EARLY DETECTION OF BROWN ROT DECAY IN DOUGLAS-FIR AND SOUTHERN YELLOW PINE BY INFRARED SPECTROPHOTOMETRY

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(Received May 1984)

ABSTRACT

Samples of Douglas-fir heartwood and southern yellow pine sapwood were incubated with several brown rot rungi and examined for decomposition as shown by weight losses (to 10%). Warm-water extracts from the samples were analyzed by infrared spectrophotometry. An absorption peak at 1,720 cm^{-1} appeared in infrared spectra of decayed specimens but was not present in nondecayed specimens. As weight loss increased, the ratio of the absorbance at 1,720 cm^{-1} to the absorbance at 1,630 cm^{-1} increased. The ratio correlated with days of incubation and modulus of rupture.

Keywords: Douglas-fir, southern yellow pine, brown rot, infrared analysis, strength loss.

INTRODUCTION

Brown rot fungi can cause major strength reduction in wood species used for structural purposes. At weight losses of only 1 or 2%, some strength properties may be reduced 50% (Wilcox 1978); therefore, brown rot fungi should be detected early so that control procedures, such as fumigation (Scheffer and Graham 1975), can inhibit fungal growth and prolong the life of a structure. Current inspection methods such as sonic and electrical resistance or direct culturing of fungi from increment cores do not effectively detect the decay before major strength reductions occur. Inwards and Graham (1980) have evaluated six techniques for detecting decay in Douglas-fir utility poles. None except culturing, which was used as the basis for evaluation, detected decay in the incipient stage. Culturing is time-consuming, and while it permits identification of the fungi, it does not indicate the extent of decay.

Some techniques show promise for early detection. Wilcox (1968), using a microscopic technique to identify decay by the presence of fungal hyphae in the cell lumens and of bore holes in the cell walls, could detect decay with confidence at weight losses as low as 5%. Krahmer et al. (1982), using fluorescence microscopy with acridine-orange stain, detected early decay by the brown rot fungus *Gloeophyllum trabeum* in southern pine sapwood. With carefully controlled staining procedures, nondecayed wood sections fluoresced green, while wood with more

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Wood and Fiber Science, 17(4), 1985, pp. 522–528

TABLE 1. *Correlation of the ratio of infrared peaks to the days of incubation with brown rot fungi and to the percentage of loss in strength of Douglas-fir and southern yellow pine samples.*

	Wafers			Beams				
	n	Days of incubation	r[a]	n	Range of MOR loss	r[a]	Range of MOE loss	r[a]
Douglas-fir								
Poria placenta (Fr.) Cke.	14	2–14	0.90**	10	9–79	0.20[NS]	0–40	−0.19[NS]
Poria xantha (Fr.) Cke.	18	2–22	0.75**	10	0–45	0.65*	0–22	0.24[NS]
Lentinus lepideus Fr.	12	2–14	0.84**	10	5–56	0.90**	0–24	0.42[NS]
Nondecayed control	15							
Southern yellow pine								
Poria placenta (Fr.) Cke.	12	2–14	0.84**					
Lentinus lepideus Fr.	14	2–14	0.70**					
Gloeophyllum saepiarium (Wulf ex Fr.) Karst.	18	2–14	0.69**					
Gloeophyllum trabeum (Pers. ex Fr.) Murr.	18	2–17	0.50*					
Nondecayed control	15							

[a] Correlation coefficient for relationship of IR peak ratio.
[NS] Not significant.
* Significant at the 0.95 level of probability.
** Significant at the 0.99 level of probability.

than 3% weight loss fluoresced orange. However, procedures for detecting incipient decay with this technique could not be developed for Douglas-fir heartwood samples.

Decay fungi utilize the chemical components of wood cell walls in order to grow and reproduce. The decomposition process brings about changes in the high-molecular-weight polymers that make up wood (Cowling 1961). Brown rot fungi attack the entire cell wall, even in initial stages of decay; therefore, changes in the major chemical components of wood or the by-products of decay could become useful for locating incipient decay. Chemical breakdown of the wood cell wall occurs before weight loss can be detected, and the breakdown products are extractable with water. The infrared (IR) spectrophotometer provides an effective means for examination of chemical variations in wood (Marchessault 1962; Chow 1972). Takahashi and Nishimoto (1967) used it to examine the decomposition of ground wood samples by brown, white, and soft rot fungi. They noted changes throughout the spectra as decay progressed, but the absorption at 1,730 cm^{-1} weakened rapidly in wood exposed to the fungi.

The objectives of this study were to examine the use of infrared spectrophotometry to detect the early presence of brown rot decay in Douglas-fir [*Pseudotsuga menziesii* (Mirb.) Franco] and southern yellow pine (*Pinus* spp.) and to relate any quantitative IR results to decomposition as indicated by weight loss or days of incubation and by strength loss.

MATERIALS AND METHODS

Sample preparation

The samples used in this study were small wafers of Douglas-fir heartwood and southern pine sapwood and end-matched beams of Douglas-fir (Table 1). The

wafers were used to determine if decay could be detected with IR analyses and if it could be related to incubation factors, while the beams were used to examine strength losses.

Wafers cut from a single board of each species were approximately 2.5 cm square in cross-section and 4 mm thick along the grain. End-matched beams were 10.2 cm along the grain, 0.25 cm in the tangential direction, and 1.3 cm in the radial direction. All samples to be incubated with fungi were conditioned in a controlled environment at 26 C and 30% relative humidity. After their weights were recorded, samples were steam sterilized for one-half hour at 121 C/15 psf.

The wafers were aseptically placed in petri-dish cultures of five representative brown rot fungi known to invade Douglas-fir and southern pine utility poles (Table 1) (Eslyn 1970; Zabel et al. 1980). They were separated from the mycelial mat by a fiberglass screen. All plates were incubated at 27 C. Four wafers were removed from each culture after 2 days of incubation, and the remaining wafers were removed in groups of four at intervals of 2 days for the first 10 days and thereafter at intervals of 3 to 5 days until weight losses reached approximately 10%. Any fungal growth was brushed from the wafers before they were air-dried for 1 week and returned to the conditioning room to equilibrate. Each wafer was then weighed, and the weight loss was calculated as a percentage of the original weight.

One-half of the Douglas-fir beams were nondecayed control samples. End-matched portions were placed in modified soil-block jars, each containing southern pine feeder strips inoculated with one of the fungi listed in Table 1. The first beams were removed after 14 days of incubation at 27 C, and succeeding groups of beams were removed every 4 to 6 days until all had been removed. The percentage of weight loss of each beam was calculated by the method previously described. Beams selected for strength testing were from the last three incubation periods (14, 17, and 22 days for *Poria placenta* and *Poria xantha*; 21, 24, and 27 days for *Lentinus lepideus*) because weight losses were then in the desired range.

The nondecayed control and the end-matched decayed beams were tested in static bending with a Universal Instron testing machine located in a controlled-environment room (23 C/50% RH). Beams were equilibrated in this room for 1 week before testing. They were center loaded on the radial face over a span of 8.9 cm at a rate of 0.1 cm/min. Load-deflection curves were plotted, and modulus of rupture (MOR) and modulus of elasticity (MOE) were calculated. The percentage of loss in strength of the decayed beam was based on the strength value of the end-matched, nondecayed beam.

Preparation of the warm-water extract

A 0.5-gram solid sample of wood was cut from control samples, from each of two or three of the four wafers that had been removed at each incubation interval, and from the center of each decayed beam after testing in static bending. These were placed in separate Erlenmeyer flasks containing 10 ml of distilled water. The flask with an attached condenser was heated to approximately 85 C for 3 h. The warm-water extract was then filtered through Number 1 Whatman filter paper and the filtrate poured into a 10-ml beaker. The water was then evaporated, leaving only the dried extract for IR analysis.

Infrared analysis

A Beckman IR-20A Double-Beam Infrared Spectrophotometer was used to obtain normal scans between 4,000 and 250 cm^{-1}. Potassium bromide (KBr) pellets were prepared by thoroughly mixing approximately 200 mg of dried IR-grade KBr and 1.0 mg of the extract. This mixture was made into a transparent disc approximately 1 mm thick and 1 cm in diameter by means of a pellet-making die (10,000 psi for 2 min; 20,000 psi for 1 min).

RESULTS AND DISCUSSION

Decay rates and strength losses

The progress of decay of wafers determined by weight loss of Douglas-fir heartwood and southern pine sapwood attacked by the five brown rot decay fungi is shown in Fig. 1. Weight losses of Douglas-fir wafers began to appear after 10 days' incubation with two of the three brown rot fungi. *Poria placenta* was the fastest acting, the average weight loss more than 10% after only 14 days. *Gloeophyllum trabeum* produced weight losses in southern pine wafers after only 6 days of incubation, the losses averaging more than 12% at 14 days.

Weight losses in the decayed beams of Douglas-fir ranged from 0.0 to 5.2%. *Poria placenta* caused up to 60% reduction in MOR with only a 2% weight loss. MOR losses in the decayed beams ranged from 0 to 79% (Table 1). MOE values were not as drastically reduced.

Infrared analysis of the warm-water extracts

Infrared spectra of warm-water extracts from decayed and nondecayed wafers of Douglas-fir heartwood and southern pine sapwood show a major difference for all fungi in the region from 1,800 to 1,500 cm^{-1} (Fig. 2). An absorption peak at 1,720 cm^{-1} was present for all decayed wood, but absent for all control samples and for all samples incubated 2 days or less. A shoulder appeared in the 1,720 cm^{-1} region for nondecayed southern pine, and a peak appeared at 1,680 cm^{-1} for nondecayed Douglas-fir; however, these did not preclude the more pronounced peak at 1,720 cm^{-1} for all decayed wood.

The peak at 1,720 cm^{-1} first appeared in spectra of extracts from incubated wood samples that had not yet shown weight loss after 2 days of incubation. Generally, it became evident 2 or more days before matched samples started showing weight loss as a result of decay. All spectra of specimens with recorded weight loss had a peak at 1,720 cm^{-1}.

As the number of days of incubation or the weight loss of the wood sample increased, the magnitude of the absorption peak at 1,720 cm^{-1} also increased. To quantify this, we calculated the ratio of the distance between the IR base line (horizontal portion between 2,000 and 1,900 cm^{-1}) and the 1,720 cm^{-1} peak to the distance between the base line and the 1,630 cm^{-1} peak. The latter peak occurred in all spectra and appeared to relate to the quantity of extract in a KBr pellet. The ratio 0.0 indicates absence of a peak at 1,720 cm^{-1}; a positive ratio indicates its magnitude. The trend of an increase in the IR peak ratio with the number of days in incubation is readily apparent (Fig. 3). Table 1 gives correlation coefficients (r) for simple linear regressions between the ratio of IR peaks and

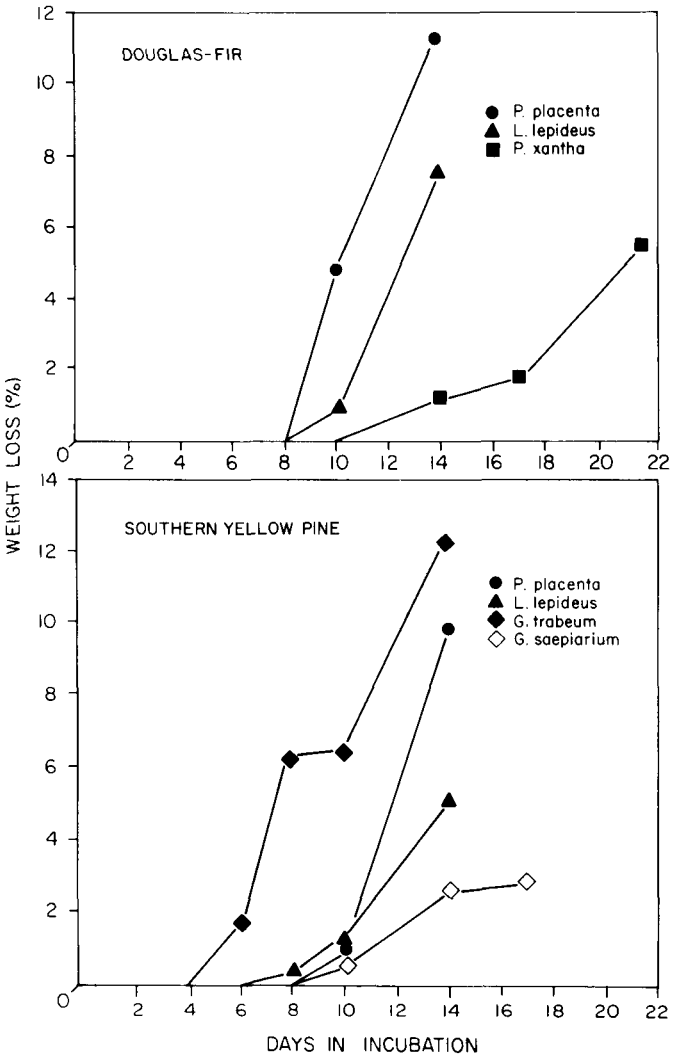

FIG. 1. Percentage of weight loss of Douglas-fir and southern yellow pine wood incubated with brown rot fungi. Each point is the average of four samples.

days of incubation with each fungus. All r-values, ranging from 0.90 for *P. placenta* in Douglas-fir to 0.50 for *G. trabeum* in southern pine, are significant at the 5% level.

Simple linear regression analyses were also made between IR peak ratios and the percentage of loss of MOR and MOE from spectra of decayed Douglas-fir beams. The strongest relationships are for loss in MOR in beams decayed by *Lentinus lepideus* and *Poria xantha* (Table 1). Relationships for which peak ratios were the predictor of loss in MOE were not significant.

During this study, some samples were incubated until they were in an advanced stage of decay (weight losses well above 10%). The IR absorption peak at 1,720 cm^{-1} was usually missing from spectra of these samples. Further research should identify the chemical components responsible for the characteristic peak in water

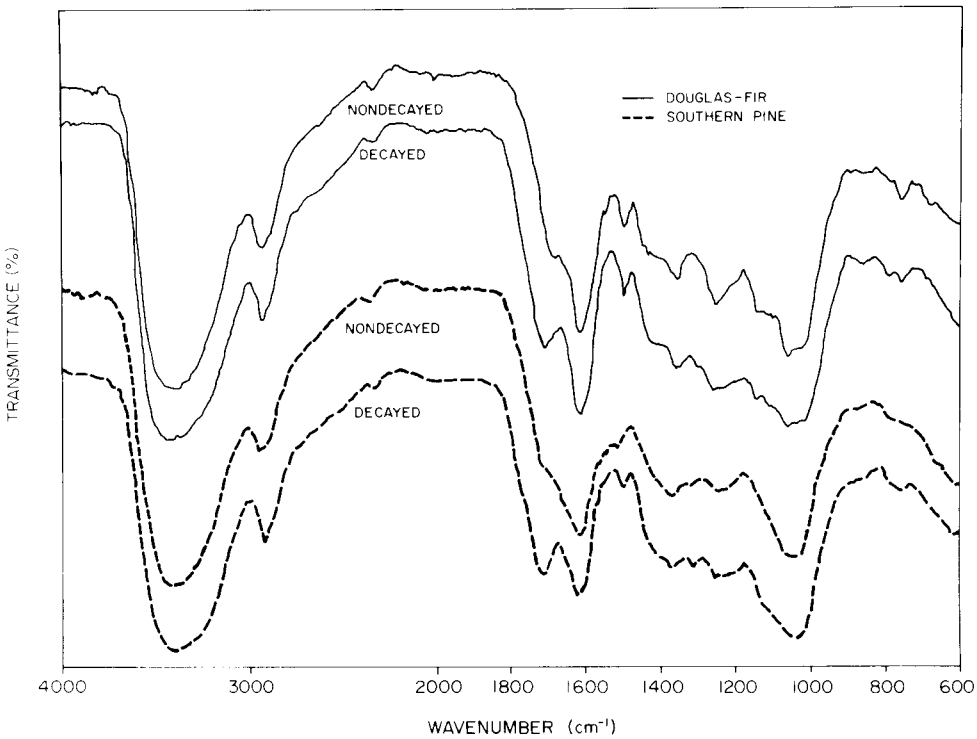

FIG. 2. Infrared spectra of warm water extracts from nondecayed and decayed wood. Decayed wood shows an absorption peak at 1,720 cm^{-1}; nondecayed wood does not. The peak for decayed Douglas-fir occurred after 8 days of incubation; weight loss is 0%. The peak for decayed southern yellow pine occurred after 10 days of incubation; weight loss is ~1%.

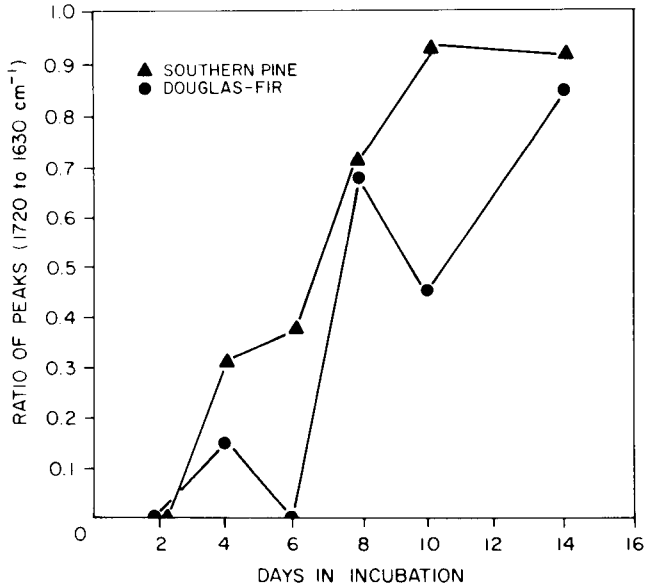

FIG. 3. Average absorption peak ratios in infrared spectra of Douglas-fir and southern pine wood extracts after incubation with fungi. Points are the average of three fungi for Douglas-fir and four fungi for southern pine.

extracts from incipiently decayed wood and should determine whether other biological, physical, and chemical conditions of wood deterioriation show an IR absorption peak at 1,720 cm^{-1}.

CONCLUSIONS

Infrared spectroscopy detected incipient brown rot decay of laboratory-prepared Douglas-fir heartwood and southern pine sapwood samples several days after incubation began but usually before weight losses were measurable. The ratio of the height of two IR absorption peaks was shown to increase with the progression of early decay and to correlate with loss of MOR in small Douglas-fir beams. No relationship was found with loss in MOE.

ACKNOWLEDGMENTS

The authors thank Bessie Earthly, Forest Products Laboratory, Madison, Wisconsin, for preparation of the decayed-wood samples, and Terry Stock, Department of Forest Products, Oregon State University for much of the analysis of the southern pine samples. This is Paper 1887, Forest Research Laboratory, Oregon State University. It was presented at the Biology Technical Session of the 37th annual meeting of the Forest Products Research Society, Norfolk, Virginia, June 1983.

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