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FUNGAL PRETREATMENT OF WOOD CHIPS FOR SULFITE PULPING[1]

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ABSTRACT

In this study, we examined the effect of fungal pretreatment of loblolly pine chips *Pinus taeda* prior to sodium- and calcium-based sulfite pulping. The pretreatment involved a 2-week incubation of the chips with two strains (CZ-3 and L-14807 SS-3) of the white-rot fungus *Ceriporiopsis subvermispora*. Focus was on the kappa number, yield, and liquor consumption. During sodium-bisulfite pulping, the fungal pretreatments reduced the kappa number by 27% with slightly lower pulp yield compared to the control, regardless of the strains. However, during calcium-based sulfite pulping, strains CZ-3 and SS-3 reduced the kappa number by 48% and 21%, respectively, compared to the control, but had the same pulp yield as that of the control. Liquor consumption was not appreciably affected by the fungal pretreatment. A simple kinetic model can be fit to the data that helps in the interpretation of the results. Thus, the fungal pretreatment is advantageous for sulfite pulping.

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INTRODUCTION

Increased public concern about the environment is having a rather large impact on the pulp and paper industry. The recently issued Environmental Protection Agency (EPA) regulations propose a dramatic reduction in allowable air and water discharges from chemical pulp and paper mills (1,2). In addition to kraft pulp mills, about 14 sulfite pulp mills are located in the United States (Table I) that need to reduce the adverse environmental impact of their effluents. The sulfite pulping process is used world-wide, especially in Europe. Paper-grade sulfite producers will soon be required to produce totally chlorine-free bleached pulp (3,4); therefore, the sulfite industry must look for alternative technologies to meet EPA regulations. Mills will also need to control costs in the form of chemicals and energy needs. Thus, new technology to reduce costs would also benefit the industry.

The sulfite cooking process includes several pulping processes that differ in the base used for the pulping chemicals as well as the pH of the resulting liquor. Historically, sulfite pulping has been dependent on calcium-based liquor of high acidity (pH 1-2). Several U.S. pulp and paper mills still rely on this technology. However, in the last half century, the more soluble bases of sodium, magnesium, and ammonium have come into use. Furthermore, the use of these bases has extended the possible pH range to less acidic conditions so that we now have bisulfite pulping at a pH of 3-5 and neutral sulfite semichemical (NSSC) at a pH of 7-9, which uses a sodium base.

Table I. Sulfite Mills in the United States (14)

Mill	Location	Production (ton/day)
Ketchikan Pulp Co.	Ketchikan, AK	600
ITT Rayonier	Fernandina Beach, FL	430
Great Northern Paper Co.	Millinocket, ME	540
Finch, Pryn, and Co.	Glen Falls, NY	350
Proctor & Gamble	Mehoopany, PA	N/A
James River Corp.	Camas, WA	450
Weyerhaeuser Paper Co.	Cosmopolis, WA	450
Scott Paper Co.	Everett, WA	500
ITT Rayonier	Port Angeles, WA	440
Wausau Paper Mills	Brokaw, WI	230
Cross Pointe Paper	Park Falls, WI	170
Badger Paper Mills	Peshtigo, WI	155
Georgia–Pacific Corp.	Port Edwards, WI	235
Weyerhaeuser Paper Co.	Rothschild, WI	200

Biopulping, defined as the fungal treatment of wood chips prior to pulping, may solve some problems associated with the traditional sulfite pulping processes. To date in our laboratory, we have focused only on the effect of fungal pretreatment of chips prior to refiner mechanical pulping. The 2-week process saved at least 30% electrical energy during refining, improved paper strength properties, and reduced the environmental impact of pulping (5–9). However, a study (10) done in collaboration with the Technical University, Vienna, Austria, and a recent review (11) indicate that fungal pretreatment also offers a great advantage for magnesium-based sulfite pulping. We explored this approach and extended the use of fungal pretreatment for sodium- and calcium-based sulfite pulping.

We hypothesized that the fungal pretreatment can be beneficial in several ways. The treatment can reduce the amount of cooking time, hence the energy that is required for pulping. The chemical demand can also be lessened due to prior degradation of the lignin by the fungus. It can also reduce the amount of bleaching that is required by selectively removing lignin from the wood by the fungus. Using these criteria, the effect of the best biopulping fungus *Ceriporiopsis subvermispora* (5) was studied on kappa number and pulp yield after sulfite pulping. Previous work with the fungal pretreatment emphasized reducing the environmental impact of the sulfite pulping processes (12).

EXPERIMENTAL PROCEDURE

Wood Chips

Freshly cut loblolly pine (*Pinus taeda* L.) pulpwood-size logs were obtained from the Talladega National Forest in Talladega, Alabama. The logs were debarked and chipped to a size averaging 16 mm. The chips were bagged in plastic bags and frozen until used to prevent the growth of contaminating microorganisms.

Fungus

Two strains (CZ-3 and L-14807 SS-3) of the best biopulping fungus *Ceriporiopsis subvermispora* were used based on their superior mechanical biopulping performance (7,13). These strains were obtained from the Center for Forest Mycology Research of the USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin. Cultures were continuously maintained in cereal culture and potato dextrose agar slants. Working cultures were prepared from the stock cultures as needed and refrigerated until used. Potato dextrose agar plate cultures were inoculated from a working culture and incubated at 27°C and 65% relative humidity for 10 days. Most results shown used the CZ-3 strain.

In preparing the liquid inoculum, potato dextrose broth (4.8 g) and yeast extract (1.46 g) were added to 200 ml of distilled water and mixed well; 100 ml of this medium was poured into two 1-L flasks. Each flask was autoclaved for 20 min at 121°C. After cooling to room temperature, each flask was inoculated with 10 plugs cut with a number 9 size cork bore from a 10-day-old potato dextrose agar plate of the fungal culture. The flasks were then incubated at 27°C at 65% relative humidity for 10 days. Prior to use, the flasks containing the fungal biomass were decanted and washed with sterile distilled water to remove excess medium from the fungal biomass. The fungal biomass was then placed in distilled water and blended in a Waring blender twice for 15 seconds, each time at high speed; distilled water was then added to the suspension to make the total volume 100 ml (stock). About 0.86 ml of CZ-3 stock and 1.23 ml of L-14807 SS-3 stock producing 0.0005% (5 g/ton of wood) inoculum of each strain (dry-weight basis) was diluted to 100 ml suspension with 15 g of unsterilized corn steep liquor (0.5% on a dry weight basis) from Corn-Products, Summit-Argo, Illinois, and an appropriate amount of sterilized water.

Chip Preparation and Bioreactor Inoculation

Frozen loblolly pine chips were thawed and thoroughly mixed to obtain uniform samples. Three static-bed reactors (13), each containing 1,500 g of chips (dry-weight basis), were decontaminated by using atmospheric steam for 10 min and then cooled to room temperature. These bioreactors were inoculated with 100 ml of inoculum suspension of each fungal strain as previously mentioned. One noninoculated bioreactor served as control. About 55% moisture (wet weight basis) in wood chips was maintained during fermentation. After receiving inocula, the bioreactors were shaken vigorously for uniform mixing. Each bioreactor was sealed and placed in a incubator at 27°C for 2 weeks and aerated with a specific aeration rate of 0.0227 L/L/min based on the volume of the reactor. At harvest, the fungus-treated and control chips were made into pin chips by processing them in a laboratory-scale atmospheric refiner equipped with "devil's teeth" plates. The chips were screened on an 3.2-mm mesh screen to remove fine material. The resulting chips averaged approximately 4 by 4 by 20 mm.

Sulfite Cooking

We pulped the chips in 90-ml bombs, which were indirectly heated in an oil bath. The temperature was ramped from the initial 70°C to the final temperature over a period given in Table II, which also details the typical liquor conditions used. In addition, the conditions given in Table II were varied to determine their effects on the process. For the calcium-based cooks, the liquor was obtained from Weyerhaeuser Paper Company (Rothschild, Wisconsin) and titrated according to Tappi test method T 604. Results of this analysis is shown in Table III. Note that the liquor decreased in potency with time, requiring reanalysis for the later runs. The sodium-based liquor was prepared as needed from reagent grade sodium hydrogen sulfite ($NaHSO_3$).

Table II. Nominal Cooking Conditions Used for Sulfite Pulping

Sulfite cooking conditions	Sodium-bisulfite	Calcium-acid sulfite
Ovendried charge of wood (g)	10	10
Total SO_2 on wood (%)	20.0	32.0
Combined SO_2 on wood (%)	10.0	9.7–10.9
Liquor-to-wood ratio	4.0:1	5.9–6.8:1
Maximum temperature (°C)	161	140
Time to temperature (h)	1.5	5
Total cooking time (h)	5–6	9–10

Table III. Titration Analysis of Calcium-Based Cooking Liquor

	Concentration (%)	
	Cooks 16 to 25	Cooks 26 to 28
Total SO_2	6.04	5.45
Free SO_2	3.98	3.80
Combined SO_2	2.06	1.65

After charging the bombs with the wood chips and liquor, they were sealed and placed in the oil bath. The initial temperature of the oil bath was 70°C. The temperature was then ramped and held according to the specific cooking conditions. During the cook, the bombs rotated end-over-end to facilitate mixing and uniform heat transfer. No gas relief (as is commonly done in the industry) was performed during the cook.

At the conclusion of the cook, we removed the bombs from the oil bath and quenched the reaction by cooling the bombs to 90°C for 10 min in a water bath. The contents of the bombs were then disintegrated in a Waring blender for 5 min. The resulting pulp was washed over filter paper, dried, and the yield and kappa number determined. The kappa number was determined using Tappi test method T 236. The spent liquor was analyzed using Tappi test method T 604 for residual pulping chemicals.

RESULTS AND DISCUSSION

We performed 28 sets of four cooks, varying the chemistry, temperature, cooking time, chemical charge, and ramp speed. Table II details the conditions for the cooks discussed in this work. Cooks under other conditions were performed but are not discussed in this paper. The effects of the fungal pretreatment on bleachability were previously presented (12).

Table IV. Summary of Sodium-Bisulfite Cooks at Cooking Times of 5.12 to 5.25 h

Treatment	Yield (%)	Kappa
Control	49.93 ± 0.39	31.27 ± 0.28
Pretreated (CZ-3)	48.16 ± 0.21	22.84 ± 0.98
Pretreated (SS-3)	48.20	22.90

Kappa and Yield

The objective of sulfite pulping is to remove the lignin from the wood, while leaving the cellulose and hemicelluloses unreacted. However, the sulfite process tends to somewhat degrade the carbohydrate portion of the wood as the lignin reactions occur. We speculated that the fungal pretreatment either consumes some of the lignin or modifies the lignin in such a way that it is easier to remove in the subsequent pulping process. The efficiency of the pulping is measured by the yield (dry weight of pulp per dry weight of wood charged) and the kappa number, a relative measurement of the residual lignin in the pulp. The goal of these cooks was to reduce the lignin sufficiently to produce a more bleachable pulp.

Table II gives the nominal cooking conditions used for both sodium-bisulfite and calcium sulfite pulping. Table IV summarizes the results for the sodium-bisulfite cook at a cooking time of 5.12 to 5.25 h. The fungally treated chips resulted in a slightly lower yield and a 27% reduction in kappa number. Both of these differences are statistically significant ($p < 0.01$ and $p < 0.0001$ for the yield and kappa, respectively). During the cook, both the yield and the kappa decreased with time. The yield decrease was due both to the dissolution of lignin as well as the concurrent attack on the carbohydrates. The fungal pretreatment in this case seemed to accelerate the pulping process but did not seem to change the selectivity between lignin and carbohydrates. Figure 1 shows the kappa numbers for each of the pulping treatments at two different times. An additional 30 min of cooking

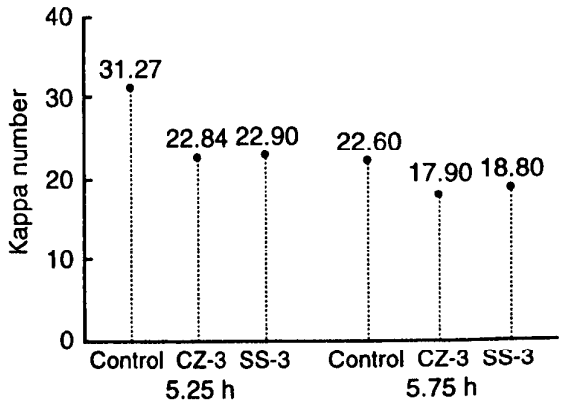

Fig. 1. Pulp kappa numbers for sodium-bisulfite cooking

Table V. Summary of Calcium-Acid Sulfite Cooks at Cooking Times of 9.50

Treatment	Yield (%)	Kappa
Control	47.63 ± 0.15	26.75 ± 0.70
Pretreated (CZ-3)	47.70 ± 0.12	13.72 ± 1.78
Pretreated (SS-3)	47.80 ± 0.69	21.05 ± 0.84

reduced the kappa number of the control to the same level as that of the fungally treated chips. As the reaction continued, the differences between the control and the fungal-treated chips decreased. This could be due to the difficulty in removing the residual lignin in this pulping process. Finally, differences were not seen between the two fungal strains; both CZ-3 and SS-3 gave comparable yields and reduced kappa numbers.

Table V summarizes the results for the calcium-acid sulfite cooks at a cooking time of 9.50 h. Again, the fungal treatment resulted in a significant reduction in the kappa number. For strain CZ-3, the kappa number was reduced by 48%, from 27 to 14. The SS-3 strain reduced the kappa number by 21%. These differences are highly significant ($p < 0.0001$). In contrast to the sodium-based cooks, the control and the fungally treated chips had the same yield. This indicates that calcium-based sulfite pulping is more selective towards lignin degradation. Figure 2 shows the kappa number for each treatment at 9.50 and 10.00 h. An additional 30 min of cooking was also required for the control to reach the same kappa number as the biotreated chips (SS-3). As in the case of the sodium-based cooking, the difference between the control and the treatments was reduced to approximately 20% in kappa.

As a result of the timing of the experiments, the potency of the cooking liquor differed between several of the calcium-based cooks through degradation of the chemicals (Table III). Statistical analysis of the data showed that the change in liquor did not produce any statistically significant differences in the results.

Cooking Liquor Consumption

In addition to reductions in the kappa number of the pulp, the possibility also existed that the fungal treatment would reduce the amount of pulping chemicals that were consumed. Figure 3 summarizes the amount of liquor charged and consumed for the control and for CZ-3 treated chips with the calcium-based cooking. Each data bar represents the average of two cooks. The same amount of cooking chemicals was consumed in each case: approximately 0.24 g SO_2/g oven-dried pulp. Thus, the fungal pretreatment did not increase the amount of pulping chemicals needed for this process. Remember that the treatment resulted in a 48% reduction in the kappa number, which would presumably make the pulp easier to bleach. This supports the theory that the fungal treatment modifies the lignin in the pulp, making it easier to remove in the subsequent pulping step.

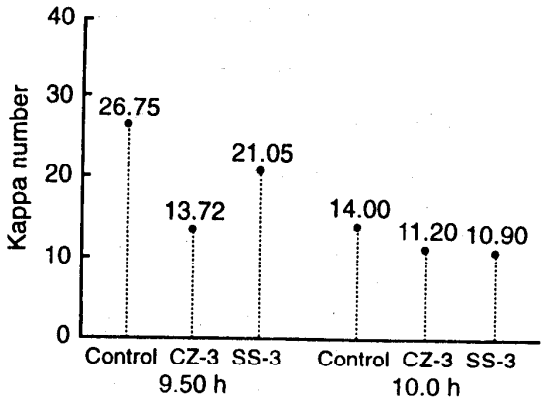

Fig. 2. Pulp kappa numbers for calcium-acid sulfite cooking

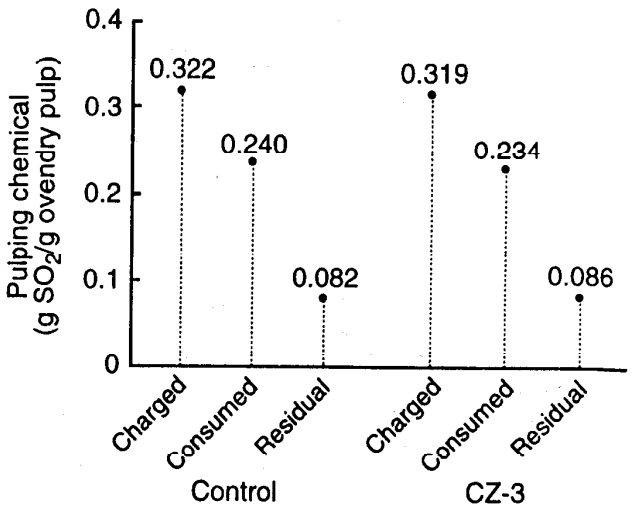

Fig. 3. Comparison of the consumption of pulping liquor by cooks

Kinetic Modeling

It is possible to fit the data acquired to a simple kinetic model of the process. With this model, we can discuss the changes that occur in the reaction in general way. For this model, wood is considered to be a two-component system consisting of lignin, L, and carbohydrates (cellulose and hemicellulose), C. L and C are the fraction of each of the two components in the wood. The rate of dissolution of each of these components is assumed to be an nth order reaction.

Because the data being fit are at a constant temperature (that is, after the completion of the ramp to the final cooking temperature), temperature effects on the kinetics are not considered. Also, the effect of liquor concentration is not considered in this simple model. The model is therefore given by the following:

$$dL/dt = k_L(L - L_0)^{n_L}$$

$$dC/dt = k_C(C - C_0)^{n_C}$$

where k_L and k_C are the reaction rate constants; L_0 and C_0 are the amount of lignin and carbohydrates that are "unreactive;" and n_L and n_C are the reaction orders; and t is the time of the reaction. Because of our simplifying assumptions, these two equations can be directly integrated to give

$$L = [k_L(1 - n_L)t + c_L]^{(n_L - 1)} + L_0$$

$$C = [k_C(1 - n_C)t + c_C]^{(n_C - 1)} + C_0$$

where c_L and c_C are constants of integration. These values may then be used to calculate the resulting yield and kappa number as

$$\text{Yield} = L + C$$

$$\text{Kappa} = 650\ L/(L + C)$$

Using data from the cooks, the various parameters of the model were fit by minimizing the weighted sum of squared error. As a result of the range of the experimental data, this produces a model that describes the behavior of the pulping process near the end of the cook. That is, the model is only valid in the region of 50% yield in both cases. Tables VI and Table VII summarize the fitted parameters to this model. The values given for L and C at the given time in each table are related to the constants of integration (c_L and c_C) and are essentially the initial conditions of the model. Figures 4, 5, and 6 display the model fit for the sodium-bisulfite cooks. Figures 7, 8, and 9 display the results for the calcium-acid cooks.

We noted in the sodium-bisulfite pulping that the fungal pretreatment had little effect on the relative values of the kappa number when plotted against yield. Figure 4 shows that both the control and the fungally treated pulp were similar in their relationship of kappa with the yield. That is, for a given yield, both the control and the fungally treated had similar kappa numbers. However, the fungal treatment allowed the lower kappa and yield to be reached with less cooking time. Figures 5 and 6 show that for any given time, both the yield and the kappa were lower for the treated chips, indicating a greater extent of cooking.

The parameters that are fit to the simple kinetic model also show some differences in the case of sodium-bisulfite pulping (Table VI). For the biosulfite pulping, the value of L (3h) (the amount of lignin remaining after 3 h of cooking) is significantly less than the control. Likewise, the amount of carbohydrate, C (3h) , is slightly less for the treated sample at this time. This indicates that for the treated chips, the extent of reaction is greater than the control at this time. Interestingly, the rate constant for both lignin and carbohydrate degradation is less for the treated chips in this case. However, it is generally understood that as cooking proceeds, the lignin becomes more difficult to remove. This

Table VI. Best Fit Parameters to the Kinetic Model for Sodium-Bisulfite Cooks

Parameter	Control	Fungal treatment
L_o	0.0160	0.0122
k_L	0.0550	0.0440
n_L	3.70	3.23
$L(3h)$	0.155	0.089
C_o	0.468	0.458
k_C	0.0600	0.0485
n_L	2.81	3.00
$C(3h)$	0.592	0.554

Table VII. Best Fit Parameters to the Kinetic Model for Calcium-Acid Sulfite Cooks

Parameter	Control	Fungal treatment
L_o	0.00507	0.00709
k_L	0.0160	0.0366
n_L	1.94	2.46
$L(7h)$	0.0635	0.0724
C_o	0.486	0.480
k_C	0.0507	0.0493
n_L	2.89	3.00
$C(7h)$	0.496	0.497

may indicate that the extent of reaction is such in the case of the fungally treated chips; the reaction has proceeded to the point where the more difficult lignin is being removed.

In contrast to the sodium-bisulfite pulping, we noted that the calcium-acid cooks had a significant effect on the relative values of kappa and yield. As previously mentioned for calcium-based cooking, the two strains of fungus showed distinctly different results. Only the CZ-3 data were used to fit the model. Figure 7 shows that for yields from 46% to 50%, the fungally treated chips had a kappa number approximately 10 points less than the control for the same yield. Figure 8 shows that the at the same cooking time, the kappa number for the fungally treated chips was approximately 10 points less. Figure 9 shows the evolution of the yield with time. As can be seen, there is little difference between the yields for the control and the treatment. In fact, at 9.5 h, the lines representing the models cross and both predict the same yield. As can be seen in Figure 8, the kappa is still significantly less than the control at this point.

Table VII gives the fitted model parameters for the calcium-acid sulfite process. As can be seen, there is little change in the parameters representing the degradation of the carbohydrates. This indicates that the fungal pretreatment has little

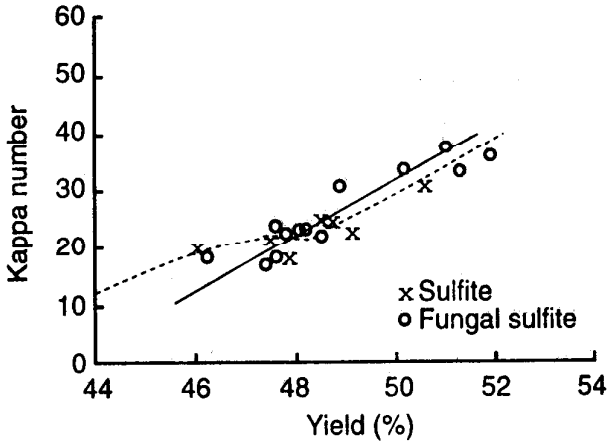

Fig. 4. Kappa compared with yield for sodium-bisulfite pulping. (Dotted and dashed lines show the fit of the respective models for the control and biosulfite pulping.)

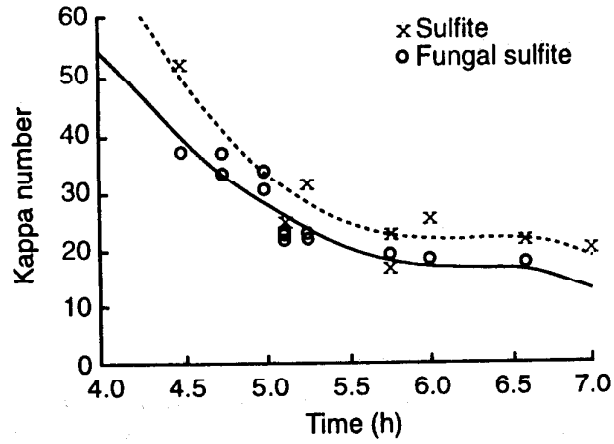

Fig. 5. Kappa compared with time for sodium-bisulfite pulping.

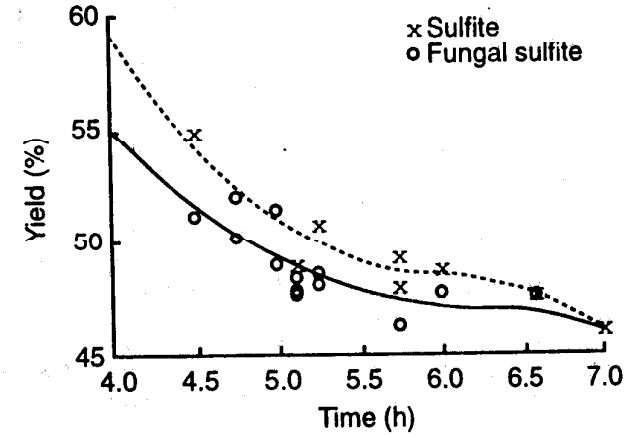

Fig. 6. Yield compared with time for sodium-bisulfite pulping.

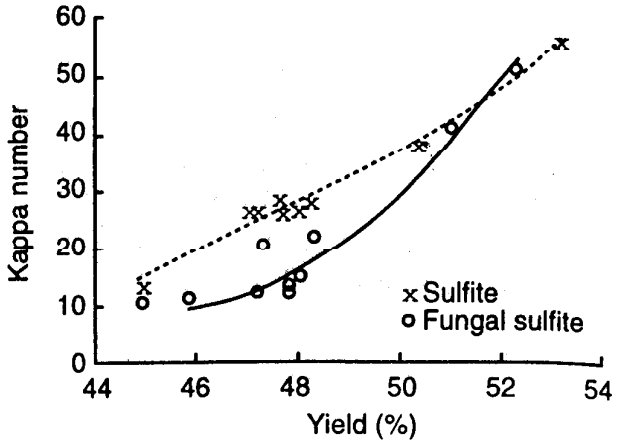

Fig. 7. Kappa compared with yield for calcium-acid sulfite pulping.

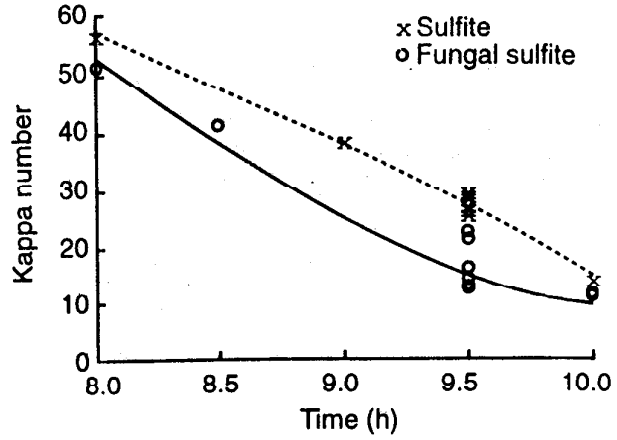

Fig. 8. Kappa compared with time for calcium-acid pulping.

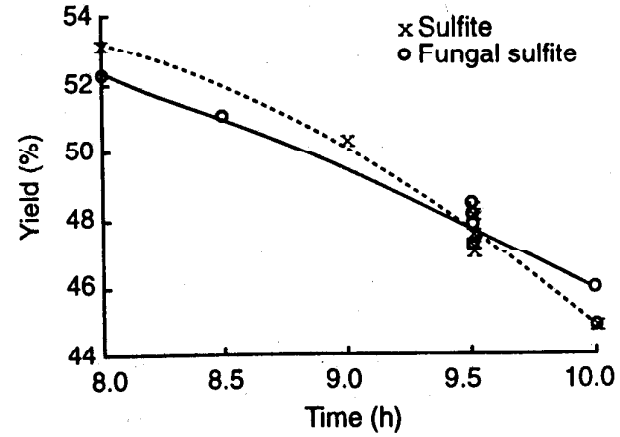

Fig. 9. Yield compared with time for calcium-acid acid pulping.

effect on the carbohydrates and their subsequent reactions in sulfite pulping according to this model. However, the parameters for lignin degradation did change, and the fungal pretreatment resulted in an increase in the reaction rate constant, k_L, and the order of the reaction, n_L. Both of these effects would lead to an increase in the rate of lignin degradation.

CONCLUSIONS AND RECOMMENDATIONS

Results show that the fungal pretreatment of the wood had several beneficial effects on the pulping process. The fungal pretreatment significantly reduced the kappa number of the resulting pulp in both the bisulfite and acid sulfite cooks. In the case of the acid sulfite cooks, this was done without adversely affecting the yield. For the same cooking time, the pulp can be cooked to a lower kappa number. Alternatively, shorter cooking times can be used to reach the same kappa number as the control, thus increasing throughput and reducing energy consumption. We also noted that in the case of the acid sulfite process, the amount of shives in the pulp was qualitatively reduced compared to the control. This resulted in a greater screened yield for the biosulfite process. The consumption of pulping liquor was not significantly affected. Finally, a simple kinetic model can be fit to the data to help in the interpretation of the results. Previous work has shown that the fungal pretreatment has several beneficial aspects, including color improvement, lower effluent toxicity, and improved bleaching response (12).

Future work needs to be directed at further assessing the effects of the biotreatment on the pulp and optimization of the process. Brightness and color stability of the resulting pulp need to be investigated It is expected that the significantly decreased lignin content will produce a pulp that is more resistant to color reversion. Strength and mechanical properties of the pulp also need to be explored. The fungus used, *C. subvermispora*, is the best mechanical biopulping fungus discovered to date. However, it may not necessarily be the best fungus to use for biochemical processes. Thus, additional fungi need to be screened for their biochemical pulping efficacy. Finally, the biopulping process needs to be extended to additional species as well as different pulping chemistries, including neutral sulfite and kraft. Sophisticated models, such as those that account for changes in the liquor composition, would aid in the interpretation and optimization of the biopulping process.

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