

## Mould and Termite Resistance of Heat-Treated Loblolly Pine Wood (*Pinus taeda*) and Rubberwood (*Hevea brasiliensis*) in Mixture of Oleoresin and Used Cooking Oil

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### ABSTRACT

This study investigated the effects of heat treatment using a mixture of natural oleoresin and used cooking oil on the mould and termite resistance of loblolly pine wood (*Pinus taeda*) and rubberwood (*Hevea brasiliensis*). Oleoresin, a non-timber forest product derived from Dipterocarp species native to Southeast Asia, was sourced from central Vietnam, while used cooking oil was obtained from an oil-recycling facility. Wood samples were heat-treated at varying oleoresin ratios (20 – 40 %), temperatures (130 – 180 °C), and durations (90 – 180 min). Response surface methodology was applied to assess the influence of these factors on mould and termite resistance under laboratory conditions. For mould resistance, specimens were evaluated at intervals of 1, 2, 4, and 8 weeks following inoculation with a conidial mixture of five mould species isolated from infected wood. Termite resistance was assessed according to Vietnamese standard TCVN 11355:2016, which aligns with the European standard EN 118:2013, using subterranean termites (*Coptotermes* spp.). Results demonstrated significant improvements in the mould and termite resistance of both loblolly pine wood and rubberwood, allowing for the identification of optimal treatment parameters for heat treatment using oleoresin combined with used cooking oil.

**Keywords:** oleoresin, heat treated loblolly pine wood, heat treated rubberwood, mould termite resistance

### INTRODUCTION

Loblolly pine wood (*Pinus taeda*), imported from the Southeastern United States, and Rubberwood (*Hevea brasiliensis*), sourced from Southeast Asian plantations, are widely used in Southeast Asia's wood industries. Loblolly pine wood is essential for furniture and construction, while rubberwood is valued for its sustainability and export potential. However, both face limitations: they are largely restricted to interior applications due to low strength, decay resistance, and susceptibility to biological degradation in exterior use. These challenges highlight the need for improved processing or treatment to enhance durability and expand their applications.

Oil heat treatment has been recognized as an effective technique for enhancing wood properties, particularly dimensional stability and decay resistance, thereby increasing its suitability for both interior and exterior applications (Cao et al., 2020; H'ng et al., 2012). This environmentally friendly approach improves the physical and mechanical properties of wood while mitigating the ecological impact associated with conventional chemical treatments (Lee et al., 2018; Mandraveli et al., 2024). Recent studies have demonstrated that the application of oils such as rapeseed, palm, linseed, and thyme significantly enhances water repellency, dimensional stability, and natural durability in various wood species (Bessala et al., 2023; Kaya et al., 2023; Mastouri et al., 2021; Suri et al., 2023; Umar et al., 2016).

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Natural oleoresins, primarily derived from *Dipterocarpus* species in Southeast Asia, have long been utilized in industrial applications such as varnishes, paints, and sealants (Dao, 2004; Luu & Pinto, 2007). Recent studies have demonstrated that oleoresin-based heat treatments enhance the physical properties and biological resistance of bamboo (Tang & Nguyen, 2021; Tang et al., 2022).

Expanding on these findings, this study investigates the effects of heat treatment using a mixture of oleoresin and used cooking oil on the physical, mechanical, and biological properties of loblolly pine wood and rubberwood. Our previous research highlighted the efficacy of this treatment in improving dimensional stability and hardness in both species. To further evaluate its potential, this study investigates the mould and termite resistance of heat-treated wood in mixture of oleoresin and used cooking oil. Mould resistance was assessed under both laboratory and field conditions, while termite resistance was evaluated under controlled laboratory conditions.

## MATERIALS AND METHODS

### Loblolly Pine and Rubberwood Samples

Loblolly pine (*Pinus taeda*) samples were obtained from a timber and wood product manufacturing factory in Ho Chi Minh City and rubberwood (*Hevea brasiliensis*) samples were sourced from a rubberwood product manufacturing facility in Binh Duong Province, Vietnam.

Samples were cut to standardized dimensions of 25 mm in thickness, 90 mm in width, and 600 mm in length, then kiln-dried to a moisture content of 10 – 12 %. The samples were subsequently divided into 15 treatment groups and one control group (untreated samples), with each group consisting of five replicates.

### Oleoresin and Used Cooking Oil

The oil mixture for the treatment consisted of oleoresin and used cooking oil, applied in the investigated proportions. Oleoresin, extracted from *Dipterocarpus alatus* with a viscosity of 190 cP, was sourced from a company exploiting natural oil in Quang Nam Province, Vietnam. Used cooking oil, obtained from a recycled oil production facility in Ho Chi Minh City, had a viscosity of 30 cP at room temperature.

### Response Surface Methodology and Central Composite Design

In this study, the central composite design (CCD) within the framework of response surface methodology (RSM) was utilized to analyse the effects of oleoresin ratio (%), treatment temperature (°C), and treatment duration (min) on mould and termite resistance. A 15-run experimental design, incorporating cube, centre, and axial points, was implemented using Minitab version 21.2. The cube points were set at 20 % and 40 % for oleoresin ratio, 130 °C and 180 °C for treatment temperature, and 90 min and 180 min for treatment duration. Axial points extended beyond these levels, ranging from 13 % to 47 % for oleoresin ratio, 113 °C to 197 °C for temperature, and 59 min to 211 min for duration. The experimental conditions, determined through RSM with CCD, are summarized in Table 1.

**Table 1: The treatment schedules developed using central composite design**

Run	Oleoresin ratio (%)	Temperature (°C)	Duration (min)
1	20	130	90
2	40	130	90
3	20	180	90
4	40	180	90
5	20	130	180
6	40	130	180
7	20	180	180
8	40	180	180
9	13	155	135
10	47	155	135
11	30	113	135
12	30	197	135
13	30	155	59
14	30	155	211
15	30	155	135

### Oleoresin Heat Treatment

The loblolly pine and rubberwood samples were fully submerged in a custom-designed apparatus for oil curing, following the specific heat treatment schedules outlined in Table 1. This specialized oil-curing apparatus consisted of a rectangular stainless-steel container with internal dimensions of 680 mm (length) × 380 mm (width) × 200 mm (depth). The system was equipped with an electric resistance heater for precise temperature control, which was monitored using a thermocouple connected to a digital temperature controller.

### Mould Testing

Specimens with dimensions of 20 mm × 20 mm in cross-section and 70 mm in length were taken from both treated and control wood samples. Five specimens from each experimental run were placed in plastic boxes (200 mm × 130 mm × 65 mm) and exposed to artificial infection with a water-based mixture of conidia from five mould species: *Aspergillus niger*, *Aspergillus flavus*, *Aspergillus oryzae*, *Aspergillus* sp., and *Penicillium* sp. using a small brush. These moulds were isolated from infected loblolly pine wood and rubberwood and identified at the Institute for Biotechnology and Environment, Nong Lam University – Ho Chi Minh City, Vietnam. The exposure was conducted in an incubation room at 30 °C and 75 % relative humidity. Table 2 outlines the rating method used to assess mould growth, which was evaluated after 1, 2, 4, and 8 weeks based on CEN/TS 15082 (British Standards Institution [BSI], 2005).

**Table 2: Standard method for rating the infection on the surface**

Rating	Description	Definition
0	0 %	Clean
1	< 10 % coverage	Slight
2	10 % - 25 % coverage	Medium
3	26 % - 50 % coverage	Heavy
4	> 50 % coverage	Severe

### Termite Testing

Termite resistance was assessed following the Vietnamese standard TCVN 11355:2016 (Commission for Standards, Metrology and Quality of Vietnam, 2016), which is based on the European standard EN 118:2013 (BSI, 2013), using subterranean termites (*Coptotermes* spp.). Test specimens with dimensions of 50 mm × 50 mm × 15 mm were prepared and conditioned at 20 ±2 °C and 65 ±5 % relative humidity for two weeks prior to testing.

Each test specimen was attached to a glass tube (110 mm in length, 25 mm in interior diameter) using adhesive. The tube contained moistened sand, prepared at a ratio of one volume of water to four volumes of sand, with a 1 mm-thick untreated loblolly pine or rubberwood disc (23 mm in diameter) placed at the bottom. Additionally, a small wood fragment (approximately 0.5 g) was buried in the sand 40 mm above the test specimen, while the moistened sand extended to a height of 70 mm.

A colony of 250 worker termites, along with 3 soldiers and nymphs, was introduced into each glass tube. To prevent water evaporation and termite escape, the tube was covered with aluminium foil. All test assemblies were placed on individual trays for containment and kept in a controlled environmental chamber at 26 ±2 °C and 70 ±5 % relative humidity, under ventilated conditions and protected from light. The moisture content of the sand was regularly monitored and re-moistened throughout the test period.

At the end of the eight-week exposure period, each tube was uncovered, and the number of live worker termites was counted to determine worker termite mortality. The specimens were then removed from the test assemblies and visually assessed for termite damage, using the rating scale defined in Table 3.

**Table 3: Standard method for rating the termite damage**

Rating	Description
0	No attack
1	Attempted attack
2	Slight damage
3	Moderate damage
4	Severe damage

## RESULTS AND DISCUSSION

### Mould Resistance Test

The mould infection grading results for loblolly pine wood and rubberwood, heat-treated with a mixture of oleoresin and used cooking oil after 1, 2, 4, and 8 weeks of exposure, are summarized in Tables 4 and 5. For loblolly pine wood, six treatment runs completely prevented mould infection, while the remaining nine exhibited slight to moderate growth. In contrast, only three treatment runs fully inhibited mould infection in rubberwood, while one run showed heavy infection, and the others ranged from slight to moderate growth. Control specimens of both wood species developed severe mould infections. However, rubberwood controls exhibited severe infection faster, showing extensive mould growth by week 4. At the same time, loblolly pine wood controls displayed only heavy infection.

To evaluate the key factors influencing mould infection grade, a second-order polynomial model was developed using the CCD. This approach allowed for the assessment of linear, quadratic, and interaction effects. Significant factors were identified through Analysis of Variance (ANOVA) and a Pareto chart of standardized effects, with the vertical line on the chart representing the 95 % confidence threshold.

**Table 4: Mould infection grade of loblolly pine wood heat-treated with a mixture of oleoresin and used cooking oil**

Run	Oleoresin ratio (%)	Temperature (°C)	Duration (min)	Exposure time			
				After 1 week	After 2 weeks	After 4 weeks	After 8 weeks
1	20	130	90	0.2 ±0.4	0.4 ±0.5	0.6 ±0.5	1.2 ±0.4
2	40	130	90	0 ±0	0.2 ±0.4	0.4 ±0.5	0.8 ±0.4
3	20	180	90	0 ±0	0 ±0	0 ±0	0.2 ±0.4
4	40	180	90	0 ±0	0 ±0	0 ±0	0 ±0
5	20	130	180	0 ±0	0 ±0	0.2 ±0.4	0.6 ±0.5
6	40	130	180	0 ±0	0 ±0	0.2 ±0.4	0.4 ±0.5
7	20	180	180	0 ±0	0 ±0	0 ±0	0 ±0
8	40	180	180	0 ±0	0 ±0	0 ±0	0 ±0
9	13	155	135	0 ±0	0 ±0	0.2 ±0.4	0.4 ±0.5
10	47	155	135	0 ±0	0 ±0	0 ±0	0 ±0
11	30	113	135	0.2 ±0.4	0.4 ±0.5	0.6 ±0.5	1.4 ±0.5
12	30	197	135	0 ±0	0 ±0	0 ±0	0 ±0
13	30	155	59	0 ±0	0 ±0	0 ±0	0.2 ±0.4
14	30	155	211	0 ±0	0 ±0	0 ±0	0 ±0
15	30	155	135	0 ±0	0 ±0	0 ±0	0.2 ±0.4
Control	-	-	-	0.8 ±0.4	1.6 ±0.5	2.6 ±0.5	4 ±0

Note: Values after ± are standard deviations.

**Table 5: Mould infection grade of rubberwood heat-treated with a mixture of oleoresin and used cooking oil**

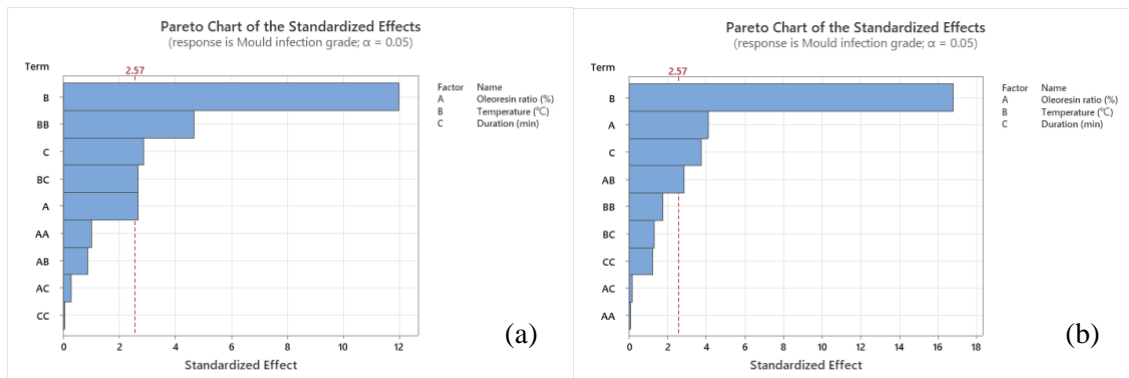
Run	Oleoresin ratio (%)	Temperature (°C)	Duration (min)	Exposure time			
				After 1 week	After 2 weeks	After 4 weeks	After 8 weeks
1	20	130	90	0.4 ±0.5	0.8 ±0.4	1.2 ±0.4	2 ±0
2	40	130	90	0.2 ±0.4	0.4 ±0.5	1 ±0	1.4 ±0.5
3	20	180	90	0 ±0	0 ±0	0 ±0	0 ±0
4	40	180	90	0 ±0	0 ±0	0 ±0	0 ±0
5	20	130	180	0.4 ±0.5	1 ±0	1.6 ±0.5	2.4 ±0.5
6	40	130	180	0.2 ±0.4	0.6 ±0.5	1.2 ±0.4	1.6 ±0.5
7	20	180	180	0 ±0	0 ±0	0.2 ±0.4	0.6 ±0.5
8	40	180	180	0 ±0	0 ±0	0.2 ±0.4	0.6 ±0.5
9	13	155	135	0.2 ±0.4	0.4 ±0.5	1 ±0	1.6 ±0.5
10	47	155	135	0 ±0	0.2 ±0.4	0.4 ±0.5	0.6 ±0.5
11	30	113	135	0.8 ±0.4	1.2 ±0.4	2 ±0	3 ±0
12	30	197	135	0 ±0	0 ±0	0 ±0	0 ±0
13	30	155	59	0 ±0	0.2 ±0.4	0.4 ±0.5	0.6 ±0.5
14	30	155	211	0.2 ±0.4	0.4 ±0.5	0.8 ±0.4	1 ±0
15	30	155	135	0 ±0	0.2 ±0.4	0.8 ±0.4	1 ±0
Control	-	-	-	2.2 ±0.4	3 ±0	4 ±0	4 ±0

Note: Values after ± are standard deviations.

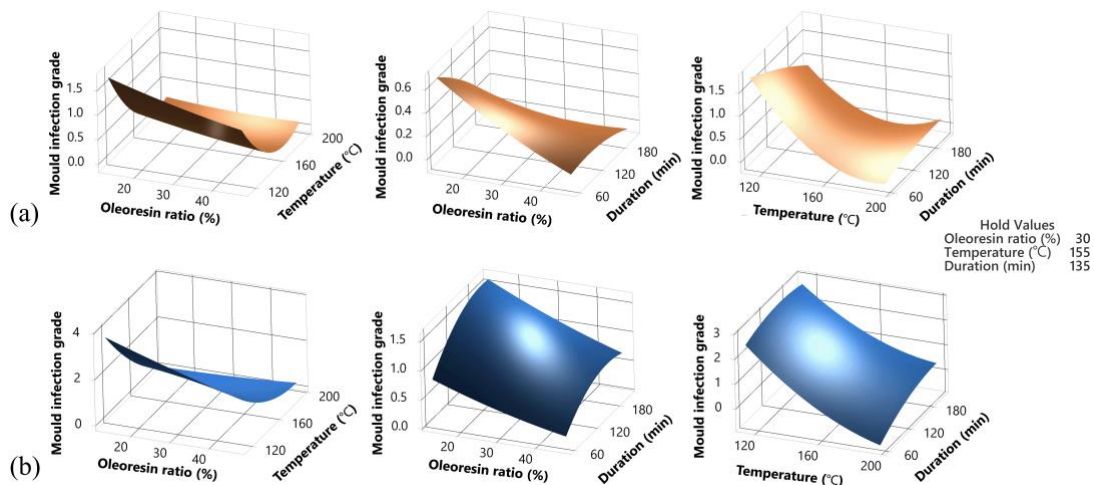
Analysis of ANOVA results and the Pareto chart (Figure 1) revealed that treatment temperature, duration, and oleoresin ratio significantly influenced the mould infection grade

of both loblolly pine and rubberwood, with temperature exerting the greatest effect. Higher treatment temperature resulted in lower mould infection grades, indicating improved resistance (Figure 2). These findings align with previous studies on the antifungal properties of oil heat treatment (Lacić et al., 2014; Suri et al., 2023; Tang et al., 2022).

The improvement in mould resistance can be attributed primarily to the blocking of internal channels responsible for nutrient exchange, as well as the degradation of polysaccharides and starch following oil heat treatment (Hao et al., 2021; Okon & Udoakpan 2019). Furthermore, the treatment reduces hygroscopicity and facilitates oleoresin penetration into wood cavities, limiting moisture absorption and creating an environment unfavourable for mould growth (Hill et al., 2021; Lee et al., 2018; Mandraveli et al., 2024).



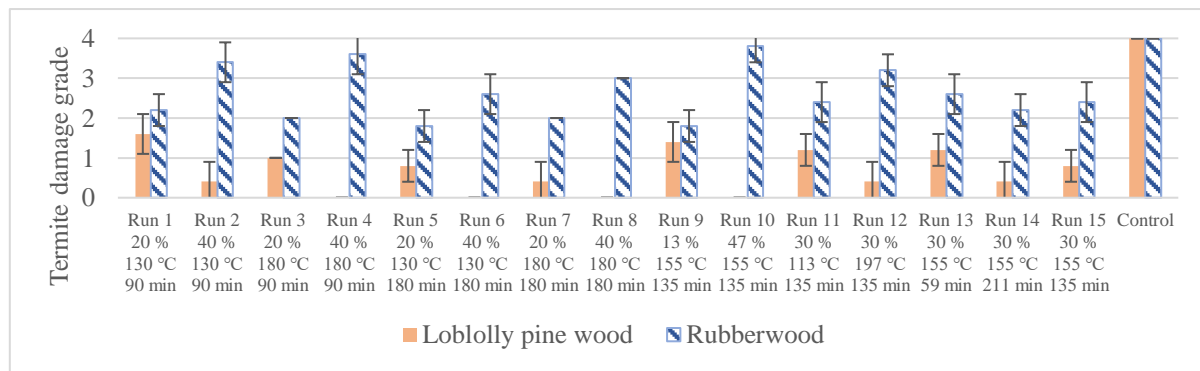
**Figure 1: Pareto chart illustrating the standardized effects of the treatment variables on the mould infection grade of loblolly pine wood (a) and rubberwood (b) heat-treated with a mixture of oleoresin and used cooking oil**



**Figure 2: Three-dimensional surface plot of the mould infection grade for the loblolly pine wood (a) and rubberwood (b) as a function of oleoresin ratio, temperature, and duration**

### Termite Resistance Test

Termite damage grades for loblolly pine and rubberwood, heat-treated with a mixture of oleoresin and used cooking oil after eight weeks of exposure, are illustrated in Figure 3. For loblolly pine, four treatment runs completely prevented termite attack, while the remaining runs exhibited varying degrees of damage, ranging from attempted attacks to slight damage. In contrast, rubberwood showed slight termite damage in seven treatment runs, with the remaining runs experiencing moderate to severe damage.



**Figure 3: Termite damage grade of the loblolly pine wood and rubberwood samples heat-treated with a mixture of oleoresin and used cooking oil**

Regarding termite mortality, termites were completely eradicated in three experimental runs for loblolly pine, while the remaining runs had survival rates below 5 %. In rubberwood, termite mortality ranged from 61 % to 80 %, with the highest mortality rates recorded in runs 5 and 9 (Table 6).

**Table 6: Termite mortality of the test for loblolly pine wood and rubberwood samples heat-treated with a mixture of oleoresin and used cooking oil after 8 weeks of exposure**

Run	Oleoresin ratio (%)	Temperature (°C)	Duration (min)	Termite mortality for loblolly pine wood test (%)	Termite mortality for rubberwood test (%)
1	20	130	90	95 ±2	75 ±1
2	40	130	90	98 ±1	63 ±2
3	20	180	90	96 ±1	75 ±1
4	40	180	90	100 ±0	65 ±2
5	20	130	180	97 ±1	80 ±2
6	40	130	180	100 ±0	72 ±2
7	20	180	180	98 ±2	75 ±1
8	40	180	180	100 ±0	71 ±1
9	13	155	135	95 ±2	80 ±3
10	47	155	135	100 ±0	61 ±2
11	30	113	135	97 ±2	74 ±1
12	30	197	135	99 ±1	70 ±3
13	30	155	59	95 ±3	72 ±2
14	30	155	211	98 ±1	74 ±1
15	30	155	135	97 ±2	71 ±2
Control	-	-	-	36 ±3	27 ±2

*Note: Values after ± are standard deviations.*

The ANOVA results and Pareto charts (Figures 4 & 5) indicated that oleoresin ratio, treatment duration, and temperature significantly influenced termite damage grade and termite mortality in both loblolly pine wood and rubberwood. However, termite mortality for rubberwood test was primarily affected by oleoresin ratio and treatment duration, with the oleoresin ratio exerting the most substantial impact on both termite damage grade and mortality.

For loblolly pine, increased oleoresin ratio resulted in lower termite damage grades and higher termite mortality rates. In contrast, for rubberwood, optimal termite resistance was

achieved with lower oleoresin ratios. These findings align with previous research on the anti-termite properties of oil heat treatment (Lee et al., 2018; Lyon et al., 2007; Manola & Garcia 2012).

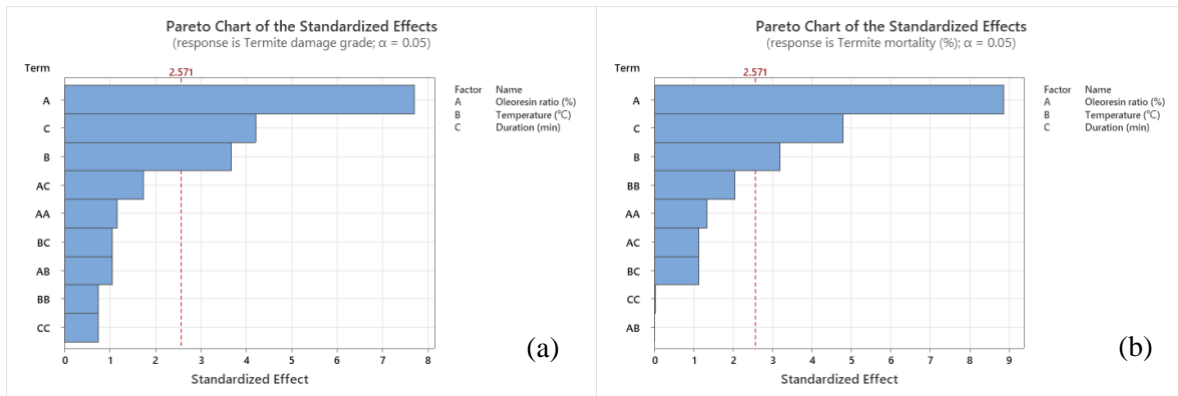


Figure 4: Pareto chart illustrating the standardized effects of the treatment variables on the termite damage grade (a) and the termite mortality (b) of the loblolly pine wood heat-treated with a mixture of oleoresin and used cooking oil

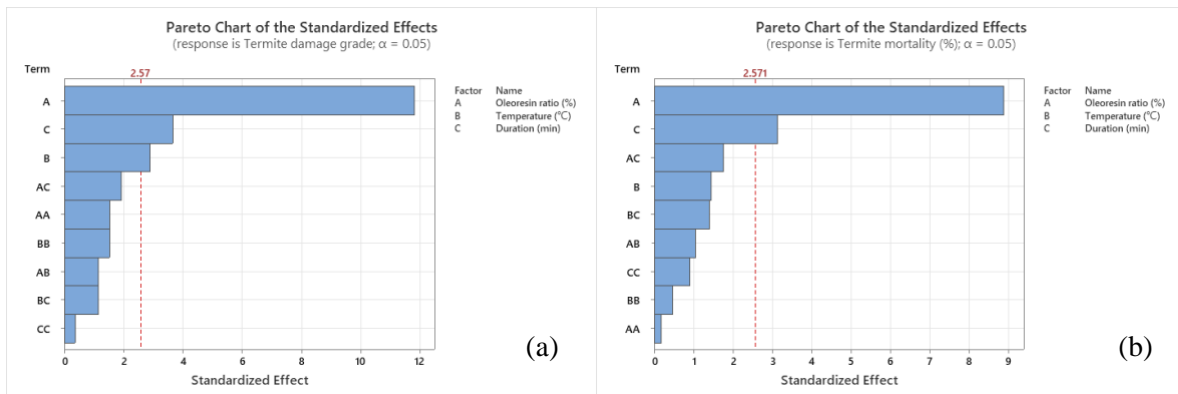


Figure 5: Pareto chart illustrating the standardized effects of the treatment variables on the termite damage grade (a) and the termite mortality (b) of the rubberwood heat-treated with a mixture of oleoresin and used cooking oil

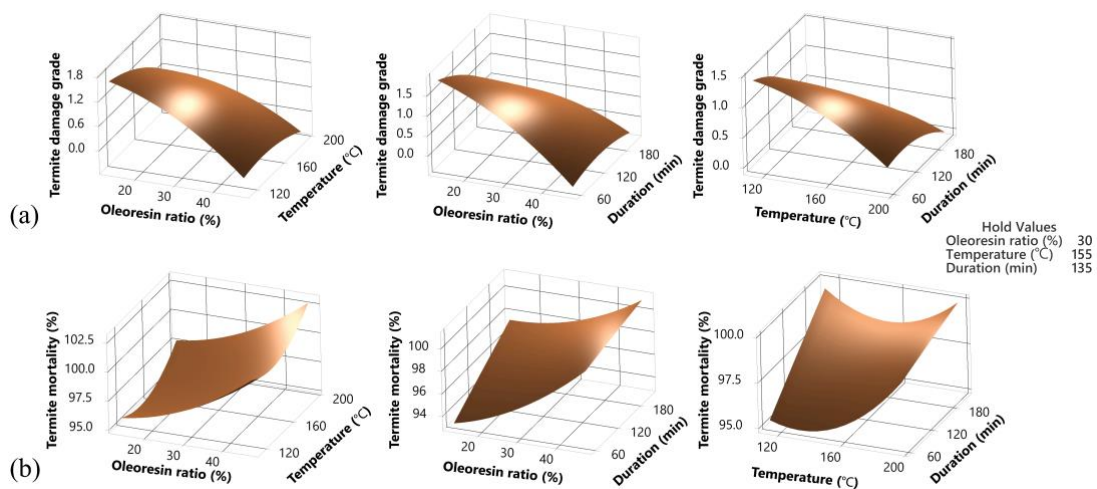
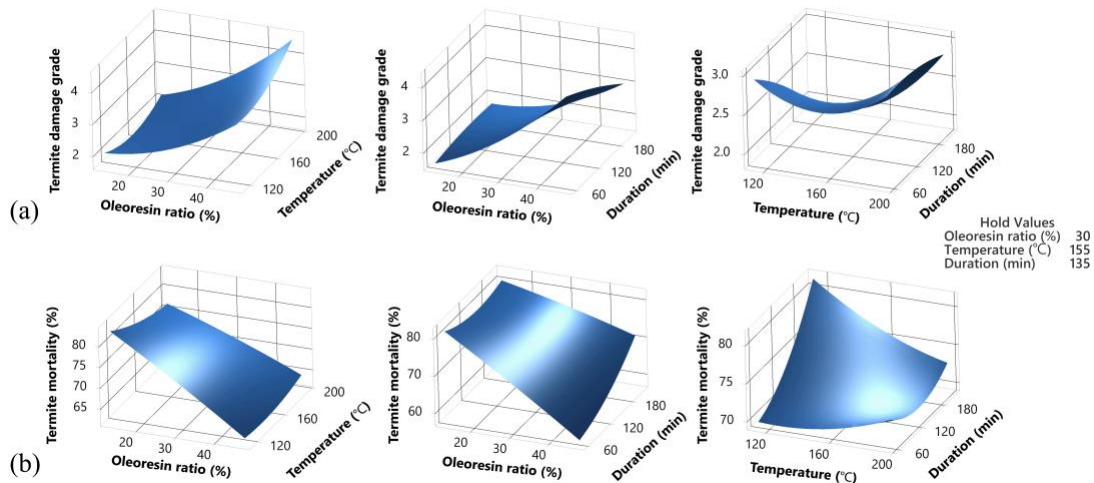


Figure 6: Three-dimensional surface plot of the termite damage grade (a) and termite mortality (b) for the loblolly pine wood as a function of oleoresin ratio, temperature, and duration





**Figure 7: Three-dimensional surface plot of the termite damage grade (a) and termite mortality (b) for the rubberwood as a function of oleoresin ratio, temperature, and duration**

### Regression Analysis and Model Adequacy

Model adequacy was evaluated using the coefficient of determination (R-squared), a statistical measure that indicates the goodness of fit. The R-squared values for mould infection grade, termite damage grade, and termite mortality in heat-treated loblolly pine wood were found to be 98.14 %, 95.1 %, and 96.06 %, respectively. Similarly, for rubberwood, the corresponding R-squared values were 98.67 %, 97.22 %, and 95.17 %. These high values demonstrate strong agreement between the predicted and experimental data, indicating that the model accounts for more than 95 % of the variability, with less than 5 % residual error.

To further confirm the robustness of model, the adjusted R-squared values were computed, yielding 94.78 %, 86.28 %, and 88.97 % for loblolly pine wood, and 96.28 %, 92.21 %, and 86.49 % for rubberwood, respectively. These results reinforce the reliability of model and its applicability to field conditions.

Following model fitting, ANOVA assumptions were verified to ensure statistical validity. The normality of residuals was assessed using the Ryan-Joiner normality test, which is well-suited for small sample sizes (<50). A p-value greater than 0.05 indicates that the residuals follow a normal distribution, while a value below this threshold suggests significant deviation. In this study, the Ryan-Joiner test produced non-significant results (p-value > 0.1), and the residuals closely followed a straight-line pattern, confirming normality and the reliability of model predictions (Figure 8 & 9).

The final predictive equations, expressed in terms of significant actual factors, describe the influence of key variables on mould infection grade, termite damage grade, and termite mortality in loblolly pine and rubberwood heat-treated with a mixture of oleoresin and used cooking oil.

For loblolly pine wood, the regression models are as follows:

$$\text{Mould infection grade} = 12.51 - 0.01074 R - 0.1235 T - 0.01627 t + 0.000311 T^2 + 0.000089 T \times t$$

$$\text{Termite damage grade} = 3.835 - 0.04194 R - 0.00804 T - 0.00511 t$$

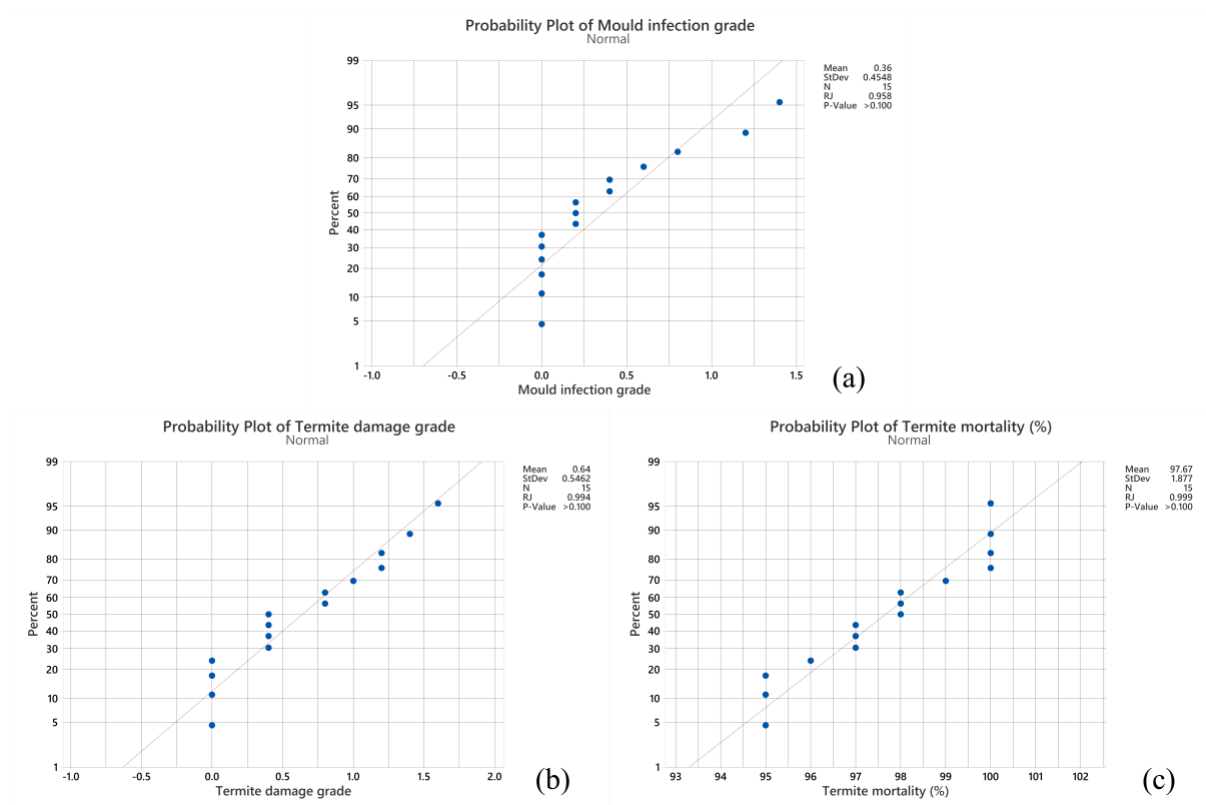
$$\text{Termite mortality (\%)} = 87.44 + 0.1488 R + 0.02158 T + 0.01794 t$$

For rubberwood, the corresponding models are:

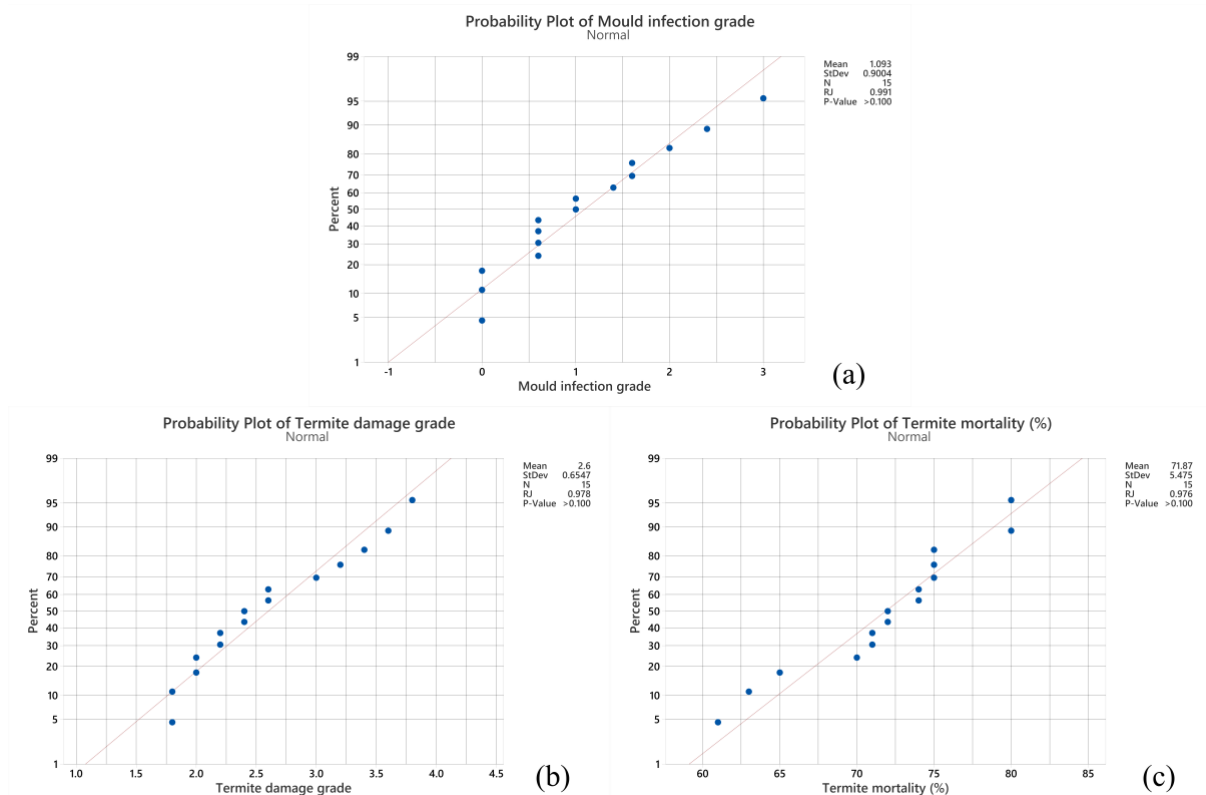
$$\text{Mould infection grade} = 16.59 - 0.131 R - 0.1465 T + 0.00401 t + 0.000298 T^2 + 0.0007 R \times T$$

$$\text{Termite damage grade} = 1.4 + 0.05806 R - 0.00401 t$$

$$\text{Termite mortality (\%)} = 81.18 - 0.4811 R + 0.0379 t$$



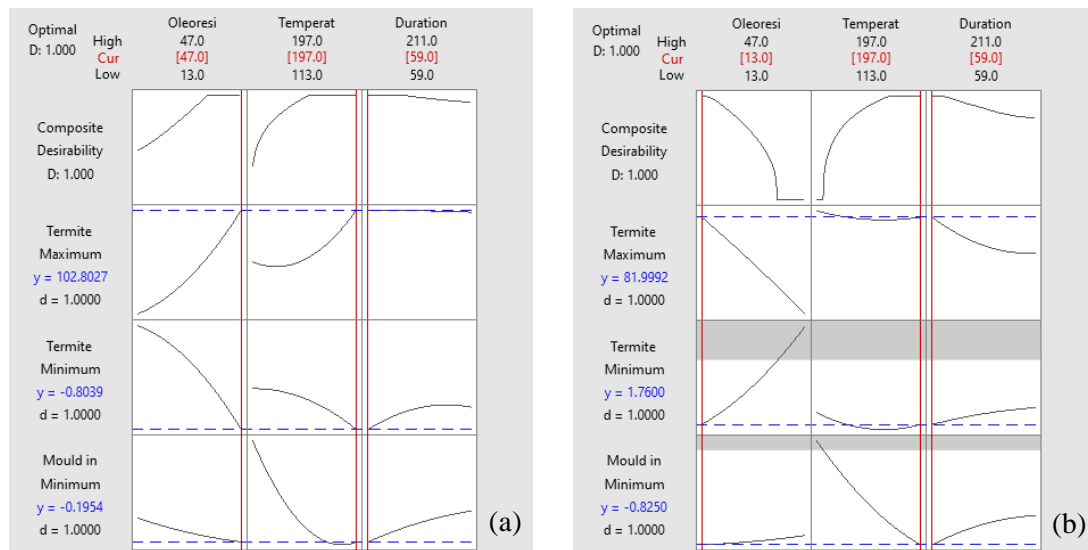
**Figure 8: Ryan-Joiner normality test results for the mould infection grade (a), termite damage grade (b) and termite mortality (c) of loblolly pine wood**



**Figure 9: Ryan-Joiner normality test results for the mould infection grade (a), termite damage grade (b) and termite mortality (c) of rubberwood**

### Optimal Treatment Conditions

Optimal treatment conditions were identified based on composite desirability, achieving values of 1.000 for both loblolly pine and rubberwood (Figure 10). For loblolly pine, these parameters were an oleoresin ratio of 47 %, a temperature of 197 °C, and a duration of 59 min. Under these conditions, mould infection is expected to be fully inhibited, with no termite damage and termites completely eradicated. For rubberwood, optimal parameters were determined as an oleoresin ratio of 13 %, a temperature of 197 °C, and a duration of 59 min. Under these conditions, mould infection is effectively prevented, although slight termite damage remains possible, with termite mortality estimated at approximately 82 %.



**Figure 10: Cross-sectional surfaces showing the corresponding values of mould infection grade, termite damage grade, and termite mortality for loblolly pine wood (a) and rubberwood (b) treated under optimal conditions**

### CONCLUSIONS

This study confirmed that heat treatment with a mixture of natural oleoresin and used cooking oil effectively enhances mould and termite resistance in loblolly pine wood and rubberwood. Loblolly pine wood exhibited greater overall improvement in biological resistance compared to rubberwood.

Optimal treatment for loblolly pine wood was an oleoresin ratio of 47 %, a temperature of 197 °C, and a duration of 59 min, predicting total inhibition of mould infection and termite damage, with termites completely eradicated. For rubberwood, optimal conditions were 13 % oleoresin, 197 °C, and 59 min, predicting effective mould prevention, although slight termite damage remains possible, with termite mortality estimated at approximately 82 %.

These findings highlight the potential of oleoresin heat treatment loblolly pine wood and rubberwood demonstrates strong resistance to mould and termite infestation, making it well-suited for applications such as flooring, garden furniture, and cladding, where enhanced biological resistance is essential.

### ACKNOWLEDGMENTS

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