KRAFT PULP AND PAPERMAKING PROPERTIES OF *PHANEROCHAETE CHRYSOSPORIUM* DEGRADED RED OAK

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(Received June 1989)

ABSTRACT

The kraft pulp and papermaking properties of *Phanerochaete chrysosporium* degraded red oak (*Quercus rubra*) were investigated. *Phanerochaete chrysosporium* was grown on rye media, and the rye spawn was used to establish mycelia growth on glucose-supplemented red oak wood chips for 0-, 10-, 20- and 30 days, respectively. Kraft pulps were produced from biodegraded and nondegraded red oak wood chips and evaluated for pulp yield, pulp refinability, and handsheet properties.

Results showed that as time (0, 10, 20, and 30 days) of vegetative mycelial growth on wood chips increased, significant changes in pulp yield, kappa number, water retention value, and handsheet properties occurred. At a given pulp kappa number, higher kraft pulp yields were obtained from wood chips fungally degraded for 30 days (3–5% yield advantage) compared to pulps obtained from non-degraded red oak wood chips. Data indicated that pulps prepared from *P. chrysosporium* degraded red oak wood chips were more hydrophylic, responded faster to beating, and at comparable freeness levels had higher tensile, burst, and fold properties than pulps prepared from nondegraded wood. Sheet opacity was not affected by fungal degradation. Handsheets made from fungally degraded wood, however, showed marked reductions in brightness as fungal incubation time increased.

Keywords: Red oak, *Phanerochaete chrysosporium,* papermaking.

INTRODUCTION

Lignin provides the structural support in wood and it consists of a chemically resistant molecular web of 9 random inter-unit linkages of phenyl propane units (Browning 1963; Sjostrom 1981). The lignin macro-molecule may be modified or weakened somewhat through demethoxylation and oxidative cleavage of some of the beta-o-4 arylglycerol-beta-aryl ether bonds by the action of specific white rot enzymes (Chen et al. 1982, 1983). This condition may make the lignin molecule

Wood and Fiber Science, 23(3), 1991, pp. 316–327

more susceptible to the action of chemical agents used in the pulping industry. In the pulping industry, wood with a weakened lignin moiety may be converted to pulp by using less energy and chemicals. The production of an improved pulp may also be possible (Eriksson and Kirk 1980; Eriksson and Vallander 1980). In order to take full advantage of the potential benefits identified with controlled fungal degradation of wood chips, studies are needed to investigate the combination of selective fungal degradation of wood chips with existing chemical pulping processes. At present, most research has been concentrated on the identification, isolation, purification and characterization of fungi and fungal enzymes that selectively degrade lignin (Tien and Kirk 1984; Setliff and Eudy 1981). The pulping potentials of some of these fungi and their enzyme systems have been evaluated, but only with mechanical pulping processes (Eriksson and Kirk 1980; Eriksson and Vallander 1980; Myers et al. 1988; Setliff et al. 1983; Bar-Lev et al. 1982).

The objective of this study was to evaluate the kraft pulp and papermaking properties of red oak (*Quercus rubra*) wood chips degraded by *Phanerochaete chrysosporium*. *P. chrysosporium* was selected because it is extensively studied and chemically characterized ligninase producing white rot fungus (Tien and Kirk 1984). The kraft process was selected because it accounts for over 70% of the world's annual pulp production and it is the most versatile method of wood delignification known to date (Sjostrom 1981).

MATERIALS AND METHODS

Red oak (*Quercus rubra*) wood chips were obtained from a local sawmill, and the fines, oversized wood chips, and knots were discarded after screening. The wood chips were innoculated with rye spawn of isolate BKB 1767 of *Phanerochaete chrysosporium* and incubated according to procedures described by Oriaran et. al. (1989). Glucose-supplemented red oak wood chips were used and incubated with fungi for periods of 0, 10, 20, and 30 days prior to pulp evaluation studies.

Five hundred grams (based on oven-dry weight) of either fungally degraded or nondegraded wood chips were placed in a laboratory digester and pulped according to the conditions described in Table 1. Time at temperature and effective alkali levels were varied to produce pulps ranging in pulp yield, kappa number, and papermaking characteristics.

Pulp yields were determined based on the oven-dry weight of wood chips initially charged to the digester. The residual carbohydrate in the pulp was assumed to be equal to one hundred less the percent lignin in pulp. In making this assumption, minor constituents in pulps such as resins and residual metal salts were neglected. A total of 96 cooks were prepared from control and biodegraded wood chips using a $2 \times 3 \times 4$ (effective alkali concentration, cooking time and fungal incubation time) full factorial design with 4 replications per treatment (Steel and Torrie 1980).

Pulps were evaluated for kappa number (Tappi Standard T-236-60), water retention value (Jayme 1958), Bauer-McNett fiber classification (Brandon 1979), and pulp refinability (Tappi standard T-200-ts-66). Pulp samples were withdrawn from the valley beater after beating intervals of 5, 15, 30, 45, and 60 minutes, respectively, for Canadian standard freeness (Csf) testing (Tappi standard T 227 m-58) and handsheet formation (Tappi standard T 205 os-71). Handsheets were conditioned and tested for tensile, tear, burst, MIT fold, brightness, and opacity.

TABLE 1. *Kraft pulping conditions used for biodegraded red oak wood chips.*

Minutes to temperature	40
Minutes at temperature	20, 80, 140
Minutes to cool down	30
Pulping temperature (C)	173
Wood chips charge (grams over dry basis)	500
Effective alkali (%)	12, 16
Sulfidity	25
Liquor to wood ratio	5:1

Simple linear regression was used to relate measured handsheet strength values to pulp freeness. Strength values at 300, 400, 500, and 600 Csf levels were estimated for different beating times, cooking times, and effective alkali concentrations. Means separation was accomplished by the Student-Newman-Keuls (SNK) procedure using the Statistical Analysis Systems (S.A.S. Institute 1983).

RESULTS AND DISCUSSIONS

Pulp yield and kappa number

Statistical differences ($P < = 0.05$) in total pulp yields and kappa number values were found among kraft digested fungally degraded red oak wood chips and the controls (Table 2). Pulp yields increased as fungal incubation time increased from 0 to 30 days. An increase in pulp yield with fungal incubation time occurred regardless of the cooking time or effective alkali concentration used.

As expected, a progressive decrease in kappa number was associated with an increase in either fungal incubation time, cooking time, or effective alkali concentration. Data in Table 2 suggest that it would take less time to kraft cook fungally degraded wood chips to a given kappa number compared to the time required to cook nondegraded wood chips. With but a few exceptions, there were

TABLE 2. *Total pulp yield (%) and average kappa number values(*).*

	Effective alkali (%)	Cooking time (hours)	Pretreatment Time (days) 0	10	20	30
Pulp yield	12	1	62.9aA	63.9aB	65.0aC	69.9aD
		2	57.4bA	59.9bB	62.3bC	65.2bD
		3	58.0bA	57.6cA	60.7bC	63.0bC
	16	1	48.9aA	49.4aA	51.1aB	53.1aC
		2	47.4aA	48.4aA	49.9aB	52.5aB
		3	43.4bA	44.0bA	47.3bB	49.1bC
Kappa number	12	1	135aA	134aA	132aB	129aC
		2	128bA	127bB	125bB	122bC
		3	122cA	122cA	120cB	117cB
	16	1	81aA	81aB	76aC	74aD
		2	69bA	68bA	66cB	64bC
		3	52cA	51cA	49cB	47cC

* Based on 2 replicate cooks. Means with the same small letter (a, b, c) within each treatment column and means with the same capital letter (A, B, C, D) within each treatment row indicate that no significant differences occurred at the 0.05 level of probability (SNK).

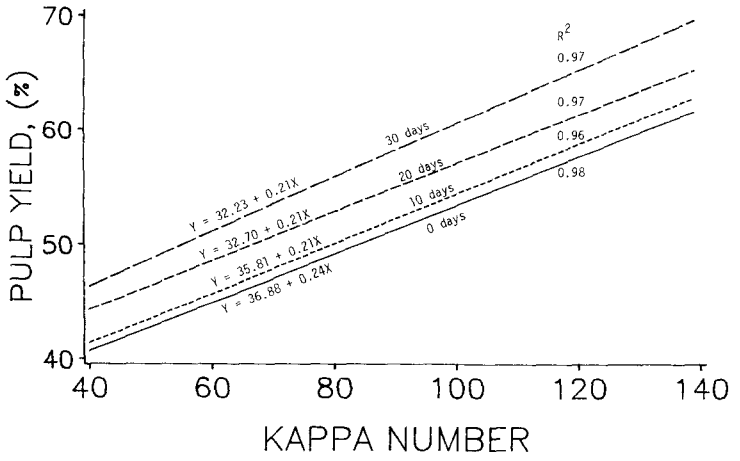

FIG. 1. Pulp yield as a function of kappa number for red oak chips subjected to fungal incubation of 0, 10, 20 and 30 days.

no statistical differences ($P < = 0.05$) between pulp yield and kappa number values of pulps prepared from control and kraft pulps fungally degraded for a period of 10 days.

Pulp yields from sound and fungally degraded red oak wood chips were compared at the same kappa numbers (Fig. 1). Wood chips that were fungally degraded for 30 days and then kraft digested exhibited the highest pulp yields and the lowest kappa numbers compared to the pulps obtained from wood fungally degraded for periods of 10 and 20 days. Pulps obtained from wood chips fungally degraded for 20 and 10 days, respectively, exhibited intermediate yields and kappa numbers, whereas pulps from nondegraded wood chips exhibited the lowest pulp yields and the highest kappa numbers. For example, at comparable kappa numbers, pulps prepared from wood chips fungally degraded for 30 days exhibited about a 5% yield advantage over pulps prepared from nondegraded wood chips (Fig. 1).

Two explanations may be given to describe the reasons why differences in pulp yield and kappa number values occurred for pulps prepared from kraft digested sound and fungally degraded wood chips. First, it may be possible that there was improved cooking liquor penetration into the biodegraded wood chips during kraft digestion. The removal of woody mass (4%, 15% and 18%) prior to pulping (10, 20, and 30 days fungally degraded wood, respectively) (Oriaran et al. 1989), may have left larger and/or additional pore structures capable of enhancing access of the cooking liquor to the cell-wall components. Undoubtedly, the efficiency of the kraft pulping process was improved, since the initial delignification phase in kraft pulping process is diffusion controlled (Sjostrom 1981). This condition could explain, in part, the observed variations in kappa number obtained for biodegraded wood pulp. Secondly, the pretreatment of wood chips with a ligninase producing fungus increased the holocellulose to lignin (H/L) ratio of wood chips charged into the digester. An earlier study (Oriaran et al. 1989) showed that the klason lignin content of wood chips degraded by *P. chrysosporium* was reduced by 5.17%, 4.20%, and 1.5%, respectively, after 30, 20, and 10 days, of fungal incubation time. In addition, the ratios of holocellulose to lignin determined prior

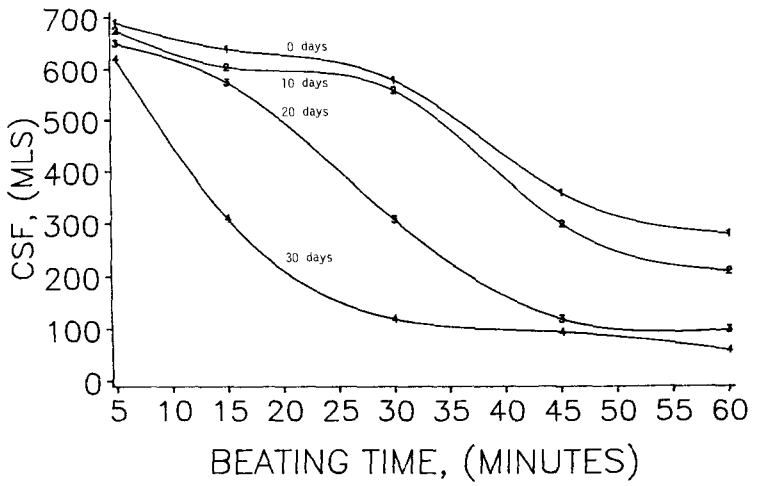

FIG. 2. Canadian Standard Freeness as a function of beating time for red oak chips subjected to fungal incubation of 0, 10, 20 and 30 days.

to digestion of wood chips were 3.2, 3.4, 3.8 and 4.0 for 0, 10, 20, and 30 days of fungal pretreatments, respectively. Therefore, a digester charge of degraded wood chips contained a higher proportion of carbohydrates than a digester charge of nondegraded wood chips. Stoichiometrically, the proportion of chemical components in wood for sound and fungally degraded wood chips was not the same per unit digester charge. A loss in lignin and holocellulose had occurred for fungally degraded wood prior to digester loading.

REFINING PROPERTIES

Less beating time was required to reach a given freeness for pulps obtained from fungally degraded wood chips in comparison to the control pulps obtained from nondegraded wood chips (Fig. 2). A functional relationship between pulp freeness development and fungal incubation time was established by a linear regression analysis (Table 3). There was a high level of association between fungal incubation time and pulp freeness development. In some cases, over 90% of the variation in pulp freeness development could be explained by incubation time (Table 3).

The relative amounts of holocellulose and lignin components in the pulp fibers could account for differences in pulp refinability for fungally degraded and nondegraded wood. Pulps obtained from fungally degraded chips had a lower kappa number (lignin content) and a higher carbohydrate content than did pulps prepared from sound wood chips (Table 2). As a general rule, the lower the lignin content (or kappa number) of a pulp, the faster the rate of refining (Brandon 1979).

The results observed in this study wee in general agreement with the findings reported by Eriksson and Vallander (1980), Setliff et. al. (1983) and Myers et. al. (1988), who prepared mechanical pulps from fungally degraded wood chips. Eriksson and Vallander (1980) showed that the longer the treatment of spruce wood chips with *Sporotrichum pulverulentum,* the lower the fiberizing energy required to refine TMP fibers to a given freeness.

TABLE 3. *Regression analysis of pulp freeness (Csf) as related to fungal incubation time.*

Form of equation: Y = a + bx (x = pretreatment time)					
Effective alkali (%)	Beating time (min)	Cooking time (h)	a	b	R^2
12	5	1	711.00	−0.90	0.85
12	5	2	702.00	−0.72	0.90
12	5	3	701.00	−1.40	0.25
16	5	1	722.00	−2.80	0.78
16	5	2	701.00	−2.40	0.99
16	5	3	694.00	−2.35	0.97
12	15	1	683.00	−1.76	0.69
12	15	2	690.00	−3.75	0.94
12	15	3	661.00	−2.40	0.42
16	15	1	720.00	−8.00	0.73
16	15	2	676.50	−4.10	0.95
16	15	3	685.50	−10.20	0.76
12	30	1	629.00	−1.60	0.44
12	30	2	670.00	−5.75	0.90
12	30	3	649.00	−7.10	0.96
16	30	1	704.00	−11.10	0.94
16	30	2	688.00	−10.70	0.98
16	30	3	636.80	−16.32	0.92
12	45	1	664.00	−5.60	0.93
12	45	2	700.00	−11.75	0.89
12	45	3	688.20	−15.43	0.93
16	45	1	637.00	−10.80	0.89
16	45	2	601.50	−11.35	0.88
16	45	3	365.00	−9.75	0.92
12	60	1	684.00	−9.60	0.86
12	60	2	699.00	−14.37	0.88
12	60	3	682.00	−17.28	0.91
16	60	1	566.00	−13.10	0.83
16	60	2	544.50	−14.05	0.90
16	60	3	278.00	−7.70	0.97

The water retention values of pulp fibers obtained from fungally degraded wood chips were determined and were found to be significantly higher than WRV of pulp fibers from sound wood chips (Table 4). This observation suggests that biodegraded pulp fibers were able to swell more readily during refining. This condition could explain why pulps prepared from biodegraded wood fibers responded more readily to beating.

Cell-wall separations resulting from fungal degradation could also explain why

TABLE 4. *Water retention values (%) of pulps (*).*

Incubation time (days)	Water retention value (WRV)
0	95a
10	101a
20	113b
30	125c

* Pulp fractions were digested for 3 hours at 16% effective alkali. Each measurement is an average of two replicates. Means with the same letter within a column indicate that no significant difference was observed at the 0.05 level of probability (Student-Newman-Keuls test).

TABLE 5. *Bauer-McNett classification of pulps.*

Screen (*) mesh	Screen opening (in.)	Incubation time (days) 0	10	20	30
		Pulp fractions retained (%)**			
+20	0.0328	18a	16a	10b	9b
−20, +35	0.0164	30a	28a	32b	31b
−35, +65	0.0082	34a	34a	37b	40c
−65, +150	0.0041	17a	19a	21b	22b
Sedimentation value (ml)		190a	246b	362c	390d

* + indicates that pulp fibers were retained and − indicates the pulp fibers passed through screen openings.
** Pulp fractions were digested for 3 hours at 16% effective alkali. Each measurement is an average of two replicates. Means with the same letter within a row indicate that no significant difference was observed at the 0.05 level of significance (Student-Newman-Keuls test).

kraft fibers from degraded wood chips refined faster than kraft fibers from non-degraded chips. Cell-wall separations, as well as increased fiber flexibility, could have enhanced the "peeling off" and liberation of cell-wall materials from restrictive forces in the cell wall during refining. Blanchette et al. (1985), observed through transmission electron microscopy (TEM), a thinning of the S2 layers of birchwood degraded by *G. applanatum*. They noted severe degradation of the middle lamellae to the extent that the boundary walls frequently separated when specimens were being prepared for TEM. These observations were consistent with the findings of Ruel et al. (1981). During refining it can be conjectured that it was possible to separate the S1 and S2 layers of biodegraded wood pulp more readily.

Bauer-McNett classification studies performed on selected pulps indicated that pulps from fungally degraded wood chips contained more fines and fibrillar materials than pulps from sound wood chips. These observations were based on the amount of fines retained or lost during classification (Table 5). A larger proportion of the pulp fibers obtained from fungally degraded wood chips passed through the 0.0041 screen opening in comparison to pulp fibers obtained from control wood chips.

Sedimentation volume determinations also showed that there were significant differences between pulp fibers obtained from sound and fungally degraded wood

TABLE 6. *Average unbleached brightness (GE) and average opacity values.*

Treatment time (days)	Brightness (GE) Cooking time (hours) 1	2	3	Opacity Cooking time (hours) 1	2	3
		16% Effective alkali concentration				
0	20	25	28	99	97	98
10	16	18	15	99	98	98
20	13	15	14	97	96	97
30	9	10	16	98	98	97
		12% Effective alkali concentration				
0	−(*)	20	23	100	98	99
10	−(*)	16	17	99	99	99
20	9	9	10	98	99	97
30	9	10	12	99	98	96

* Data not collected because of the preponderance of shives in handsheets.

TABLE 7. *Average predicted handsheet strength properties obtained from sound and* P. chrysosporium *degraded red oak wood chips digested at 16% effective alkali concentration.*

Cooking Time (hours)	Incubation Time (days)	CSF															
		MiT Fold				Tear (g)				Tensile (kg/m)				Burst (kG/m)			
		600	500	400	300	600	500	400	300	600	500	400	300	600	500	400	300
1	0	401d[1]	412c	423d	431b	108c	100c	92c	84c	228b	315b	402b	488b	93a	148a	203e	258a
2		477c	486c	495c	504b	124c	115c	106c	98c	268a	352a	436a	520a	235b	322a	343a	316a
3		502c	510c	518b	526b	156c	142d	128d	114d	547b	587b	627b	667b	369a	388a	428a	456a
1	10	286c	332b	378b	424a	117c	107c	97c	81c	94a	213a	330a	447a	318b	350b	402b	411b
2		138a	209a	280a	351a	123c	113c	108c	93c	301b	388b	485b	577b	374b	412b	450b	488b
3		241a	296a	351a	406a	137c	125c	113c	101c	360a	413a	538a	627a	462b	431b	520b	518b
1	20	121a	206a	291a	376a	93b	85b	77b	69b	452c	525c	518c	671c	460c	500c	540c	580c
2		280b	339b	398b	457b	95b	87b	79b	71b	606c	623c	740c	807c	430c	535c	580c	625c
3		356b	413b	470c	527b	90b	91b	84b	77b	654c	715c	776c	837c	617c	651c	688c	725c
1	30	250b	326b	402c	478c	61a	59a	54a	43a	591d	677d	763d	813d	731d	773d	815d	857d
2		730d	801d	878d	952c	73a	67a	61a	55a	760d	819d	878d	937d	750d	797d	844d	891d
3		795d	861d	933d	1,002c	80a	74a	68a	62a	1,137d	1,173d	1,221d	1,263d	1,111d	1,107d	1,233d	1,279d

[1] Means with the same small letter within a column indicate no significant difference between fungal incubation time at the 0.05 level of probability.

TABLE 8. *Average predicted handsheet strength properties obtained from sound and* P. chrysosporium *degraded red oak wood chips digested at 12% effective alkali concentration.*

Cooking time (hours)	Incubation (time (days)	CSF															
		Mit fold				Tear (g)				Tensile (kg/m)				Burst (kH/m)			
		600	500	400	300	600	500	400	300	600	500	400	300	600	500	400	300
1	0	—	—[2]	—	—	—	—	—	—	—	—	—	—	—	—	—	—
2		41a	52a	63a	74a	93bc	83c	73b	63b	44a	106a	168a	230a	10a	11a	61b	111a
3		70a	79a	88a	87a	118b	105b	92a	79a	47a	110a	173a	236a	12a	25a	95c	165c
1	10	203a	210a	281a	221a	77b	88a	59a	50a	182b	248b	314b	300a	14a	16a	23a	95a
2		195b	208b	223b	237b	88a	78a	68a	58a	195c	237b	273b	321b	20a	26b	65b	120b
3		235b	248b	251b	259b	96a	85a	74a	63a	270b	328b	382b	430b	22a	26a	39a	116a
1	20	300b	306b	312b	318b	71a	62a	53a	44a	125a	212c	299a	386a	16a	18a	48b	124b
2		248c	263c	278c	293c	82a	72a	62a	52a	175b	252c	329c	406c	18a	20ab	22a	107a
3		342c	351c	360c	369c	96a	81a	72a	60a	433c	476c	519c	502c	24a	32a	59b	143b
1	30	366c	373c	380c	387c	84b	73b	62a	51a	362c	429d	486c	548b	01b	130b	195c	252c
2		341d	353d	365d	377d	81a	71a	61a	51a	436d	496d	536d	586d	242b	281c	320c	359c
3		397d	408d	419d	430d	102ab	89a	76a	63a	467d	512d	557d	602d	209b	332b	305d	398d

[1] Means with the same small letter within a column indicate no significant difference between fungal incubation time at the 0.05 level of probability.
[2] Data not collected because of the preponderance of shives in handsheets.

chips (Table 5). Sedimentation values were 190 ml and 390 ml per gram from pulps of wood chips degraded for 0 and 30 days, respectively. Higher sedimentation values obtained for pulps from degraded wood chips indicated a higher degree of fines generation and fibrillation in comparison to pulps obtained from sound wood chips.

PULP BRIGHTNESS AND OPACITY

Significant differences ($P < = 0.05$) in pulp brightness of unbleached handsheets were observed to occur among pulps made from sound and fungally degraded red oak. Unbleached brightness decreased as fungal incubation time increased, irrespective of the length of cook or effective alkali concentration used during pulp preparation (Table 6). Brightness reduction ranged from 42% to 62% for pulps obtained from wood chips digested after 30 days of fungal degradation, depending on kraft cooking conditions. In this study opacity was not affected by the fungal degradation of red oak wood chips (Table 6).

Similar reductions in pulp brightness had been observed by Setliff et al. (1983) and by Eriksson and Kirk (1980); Eriksson and Vallander (1980) for mechanical pulps prepared from fungally degraded aspen and spruce.

HANDSHEET PROPERTIES

At a given freeness level, significantly higher ($P < = 0.05$) tensile strength, burst, and fold properties were obtained for handsheets prepared from kraft digested fungally degraded red oak wood chips compared to control handsheets (Tables 7 and 8). The highest tensile strength, burst, and fold properties were observed to occur for handsheets prepared from wood chips that were fungally degraded for 30 days. On the other hand, the lowest tensile strength, burst and fold properties were observed to occur for handsheets prepared from 10-day fungally degraded and nondegraded wood chips (controls). A decrease in tear strength was associated with an increase in fungal incubation time. These changes in measured strength properties with incubation time occurred irrespective of cooking time or effective alkali concentration used during pulp preparation (Tables 7 and 8).

Improvements in handsheet tensile strength, burst and fold properties occurred with an increase in fungal incubation time, and this observation could be explained, in part, by the increase in fiber swelling as indicated by higher WRV results (Table 4). The increased contact and intertwining of microfibrils resulting from enhanced swelling, collapsibility, and fibrillation during pulp refining has been shown to improve both interfiber bonding and web formation (Labosky 1970).

Brandon (1979) had shown that the amount and quality of fiber bonding was the most important factor affecting tensile strength development of paper. Since pulped, fungally degraded wood contained more holocellulose and less lignin (Table 2) compared to pulps obtained from nondegraded wood, it can be expected that more fines and bonding sites (or fibrillar materials necessary for bonding) may have been generated during refining. This apparently was the case because more fibrillar material was generated during refining of pulped, fungally degraded wood compared to control pulp (Table 5).

A decrease in handsheet tear properties occurred for fungally degraded wood as incubation time, cooking time, and beating time increased (Tables 7 and 8). A loss in tear strength for chemically pulped wood was expected, because tear is inversely proportional to tensile or burst properties in a sheet (Brandon 1979). The observed loss in tear properties with refining or freeness development may be explained on the basis of an increase in fiber to fiber bonding and the reduction in fiber length due to refining. An increase in tear properties was reported by Myers et al. (1988), and Setliff et al. (1983) for fungally degraded mechanical pulps.

SUMMARY AND CONCLUSIONS

The following conclusions can be drawn from this study:

1. Significantly higher ($P < = 0.05$) kraft pulp yields and lower kappa numbers were obtained from biodegraded red oak wood chips compared to nondegraded wood. At comparable kappa numbers, higher pulp yields (3% to 5%) occurred for pulps obtained from wood chips fungally degraded for 20 and 30 days as compared to nondegraded wood or wood chips fungally degraded for 10 days.
2. Red oak pulps prepared from wood biodegraded for 30 days refined the fastest, followed by pulps from 20-, 10-, and 0- day fungal incubation time.
3. At a given freeness level, significantly higher ($P < = 0.05$) tensile strength, burst, and fold properties were measured for handsheets prepared from 20- and 30-day fungally degraded wood compared to handsheets prepared from control pulps or those biodegraded for a 10-day period. In all cases, at a given freeness level, significantly lower tear properties were obtained from handsheets prepared from fungally degraded wood compared to the control.
4. Lower unbleached brightness values were measured for handsheets prepared from fungally degraded red oak wood at all decay times compared to handsheets prepared from pulps of nondegraded red oak wood.
5. Handsheet opacity was not affected by fungal incubation time.

ACKNOWLEDGMENTS

This study was supported by McIntire-Stennis Project and has been approved as Journal Series No. 8210 of The Pennsylvania Agricultural Experiment Station and was authorized for publication on May 11, 1989. The authors would like to express their appreciation to Dr. Ming Tien, Associate Professor of Molecular and Cell Biology, The Pennsylvania State University for making available the fungus used in this study and to Westvaco Corporation, Tyrone, Pennsylvania for making available their laboratory facilities for the testing of handsheets.

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