analyses of the data to examine the presence of and extent of effects from Cp treatment. The coefficients of the multiple regression equations were all significant at the 95% confidence level and yielded strong multiple coefficients of correlation (see **Table I** for laboratory-scale results and **Table II** for pilot-scale results). Pulp and handsheet properties will be discussed in the order presented in Tables I and II.

Although the presence of the Cp was significant for the multiple regression equations describing freeness development for both the laboratory- and pilot-scale work, equivalent refining-energy input to treated and nontreated chips and resulting pulps did not produce pulps with meaningfully different freeness levels in either case. Fungal treatment did not reduce energy usage to produce these pulps in laboratory-scale refining runs. The pilot-scale trial, however, did reflect decreased energy requirements for pulp pro-



7. Effect of increasing energy consumption on fiber length for controls and O. piliferum*-treated pulps on the pilot scale*



8. Tear–tensile relationship for laboratory-scale work

duction.

Strength, power, and length relationships given in **Figs. 2–7** represent upper and lower limits for the multiple regression equations (lower limit: fungal treatment/incubation time equals zero; upper limit: Cp treatment/incubation time equals 5 weeks). Additional regression lines were not included in the graphs to maintain readability. These relationships are summarized in **Table III**.

The multiple regression analysis, with tensile index of handsheets as the dependent variable for the laboratory-scale study, showed that specific energy consumption and incubation time were the significant variables for tensile development (Table I). The tensile strength of the 5-week Cp treatment increases at a greater rate than the time-zero control for given increments of energy consumption (Fig. 2).

Handsheets from treated chips in the pilot-scale work exhibited increased tensile strength over the control pulps (Fig. 3). The multiple regression analysis also showed that the presence of Cp was significant.

On the laboratory scale, zero-span tensile strength increased with specific-energy consumption, incubation time, and Cp treatment (Table I). Differences in zero-span strength are probably related to increased fiber length and increased bonding. For mechanical pulps, an increase in zero-span with specific energy consumption was expected and has been documented by many researchers *(5)*. Increased intrinsic fiber strength is not likely, due to minimal yield losses and evidence that the fungal treatment does not break down lignin, cellulose, or hemicellulose *(6)*.

The zero-span tensile index of handsheets produced from Cp-treated pilot-scale chips increased (Fig. 5), as was observed in the laboratory-scale work. The multiple regression analysis, however, did not suggest that fungal treatment made a significant contribution to zero-span tensile index (Table II).

Fiber lengths in the pilot-scale-produced pulps (Fig. 7) were not as distinctively different as in the laboratory case (Fig. 6). Multiple regression analysis also showed that fungal treatment contributed significantly to fiber length on the laboratory scale (Table I) but not on the pilot scale (Table II). These differences could be related to the extent of fungal growth on the laboratory-scale chips, which was greater than growth on the pilot-scale chips. Pilot-scale *O. piliferum*-treated pulps at equal energy input levels are at most 0.275 mm shorter than the control pulps, while laboratory-scale Cp pulps are generally longer than the controls. Both the laboratory and pilot data appear to converge as energy input is maximized.

Generally, tensile, tear, and zero-span tensile strengths increased with *O. piliferum*-treatment over nonaged and aged controls. Multiple regression analysis showed that Cp contributed significantly to tear strength in the laboratory (Table I) and in the pilot (Table II) scale. Tear strength in laboratory-scale Cp-treated pulps increased by 18% and 35% over strengths of 3- and 5-week-aged control pulps, respectively (Fig. 8).

Tear strength increases for the pilot-scale trial were not as dramatic, as evidenced in the laboratory-scale work (Fig. 5). At pilot-scale tensile index values greater than 18 N·m/g, the tear strength of the Cp-treated sheet is greater than the control. At a tensile index value of 25 N·m/g, the tear reaches its maximum, which is 17% greater than the tear strength of the control.

As shown in **Figs. 8 and 9**, tear at a given tensile strength increased, indicating a probable increase in individual fiber strength, supported by zero-span data (Figs. 4 and 5). Another result that differed between the two trials was the extent of fungal growth. Chips in the laboratory-

Dependent variable, y	Regression equation	r^2
ln CSF, mL	$y = -2.986\ x_1 + 0.24\ x_3 + 8.629$	0.962
Tensile index, N·m/g	$y = 31.54\ x_1 + 0.83\ x_2 + 26.147$	0.969
Zero-span tensile index, N·m/g	$y = 26.87\ x_1 + 0.939\ x_2 + 3.895\ x_3 + 23.918$	0.918
Fiber length, mm	$y = -0.406\ x_1 + 0.0175\ x_2 + 0.095\ x_3 + 1.246$	0.876
Percent fines, P100–R200, P200	$y = 21.85\ x_1 - 1.212\ x_2 - 3.527\ x_3 + 16.827$	0.903
Tear index, mN·m²/g	$y = 1.327\ x_1 + 0.1182\ x_2 + 0.7973\ x_3 + 0.8403$	0.921

*x_1=SEC in GJ/o.d. metric ton, x_2=incubation time (0, 1, 3, or 5 weeks), x_3=Cp presence (0 or 1)

I. Multiple regression equations and r^2 for dependent variables from laboratory-scale refining runs (95% confidence level)*

Dependent variable, y	Regression equation	r^2
ln CSF, mL	$y = -128.05\ x_1 - 64.84\ x_2 + 1448.14$	0.934
Tensile index, N·m/g	$y = 3.701\ x_1 + 3.21\ x_2 - 11.47$	0.934
Zero-span tensile index, N·m/g	$y = 15.755\ x_1 + 0.297\ x_{12} - 0.876\ x_1^2 + 7.478$	0.794
Fiber length, mm	$y = -0.1076\ x_1 + 2.227$	0.797
Percent fines, P100–R200, P200	$y = 3.93\ x_1 + 1.99\ x_2 - 4.045$	0.778
Tear index, mN·m²/g	$y = 10.596\ x_1 + 0.95\ x_2 - 0.584\ x_1^2 - 41.022$	0.802

*x_1=SEC in GJ/o.d. metric ton, x_2=Cp presence (0 or 1), x_{12}= interaction term (SEC – Cp presence product)

II. Multiple regression equations and r^2 for dependent variables from pilot- scale refining runs (95% confidence level)*

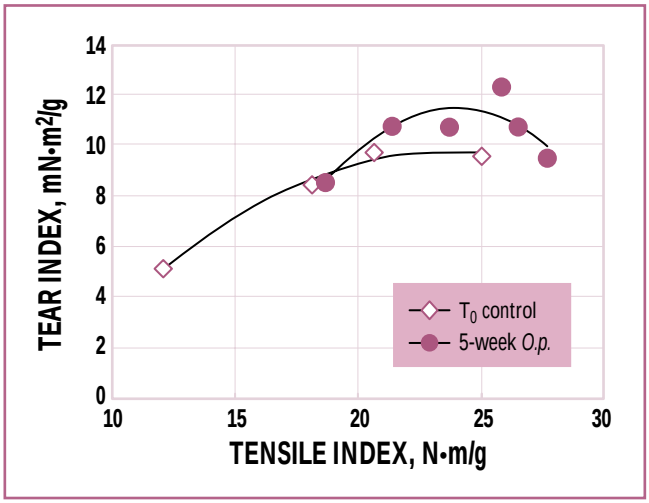
Dependent variable, y	Regression equation	r^2
Laboratory scale		
Tensile index, N·m/g (lower limit)	$y = 8.4\ x_1 - 25.6$	0.991*
Tensile index, N·m/g (upper limit)	$y = 12.4\ x_1 - 45.9$	0.997*
Zero-span tensile index, N·m/g (lower limit)	$y = 6.9\ x_1 + 24.9$	0.964*
Zero-span tensile index, N·m/g (upper limit)	$y = 11.2\ x_1 + 8.1$	0.898*
Fiber length, mm (lower limit)	$y = -0.12\ x_1 + 1.272$	0.976
Fiber length, mm (upper limit)	$y = -0.22\ x_1 + 2.066$	0.995*
Pilot scale		
Tensile index, N·m/g (lower limit)	$y = 5.5\ x_1 - 26.3$	0.969*
Tensile index, N·m/g (upper limit)	$y = 5.0\ x_1 - 18.2$	0.947*
Fiber length, mm (lower limit)	$y = -0.13\ x_1 + 2.45$	0.930*
Fiber length, mm (upper limit)	$y = -0.078\ x_1 + 1.93$	0.667

*Coefficient of determination is significant at 95% confidence level.

III. Simple linear regression equations and r^2 for strength, power, and length relationships

scale trial exhibited 95% Cp growth, while about 59% of the chips in the pilot-scale chips showed Cp growth. This may account for larger differences between Cp treatment and control sheet properties in the laboratory-scale study than differences between pilot-scale treatments and controls. Wild-type *O. piliferum* growth was found on 85% of the chips used as aged, nontreated controls in the laboratory-scale study. Wild-type *O. piliferum* was also present on treated chips in both studies. The laboratory-scale nonstaining *O. piliferum*–treated chips had 9% wild-type growth, while the pilot-scale chips had 14% wild-type growth.

Wild-type *O. piliferum* ultimately results in the same pulp and handsheet properties as induced fungal treatment but not to the same extent (Figs. 2, 4, 6, and 8). Examination of the laboratory-scale results suggests that fiber-to-fiber bonding and fiber length are significant contributors to the additions in strength. The pilot-scale trial might suggest that fiber length is not an essential component of the strength-forming mechanism



9. Tear–tensile relationship for pilot-scale work

present with fungal treatment. However, the dense fungal coverage visible in the laboratory-scale trial after 3 and 5 weeks was not that extensive in the pilot-scale trial. This visual observation was supported by the fungal growth analysis described in the preceding paragraph. As a result, fungal treatment may well yield pulps with increased fiber length. This could take place by allowing fibers to refine more easily by emptying resin canals, yielding free passages for steam diffusion and chip softening. Another possible explanation for the difference in fiber lengths may have to do with the refiners used for the studies. Laboratory-scale refiners have been shown to yield pulps with lower overall tear strengths than pilot-scale refiners *(7)*.

SUMMARY AND CONCLUSIONS

Fungal treatment effects of *O. piliferum* on handsheet strength properties were studied by producing refiner mechanical pulps from chips treated with fungus for varying time periods. Cp treatment decreased extractive levels and increased tear, tensile, and zero-span tensile strengths over and above growth with *O. piliferum* wild types. *O. piliferum*–treated chips refined with greater fiber lengths, which may partly account for increased tear strength in laboratory-scale studies. Intrinsic fiber strength is probably also a major contributor to increased tensile and tear strengths. Fiber and/or extractive chemical compositional changes may ultimately explain strength differences. The *O. piliferum*–treated pulps resemble chemimechanical pulps *(8)*, where strength properties increase while freeness changes minimally. Strength development in chemimechanical pulps is caused in part by larger numbers of acid groups on the fiber surface *(8)*. **TJ**

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KEYWORDS

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