ANTITERMITIC PROPERTIES OF CELLULOSE PADS TREATED WITH BARK EXTRACTIVES[1]

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ABSTRACT

A study was undertaken to evaluate the feasibility of using bark extractives as a preservative treatment for wood to inhibit subterranean termite activity. Bark samples were collected from eleven species: *Pinus strobus, P. virginiana, Tsuga canadensis, Quercus alba, Q. velutina, Q. prinus, Sassafras albidum, Juglans nigra, Carya ovata, Liriodendron tulipifera,* and *Robinia pseudoacacia.*

Cellulose paper pads (unbleached kraft) were treated with one of two bark extracts (A:H:W or 1% $NaOH/Na_2S$) at 0.16, 0.67, and 0.83 g/ml treatment levels and evaluated for antitermitic properties.

Complete termite mortality was observed after 4 weeks for paper pads treated with A:H:W bark extracts from the species *Q. prinus, P. strobus, C. ovata,* and *S. albidum* at both the 0.67 and 0.83 g/ml treatment levels. In addition, no termite survival was observed for paper pads treated with the A:H:W bark extracts from the species *Q. alba, L. tulipifera,* and *T. canadensis* at the 0.83 g/ml treatment level. Termite survival was observed for paper pads treated with A:H:W extracts at the 0.16 g/ml treatment level and with alkali extracts at the 0.16, 0.67, and 0.83 g/ml treatment levels for all bark species examined.

No significant difference in termite mortality was observed between A:H:W bark extracts obtained from fresh or 1-year-old harvested bark. Feeding preference trials indicate that termites are attracted to A:H:W extracts obtained from *C. ovata* and *J. nigra.* Antitermitic trials with pentachlorophenol, copper naphthenate, and bark extracts at the 0.67 g/ml treatment level exhibited complete termite mortality; but the termites consumed more cellulose treated with bark extractives than with commercial preservatives.

These results indicate that A:H:W bark extracts from the species *Q. prinus, S. albidum, P. strobus* and *C. ovata* show promise as a wood preservative, but additional studies are needed to isolate and identify the antitermitic extracts.

Keywords: Termite control, cellulose pads, bark extractives, wood preservatives.

INTRODUCTION

Among wood-destroying insects, subterranean termites are by far the most destructive. The U.S. Forest Service estimates that about $1 billion worth of wood products deteriorate from decay and termites each year. This waste is the equivalent to the timber volume from 300,000 acres of commercial production, annually (Koch 1972).

Investigators have long noted that certain woods are naturally resistant to subterranean termite attack (Beal et al. 1974; Carter et al. 1975; Carter and Smythe 1974; Carter et al. 1978; Koch 1972; Nelson 1975; Rust and Reierson 1977; Smythe and Carter 1969). This resistance results primarily from chemical substances that are distasteful, repellent, or toxic to the termites rather than from the high lignin content or hardness of the wood.

Carter et al. (1975), in a recent study, reported that many extracts prepared

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from naturally resistant woods contained substances detrimental to termite survival. They found that termites would not survive on absorbent paper pads treated with extracts from 14 different woods. Carter and Smythe (1974) in a similar study concluded that termite survival varied by wood species and solvent systems employed when *Reticulitermes flavipes* (Kollar) was fed on selected solvent-extracted sawdusts prepared from redwood, black walnut, and baldcypress. The best solvent system for removing the termiticidal materials from all three woods appeared to be a mixture of acetone, hexane, and water (A:H:W—53:44:2, by volume). Carter et al. (1975) concluded from their studies that no single solvent can extract all detrimental materials from wood. Most recently, Carter et al. (1979) studied the termiticidal properties of slash pine wood. It was found that individual slash pine trees varied in termite resistance. In general, the heartwood zone nearest the pith contained substances that were more resistant to termites than the heartwood zone at the sapwood-heartwood interface. It was found that both the pentane and the A:H:W (54:44:2, by volume) extractable fraction of slash pine heartwood contained components that were termiticidal. The authors concluded that the heartwood components responsible for resistance against the termites were the phenolic compounds, such as the flavones and stilbenes. Although the function of tannins, phlobaphene, and other phenolic substances in bark is not clear, it has been suggested by investigators that these materials act as an inhibitor of fungal growth (Browning 1963; Carter et al. 1979; Howard 1971). In the south, it is quite common to see remains of southern yellow pine stumps with the bark still intact. Apparently, the bark contains substances that are detrimental to both fungi and termites.

Considering the importance to isolate and identify new materials for the preservative treatment of wood, information concerning extractives that make commercially important wood resistant to termites and fungi would be of great value. Therefore, a study was undertaken to investigate the antitermitic properties of selected bark extractives. To date, data are limited dealing with the use of bark extracts as a preservative treatment for wood to inhibit termite attack.

MATERIAL AND METHODS

Termites

The eastern subterranean termite *Reticulitermes flavipes* (Kollar) was used in this study. Termites were collected from oak tree stumps near State College, Pennsylvania, and were maintained in an environmental chamber at 21 C, 55% RH until used (Smith et al. 1969). As needed, undifferentiated functional worker stages were selected and exposed to test materials. Care was taken to select termites that were actively feeding.

Bark collection and preparation

Bark was stripped from trees located near State College, Pennsylvania, during the fall of 1980. Bark was hewed from three trees for each species (Table 1) selected for study and dried separately in paper bags. Care was taken not to include the cambium. A composite sample from each species was prepared by blending equal amounts (by weight) of bark from each of three trees. Each composite was Wiley-milled to pass a 20-mesh screen. The bark composites were then stored in

TABLE 1. *Average percent alcohol-benzene extractives content[1] yields of eleven bark composites.*

Bark composite		
Scientific name	Common name	Yield (%)
Tsuga canadensis	Hemlock	21.41 a[2]
Sassafras albidum	Sassafras	20.41 a
Carya ovata	Shagbark hickory	13.86 b
Pinus virginiana	Virginia pine	8.72 c
Robinia pseudoacacia	Black locust	8.72 c
Liriodendron tulipifera	Yellow-poplar	7.96 c
Quercus prinus	Chestnut oak	7.88 c
Quercus velutina	Black oak	7.62 c
Juglans nigra	Black walnut	7.56 c
Pinus strobus	White pine	5.25 d
Quercus alba	White oak	4.37 d

[1] Yields based on the dry weight of unextracted bark.
[2] Means followed by the same letter are not significantly different at the 0.05 level.

paper bags prior to extraction. Bark from individual trees was also Wiley-milled for alcohol-benzene (1:2 volume) yield determinations (Moore and Johnson 1967).

Bark composites were sequentially extracted in a Soxhlet extractor with A:H:W (54:44:2 by volume to form an azeotrope) and an alkali mixture of 1% sodium hydroxide-sodium sulfide. Four 20-g samples of air-dried (11.5% MC) bark was extracted for each species. The extraction rate was four siphons per hour for 4 hours. The four extractions were combined, filtered, and stripped of solvent in a rotary vacuum evaporator at about 30 C. Sufficient solvent was added to obtain the desired concentration level for treatment of cellulose pads. The A:H:W extracted bark was air-dried to remove excess solvent before alkali extraction. Prior to antitermitic trials, preliminary tests were performed to determine extractive treatment levels to be used on the cellulose pads (unbleached kraft paper).

Termiticidal trials

Air-dried cellulose paper pads (2.5 × 1.3 × 0.6 cm and about 500 mg) were placed into circular plastic petri dishes (5.08 × 1.27 cm) and treated with 5 ml of solvent extractables from each bark species at three treatment levels of 0.16, 0.67, and 0.83 g/ml. Pads were air-dried and then 5 ml of distilled water was added to maintain relative humidity at saturation. Fifty termites were added to each dish. The containers were covered and placed in the environmental chamber for 4 weeks. Surviving termites were counted at the end of each week. Pad weight loss after 4 weeks was also used to quantify termite attack. No attempt was made to remove dead termites from the dish during the trial period.

Each test condition (treatment level) was replicated in triplicate for each species and extract type (A:H:W and alkali extracts). Nine replications were measured for a test condition for a total of 117 observations per trial (13 conditions × 3 trials × 3 replicates). Controls using pads treated with and without solvents were also evaluated to determine the solvent effect.

Preference trial

Preference trial tests were performed to determine if the feeding propensity of termites was toward one or more particular bark extracts. A plastic container

(30.5 × 15.2 × 10.2 cm) was filled with 1,000 g of autoclaved sand. Cellulosic pads were treated at 0.67 g/ml with each species of bark extracted with A:H:W and were randomly placed without a control into the container with 500 termites. The sand was moistened with 150 ml of distilled water to maintain a high relative humidity in the container. Each test condition was replicated in triplicate using A:H:W bark extracts. Containers were kept in the environmental chamber at 21 C and 50% RH. After 4 weeks, the pads were air-dried and weighed to obtain weight losses. The loss in weight served as a measure of termite attack. No attempt was made during these trials to monitor and determine attraction responses of the termites to the treated pads.

Filtered bark extract trial

A trial was run to determine the effect of the waxlike material suberin on termite survival. It was noted that this material coated the paper pads. Fresh A:H:W bark extracts were cooled to solidify the wax and then filtered in a Buchner funnel. The filtrate was concentrated in a rotary vacuum evaporator, and cellulose pads were treated at the 0.67 g/ml level. Termites were added to the petri dishes containing the cellulose pads and 5 ml of distilled water and tested for termiticidal properties.

Comparative termiticidal trials with fresh and one-year-old harvested bark

A trial was also performed to determine if any differences in termiticidal properties existed between fresh bark and 1-year-old harvested bark. The purpose of this trial was to examine what effect the time between bark removal and extraction may have on bark termite-resistant properties since the initial trials employed 1-year-old harvested bark. Termiticidal trials were performed as described above using cellulosic pads treated with 5 ml of A:H:W extract at the 0.67 g/ml treatment level.

Data analysis

The data were analyzed by one-way analysis of variance ($P \leq 0.05$). Termite survival and weight loss data were transformed to arc sine for analysis but were presented as untransformed values to facilitate interpretation. Differences in termite survival and pad weight loss were compared at the 0.05 level by Duncan's new multiple range test.

RESULTS AND DISCUSSION

Alcohol-benzene extractive yields

To assess the normality of extractive content in the eleven experimental barks, alcohol-benzene extractive yields were determined, and significant differences ($P \leq 0.05$) in extractives content were measured (Table 1). Hemlock and sassafras barks exhibited the highest extractive yields (21 and 20%), and these values were significantly higher than those observed for all other species. White pine (5%) and white oak (4%) exhibited the lowest alcohol-benzene extractive yields, and these yields were significantly lower than Virginia pine (9%), black locust (9%), yellow-poplar (8%), chestnut oak (8%), black oak (8%), and black walnut (8%). In general, these figures agree with the alcohol-benzene bark extractive data reported in the

TABLE 2. *Feeding (pad weight loss %) and survival (%) of* R. flavipes *exposed to cellulose pads treated with A:H:W*[1] *bark extracts at three treatment levels.*[2]

	Treatment level (g/ml)					
	0.16		0.67		0.83	
Bark extract	Survival (%)	Weight loss (%)	Survival (%)	Weight loss (%)	Survival (%)	Weight loss (%)
Black oak	95.3 abc	8.8 dce	9.3 e	25.6 bcd	19.1 c	22.9 c
Hemlock	93.0 abc	9.0 dce	61.8 cd	16.5 efg	0.0 e	5.7 e
Black walnut	91.0 abc	3.6 g	66.9 bc	10.7 ghi	5.0 d	22.8 c
Chestnut oak	60.7 f	7.7 dfe	0.0 f	19.0 def	0.0 e	4.0 e
Yellow-poplar	91.3 abcd	13.8 ab	59.3 d	29.7 bc	0.0 e	15.3 cd
Virginia pine	79.7 e	7.2 ef	6.8 e	13.8 fgh	18.5 c	7.0 e
White pine	13.6 i	7.2 ef	0.0 f	4.2 i	0.0 e	16.8 cd
White oak	52.3 g	15.7 a	63.8 cd	32.8 b	0.0 e	6.3 e
Shagbark hickory	30.7 h	6.6 ef	0.0 f	6.8 hi	0.0 e	17.4 cd
Black locust	86.3 d	5.5 fg	71.6 b	32.3 b	24.5 b	14.7 d
Sassafras	89.6 cd	10.7 cd	0.0 f	7.3 hi	0.0 e	6.9 e
Control	97.6 a	11.8 bc	92.8 a	41.9 a	91.7 a	45.1 a
A:H:W control	97.3 ab	11.7 bc	93.1 a	22.5 cde	92.3 a	33.7 b

[1] Acetone : hexane : water (A:H:W 54:44:2 by volume).
[2] Figures are averages of nine observations. Means with the same letter are not significantly different at the 0.05 level.

literature (Harkin and Rowe 1971; Labosky 1978; Ottone and Baldwin 1981). On the basis of these observations, the extractives yield from one ton of oven-dry bark (excluding the cambium) may vary from a high of about 400 pounds to a low of about 100 pounds.

Antitermitic trials with A:H:W bark extracts

The termite survival (%), and feeding (pad weight loss, %), results are summarized in Table 2. At the lowest treatment level (0.16 g/ml), test units containing pads treated with white pine bark extract were the only ones exhibiting termite survival below 15% after the 4-week trial period. Pads treated with chestnut oak, white oak, or shagbark hickory extracts exhibited antitermitic properties, but termite survival after 4 weeks was still above 20%. Test units containing pads treated with 0.67 g/ml bark extracts, however, exhibited complete termite mortality on pads treated with extracts of chestnut oak, white pine, shagbark hickory, and sassafras. At the highest treatment level, 0.83 g/ml complete termite mortality was also observed in test units containing pads treated with extracts of hemlock, yellow-poplar, and white oak in addition to the four species observed at the 0.67 g/ml trial. Throughout these trials, the control pads exhibited termite survival above 90%, indicating that the solvent used and cellulose pads had no effect on termite mortality. Similar observations were reported by Carter et al. (1975) in which 83% of the termites survived on the solvent-treated control pads using the same ratio of A:H:W mixture used in this study.

During the 0.67 g/ml trials, it was observed that termite mortality occurred gradually during the 4-week exposure period for white pine, shagbark hickory, and sassafras bark extracts. However, all termites were dead after 2 weeks of exposure to pads treated with chestnut oak bark. It was thought that the death caused by the extracts from white pine, shagbark hickory, and sassafras bark may have been due to a toxic action on the intestinal protozoa after ingesting the

TABLE 3. *Feeding (pad weight loss %) and survival (%) of* R. flavipes *exposed to cellulose pads treated with alkali*[1] *extracts at three treatment levels.*[2]

Bark extract	Treatment level (g/ml) 0.16 Survival (%)	0.16 Weight loss (%)	0.67 Survival (%)	0.67 Weight loss (%)	0.83 Survival (%)	0.83 Weight loss (%)
Black oak	40.0 e	24.0 e	38.5 f	24.2 de	43.2 e	33.8 b
Hemlock	70.8 b	12.3 g	60.8 de	16.0 f	51.2 e	16.1 def
Black walnut	64.2 b	11.2 gh	61.8 de	15.7 f	74.3 b	12.9 ef
Chestnut oak	47.2 d	16.7 f	58.5 e	21.6 e	69.7 bcd	17.6 de
Yellow-poplar	64.5 b	38.0 c	69.0 bcd	36.8 c	65.5 bcd	36.9 ab
Virginia pine	50.8 cd	16.3 f	76.2 b	22.2 de	61.5 d	19.6 d
White pine	27.0 f	8.0 hi	71.8 bc	8.7 g	63.0 cd	29.1 c
White oak	70.2 b	34.3 d	74.5 b	38.1 bc	75.7 b	40.0 a
Shagbark hickory	54.6 c	7.0 i	64.6 cde	9.0 g	65.0 bcd	10.9 f
Black locust	36.5 e	52.3 a	76.3 b	49.8 a	73.2 bc	41.6 a
Sassafras	67.8 b	10.9 gh	69.3 bcd	13.1 fg	68.5 bcd	13.0 ef
Control	91.5 a	44.1 b	92.8 a	43.0 b	90.6 a	41.4 a
Alkali control	95.5 a	26.5 e	96.0 a	27.1 d	96.5 a	27.5 c

[1] Alkali = 1% sodium sulfide–sodium hydroxide.
[2] Figures are averages of nine observations. Means with the same letter are not significantly different at the 0.05 level.

treated cellulose pad because of heavy feeding and a high pad weight loss (10%). If this was the case, the termites would continue to feed on the cellulose. The protozoa would not be able to break down the cellulose into usable sugars; the insect would slowly starve to death. Similar observations have been reported by Smythe and Carter (1969) in their experiment with feeding of *R. flavipes* on the sound wood extractives of redwood, black walnut, and baldcypress. They concluded that these wood extractives caused a slow termite death rate, which finally resulted in complete or almost complete mortality over an 8-week exposure period.

Antitermitic trials with alkali bark extracts

The alkali bark extracts were evaluated for antitermitic properties at treatment levels of 0.16, 0.67, and 0.83 g/ml and the termite survival (%) and feeding pad

TABLE 4. *Choice feeding (pad eaten mg) preferences of* R. flavipes *exposed to A:H:W*[1] *bark extracts at the 0.67 g/ml treatment level.*[2]

Bark extract	Cellulose eaten (mg)
Black oak	57 ef
Hemlock	43 f
Black walnut	210 b
Chestnut oak	147 c
Yellow-poplar	67 def
Virginia pine	100 d
White pine	102 d
White oak	65 def
Shagbark hickory	360 a
Black locust	53 ef
Sassafras	95 de

[1] Acetone : hexane : water (A:H:W 54:44:2 by volume).
[2] Figures are averages of three observations. Means with the same letter are not significantly different at the 0.05 level.

TABLE 5. *Feeding and survival of* R. flavipes *exposed to cellulose pads treated with A:H:W*[1] *filtered bark extracts at a treatment level of 0.67 g/ml.*[2]

Bark extract	Survival (%)	Weight loss (%)
Black oak	10.2 e	27.5 bc
Hemlock	25.6 cd	16.6 de
Black walnut	27.2 c	11.2 ef
Chestnut oak	0.0 f	23.5 cd
Yellow-poplar	21.5 d	35.4 ab
Virginia pine	9.0 e	16.8 def
White pine	8.0 e	10.2 f
White oak	41.0 b	38.8 a
Shagbark hickory	0.0 f	10.0 ef
Black locust	45.7 a	38.6 a
Sassafras	0.0 f	10.0 ef
Control	43.9 a	39.5 a
A:H:W control	45.6 a	37.4 a

[1] Acetone : hexane : water (A:H:W 54:44:2 by volume).
[2] Figures are averages of nine observations. Means with the same letter are not significantly different at the 0.05 level.

weight loss (%) results are summarized in Table 3. Results were erratic, but termite survival above 40% was observed even at the highest treatment level for all barks examined. These results indicate that the alkali extracts had little effect on termite mortality. In fact, it appeared that in some instances the termites consumed more of the alkali extractable materials. On the basis of these observations, it can be concluded that the A:H:W extraction removes essentially all the termiticidal extractives in bark.

Feeding preference trials

Feeding preference trials were conducted with A:H:W bark extracts at the 0.67 g/ml treatment level. The results of this trial are shown in Table 4. Significant differences in pad weight loss were observed, and the data showed that the termites preferred to feed on the cellulose pads treated with bark extracts from shagbark hickory and black walnut.

It is peculiar that the bark extractives that appeared effective in terms of termite mortality in early A:H:W trials also exhibited heavy termite feeding. The termites consumed large amounts of the cellulose pads treated with the bark extracts of shagbark hickory, chestnut oak, white pine, and Virginia pine. It is difficult to explain the feeding selectivity for termites since earlier investigations indicate that the food-finding behavior of termites is random (Smith et al. 1969).

Antitermitic trials using filtered A:H:W bark extracts

Initial observations indicated that the solvent removed a waxy substance believed to be suberin, which coated the cellulose pads and prevented water absorption particularly for the species Virginia pine and white pine. Since water is a requirement for termite survival, a question was asked as to what effect this waxlike material had on termite survival. Therefore, a trial was performed to remove this substance, and the remaining solvent extract was evaluated for antitermitic properties. Pads were treated at 0.67 g/ml and exposed to termites. The results are summarized in Table 5. Although termite survival was lower than that

TABLE 6. *Feeding and survival*[1] *of* R. flavipes *exposed to cellulose pads treated with commercial preservatives (Seal-Treat and Coppo) at a treatment level of 0.67 g/ml.*

Preservative	Survival (%)	Weight loss (%)
Pentachlorophenol	0.0 a	5.0 b
Copper naphthenate	0.0 a	6.8 b
Shagbark hickory	0.0 a	20.0 a
Chestnut oak	0.0 a	19.1 a
White pine	0.0 a	20.3 a
Sassafras	0.0 a	20.3 a

[1] Figures are averages of eight observations. Means with the same letter are not significantly different at the 0.05 level.

observed in earlier A:H:W trials, it can be observed that the termiticidal properties parallel earlier observations. It can thus be concluded that the waxlike material had little effect on termite mortality.

Comparative termiticidal trials using commercial preservatives and A:H:W bark extractives

Results of a comparative termiticidal trial between commercial preservatives (pentachlorophenol and copper naphthenate) and A:H:W bark extracts from shagbark hickory, chestnut oak, white pine, and sassafras are summarized in Table 6.

Complete termite mortality was observed for the wood preservatives (Seal-Treat and Coppo) and bark extractives evaluated. However, more cellulose (about 4 times as much) was consumed by termites feeding on pads treated with the A:H:W bark extracts than on those treated with commercial preservatives. In addition, it was noted that complete termite mortality occurred within 1 week rather than the 4-week trial period.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. When compared with termite survival in controls, all eleven A:H:W bark extracts gave mortality at the 0.67 and 0.83 g/ml treatment levels, but the extracts from chestnut oak, white pine, shagbark hickory, and sassafras gave complete mortality at these levels.
2. Although all of the alkali extracts gave some reduction in termite survival as compared with controls, increased treatment levels did not show an increase in termite mortality.
3. Feeding preference trials indicate that termites favored the A:H:W bark extracts from shagbark hickory and black walnut.
4. A:H:W bark extracts from *Q. prinus, S. albidum, P. strobus,* and *C. ovata* show promise as a wood preservative.

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