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**Potential of heartwood extracts as wood preservatives combined with linseed oil in 8-year above-ground and in-ground field trials**

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# Potential of heartwood extracts as wood preservatives combined with linseed oil in 8-year above-ground and in-ground field trials

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## ABSTRACT

The current study assessed the efficacy of heartwood extracts from *Tectona grandis*, *Dalbergia sissoo*, *Cedrus deodara*, and *Pinus roxburghii*, combined with linseed oil, as protectants for two non-durable wood species against termites and decay fungi. Stakes measuring 45.7 × 1.9 × 1.9 cm and blocks measuring 12.5 × 3.75 × 2.5 cm of the sapwood of southern pine (*Pinus* sp.) and cottonwood (*Populus* sp.) were impregnated via vacuum pressure with individual heartwood species extract, linseed oil, or a combination of both. For comparison, solid heartwood stakes and blocks of the wood species used to obtain extracts were also included in the tests. All samples were exposed to decay and termites for eight years at a test site in southern Mississippi using ground contact (AWPA E7) and ground proximity (AWPA E26) tests. Results showed that combining heartwood extract and linseed oil resulted in greater resistance to termites and decay in cottonwood and southern pine compared to using only linseed oil or the individual heartwood species extract in both tests. However, most of the samples that were treated with the combination treatment failed and, in either test, these treatments were not as effective as commercially used wood preservatives, such as copper naphthenate (CuN) or disodium octaborate tetrahydrate (DOT). Additionally, solid heartwood *P. roxburghii* stakes and blocks decayed completely and failed due to termite attack after eight years in the field in both tests. After eight years in the field, *C. deodara* and *D. sissoo* stakes were severely attacked by decay and moderately to severely attacked by termites. Blocks of these two species exposed in a covered ground proximity exposure showed slight attack by termites and decay fungi. *Tectona grandis* stakes showed moderate decay damage and slight termite attack in the ground contact test. Blocks of *T. grandis* showed slight damage from fungi and termites in the ground proximity test after eight years in the field.

**Keywords:** Wood extractives, heartwood, termites, decay fungi, field tests, oil, preservation

## 1. INTRODUCTION

Wood is one of the most widely utilized, adaptable, and environmentally sustainable construction materials worldwide. However, many wood species are susceptible to deterioration from decay fungi and insects, necessitating suitable protection measures to prolong their lifespan (Hwang *et al.* 2007). For decades, traditional chemical wood preservatives have been employed to mitigate the susceptibility of wood to biological degradation, owing to their affordability and demonstrated effectiveness across different environmental conditions. However, regulations are increasingly limiting the use of traditional chemical wood preservatives to prolong the lifespan of wood, driven

by concerns regarding their potential human toxicity and environmental impact (Singh and Singh 2012, Kirker *et al.* 2024).

Certain wood species have evolved mechanisms to withstand attack from biotic agents. Resistance against termites or decay fungi is often attributed to the production of toxic extracts within the heartwood of these wood species as the tree matures (Singh and Singh 2012, Francis *et al.* 2024). Some heartwood extracts have been found to exhibit toxic, antifeedant, antioxidant, antiviral, bactericidal and fungicidal properties. Using heartwood extract from durable species as wood preservatives for less durable wood species is one strategy to reduce environmental and health hazards. These compounds could be extracted and utilized to treat less resistant wood species, protecting against termite and fungal infestations. Extracts of naturally resistant woods are easy to detoxify and dispose of without impairing the quality of the environment (Hassan *et al.* 2021).

There are doubts regarding the ability of these natural compounds to match the effectiveness of traditional treatments like chromated copper arsenate (CCA) in wood protection (Hwang *et al.* 2007, Hassan *et al.* 2021). One approach to enhance natural wood protectants' efficacy is combining one or more organic biocides. The advantages of such combinations have been recognized for some time, and when coupled with an appropriate additive formulation, they can decrease production costs and improve effectiveness against organisms that degrade wood. One approach to enhance the effectiveness of heartwood extracts is by combining them with other natural compounds, such as hydrophobic plant oils. Similar to wood extracts, oils derived from seeds and foliage of various plant species possess insecticidal, antimicrobial, antioxidant, antifeedant, and repellent properties (Hwang *et al.* 2007, Terziev and Panov 2010, Hassan *et al.* 2020).

In the current study, we combined heartwood extracts from four durable wood species with linseed oil to treat two non-durable wood species, aiming to create a multi-component wood preservative system derived from natural sources. We then subjected treated samples to field testing for eight years in ground proximity and ground contact exposures. The results from the first five years of field exposure have been previously published (Hassan *et al.* 2021). In this article, we only report on results obtained from a final eight-year rating and juxtapose this latest data set with published five-year data.

## **2. EXPERIMENTAL METHODS**

### **2.1 Procurement of wood and extract preparation**

Extracts were extracted from the heartwood of four naturally grown durable wood species: *Tectona grandis* L.f. (Teak), *Dalbergia sissoo* Roxb (Shisham), *Cedrus deodara* (D. Don) (Deodar), and *Pinus roxburghii* Sargent (Chir Pine). To assess the efficacy of heartwood extracts on non-durable species, southern pine (*Pinus* sp.) and cottonwood (*Populus* sp.) were chosen. Heartwood logs free from defects were obtained from a timber market on Jhang Road in Faisalabad, Pakistan, for all test species except *T. grandis*, which was sourced as marine grade from a supplier in the USA (McIlvain, Pittsburgh, PA) and shipped to the Forest Products Laboratory in Starkville, MS (Hassan *et al.* 2017b).

Heartwood extracts were prepared following the method described by Hassan *et al.* (2020). Briefly, the heartwood of all species was cut into boards measuring 457 × 127 × 19 mm, air-dried for four weeks, and later converted into wood shavings. Wood shavings were air-dried in the laboratory for four weeks and then divided into batches of 12–15 grams each, which were placed in 20 Soxhlet extractors. The extraction process was carried out according to ASTM D1105-96 (Standard Test Method for Preparation of Extractive-Free Wood (ASTM, 2014)).

Further details regarding the preparation and storage of extracts can be found in (Hassan *et al.* 2020, Hassan *et al.* 2021). The obtained extracts were diluted using a mixture of ethanol/toluene (2:1) to achieve final concentrations suitable for treating non-durable wood.

## 2.2 Treatment of non-durable wood species and field tests

Cottonwood (CW) and southern pine (SP) sapwood boards were sourced locally and cut into blocks measuring  $12.5 \times 3.75 \times 2.5$  cm for the ground proximity test (AWPA 2014b), as well as stakes measuring  $45.7 \times 1.9 \times 1.9$  cm for the field stake test (AWPA 2014a). Non-durable wood blocks intended for field tests were conditioned at  $33^\circ\text{C}$  and  $62 \pm 3\%$  relative humidity (RH) and then treated using the method outlined by Hassan *et al.* (2021). All test samples underwent full cell vacuum pressure treatment, beginning with an initial vacuum of 91 kPa for 30 minutes and then applying pressure at 1034 kPa for 60 minutes.

Table 1: Treatment details for both field tests

Treatment	Concentration/details	Treated wood
<i>T. grandis</i>	7.5 mg/ml	SP and CW
<i>D. sissoo</i>	7.5 mg/ml	SP and CW
<i>C. deodara</i>	7.5 mg/ml	SP and CW
<i>P. roxburghii</i>	7.5 mg/ml	SP and CW
<i>T. grandis</i> + oil	4.25 mg/l + 20%	SP and CW
<i>D. sissoo</i> + oil	4.25 mg/l + 20%	SP and CW
<i>C. deodara</i> +oil	4.25 mg/l + 20%	SP and CW
<i>P. roxburghii</i> + oil	4.25 mg/l + 20%	SP and CW
oil	20%	SP and CW
Solvent (ETOH:Tol) (control)	-	SP and CW
Untreated CW and SP (control)		-
DOT (positive control)	67% (E26 only)	SP and CW
CuN (positive control)	9% (E7 only)	SP and CW
<i>T. grandis</i>	Solid heartwood	-
<i>D. sissoo</i>	Solid heartwood	-
<i>C. deodara</i>	Solid heartwood	-
<i>P. roxburghii</i>	Solid heartwood	-

Retentions of CW and SP impregnated with linseed oil (20%), heartwood extracts (7.5 mg/ml), or a mixture of each heartwood extract and oil separately (4.25 mg/ml + 20% oil) are provided in previous publications (Hassan *et al.* 2017a, Hassan *et al.* 2021). Heartwood stakes and blocks of the four durable test species were also exposed in the field for comparison.

After treatment, wood blocks and stakes were conditioned at  $33^\circ\text{C}$  with  $62 \pm 3\%$  RH and labelled with durable metal tags. Treated blocks of non-durable wood species and untreated blocks of heartwood from durable species were then exposed in a ground proximity test following the AWPA E26 standard (AWPA 2014b). Treated stakes of both non-durable wood species and untreated stakes of heartwood of the durable species were exposed in the field using ground contact field stake tests according to the AWPA E7 standard (AWPA 2014a). Each treatment was replicated five times for both tests. All samples were exposed at the Harrison Experimental Forest, north of Gulfport, Mississippi ( $30^\circ38' \text{N}$ ,  $89^\circ03' \text{W}$ ), which is within the American Wood Protection Association Deterioration Zone 5 and is considered a severe biodeterioration hazard zone. This site is dominated by pine forests and experiences a humid, subtropical climate. The dominant termites at the test location are *Reticulitermes* species, with *Reticulitermes flavipes* (Kollar) being the most common. All blocks and stakes were rated visually using a 0–10 scale as described in the AWPA standards annually for five years and then after 8 years (Hassan *et al.* 2021). After 8 years, the study was dismantled, and DOT-treated samples from the E26 ground

proximity test were stained for the presence of boron according to the AWP A68-22, *Standard method for determining penetration of boron-containing preservatives and fire retardants* (AWPA 2022)

## 2.3 Statistical Analysis

Data were averaged in Microsoft Excel and the standard error of mean was calculated.

## 3. RESULTS AND DISCUSSION

### 3.1 Ground Proximity Test

#### 3.1.1 Decay

Cottonwood blocks treated with extracts from all wood species except *C. deodara* were severely attacked by decay fungi after five years of exposure, and failed after eight years, with an average rating below 1. Conversely, after five years of exposure, cottonwood blocks treated with *C. deodara* extracts showed slightly higher ratings than blocks treated with extracts from other wood species. However, these blocks received ratings of < 4 but did not fail. Southern pine wood blocks treated with *D. sissoo* extracts failed after five years in the field. After five years, SP samples treated with extracts from three other wood species were severely attacked by decay fungi, receiving ratings of less than 4, but failed after eight years of exposure, except samples treated with *T. grandis* extracts that showed a damage rating of greater than 1 (Table 2).

Cottonwood and SP blocks treated with a combination of heartwood extract and linseed oil, or linseed oil alone, exhibited higher damage ratings than samples treated with extracts alone. Cottonwood samples were severely attacked by decay fungi, with damage ratings between 4 and 5 after five years and < 4 after eight years of exposure in the field. However, CW samples treated with *C. deodara* extract + oil showed higher resistance to decay fungi after eight years than those treated with other extract-oil mixtures or oil alone. Similarly, SP wood blocks treated with *C. deodara* extract + oil showed higher resistance than others. They showed moderate to severe damage after five and eight years of exposure, with an average rating between 7.2 and 6.4. In contrast, SP blocks treated with other extracts and oil mixtures showed lower ratings (between 4.4 and 3.2) and were severely attacked by decay fungi. DOT-treated SP and CW samples showed trace damage to no damage by decay fungi after 8 years in the field (Table 2).

Table 3 shows the average decay damage rating for solid untreated durable wood blocks exposed in the ground proximity test. After eight years of exposure, heartwood blocks of *D. sissoo* exhibited only a slight attack. *Tectona grandis* and *C. deodara* blocks were moderately attacked by decay fungi during this period. However, *P. roxburghii* blocks suffered severe attacks after five and eight years, with an average rating of <2.

#### 3.1.2 Termite

Within five years of exposure, CW treated with *D. sissoo* extracts failed (average rating <1), and blocks treated with the other three types of heartwood extracts showed slightly higher resistance against termites. Still, these were severely attacked by termites after five and eight years of exposure. Cottonwood blocks treated with *C. deodara* extracts were more resistant to termites than samples treated with the other three wood extracts. Like CW blocks, SP blocks treated with *D. sissoo* extracts showed the least resistance against termites, and all blocks treated with all types of wood extracts failed after eight years of exposure in the field except samples treated with *T. grandis* extracts that showed an average damage rating of 2 after eight years in the field (Table 2).

As seen in decay tests, the mixture of oil and extracts or oil only provided more resistance to CW and SP blocks than the blocks treated with extracts. All CW samples treated with oil + extract mixture were severely attacked by the termites with an average rating of <5 except samples treated with *C. deodara* + oil, which showed moderate to severe attack by termites with an average rating of >6 after eight years of exposure in the field. Southern pine wood blocks treated with *C. deodara* + oil showed more resistance against termites, with an average rating of 7, than samples treated with other extracts and oil mixture, with a rating score between 3.2 and 6.

No termite damage was observed on DOT-treated SP samples, and DOT-treated CW blocks exhibited only slight attack after eight years in the field. The presence and diffusion of DOT were confirmed by spraying a crosscut section of both cottonwood and southern pine (Fig. 1).

Table 3 shows the average termite damage rating for solid untreated durable wood blocks exposed in the ground proximity test. After eight years of exposure, heartwood blocks of *T. grandis*, *D. sissoo*, and *C. deodara* exhibited only a slight attack by termites. However, *P. roxburghii* blocks suffered severe attacks after five and eight years, with an average rating of <2.

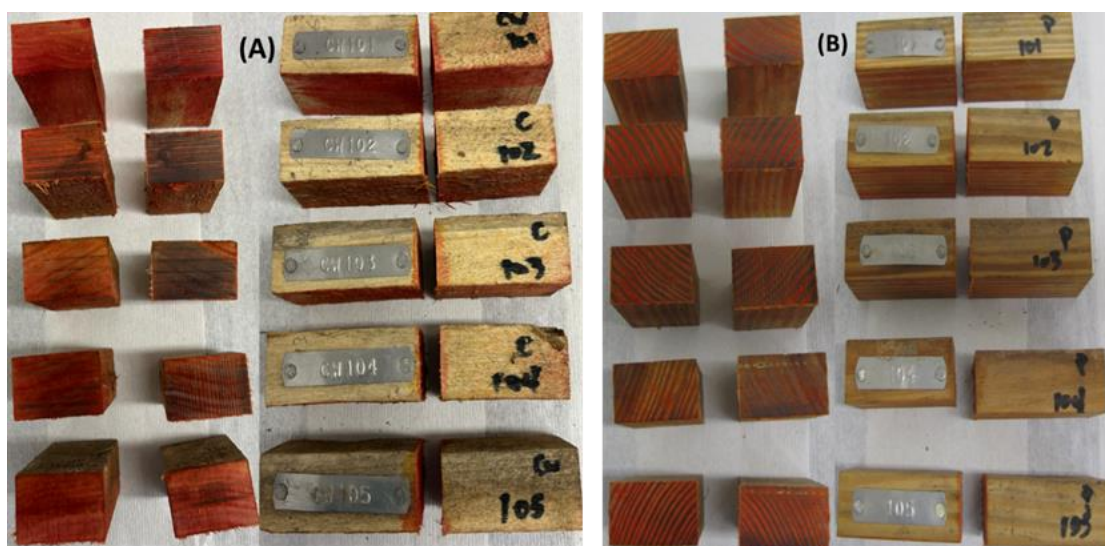


Figure 1: Examples of crosscut samples of cottonwood (A) and southern pine (B) positioned outside with tags, followed by cross-sections of turned-up samples stained with boron indicator solution.

Table 3: Average decay and termite damage ratings for solid untreated durable and non-durable wood species exposed for eight years in an AWP E26 ground proximity test.

Wood type	AWPA E26			
	Decay		Termite	
	5Yrs	8Yrs	5Yrs	8Yrs
<i>T. grandis</i>	8.0	8.4	10	9.1
<i>D. sissoo</i>	9.6	9.2	9.8	9
<i>C. deodara</i>	8.2	8.4	10	8.8
<i>P. roxburghii</i>	1.6	1.2	1.4	1.2
Untreated SP	0	0	0	0
Untreated CW	0	0	0	0

Table 2: Average decay and termite damage rating for treated cottonwood and southern pine exposed in the field for eight years following the AWP E26 ground proximity test.

Treatment	Southern pine				Cottonwood			
	Decay		Termite		Decay		Termite	
	5Yrs	8Yrs	5Yrs	8Yrs	5Yrs	8Yrs	5Yrs	8Yrs
<i>T. grandis</i>	3.2	1.8	2.0	1.6	2.4	0.8	3.2	2.4
<i>D. sissoo</i>	0	0	0.8	0	0.8	0	0	0
<i>C. deodara</i>	2.8	0	1.2	0	4.2	2.0	4.8	4.2
<i>P. roxburghii</i>	1.6	0	1.2	1.2	1.6	0	2	0.8
<i>T. grandis</i> + oil	4.8	3.6	8.0	3.2	4.4	3.4	6.6	4.2
<i>D. sissoo</i> + oil	6.8	4.4	7.6	5.0	4.6	1.4	5.4	4.6
<i>C. deodara</i> +oil	7.2	6.4	7.8	6.8	5.0	4.8	7.2	6.4
<i>P. roxburghii</i> + oil	5.6	3.2	7.0	4.4	2.4	0	5.2	2.4
Oil	6.6	4.4	7.6	6.0	4.4	2.4	6.6	3.6
Solvent	0	0	0	0	0.8	0	2.4	0
DOT	9.6	10	10	9.9	9.0	9.6	10	9.0

## 3.2. Field Stake Test

### 3.2.1 Decay

Table 4 illustrates the average decay damage ratings at years five and eight for CW and SP test specimens exposed in the ground contact field stake test. Cottonwood stakes treated with various wood extracts or extracts + oil combinations failed after eight years, except those treated with *D. sissoo* + oil, demonstrating slightly higher resistance with an average rating of 1.5. Similarly, SP blocks treated with extracts alone or mixed with oil showed severe decay by fungi at five years and eventual failure by eight years, except for those treated with *D. sissoo* + oil and *P. roxburghii* + oil, which had slightly higher decay damage ratings (<1.5) (Table 4).

Cottonwood stakes treated with CuN showed minor decay after five years but experienced severe decay with an average rating of <5 after eight years. Southern pine stakes treated with CuN exhibited minimal damage after five years but were moderately attacked by fungi after eight years (Table 4).

Table 5 shows the average decay rating for untreated durable and non-durable solid wood stakes exposed in the field stake test. Untreated solid heartwood of *Dalbergia sissoo* and *C. deodara* heartwood stakes were severely attacked, with 50-75% of the cross-section area impacted by decay fungi. *Tectona grandis* stakes showed moderate resistance and were moderately attacked by decay fungi. Untreated CW, SP failed after five years, while *P. roxburghii* stakes failed after eight years of exposure in the field.

### 3.2.1 Termite

Table 4 presents the average termite damage ratings over eight years for CW and SP test specimens subjected to the ground contact field stake test. Like decay, CW stakes treated with various wood extracts or extracts + oil combinations failed after eight years, except those treated with *D. sissoo* + oil, which exhibited slightly higher resistance against termites with an average rating of 2.2. Likewise, SP blocks treated with extracts alone or mixed with oil showed severe termite damage at five years and eventual failure by eight years, except for those treated with *D. sissoo* + oil and *P. roxburghii* + oil, which had slightly higher damage ratings (1.7-1.2).

Cottonwood stakes treated with CuN experienced surface etching by termites after eight years. SP stakes treated with CuN were moderately attacked by termites during this period, with an average rating of 8 (Table 4).

Table 5 shows termite resistance ratings for solid untreated durable wood assessed in the field stake test. *Pinus roxburghii* heartwood stakes failed after eight years due to termite attack, whereas *D. sissoo* heartwood stakes were severely impacted, with 30-50% of the cross-section area affected by termite activity. *Cedrus deodara* heartwood stakes showed moderate termite damage after eight years in the field, while *T. grandis* stakes exhibited slight surface nibbling by termites with an average rating of 9.2 after eight years in the field.



Table 4: Average decay and termite damage ratings for treated cottonwood and southern pine exposed for eight years in an AWP A E7 field stake test.

Treatment	Southern pine				Cottonwood			
	Decay		Termite		Decay		Termite	
	5Yrs	8Yrs	5Yrs	8Yrs	5Yrs	8Yrs	5Yrs	8Yrs
<i>T. grandis</i>	0	0	0	0	0	0	0	0
<i>D. sissoo</i>	1.6	0	1.2	0	0	0	0	0
<i>C. deodara</i>	0	0	0	0	0	0	0	0
<i>P. roxburghii</i>	0	0	0	0	0	0	0	0
<i>T. grandis</i> + oil	2.8	0	3.8	0	0.8	0	0	0
<i>D. sissoo</i> + oil	4.4	1.0	5.4	1.0	3.0	1.5	4.8	2.2
<i>C. deodara</i> +oil	2.4	0	3.2	0	0	0	0	0
<i>P. roxburghii</i> + oil	3.0	1.5	6.0	1.7	0	0	0.8	0
Oil	3	0	3.4	1.2	0.8	0	4.2	0
Solvent	1.6	0	1.4	0	0	0	0	0
CuN	9.4	8.0	10	8	8.8	4.4	10	9.6

Table 5: Average decay and termite damage ratings for solid untreated durable and non-durable wood species exposed for eight years in an AWPA E7 field stake test.

Wood type	AWPA E7			
	Decay		Termite	
	5Yrs	8Yrs	5Yrs	8Yrs
<i>T. grandis</i>	9.0	7.6	10	9.2
<i>D. sissoo</i>	9.2	4.2	10	5.6
<i>C. deodara</i>	7.8	4.0	9.8	8.33
<i>P. roxburghii</i>	2.6	0	2.0	0
Untreated SP	0	0	0	0
Untreated CW	0	0	0	0

The increasing focus on environmental regulation and public concerns regarding the toxicity of synthetic wood preservatives has prompted endeavours to reduce biocide usage and explore new, less harmful wood preservation methods utilizing natural compounds. Heartwood extracts derived from naturally durable wood species can be considered substitutes for chemical wood preservatives; however, there is a dearth of field data on their long-term performance. The current study explored the combined effectiveness of extracts and linseed oil as wood preservatives. Prior studies have indicated that supplementing heartwood extracts with other substances may enhance their potency against termites and decay fungi (Hassan *et al.* 2021). Mixing linseed oil (at a concentration of 20%) with the extracts boosted their efficacy as wood protectants. Synergistic effects of plant oils, sharing similar modes of action with certain compounds, have been tested against termites, showing lethal effects and facilitating penetration of toxicants into wood, thereby protecting it from termites and fungi (Terziev and Panov 2010). One plausible mechanism suggests that the oil creates a hydrophobic barrier, displacing water within treated wood and thus hindering biological attack (González-Laredo *et al.* 2015, Singh and Singh 2012). Our findings revealed that combining oil and heartwood extracts outperformed the extracts alone in wood protection. However, they were not as effective as oil-based CuN or water-based disodium DOT in ground contact and protected ground proximity tests, respectively. This disparity could stem from the tested extracts or oil concentrations falling below the necessary threshold for effective defence against termites and decay fungi. Some CuN-treated CW and SP samples also experienced decay, possibly attributable to copper-tolerant decay fungi (Green III and Clausen 2005).

#### 4. CONCLUSIONS

The current study suggests that combining certain heartwood extracts with linseed oil provides some level of protection against termite and decay attack in field exposure tests for treated non-durable wood species, compared to untreated or extract-only treated non-durable wood. However, these protective effects were not as robust as those observed with synthetic preservatives such as CuN or DOT in protecting non-durable wood species.

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